DEWATERING AND SCALE DOWN OF SOLIDS RECOVERY IN INDUSTRIAL CENTRIFUGES

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ABSTRACT

The scale up of centrifugal operations has been carried out in the past using a combination of Σ theory applied to laboratory bottle centrifugation and expert experience. The aim of the work presented in this thesis is to scale down industrial centrifuges so that they can be used with small amounts of material to obtain process information at an early stage in process development. Practical work was carried out using real process systems such as homogenised yeast flocculated with polyethylenimine (PEI) to selectively remove contaminating lipids, nucleic acids and cell debris from a solution of soluble protein.

The separation capacity of a scroll decanter centrifuge is limited by solids conveyance, a turbulent settling zone and shear breakage. Rheological instrumentation was used to predict dewatering performance of biological sediments in a series of mass balanced pilot scale trials. Laboratory scale separations yield enough information to predict initial pilot scale trials using an operating line for the scroll decanter centrifuge where the viscoelastic properties of dewatered sediments are related to the turning force per revolution of the conveyor:

$$\log_{10} G \ast M_{\text{sed}} \propto \Delta N(T - T_p)$$

Operation is then constrained only by economic and machine limitations.

Scale down experiments revealed that shear of suspension in the feed zone creates permanent floc damage and also that the shearing action of the conveyor assists dewatering and contributes to softening of the sediment.

The separation performance of a disc stack centrifuge with a bowl volume of 0.6 L was found to be the same as for a scaled down disc stack centrifuge with a bowl volume of 3 L used in previous studies. 25% scale down of separation area in the smaller disc stack centrifuge was achieved according to Σ theory using a dilute suspension of polyvinylacetate. The position of the active discs was adjusted to compensate for variations in flow conditions across the stack using this robust system before applying the method to shear sensitive systems. Solids recovery of homogenised yeast flocculated with PEI was slightly higher in the scaled down stack due to the lower flow rates which reduce shear break up in the feed zone. Dewatering was over estimated in the scaled down stack due to longer sediment residence time.
Acknowledgements

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Mr John Betts, Mr Billy Doyle, Mr Derek Webb and all the workshop staff for their friendly technical assistance,

Ms Somaiya Siddiqi, Dr Josh King and Mr John Maybury for their humorous comments and patient help with my computer literacy,

and the Biotechnology and Biological Sciences Research Council for their financial support without which this project could not have taken place.
A man walks on the prairie. He does not move - there is no mark to measure his progress. In despair, he digs a hole with his heel, leaves it, and before a week reaches the far edge. "I dug a hole," he tells his friend, swallowing beer.

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<td>h</td>
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hs  depth of settling layer  m
hw  weir height  m
J  mass flux  kg m\(^{-2}\)
K  network modulus  Pa
k\(_1\)  constant
k\(_2\)  constant
k\(_3\)  constant
K\(_1\)  Dimensionless rate constant
K\(_2\)  constant
L  bowl length  m
L\(_c\)  length of scroll decanter centrifuge  m
m  mass  kg
M  Mass flow rate  kg s\(^{-1}\)
n  rotational speed  rev s\(^{-1}\)
\(\Delta n\)  conveyor differential speed  rev s\(^{-1}\)
N  power  W
N, n  Whole number
n\(_1\)  constant
n\(_2\)  constant
n\(_3\)  power law index
p  pitch  m
P  fractional resistance to penetration
P  Pressure  N m\(^{-2}\) or Pa
P  centrifugal pressure  N m\(^{-2}\) or Pa
q  single passage throughput  m\(^3\) s\(^{-1}\)
q\(_s\)  specific throughput capacity  m s\(^{-1}\)
Q  volumetric flow rate  m\(^3\) s\(^{-1}\)
Q\(_b\)  equivalent flow throughput in a bottle centrifuge
r  radial position of a settling particle  m
r'  radius to tip of conveyor  m
r\(_1\)  radius from axis of rotation to top of liquid  m
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<td>Volume</td>
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<td>v_r</td>
<td>Radial particle settling velocity</td>
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<td>Conveyor ribbon width</td>
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<td>Y</td>
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### Greek Symbols

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<tr>
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<td>screw angle</td>
<td>radians</td>
</tr>
<tr>
<td>γ</td>
<td>shear strain</td>
<td>m</td>
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<td>γ̇</td>
<td>shear (strain) rate</td>
<td>s⁻¹</td>
</tr>
<tr>
<td>δ</td>
<td>loss angle (phase shift, phase lag)</td>
<td>°</td>
</tr>
<tr>
<td>ε</td>
<td>solids path angle</td>
<td>radians</td>
</tr>
<tr>
<td>θ</td>
<td>half cone angle of the disc</td>
<td>radians</td>
</tr>
<tr>
<td>λ</td>
<td>dimensionless hydrodynamic parameter</td>
<td></td>
</tr>
<tr>
<td>π</td>
<td></td>
<td>3.142</td>
</tr>
<tr>
<td>μ</td>
<td>dynamic viscosity</td>
<td>Pa s</td>
</tr>
<tr>
<td>ρ</td>
<td>density</td>
<td>kg m⁻³</td>
</tr>
<tr>
<td>ρₐ</td>
<td>cell density</td>
<td>kg m⁻³</td>
</tr>
<tr>
<td>Δρ</td>
<td>solid-liquid density difference</td>
<td>kg m⁻³</td>
</tr>
<tr>
<td>σ</td>
<td>particle shape factor</td>
<td></td>
</tr>
<tr>
<td>Σ</td>
<td>Sigma factor</td>
<td>m²</td>
</tr>
<tr>
<td>τ</td>
<td>residence time</td>
<td>s</td>
</tr>
<tr>
<td>τ</td>
<td>shear stress</td>
<td>Pa</td>
</tr>
<tr>
<td>τᵧ</td>
<td>yield stress</td>
<td>Pa</td>
</tr>
<tr>
<td>ν</td>
<td>kinematic viscosity</td>
<td>m² s⁻¹</td>
</tr>
<tr>
<td>φ</td>
<td>cell or solids volume fraction</td>
<td></td>
</tr>
<tr>
<td>ψ</td>
<td>power law constant</td>
<td></td>
</tr>
<tr>
<td>ψ</td>
<td>constant</td>
<td></td>
</tr>
<tr>
<td>ω</td>
<td>angular (radial) velocity</td>
<td>rad s⁻¹</td>
</tr>
<tr>
<td>Δω</td>
<td>conveyor differential angular velocity</td>
<td>rad s⁻¹</td>
</tr>
</tbody>
</table>

### Subscripts

- **max**: maximum
- **min**: minimum
- **c**: critical
- **l**: liquid
- **s**: suspension
sed  sediment
sup  supernatant
o    initial (feed)
avc  average
f    fine particles
c    coarse particles
1. INTRODUCTION

1.1 Downstream Processing of Globular Proteins

Recent advances in biotechnology have led to an increase in worldwide production of biological products, particularly the globular proteins. These range from extremely high value and potent therapeutic proteins such as Factor VIII which is produced in very small quantities, to antibodies, subunit vaccines, industrial and diagnostic enzymes and food proteins such as soya protein isolate, which is produced relatively cheaply in large quantities. In addition, recombinant DNA techniques have opened up the prospect of many human proteins becoming available in quantity either for use as therapeutic agents or for research. The downstream processing and extraction of proteins is reviewed by Hoare and Dunnill (1989).

Extracellular products are suited to integrated fermentation and product extraction, such as perfusion culture of mammalian cells (Kearns 1990). Proteins may also be secreted into and protected by the periplasmic membrane, or their extraction may be enhanced by fusion with signal peptides (Flaschel and Friehs 1993). However, the majority of products to date are intracellular proteins (Hoare and Dunnill 1989), which may be cultured in recombinant microbial systems. The cells are disrupted as part of the recovery process.

1.1.1 Cell disruption

Intracellular proteins may be extracted into solution by sonication, selective membrane lysis or mechanical disruption in high speed bead mills, as reviewed in Beter et al (1988). However, the most widely used method for laboratory and large scale cell disruption is by high pressure homogenisation (Bonnerjea et al 1986, Scawen and Hammond 1986). The mechanisms for cell disruption in high pressure homogenisers are discussed in Keshavarz et al (1987). Hetherington et al (1971) put forward a kinetic model for the disruption of yeast cells based on the operating parameters of pressure, number of passes, temperature and cell concentration as follows:
\[
\log \left( \frac{R_{\text{max}}}{R_{\text{max}} - R} \right) = K_1 N
\]

where:

\( R_{\text{max}} \) = maximum protein available for release

\( = (96 \text{ mg/g packed yeast}) \)

\( R \) = protein release (mg/g yeast)

\( N \) = number of discrete passes

\( K_1 \) = dimensionless rate constant

\( = 0.23 \) for packed yeast homogenised at \(5^\circ\text{C}, 500\) barg.

\( K_1 \) may be rewritten \( K_1 = k_1 P^a \), i.e. it depends on pressure, where the exponent \( a \) indicates the degree of dependency on pressure.

\( a = 2.9 \) for \( S.\ cerevisiae \) disrupted at \(5^\circ\text{C}\).

The dependence on cell characteristics and process temperature is inherent in \( k_1 \).

The constants \( k_1 \) and \( a \) are specific to cell type, and the expression may only be applied to solutions of baker's yeast between 30\% and 60\% by volume. The use of such highly concentrated product streams reduces processing time and hence the risk of product degradation caused by the simultaneous release of proteolytic enzymes from the cell contents.

The kinetics of enzyme release and activity in high pressure homogenisers were shown to be first order with respect to the number of passes, \( N \). However, the rate of release depended on the location of the enzyme within the cell (Follows et al 1971). Membrane associated enzymes such as gramicidin synthetase in \( B.\ brevis \) may be prone to shear damage (Keshavarz et al 1987).

High pressure homogenisation releases lipids and nucleic acids into the product solution and it creates large amounts of cell debris which also contaminate the suspending medium. Cell debris is partially composed of sub-micron particles which form a colloidal suspension and are notoriously difficult to separate in conventional process equipment.
1.1.2 Primary separation

The physical separation of cell debris from protein solution may be carried out by process equipment such as centrifuges, filters or microfilters, none of which are wholly satisfactory and the relative merits of which are under constant review (Bentham 1989, Devereux et al 1984).

Filters tend to become fouled by cell debris or form ill-defined surface gels which become the active filtration component. Centrifuges tend to damage shear sensitive proteins at the air liquid interfaces of feed or collection zones and their scale up is unpredictable (Mackay and Salusbury 1988).

However, centrifuges have a number of relative advantages over filtration methods in that they are:

* versatile with respect to particle size
* able to achieve a high level of dewatering
* insensitive to variations in the product stream and foulants
* suited to large scale operation whilst maintaining a small process area "footprint".

For these reasons, centrifuges are the main object of study in this thesis, where the problem of predicting scale-up from laboratory to pilot scale is examined.

1.1.3 Flocculation to enhance separation

Brunner and Hemfort (1988) have reviewed protein extraction and put forward a generalised flowsheet for the recovery of intracellular proteins, shown in Figure 1.1.1 (a). If this route of product extraction is chosen, then the precipitating agents must be highly selective, affordable in adequate quantity and acceptable as process aids with respect to toxicity. Process aids may also be used to remove selectively contaminants such as colloidal cell debris from solution and thereby ease their recovery from the product suspension by the route shown in Figure 1.1.1 (b).

Bonnerjea et al (1988) showed how sodium borate solution enhanced the recovery of total protein and enzyme activity from yeast cell debris. Bentham et al (1990) applied the flocculation successfully at pilot scale in a scroll decanter
Figure 1.1.1: *Downstream methods for fermentation based intracellular products*

(a) Protein precipitation

```
Cell harvest → Cell disruption → Selective product precipitation → Purification
```

Precipitating agent

(b) Selective flocculation

```
Cell harvest → Cell disruption → Selective removal of contaminants → Purification
```

Flocculant
The mechanism of this flocculation is a specific reaction of the borate anion with the cis-diol mannose groups present in the cell walls of yeast, as illustrated in Figure 1.1.2.

![Figure 1.1.2: Interaction of the borate anion with 1,2 cis-diols at high diol to borate ratios to form spirine type complexes comprising two 5-membered ring structures.](image)

The flocculating agent polyethylenimine (PEI) is more generally applicable to a range of microorganisms (Salt et al., 1996), and whilst soluble proteins are retained, cell debris, lipids and nucleic acids are selectively removed from solution (Milburn et al., 1990). PEI is a highly branched water soluble polyamine of variable molecular weight. It acts as a cationic polyelectrolyte which is attracted to negatively charged organic solids (Horn, 1980). It flocculates cell debris by an electrostatic patch mechanism, while nucleic acids are removed as precipitates (Bulmer, 1993). This is illustrated in Figure 1.1.3.

![Figure 1.1.3: Polyethylenimine and mechanism of flocculation of cell debris particles.](image)
1.2 SCALE DOWN

Scale down is used in biotechnology for both process optimisation and design. Production trials based on laboratory tests should speed up the development stages of new processes. Some examples where scale down techniques have been used for process optimisation and design are discussed below, followed by some scale down theory.

1.2.1 Scale down for process optimisation

Process optimisation is concerned with making observations on the effects of changing process variables in the pilot plant or laboratory, and transferring the results to a production scale. For example, Bosnjak et al (1985) found that transferring growing fermentation broth (for oxytetracycline biosynthesis) directly from industrial to pilot (1000 L), and laboratory (10 L) fermenters, gave them a reliable comparison of results under different culture conditions. These scale down experiments led directly to improvement of the industrial process.

1.2.2 Scale down for process design

Scale down for design is used to solve scale up problems by theoretical analysis and small scale investigations.

Gooding (1991) has shown how laboratory scale batch membrane filter tests can be used to design industrial scale filtration units. He stressed the importance of carrying out mass balance closure analysis on batch lab data in order to eliminate erroneous results. The simplified equation for the mass balance applied to a single component (usually the solute) is:

$$ \rho \frac{d(VX)}{dt} = -JAY $$

where

- $\rho$ = density \hspace{1cm} (kg m$^{-3}$)
- $V$ = volume \hspace{1cm} (m$^3$)
- $t$ = time \hspace{1cm} (s)
- $J$ = mass flux \hspace{1cm} (kg m$^{-2}$)
- $A$ = membrane area \hspace{1cm} (m$^2$)
and

\[ X = \text{mass fraction solute in feed or retentate} \]
\[ Y = \text{mass fraction solute in permeate} \]

The aim of batch testing is to determine values for J, Y and X, which can be substituted into design equations for large-scale batch, fed-batch, once-through or feed-and-bleed membrane systems.

Gooding used simple system components to test his theory: the pervaporation of acetone and methanol from aqueous solution using a silicone rubber membrane. The possible adsorption of organics onto the membrane surface during a batch test was considered to have a negligible effect on the mass balance, but the effect on flux would be accounted for in the design. Gooding anticipated problems in using this method for enzyme systems, where loss in activity would not be apparent from mass balance data.

There are some major assumptions made in this design method which limit its applicability in practice: It is assumed that the membrane characteristics, degree of fouling, pressure, temperature and crossflow velocity will be duplicated in the larger model to give the same observed flux and separation factor as in laboratory tests. The emphasis on using scale down technique in this example is that it is a design tool, rather than a scale up protocol.

1.2.3 The principle of similarity

The principle of similarity is concerned with the relations between physical systems of different sizes, and it is thus fundamental to the scaling up or down of physical and chemical processes. It is concerned with discovering the ratios of magnitudes within the system which govern its spatial and temporal configuration irrespective of the measured scale.

There are four states of similarity as reviewed by Johnstone and Thring (1957): geometric, mechanical, thermal and chemical.

Geometric similarity is achieved when to every spatial point in one body there exists a corresponding point in the other. The points are linked by a scale ratio. If the scale ratio is not the same along every spatial axis then a distorted
body is the result. An example of a criterion for geometric similarity is maintaining constant length/diameter ratio in scroll decanter centrifuges.

Mechanical similarity is an extension of geometric similarity to stationary or moving bodies which are subjected to forces. It is divided up into static, kinematic and dynamic similarity. Static similarity is achieved when geometrically similar bodies undergo similar deformation under constant stress. Kinematic similarity is achieved when the particles within geometrically similar moving systems trace out geometrically similar paths in corresponding intervals of time. Dynamic similarity is achieved when the ratios of all corresponding forces are equal in geometrically similar moving systems.

Thermal similarity is concerned with systems in which there is a flow of heat. It is achieved when corresponding temperature differences are in a constant ratio. It requires geometric and kinematic similarity.

Chemical similarity is concerned with reacting systems in which composition varies. It is achieved when corresponding concentration differences are in a constant ratio. It requires geometric, thermal and kinematic similarity.

Similarity criteria are dimensionless quantities and may be derived by dimensional analysis provided that all the variables that govern the system are known. They may otherwise be derived from setting up differential equations around the system, or those of importance to a particular study may be selected by regime analysis.

1.2.4 Techniques for the application of the principle of similarity

There are a variety of known protocols for scale-up of processes which are listed in Sweere (1987) and include:

1. Fundamental method, such as using a rate equation where physical and chemical properties are fed into the design equation from literature or laboratory experiment.

2. Rules of thumb, such as either maintaining constant volumetric mass transfer coefficient \((k_a)\) or constant dissipated power per unit volume \((N/V)\) in scale up of fermentations limited by oxygen absorption.
3. Dimensional analysis:

Dimensional analysis begins with a review of the many variable parameters which can have a significant effect on, say, a fluid dynamics problem. In most instances these parameters can be grouped together in dimensionless groups, thus reducing the effective number of independent variables which the experimenter must examine. Dimensional analysis can lead to false conclusions if a significant variable is omitted from the problem, and alone gives no information about the forms of the functions which link individual parameters (Coulson and Richardson 1984).

Dimensionless numbers can be used to ensure that a scale up or down model is fundamentally similar to the original. This can lead to a compromising situation, where the least significant dimensional constraints are neglected to ease the demand for specialised experimental equipment.

4. Regime analysis:

Regime analysis is concerned with defining the most important mechanism of a scale up or down problem, rather than eliminating insignificant variables. For example, in a chemical reaction where the resistance to conversion is the rate-limiting step, then the system is subject to a chemical regime. If the controlling factor is dependent on the fluid dynamics of the system due to resistance to diffusion, then the regime is dynamic. Where there is a chemical regime, the scale up relation calls for chemical similarity; where there is a dynamic regime, for dynamic similarity.

Hence parameter sensitivity analysis is important in finding a characteristic parameter on which to base a scale down model. Sweere et al (1987) reviews the use of regime analysis based on characteristic times for scale down of microbial fermentations.

A certain amount of flexibility is apparent in the choice of theoretical approach to scale down projects. For example, Bolle et al (1987) used dimensional analysis to scale down an upflow anaerobic sludge blanket (UASB) reactor. They found that most of the fourteen dimensionless numbers relevant to flow in a 800 m³ pilot scale reactor could be neglected on scale down, which
was based on constant Froude number in order to control liquid flow and maintain the characteristic time for substrate consumption. Hence the effect of gravity on governing the reaction rate was the criterion upon which scale down was based and the ruling regime was dynamic.

The power of scale down experiments in this case is quite clear: a 0.12 m³ UASB model reactor was used to test fluctuating flows of influent to the sludge bed. Application of a fluctuating influent flow rate led directly to the improvement of reactor performance in a production scale UASB with a volume of 1340 m³.

1.2.5 Schemes for scale down

Oosterhuis (1985) summarised how regime analysis and scale down can be used to solve scale up problems in the following scheme:

<table>
<thead>
<tr>
<th>PRODUCTION SCALE</th>
<th>LABORATORY SCALE</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Regime analysis</td>
<td>4. Application</td>
</tr>
<tr>
<td>2. Simulation</td>
<td>3. Optimisation</td>
</tr>
</tbody>
</table>

In regime analysis, the first stage is to ascertain which is the ruling regime in a particular operation. There may be one (pure) or two (mixed) regimes, or even a change of regime during a time-related process or on scale-up. It is important to define a characteristic parameter on which to base laboratory scale experiments, especially before the construction of any specialised equipment.

Geometric similarity of the equipment simulating the real operation may not be required if the ruling regime is better modelled with an alternative geometry: the important criteria at this stage is that laboratory experiments must be representative of what goes on at production scale.

Optimisation of the process at laboratory scale must be translatable to production scale: any extrapolation of empirical or mechanistic models will be limited by practical constraints of large scale operation.

Sweere et al (1987) proposed an extension to the scheme where the first step in the scale down procedure not only includes regime analysis, but also
dimensional analysis, mechanistic analysis and the similarity principle. Complementary knowledge about the process was also supplied from rules of thumb, literature data, correlations and experience.

If all these considerations have been taken into account, then scale up is based upon the application of a reversal of the methods used to scale down. Further examples where scale down techniques have been applied are: gas-liquid flow in pipes (Chesters 1977), trickle flow reactors (Gierman 1988), chromatography (Naveh 1991, Hinrichsen 1985), testing of automated production software (Young et al 1984), and disc stack centrifuges (Mannweiler and Hoare 1992).

1.3 SCALE DOWN OF CENTRIFUGE OPERATIONS

There are several types of industrial centrifuge, which are reviewed in standard texts such as Perry (1986), Coulson and Richardson (1985), Svarovsky (1990), and the main types are listed in Table 1.3.1. Centrifuges are frequently selected on the basis of their solids handling capacity and ability to recover small particles as shown in Table 1.3.1 and Figure 1.3.1. Centrifuge modifications for biotechnology include bowl cooling, clean in place and steam sterilisation.

Table 1.3.1: Types of industrial centrifuge and their selection with respect to solids handling.

<table>
<thead>
<tr>
<th>Centrifugal separation equipment</th>
<th>Solids handling capacity (% v/v)*</th>
<th>Solids discharge type</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tubular</td>
<td>&lt;5</td>
<td>manual</td>
</tr>
<tr>
<td>Multichamber</td>
<td>&lt;5</td>
<td>manual</td>
</tr>
<tr>
<td>Scroll decanter</td>
<td>15-60</td>
<td>continuous</td>
</tr>
<tr>
<td>Disc stack</td>
<td>Solids ejecting</td>
<td>0-10</td>
</tr>
<tr>
<td>Nozzle</td>
<td>10-30</td>
<td>continuous</td>
</tr>
</tbody>
</table>

*Data taken from Brunner and Hemfort (1988)
The centrifuges examined in this study can be operated with continuous throughput. The intermittent solids discharge disc stack centrifuge is a high capacity clarifier, which is able to recover small particles. The scroll decanter centrifuge can handle high feed solids concentrations and discharges highly dewatered solids. An understanding of the parameters which are likely to govern their scale down may be found from established scale up procedures and flow path information found in the relevant literature.

### 1.3.1 Scale up or down using sigma theory

The sigma value concept first proposed by Ambler (1952) is used for predicting separation characteristics in industrial centrifuges.

In general:

\[ Q = 2u_g \Sigma \]  \hspace{1cm} (3)

Where

- \( Q \) = volumetric flow rate \( (m^3 \text{s}^{-1}) \)
- \( u_g \) = terminal velocity of a solid particle settling under gravity through a liquid \( (m \text{s}^{-1}) \)
- \( \Sigma \) = sigma factor \( (m^2) \)

Terminal settling velocity \( (u_g) \) is found from Stoke's law for a free particle moving through a liquid under ideal settling conditions (section 3). \( \Sigma \) is the
factor by which centrifugal acceleration increases settling capacity relative to the area of a gravity settling tank.

When scaling up or down with the sigma value, it is assumed that $u_g$ remains constant in geometrically similar centrifuges, and that separation is a function of flow rate capacity and equivalent settling area alone:

$$\frac{Q_1}{\Sigma_1} = \frac{Q_2}{\Sigma_2}$$

Large discrepancies between prediction and practice using this method are usually attributed to deviations from Stoke's law due to changes in the degree of turbulence, or a change in the break-up of aggregated particles due to shear.

Efficiency factors reduce $\Sigma$ on scale up, and vary in magnitude according to the extent of departure from idealised sedimentation in each type of centrifuge.

The efficiency factor for scaling up bottle centrifuges used for laboratory analysis is 90-100% (Perry 1986). However, in scale up of industrial centrifuges such as tubular bowls, there is a deterioration in performance with an efficiency factor of 80% (Perry 1986). For disc stack centrifuges, efficiency factors vary from 73% for a dilute monodisperse suspension (Murkes and Carlsson 1978) to 55% (Ambler 1959/1961 and Frampton 1963), 45% (Morris 1966) and 40% (Purchas 1981). The reduction in effective clarification area on scale up of helical conveyors varies from 54%-67% (Ambler 1959, Morris 1966, Sokolov 1971) depending on the volume occupied by sediment. However, in this case scale up is also limited by the ability to convey solids. The magnitude of the efficiency factor becomes more pronounced in larger centrifuges. The problem faced in scale down to a small centrifuge is how to mimic the performance of a large machine without being unduly optimistic.

Disc stack centrifuges have also been scaled up using "KQ" value which is inversely proportional to $u_g$ (Sullivan and Erikson 1961). K is found from empirical measurements on a bowl with known geometry and particle-related constants.
1.3.2 Disc stack centrifuge: flow path considerations

The discs in disc stack centrifuges split the incoming liquid into thin layers. The flow between the discs becomes laminar and the settling distance is drastically reduced, which makes particle settling more rapid.

The liquid is distributed evenly between the discs (Carlsson 1979, Rachitskii and Skvortsov 1980)). The velocity distribution across the disc spacing depends on a dimensionless hydrodynamic parameter \( \lambda \) (Sokolov 1971, Bohman 1974, Brunner and Molerus 1979, Carlsson 1979):

\[
\lambda = h \sqrt{\frac{\omega \sin \theta}{v}}
\]

Where:
- \( h \) = disc spacing (m)
- \( \omega \) = angular velocity of the bowl (rad s\(^{-1}\))
- \( v \) = kinematic viscosity (m\(^2\) s\(^{-1}\))
- \( \theta \) = half cone angle of the disc (radians)

If \( \lambda < \pi \), then the velocity profile is parabolic in both radial and circumferential directions. If \( \pi < \lambda < 2\pi \), then the velocity in the radial direction is at a minimum in the centre of the disc gap. If \( \lambda > 2\pi \), then there is a radial back-flow and circumferential velocity can reach \( \lambda \) times the average radial velocity in the middle of the disc spacing.

Reynolds numbers calculated from the circumferential velocity increase with increasing \( \lambda \), and transition to turbulence can take place. The reason for this behaviour is due to interplay between Coriolis, friction and centripetal forces.

There may be an uneven distribution of particles entering the disc stack when coarse particles are separated out in the outer disc space (Skvortsov 1984).

This additional clarification provided by the sediment holding region has no beneficial effect where feed enters the disc stack via feed channels, as shown in Figure 1.3.2.

The role of feed channels is to prevent the back mixing of solids sliding down the disc wall into the sediment holding space. There is a reduction in the active separation area using this arrangement, but the intention is that this is
compensated for by reduced re-entrainment of sedimented solids flowing across
the incoming feed. However, around each distribution hole a vortex is formed
due to Coriolis forces. Sokolov (1971) found that the vortex size increases with
bowl speed and feed flow rate. Willus and Fitch (1973) used a rotating camera
to observe the flow patterns inside a disc stack centrifuge, and found that these
vortices occupied a substantial part of the separation area, and were responsible
for the re-entrainment of coarse particles.

Brunner and Molerus (1979) discovered that long radial spacer ribs
(caulks) between discs had a beneficial effect on particle recovery relative to
point spacers. They used dye tracer experiments observed through a transparent
bowl lid to observe flow patterns in both configurations, as shown in Figure
1.3.3. They found that radial spacer ribs suppressed both flow in the
circumferential direction and also vortex formation. Hence they had a beneficial
effect with respect to suppressing transition from laminar to turbulent flow.
The flow velocity at which the transition from regular vortices to turbulence
occurred was used to calculate a critical rotation number (Rossby number, Ro)
for a variety of equipment settings.

\[ Ro = \frac{u_{ave}}{R_o \omega} \]  

where \( u_{ave} = \) average velocity between discs calculated at outer disc
radius \( (\text{ms}^{-1}) \)
\( R_o = \) outer disc radius \( (\text{m}) \)

Ro is a dimensionless number which when plotted against the hydrodynamic
parameter \( \lambda \) produced critical curves for centrifuge operation as shown in Figure
1.3.4. This figure shows that with ribbed spacers relatively high throughputs
could be achieved before transition to turbulence than with point spacers. Hence
industrial centrifuges are designed to operate at throughputs below the critical
curve and typically at \( \lambda \) values of 5-28, in order to maintain laminar flow
characteristics (Axelsson 1985).

Sokolov and Dolzhanova (1972) showed that disc stack centrifuges with
feed channels and spacer ribs have asymmetrical solids loading at entry to the
discs. This is due to Coriolis forces which cause the net flow at the leading side of a caulk (with respect to the direction of rotation) to be lower than that on the following side. Hence particles settling on the leading side are subjected to a higher throughput than those on the following side, and are less likely to settle out. This phenomenon contributes to the departure from ideal settling according to Stoke's law.

1.3.3 Hindered settling and scale up

The above discussion only applies to dilute particle suspensions. There are further deviations from ideal settling due to operation at high solids concentrations.

For solids concentrations > 2% (v/v), the free settling velocity of a particle under gravity is hindered by interactions with other particles and the suspending liquid. The reduction in settling velocity increases with solids concentration (Muschelknautz 1987). According to Richardson and Zaki (1954):

\[ u_g^* = u_g(l - C_v)^\sigma \]

where
- \( u_g^* \) = hindered settling velocity (m/s)
- \( C_v \) = volume concentration of solids in suspension (m^3/m^3)
- \( \sigma \) = particle shape factor

For rigid, spherical particles \( \sigma = 4.6 \), but it can increase to 10-100 for non-rigid or irregular particles. This can lead to large errors where Stoke's law is assumed (Datar and Rosen 1987). For flocculated suspensions of yeast, \( \sigma = 12-20 \) (Brohan and MâLoughlin 1984).

Barnea and Mizrahi (1973) made further reductions to \( u_g^* \) by taking into account wall effects where the vessel diameter is only 1-2 orders of magnitude larger than the particle size. However, centrifugal sedimentation at high solids concentrations is not hindered as much as in gravity settling due to the continuous dilution effect of the feed throughput (Svarovsky 1990).
Figure 1.3.2: *The role of feed channels in a disc stack centrifuge.*

Figure 1.3.3: *Fluid flow within the gap of a disc stack centrifuge.*

Figure 1.3.4: *Critical Rotation number Ro as a function of the hydrodynamic parameter λ in a disc stack centrifuge.*

where
- ○ critical values in the free gap (point spacers)
- ● critical values in the free gap with 8 rib spacers
With Σ theory scale up, corrections for hindered settling can be produced simply by substituting the reduced settling velocity expression into the derivation for critical particle diameter, $d_c$ (see Section 3.2) to obtain:

$$d_c = \sqrt{\frac{18\mu Q}{\Delta \rho A_e g (1 - C_s)^2}}$$

where
- $m$ = dynamic viscosity (kg m$^{-1}$ s$^{-1}$)
- $\Delta \rho$ = solid-liquid density difference (kg m$^{-3}$)
- $A_e$ = equivalent settling area (m$^2$)
- $g$ = acceleration due to gravity (m s$^{-2}$)

Grade efficiency curves (Section 3.2) for hindered settling can in theory be superimposed onto those for unhindered settling using this method. One of the difficulties to overcome in the application of hindered settling theory to centrifugal separation of biological solids is the often poorly defined density difference between suspending liquid and solid particles, as in the case of cell debris.

### 1.3.4 Scale down of the disc stack centrifuge

Work was carried out by Obeng (1983) on a small, scaled down disc stack centrifuge (Westfalia SA00H-205) to investigate if it could be used to predict the under-run capabilities of a larger disc stack by reducing its separation area. The positions of the active discs were found to have a profound impact on the separation.

Obeng used two sets of active disc arrangements: 6 active discs at the top of the stack, and 6 active discs at the bottom. 53 blanking discs, ie. with no separator caulks, were used to replace the equivalent of 32 missing active discs. Breakthrough curves were plotted using turbidity measurements for a 6.8% soya protein precipitate at flow rates 9 - 30.6 Lh$^{-1}$ for both arrangements.

With the active discs placed at the top of the stack, initial clarification was high at all flow rates. Particle breakthrough into the supernatant was observed after the equivalent of approximately 2 total bowl volume changes at all flow rates, although turbidity of the low flow rate stream remained half that
of the high flow rate and occurred less suddenly. Active discs placed at the bottom of the stack gave poor initial clarification, which declined further during 2.5 total bowl volume changes. The same result was observed at all flow rates.

An explanation was offered relating to fluid flow patterns inside the centrifuge. Improved separation observed in the active top stack arrangement was thought to be due to a large contribution to separation in the solids hold up region of the centrifuge bowl.

The solids dewatering in the top stack arrangement was 51-52 % dwt/wwt, compared to 43-44 % in the bottom stack arrangement. It was thought that this corresponded to a significant amount of recovery occurring in the sediment holding space. However, the total run time and hence solids residence time was nearly twice as long in the experiments with the top stack arrangement and is therefore more likely to have been the real cause for the increased dewatering.

This work preceded that of Mannweiler (1990), who used a larger disc stack centrifuge, and found that active discs placed near the bottom of the stack gave comparable performance to a separator with a complete set of active discs.

Mannweiler (1990) based his scale down method on observations of particle separation efficiency of the clarified effluent. He used a disc stack centrifuge with a bowl volume of 3L normally fitted with 72 active discs. Using an assortment of "blanks", ie. solid disc blocks which obstruct fluid flow, he reduced the active settling area by up to 90%.

Comparisons of the grade efficiency curves obtained in the scaled down and fully active stacks showed that separation was reproducible at all flow rates if the active discs were placed at a height equivalent to 7 active discs from the bottom of the stack. This corresponds to a position approximately 1/10 th of the way up the full stack. Active discs placed below this position in close proximity to the inlet stream were subject to turbulent flow, which disturbed the flow of solids down the discs. Hence solids re-suspension occurred, causing uncharacteristically poor separation, especially at high flow rates. Active discs placed too high gave rise to better separation than that observed in the fully
active stack, due to pre-settling in the sediment holding region, i.e. the centrifuge behaved as a low capacity tubular bowl.

Dye tracer studies revealed that the fluid volume required for nearly all the tracer material to be removed, \( V_{99} \), was 8.2 L, irrespective of the active discs’ position. However, experiments were carried out using 15 L feed. This was equivalent to 5 bowl volume changes required to reach steady state according to a rule of thumb for continuous stirred tank reactors (CSTR). However, 15 L was more than 10 bowl volume changes if the volume occupied by the blank discs is taken into account.

Since scale down of the process stream was based on bowl volume, then there was no reduction in the amount of test material actually required for each experiment. Any saving of process material was therefore made simply by the use of lower flow rates in the scaled down stack contributing to the ease of operation.

Comparisons of performance were made in terms of separation efficiency between pilot and scaled down stacks on the basis of the particle size distribution of the clarified supernatant relative to that in the feed. The full separation effect, including the extent of dewatering and the ability to discharge solids cannot be predicted using this method. For example, Milburn et al (1990) found that yeast cell homogenate flocculated with PEI gave good initial clarification in a disc stack centrifuge, but that blockage of the discharge nozzle due to the rheological nature of the dewatered solids hindered continuous operation.

1.3.5 Scroll decanter centrifuge: flow path considerations

Existing scale up strategies are based on a plug flow of the liquid phase through the bowl of the centrifuge from the feed zone to the outlet weir. Σ theory is essentially a plug-flow theory where all particles are assumed to have equal rotational speed. However, it is more likely that the particles never do attain this speed, and are continually being accelerated across a turbulent flow path. This can be illustrated by considering the energy distribution and flow profiles within the machine.
1.3.5.1 Energy considerations

Gosele (1980) introduced the concept that half the energy supplied by the motor to the decanter for accelerating the fluid to the outlet velocity is dissipated within the centrifuge. This can be explained by considering the energy balance across the centrifuge.

At the inlet pipe, a unit of feed has negligible rotational velocity and its total energy is zero compared to the unit at the overflow weir, which rotates with a tangential velocity proportional to the rotational velocity of the bowl. The kinetic energy \( E_k = \frac{1}{2}m u^2 \) of this unit together with its associated dissipated energy \( E_d \) are equal to the total energy \( E = mu^2 \) supplied by the motor. Hence the dissipated energy is equivalent to half the total:

\[
E_d = \frac{1}{2}m u^2 = \frac{1}{2} \rho_s \omega r_1^2
\]

where
- \( m \) = mass (kg)
- \( u \) = velocity (m s\(^{-1}\))
- \( \rho_s \) = suspension density (kg m\(^{-3}\))
- \( r_1 \) = radius to liquid suspension (m)

It immediately becomes clear that any theories based on a simple flow concept such as Stoke's settling velocity, are likely to give very misleading results on scale up.

Gosele stated that the dissipated power was identical to the energy dissipated during the acceleration of the slurry, and that the friction of the conveyor was comparatively small. In their experiments relating floc break up to dissipated power, Bell and Brunner (1983) assumed that all the power was dissipated within the volume occupied by the feed zone. However, Madsen (1989) proposed that the dissipated energy was not restricted to the inlet zone, but was distributed throughout the bowl. Scroll friction played a significant role in redistribution of the dissipated energy.

1.3.5.2 Observations on flow profiles

Zeitsch (1978) predicted that the exchange of momentum between feed entering the bowl and the charge already rotating would cause the liquid surface
to lag behind the bowl by 3-10\% of the angular velocity and that this would affect the separation capacity of the machine.

Faust and Gosele (1986) observed flow profiles in a short, transparent bowl decanter centrifuge rotating at low speeds (equivalent to 80-700g) with tracer dye. They noted that in a solid scroll conveyor rotating faster than the bowl, then feed was forced into the conical section of the bowl. Supernatant flowed between the scroll blades in order to reach the overflow weir, and was therefore forced to rotate slightly faster and in the opposite direction to the conveyor. The result was the formation of a stratified liquid ring, in which the outer half rotated nearly as a rigid cylinder, and the fast-flowing inner half was exposed to a relatively higher gravitational acceleration.

With the same conveyor rotating slower than the bowl, then an extremely stable stratification was observed, with a deeper outer cylinder of liquid rotating with the bowl. The inner liquid layer occupied a quarter of the pond depth in this instance and flowed immediately to the overflow weir. Although there was less turbulence in the slowly rotating scroll, better clarification in the faster scroll was thought to be due to the greater effective centrifugal force on the flowing layer.

A conveyor running faster than the bowl is the normal case for centrifuges fitted with cyclo gear boxes, and a conveyor running slower than the bowl is the case for planetary gear boxes.

In either configuration, turbulence caused by the impact of a poorly accelerated feed was restricted to a small area around the feed zone, without penetration into the deeper liquid layers. Instead, the low kinetic energy of the incoming feed caused it to spread on the surface of the inner layer as a thin film.

Like Faust and Gosele, B.Madsen (1989) observed flow profiles in a scroll decanter centrifuge rotating at low speed (equivalent to 28g), but with a relatively longer, transparent bowl. Similarly, he noted that the poorly accelerated feed was transported axially away from the inlet zone at the surface in the channel, at high velocities relative to the conveyor flights. However, with a conveyor rotating slower than the bowl, frictional forces at the liquid flights...
continuously reduced flow velocities along the length of the bowl towards the liquid outlet. The result was that the depth of the flowing area increased, and was counterbalanced by a return flow area underneath the surface flow.

Madsen and Madsen (1989) carried out separation experiments with dilute suspensions of kaolin in a range of scroll decanter geometries, and compared the results by means of grade efficiency curves. They found that scroll decanter centrifuges with high (L/D) values gave consistently better separation than could have been expected due to the increased settling area alone. They also showed results which indicated that increasing conveyor pitch and hence the total contribution of flight friction as well as path length and liquid velocity also improved clarification.

Madsen proposed that the separation capacity dependence on length was not linear because the velocity profile was continuously developing throughout the screw channel. Only in very long bowls was the velocity profile seen to approach plug flow and ideal settling conditions as illustrated in Figure 1.3.5. This explained why $\Sigma$ theory, which is derived from a constant velocity profile, fails to predict separation capacity, particularly in short decanters.

![Figure 1.3.5: Tangential velocity profiles of fluid relative to the conveyor flights in a scroll decanter centrifuge.](image)

In Figure 1.3.5, the directions of the radial velocities are indicated with arrows. The relative bowl length of the P600 centrifuge used in this practical study is also shown (see section 5.9.1).
1.3.6 Scale up limited by clarification in the scroll decanter centrifuge

1.3.6.1 Drag effect theory

Stahl and Langeloh (1984) noticed that at low feed flow rates in a scroll decanter centrifuge, clarification efficiency was highly reproducible in a variety of test systems, and decreased linearly with increasing throughput. However, at a break-point flow rate, which they termed the critical flow rate \( Q_c \), the separation effect declined rapidly. The permissible flow rate before reaching \( Q_c \) could be raised disproportionately by increasing the pond depth. This effect had been observed in many centrifuge geometries and scales.

An explanation according to drag theory was proposed: when \( Q > Q_c \), then shear stress in the liquid at the surface of the deposited solids layer causes settled particles to become entrained in the liquid, and to migrate along the solid surface to the discharge weir. A particle is restrained only by its friction on the sediment surface. At the throughput limit \( (Q_c) \), drag force just overcomes frictional force and the particle is swept away. Stahl and Langeloh likened the drag effect to the rolling of pebbles along the surface of a river bed. A friction coefficient characteristic of the process material can only be derived from experimental data where the particle size distribution is known.

In solid conveyors, the liquid flows at high velocity down a long path through the screw blades. Faust and Gosele (1986) suggested that a conveyor design where the liquid flows along a short axial path, as in a ribbon screw conveyor, might help to limit drag effects by reducing surface velocity without shortening the residence time of liquid in the centrifuge.

Stahl and Langeloh (1984) observed that relatively high solids loading capacity was frequently feasible in larger decanters operating at the same centrifugal acceleration on scale up. This was in proportion to the cube of the bowl diameter, which explains why a scale up "rule of thumb" exists where the liquid volume of the drum is used as a reference value for throughput. It also explains why it is not always possible to pre-test the area loading capacity of large machines: If \( Q_c \) is \( d^3 \) times larger for the same centrifugal field at full scale, then using \( \Sigma \) theory on a pilot scale, this flow rate will not fall within the \( Q < Q_c \).
range, and only scattered clarification data will be collected. Reif, Stahl and Langeloh (1990) give an empirical formula for determining whether \( Q_c \) is exceeded on scale up.

### 1.3.6.2 Scale up using sigma theory

Bowl length appears in all the expressions for \( A_e \), and so \( \Sigma \) theory predicts that increasing \( L/D \) ratio improves separation. This is what prompted the studies by Madsen and Madsen (1989) on slender decanter centrifuges with high clarification capacity: hence existing scale-up strategies differ for long and short scroll decanters.

Gosele (1980) proposed that for a given separation process operating satisfactorily at pilot scale, then \( A_e \) was unimportant if the only requirement was to specify appropriate centrifuge size and speed for scale up. For equivalent sedimentation in geometrically similar centrifuges the settling area (size, bowl diameter \( D \)) and gravitational acceleration (rotational speed, \( n \)) ratios are as given below:

\[
\frac{A_{e1}}{A_{e2}} = \left( \frac{D_1}{D_2} \right)^2 \quad \text{and} \quad \frac{g_{e1}}{g_{e2}} = \left( \frac{n_1}{n_2} \right)^2 \frac{D_1}{D_2}
\]

hence:

\[
\frac{\Sigma_1}{\Sigma_2} = \left( \frac{D_1}{D_2} \right)^3 \left( \frac{n_1}{n_2} \right)^2 = \frac{Q_1}{Q_2} = \frac{M_1}{M_2} \quad \text{for constant } \rho
\]

where

- \( g_c \) = relative centrifugal acceleration
- \( n \) = rotational speed (rpm)
- \( D \) = bowl diameter (m)
- \( M \) = mass flow rate (kg s\(^{-1}\))

This follows standard settling theory, where the same separation effect with respect to clarification can be achieved on scale-up by keeping the ratio of the sigma values equal to the ratio of feed flow rates.
1.3.7 Influence of dissipated power on separation: shear break up of flocculated suspensions

From Gosele's (1980) observations that separation was dependent on the energy dissipated within the centrifuge, he proposed that it was necessary to maintain constant power per unit volume (N/V) on scale up:

\[ \frac{N}{V} = \frac{1}{2} \frac{M u^2}{V} \]

hence:

\[ \frac{N}{V_i} = \left( \frac{M_1}{M_2} \right) \left( \frac{u_1}{u_2} \right)^2 \left( \frac{V_i}{V_2} \right)^{1/2} \] (13)

In geometrically similar centrifuges:

\[ \frac{V_i}{V_2} = \left( \frac{D_1}{D_2} \right)^3 \] (14)

and for constant power per unit volume:

\[ \left( \frac{\frac{N}{V}}{\frac{N}{V_2}} \right)_1 = 1 \] (15)

substituting into equation 13 and rearranging:

\[ \frac{M_1}{M_2} = \frac{D_1}{D_2} \left( \frac{n_1}{n_2} \right)^2 \] (16)

Thus the condition for maintaining constant dissipated power per unit volume on scale-up follows a trend directly opposed to that for ideal sedimentation using sigma theory (see equation 11).

Bell and Brunner (1983) conducted experiments with a flocculated suspension of PVAc which showed that a change in fluid volume available for power dissipation had a significant effect on separation efficiency. They confirmed that the number of newly formed (smaller) flocs was proportional to the square root of the dissipated power per unit volume of feed zone.
1.3.8 Scale up with both equal ideal sedimentation and equal power per unit volume

Gosele (1980) combined the power per unit volume and Σ theory scale-up methods to predict the separation capacity of a large scale machine for a flocculated suspension. By examining the constraints on both conditions he found that for both equal ideal sedimentation and equal power dissipation, then the centrifugal field must be kept constant on scale up, and the ratio of flow rates should be equivalent to the ratio of settling areas:

\[ 1 = \frac{D_i}{D_f} \left( \frac{n_i}{n_f} \right)^2 = \frac{g_{e_1}}{g_{e_2}} \text{ and } \frac{M_i}{M_f} = \left( \frac{D_i}{D_f} \right)^2 \]

Gosele called these scale-up constraints the "preferred set of operating parameters". He found that these conditions gave satisfactory scale up for systems where particle size distributions were constant and where centrifuge operation was not limited by solids transport.

However, Gosele showed that where there is significant reagglomeration of particles broken by shear on entry to the centrifuge, then residence time of the liquid layer must be kept constant on scale up. In this case, the flow rate ratio is proportional to the bowl volume ratio:

\[ \frac{M_i}{M_f} = \left( \frac{D_i}{D_f} \right)^3 \]

1.3.9 Scale up limited by solids transport in the scroll decanter centrifuge

Wiesmann and Binder (1982) stated that centrifuge capacity can be limited by solids transport as well as clarification. For equal values of the mean residence time of thickened solids and the conveying rate, all solids are just discharged by the screw:

\[ \frac{V_{sed}}{Q_{sed}} = \frac{L}{\Delta \omega \rho} \]

where

- \( V_{sed} \) = sediment
- \( Q_{sed} \) = conveyor differential angular velocity (rad s\(^{-1}\))
\( p \) = pitch of conveyor blades \((m)\)

For maximum dewatering:

\[ Q_{\text{sed}} C_{\text{sed}} = Q_{\text{sed}} C_{\text{max}} \]  \hspace{1cm} (19)

where \( C_{\text{sed}} \) = solids concentration of sediment \((\text{kg}\ \text{m}^{-3})\)

\( C_{\text{max}} \) = maximum solids concentration \((\text{kg}\ \text{m}^{-3})\)

and assuming that all the material in the bowl of the centrifuge which is in contact with the scroll to the level of the pond is conveyed:

\[ V_{\text{sed}} = (r' - r_1) 2\pi R L \]  \hspace{1cm} (20)

where \( r' \) = radius to tip of conveyor \((m)\)

\( R \) = bowl radius \((m)\)

then:

\[ Q_{\text{sed}} C_{\text{sed}} = (r' - r_1) 2\pi R \Delta \omega p C_{\text{sed,max}} \]  \hspace{1cm} (21)

If \( Q_{\text{sed}} C_{\text{sed}} \) exceeds the above condition, then the machine is overloaded and solids are discharged with the clarified liquor. If the machine is under-loaded due to a high rate of conveyance or low feed concentration, then more liquid is conveyed by the screw and a lower solids discharge concentration is obtained.

Wiesmann and Binder assumed that \( C_{\text{max}} \) was unknown at pilot scale and the appropriate flow rate of thickened solids and scroll differential speed would be determined with experiments near maximum load. Equation (21) could then be used to scale up from a successfully operating pilot to a production scale machine:

\[ \frac{Q_{\text{sed}}}{(r'_1 - r_1) R_1 \Delta \omega_1 p_1} = \frac{Q_{\text{sed}}}{(r'_2 - r_1) R_2 \Delta \omega_2 p_2} \]  \hspace{1cm} (22)

This method assumes total geometric similarity on scale up, including proportional sludge depth ratios. However, the settling layer is thinner in the smaller centrifuge and so the same centrifugal field does not produce the same compressive pressure. Hence the dewatering of compressible solids cannot be predicted.
The solids path in the equation is based on a linear movement of solids in an axial direction in the centrifuge with 100% scrolling efficiency. However, force balances carried out by Records (1974) show that this is not the case, since even with maximum scrolling efficiency, the solids path would follow the direction of the screw angle.

Reif and Stahl (1989) visualised transport of sediment up the cone of a scroll decanter centrifuge as having to take place in two distinct layers: a totally saturated film which becomes linearly smaller towards the cone, and a portion which is dewatered to the product-specific equilibrium saturation. The trend of the liquid film height along the cone remains constant at steady state.

At low feed flow rates, the bulk height remains low, and equilibrium saturation may be obtained. As the mass throughput increases, the saturation also tends to increase, until it reaches a point at which the dewatering capacity of the product is exceeded, and under these conditions, saturated liquor is transported up the cone to discharge with the solids. The limiting mass throughput is a function of the machine geometry, operating conditions and friction coefficient of the sediment, and must be determined by experiment.

Reif and Stahl suggested that dewatering in the cone would not be affected by changes in scroll differential speed, since although the residence time of solids increases with decreasing scroll differential, the bulk height simultaneously increases. However, Ward (1989) showed that this is not the case for *Fusarium graminearum*, where dewatering is highly dependent on scroll differential speed.

1.3.9.1 Scale up for compressible solids

Gosele (1980) showed that where solids such as activated sludge dewater by compression (section 1.4.2) then the scale up requirement is to maintain constant centrifugal pressure. A settling layer of thickness $h_s$, rotating at a mean radius $r$ produces a pressure $P = h_s r (2\pi n)^2$. The ratio of pressures in the two centrifuges is:
If the two machines are geometrically similar then \( h_1/h_2 = r_1/r_2 = d_1/d_2 \) and the pressures are equal \( (P_1/P_2 = 1) \) if:

\[
\frac{n_1 \, D_1}{n_2 \, D_2} = 1
\]

Hence the same compression can be obtained in geometrically similar centrifuges by running them at the same peripheral velocity.

1.3.9.2 Scale up with scrolling torque

Records (1974) found that the rate at which solids were transported and the turning force required to transport them could limit centrifuge capacity. This turning force or scrolling torque is \((T-T_0)\) where \(T\) is the torque output, and \(T_0\) is the residual or heel torque in the absence of solids. Scrolling torque was found to be proportional to the solids mass transport rate and to the differential speed of the conveyor.

The following criteria are used for scale up where solids transport limits the machine capacity:

\[
\frac{M_2}{M_1} = \frac{(T-T_0) \, g \, \Delta n \, D_2 \, \Psi_1}{(T-T_0) \, g \, \Delta n \, D_1 \, \Psi_2}
\]

where \( \Delta n \) = conveyor differential speed \((\text{rev s}^{-1})\)

\( \Psi \) = a dimensionless constant

\( \Psi \) is a function of the wet or dry beach configuration, or pond depth in machines fitted with a negative ring dam. It is highly dependent on beach (cone) angle and scrolling efficiency of a particular solid sludge. \( \Psi \) factors are known only to the associated manufacturers (Alfa-Laval Sharples), who claim to have found empirical values of \( \Psi \) for a variety of industrial sludges.

Each of the scale up methods outlined so far deal with problems of scale up from an already well-defined pilot scale trial. Very little work has been
carried out on predicting what would happen in a pilot scale trial from laboratory bench studies.

1.3.10 Scale down of the scroll decanter centrifuge

At present it is usual to carry out extensive tests on pilot scale centrifuge equipment with potential for a particular application at production scale: tests which may be carried out as a service by the centrifuge manufacturers themselves. This is only feasible when process design and development can supply large volumes of sample material at low cost, for example in waste water treatment. Alfa-Laval Sharples regularly carry out such tests on a pilot decanter centrifuge, typically using 100-200 L material to optimise each operation. The advent of high value, low volume biotechnological products demands a method for predicting centrifuge performance using very small amounts of material.

Reif and Stahl (1988) have published initial studies where the friction coefficient of moist bulk solids is determined in a small experimental rig. The coefficient is obtained by measuring the torque generated by a sample of material held in a rotating wedge by a scraper device. The method has been applied to glass beads and PVAc with the intention of investigating centrifugal effects on the friction coefficient and scaling up to an industrial centrifuge.

Vesilind (1974) proposed that process design based on laboratory tests must mimic both the liquid retention time and the gravitational acceleration in a prototype centrifuge. Tests would have to be devised which examined if a suspension would clarify at a particular relative centrifugal force (RCF), and to estimate how well sludge would be moved out of the bowl.

Solids recovery in a pilot scale machine can be described with:

\[
\% \text{ solids recovery} = \left[ \frac{C_o - C_{\text{sup}}}{C_o} \right] \times 100\% \tag{26}
\]

where:

- \( C_o \) = feed solids concentration
- \( C_{\text{sup}} \) = supernatant solids concentration

Solids recovery in the laboratory tests was found for a variety of residence times at different centrifugal accelerations. Using a bottle centrifuge, 15 mL samples
of process feed material were spun at centrifugal accelerations equivalent to a prototype industrial centrifuge, as defined by:

\[ RCF = \left( \frac{r_2 + r_3}{2} \right) \frac{\omega^2 l}{g} \]  

where:  
\[ r_2 = \text{radial distance to top of sludge layer (m)} \]  
\[ r_3 = \text{radial distance to bottom of tube (m)} \]

The supernatants were decanted off and spun at high speed in a laboratory centrifuge to record the remaining suspended solids. These results gave the solids concentrations of the supernatants (\(C_{sup}\)) for substitution into the estimated solids recovery equation.

The mean residence time of the liquid was calculated for different flow rates in the industrial centrifuge, and estimated in the batch experiment by the "power on" time. The final solids recovery was then estimated by choosing the appropriate g-force for the prototype centrifuge, and interpolating between liquid residence times according to estimated liquid residence time in the prototype.

Vesilind recognised problems in determining scrolling efficiency and that this was related to solids consistency: "A light, fluffy sludge of poor consistency cannot be scrolled". He used a "penetrometer" (a brass rod dropped into sludge from a known height) to quantify solids consistency. Penetration (\(P\)) was given as the fraction of sludge not penetrated by the rod. Soft sludges with a low resistance (\(P\) close to zero), scrolled worse than tough sludges with a high resistance (\(P\) close to 1).

Tests with the penetrometer were carried out on samples of solids sludge sedimented at the same gravitational acceleration as the clarified liquor, except that the spin time was arbitrarily set at a constant 60 seconds.

Vesilind thought that \((C_o - C_{sup})/C_o\) was a reasonable measure of sludge settling characteristics and that \(P\) was a reasonable measure of the ability of a sludge to be discharged from the machine. He combined the results from both sets of laboratory tests into one equation for estimated solids recovery (ER) in the prototype:
Tailored to wastewater sludges, the model was claimed to have a high 10% accuracy in predicting centrifuge recovery performance.

However, there are some fundamental difficulties to overcome in the analysis procedure. Firstly, it is difficult to estimate the appropriate residence time of liquid inside the prototype centrifuge from a simple flow rate and volume calculation according to pond depth in the pilot centrifuge. A significant part of this volume may be occupied by sedimenting solids. There is also a marked deviation from plug flow due to a stratified surface layer in short decanters. There may also be shear break up of flocculated suspensions in the feed zone of the prototype which is not mimicked in the laboratory.

Vesilind used the laboratory centrifuge "power on" time for the residence time, and suggested that any loss of separation effect in speed-up time was compensated for in braking time. However, the full separation effect occurs only at the maximum speed attained, and so a more appropriate measure of residence time would be the time spent at speed.

Vesilind acknowledged that clarification was dependent on flow rate in the centrifuge by suggesting that tests were carried out at a variety of residence times in the laboratory centrifuge, and yet he did not treat the solids in a similar way. Solids were spun for a standard time set at an arbitrary 60s for the penetrometer test. However, he later went on to show that the rate of solids compaction increases with gravitational acceleration.

Using a strobe light to "freeze" the image of settling solids in a specially adapted centrifuge, Vesilind showed that compression of solids continues until an equilibrium value is reached where further compaction is negligible. Buscall (1982) used compression tests like these to examine the collapse of sediment network structure in a gravitational force field over periods of time which could extend into hours.
New rheometers make it possible to describe sludge characteristics in a more sophisticated way than using a penetrometer. Ward and Hoare (1990) showed how the elasticity and toughness of a dewatered sludge can be quantified according to standard viscoelastic theory. He went on to investigate the rheological nature of solids emerging from a scroll decanter centrifuge under different operating conditions. Using viscoelastic characterisation, he was able to differentiate between those solids with a high scrolling efficiency, and those which required extra hydraulic force (ie. a deep pond) to help them scroll up the beach without slipping. It may be possible to use rheological analysis of sediments produced in small scale laboratory tests to help predict the correct operating parameters required for successful scroll centrifuge operation.

1.4 SYSTEM CHARACTERISATION FOR DEWATERING

Sedimentation processes dewater by compression or drainage mechanisms according to characteristics such as particle morphology and surface properties. Ward (1989) found that sedimentation tests under gravity, network strength, resistance to shearing forces and centrifugal drainage tests were important in characterising biological sediments.

1.4.1 Solids volume fraction

Dewatering cannot always be directly related to solids concentration measurements, particularly in the case of whole cells where intracellular water is lost by drying but not by dewatering. In this case, solids volume fraction ($\phi$) is used which may be converted to dwt/wwt% where the cell wet weight to dry weight ratio is known (0.35 for yeast according to Harrison 1967). Ward (1989) found that this ratio was 0.36 for yeast, 0.30 for $E.coli$ and 0.27 for $F.graminearum$.

$\phi$ can also be measured using packed volumes under high speed centrifugation, where the interstitial fluid remaining between particles is considered negligible. Dye tracer methods were found to be unsuitable for biological suspensions due to adsorbance onto the cell surface (Ward 1989).
1.4.2 Compression and drainage dewatering

Compression dewatering occurs as liquid is squeezed out of saturated pores of sediment as it collapses under increasing centrifugal pressure. The liquid moves in the opposite direction to the applied centrifugal pressure. The degree of dewatering attainable by compression is affected by the pressure generated due to accumulated solids as shown in section 1.3.9.1.

Drainage dewatering occurs as liquid is expelled through an open network of compact particles, and it may be displaced by air. Liquid flow is in the same direction as the applied pressure. For example, in centrifugal filters the resistance to dewatering is proportional to the cake thickness and its porosity (Coulson and Richardson 1985).

The predominant dewatering mechanism depends on the morphology of the particles in a sediment. Fine, gelatinous materials tend to dewater by compression, while coarse, granular, or rigid materials tend to dewater by drainage.

Sediments of sub-micron particles tend to occupy relatively large volumes and dewatering becomes increasingly difficult as the primary particle size diminishes. Asymmetric particles tend to form space-filling network structures at lower concentrations than spherical particles (Buscall et al 1984). These phenomena are related to the packing characteristics of such particles.

Flocculation increases the size and hence the settling rate of particles at low concentrations. However, at high concentrations individual floccules may pack together to form a network structure or "one large floc". The compression rate may be retarded as a result (Buscall et al 1984).

The size distributions of aggregated particles, such as compressed flocs composing a dewatered sediment, are highly dependent on solids concentration. Hence it is not possible to describe them using particle sizing techniques which require very dilute samples.

1.4.3 Particle morphology

Ward (1989) used a combination of freeze-etch microscopy (FEM) and a study of particle surface properties to examine particle morphology.
Ward found from FEM observations that organisms with a low aspect ratio such as *S. cerevisiae* (yeast) and *E. coli* form sediments of discrete particles with a randomly close-packed structure. This kind of structure has no network, and dewaterers by compression. Yeast cells flocculated with borax had a weak network bridging structure which rapidly collapsed on compression, to give a randomly close packed, but rigid sediment. In contrast to these organisms, *F. graminearum*, a mycelial organism of hyphae with an aspect ratio of 50, had an open network structure after compaction. A rigid, porous structure such as this dewatered by drainage.

Surface properties of particular significance to dewatering are those concerned with particle interaction and agglomeration mechanisms. Particle surface charge can affect particles up to about 1 μm in diameter. Ward considered its influence on small cells such as *E. coli* (2 μm). He found that both yeast and *E. coli* were charge stabilised at most pHs, forming stable suspensions of discrete particles, unless treated with a flocculating agent such as borax. The large cell size of *F. graminearum* (approximately 600 μm long), prevents colloidal and surface properties having any effect on bulk suspension flow properties.

### 1.4.4 Sedimentation tests under gravity

Ward (1989) observed the settling characteristics of various biological suspensions under gravity in order to ascertain whether useful information could be obtained for sediments forming under an artificial gravitational field.

The position of the solid/liquid interface for a suspension of whole yeast cells fell gradually under gravity over a 22 hour period. Yeast cells have a narrow size distribution and their settling rate was limited only by the resistance of the suspending medium as described by the Richardson and Zaki equation (1974). However, the settling rate of yeast flocculated with borax was highly dependent on yeast:borax concentration. At borax concentrations greater than 70 mM large, rapidly settling flocs were formed, and at less than 35 mM, small flocs caused slow, hindered settling. *F. graminearum* formed space filling networks at solids volume fractions as low as 0.05, which did not settle under
gravity alone. However, increasing centrifugal pressure led to rapid compaction. These simple experiments illustrated the importance of sediment properties in affecting the settling rate.

1.4.5 Compaction of flocculated sediments under centrifugal pressure

A typical compression curve is shown in Figure 1.4.1 for flocculated wastewater sludge (Wiesmann and Binder 1982). As relative centrifugal force is increased, solids volume fraction decreases in stages which correspond to the release of the specific water fractions described below.

![Classification of water fractions in a flocculated sediment by gravitational and centrifugal settling.](image)

**Figure 1.4.1:** Classification of water fractions in a flocculated sediment by gravitational and centrifugal settling.

Easily extracted free water took 74% of the volume. 21% of the sample volume was taken by floe water; that which is hindered from flowing through the sample by the fibrous nature of the solid substance. The floe water was pressed out increasingly as the flocs compacted up to an RCF of 3000 g. To separate the capillary water, relative centrifugal force was increased to 15 000 g, obtainable only in laboratory centrifuges and some industrial scale tubular bowl centrifuges.

The particle water was chemically bound or intracellular, and could not be removed except by drying. In general, successive water fractions become increasingly difficult to remove, requiring high centrifugal speeds and specialised dewatering equipment.
1.4.6 Network strength

Compressive dewatering of a sediment is characterised by its network strength, which may be determined using a pressure filtration cell or under centrifugal pressure as proposed by Buscall (1982).

Buscall (1982) observed the relative position of a solid / liquid interface with increasing centrifugal pressure using a stroboscopic centrifuge at low centrifugal speeds. Each step increase in the compressive force gave rise to a corresponding reduction in the height of the solid/liquid interface. Hence at each new step the solids network compresses to an equilibrium level at which the strength of the network is equal to the applied compressive force.

Data collected from step increases in RCF was approximated to an incremental change in solids volume fraction caused by an incremental change in the applied compressive pressure. This was used to calculate a value for the network strength, called the "network modulus" (K):

\[
K = \frac{\phi}{\ln \phi} = \frac{dP}{d\ln \phi}
\]

where \( \phi \) = solids volume fraction
\( P \) = centrifugal pressure (Pa)
\( K \) = network modulus (Pa)

A plot of network modulus against solids volume fraction yielded a smooth curve which could be fitted according to:

\[
K = a_2 \phi^n
\]

where \( a_2 \) and \( n_i \) are system dependant constants, ie. network strength varies for different systems at the same solids concentrations.

Ward (1989) tried to find the network modulus of a flocculated suspension using the above method. He found that the network structure of whole yeast flocculated with borax was so weak that it was insufficient to resist compressive dewatering even under gravity. The final degree of dewatering attainable in this case was not affected by increasing centrifugal pressure since the flocs collapsed rapidly to form a randomly close packed network. In this
system, the only influence of small increases in centrifugal pressure was on the settling rate, not the equilibrium compression level.

Network strength may also be determined under shear by rheological characterisation.

1.4.7 Rheological Characterisation

Rheological characterisation is commonly used to find correlations between molecular structure and material behaviour. Historically, this has been the main driving force behind commercial research into the rheology of elastic liquids, such as synthetic fibres and polymer melts. However, simple rheological measurements can also be used to give information about material behaviour in non simple flow situations (Walters 1975). There are several techniques for rheological characterisation which include tests under continuous and oscillatory shear.

Tests carried out under continuous shear by a viscometer give information about how a liquid responds to various shear strain rates. The shear stress response is plotted against shear strain rate, and the gradient is called the viscosity, or "apparent viscosity" if the curve is not a straight line.

The application of an oscillating shear strain results in a dynamic shear stress response, of which two independent parameters, both the timing and the amplitude are important. These tests are carried out in rheometers.

The shear stress $\tau$ experienced by any material is equivalent to the shearing force applied, divided by the area of the surface plane which is affected.

1.4.7.1 Tests under continuous shear

In viscous flow, the resulting angular displacement of the plane to which a shearing force is applied is called the strain (\(\gamma\)). In Newtonian (ideally viscous) liquids such as water or glycerol, the rate of strain (called the shear rate, \(\dot{\gamma}\)) is directly proportional to the stress (\(\tau\)) due to the shearing force applied, but independent of the strain magnitude:

$$\mu = \frac{\tau}{\dot{\gamma}}$$
where \( \mu = \text{viscosity} \) (Pa s)

Newtonian liquids such as water or glycerol have a constant viscosity coefficient with respect to shear strain rate. If the viscosity is not constant, it is usually referred to as the "apparent viscosity". When this is the case, then it describes pseudoplastic (shear thinning) or dilatant (shear thickening) liquids. These properties may be adequately described using one of the many inelastic flow models, such as the power law model:

\[
\tau = \psi \dot{\gamma}^n
\]

\( \psi \) is a constant and \( n \) is an empirically found exponent which is system dependent. For \( n=1 \), the material is an ideal, perfectly viscous liquid (Newtonian), as described by the straight line in Figure 1.4.2 (a).

For \( n<1 \), such as with most polymer melts, the viscosity decreases as the shear rate increases, and the material is said to be shear thinning, or pseudoplastic as shown in Figure 1.4.2 (c).

For \( n>1 \), such as with aqueous solutions of cornflour, the viscosity increases as the shear rate increases, and the material is said to be dilatant as shown in Figure 1.4.2 (d). However, such materials often behave in a way such that an abrupt "solidification" occurs as molecules are rearranged under high shear. The model is no longer satisfactory when this point is reached.

Some materials behave as elastic solids at low stress levels; that is, they do not flow until a certain specific yield stress is reached, (Figure 1.4.2.(b)). If the yield stress is reached and the flow behaviour is then Newtonian, then they may be described by the Bingham plastic model, which incorporates a yield stress \( (\tau_y) \) into the flow curve equation:

\[
\tau - \tau_y = \mu \dot{\gamma}
\]

The Herschel-Bulkley model incorporates a yield stress into the power law equation for materials which exhibit shear-thinning behaviour after a yield stress is reached: Figure 1.4.2 (e).

\[
\tau - \tau_y = \psi \dot{\gamma}^n
\]
1.4.7.2 Elasticity

The force applied to a surface plane in a perfectly elastic solid causes deformation proportional to the magnitude of the force applied. The rate at which the resultant strain occurs is irrelevant, since all the energy is stored, allowing the solid to spring back to its original shape once the strain is removed. Shear stress plotted against shear strain yields a straight line graph for a perfectly elastic, Hookean solid such as rubber. The gradient of such a plot gives rise to the "modulus of rigidity" (G):

\[ G = \frac{\tau}{\gamma} \]

Most solid materials have an elastic limit. If the applied strain exceeds this limit, then a solid becomes brittle and may fracture.

Some solids can exhibit liquid-like properties when exposed to strain, where part of the shearing force is dissipated as viscous flow, and part is stored as elastic energy. Sludge-like materials such as this are called viscoelastic.

1.4.8 Viscoelasticity

Generally speaking, rheology is a function of stress, strain and time, and unless a material is Hookean or Newtonian, then more than one parameter is required to describe its flow properties. Ward (1989) found that a variety of biological sediments including yeast, *E.coli*, borax-flocculated yeast and
F. graminearum exhibited rheological characteristics typical of a viscoelastic material. He also found that these biological sediments have an increasingly significant elastic component as they dewater and the solids concentration increases.

Viscoelastic characterisation is the measurement of the relative effects of shear rate dependent stresses (viscous) and shear magnitude dependent stresses (elastic) on a sample exposed to shear.

The standard rheometric test for describing viscoelastic behaviour is to apply an oscillating strain to a sample of material held, for example, between two parallel plates. The strain originates at the bottom plate, and is transferred to the top plate by the stresses inside the material. A turning force couple is formed between the bottom and top plate with each new oscillation. The top plate is constrained only by a calibrated torsion bar, from which torque signals give information about the material structure according to the viscoelastic theory outlined in Appendix II.

The strain applied to the sample is varied periodically with a sinusoidal alternation at a specified frequency, illustrated in Figure 1.4.3 (a). Standard tests are carried out at a range of frequencies, for example between 0.01 and 10 Hz.

For small amplitude oscillating strains, the sample stress response is harmonic but slightly out of phase, i.e. the response of the sample picked up by the top plate oscillates at the same frequency as the bottom plate, but lags behind it slightly. The delay in the timing of the response is called the phase-shift (also lag angle or loss angle δ), measured between 0° and 90°. It gives information about the relative proportion of suspension strength attributable to interactions of a viscous (liquid-like) nature and an elastic (solid-like) nature.

An elastic, Hookean solid oscillates wholly in phase with the applied strain since its stress response is directly proportional to the magnitude of the deformation. Its phase shift is 0°, as shown in Figure 1.4.3 (b).
Figure 1.4.3: Viscoelastic characterisation by application of an oscillating strain ($\gamma$) to obtain a stress response ($\tau$). (a) applied strain, (b) elastic response, (c) viscous response, (d) viscoelastic response.
A viscous, Newtonian liquid oscillates completely out of phase with the applied strain since its stress response is directly proportional to the deformation rate. The maximum rate of deformation occurs wherever the magnitude of the deformation is zero. Here the phase shift is 90°, as shown in Figure 1.4.3 (c).

The behaviour of a viscoelastic material lies somewhere in between these two ideals as shown in Figure 1.4.3 (d).

The second parameter measured in a viscoelastic oscillation is the amplitude of the response relative to the input. The amplitude ratio is the ratio of peak stress to peak strain (τ/γ), as shown in Figure 1.4.3(d). It is independent of the phase shift, and gives information about how much elastic energy is stored by the sample, and how much is dissipated by viscous flow and heat. By linear viscoelastic theory, (see Appendix II), the amplitude ratio and phase shift are combined to yield 3 distinct variables:

Storage modulus (G'), is the stress in phase with the strain in a sinusoidal deformation divided by the strain. It is a measure of the energy stored and recovered per cycle, when different systems are compared at the same strain amplitude.

Loss modulus (G'″), is the stress 90° out of phase with the strain divided by the strain. It is a measure of the energy dissipated per cycle of sinusoidal deformation, when different systems are compared at the same strain amplitude.

Complex shear modulus (G*), is the sum of the previous two moduli in complex format. Unlike the amplitude ratio, it is time independent. Its magnitude gives a measure of the total shear stiffness of the sample arising from both viscous flow and elastic strain.

\[ G^* = G + iG'″ \]

The loss modulus is often called the imaginary part of the complex whole. However, G'″ is actually a real quantity which is the coefficient of an imaginary term. All shear moduli are expressed in units of pascals (Pa).
Materials are generally referred to as "elastic liquids" if they continually change their shape when subjected to stress, and "viscous solids" if they do not (Walters 1975). Solid-like properties dominate when:

\[ G' \geq 3G'' \]

which occurs at around a loss angle of 18.4° since:

\[ \tan \delta = \frac{G''}{G'} \]

At appropriate frequencies, oscillatory flow is said to simulate bulk flow conditions. However, this is not well defined, with a rough approximation at high frequencies (>1 Hz), corresponding to rapid "forced" flow, and at low frequencies (<0.1 Hz), corresponding to slow "creeping" flow (Ward and Hoare 1990).

Storage modulus measurements at 1 Hz have been used to quantify biscuit texture (Oliver 1992). Ward (1989) processed viscoelastic data at 5 Hz when considering the bulk flow of sediment in a scroll decanter centrifuge.

1.4.9 Network strength using the pulse shearometer.

Network strength may be determined using wave propagation methods in a pulse shearometer. A sediment sample is subjected to instantaneous shear at a fixed amplitude and its attenuation to a lesser amplitude is measured. At low strains the pulse shearometer yields a value for the storage modulus of the sample according to linear viscoelastic theory. The shearometer operates at high frequencies of around 200 Hz where the storage modulus is the dominant part of the complex shear modulus, hence:

\[ G' \approx G^* \]

Buscall et al (1984) found that the shear modulus as measured in the pulse shearometer was identical to the network modulus found by compression for some non-biological sediments. For example, rod-like particles of attapulgite at \( \phi = 0.08 \) had \( G' \) and \( K \) of \( 10^4 \) Pa, and spherical particles of latex at \( \phi = 0.08 \) had \( G' \) and \( K \) of \( 10^2 \) Pa.
However, Buscall and White (1987) found that a measure of strength under compression was not necessarily the same as a measure of strength under shear in all systems. In general:

\[ K \approx \frac{2}{3} G' \]

Ward (1989) found that for *F. graminearum* the network modulus was 1/3 to 2/3 the shear modulus depending on cell volume fraction.

1.4.10 Rheological Characterisation of Biological Suspensions

Ward (1989) studied the rheological behaviour of concentrated suspensions of yeast, *E. coli*, yeast flocculated with borax and *F. graminearum* under both continuous and oscillatory shear. The concentrations studied ranged from dilute suspensions equivalent to those exiting a fermenter, to highly dewatered sediments. He proceeded to show how information from these studies was relevant to both design and optimisation of solid/liquid separation in an industrial centrifuge.

1.4.10.1 Viscosity of concentrated biological suspensions

Under continuous shear, suspensions of yeast with volume fractions 0.27 showed shear-thinning behaviour at low shear rates below 100 s⁻¹. Above this critical shear rate, the viscosity coefficient tended to increase, and shear thickening behaviour was observed. This dilatancy was more pronounced in concentrated suspensions up to a maximum packing fraction of 0.64, where viscous flow was limited and elastic forces began to dominate.

Ward explained that low shear rates reduce cell:cell surface interactions, thereby reducing the apparent viscosity of the bulk suspension. At high shear rates, and in more concentrated suspensions, interstitial water is excluded and becomes insufficient to lubricate rapid cell movement. Similar results were observed for *E. coli*, although the maximum volume fraction achieved was higher at 0.75.

The addition of borax to suspensions of yeast resulted in a dramatic rise in the apparent viscosity as compared to free yeast suspensions of the same
solids concentration. This rise in viscosity was maintained at low shear rates (1-20 s\(^{-1}\)), depending on yeast and borax concentrations. At high shear rates (20-1000 s\(^{-1}\)), shear thinning behaviour was observed.

Increasing the borax concentration from 7.5 mM to 30 mM or increasing the yeast concentration had similar effects, i.e. the step increase in apparent viscosity tended to occur at higher shear rates as the concentrations increased. Borax concentrations in excess of 40 mM yielded non-homogenous suspensions which could not be measured accurately due to wall slip.

The explanation offered by Ward for these phenomena, was that at low shear rates, shear thickening occurs due to the yeast-borax reaction. High shear rates provide enough energy to break down the agglomerating effects of the reaction, and the apparent viscosity is thereby reduced. Apparent viscosity changed with borax or yeast concentration due to shifts in the equilibrium of the flocculation reaction.

Rheological studies of \textit{F.graminearum} are problematic due to the large cell size which is greater than 20\% of a narrow gap in conventional equipment, giving rise to phase separation and wall slip.

Ward stated that viscosity considered as resistance to flow could indicate the likelihood of slip on the beach section of a decanter centrifuge. However, it is difficult to relate shear conditions inside the centrifuge to continuous shear measurements on a viscometer. In addition, continuous shear studies are not suitable for highly concentrated suspensions such as dewatered sediments, which exhibit significant elastic behaviour. Viscoelastic characterisation of concentrated sediments gave more information about dewatering in this type of centrifuge.

1.4.10.2 Viscoelastic behaviour of dewatered sediments from biological suspensions

Ward (1989) found that viscoelastic parameters could be used as a measure of the level of dewatering in biological sediments. Sediments tended to become more solid-like, with higher storage moduli and lower loss angles as
they dewatered. This is illustrated in Figure 1.4.4 for both yeast and *E.coli* at various cell volume fractions.

Ward found that both yeast and *E.coli* displayed viscoelastic behaviour at cell volume fractions greater than 0.6. The loss angles of *E.coli* tended to be higher than those in yeast at equivalent concentrations. This accounts for the "slimy" (liquid-like) texture of *E.coli* compared to the more "pasty" (solid-like) texture of yeast.

The storage modulus of both yeast and *E.coli* was frequency dependent, which accounted for a difference in values obtained by the pulse shearometer and oscillatory shear measurements at 200 Hz and 5 Hz respectively. A substantial degree of error was found in the pulse shearometer measurements due to wave attenuation i.e. waves decaying within the time taken for the first wavelength cycle.

The effect on viscoelastic parameters of adding a small amount of borax (7.5 mM) to yeast suspensions and then sedimenting, was to increase the storage modulus by an order of magnitude from $10^2$ Pa to $10^3$ Pa at the same cell volume fraction. The loss angle also dropped from around 15° to 7°, showing a marked tendency towards more solid-like behaviour with borax present. Increasing the borax concentration to 70 mM caused a further rise in storage modulus to $10^5$ Pa and a drop in loss angle to 4°.

There was also a drop in loss angle at higher frequencies, indicating an increasing resistance to deformation, with fracture occurring at very high rates of deformation (10 Hz). The borax yeast sediments showed a close similarity to the classic Maxwell model (Whorlow 1980) for viscoelastic behaviour, over an unusually narrow frequency range. Ward took this as evidence of the breakdown of a single cell:cell interaction bond strength under shear.

In his viscoelastic studies of *F.graminearum*, Ward found little variation in shear modulus with a low or high degree of cell branching. At cell volume fractions of 0.19, typical parameters at 10 Hz were $G' = 10^5$ Pa and $\delta < 5°$, and showed little frequency dependence.
Figure 1.4.4: Viscoelastic spectra with respect to cell volume fraction for Baker's yeast (S. cerevisiae) and E. coli. Data taken from Ward (1990).

Open symbols E. coli, closed symbols S. cerevisiae.

- □ Loss angle $\delta (^\circ)$
- ■ Storage modulus $G'$ (Pa)
1.4.11 Viscoelastic characterisation for dewatering in industrial centrifuges

Ward observed a change in the dewatering of yeast solids discharge in the scroll decanter centrifuge on the prior addition of borax, and related this change to a change in the viscoelastic properties of the sediment. Yeast suspensions in the centrifuge required deep pond operation to scroll up the beach section resulting in a poorly dewatered sediment, whereas borax-flocculated sediment could be discharged using a dry beach, producing a highly dewatered cake. He also noted that this type of discharge was accompanied by a rise in the torque output of the conveyor.

In general, Ward found that solid-like sediments with a low loss angle were more likely to scroll up a dry beach (shallow pond operation) in a scroll decanter centrifuge. This was true for both borax flocculated yeast and F.graminearum. In each of these cases the complex shear moduli were also relatively high, (>10^3 Pa measured at 5 Hz).

Ward noted that both yeast and soya protein precipitates had higher loss angles (>18°), and required the use of a deep pond, where hydrostatic pressure assists in forcing the sediment up the beach section. He described yeast as a fluid sediment, and compared this to his observations that it is discharged easily from a disc stack centrifuge.

Ward constructed Figure 1.4.5, in which he divided up dewatered sediments into four categories, based on their viscoelastic characteristics.

The position of the axes are somewhat arbitrary. The vertical axis relies on the 18.4° loss angle distinction between viscoelastic solids and liquids described in section 1.4.8. Ward placed the horizontal axis at 10^3 Pa, setting this as the critical "toughness" of material required to scroll up the beach section in the centrifuge configuration he used.

Figure 1.4.5 was constructed purely from observations of centrifuge operation on a range of suspensions which were deliberately chosen for study because of their differing flow properties.
Figure 1.4.5: Viscoelastic characterisation in relation to dewatering in a scroll decanter centrifuge. Viscoelastic characteristics measured at 5 Hz, and carried out on samples of sediment discharged from a scroll decanter centrifuge under various operating conditions.

2 Viscous, difficult to scroll

M Pa

Highly structured, should filter

F. graminearum

Soya Protein Precipitate

kPa

Whole yeast cells flocculated with borax

δ (Loss angle)

90 45 20

Whole yeast cells

3 Fluid, settles easily

Pa

Elastic fluid, hindered settling

51
Using this information, Ward felt able to make a prediction that solids falling into sector 4 would be too soft to scroll up a beach without long residence times in the centrifuge, i.e. they would require low conveyor differential speeds. The lowest scroll differential Ward was able to achieve with his equipment was 5 rpm, whereas more modern machines can support differentials of 0.5 rpm.

The characterisation procedures described above could be used as a design tool for predicting the appropriate operating parameters for dewatering of biological solids in the scroll decanter centrifuge.

1.4.12 Limit of compression dewatering

The strength of suspensions as determined by Buscall et al (1984) for the network modulus lead to correlations between the degree of dewatering of any suspension under a given compressive pressure. Ward (1989) used this method to estimate the maximum degree of compressive dewatering of suspensions at a centrifugal pressure equivalent to that in his industrial centrifuge.

For \( F. graminearum \):
\[
K = 2.96 \times 10^6 \phi^{3.48} \text{ Pa}
\]

or
\[
P = 8.51 \times 10^5 \phi^{3.48} \text{ Pa}
\]

In a centrifuge \( P = \Delta \rho g h_s \phi \)

hence:

\[
\phi = \frac{2.48 \sqrt{\frac{\Delta \rho g h_s}{8.51 \times 10^5}}}{41}
\]

where \( h_s \) was taken as 3/4 the pond depth for the stagnant layer available for settling (Gosele 1980).

Ward found that the theoretical maximum solids concentration for yeast was 24%, yeast flocculated with borax was 27%, and \( F. graminearum \) was 6%.

In pilot scale trials with a scroll decanter centrifuge operating with a flooded beach, the actual dry weight of the solids discharged was 5-24% for yeast, 18% for borax-flocculated yeast and 5-12% for \( F. graminearum \), depending on conveyor differential speed. Hence compression test measurements over-estimated the maximum degree of compressive dewatering in the case of yeast and yeast flocculated with borax, and under-estimated it in
the case of *F. graminearum*, where drainage dewatering was also thought to take place.

### 1.4.13 Limit of drainage dewatering

Ward (1989) carried out simple scale down experiments to investigate drainage dewatering in biological solids. He fitted swing-out rotor centrifuge tubes with a loose fitting angled base which acted as a beach simulation device. Sediment with a rigid network structure remained on the "beach", whilst any water draining through the sediment slipped down to the compartment below, through the narrow clearance between the beach insert and the tube wall.

He found that neither yeast nor *E. coli* yielded any water by drainage, but were viscous enough for the entire suspension to slip down the narrow clearance. Borax-flocculated yeast formed a rigid network which remained on the beach section, but an insignificant amount of water drained to the compartment below since the sediment compressed to a close packed structure. With its open, porous network, *F. graminearum* was the only material examined through which water drained freely. Once the sediment had formed a rigid cake within a stable structure, dewatering occurred by drainage, and much drier sediments were obtained than those found by compression dewatering alone. These experiments were carried out at a range of relative centrifugal forces, from 100-2500g.

The method used by Ward for predicting the limit of drainage dewatering in *F. graminearum* was to use data for the dry weight of sediment samples from the beach simulation devices. The data for a residence time of 10 minutes, from 100-2500 g was extrapolated to 3000 g, to give a dry weight estimate of 21%. A scroll decanter centrifuge with a dry beach yielded sediments of 11-15 dwt/wwt %. Hence extrapolation of drainage test data overestimated drainage dewatering capability for pilot scale trials in this case.

The highest levels of dewatering in the scroll decanter centrifuge were attained at the lowest conveyor differentials used. Hence the calculated limits for drainage and compressive dewatering are approached as the conveyor differential tends towards zero, ie. as the continuous solids-conveying centrifuge...
approaches flow (or non-flow) conditions similar to those of sediment in a laboratory centrifuge.

The basis of these scale down tests was to keep compressive pressure or relative centrifugal force constant as appropriate to the dewatering mechanism (Gosele 1980). There was no reference to the mean residence time of solids in the centrifuge, nor to the shear experienced by the conveyed solids, which may also affect the dewatering process.

1.5 Summary

The literature has shown a number of important points to consider for the dewatering and scale down of solids recovery in industrial centrifuges. Selective flocculation of disrupted microbial cells can be used to enhance the removal of contaminants such as cell debris, lipids and nucleic acids by centrifugal separation. The choice of centrifuge will depend on the application such as for high or low solids throughput.

It should be possible to bring the design and optimisation of centrifugal separation processes to an early stage of product development by applying scale down techniques. Scale change in industrial centrifuges can follow any or a combination of the following conditions: equivalent settling area, dissipated power, peripheral velocity or total bowl volume, depending on the nature of the process. Clarification is profoundly affected by the liquid flow profile within the centrifuge and can be complicated by solids dewatering and transport.

Liquid viscosity is an important parameter for investigating how particles settle in suspension. Complex shear modulus and loss angle are important parameters for characterising dewatered sludges which are typically solid like or visco-elastic. The most convenient way of rapidly measuring visco-elastic parameters under controlled conditions is by using an instrument such as the Bohlin (VOR) rheometer. Such measurements can be used to characterise dewatering in industrial centrifuges.
2. AIMS AND OBJECTIVES:

The aim of this study is to devise methods for prediction of operating parameters for biological suspensions in industrial centrifuges using small amounts of process material. This will be achieved by adopting a combination of scale down techniques and instrumental analysis to investigate both solids recovery and sediment dewatering.

The study will focus on two types of centrifuge, both currently used in the primary recovery stages of bio-processes:

The scroll decanter centrifuge (primarily used for dewatering of bulk solids with a high feed solids concentration)

The disc stack centrifuge (primarily used for clarifying relatively dilute suspensions)

The literature indicates that laboratory spin tests do not give enough information about dewatering properties, since a qualitative assessment of sediment flow properties is always called for. Rheological instrumentation may be used to give a quantitative assessment of the flow behaviour of sediments using small amounts of process material.

Viscoelastic properties of dewatered sediments discharged from a scroll decanter centrifuge should give insight into how the centrifuge works: Ward (1989) observed a rise in torque with increased solids dewatering. Torque values from the scroll decanter centrifuge will be recorded in the practical parts of this study, to facilitate investigation into the relationships between torque output and viscoelastic parameters of dewatered sediment.

Solids recovery in a moderately sized disc stack centrifuge has been scaled down by separation area (Mannweiler and Hoare 1992) using dilute suspensions of PVAc. This study aims to apply the same scale down protocol to a smaller disc stack centrifuge using biological suspensions.

This study also aims to investigate the limitations of scaling down industrial centrifuges with respect to shear of flocculated biological suspensions. It is anticipated that shear in the feed zone will affect solids recovery in both
types of centrifuge, and that viscoelastic properties or dewatering of sediment may be affected by conveyance in the scroll decanter centrifuge.

The first step in this study is to demonstrate the importance of dewatering in biotechnology by constructing a mass balance across a typical process. The process chosen for study involves the recovery of a soluble protein from an intracellular, microbial fermentation followed by cell disruption and flocculation to remove contaminating material from solution.

Mass balances are used as design tools by engineers when trying to understand the nature and influence of major process variables. They may be used to find an area of research interest or to help define an operating window for a new process.

Mass balances were set up in a commercially available personal computer spreadsheet software package (Borland's Quattro Pro) to predict the performance of a downstream process proposed for the recovery of intracellular proteins from a microbial fermentation. Two spreadsheets were devised: one to examine the effect of dewatering on product yield in an ideal separation step, and another to estimate the volume of material required for a scaled down process and hence aid equipment specification.

The mass-balance spreadsheets were written to facilitate easy alteration of the main variables of interest. The entire spreadsheet was recalculated whenever a variable was changed.

2.1 Mass balances for dewatering

Mass balances were used to study the significance of different levels of dewatering around the primary separation stage for the recovery of intracellular protein from a fermentation of baker's yeast, as shown in Figure 2.1. A sample spreadsheet is shown in Appendix 1 (a).

2.1.1 Format

The volume basis used for examining interaction of process variables in the dewatering spreadsheet was the process analysis standard of 1 L.
Figure 2.1.1: Mass balance flow sheet. Boxed section indicates operations considered in dewatering mass balance.
Yeast fermentations typically produce 60 wwg L\(^{-1}\) whole cells (Dehgani 1996). Cells are concentrated during the harvesting process by a factor of up to 6x (10 x is possible according to Aronsson 1987) in this spreadsheet. This factor was varied during experiments with the spreadsheet in order to examine the effect of feed concentration on dewatering.

Yeast cell composition was obtained from selected texts (Mateles and Tannenbaum 1968, Harrison 1967, Moo Young and Gregory 1986) and could be altered for a different microorganism, or for a recombinant strain with increased DNA content (rDNA process). Intracellular water occupied 70% of the total volume (Ward 1989). Cell components shown fed to the homogeniser were included in the total sum only as components of the whole cells. Concentrations were calculated throughout as fractions of the total mass.

The harvested process stream was disrupted in an industrial homogeniser and cell contents release calculated according to Hetherington et al (1971) for protein release. Protein release is linearly proportional to cell concentration not exceeding 60% by volume (660 g L\(^{-1}\)). It was assumed throughout that the process temperature would be controlled in a way to minimise product degradation due to protease action. Neither temperature nor product degradation were not variable parameters in the spreadsheet mass balance.

A flocculation step using either borax or PEI extracted cell debris and the major soluble contaminants into a solid phase of the process stream according to data from Milburn et al (1990). The process stream was divided into liquid and solid phases at this stage, where the composition of the liquid product phase was calculated first. It was assumed that no cells would remain in solution, no protein product would be precipitated, 50% carbohydrates would remain in solution and 4.4% of the extracellular water was chemically bound to the flocculated material (Wiesmann and Binder 1982). The amount of PEI required was found from a stoichiometric assessment of yeast cell debris flocculation (Milburn et al 1990). Flocculant was added at 10% of the process stream volume according to standard industrial practice.
The physical separation of the solid and liquid phases was said to occur in a single unit operation where there was no carryover of contaminating solids into the product stream. In this ideal separation step, the only product loss was through liquor entrained in the solids discharge.

Having determined that no solids were present in the product stream, then all were present in the sediment discharge. The solids dryness specification determined the total mass of solids discharged and hence the mass of entrained liquor. Any liquor not entrained remained in the product stream.

The step protein yield for this and other unit operations was calculated as the fraction of protein dissolved in the product stream relative to the total present in the feed.

Experiments were carried out with the spreadsheet where the feed stream cell concentration was varied from a typical yeast fermentation broth (20 gdcw L\(^{-1}\)) to cells harvested using a centrifuge (200 gdcw L\(^{-1}\)). This was repeated for a range of sediment solids concentrations from 20-60 % dwt/wwt, given as kg solids / kg total mass.

2.1.2 Results

Figure (2.1.2) shows different levels of dewatering as a function of the input solids concentration and the overall yield of the operation. Taking typical values as an input concentration of 100 gdcw L\(^{-1}\) and a step yield in excess of 90%, then it is clear that this can only be achieved if a level of dewatering greater than 60% (expressed as 0.6 kg dry solids per kg total sediment) is realised. Current dewatering technology using the scroll decanter centrifuge is limited to 0.3 kgkg\(^{-1}\).

Overall recovery may be enhanced by dilution of the process stream at the flocculation stage to avoid entrainment of concentrated product in poorly dewatered solids. However, dilution may not be a feasible option for rDNA processes where the cost of containing large volumes of material is prohibitive. This study will aim to investigate how dewatering can be maximised in a typically concentrated process system of homogenised yeast flocculated with PEI.
Figure 2.1.2: Simulation of the effect of dewatering on the recovery of intracellular yeast protein. Sediment dewatering given in kg dry sediment per kg total sediment.
2.2 Mass balances for scale down

This mass balance was carried out as an extension of the previous mass balance for dewatering, but with a series of non-ideal separation stages operating at fixed feed and solids concentrations. The flow sheet of the process used in this mass balance is shown in Figure 2.1.1, with the results shown in Figure 2.2.1. The spreadsheet is shown in Appendix I (b).

2.2.1 Format

In the spreadsheet for scale down, the actual volumes of material were of interest in relation to the size of the equipment available. In this case the fermentation volume required was back calculated on the basis of using a 40 mL homogeniser at a fixed cell concentration. Fermenter volume was based on a 20% increase of the estimated working volume. Cell concentration factor was fixed at 3.45 according to Bartholomew and Reisman (1979).

The enzyme ADH was chosen as a typical example of a soluble protein for the product yield calculations. ADH activity units were taken from Milburn (1990), where activity increased by 45% on flocculation with PEI. Estimates of enzyme activity were calculated from the completed mass balance.

The volumetric mixing ratio for flocculant was increased to 1:1 for ease of handling at this scale.

Solids phase removal was carried out in two primary stages: bulk solids removal modelled on the scroll decanter centrifuge, followed by clarification modelled on the disc stack centrifuge. The scroll decanter centrifugation allowed 40% carryover of solids into the product stream and sediment dewatering at 30% dwt/wwt (section 5.5.3). The supernatant was clarified in a disc stack centrifuge with 10% solids carryover and 14% dwt/wwt sediment (Mosquira et al 1981).

A final polishing step based on filtration was introduced before purification. The solids load on the filters was reduced four-fold by the centrifugation stages. The filter polish prevented any contaminating solids material from continuing in the process stream.
A chromatographic purification was used where the size of the column was calculated according to the protein contained in the filtrate and a typical column loading capacity (Q-Sepharose, Pharmacia, Sweden). The manufacturer also supplied details of typical protein yield, purification factor and DNA clearance. Protein yield was used to calculate the mass of protein recovered in the elution buffer and that retained on the column. DNA clearance was used for the removal of all nucleic acids. All other contaminants were assumed to be cleared in 8x the quantity of those eluted (Manufacturer's typical purification factor). The overall product yield was found by multiplying all individual step protein yields.

2.2.2 Results

This was a first attempt at a scale down study of an entire process, completed with the intention of getting an approximate idea of the volumes involved. Each individual unit operation would have to be scaled down in sequence to test if the overall yield of soluble protein is 53% in practice.

The scaled down mass balance was based on using a small high pressure homogeniser (Alfa Laval Microlab 40) with a 40 mL sample of concentrated yeast cells (207 g L\(^{-1}\)). This would require a 480 mL fermenter: 500 mL miniature fermenters are commercially available. Alternatively, packed yeast re-suspended in buffer could be used for initial practical studies of the downstream operations, omitting the fermentation stage.

It was anticipated that mixing for flocculation could be scaled down using a suitable power correlation, and filtration and chromatography could be scaled down using small batch tests as described by Gooding (1991) and Hinrichsen (1985) respectively.

The greatest problem in scaling down the process was anticipated in finding centrifuges which handle 66-80 mL feed, which at the same time operate in such a way as to mimic pilot scale equipment in terms of solids recovery. However, it may be possible to use instrumentation to scale down dewatering. From the mass balance calculations, the scaled down process should yield 10-12 mL sludge, which is just enough for tests using the Bohlin VOR rheometer.
Figure 2.2.1: Scale down mass balance flow sheet

Fermentation  Harvest  Homogenisation  Flocculation  Dewatering  Clarification  Filtration  Chromatography

Mini-fermentation or cell resuspension  Microlab 40 homogenisation  Mixing Gt correlation  ?  ?  Batch filter  Batch chromatography

Numeric values refer to volumes, given in mL.
3. THEORY

3.1 Separation efficiency

3.1.1 Mass balance Equations

A separating unit operation recovers solid particles from a continuous liquid phase. Assuming no accumulation of particulate material then:

\[ M_o = M_c + M_f \]

where

- \( M \) = mass flow rate \( (\text{kg s}^{-1}) \)
- \( o \) = initial (feed) particles
- \( c \) = coarse particle fraction
- \( f \) = fine particle fraction

If there is no change in particle size inside the separator (no disruption nor agglomeration), then the mass balance also applies to each particle size component. The particle size distribution frequency gives the fraction of particles of size \( x \) in a sample. Therefore the total mass of particles of a certain size range \( \Delta x \) present in a process stream is the mass flow rate multiplied by the appropriate size fraction. Hence:

\[ M_o \frac{\Delta F_o}{\Delta x} = M_c \frac{\Delta F_c}{\Delta x} + M_f \frac{\Delta F_f}{\Delta x} \]

3.1.2 Total efficiency

The total separation efficiency of particles or mass yield \( (E_t) \), is defined as:

\[ E_t = \frac{M_c}{M_o} \quad \text{or} \quad E_t = 1 - \frac{M_f}{M_o} \]

These equations for separation efficiency can be substituted into the mass balance equation (45) in terms of the particle components:

\[ \frac{\Delta F_o}{\Delta x} = E_t \frac{\Delta F_c}{\Delta x} + (1 - E_t) \frac{\Delta F_f}{\Delta x} \]
3.1.3 Grade efficiency

It is very rare for a unit operation to yield a sharp separation where the process stream is divided entirely into particles above and below a specified size.

More diffuse separations are achieved in practice where the separation efficiency of a particular piece of equipment changes with particle size. This changing performance is described by the grade efficiency $T(x)$ and grade efficiency curves are obtained by plotting the separation efficiency for every particle size $x$:

$$T(x) = \frac{(M_c)_x}{(M_o)_x}$$

or in terms of each particle size component:

$$T(x) = M_c \frac{\Delta F_c}{\Delta x} = M_o \frac{\Delta F_o}{\Delta x} = E_i \frac{\Delta F_c(x)}{\Delta F_o(x)} = 1 - (1 - E_i) \frac{\Delta F_f(x)}{\Delta F_o(x)}$$

Grade efficiency curves are typically S-shaped as shown in Figure 3.2.2 in section 3.2.1. They show the probability of particles of a certain size being recovered. For example, if many particles of size $x_{50}$ enter the separator, then it is likely that 50% of them will follow trajectories which will allow them to settle out. "$x_{50}$" is called the equiprobable cut size for separation. By contrast, $x_{100}$ or $x_{\text{max}}$ is the largest particle which may remain in the fine particle stream, and is called the limit of separation. Where diameter is used as a measure of the size of spherical particles, then $x_{50}$ and $x_{100}$ are usually termed $d_{50}$ and $d_{100}$ respectively.

In applying the grade efficiency concept to centrifugal separation, particle size analysis is usually carried out on the feed and supernatant. The mass yield is first calculated from:

$$E_i = 1 - \Sigma_{\Delta d_{\text{max}}} [1 - T(d)] \Delta F_o(d)$$

65
where \( d_{\text{min}} \) and \( d_{\text{max}} \) are the minimum and maximum particle sizes present in the feed suspension. It is assumed that negligible fluid is lost in the solids discharge. Equation (48) then takes the form:

\[
T(d) = 1 - (1 - E_i) \frac{\Delta F_f(d)}{\Delta F_o(d)}
\]

The particle size distributions shown here are the percent-in-range distributions so that the sum over all the size fractions is 100%. Particle size measurement is restricted to particles with diameter 1 \( \mu m \). It is therefore necessary to assume that sub-micron particles do not make a significant contribution to the total mass of particles. For this reason, particle size distributions are expressed in terms of particle volume, where the population of each size fraction is given spherical volume \( (4/3)\pi(d^3/9) \). This tends to weight the size distributions in favour of the larger particles.

Grade efficiency curves are constant for any given set of operating parameters such as fluid viscosity, flow rate and initial solids concentration. However, it is possible to make grade efficiency curves applicable to changes in solid-fluid density difference (\( \Delta \rho \)) and viscosity (\( \mu \)) by converting the particle size scale using Stoke's law so that:

\[
\frac{x_1}{x_2} = \sqrt{\frac{\mu_1 \Delta \rho_2}{\mu_2 \Delta \rho_1}}
\]

When generating grade efficiency curves to describe separation efficiency in a centrifuge, the particle size axis is divided by \( d_c \) to create a dimensionless parameter “\( d/d_c \)” (see section 3.2.1). This enables comparison of the grade efficiency curves for many systems at any flow rate, provided that the correct values for liquid viscosity and particle density inside the centrifuge are known constants. The conversion contains all the assumptions associated with Stoke's law (section 3.2), as well as assuming that particle trajectories in the equipment remain unaffected by changes in fluid viscosity.
3.1.4 Reduced efficiency

In some centrifuge operations where there is a significant underflow it is necessary to take into account the volumetric split in order to observe the net separation effect:

\[ E^*_t = \frac{E_t - R_f}{1 - R_f} \]  

where

- \( E^*_t \) = "reduced" efficiency
- \( R_f = \frac{Q_{sed}}{Q_o} \) underflow to throughput ratio

In cases where \( Q_{sed} \) contains compressible solids or is difficult to measure, then \( R_f \) can be found from a mass balance against the supernatant:

\[ R_f = 1 - \frac{Q_{sup}}{Q_o} \]  

In practice, \( E^*_t \) is usually determined from the solids concentration in the supernatant \( C_{sup} \) relative to the solids concentration in the feed \( C_o \), (Svarovsky 1990). This form is also called the clarification number \( C_N \):

\[ E^*_t = C_N = \frac{C_o - C_{sup}}{C_o} \]

Reduced separation efficiency may also be found using:

\[ E^*_t = \frac{(C_{sed} - C_o)(R_f)}{C_o(1 - R_f)} \]  

where \( C_{sed} \) = solids concentration in the underflow

and:

\[ E^*_t = \frac{C_{sed} - C_{sup}}{C_{sed} + C_{sup} + \left( \frac{C_{sup}}{R_f} \right)} \]

Hence there are three alternative ways of conveniently measuring the reduced efficiency.
3.2 Sigma factor theory for centrifugal separation

The sigma value concept first proposed by Ambler (1952) is used for predicting separation characteristics in industrial centrifuges. It may be derived by considering the trajectory of a particle in a simple tubular centrifuge as shown in Figure 3.2.1. It is assumed that the particle suspension entering the centrifuge is instantaneously accelerated to the angular velocity of the rotating cylinder, and that it is homogenously distributed in the annulus between the radii \( r_1 \) and \( R \).

**Figure 3.2.1:** Schematic diagram of suspension flow through a tubular bowl centrifuge for the recovery of a single particle.

![Diagram](image)

where \( r \) = radial position of a settling particle

\( z \) = axial position of a settling particle

A particle enters the bottom of the centrifuge at the smallest radius at which the sedimentation process may begin, and continues until it is just separated out at the bowl radius at the top end of the centrifuge. The resulting
parabolic velocity profile of the particles may be resolved into radial and axial components.

Assuming the suspension is dilute, and there is no particle interaction, then Stoke's law can be used to define the radial velocity component:

\[
\frac{dr}{dt} = \frac{\Delta \rho x^2 r \omega^2}{18 \mu}
\]

56

It is assumed by using Stoke's law that as particles move from \( r_1 \) to \( R \), their instantaneous velocity at any point is equal to the terminal settling velocity corresponding to the centrifugal acceleration \( r \omega^2 \).

The axial velocity component is assumed to evolve from "plug flow" in which the velocity profile in the liquid shell is uniform:

\[
\frac{dz}{dt} = \frac{Q}{\pi (R^2 - r_1^2)}
\]

57

The ratio of equations 58 and 59 are integrated between the limits \( r = R \) at \( z = L \), and \( r = r_1 \) at \( z = 0 \). The position of the particle at any radius \( r \) may then be described

\[
\ln\left(\frac{R}{r}\right) = \frac{K_2 x^2 L \pi (R^2 - r_1^2)}{Q}
\]

58

where

\[
K_2 = \frac{\Delta \rho \omega^2}{18 \mu}
\]

59

and

\[
\frac{L \pi (R^2 - r_1^2)}{Q} = \frac{V}{Q}
\]

60

Hence the position of settling particles is proportional to their sedimentation velocity and the liquid residence time inside the centrifuge.
At the limit of separation this yields:

\[ x_{\text{max}} = \frac{Q \ln \left( \frac{R}{r_1} \right)}{K_2 \pi L (R^2 - r_1^2)} \quad 61 \]

Alternatively, the equiprobable cut size \( x_{50} \) has a corresponding radius \( r_{50} \) which splits the annulus between \( r_1 \) and \( R \) into equal areas:

\[ R^2 - r_{50}^2 = r_{50}^2 - r_1^2 \quad 62 \]

If \( r_{50} \) is substituted into equation 61, then it is possible to find \( x_{50} \) from:

\[ x_{50}^2 = \left( \frac{Q}{2 \pi L K_2} \right) \left[ \ln \left( \frac{2 R^2}{R^2 + r_1^2} \right) \right] \left( \frac{1}{R^2 - r_1^2} \right) \quad 63 \]

The cut-size \( x_{50} \) was represented by its terminal velocity \( u_g \) in the given liquid under gravity so that from Stoke's law:

\[ u_g = \frac{x_{50}^2 \Delta \rho g}{18 \mu} = x_{50}^2 K_2 \frac{g}{\omega^2} \quad 64 \]

Combining equations 63 and 64 yields:

\[ Q = 2 u_g \left( \frac{\omega^2}{g} \right) \pi L \left[ \frac{R^2 - r_1^2}{\ln \left( \frac{2 R^2}{R^2 + r_1^2} \right)} \right] \quad 65 \]
This expression may be simplified to the general form for $\Sigma$ theory as shown in equation 66.

$$Q = 2u_s \Sigma$$

where:

$$\Sigma = \left( \frac{\omega^2}{g} \right) \pi L \left[ \frac{R^2 - r_i^2}{R^2 + r_i^2} \ln \left( \frac{2R^2}{R^2 + r_i^2} \right) \right]$$

This equation simplifies the expression of centrifuge performance in terms of $x_{50}$ into a relation between volumetric flow rate and an index of centrifuge size, the $\Sigma$-factor.

The dimensions of $\Sigma$ are [Length$^2$]. It is said to yield a settling area equivalent to a gravity settling tank of the same separation capacity. Hence a centrifuge with equivalent settling area ($A_e$ or $\Sigma$) 10 000 m$^2$ has the same separation performance as a gravity settling tank of 100x100 m.

In applying Stoke's law to centrifugal settling it is assumed that the suspended particles are discrete, small, inert spheres settling under laminar flow conditions (Reynolds number less than 0.5). It is also assumed that the suspension is evenly distributed, dilute enough to preclude any interference between settling particles (no hindered settling), and that there is no solids re-entrainment once settled.

### 3.2.1 Theoretical grade efficiency for ideal settling according to Stoke's law

It is possible to define a specific throughput capacity ($q_s$) where particles with settling velocity $u_g$ greater or equal to $q_s$ are removed from the continuous liquid phase:

$$q_s = \frac{Q}{A_e} = u_{g,c} = \frac{d_i^2 \Delta \rho g}{18 \mu}$$

71
The gravitational settling velocity of a particle must be at least equivalent to a critical rate \( u_{g,c} \) to be certain of settling out. Since the characteristic settling velocity of a particle depends most importantly on its size, then the above expression is usually written in terms of a critical particle diameter \( d_c \):

\[
d_c = \sqrt{\frac{18 \mu Q}{A_c \Delta \rho g}}
\]

Only particles of diameter greater or equal to \( d_c \) will be recovered in the centrifuge. The probability of particles being recovered is described by the grade efficiency \( T \):

\[
T = \frac{u_g}{u_{g,c}}
\]

For particles with \( u_g > u_{g,c} \) \( T=1 \)

ie. for particles with \( d > d_c \) \( T=1 \).

For particles with \( u_g < u_{g,c} \) \( T<1 \)

and according to Stoke's law if \( d < d_c \) \( T=(d/d_c)^2 \).

The grade efficiency curve generated by ideal settling according to Stoke's law is shown in Figure 3.2.2.

In practice some particles with a velocity less than \( u_{g,c} \) or diameter less than \( d_c \) may settle out due to variations in particle trajectories. In the disc stack centrifuge, particles are evenly distributed at the entrance to the disc space, and so a small particle starting close to the lower surface of the upper disc at \( R_o \) follows a short particle trajectory which allows it to be recovered. The grade efficiency curve showing such a variation in performance is also shown in Figure 3.2.2.
Figure 3.2.2: Theoretical and real grade efficiency curves for centrifugal separation with definitions of $d_{50}$ and $d_{\text{max}}$.

---

Theoretical curve for ideal settling of spherical particles according to Stoke's law,

Standard curve for a dilute aqueous suspension of PVAc settling in a disc stack separator (Westfalia BSB, Mannweiler 1992)
3.2.2 Curve-fitting for grade efficiency

Grade efficiency curves may be transformed into mathematical expressions which can be described accurately with only a few parameters. The two parameter Rosin-Rambler-Sperling-Bennet (RRSB) function is normally used for describing particle size distribution curves (Svarovsky 1990). The function was converted for use with grade efficiency curves as described in Mannweiler (1990):

\[ T(d) = 1 - \exp\left[-\left(k_3 \frac{d}{d_c}\right)^{n_2}\right] \]

where \( n_2 \) is found from the slope of the straight line:

\[ \ln\left[\ln(1 - T(d))\right] = n_2 \ln(k_3) + n_2 \ln\left(\frac{d}{d_c}\right) \]

and \( k_3 \) is found from the intercept on the ordinate:

\[ k_3 = \exp\left(\frac{\text{intercept}}{n_2}\right) \]

The curve given by Mannweiler (1992) for a scaled down disc stack centrifuge (Westfalia BSB) had parameters \( k_3 = 0.865 \) and \( n_2 = 2.08 \) and it is this curve which is shown in Figure 3.2.2.

3.2.3 \( \Sigma \) value for a scroll decanter centrifuge

In a scroll decanter centrifuge fitted with a negative ring dam, where feed suspension does not enter the conical section of the bowl, \( \Sigma \) is essentially the same as for a tubular bowl centrifuge (equation 69). This can be simplified by an expansion of a logarithmic function as described in Svarovsky (1990). Hence:

\[ \Sigma = \frac{\omega^2}{g} \pi L \left( \frac{3}{2} R^2 + \frac{1}{2} r_l^2 \right) \]

In batch centrifugation, both the area available for settling and the acceleration radius tend to decrease as the bowl fills with solids. Ambler (1959)
suggests a reduction factor of 0.67 for a cylindrical bowl to account for this effect. In addition, the conveyor flights also displace their own volume of liquid inside the centrifuge. Ambler (1959) suggests that 6% should be deducted from the theoretical Σ value to compensate for a solid type conveyor.

3.2.4 Σ value for a laboratory bottle centrifuge

The derivation of Σ value in a laboratory bottle centrifuge is given in Trowbridge (1962):

$$\Sigma_b = \frac{\omega^2 V}{2g \ln \left( \frac{2r_2}{r_1 + r_2} \right)}$$

where

\begin{align*}
  r_1 &= \text{radius to top of liquid suspension (m)} \\
  r_2 &= \text{radius to top of sedimenting solids (m)}
\end{align*}

The bottle centrifuge has no volumetric throughput, but residence time inside the centrifuge can be related to Q/Σ as shown in Trowbridge (1962):

$$\frac{Q_b}{\Sigma} = \frac{4.6 \log_{10} \left( \frac{2r_2}{r_1 + r_2} \right)}{\omega^2 t}$$

3.2.5 Σ value for a disc stack centrifuge

In the disc stack centrifuge, the settling area occupies a much smaller volume than that of the equivalent tubular bowl or multichamber centrifuge, i.e. it is more compact. The equivalent settling area is derived by considering the trajectory of a particle starting at the outside radius (R_o) on the upper surface of two discs and settling on the lower edge of the upper disc at the inner disc radius (R_i) (Ambler 1952). The particle trajectory is resolved into radial and axial velocity components (relative to the discs) as shown in Figure 3.2.3.
Figure 3.2.3 Schematic diagram of suspension flow through a single gap of a disc stack centrifuge for the recovery of a single particle.

The radial particle settling velocity \( v_r \) is:

\[
v_r = \frac{dh}{dt \cos \theta}
\]

and the axial particle velocity relative to the discs is assumed to be the same as the velocity of the continuous liquid phase under plug flow conditions:

\[
v(h) = \frac{dr}{dt \sin \theta}
\]

where
- \( h \) = disc gap width (m)
- \( Q \) = throughput for which a particle is just recovered \((m^3 \cdot s^{-1})\)
- \( \theta \) = half disc angle (radians)
- \( z \) = No. discs in stack
The ratio of the particle velocity components yields a differential quotient in terms of radius \( r \):

\[
\frac{v_r}{v(h)} = \frac{dh}{dr} \tan \theta \tag{79}
\]

for \( 35^\circ > \theta > 50^\circ \), as in all disc stack centrifuge designs.

However, the flow velocity may also be described as a function of the radius of rotation:

\[
v(h) = \frac{1}{2\pi r} \frac{dq}{dh} \tag{80}
\]

where \( q = \) single passage throughput \( (m^3 \text{ s}^{-1}) \)

Substituting \( v(h) \) into equation 79 and rearranging for \( dq \) yields:

\[
dq = v_r, 2\pi r \cot \theta dr \tag{81}
\]

Using Stoke's law, the centrifugal settling velocity of a particle \( (v_r) \) is:

\[
v_r = u_s \frac{\omega^2 r}{g} \tag{82}
\]

and substituting this into equation 81 yields:

\[
dq = u_s \frac{2\pi \omega^2}{g \tan \theta} r^2 dr \tag{83}
\]
This equation is now ready to be integrated between the limits \( q = q \) at \( r = R_o \), and \( q = 0 \) at \( r = R_i \):

\[
q = u_s \frac{2\pi \omega^2}{3g \tan \theta (R_o^3 - R_i^3)}
\]

84

The total throughput capacity is equivalent to the whole stack containing \((z)\) number of discs:

\[
Q = u_s \frac{2\pi \omega^2 z}{3g \tan \theta (R_o^3 - R_i^3)} = u_s \Sigma
\]

85

\( \Sigma \) again has the dimensions \([\text{Length}^2]\), and can be compared to a gravity settling tank.

In practice, \( \Sigma \) values tend to be lower than those given in equation 85 due to the spacer ribs which support and separate the active discs. Not only do they occupy a significant area on the discs, but they also cause flow vortices along the axis of rotation close to the disc surfaces (Fitch 1965). For this reason a correction factor \((f_i)\) is normally applied to the equivalent settling area:

\[
\Sigma = \frac{2}{3g} \frac{\pi \omega^2}{\cot \theta (R_o^3 - R_i^3)} f_i
\]

86

where

\[
f_i = 1 - \frac{3z_i b_i}{4\pi R_o} \frac{1 - (R_i/R_o)^2}{1 - (R_i/R_o)^3}
\]

87
and \( z_l = \) number of caulks
\( b_l = \) caulk width (m)

### 3.3 Scrolling efficiency in a scroll decanter centrifuge

A number of forces act against the movement of solids towards the conical section and to discharge in a scroll decanter centrifuge. They are centrifugal force (particularly in the conical section), friction from the bowl and friction from the scroll flights. If solids are to move in an axial direction along the bowl then the normal force from the scroll (supplied by its torque) must be greater than all the above forces when they are resolved perpendicularly to the scroll flight. Figure 3.3.1 shows the path of a solid particle during one revolution of the scroll.

**Figure 3.3.1:** Movement of solids during one revolution of the conveyor in a scroll decanter centrifuge

![Diagram](image)

where \( \beta = \) screw angle \((p/2\pi R)\) (°)
\( \delta = \) solids path angle (°)
\( a = \) axial distance moved by solids (m)

The actual path taken by the particle is from A to B, so that it covers a distance \( a \) in the axial direction. Using simple trigonometric relationships, it is possible to find the distance BC:
Scrolling efficiency is defined as the ratio of the axial movement of solids per scroll revolution divided by the pitch, \((a/p)\), so that:

\[
BC = a \tan \delta = \frac{p-a}{\tan \beta}
\]

The highest scrolling efficiency is obtained when the solids path angle \((\delta)\) is equal to the screw angle \((\beta)\):

\[
\left( \frac{a}{p} \right)_{\text{max}} = \frac{1}{1 + (\tan \beta)^2}
\]

It would appear from these relations that scrolling efficiency could be increased by decreasing the pitch, in theory down to the limit of the finite volume of the scroll winding. However, in practice this also tends to increase the scroll friction so that "bridging" occurs, where solids turn with the conveyor.

### 3.3.1 Solids residence time

The number of revolutions a particle makes when travelling down the entire length \((L_c)\) of a scroll decanter centrifuge is found from:

\[
\frac{\text{No. revs}}{\text{distance}} = \frac{\text{No. turns}}{\text{length}} = \left( \frac{L_c}{p} \right) = \frac{l}{p}
\]
It is assumed that the scrolling efficiency in the conical section of the centrifuge is the same as in the bowl section ie. there is no solids accumulation.

The velocity of the particle is found from the number of revolutions it makes per second, ie. the conveyor differential speed relative to the bowl ($\Delta \omega$) and the number of turns made per unit distance:

$$Velocity \ of \ a \ solid \ particle = \frac{\Delta \omega}{\frac{l}{p}} = \Delta \omega p$$

The residence time of the particle inside the centrifuge can be found from:

$$Residence \ time \ of \ sediment = \frac{d}{\Delta \omega p}$$

where $d = \text{solids path length} \ (L_e / \cos \delta)$

and $\delta$ is found from the scrolling efficiency:

$$\tan \delta = \frac{1}{\left(\frac{a}{p}\right) \tan \beta} - \frac{1}{\tan \beta}$$

Estimates of scrolling efficiency can be used to generate solids residence time data for a range of conveyor differential speeds using this method.
4. MATERIALS AND METHODS

4.1 Materials

*Saccharomyces cerevisiae* (baker's yeast) of consistent quality was supplied by Distillers Co. Ltd., Surrey.

Phosphate buffer was prepared from general purpose NaH$_2$PO$_4$ (MW 142) and Na$_2$HPO$_4$ (MW 156) supplied by Sigma Chemical Co., Poole, Dorset.

Other general purpose reagents used were: sodium hydroxide pellets (NaOH), concentrated hydrochloric acid (HCl) and concentrated phosphoric acid (H$_3$PO$_4$) all supplied by BDH, Merck Ltd., Poole, Dorset.

Polyethylenimine (PEI) of nominal MW 10 000 was supplied in 50% aqueous solution both in bulk (BASF, Cheadle Hulme, Cheshire) for pilot scale experiments, and in smaller 1 L quantities (Sigma) for laboratory and some pilot scale work. All given concentrations of PEI are absolute values.

Hydrated crystals of sodium tetraborate (Borax) (Na$_2$B$_4$O$_7$.10H$_2$O, MW 381.4) supplied by Sigma as above.

An aqueous suspension of polyvinyl acetate (PVAc or "latex") particles was kindly donated by Hoechst, Frankfurt, Germany.

4.2 Equipment

4.2.1 Laboratory centrifuges

Experimental work involved the use of several different laboratory centrifuges:

- the bench-top B12401 centrifuge (Denley) fitted with an angled rotor, capacity 8x20 mL, with maximum speed 5600 rpm, equivalent to 3000g.

- the Europa 24M laboratory centrifuge (MSE, Crawley, West Sussex) fitted with an angled rotor of 8x20 mL capacity, in experiments here used at speeds not exceeding 8000 rpm (maximum RCF 10 000g).

- the bench-top variable speed 14x1.5 mL Eppendorf centrifuge (Eppendorf Geratebau, Netheler + Hinz GmbH, 2000 Hamburg 63), maximum RCF 14 000g, with $r_1$ 0.042m and radius to bottom of tube ($r_3$) 0.073 m.
4.2.2 Homogenisers

For experimental and pilot scale work requiring not more than 2 L of material, the Lab60 homogeniser (Lab60, APV, Crawley, W.Sussex) was used to disrupt suspensions of yeast cells. This was a two-piston, positive-displacement homogeniser equipped with a knife-edged valve and facility for operating discrete passes.

For pilot scale work requiring more than 20 L homogenised yeast, the K3 homogeniser (K3, APV) was used. This is a three-piston, positive-displacement industrial-scale homogeniser with flow capacity 280 L h⁻¹.

Both homogenisers were equipped with plate heat exchangers downstream for cooling the disrupted cells to 5°C. Experimental work was continued at ambient temperatures (20-25°C) since neither centrifuge was equipped with a cooling facility.

4.2.3 Feed Pumps to Centrifuges

Suspensions were fed to the pilot scale centrifuges at constant flow rates by positive displacement lobe pumps (Model 50 NDM, SSP Pumps Ltd., Eastbourne, East Sussex). These were controlled by a microprocessor (System 6366, Turnbull Control Systems Ltd., Worthing, West Sussex) coupled to magnetic flow meters (Turbo-Werk GmbH, Cologne, Germany).

Pump No.1 alone was sufficient to supply the pilot scale centrifuges at flow rates of 480 L h⁻¹ or less, as shown in Figure 4.2.1 a. Where flow rates greater than 480 L h⁻¹ were required, then a coupling system was used after two pumps, in order to double the flow rates up to a maximum 960 L h⁻¹, as shown in Figure 4.2.1 b. Non-return valves positioned downstream of the flow meters ensured correct direction of the process stream.

In one experiment, PEI and disrupted yeast suspensions were mixed at the point of entry to the centrifuge bowl using a concentric feed pipe arrangement as in Figure 4.2.1 c, in order to investigate the effect of shear in the feed zone.
Figure 4.2.1: Flow diagrams for industrial centrifuges. (a) scroll decanter or disc stack centrifuges, (b) disc stack centrifuge, (c) scroll decanter centrifuge flocculating in the feed zone.

Key: FE = flow element  FT = flow transmitter
M = microprocessor  SC = speed control

(a) 30-540 L/h

(b) 60-1080 L/h

(c) 60-1080 L/h
Figure 4.2.1 also shows the mixing tanks used in the pilot scale trials. The primary tank was a 100 L vessel fitted with an angled 2-bladed marine propeller. A 250 L tank fitted with a similar mixing device was used as a secondary vessel.

4.2.4 Pilot scale scroll decanter centrifuge

4.2.4.1 Centrifuge dimensions

The centrifuge used primarily for dewatering studies was a P600 Decanter Centrifuge (Alfa-Laval Sharples, Camberley, Surrey) operated at a bowl speed of 6000 rpm and generating a relative centrifugal force of 3000 g. The centrifuge dimensions are given in Figure 4.2.2 and Table 4.2.1.

<table>
<thead>
<tr>
<th>L</th>
<th>total length</th>
<th>360</th>
<th>mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>D</td>
<td>bowl diameter</td>
<td>150</td>
<td>mm</td>
</tr>
<tr>
<td>P</td>
<td>conveyor pitch</td>
<td>50</td>
<td>mm</td>
</tr>
<tr>
<td>t</td>
<td>conveyor ribbon thickness</td>
<td>12</td>
<td>mm</td>
</tr>
<tr>
<td>$h_w$</td>
<td>weir height (pond depth)</td>
<td>13.1, 16.2, or 19.3</td>
<td>mm</td>
</tr>
<tr>
<td>a</td>
<td>beach angle</td>
<td>$10^\circ$</td>
<td></td>
</tr>
<tr>
<td>b</td>
<td>screw angle</td>
<td>$6^\circ$</td>
<td></td>
</tr>
<tr>
<td>L/P</td>
<td>No. turns of conveyor</td>
<td>7.2</td>
<td></td>
</tr>
<tr>
<td>L/D</td>
<td>$\lambda$ ratio</td>
<td>2.4</td>
<td></td>
</tr>
</tbody>
</table>

The bowl incorporated a soft feed unit which doubled as a negative ring dam to prevent clarified liquor entering the beach section. The gear box was of planetary type hence conveyor differential speeds are given where the conveyor rotates slower than the bowl. The scroll winding was of a helical ribbon type, allowing axial flow of supernatant to the overflow weir. In the majority of
centrifuge trials a deep pond setting of 19.3 mm was used, although shallower ponds of 16.2 mm and 13.1 mm were also used.

4.2.4.2 Centrifuge control

An electronic "Advanced Brake Control" (ABC) unit was used in early experiments to control the conveyor differential speed relative to the bowl speed. A torque probe on the conveyor hub sent current signals to the ABC so that torque developed by scrolling solids was used to control the conveyor differential speed via an eddy current brake attached to the conveyor, as shown in Figure 4.2.3 a. This system gave adequate control within a 10-50 rpm differential speed range. In all experiments with the ABC, the centrifuge was operated by choosing a differential speed setpoint, and monitoring the torque generated.

In order to gain greater control at very low conveyor differential speeds, an "Advanced Back Drive Controller" (ABDC) was fitted, together with a bowl speed probe and back drive motor attached to the conveyor. The bowl speed probe enabled the ABDC to monitor and reduce fluctuations in bowl speed via the main drive motor. The torque current signals from the probe on the conveyor hub were then used to control the differential speed via the back drive motor, as shown in Figure 4.2.3 b. This system gave close control of the conveyor and bowl speeds, so that conveyor differentials as low as 1 rpm could be achieved.

With the addition of the ABDC, it was also made possible to operate the centrifuge by choosing a controlling torque setpoint, and monitoring the resultant differential speed.
Figure 4.2.2  Lengthways cross section schematic diagram of P600 scroll decanter centrifuge. Dimensions given in mm.
**Figure 4.2.3:** Schematic control diagrams for the scroll decanter centrifuge, (a) ABC system drives bowl and controls conveyor speed by braking. (b) ABDC controls bowl speed and conveyor speed via independent motor drives.
4.2.5 **Pilot scale disc stack centrifuge**

The centrifuge used primarily for scale down studies was a high speed SA00H-205 disc stack (Westfalia, Oelde, Germany) operating at 10 000 rpm and generating a maximum RCF of 8660g at the bowl radius. The feed zone was non-hermetic, and the supernatant was discharged under a back pressure of 1.5 bar g via a centripetal pump located above the stack. The dimensions of the bowl and discs are shown in Figures 4.2.4 and 4.2.5 respectively.

The SA00H-205 was fitted with a full stack of 38 active discs, or a 25% stack of 9 active discs. An active disc is shown in Figure 4.2.5. It has spacer ribs which allowed fluid to flow between the stacked discs. The inactive section of the stack was filled with 48 "blank" discs, which did not have spacer ribs and formed an impenetrable block.

The active discs had six, radially-arranged holes, which when stacked together formed riser channels through which bulk fluid could flow, following the path of least resistance to centrifugal pressure. In one experiment, the blank discs positioned below the active discs in the scaled down stack also contained channel risers.

4.3 **Instrumentation**

4.3.1 **Rheological characterisation**

A model VOR Bohlin rheometer (Bohlin Reologi, Lund, Sweden) interfaced with a Dell 386 personal computer was used for continuous (unidirectional) and dynamic (oscillatory) measurements. Unidirectional shear measurements were used to measure the viscosity of flocculated suspensions of homogenised yeast, whereas oscillatory measurements were used to characterise the viscoelastic nature of their sediments.

Viscous suspensions of flocculated yeast homogenate were sheared at rates between 0 and 1600 s⁻¹ using a calibrated torsion bar of nominal size 40 g mm to measure the resultant shear stress in the C25 cup and bob system (capacity 17 mL). Viscosity measurements were collected within 1-99% of the operating range of the torsion bar.
Figure 4.2.4: *Westfalia SA00H-205 disc stack centrifuge.*
Dimensions given in mm.

\[ \begin{align*}
\theta &= \text{disc angle} = 0.68 \text{ radians} \\
b_1 &= \text{caulk width} = 0.5 \text{ mm} \\
z_1 &= \text{No. caulks} = 6 \\
z &= \text{No. discs} = 38 \\
V &= \text{bowl volume (including sediment holding space)} = 0.6 \text{ L}
\end{align*} \]
Figure 4.2.5: Westfalia SA00H-205 disc stack centrifuge: detail of active disc.

(a) Elevation

(b) Plan

Channel riser
Caulk

All dimensions given in mm.
In oscillatory experiments, interchangeable torsion bars of nominal sizes 40, 180, 900 and 3000 g mm were used to cover a range of shear modulus measurements from $10^{-4}$ to $10^4$ Pa. Correction factors for torque measurement errors were maintained below 0.3, and operating range restricted to between 1-99%.

Samples of dewatered sediment (approximately 1 mL) were placed onto the lower of a pair of parallel plates (pp30), and the top plate lowered gently until the gap width was 1 mm, and filled with homogenous sediment. Any excess material was carefully trimmed and wiped away before covering the plates with a metal casing to limit further drying of the sample. Torque was checked for a steady reading to ensure that the sample was at rest, and readjusted to zero before the start of each experiment.

A strain sweep test at a fixed frequency of 1 Hz was carried out for each sample in order to measure rheological parameters as a function of strain amplitude. At very small strains, $G^*$, $G'$, $G''$ and $\delta$ are independent of the applied strain magnitude at any given frequency. This operating region is called the linear viscoelastic region (LVR). Viscoelastic theory as outlined in Appendix II is applicable below the limit of the LVR, which is determined during the strain sweep test.

Once the LVR limit was indicated, then more oscillatory measurements were made, but at a range of frequencies at a fixed strain amplitude set within the LVR. The frequency range used was 0.01-10 Hz. Data for further processing with respect to bulk flow of sediment in the centrifuge was taken at 5 Hz (Ward and Hoare 1990).

Oscillatory shear experiments were repeated twice, to give three consistent measurements within +/- 5% of the mean.

4.3.2 Particle size analysis

Simultaneous measurement of particle size distribution and concentration was achieved using the electrical sensing zone method (Elzone 280PC, Particle Data Ltd., Kingstone).
Size analysis was carried out using a 18 µm orifice tube, suitable for measuring particles between 1.08 µm and 7.2 µm in diameter. The tube was calibrated using monodisperse particles of latex (PVAc) of diameters 1.09 µm and 2.02 µm. A narrow logspan was used to ensure that the majority of the 128 size channels measured lay within the calibrated range.

Prior to size analysis the supernatant or feed sample was accurately diluted in 10% (w/w) 0.1 µm filtered sodium chloride (Polycap 75TF PTFE membrane filter capsules, Whatman International Ltd., Maidstone, Kent). This is a low resistance solution which allowed the use of a high aperture current, which is required for such a small orifice size.

20 mL of suspension was analyzed at under constant vacuum of 160 mbarg, to give a maximum 6000 counts. This was well below the 22 000 limit for 1% coincidence during the time for a fixed 20 mL volume measurement. The counting procedure was repeated twice and averaged with a correction for electrolyte background noise if necessary. However, this was generally found to be negligible at a maximum 300 counts per sample.

4.4 Laboratory Analytical Techniques

4.4.1 Clarity of supernatants
Absorbance or light transmission of feed and centrifuge supernatants was read at 690 nm against water in a spectrophotometer (PU 8800 Philips). Three samples of supernatant were deposited in disposable cuvettes and the result automatically averaged over a 2 second period. Samples were diluted if necessary to read within the linear range of absorbance.

4.4.2 Dry Solids Concentration Analysis
The dry solids concentrations of centrifuge supernatants were determined by pipetting 3x 1.5 mL samples into pre-weighed plastic Eppendorfs. The mass of Eppendorfs containing supernatant was recorded before spinning down the solids at 14 000 g for 300 s. The sediments were resuspended in 0.2 µm filtered water to rinse out soluble protein entrained in the wet solids. The solids were resedimented, again at 14 000 g for 300 s, and stored in a drying oven at 105°C
for 48 h to constant dry weight. The dry weight as a percentage of wet weight was determined using the formula (Vesilind 1990):

\[
\% \frac{Dwt}{Wwt} = \frac{Drymass - \text{eppendorf}}{Wetmass - \text{eppendorf}} \times 100 \%
\]

Since a known volume of material was used, then this could be recalculated to give a solids concentration in (g L\(^{-1}\)).

Dewatered sediments were generally too solid-like to pipette a specific volume of material. Approximately 3 x 1 g each sample was placed on a pre-weighed aluminium foil square, and weighed before drying at 105°C for 48 h. The dried samples were reweighed to constant weight. The analysis was repeated twice for each sample, and the dwt/wwt % was determined according to equation 1 above, substituting the mass of foil for the Eppendorf mass.

Samples for dry solids analysis were occasionally stored at -20°C for 24 h before analysis. According to a comparative analysis experiment, this did not affect the solids concentration results.

4.5  Experimental Procedure

4.5.1  Optimisation of Flocculation
Fresh stock solutions of 20 g L\(^{-1}\) PEI were prepared for laboratory use in optimising PEI concentration by its ability to clarify homogenised yeast.

The effect of PEI concentration on flocculation was examined by preparing a dilution series of PEI from 0-20 g L\(^{-1}\) in 2 g L\(^{-1}\) intervals, and mixing by inversion in a 1:1 volume ratio with 60% (vol) disrupted yeast. This was repeated twice for each source of PEI. The supernatant collected from spinning down the solids at 3000 g for 300 s (Denley) was tested for clarity at 650 nm.

Similar experiments were carried out where the sediment was collected instead of the supernatant, after a 0.5 h spin. The sediments were examined for variation in viscoelastic properties with changes in PEI concentration.

4.5.2  Viscosity of biological suspensions
The viscosities of 60% (v/v) homogenised yeast suspensions mixed with equal volumes of 10 g L\(^{-1}\) PEI, or 0.1 M borax, or water were measured in the
Bohlin VOR as outlined above, taking the mean of 3 consistent 17 mL samples. The samples were sheared for 13 s at each shear rate. The viscosities of the flocculant solutions were also measured using the same equipment.

4.5.3 Viscoelastic characterisation of dewatered sediments

Experiments were carried out to examine the relationship between complex shear modulus and dry solids concentration of sediments formed from flocculated suspensions of homogenised yeast.

60% (v/v) homogenised yeast was mixed with equal volumes of 10 g L\(^{-1}\) PEI, or 0.1 M borax and transferred to sets of 6x20 mL centrifuge tubes. The tubes were balanced, and each set spun in the Denley or Europa centrifuge at a constant RCF within the range \(10^3\)-\(10^4\) g. The supernatants were decanted off and the sediments collected for viscoelastic characterisation on the Bohlin VOR and dry solids analysis, taking the mean of 3 consistent samples in each case.

In one set of experiments, the supernatant from a borax flocculation was collected and flocculated with 10 gL\(^{-1}\) PEI, and sedimented at a range of RCF as before.

4.5.4 Scroll Decanter Centrifuge

4.5.4.1 Preparation of yeast suspension

The preparation of feed material to the scroll decanter centrifuge began with suspending yeast in phosphate buffer. Phosphate buffer was prepared from 0.0574 M NaH\(_2\)PO\(_4\) and 0.0426 M Na\(_2\)HPO\(_4\) to give a 0.1 M solution at pH 6.5.

In a typical pilot scale trial with the scroll decanter centrifuge, 33.3 kg (30 L) yeast were crumbled into 20 L 0.1 M phosphate buffer whilst mixing in a 100 L tank (250 L tank for larger batches) to make 50 L of a 60% (v/v) suspension.

4.5.4.2 Homogenisation

The yeast was disrupted using high pressure homogenisation at 500 bar g in the K3 homogeniser for 5 discrete passes at a flow rate of 280 L h\(^{-1}\). Temperature was maintained at 5°C to minimise protein degradation. This method yields complete disruption of whole cells in terms of protein release.
(Hetherington et al. 1971). 2 M NaOH was used to adjust to pH 7.4 after disruption.

4.5.4.3 Flocculation

Two different flocculating agents were used in the scroll decanter centrifuge experiments: PEI and borax. In a typical scroll decanter centrifuge experiment, 1 kg PEI was dissolved in 20 L deionised water and diluted to 50 L to prepare a 20 g L\(^{-1}\) solution. This required approximately 2 L 50% hydrochloric acid to adjust to pH 7.4.

0.1 M borax solution was adequate to effect complete flocculation when mixed with an equal volume of 60% (v/v) disrupted yeast (Ward 1990). Borax crystals were dissolved in deionised water before dilution to 50 L, and pH adjustment carried out to pH 6 after flocculation using 50% (vol/vol) phosphoric acid. The sediment properties are known to be strongly dependent on pH, with very stable flocs forming at pH 4 (Ward and Hoare 1990). However, pH 6 was used in these experiments since therapeutic protein recovery processes typically avoid extremes of pH in order to reduce product inactivation.

60% (v/v) disrupted yeast suspensions were mixed with flocculant (10 g L\(^{-1}\) PEI or 0.1 M borax) in a 1:1 volumetric ratio in a 100 L tank for 0.5 h. Mixing continued during the time for carrying out an experiment, which was typically 1.67 h, depending on flow rates.

4.5.4.4 Scroll decanter centrifuge trials

The feed arrangements to the scroll decanter centrifuge were as shown in Figure 4.2.1 a, with the experiment for flocculation at the point of entry to the centrifuge bowl in Figure 4.2.1 c. Timing of the centrifuge run was started from the point of entry of the feed to the centrifuge feed pipe.

Under differential speed control, torque readings from the centrifuge were monitored throughout the run at 120 or 300 s intervals. Due to slight fluctuations in the continuous readout, the torque measurement was recorded 10 s prior to, at, and 10 s past the specified time. The mean reading was taken as a representative torque measurement, and used to give an on-line estimate of
centrifuge operation approaching steady state. Torque is generated by the conveyor even when there is no process material present i.e. when the centrifuge is run dry, due to friction in the gear box bearings. Experiments were therefore carried out where torque measurements were recorded at varying differential speed setpoints without process material present.

Dewatering trials were carried out in the scroll decanter centrifuge using a variety of systems with different rheological properties. These were whole yeast cells, disrupted yeast cells, and disrupted yeast cells flocculated with either PEI or Borax. Feed solids concentrations were all at or disrupted equivalent to 30% (v/v) whole cells.

The centrifuge was operated in differential speed control mode to investigate the effect of changes in feed flow rate, pond depth and differential speed. Experiments in torque control mode were also carried out for disrupted yeast cells flocculated with borax.

Sampling of the material produced by the centrifuge was carried out at approximate steady state (i.e. constant torque reading) for both the sediment and supernatant for further analysis. The mass and volumetric flow rates of the supernatant were determined by collection in a tared measuring cylinder over a period of 30 s.

Measuring the mass flow rate of dewatered sediment discharge posed severe practical problems due to solids accumulation in the collection area close to the bowl. A novel collection method was tested where a tray was inserted into the solids discharge hopper close to the bowl, but was unsuccessful due to solids being sliced off the tray surface as it was extracted. The most reliable method was found to be placing a tared bucket under the discharge hopper, and collecting the solids produced under steady conditions over a 300 s period.

Mass balances were carried out on total and dry solids material around the centrifuge for each run. Results for further processing were taken only when the total mass flow rate collected from the overflow and underflow streams was within +/-10 % of the feed mass flow rate.
4.5.5 Disc stack centrifuge

4.5.5.1 Preparation of feed material

Suspensions of Polyvinylacetate (PVAc) particles were used to establish characteristic separation performance for the SA00H-205 operating at full and quarter separation capacities.

PVAc forms stable suspensions of robust, spherical particles in water, which sediment according to Stoke's law in ideal settling conditions. In each experiment, 0.05 kg PVAc was suspended in 1 L deionised water, and sonicated for 1 h. This amount of sonication was sufficient to break up any agglomerated particles (Mannweiler 1990) which might otherwise be broken up by shear effects in the centrifuge after sampling the feed. The suspension was diluted to 100 L with deionised water, and mixed for 0.5 h to a final concentration of 0.055% (w/v). Feed samples were taken before and after passage through the gear pumps used to feed the centrifuge. The feed pump configurations were as those described in sections 4.2.1 (a-b).

In further experiments with the disc stack centrifuge, a variety of yeast suspensions were used as a feed material. Dilute suspensions of material (<1% w/vol) were used throughout, to avoid hindered settling effects where high concentrations of particles lead to the obstruction of the pathway for free settling.

The feed preparation for the experiments with whole yeast began with crumbling 0.55 kg yeast into 100 L of 0.1 M phosphate buffer at pH 7.4 in a 100 L mixing tank. The yeast was mixed for 0.5 h to yield a 0.55% (w/v) homogenous suspension of discrete particles.

Feed for experiments with cell debris took 0.92 L of 600 gL\(^{-1}\) homogenised yeast in 0.1 M phosphate buffer at pH 6.5 diluted to 100 L in a 100L mixing tank with deionised water. The resultant 0.55% (w/v) suspension was mixed for 0.5 h.

In the experiments with PEI flocculant, 1 L 55% (v/v) homogenised yeast was diluted and mixed with deionised water to 99 L in a 100 L tank before adding 1 L of 18.3 g L\(^{-1}\) PEI solution, and mixed for 0.5 h.
4.5.5.2 Disc stack centrifuge trials

Scale down of the disc stack centrifuge was based on constant \( Q/\Sigma \), and to this end experiments were designed so that the critical particle diameter (\( d_{\text{cut size}} \)) was the same in both full and \( \frac{1}{4} \) size stacks.

The flow rates were chosen to coincide with a range around a critical particle diameter of 1 \( \mu \text{m} \), which is the smallest size for reliable analysis using the electronic particle sizing equipment available. The values for the calculation of separation area and cut size are shown in Figures 4.2.4, 4.2.5 and Table 4.5.1. The appropriate mid-flow rate was repeated for each experiment.

15 L PVAc feed material were allowed to flow through the centrifuge before sampling the supernatant at each flow rate. This was achieved by timing the flow from the onset of supernatant discharge. Two full discharges of sediment were executed after each flow rate change without terminating the feed supply. Particle size analysis was carried out on the feed and supernatant samples as described above.

The separation efficiency of the \( 1/4 \) stack was tested in three different configurations as shown in Figure 4.5.1.

Configuration "a" had 12 blank discs on the bottom, 9 active discs and 36 blank discs on top. This resulted in the active discs being located 1/4 off the base of the stack.

Configuration "b" had 6 blank discs with channel risers on the bottom, 9 active discs and 42 blank discs on top. The active discs were situated 1/10 off the base of the stack.

Configuration "c" had 6 blank discs without channel risers on the bottom, 9 active discs and 42 blank discs on top. The active discs were situated 1/10 off the base of the stack.

The full stack with 38 active discs was also tested for comparison.

Once the scale-down configuration had been established with PVAc, then similar experiments with full and \( \frac{1}{4} \) stacks were performed using dilute suspensions of whole yeast cells, homogenised yeast cells, and homogenised yeast cells flocculated with PEI.
Figure 4.5.1: Diagram of disc stack separator showing the position of the active discs within the dummy disc stack.
Table 4.5.1: Flow rates to the Westfalia SA00H-205 disc stack centrifuge.

<table>
<thead>
<tr>
<th>System</th>
<th>Particle density (kg m(^{-3}))</th>
<th>dc (μm)</th>
<th>Flow rate (L h(^{-1}))</th>
<th>1/4 stack</th>
<th>Full stack</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PVAc</td>
<td>1191 (Mannweiler 1992)</td>
<td>1.6</td>
<td>137</td>
<td>554</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.2</td>
<td>74</td>
<td>302</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.9</td>
<td>199</td>
<td>793</td>
<td></td>
</tr>
<tr>
<td>Homogenised yeast</td>
<td>1050 (Mosquira et al 1981)</td>
<td>1.6</td>
<td>35</td>
<td>148</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.2</td>
<td>19</td>
<td>81</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.9</td>
<td>50</td>
<td>212</td>
<td></td>
</tr>
<tr>
<td>Homogenised yeast flocculated with PEI</td>
<td>1140 (Clarkson et al 1993)</td>
<td>1.6</td>
<td>89</td>
<td>405</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.2</td>
<td>49</td>
<td>221</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.9</td>
<td>127</td>
<td>580</td>
<td></td>
</tr>
<tr>
<td>Whole yeast</td>
<td>1112 (Ward 1989)</td>
<td>1.6</td>
<td>77</td>
<td>326</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.2</td>
<td>42</td>
<td>178</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.9</td>
<td>110</td>
<td>467</td>
<td></td>
</tr>
</tbody>
</table>

6 L material were allowed to pass through the centrifuge before sampling supernatant.

Samples of the sediment were collected from the bowl section for dry weight analysis and viscoelastic characterisation at the end of each centrifuge trial, by dismantling the bowl without a final solids discharge.

4.5.6 Scale down experiments and laboratory mimics

A variety of experiments were carried out on flocculated material in the laboratory, in an attempt to find the controlling regime for scale down experiments with respect to design of pilot scale trials.
4.5.6.1 Effect of continuous shear on flocculated suspensions

Particle size analysis was carried out on samples of flocculated suspensions to investigate if the size distribution of the flocs was affected by prolonged exposure to continuous shear.

17 mL samples of yeast cell debris flocculated with PEI at an equivalent concentration of 30% (v/v) whole cells were placed in a C25 cup and bob measuring system on the Bohlin VOR. The samples were sheared at 1160 s⁻¹ for 0, 30, and 300 s, and each experiment was repeated twice. Particle size analysis was carried out on the diluted samples as described in section 4.3.2.

4.5.6.2 Effect of continuous shear on flocculated sediment

The aim of this experiment was to determine if a continuous shearing action performed in the laboratory could affect the structure of a dewatered sediment.

Viscoelastic characterisation and dry weight analysis was carried out on samples of dewatered sediment prepared from suspensions of homogenised and PEI-flocculated yeast spun at 3000 g for 600 s in a bench top centrifuge (Denley).

3x the samples were mounted between parallel plates (pp30 Bohlin VOR) and sheared at 0, 2, and 20 s⁻¹ for a total of 1000 s with viscoelastic tests carried out at 0, 10, 300, 600 and 1000 s. Operating files for continuous shearing and viscoelastic tests were linked so that each experiment was conducted in one continuous operation (Bohlin VOR "jobstream"). The remaining 3x samples were analyzed for dry weight of sediment as described in section 4.4.2.

4.5.6.3 Comparative clarification and sedimentation in the scroll decanter centrifuge

Samples of feed material from the pilot scale scroll decanter were sedimented in laboratory centrifuges and examined for clarification and dewatering properties.

The basis of scale down for the clarification tests was to keep the liquid residence time inside the centrifuge constant in a fixed centrifugal field. At pond
depths of 13.1, 16.2 and 19.3 mm, and a feed flow rate of 60 L h\(^{-1}\), the liquid residence time inside the scroll decanter was estimated to be 76, 92, and 111 s respectively. 3x 1.5 mL triplicate samples of feed were clarified at 3000 g in an Eppendorf centrifuge for 76, 92 and 111 s and the supernatants were decanted off and tested for clarity at 690 nm.

3x 20 mL samples of feed material were also spun at 3000 g in a bench top centrifuge (Denley) for 300, 600, 1200 and 2400 s. The supernatants were decanted off, and the sediments analyzed for dry weight and viscoelastic properties. The results were compared to the dry weight and viscoelastic properties of sediment discharged from the scroll decanter centrifuge.

### 4.5.6.4 Comparative clarification in the disc stack centrifuge

Samples of dilute feed material of homogenised yeast flocculated with PEI from the pilot scale disc stack centrifuge were sedimented in an Eppendorf centrifuge according to two different scale down protocols.

3x 1.5 mL samples were clarified for residence times in an Eppendorf centrifuge appropriate to equivalent Q/Σ in a disc stack centrifuge using Q_0/Σ as defined in section 3.2.4. Q/Σ was calculated for a centrifugal field of 2350 g, corresponding to the worst case for clarification in the disc stack centrifuge at the innermost disc radius.

3x 1.5 mL samples of feed material were also clarified for a time equivalent to the liquid residence time inside a disc stack centrifuge, calculated according to \( \tau = V/Q \) where V was the bowl volume. The centrifugal field chosen was also 2350 g.

Supernatants were collected and examined for clarity at 690 nm.
5. RESULTS

5.1 Optimisation of flocculation

Selective flocculation of yeast cell homogenate with the cationic polyelectrolyte PEI is strongly dependent on flocculant dosage. Figure 5.1.1 shows the effect of increasing concentration of two different commercial brands of PEI with the same nominal molecular weight (10 000) on the clarity of the supernatant from a yeast cell homogenate.

With no PEI present, there is zero light transmission though the supernatant sample at 650 nm due to fine solids such as cell debris remaining in suspension. PEI concentrations as low as 1 gL⁻¹ enhance clarification, with a steady improvement to over 80% transmission at 5 gL⁻¹ in comparison to 100% transmission for water. After this maximum, clarification is reduced due to excess PEI causing the restabilisation of debris particles and precipitation of proteins previously held in solution, as discussed by Bulmer (1993).

The optimum PEI concentration of 5 gL⁻¹ for 60% (vol) homogenised yeast suspension was chosen in all experimental work for maximum clarification without incurring protein precipitation. Sigma PEI was used throughout, unless otherwise stated.

5.2 Viscosity of biological suspensions

Figure 5.2.1 shows flow curves for a variety of flocculated yeast suspensions. Solutions of the flocculating agents Borax and PEI had viscosities of 1.05 and 1.5 mPa s respectively which are close to water at 1.005 mPa s when compared with the disrupted suspensions.

Yeast homogenate flocculated with PEI showed slight shear thinning behaviour with a viscosity of 5.3 mPa s at a shear rate of 73 s⁻¹ and 3.9 mPa s at 1465 s⁻¹. This was probably due to shear breakage of the flocs, with less particle interaction at higher shear rates.

Yeast homogenate flocculated with borax has a yield stress with a viscosity tending towards a high value as the shear rate tends towards zero, as shown in Figure 5.2.1.
Figure 5.1.1: The effect of PEI concentration on flocculation of homogenised yeast given by % transmission at 650nm against water. Supernatants prepared from 60% homogenised yeast suspension mixed with an equal volume of PEI solution and spun for 300 s at 3000 g in a laboratory centrifuge (Denley B12401).

- □ SIGMA laboratory supply,
- ■ BASF bulk supply
Figure 5.2.1: Viscosity of flocculated yeast suspensions. 17 mL suspensions exposed to continuous uni-directional shear for 13 s at each shear rate in the Bohlin VOR using C25 cup and bob at 25 °C.

- □  Water
- ○  5 gL⁻¹ PEI solution
- ▲  0.05 M Borax solution
- ■  30% homogenised yeast in water
- ●  30% homogenised yeast in 5 gL⁻¹ PEI
- ▲  30% homogenised yeast in 0.05 M Borax
This behaviour is due to the large, space-filling nature of the sedentary flocs, which become separated as the yield stress is exceeded. Flocs become visibly smaller at high shear rates, where the suspension has the same flow curve as that for non-flocculated yeast suspension at the same concentration.

Yeast homogenate without a flocculating agent had a viscosity of about 2.8 mPa s at the shear rates measured.

5.3 Viscoelastic characterisation of sedimented flocs

Figure 5.3.1 shows the results of two typical strain sweep tests on sediments of flocculated yeast debris at different solids concentrations. A smaller, more sensitive torsion bar was required for the less well dewatered sediment. Both the complex shear modulus and loss angle are independent of oscillating strain magnitude at strains less than 0.002. This is the limit of the linear viscoelastic region (LVR), and within this range of small strains, the equations for linear viscoelastic theory apply.

Figure 5.3.2 shows the complex shear modulus and loss angle for sediment samples at a range of oscillating frequencies i.e. shear strain rates, at small strain magnitudes within the LVR. At all frequencies, $G^*$ is lower, and $\delta$ is higher for the wetter sediment. This is because the liquid-filled spaces between the particles of compact sediment are larger and perform a lubricating function which causes the bulk sample to flow more readily. The drier sediment has a greater resistance to shear (higher $G^*$) and is more elastic (lower $\delta$). As a point for comparison, $G^*$ and $\delta$ are chosen at 5 Hz. This relatively high frequency is thought to be related to fast or bulk flow of sediment compared to creeping flow at 0.1 Hz (see section 1.4.7.3). Ward (1989) processed viscoelastic data at 5 Hz when considering viscoelastic characteristics relevant to the bulk flow of sediment in the decanter centrifuge.

5.3.1 Viscoelasticity as a characteristic of dewatering

Shown in Figure 5.3.3 are the viscoelastic characteristics of various flocculated sediments at an oscillating frequency of 5 Hz.
Figure 5.3.1: Viscoelastic characterisation of sediments prepared from suspensions of homogenised yeast flocckulated with PEI: Strain sweep test for limit of linear viscoelastic region (LVR), for sediments of 60% homogenised yeast flocckulated with an equal volume of 10 gL⁻¹ PEI solution at pH 7.4. Measured on the Bohlin VOR using pp30 parallel plates, calibrated torsion bars 3.84 g cm and 21.24 g cm, at an oscillation frequency of 10 Hz. Temperature 25°C.

- • – O — Loss angle, (δ), - ■ — □ — Complex shear modulus, (G*),
Open symbols dwt/wwt 16%. Closed symbols dwt/wwt 21%.
Figure 5.3.2: Viscoelastic characterisation of sediments prepared from suspensions of homogenised yeast flocculated with PEI: Frequency sweep test in ascending and descending frequency of oscillating strain. Sediments prepared from 60% homogenised yeast flocculated with an equal volume of 10 g L\(^{-1}\) PEI solution. Bohlin VOR measurement as for Figure 5.3.1, nominal strain amplitude 0.002, within LVR. Temperature 25 °C.

— — — Loss angle, (\(\delta\)), — — — Complex shear modulus, (G\(^*\)).

Open symbols dwt/wwt 16%. Closed symbols dwt/wwt 21%. 
Figure 5.3.3: Viscoelastic spectra with respect to solids concentration for sediments prepared from suspensions of homogenised and flocculated yeast. Sediments prepared at a range of relative centrifugal force $10^3$-$10^4$ g. Loss angle and shear modulus measurements using the Bohlin VOR as for Figure 5.3.1 given at an oscillation frequency of 5 Hz. Open symbols loss angle $\delta$, closed symbols complex shear modulus, $G^*$. 

- ○: 60% homogenised yeast mixed with an equal volume of 10 gL$^{-1}$ PEI solution
- △: 60% homogenised yeast mixed with an equal volume of 100 mM Borax solution
- ▲: 60% homogenised yeast pre-clarified with 100 mM Borax, and then mixed with an equal volume of 10 gL$^{-1}$ PEI.
The loss angle (relative contribution of viscous and elastic properties) and complex shear modulus (total resistance to shear) are plotted against Dwt/Wwt % as a measure of solids concentration. In general, the loss angle decreases slightly, and the complex shear modulus tends to increase as the solids concentration increases. Hence viscous, liquid-like biological sediments behave increasingly more like elastic solids as they dewater and compact. Complex shear moduli are plotted on a log scale since the changes in sample strength can vary by orders of magnitude over a narrow range of solids concentrations.

The rate at which a change in viscoelastic properties occurs relative to solids concentration varies between different types of sediment. For example, in Figure 5.3.3, the loss angles of sediments flocculated with PEI or borax are low (ie. between 0° and 18°) at concentrations of 16% and 14% dwt/wwt respectively. These sediments behave more like solids than liquids. However, even at solids concentrations greater than 30% dwt/wwt, protein precipitate formed by flocculating yeast homogenate already clarified with borax yields a more viscous, liquid-like sediment with loss angles greater than 18°.

Figure 5.3.3 also shows complex shear moduli measured for the same sediments, together with the different levels of dewatering achieved specifically at 3000 g in each case. Sediments such as PEI-flocculated yeast cell homogenate generated at 3000g are of 19% dwt/wwt, have a low G* (1000 Pa or less), and are considered soft. Similar sediments formed from yeast homogenate flocculated with borax, or yeast homogenate pre-clarified with borax and flocculated with PEI have complex shear moduli greater than 1000 Pa at 14% and 22% dwt/wwt respectively, and so are considered to be relatively tough.

Combining these characteristics enables each sediment to be qualified under the following categories, which were first laid out by Ward (1989):

- **Yeast homogenate flocculated with PEI**: soft, elastic
- **Whole Yeast (Ward 1990)**: soft, viscous
- **Yeast homogenate flocculated with Borax**: tough, elastic
- **Yeast homogenate pre-clarified with Borax and flocculated with PEI**: tough, viscous
These categories were observed in biological suspensions discharged from a scroll decanter centrifuge by P.N. Ward at UCL (1989). He used whole yeast flocculated with borax, soya protein precipitates and *F. graminearum* as described in section 1.4.10.

5.4 Effect of flocculant dosage on sediment structure.

PEI concentration affects the viscoelastic properties of a sediment. Figure 5.4.1 shows that both loss angle and complex shear modulus increase as sediments form under the same conditions with increasing PEI flocculant concentration. In fact a 1% change in PEI concentration can change the sediment characteristics from a soft, elastic solid (at 4 g L\(^{-1}\) PEI, \(G^*\) is 600 Pa and loss angle is 10\(^{\circ}\)), to a tough, viscous liquid (at 14 g L\(^{-1}\) PEI, \(G^*\) is 1100 Pa and loss angle is 18\(^{\circ}\)). This behaviour is in contrast to that where dewatering or drying of the sample occurs, when the loss angle tends to decrease as the material becomes tougher and the complex shear modulus increases. Yasufumi and Koichiro (1989) also reported an increase in \(G'\) with increasing flocculant concentration for dense suspensions of silica.

Compressed flocs containing a high proportion of nucleic acids are thought to contribute to the elastic nature of the sediment formed at low PEI concentrations. Higher PEI concentrations cause the precipitation of proteins formerly held in solution which settle and cause the sediments to become tougher and more viscous. Soya protein precipitates examined by Ward (1989) formed tough, viscous sediments at 3000 g.
Figure 5.4.1: Effect of flocculant dosage on viscoelastic properties of sediments prepared from suspensions of homogenised and flocculated yeast. 60% homogenised yeast mixed with an equal volume of PEI solution. Sediments collected after a 360s spin at 3000g in a laboratory centrifuge (Denley). $G^*$ and $\delta$ given at an oscillation frequency of 5Hz.

- $\bullet$ Complex shear modulus, $G^*$, (Pa)
- $\bigcirc$ Loss angle, $\delta$, (°)
5.5 **Pilot Scale Trials with a scroll decanter centrifuge**

Figures and tables in section 5.5 show results from experiments carried out using suspensions derived from yeast cells and fed to a scroll decanter centrifuge (Alfa-Laval Sharples P600) rotating at 6000 rpm and generating a relative centrifugal force of 3000 g, as described in section 4.2.4.

5.5.1 **Heel Torque**

Torque \( T \) is experienced across the centrifuge bowl due to the turning of the scroll conveyor. The so-called heel torque \( T_o \) is obtained in the absence of solids and is dependent on scroll differential speed as shown in Figure 5.5.1. In standard centrifuges, heel torque is zero when the conveyor rotates at the same speed as the bowl, and increases linearly with conveyor differential speed and an increased load on the gearbox. However, very high heel torque values can occur at low differential speeds as shown in Figure 5.5.1. This is normal behaviour and has been attributed to oil separation in the gear box, or worn gears (Records 1992). The appropriate heel torque value was checked against the standard curve obtained in Figure 5.5.1 before each experiment.

Scrolling torque \( T-T_o \) is due to the turning force required to scroll solids up the conical section of the centrifuge under centrifugal pressure. When operating under a conveyor differential setpoint, the scrolling torque was found by subtracting the appropriate heel torque \( T_o \) value from the torque readout on the controls \( T \). When operating under torque control, the appropriate heel torque was found from the resultant conveyor differential using the fitted data as shown in Figure 5.5.1.

5.5.2 **Amount of material required**

Figure 5.5.2 shows the progress of two pilot scale trials carried out with a deep pond (19.3 mm), giving an active bowl volume of 1.94 L. The first experiment was run from an empty bowl at a feed flow rate of 120 L h\(^{-1}\) and controlled at a conveyor differential speed of 20 rpm. In the second experiment the bowl was already full of solids, the flow rate was halved to 60 L h\(^{-1}\), and the controlling setpoint for the conveyor differential was also halved to 10 rpm. It is
Figure 5.5.1: The effect of conveyor differential speed on heel torque in a scroll decanter centrifuge. P600 centrifuge fitted with a back drive motor running with a dry bowl at 6000 rpm.
Figure 5.5.2: Variation in torque and solids discharge during start-up in a scroll decanter centrifuge. P600 centrifuge fed with a suspension of 60% homogenised yeast flocculated with an equal volume of 10 g L\(^{-1}\) PEI solution. Open symbols torque, closed symbols dwt/wwt.

- □ Experiment 1: Conveyor differential speed 20 rpm, feed flow rate 120 L h\(^{-1}\). Start from empty bowl.
- ■ Experiment 2: Conveyor differential speed 10 rpm, feed flow rate 60 L h\(^{-1}\). Start with bowl already charged with sediment.
necessary to reduce the feed flow rate when reducing the conveyor differential to avoid overloading the bowl with solids.

In the first experiment, the torque output and solids discharge concentration increased simultaneously with time as the bowl filled with solids. After about 30 L material had been fed into the machine (800 s) there was a peak in the torque output after which the solids were discharged at a fixed concentration and the torque output settled around a constant value. An approximation to steady state is achieved where solids are discharged at a steady rate (in this instance 22 kg h\(^{-1}\)).

In the second experiment, there was initial variation in torque output as the conveyor and bowl readjust to the lower conveyor differential speed. During this time, solids accumulated in the bowl. The solids concentration in the sediment discharge increased to a higher value than in experiment 1, but the discharge mass flow rate was also lower at 12 kg h\(^{-1}\).

The torque output of the conveyor was higher in experiment 2 than in experiment 1 due to a combination of drier sediment and a higher solids mass flow rate per revolution of the conveyor. Hence torque is significantly affected by the resistance of the solids to discharge.

In neither experiment was an approximate steady state approached until 30 L of concentrated feed material had been fed into the centrifuge. The volume of material that would be required for multiple optimisation tests highlights the usefulness of being able to pinpoint appropriate pilot trials at an early stage in process design.

5.5.3 Effect of changes in feed flow rate

Figures 5.5.3 (a-f) show the results from feeding a concentrated suspension of yeast homogenate flocculated with PEI to the scroll decanter centrifuge operated at a constant conveyor differential speed at a range of feed flow rates.

In the deep 19.3 mm pond, the solids concentration and complex shear modulus of the sediment are independent of feed flow rate at this scale as shown in (a) and (b) respectively. However, there is an increase in the total and solids
Figure 5.5.3: The effect of feed flow rate on clarification and dewatering for a suspension of homogenised and flocculated yeast in a scroll decanter centrifuge. P600 scroll decanter centrifuge feed suspension was 60% homogenised yeast mixed with an equal volume of 10 g L⁻¹ PEL. Conveyor differential speed was 10 rpm. Open symbols 19.3 mm pond, closed symbols 16.2 mm pond.
Figure 5.5.3: (continued) Open symbols 19.3 mm pond, closed symbols 16.2 mm pond.
component mass flow rates of sediment discharged as shown in (d) and (e) respectively. This occurs at the same conveyor differential, and accounts for the slight rise in torque seen in (c). The sediment discharged is a soft, elastic material as shown in (b).

The supernatant flow rate is directly proportional to the feed flow rate as shown in (d). However, the solids component in the supernatant increases slightly with feed flow rate as shown by the slight increase in solids concentration in (a) and solids component mass flow rate in (e). This result is confirmed by the decrease in separation efficiency shown in (f) which was calculated by the methods discussed in section 3.1.5 for the reduced separation efficiency $E_t$ and the clarification number $C_N$. Values for $E_t$ follow the same trend but are omitted for clarity. Hence solids recovery improved with decreasing flow rate.

As expected from $\Sigma$ theory, increasing feed flow rate reduces separation efficiency due to the reduction in residence time. An increase in clarification at low flow rates indicates that the feed flow rates used are not greater than the critical flow rate $Q_{\text{crit}}$, above which the clarification capacity of the centrifuge is exceeded and solids recovery is independent of flow rate (Stahl and Langeloh 1984).

In the 16.2 mm pond, the sediments discharged are more highly dewatered than those in the deep pond, as shown by the higher values for solids concentration and complex shear modulus in (a) and (b) respectively. The torque values are also higher than for the deep pond as shown in (c).

The solids concentration in the supernatant is higher than for the deep pond as shown in (a). The total and solids component mass flow rates of the supernatants are also higher than for the deep pond: (d) and (e) respectively. Hence the overall separation efficiency of the 16.2 mm pond is lower than for the deep 19.3 mm pond. The separation capacity of the centrifuge is reduced with a shallower pond due to the smaller hold up volume and reduced residence time for clarification.
The mass flow rate of solids discharge is independent of feed flow rate in
the 16.2 mm pond as shown in (d) and (e). Similarly, the solids concentration,
complex shear modulus and torque are also constant. However, the solids
concentration in the supernatant increases with feed flow rate (a), and there is a
steep increase in solids carryover in the supernatant in (e), hence overall
separation efficiency decreases with increasing flow rate as shown in (f).

The separation capacity of the 16.2 mm pond has been limited by solids
conveyance. Sediment accumulates in the bowl to form a deep solids layer
which both reduces the area available for separation and occupies a significant
amount of the remaining volume available for incoming feed. Hence increasing
the rate of incoming feed simply increases the rate at which solids are discharged
in the supernatant and reduces apparent separation capacity.

5.5.4 Effect of changes in pond depth

The results discussed in this section are for concentrated suspensions of
yeast homogenate flocculated with borax and fed to the scroll decanter
centrifuge at the same feed flow rate and operated at the same conveyor
differential speed for three different pond depths. The results show similar
trends and are comparable to those shown for different pond depths using PEI as
a flocculant in the previous section.

This system yields a tough, elastic sediment as shown in 5.5.4 (b).
Figure 5.5.4(a) shows that the dewatering of sediment possibly decreases as the
pond deepens. This is paralleled by a decrease in G* and torque as shown in (b)
and (c) respectively. The total mass flow rate of sediment increases with pond
depth, (d), as large amounts of wet solids are discharged from a deep pond.

The shallow ponds yielded small amounts of highly dewatered sediment,
and the deep pond yielded large amounts of poorly dewatered sediment.

Head pressure from a deep pond forces solids out from the bowl section
quickly, leaving a thin solids layer and hence a large active bowl volume for
clarification. However, in shallow ponds, solids accumulate to a comparatively
greater depth since they are without the head pressure required for rapid
discharge. They may compress to a greater density under their own weight, and
Figure 5.5.4: Effect of pond depth on clarification and dewatering for a suspension of homogenised and flocculated yeast in a scroll decanter centrifuge. P600 centrifuge feed suspension was 60% homogenised yeast mixed with an equal volume of 0.1 M Borax. Feed flow rate was 60 L h⁻¹, and conveyor differential speed was 10 rpm.
Figure 5.5.4: (continued)
their residence time inside the centrifuge is greater, both of which factors contribute to a higher level of dewatering.

The mass flow rate of supernatant decreases as the pond deepens (d) due to entrainment in poorly dewatered sediment. The solids carryover in the supernatant from borax-flocculated yeast cell debris (a) and (e) is low at all pond depths, particularly in comparison with the material flocculated with PEI. This result is reflected in (f) which shows that overall separation efficiency is also comparatively high.

The high separation efficiency in this system may be due to the larger size or robust nature of borax flocs. However, it is also linked to the rheological properties of the sediment which enhance solids transport. Tough, elastic solids are discharged efficiently from the bowl area, leaving a large active bowl volume for clarification.

5.5.5 Effect of changes in the feed zone

Bell and Brunner (1983) used particle size analysis to show that shear damage increased the population of small particles in the feed and supernatant. However, the supernatant suspended solids concentrations used in this dewatering study are too high and too close to the feed concentration to guarantee a representative analysis using this method. There was also evidence to suggest that particle re-agglomeration could have occurred in the supernatant after discharge, which would also make it difficult to detect floc breakage.

A simple experiment was set up to examine the effect of flocculation on the sedimentation of yeast homogenate flocculated with PEI by changing the nature of the feed zone. Table 5.5.1 refers to two experiments, one where flocs were mixed in a batch stirrer prior to feeding the centrifuge, and an identical experiment where the feed zone was used as a mixer for the flocculant using concentric feed pipes. The aim was to test if possible conditioning of the flocs in the mixing vessel had a beneficial effect on solids recovery when compared to on line flocculant dosing inside the centrifuge.
Table 5.5.1: The effect of feed zone on the clarification and dewatering of a suspension of homogenised and flocculated yeast in a scroll decanter centrifuge. P600 scroll decanter centrifuge, pond depth 19.3 mm, total feed flow rate 60 L h\(^{-1}\).

Feed 1: 60% homogenised yeast was mixed with an equal volume of 10 g L\(^{-1}\) PEI in a batch stirring tank and mixed for 30 minutes prior to and during the experiment.

Feed 2: 60% homogenised yeast was mixed with an equal volume of 10 g L\(^{-1}\) PEI inside the centrifuge by feeding each stream at 30 L h\(^{-1}\) via a concentric feed pipe.

<table>
<thead>
<tr>
<th>Method of flocculation</th>
<th>Feed 1</th>
<th>Feed 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conveyor differential speed (rpm)</td>
<td>3</td>
<td>5</td>
</tr>
<tr>
<td>Solids concentration in feed (% dwt/wwt)</td>
<td>7.7</td>
<td>7.7</td>
</tr>
<tr>
<td>Solids concentration in supernatant (% dwt/wwt)</td>
<td>3.9</td>
<td>4.6</td>
</tr>
<tr>
<td>Solids concentration in sediment (% dwt/wwt)</td>
<td>28.4</td>
<td>24.5</td>
</tr>
<tr>
<td>Scrolling torque (T-To) (N m)</td>
<td>0.13</td>
<td>0.04</td>
</tr>
<tr>
<td>Complex shear modulus at 5 Hz (Pa)</td>
<td>2820</td>
<td>789</td>
</tr>
<tr>
<td>Loss angle at 5 Hz (°)</td>
<td>8</td>
<td>10</td>
</tr>
<tr>
<td>Flow rate of supernatant (kg h(^{-1}))</td>
<td>56</td>
<td>52</td>
</tr>
<tr>
<td>Flow rate of sediment (kg h(^{-1}))</td>
<td>62</td>
<td>57</td>
</tr>
<tr>
<td>Flow rate of dry solids in supernatant (kg h(^{-1}))</td>
<td>2.2</td>
<td>2.4</td>
</tr>
<tr>
<td>Flow rate of dry solids in sediment (kg h(^{-1}))</td>
<td>1.8</td>
<td>1.2</td>
</tr>
<tr>
<td>Reduced separation efficiency, (E_{\alpha}^{*})</td>
<td>31</td>
<td>40</td>
</tr>
<tr>
<td>Reduced separation efficiency, (E_{\ell}^{*})</td>
<td>39</td>
<td>40</td>
</tr>
<tr>
<td>Clarification number (C_N)</td>
<td>49</td>
<td>40</td>
</tr>
</tbody>
</table>

The table shows similar results for both experiments in terms of sediment dewatering and scrolling characteristics (i.e. solids concentration, viscoelastic properties and torque). However, the solids carryover into the supernatant is higher for Feed 2, where the feed zone is used as a mixer for the flocculating agent, as shown by the solids concentration and mass flow rate of...
solids in the supernatant. In this case, the feed zone was responsible for poor solids recovery.

Floes mixed slowly in a tank prior to centrifugation may be conditioned in a similar way to that described by Bell and Dunnill (1982) for protein precipitates. Floes may become denser, more robust and better able to withstand exposure to high shearing forces in the feed zone of a centrifuge. In contrast to this, floes instantly exposed to high shear as they form in the feed zone of a scroll decanter centrifuge such as for Feed 2, will not have the size and density required for good separation.

In each of the experiments shown in Table 5.5.1, scroll differential speed relative to the bowl affects the solids dewatering and separation efficiency of the process. This can be seen where there is an increase in both scrolling torque and complex shear modulus of sediment discharged at the lower differential of 3 rpm compared to 5 rpm.

5.5.6 Effect of changes in conveyor differential speed

Figures 5.5.5 (a-f) show the effect of changes in conveyor differential speed on the separation of a concentrated suspension of yeast homogenate flocculated with PEI at the same feed flow rate and pond depth.

The solids concentration of the sediment and complex shear modulus increase as the differential speed to which the conveyor is controlled is reduced, as shown in (a). Sediments are conveyed slowly at low differential speeds, and so have a longer residence time inside the bowl for greater dewatering. This is compensated for by an increase in torque at low conveyor differential speeds as shown in (c).

These results are consistent with those reported by Axelsson et al (1992) for baker's yeast, where a rapid decrease in the solids concentration of sediment occurred as conveyor differential was increased from 5 to 30 rpm.

The total mass flow rate of sediment discharged increases with differential speed. This occurs as the conveyor discharges more, wetter solids as shown in (d). However, the scrolling torque decreases with scroll differential due to the softness of the wetter solids.
Figure 5.5.5: Effect of conveyor differential speed on clarification and dewatering of a suspension of homogenised and flocculated yeast in a scroll decanter centrifuge. P600 centrifuge feed suspension was 60% homogenised yeast mixed with an equal volume of 10 gL⁻¹ PEI. Feed flow rate was 60 L h⁻¹, and pond depth was 19.3 mm.
Figure 5.5.5: (continued)
The total supernatant mass flow rate decreases with differential speed as shown in (d), but contains a smaller dry solids component as shown in (a) and (e). Hence dropping scroll differential speed produces a highly dewatered sediment, but compromises clarification, since a smaller portion of suspended solids are separated in the first place. This result is confirmed in (f), which shows that overall separation efficiency increases with differential speed.

At low differential speeds, the long residence times of sediment in the bowl initially cause the solids to accumulate, and the steady operating depth of solids is high. The active separation area and liquid hold up volume is thus reduced, and supernatant still contains a high proportion of solids when it is discharged.

Figure 5.5.6 shows the effect of conveyor differential speed for a number of different processed yeast suspensions. Each suspension has unique dewatering properties, but they all follow similar trends with respect to centrifuge operation.

For all the systems shown in Figure 5.5.6 (a) and (b), both dry solids and complex shear modulus decrease as the conveyor differential increases. This is because the sediments dewater poorly at higher conveyor differential speeds, due to the correspondingly short residence times in the bowl. Similarly, the mass flow rate of wet solids increases and torque decreases with conveyor differential speed for all the systems shown in Figure 5.5.6 (c).

The suspensions of yeast homogenate and yeast homogenate flocculated with PEI have relatively low complex shear moduli and sediment discharge rates, hence low operating torques as shown in Figure 5.5.6 (d). Whole yeast cells and homogenised yeast flocculated with borax have relatively high complex shear moduli and sediment discharge rates, and hence higher operating torques, also shown in Figure 5.5.6(d).

Figure 5.5.6 (a) shows that under the given conditions, homogenised yeast flocculated with PEI is discharged with a higher solids content than that flocculated with borax, and so prior to this study one might have predicted that it
Figure 5.5.6: Comparison of dewatering characteristics of processed yeast suspensions in a scroll decanter centrifuge. (a) Solids dry weight and (b) Complex shear modulus of sediments discharged from P600 scroll decanter centrifuge operating at varying conveyor differential speeds. Feed flow rate 60 L h⁻¹, pond depth 19.3 mm.

- ■ 30% (vol) homogenised yeast suspension
- □ 30% (vol) whole yeast cell suspension
- ○ 60% (vol) homogenised yeast cells flocculated with an equal volume of 10 g L⁻¹ PEI solution
- △ 60% (vol) homogenised yeast cells flocculated with an equal volume of 0.1 M Borax solution
Figures 5.5.6 (continued) to show (c) Mass flow rate of sediment discharge, and (d) scrolling torque in a scroll decanter centrifuge operating at varying conveyor differential speeds for a range of processed yeast suspensions.

- ■ 30% (vol) homogenised yeast suspension,
- □ 30% (vol) whole yeast cell suspension,
- ○ 60% (vol) homogenised yeast cells flocculated with an equal volume of PEI solution.
- △ 60% (vol) homogenised yeast cells flocculated with an equal volume of Borax solution.
would require a higher scrolling torque to discharge. However, the PEI-flocculated sediment is actually discharged with a lower scrolling torque than the borax-flocculated sediment, because it has a lower complex shear modulus (i.e. it is “softer”).

These results show that visco-elastic characterisation is crucial to understanding centrifuge operation.

5.6 Discussion of scroll decanter centrifuge trials

The results in section 5.5 indicate that the scroll decanter centrifuge requires optimisation of clarification against dewatering.

Clarification is generally improved by reducing flow rate and increasing pond depth. Both factors increase residence time and hold up of liquid inside the centrifuge bowl, and so the accompanying increase in overall separation efficiency might be predicted from Sigma theory. Flow path considerations (section 1.3.5) with a solid conveyor predicted a fast flowing layer of liquid in the central part of the bowl, with a slower flowing outer layer. This flow pattern produces a very severe surface flow of liquid in a ribbon type conveyor, as incoming feed takes the path of least resistance to centrifugal pressure along an axial path in the central part of the bowl. It contributes to the poor clarification observed for fine suspensions of yeast cell debris flocculated with PEI, particularly when flocculation takes place in the feed zone.

According to Bell and Brunner (1983), shear damage occurs in the feed zone of a decanting centrifuge, in the turn of the conveyor ribbon which surrounds the feed. Bell's method was to assume that all the power which is dissipated in the centrifuge is restricted to the volume occupied by this region. For the experiments shown here, the equivalent calculations are shown in Table 5.6.1 which shows that the potential for instantaneous flocc damage increases with flow rate and a reduction in pond depth. This occurs as the feed is accelerated to maximum bowl speed into a smaller dissipating volume.

In contrast to Bell's experiments using a solid conveyor where liquid flows around the screw thread, the experiments here were conducted using a ribbon conveyor, where liquid is thought to flow in an axial direction away from
the feed zone. With this type of conveyor, it is unlikely that the dissipated power is restricted to the feed slot volume but that it is distributed in a turbulent settling zone. However, the effect of reducing pond depth in reducing overall volume available for power dissipation would be the same.

Table 5.6.1: Power per unit volume calculations for P600 scroll decanter centrifuge at various pond depths.

Dissipated power is calculated using \( P = \frac{1}{2} M (\omega R)^2 \) (Gosele 1980), where \( \omega = 100 \, \text{rev}^{-1} \) at 6000 rpm at \( R = 0.075 \, \text{m} \). Suspension of yeast homogenate flocculated with PEI \( \rho = 1032 \, \text{kg m}^{-3} \).

<table>
<thead>
<tr>
<th>Pond depth (mm)</th>
<th>Flow rate (Lh(^{-1}))</th>
<th>P/V (kW m(^{-3}))</th>
<th>60</th>
<th>120</th>
<th>180</th>
</tr>
</thead>
<tbody>
<tr>
<td>19.3</td>
<td>1.2</td>
<td>2.4</td>
<td>3.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>16.2</td>
<td>1.4</td>
<td>2.8</td>
<td>4.3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>13.1</td>
<td>1.7</td>
<td>3.4</td>
<td>5.2</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Section 5.5.5 showed that homogenised yeast flocculated with PEI is sensitive to shear in the scroll decanter centrifuge, and poor solids recovery was found in section 5.5.3 even at low flow rates. However, there was some improvement in solids recovery for the deep pond relative to the shallower pond. This was in part due to the greater volume available for power dissipation in the deep pond, with a corresponding reduction in shear damage.

Increasing conveyor differential improves clarification and may be considered in relation to its effect on acceleration of the feed. The "BD" feature fitted in this centrifuge not only acts as a negative ring dam to prevent feed entering the conical section of the centrifuge, but it also acts as a "soft feed", reducing the acceleration rate of the incoming material. This is a design feature which reduces the damage to shear sensitive materials such as flocculated cell debris. The conveyor actually turns more slowly as its differential speed increases relative to the bowl. This may enhance the effect of the soft feed, by reducing further the rate of acceleration of feed entering the bowl section.

Scroll differential speed and pond depth also play a part in clarification limited by solids transport. If they are reduced to maximise dewatering the
resultant effect is to increase solids residence time and the depth of the solids layer. Operating with a deep solids layer reduces the separation area available for settling and restricts the active bowl volume remaining for supernatant hold up. The reasons for the effect of pond depth on dewatering are central to understanding how the solids are discharged from the centrifuge.

A peak in torque output during the progress of a centrifuge trial (as shown for Experiment 1 in Figure 5.5.2 at 700 s) was a standard observation when feed was delivered to an empty bowl. As the bowl fills with solids, conveyance of wet sediment occurs up the beach section. Eventually solids accumulate in the bowl to a depth where they meet the lower edge of the negative ring dam, and supernatant can no longer enter the beach section. At this point the negative ring dam is said to be "sealed". The maximum torque value in Experiment 1 corresponds to sealing of the negative ring dam.

In the conical section of the centrifuge dewatering may occur, but process liquid extracted here is simply conveyed and discharged with the outgoing solids: it is possible for the conveyor to scroll liquids such as water. This is shown in Figure 5.6.1, where underflow is extracted when no solids are present in the feed. The underflow to throughput ratio for water increases with conveyor differential speed in a deep pond at constant feed flow rate. This is an important result, because it shows that water cannot re-enter the centrifuge via the negative ring dam due to the pressure gradient which forces it out in the first place.

The critical point for solids dewatering lies where the negative ring dam ("BD") meets the conveyor ribbon at the bottom of the conical section or "beach". Here the centrifugal pressure component acting against solids conveyance into the beach section is at its greatest since the acceleration radius is close to that of the bowl.

In deep ponds, hydraulic pressure from the process liquid helps to meet the centrifugal pressure component which tends to push solids back down the conical section, and soft, wet solids are forced into the conical section ("beach") relatively easily by the turning force of the conveyor. The torque output
Figure 5.6.1: Conveyance of water in a scroll decanter centrifuge. P600 centrifuge, pond depth 19.3 mm, feed flow rate 150 L h⁻¹.
associated with deep ponds and soft solids is relatively low, except for high conveyor differential speeds where the mass flow rate of solids discharge is high.

In a shallow pond, this hydraulic pressure is much reduced, and solids accumulate at the bottom of the conical section until they are tough and dry enough to resist the centrifugal pressure component of the beach. In this case it is the turning force of the conveyor alone which forces solids into the conical section, which accounts for the high torque output.

Compression dewatering should increase with the depth of the settling layer as shown by network modulus theory in section 1.4.6. However, in scale down tests intended to mimic an increase in pond depth in a scroll decanter centrifuge, Munro and Van Til (1988) found that adding water to increase the hydrostatic pressure in bottle centrifuges had no effect on the dewatering of sediment. In practice, pond depth affects dewatering mainly by assisting the rapid discharge of solids.

Shallow ponds produce small amounts of highly dewatered sediment, whereas deep ponds produce large amounts of poorly dewatered sediment (section 5.5.4). If a high degree of dewatering is the single most important process requirement, then a shallow or intermediate pond can be used to enhance dewatering of a soft solid (G*<1000 Pa, loss angle< 18°), but not necessarily without compromising clarification and overall separation efficiency. The advantage with respect to overall separation efficiency, clarification and removal of bulk solids lay with the deep pond in the systems examined in this study.

Accumulation of compressible solids at the negative ring dam takes time, and is enhanced by a low conveyor differential speed which increases solids residence time inside the centrifuge. In one experiment (section 5.5.3), discharge was not achieved at all until the conveyor differential was reduced to 5 rpm. Low conveyor differential speeds also bring drier solids towards the negative ring dam, since the longer residence times involved allow more time for dewatering in the bowl section.
These results are in contrast with those shown by Devereux et al (1984) who reported increased dewatering of soya protein precipitates with increasing conveyor differential speed. This type of sediment is tough, viscous and dewatered by drainage rapidly (Ward 1989). A high conveyor differential speed which causes mixing of the sediment (section 5.10) assists dewatering in this case. Unfortunately the authors gave no information about the high torque levels which would be associated with these high levels of dewatering at high conveyor differentials.

5.6.1 Relationships between torque and sediment scrolling characteristics

Figures 5.5.5-6 have shown that torque tends to increase as scroll differential speed decreases and compressible solids dewater. This type of data yields a linear relationship when the solids dry weight multiplied by the mass flow of solids discharge per revolution of the scroll is plotted against the scrolling torque, as shown in Figure 5.6.2.

Alan Records at Alfa-Laval Sharples uses this empirical relationship to scale up from pilot to industrial scale in geometrically similar machines (Records 1974). This method is suitable for any one system, i.e. pilot scale trials must be redefined for any new type of material. This is because different materials compact and give greater resistance to bulk flow at different solids concentrations, as discussed in section 5.3.

The same trend towards greater dewatering and higher torque values as pond depth decreases was seen for both PEI and borax flocculated sediments (sections 5.5.3 and 5.5.4 respectively). The solids concentration in the borax flocculated sediment was lower than that in the PEI flocculated sediment, but the complex shear moduli were higher. This indicates that in different systems, scrolling torque is dependent on the solids resistance to flow (G*) rather than the solids concentration.
Linear regression through the origin:
\[ Y = ax \] where \( a = 9.4574 \)
Standard deviation = 0.6029

Figure 5.6.2: Turning force of the conveyor in a scroll decanter centrifuge in relation to the solids discharge: Total dry mass resistance of sediment to discharge per revolution of the conveyor plotted against scrolling torque for a range of biological feed suspensions.
- ○ 60% homogenised yeast flocculated with an equal volume of 10 gL⁻¹ PEI
- ▲ 60% whole yeast cells flocculated with an equal volume of 0.1 M Borax
- △ 60% homogenised yeast flocculated with an equal volume of 0.1 M Borax
- □ 30% whole yeast cells
- ■ 30% homogenised yeast

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Table 5.6.2 illustrates the significance of solids consistency for sediment transport in the scroll decanter centrifuge. For example, at dry solids concentrations of around 22% dwt/wwt, the complex shear modulus of homogenised yeast flocculated with PEI is less than that of homogenised yeast flocculated with borax, and the torque output of the conveyor is also higher. The interstitial and intracellular water for whole yeast cells is equivalent to the interstitial water for yeast homogenate suspensions when comparing the conveyance of solids mass in a scroll decanter centrifuge.

**Table 5.6.2: Comparison of dewatering properties and scrolling torque in a scroll decanter centrifuge.**

*Feed flow rate 60 L h⁻¹, conveyor differential speed 10 rpm, pond depth 19.3 mm. Feed material prepared from 60% v/v homogenised yeast mixed with an equal volume of water or flocculant.*

<table>
<thead>
<tr>
<th></th>
<th>G* (Pa)</th>
<th>Dwt/wwt (%)</th>
<th>(T-To) (N m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yeast homogenate</td>
<td>1380</td>
<td>22.1</td>
<td>0.034</td>
</tr>
<tr>
<td>Whole yeast</td>
<td>1530</td>
<td>22.4</td>
<td>0.055</td>
</tr>
<tr>
<td>Yeast homogenate flocculated with 10 g L⁻¹ PEI</td>
<td>1830</td>
<td>22.7</td>
<td>0.057</td>
</tr>
<tr>
<td>Whole yeast flocculated with 0.1 M borax</td>
<td>2200</td>
<td>22.0</td>
<td>0.088</td>
</tr>
<tr>
<td>Yeast homogenate flocculated with 0.1 M borax</td>
<td>25000</td>
<td>22.6</td>
<td>0.096</td>
</tr>
</tbody>
</table>

A physical characteristic such as G* takes into account sediment structure whereas solids concentration does not, hence using G* yields a relationship which is not system specific. Figure 5.6.3 shows that a linear relationship is also obtained when the complex shear modulus multiplied by the mass flow of solids discharge per revolution of the scroll is plotted against the heel torque. Thus the inherent resistance of viscoelastic solids to shear is related to the turning force required to scroll them up the conical section of the centrifuge.
Linear regression through the origin:
\[ Y = aX \text{ where } a = 1.5773 \]
Standard deviation = 0.08149

Figure 5.6.3: Total resistance of sediment to discharge per revolution of the scroll plotted against scrolling torque for a range of biological feed suspensions.
- ○ 60% homogenised yeast flocculated with an equal volume of 10 gL⁻¹ PEI
- ▲ 60% whole yeast cells flocculated with an equal volume of 0.1 M Borax
- △ 60% homogenised yeast flocculated with an equal volume of 0.1 M Borax
- □ 30% whole yeast cells
- ■ 30% homogenised yeast
Experience has shown that it is most important to ensure that the mass balance of material around the centrifuge remains closed when constructing this type of relationship. Log(G*) is used on the ordinate due to the G* data which is measured by order of magnitude.

The majority of experiments carried out in developing this relationship were based on controlling the centrifuge by means of a conveyor differential set point, and measuring the resultant torque. However with the ABDC modification, it was also possible to define a torque set point to which the centrifuge was controlled, and the resultant conveyor differential measured. The data from these experiments is also plotted in Figure 5.6.3. The advantage of using this method is that it provides a direct control for operating at maximum torque output as advised by Leung and Havrin (1992) for maximum dewatering of the solids discharge.

In all the experiments shown, the solids discharged are relatively soft, and the torque limit of the machine was not exceeded. However, in dewatering some solids such as domestic waste flocculated with sulphide (G* = 100 000 Pa), the torque limit of 1.5 Nm is approached in pilot scale operation (Records 1992). In this case, dewatering is the most important operation, and the process is scaled up on the basis of machine torque capacity, with the intention to run the centrifuge at maximum torque output for solids dewatering close to the maximum possible.

However, in many of the trials shown, the level of clarification and overall separation efficiency was unacceptable and measures taken to improve this (increasing conveyor differential and pond depth) would attain a much lower level of dewatering. If the centrifuge dewatering capabilities are to be maximised, then the clarification problem needs to be resolved, either by diluting the feed (at 30% solids, the feed loading is high in these experiments), by increasing centrifuge bowl capacity and separation area, or by reducing shear damage to flocs in the feed zone.
5.7 Pilot scale trials with a disc stack centrifuge

Previous studies at UCL (Mannweiler and Hoare 1992) have shown that it is possible to scale down a disc stack centrifuge using aluminium blocks to replace active discs and thereby reduce separation area. Mannweiler used an industrial scale disc stack centrifuge (Westfalia BSB 7 47 476) with 72 discs in the fully active stack, outer disc radius $R_o = 0.076$ m and channel risers on the edge of the discs. Figures in this section show the results from scaling down a smaller disc stack centrifuge (Westfalia SA00H-205) using a series of blank discs without spacers. The number of active discs in the full stack was 39 in this case, with $R_o 0.051$ and channel risers situated towards the centre of the discs.

5.7.1 Scale down with a defined medium

Aqueous suspensions of polyvinylacetate (PVAc or "latex") were used to characterise separation in the fully-active and scaled-down disc stacks by particle size analysis of feed and supernatant samples as described in sections 3.3 and 4.3.2.

5.7.1.1 Fully active disc stack

The grade efficiency curves obtained for the fully active stack at a range of flow rates are shown in Figure 5.7.1. The adapted Rosin-Rambler-Sperling-Bennet (RRSB) fitted curve given by Mannweiler (1990) (section 3.3.1) for the scaled down BSB centrifuge is shown for comparison. The centrifuge grade efficiency curves are S-shaped and deviate from Stoke's law due to flow vortices around the spacer ribs and uneven particle distribution at the disc openings as described in section 1.3.2.

A major part of the grade efficiency curve at each flow rate in the SA00H overlies the curve reported by Mannweiler (1990) for the larger centrifuge, indicating that separation characteristics are independent of scale for dilute suspensions of PVAc. As in Mannweiler's work, critical particle diameter $d_c$ was calculated using the radius at the inner edge of the channel riser (ie. $R_o = 0.043$ for the SA00H). Separation is restricted to the area above the channel risers where feed enters the discs.
Figure 5.7.1: Grade efficiency curves to show the separation of 0.055% (w/v) PVAc in a disc stack separator (Westfalia SA00H-205) fitted with a fully active stack.

- ■ Feed flow rate 554 L h⁻¹, dₜ 1.6 μm
- ○ Feed flow rate 302 L h⁻¹, dₜ 1.2 μm
- ▲ Feed flow rate 793 L h⁻¹, dₜ 1.9 μm
- Standard PVAc curve for scaled down BSB disc stack centrifuge (Mannweiler 1992)
The curve for a flow rate of 793 L h\(^{-1}\) shows carryover of large particles into the supernatant. Above 580 L h\(^{-1}\) the total flow capacity of the centrifuge is exceeded and feed flows into the overflow section together with supernatant. As the flow rate increases, the flow pattern inside the centrifuge becomes more turbulent and particle re-entrainment into the supernatant can occur as shown in the curves for 554 L h\(^{-1}\). Mannweiler (1994) has suggested that this is a common problem in small machines which tend to have poorly defined flow patterns.

5.7.1.2 Scaled down disc stack

Figures 5.7.2-3 show separation efficiency results using the Westfalia SA00H-205 with only 1/4 of the separation area active as described in section 4.5.5.2.

Configuration (a) with the active discs positioned 1/4 of the way up the stack clearly shows an improved separation (curve lies further to the left) not at all representative of the full stack operation. This is due to pre-settling of particles in the sediment holding space, so that the centrifuge behaves partly like a tubular bowl.

Configuration (b) is where the active discs positioned 1/10 of the way up the stack are supported from the bottom by blanking discs with identical channel risers to those in the active discs. At 74 L h\(^{-1}\) the grade efficiency curve overlies that for configuration (a) at the same flow rate, suggesting that pre-settling occurred. The marked difference in separation efficiency between high and low flow rates with this arrangement is not representative of full stack operation where all flow rates yield the same curve.

Configuration (c) has the active discs positioned 1/10 of the way up the stack supported by blanking discs without channel risers. The separation shown by this arrangement closely follows the standard PVAc curve given by Mannweiler (1992) for the larger BSB centrifuge with well defined flow patterns. It shows a slightly better separation than in the SA00H-205 full stack curve with respect to carryover of large particles at higher flow rates. The
Figure 5.7.2: Grade efficiency curves to show the separation of 0.055% (w/v) PVAc in a disc stack separator (Westfalia SA00H-205) fitted with a scaled down 25% active stack. Active discs situated 1/4 off base, with no channel risers in supporting discs.

- ■ Feed flow rate 137 L h$^{-1}$, $d_c$ 1.6 μm
- ○ Feed flow rate 74 L h$^{-1}$, $d_c$ 1.2 μm
- ▼ Feed flow rate 199 L h$^{-1}$, $d_c$ 1.9 μm
- Standard PVAc curve for scaled down BSB disc stack separator (Mannweiler 1992)
Figure 5.7.3: Grade efficiency curves to show the separation of 0.055% (w/v) PVAc in a disc stack separator (Westfalia SA00H-205) fitted with a scaled down 25% active stack. Active discs located 1/10 off base, with channel risers in the supporting discs.

- ■ Feed flow rate 137 L h⁻¹, dᵥ 1.6 μm
- ○ Feed flow rate 74 L h⁻¹, dᵥ 1.2 μm
- ▲ Feed flow rate 199 L h⁻¹, dᵥ 1.9 μm
- Standard PVAc curve for scaled down BSB disc stack separator (Mannweiler 1992)
Figure 5.7.4: Grade efficiency curve to show the separation of 0.055% (w/v) PVAc in a disc stack separator (Westfalia SA00H-205) fitted with a scaled down 25% active stack. Active discs situated 1/10 off base, without channel risers in supporting discs.

- □ Feed flow rate 137 L h⁻¹, dₐ 1.6 μm
- ○ Feed flow rate 74 L h⁻¹, dₐ 1.2 μm
- ▽ Feed flow rate 199 L h⁻¹, dₐ 1.9 μm

Standard PVAc curve for scaled down BSB disc stack separator (Mannweiler 1992)
separation area to flow rate ratio is the same in both full and quarter active stacks. However, the flow rate has decreased relative to the bowl volume in the scaled down stack and the flow capacity is not exceeded, resulting in less particle re-entrainment.

The differences in separation efficiency shown by the scale down configurations (a-c) can be explained by flow path considerations.

In the fully active SA00H-205 stack, the discs are fitted with channel risers, i.e. holes which when aligned form a vertical channel through which the bulk of process material flows at a relatively small centrifugal pressure compared to that at the disc tip as illustrated in Figure 1.3.2. The original intention of this design was to reduce the re-entrainment of settled solids into the incoming feed stream. However, riser channels can create flow vortices which disturb particle settling. Moreover, channel risers 1/3 of the way up the slope of the discs severely reduce the potential separation area since separation begins at the inner edge of the channel riser. More modern designs such as the CSA1 have semi-circular channel risers at the edges of the discs, which prevent solids re-entrainment without compromising settling area.

In scale down configuration (a), (Figure 5.7.2), the lack of channel risers forces feed to the outer edge of the discs and pre-settling of solids occurs in the central part of the sediment holding space before feed enters the active part of the stack.

At the mid flow rate used in the full stack experiments significant numbers of large particles remain in the supernatant. This is due to large vortices forming around the channel risers, thereby reducing active separation area.

Similar effects were observed in the scale down version of the full stack with channel risers (configuration b, Figure 5.7.3). Particle re-entrainment associated with a turbulent flow pattern occurred at the higher flow rates whereas at the lower flow rate of 74 L h⁻¹ re-entrainment of large particles was not observed. However, the flow pattern at 74 L h⁻¹ where pre-settling of
particles appears to occur (curve lies towards the left of the standard graph) was not duplicated at the same specific throughput in the fully active stack.

In the case of configuration (c), (Figure 5.7.4), separation was slightly better than for the full stack. This was due to the incoming feed being forced into the sediment holding space before entering the active part of the stack. Presettling of solids does not occur in this lower part of the bowl due to turbulence (Willus and Fitch 1973) but performance may have improved due to the extra separation area made available by the longer flow path. In addition, particle re-entrainment would not occur where the feed meets the channel risers in the active disc section in this case, because the feed is already evenly distributed between the discs.

5.7.1.3 Comparison of full and quarter scale disc stack performance

Figure 5.7.5 shows a comparison of grade efficiency separation for the three scale down configurations at 137 L h\(^{-1}\) fitted to the adapted RRSB function and extrapolated to \(d/d_c = 0\). The fitted full stack curves are also shown for the SA00H (at the same specific throughput) and the BSB disc stack centrifuges. The fitting procedure tends to weight the curves in favour of the larger particles so that at this flow rate configuration "b" with the channel risers, appears to be the closest mimic of the SA00H full stack curve due to large-particle re-entrainment phenomena.

Figure 5.7.6, shows similar curves at a lower specific throughput where \(d_c = 1.2\ \mu m\). At this lower flow rate there is a shift in the grade efficiency separation for configuration "b" with channel risers, so that it no longer duplicates the SA00H full stack curve. Configuration "c" without channel risers however, yields a consistent grade efficiency separation curve at all flow rates in keeping with full stack SA00H behaviour.

Configuration "a" with active discs further up the stack, yields grade efficiency curves which are dissimilar to the full SA00H curves at all flow rates.

These results were used to establish the scale down configuration for experiments using dilute suspensions of biological materials.
Figure 5.7.5: Grade efficiency curves to compare the separation of PVAc in a fully active and scaled down disc stack separator at a critical cut size of 1.6 μm. 0.055% (w/v) PVAc fed to the Westfalia SA00H-205 at a flow rate of 137 Lh⁻¹ in the 25% active stack, and a flow rate of 554 Lh⁻¹ in the fully active stack.

- - - - Active discs 1/4 off base, no channel risers.
- - - - Active discs 1/10 off base, with channel risers.
- - - - Active discs 1/10 off base, no channel risers.
- - - - Fully active stack.
- - - - Standard PVAc curve for scaled down BSB disc stack separator (Mannweiler 1992)
Figure 5.7.6: Grade efficiency curves to compare the separation of PVAc in a fully active and scaled down disc stack separator at a critical cut size of 1.2 μm. 0.055% (w/v) PVAc fed to the Westfalia SAOOH-205 at a flow rate of 74 Lh\(^{-1}\) in the 25% stack, and a flow rate of 302 Lh\(^{-1}\) in the fully active stack.

- Active discs 1/4 off base, no channel risers.
- Active discs 1/10 off base, with channel risers
- Active discs 1/10 off base, no channel risers.
- Fully active stack.

Standard PVAc curve for scaled down BSB disc stack separator (Mannweiler 1992)
5.7.2  Scale down with complex biological media

Since the density difference between biological particles and their suspending medium tends to be less than that between PVAc and water, the flow rates used to operate at cut-sizes around 1 μm are low. This rendered the 1/4 stack arrangement "c" (active discs 1/10 off the base and no channel risers) suitable for scale down experiments with biological suspensions. With this arrangement dilute PVAc separation was consistent with the full stack even at low flow rates.

5.7.2.1  Whole baker's yeast cells

Figures 5.7.7-8 show the grade efficiency curves for whole yeast cells separated in the full and 1/4 stack respectively. The curves for the lowest specific throughput with $d_s = 1.2 \mu m$ were incomplete due to almost complete recovery of large particles. Both full and 1/4 stack grade efficiencies show steep curves with a high separation efficiency for large particles, deteriorating to a negative separation efficiency for small particles.

This type of curve corresponds to a particle size distribution where more small particles appear in the supernatant than there were originally present in the feed, as shown in Figures 5.7.9. It is typical for shear-sensitive particles, where shear damage occurs in the feed zone. If shear breakage occurs in the feed zone, then the feed distribution measured prior to entering the centrifuge has fewer smaller particles than that entering the disc stack after the feed zone, and consequently separation performance is underestimated by the observed grade efficiency curve (Bell and Brunner 1983).

Low concentrations of whole yeast cells form a suspension of discrete particles (Ward 1989) and so shear breakage was due to cell disruption, on entry to the centrifuge rather than aggregate disruption. Microscopic examination could have helped to determine if the shear damage was due to loss of cell buds. The SA00H is a non-hermetic centrifuge where the incoming suspension flows through an air filled cavity and is accelerated to high speeds when it enters the bowl section. More modern hermetic designs exclude air from the feed zone so
Figure 5.7.7: Grade efficiency curves to show the separation of whole yeast cells in a disc stack separator fitted with a fully active stack. 5.5 gL⁻¹ whole yeast cells fed to the Westfalia SAOOH-205.

- Feed flow rate 326 L h⁻¹, dₜ 1.6 μm
- Feed flow rate 467 L h⁻¹, dₜ 1.9 μm
- Standard PVAc curve for scaled down BSB disc stack separator (Mannweiler 1992)
Figure 5.7.8: Grade efficiency curves to show the separation of whole yeast cells in a disc stack separator with a scaled down stack. Active discs located 1/10 off base with no channel risers. 5.5 gL$^{-1}$ whole yeast cells fed to the Westfalia SAOOH-205.

- ■ Flow rate 77 L h$^{-1}$, $d_c$ 1.6 μm
- ▲ Flow rate 110 L h$^{-1}$, $d_c$ 1.9 μm
- Standard PVAc curve for scaled down BSB disc stack separator (Mannweiler 1992)
Figure 5.7.9: Particle size distributions of feed and supernatant for whole yeast cells in a fully active and a scaled down disc stack separator. Active discs located 1/10 off base with no channel risers.
Figure 5.7.10: Grade efficiency curves to compare the sparation of whole yeast cells in a fully active and scaled down disc stack separator. Active discs in scaled down stack located 1/10 off base with no channel risers. Feed suspension 5.5 gL$^{-1}$ whole yeast cells.

- 25% stack active, flow rate 77 L h$^{-1}$, $d_c$ 1.6 μm,
- Fully active stack, flow rate 325 L h$^{-1}$, $d_c$ 1.6 μm,
- Standard PVAc curve for scaled down BSB disc stack separator (Mannweiler 1992).
that incoming flow is cushioned by liquid. Further design modifications incorporate a rotating feed pipe so that feed suspension is accelerated gently into the bowl section. These advances limit shear damage to cells and thereby render disc stack centrifuges suitable for harvesting fermentation broth.

The fitted full stack curve in Figure 5.7.10 is slightly steeper and more negative than the fitted scaled down curve showing a situation where more shear damage occurs in the full stack at higher flow rates.

5.7.2.2 Homogenised baker's yeast

The grade efficiency curves for homogenised yeast cells shown in Figure 5.7.11-12 are also negative in the smaller particle range. This result was unexpected for homogenised yeast cells, since cell debris is a robust material. However, the separation curve did not correspond to particle size distributions where more small particles were recovered in the supernatant than in the feed, as shown in Figures 5.7.13. In these figures, the total population of particles in the supernatant relative to the feed is very small, suggesting that the clarification process was very efficient at removing large particles. Hence the proportion of small particles in the supernatant was exaggerated, leading to a negative grade efficiency curve.

A combination of coincidence and shape factor where long narrow particles are streamlined through the sizing orifice may be responsible for a larger number of small particles in the dilute supernatant sample. Variations in density between particles of cell debris also hamper grade efficiency analysis since only one mean density value is taken for the calculation of $d_c$. Density difference is inversely proportional to $d_c$ and so greater proportions of large dense particles and small light particles would shift the curve in opposite directions at top and bottom, closer towards the shape of the grade efficiency curve for PVAc.

However, it is possible that aggregates of cell debris particles were present in the feed solution, and that these are susceptible to shear damage. This might also explain why the predicted separation fits the standard curve better at
Figure 5.7.11: Grade efficiency curves to show the separation of homogenised yeast cells in a disc stack separator fitted with a fully active stack. 5.5 gL$^{-1}$ yeast cell debris fed to the Westfalia SAOOH-205.

- ■ Feed flow rate 148 L h$^{-1}$, $d_e$ 1.6 μm
- ○ Feed flow rate 81 L h$^{-1}$, $d_e$ 1.2 μm
- ▽ Feed flow rate 212 L h$^{-1}$, $d_e$ 1.9 μm
- Standard PVAc curve for scaled down BSB disc stack separator (Mannweiler 1992)
Figure 5.7.12: Grade efficiency curves to show the separation of homogenised yeast cells in a scaled down disc stack separator. Active discs located 1/10 off base with no channel risers. 5.5 gL⁻¹ yeast cell debris fed to the Westfalia SAOOH-205.

- ■ Feed flow rate 35 L h⁻¹, \( d_c \) 1.6 μm
- ○ Feed flow rate 19 L h⁻¹, \( d_c \) 1.2 μm
- △ Feed flow rate 50 L h⁻¹, \( d_c \) 1.9 μm

Standard PVAc curve for scaled down BSB disc stack separator (Mannweiler 1992)
Figure 5.7.13: Particle size distributions of feed and supernatant for homogenised yeast cells in a fully active and scaled down disc stack separator. Active discs located 1/10 off base with no channel risers.
Figure 5.7.14: Grade efficiency curves to compare the separation of homogenised yeast cells in a fully active and scaled down disc stack separator. Active discs in scaled down stack located 1/10 off base, with no channel risers. Feed suspension 5.5 gL$^{-1}$ disrupted cells.

- . . . 25% active stack, flow rate 35 L h$^{-1}$, $d_c$ 1.6 $\mu$m.
- . . . Fully active stack, flow rate 148 L h$^{-1}$, $d_c$ 1.6 $\mu$m.
--- Standard PVAc curve for scaled down BSB disc stack separator (Mannweiler 1992)
low flow rates, when less de-aggregation occurs. Using the de-aggregated supernatant itself as a feed solution would indicate if this is so.

The curves from Figures 5.7.11 and 5.7.12 are overlaid at all flow rates, as shown in Figure 5.7.14, showing that the unusual separation behaviour of yeast cell homogenate in the full stack could have been predicted from a scale down experiment.

5.7.2.3 **Homogenised baker's yeast flocculated with PEI**

Figures 5.7.15-16 show the grade efficiency curves for yeast cell debris flocculated with PEI in a full and 1/4 stack. In Figure 5.7.16 the 1/4 stack grade efficiencies show the typically steep curves associated with shear breakage. A negative grade efficiency curve in this case corresponds to an increase in the population of small particles in the supernatant as shown in Figure 5.7.17. This result is a definite indication of shear breakage in the feed zone of the centrifuge. The grade efficiency curves become steeper and slightly more negative at the higher flow rates. In the full stack (Figure 5.7.15) curves are overlaid at all flow rates suggesting that at flow rates in excess of 221 L h⁻¹, breakage is uniform.

In Figure 5.7.17 the entire population of particles is not captured in the size analysis of feed material to the full stack. This does not affect the grade efficiency curves, since the recovery of larger particles is complete (T(d) = 1). There is a slight variation in the mean particle size of the feed suspensions to the full and quarter stacks, due to experimental error in controlling flocculation on a pilot scale with the available equipment.

Although the data for the full stack curve in Figure 5.7.15 is negative and indicates the formation of small particles, the complementary reduction of large particles expected in the supernatant is not shown. The usual number of larger particles are present in the supernatant in this case, possibly due to re-entrainment at the high flow rates associated with full stack operation.

The fitted full stack curve in Figure 5.7.18 shows the classic breakage shape due to the large number of negative particles. It is displaced to the right of the 1/4 stack fitted curve, showing an overall deterioration in performance on scale up. A similar deterioration in performance is observed in a comparison of
Figure 5.7.15: Grade efficiency curves to show the separation of homogenised yeast cells flocculated with PEI in a fully active disc stack separator. 5.5 gL⁻¹ yeast cell debris flocculated with 0.18 gL⁻¹ PEI.

- ■ Feed flow rate 405 L h⁻¹, \( d_c \) 1.6 μm
- □ Feed flow rate 221 L h⁻¹, \( d_c \) 1.2 μm
- ▲ Feed flow rate 580 L h⁻¹, \( d_c \) 1.9 μm
- — Standard PVAc curve for scaled down BSB disc stack separator (Mannweiler 1992)
Figure 5.7.16: Grade efficiency curves to show the separation of homogenised yeast cells flocculated with PEI in a scaled down disc stack separator. Active discs located 1/10 off base with no channel risers. 5.5 gL⁻¹ yeast cell debris flocculated with 0.18 gL⁻¹ PEI.

- Feed flow rate 89 L h⁻¹, d_c 1.5 μm
- Feed flow rate 49 L h⁻¹, d_c 1.1 μm
- Feed flow rate 127 L h⁻¹, d_c 1.8 μm

Standard PVAc curve for scaled down BSB disc stack separator (Mannweiler 1992)
Figure 5.7.17: Particle size distributions of feed and supernatant for homogenised yeast cells flocculated with PEI in a fully active and a scaled down disc stack separator. Active discs located 1/10 off base with no channel risers.

(a) Fully active stack
- Feed, ○ Supernatant at a flow rate of 405 L h⁻¹
(b) 25% active stack
- Feed, ● Supernatant at a flow rate of 89 L h⁻¹
Figure 5.7.18: Grade efficiency curves to compare the separation of homogenised cells flocculated with PEI in a fully active and scaled down disc stack separator. Active discs in scaled down stack located 1/10 off base with no channel risers. Feed suspension 5.5 gL⁻¹ disrupted cells flocculated with 0.18 gL⁻¹ PEI solution.

- - - - 25% stack, flow rate 89 L h⁻¹, dₐ 1.5 µm.
- - - - Full stack, flow rate 405 L h⁻¹, dₐ 1.6 µm

Standard PVAc curve for scaled down BSB disc stack separator (Mannweiler 1992)
Figure 5.7.19: Grade efficiency curves to compare the separation of homogenised cells flocculated with PEI in a fully active and scaled down disc stack separator. Active discs in a scaled down stack located 1/10 off base with no channel risers. Feed suspension 5.5 gL⁻¹ disrupted cells flocculated with 0.18 gL⁻¹ PEI solution.

- 25% stack, flow rate 49 L h⁻¹, dₜ 1.1 μm,
- Full stack, flow rate 221 L h⁻¹, dₜ 1.2 μm
- Standard PVAc curve for scaled down BSB disc stack separator (Mannweiler 1992)
full and scale down stacks at a lower specific throughput with $d_c = 1.2 \, \mu m$ in Figure 5.7.19. The poor comparative separation performance at full scale is due mainly to increased shear damage, but was also expected due to the slightly improved performance of configuration "c" on scale down at these flow rates as shown with PVAc in section 5.7.1.3.

5.7.3 Scale down of solids dewatering

Table 5.7.1 shows the dry solids content and rheological properties of sediment samples taken from inside the bowl of the disc stack centrifuge at the end of each experiment.

Erikson (1984) reported solids concentrations of 18-22% dwt/wwt for baker's yeast in disc stack centrifuges large enough to operate at a throughput capacity of 200 m$^3$ h$^{-1}$, indicating that dewatering could have been predicted for whole yeast cells. However, solids dewatering is clearly over-estimated in the scaled down stack in the case of yeast cell debris flocculated with PEI, which is a compressible sediment.

**Table 5.7.1: Scale down of solids dewatering in a disc stack centrifuge.**

*Sediments collected from the inside edge of the bowl before discharge.*

<table>
<thead>
<tr>
<th>Feed material</th>
<th>30% v/v homogenised yeast</th>
<th>60% v/v homogenised yeast flocculated with equal volume 10 g L$^{-1}$ PEI</th>
<th>30% v/v whole yeast</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stack activity</td>
<td>1/4</td>
<td>Full</td>
<td>1/4</td>
</tr>
<tr>
<td>$G$' (Pa)</td>
<td>1400</td>
<td>850</td>
<td>6600</td>
</tr>
<tr>
<td>$\delta$ ($)</td>
<td>4</td>
<td>5</td>
<td>11</td>
</tr>
<tr>
<td>dwt/wwt (%)</td>
<td>14</td>
<td>13</td>
<td>22</td>
</tr>
</tbody>
</table>

The sediment samples taken from the scale down stack tend to be drier, tougher and less liquid-like than those taken from the full stack experiments. This is due to the longer residence times associated with the lower flow rates used in the scaled down stack. Erikson (1984) also reported that increasing
residence time of *E. coli* debris in a disc stack centrifuge increased dewatering performance due to greater compaction of the sediment.

The higher $G^*$ values observed in the scaled down experiments could potentially give misleading results in two ways. The tougher solids could give pessimistic results with respect to hindering solids discharge, and also the level of anticipated dewatering would be optimistic.

### 5.8 Discussion of disc stack scale down results

According to sigma theory, scaling down separation area allows the use of a reduced flow rate to examine the same cut size. This eases handling of small volumes of material, and reduces the waste between runs when the bowl is emptied.

Experiments were based on the number of bowl volume changes required to reach steady state as equivalent to that derived from Mannweiler's dye tracer tests i.e. a total equivalent to 5 bowl volume changes. The volume of the SA00H-205 bowl was 0.6 L, and so it would require 3 L feed material before reaching steady state. Experiments here doubled Mannweiler's equivalent value in a conservative estimate for steady state at 6 L. It was assumed that a larger feed volume did not affect separation efficiency since at 0.05% w/w concentration the feed was extremely dilute: any build up of solids inside the bowl was considered negligible.

Mannweiler (1990) proposed that the volume of liquid held inside the solids holding region is stagnant due to hydraulic pressure differences at the bowl and outer disc edges. The flow path of feed suspension inside the machine would take the path of least resistance, and would not enter the solids holding space. In this case, the active separation volume in terms of clarification at steady state is only slightly more than the volume occupied by the disc stack, and blanking off 3/4 of the stack would significantly reduce the active volume, thereby potentially reducing the amount of material required for a scale down test.

However, the actual volume of material used in these experiments was based on the fixed number of total bowl volume changes required to reach
steady state, which includes an allowance for the amount of material required to fill the bowl. As soon as steady state was thought to have been attained, then a sample was taken, and the experiment finished. In these experiments, the actual volume of material used in full and quarter stack experiments was based on the total bowl volume, and therefore was exactly the same. A true scale down in terms of saving valuable process material can only be achieved by simultaneously scaling down the bowl volume with separation area.

What this work has shown so far is that separation area can be effectively scaled down according to sigma theory for the equipment and materials tested. Blocking off the inactive separation volume in the solids holding region with buffer or blanking inserts could significantly reduce the amount of material required for a pilot test run.

In all the above experiments, flow rate was scaled down with separation area so that the same size range of particles would be separated in both full and 1/4 stacks. An alternative way of scaling down would be to allow the centrifuge to experience the same flow rates at each scale, and to adjust the critical particle diameter for grade efficiency calculations according to sigma theory. However, although constant shear breakage conditions inside the feed zone of the centrifuge would be maintained at each scale using this method, the observed particle size distribution in the supernatant would differ. This would cause difficulties during size analysis, particularly when scaling up to industrial scale. For example, a flow rate of 74 L h\(^{-1}\) yields a cut size of 1.2 μm in the 1/4 stack SA00H-205, but at the same flow rate in an industrial centrifuge with a large separation area such as the BSB, this would decrease to 0.2 μm. Clearly, very few particles of measurable size would be extracted from the supernatant of the larger centrifuge. In addition to this, the small scale separation would yield a material which would not be suitable to start the next stage of a scaled down process because it would be different to the material which would be produced after scale up.

The data given for solids dewatering is the maximum which can be achieved in the centrifuge bowl under the given conditions before discharge. In
general, this would lead to an over estimation of dewatering in practice since at least one bowl volume of process liquid is required to discharge the solids (more in continuous discharge nozzle centrifuges). The edge of the bowl becomes a critical point for effective solids discharge where resuspension of the sediment occurs before flow through the discharge ports. Discharge frequency with possible over-dewatering and blockage must be optimised against loss of valuable process liquid in re-suspended solids. Equipment fitted with a feed bypass alleviates this problem by intermittently using clean process water to re-suspend and discharge dewatered sediment.
5.9 Laboratory scale mimics of industrial centrifuges

The figures and tables in this section refer to studies which examined scale down of centrifugation to laboratory scale.

5.9.1 Clarification in the scroll decanter centrifuge

Σ theory calculations for the scroll decanter centrifuge at constant RCF (section 3.2.3) predict better separation as pond depth decreases. This is due to the theoretically larger surface area available for separation in a shallow pond. However, a trend towards poor separation efficiency with increasing pond depth contradicts all observations on clarification in the scroll decanter centrifuge made so far in this study.

Table 5.9.1 for yeast homogenate flocculated with borax shows a comparison of supernatants from a laboratory spin and the P600 decanter centrifuge under the same conditions of feed, RCF and residence time with respect to a range of pond depths. The liquid residence times in the scroll decanter centrifuge were calculated using $t = V/Q_o$ where:

$$V = \pi L (R^2 - r_i^2)$$

Solids depth was considered negligible for these purposes since solids were rapidly discharged at a conveyor differential of 10 rpm.

There was some variation in the preparation of feed material at pilot scale on different days. However, mean results of the laboratory mimics show good comparison with the supernatants collected from the scroll decanter centrifuge. The observed tendency towards clearer supernatants with increasing pond depths is duplicated. Hence the criteria for scale down in this case appears to be liquid residence time at constant RCF.

Gosele (1980) proposed that scale up of compressible solids in geometrically similar scroll decanter centrifuges depends on constant peripheral velocity. Although not geometrically similar, if this had been maintained on scale down, then the RCF would actually have been the same due to the similar outer radius of the Eppendorf centrifuge. Hence liquid residence time at constant peripheral velocity could also have been the criteria for scale down.
Table 5.9.1: Comparison of clarification of a flocculated suspension in laboratory and scroll decanter centrifuges.

3x 3x 1.5 ml samples of feed material to the P600 scroll decanter centrifuge were spun in an eppendorf centrifuge at 3000g for 76, 92, 111 seconds. This was equivalent to the residence time of feed in the scroll decanter centrifuge bowl operating at a feed rate of 60 L h⁻¹ at pond depths of 13.1, 16.2, 19.3 mm respectively. Feed samples 1-3 prepared in the pilot plant, feed sample 4 prepared in the laboratory.

<table>
<thead>
<tr>
<th>Feed material was 60% v/v</th>
<th>Solids concentration of supernatants in 76 s</th>
<th>92 s</th>
<th>111 s</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed 1</td>
<td>2.24*</td>
<td>2.09*</td>
<td>1.93*</td>
</tr>
<tr>
<td>Feed 2</td>
<td>4.32</td>
<td>3.71</td>
<td>3.22</td>
</tr>
<tr>
<td>Feed 3</td>
<td>3.89</td>
<td>3.61</td>
<td>3.25</td>
</tr>
<tr>
<td>Feed 4</td>
<td>3.41</td>
<td>3.09</td>
<td>3.06</td>
</tr>
<tr>
<td>Mean</td>
<td>3.9</td>
<td>3.5</td>
<td>3.2</td>
</tr>
<tr>
<td>Scroll decanter centrifuge</td>
<td>3.9</td>
<td>3.7</td>
<td>2.9</td>
</tr>
</tbody>
</table>

* outlier not included in mean

Table 5.9.2: Comparison of supernatants of flocculated material from laboratory and scroll decanter centrifuges.

3x 1.5 mL samples of feed material to the P600 scroll decanter centrifuge were spun in an eppendorf centrifuge at 14 000 g for 600 s to test the potential clarification. 3x 1.5 mL samples of the same feed material were also spun at 3000 g for 111 seconds, equivalent to the residence time of feed in the scroll decanter centrifuge bowl at a pond depth 19.3 mm and a flow rate of 60 L h⁻¹. Resultant light transmission at 690 nm of the laboratory supernatants are shown compared to light transmission of supernatants from the scroll decanter centrifuge operating at conveyor differentials 3 and 5 rpm. Feed 1 material was mixed in a stirred tank for 0.5 h prior to and during the experiment. Feed 2 material was flocculated inside the centrifuge.

<table>
<thead>
<tr>
<th>Feed material prepared</th>
<th>% Light transmission of supernatants at 690 nm:</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Potential Laboratory mimic</td>
</tr>
<tr>
<td>Feed 1</td>
<td>71</td>
</tr>
<tr>
<td>Feed 2</td>
<td>67</td>
</tr>
</tbody>
</table>
The clarification of homogenised yeast flocculated with PEI in the scroll
decanter centrifuge was very poor, and could not be duplicated in the laboratory.
Table 5.9.2 shows the clarification of feed samples to the scroll decanter
centrifuge in ideal settling conditions at 14,000 g for 300 s in an Eppendorf
centrifuge. It was assumed that all suspended solids were settled under these
conditions, and that the given clarity is the maximum potential clarity of the
supernatant when containing only material held in solution. The supernatant
from the laboratory mimics of a deep pond (τ=111s) at 3000 g almost matched
this maximum clarification of feed material, showing potential for a high level of
clarification in the scroll decanter centrifuge.

The clarity of supernatants actually discharged from the scroll decanter
centrifuge is extremely poor in comparison to the laboratory mimic. This is
because yeast homogenate flocculated with PEI is a shear sensitive material
which is affected by the turbulent feed and settling zones in short scroll decanter
centrifuges. The relative length and associated flow pattern of the P600
centrifuge was compared to a slender decanter centrifuge in Figure 1.3.5 A
longer centrifuge or much reduced flow rate could be expected to improve solids
recovery in this system.

Further experiments considered the possibility of permanent shear
damage to flocs in the scroll decanter centrifuge by comparing the potential
clarification of the feed and supernatants at a range of conveyor differential
speeds. Spinning a sample of material at 14,000 g in a laboratory centrifuge for
600 seconds was considered to demonstrate total clarification of the supernatant
in terms of removing suspended solids. Recovering solids under these
conditions yields the maximum potential clarification of the sample. Table 5.9.3
shows the maximum potential clarification of the feed and supernatants for
homogenised yeast, and for homogenised yeast flocculated with PEI. In the
non-flocculated sample the potential clarification of the supernatant was poor for
all samples from a range of conveyor differentials. However, the potential
clarification of the flocculated supernatant samples was less than that of the feed
original, i.e. it was not possible for the supernatant to match the potential
clarification of the starting material. This shows that this system was adversely affected by conditions inside the scroll decanter centrifuge, and that a degree of permanent shear damage occurred.

Table 5.9.3: Comparison of the maximum potential clarification of feed and supernatants from a scroll decanter centrifuge operating at a range of conveyor differential speeds for flocculated and non-flocculated systems. 3x 1.5 ml samples of each material were spun at 14 000g in an eppendorf centrifuge for 600s. The resultant supernatants were decanted off and examined for light transmission at 690 nm.

<table>
<thead>
<tr>
<th>Feed material to the scroll</th>
<th>%Transmission at 690 nm</th>
<th>Feed</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Supernatants</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Conveyor differential</td>
<td>mean</td>
</tr>
<tr>
<td></td>
<td>(rpm) 2.5 5 10</td>
<td></td>
</tr>
<tr>
<td>30% v/v homogenised yeast</td>
<td>3 2 3 3</td>
<td>3</td>
</tr>
<tr>
<td>60% v/v homogenised yeast</td>
<td>64 60 62 62 71</td>
<td></td>
</tr>
</tbody>
</table>

5.9.1.1 The effect of shear on the particle size distribution of flocculated material

Figure 5.9.1 shows results from a particle size analysis of flocculated material exposed to a unidirectional continuous shear field. This was an attempt to mimic the shearing effects of the feed zone of an industrial centrifuge in laboratory scale equipment. Dilution of the suspension was intended to inhibit re-agglomeration of disrupted flocs as well as facilitate size analysis. The particle size data showed a trend to decrease in the number of large particles (> 4 μm) and increase in the number of small particles (< 2 μm) as the time for exposure to shear increased. There also appears to be a bi-modal size distribution building up at 300 s⁻¹ since at this shear rate the cumulative size distribution curve dips at about 3.5 μm to effectively form two separate curves.

The observations recorded here contain important considerations when attempting to mimic the settling of shear sensitive systems in laboratory centrifuges. Using Σ theory, these mimics are carried out under shear free, ideal settling conditions which are not representative of conditions in an industrial
Figure 5.9.1: The effect of shear on the particle size distribution of flocculated material. 60% homogenised yeast flocculated with 10 g L$^{-1}$ PEI solution. 17 mL samples sheared at a rate of 1160 s$^{-1}$ in a cup and bob viscometer (C25 Bohlin VOR). Particle size analysis carried out after 2x1/50 dilution in 10% sodium chloride by electrical sensing method (Elzone 280pc) using orifice size 18 μm.

- ○ — unsheared,
- □ — — 30 s shear,
- ▽ — — 300 s shear
centrifuge. A protocol is required to quantify shear sensitivity from laboratory data which can be applied to scale up procedures.

5.9.2 Clarification in the disc stack centrifuge

Table 5.9.4 (a-d) shows a comparison of solids recovery from constant $Q/\Sigma$ and constant residence time methods of mimicking a disc stack centrifuge in an Eppendorf centrifuge for yeast cell debris and yeast cell debris flocculated with PEI. The volume of the centrifuge bowl was constant for both full and 1/4 scale residence time mimics, hence residence times for clarification in the quarter stack mimic were longer than those in the full stack mimic due to the changes in flow rates. (Table 5.9.4 b,d) The $Q/\Sigma$ method was based on specific throughput with respect to separation area, so that the residence times are the same for full and 1/4 stack mimics (Table 5.9.4 a,c).

The worst case for clarification was selected in both sets of laboratory mimics, where $RCF=2350g$, equivalent to the inner disc radius on the disc stack centrifuge.

Solids recovery as given by $E_t$ for non-flocculated debris (Table 5.9.4 a-b) show similar or higher values in the disc stack centrifuge compared to the laboratory mimics. Hence the "worst case" settling at $R_i$ is a representative mimic of settling conditions for a robust material such as homogenised yeast.

The $E_t$ values for flocculated debris (Table 5.9.4 c-d) show that both sets of laboratory mimics over-estimated solids recovery in the disc stack centrifuge at both 1/4 and full pilot scale since the mass yields are similar or lower in the industrial machine.

These results confirm that solids recovery of the flocculated material cannot be predicted from laboratory scale mimics of this type because it is affected by shear damage in the disc stack centrifuge.
Table 5.9.4: Comparison of solids recovery in laboratory and disc stack centrifuges using constant specific throughput ($\Sigma$ theory), and constant residence time ($\tau$).

Solids recovery given by mass yield ($E_t$) where:

$$E_t = \left( \frac{\text{Supernatant absorbance at 690nm}}{\text{Feed absorbance at 690nm}} \right)$$

1.5 mL samples spun at 2350 g in an eppendorf centrifuge for each mimic. 1 mL disc stack samples read for light absorbance at each flow rate. Results given to within 5% deviation from the mean.

<table>
<thead>
<tr>
<th>Figure 5.9.4 (a)</th>
<th>30% v/v homogenised yeast</th>
<th>Qb/$\Sigma$ = Q/$\Sigma$</th>
<th>Full disc stack</th>
<th>25% disc stack</th>
</tr>
</thead>
<tbody>
<tr>
<td>settling time (s)</td>
<td>14</td>
<td>20</td>
<td>37</td>
<td>14</td>
</tr>
<tr>
<td>laboratory mimic</td>
<td>$E_t$</td>
<td>0.87</td>
<td>0.87</td>
<td>0.88</td>
</tr>
<tr>
<td>pilot disc stack</td>
<td>$E_t$</td>
<td>0.85</td>
<td>0.88</td>
<td>0.90</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>Figure 5.9.4 (b)</th>
<th>30% v/v homogenised yeast</th>
<th>$t_b = \tau$</th>
<th>Full disc stack</th>
<th>25% disc stack</th>
</tr>
</thead>
<tbody>
<tr>
<td>settling time (s)</td>
<td>21</td>
<td>30</td>
<td>55</td>
<td>87</td>
</tr>
<tr>
<td>laboratory mimic</td>
<td>$E_t$</td>
<td>0.87</td>
<td>0.87</td>
<td>0.88</td>
</tr>
<tr>
<td>pilot disc stack</td>
<td>$E_t$</td>
<td>0.85</td>
<td>0.88</td>
<td>0.90</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Figure 5.9.4 (c)</th>
<th>60% v/v homogenised yeast flocculated with 10 g L$^{-1}$ PEI</th>
<th>Qb/$\Sigma$ = Q/$\Sigma$</th>
<th>Full disc stack</th>
<th>25% disc stack</th>
</tr>
</thead>
<tbody>
<tr>
<td>settling time (s)</td>
<td>5</td>
<td>7</td>
<td>14</td>
<td>5</td>
</tr>
<tr>
<td>laboratory mimic</td>
<td>$E_t$</td>
<td>0.95</td>
<td>0.95</td>
<td>0.96</td>
</tr>
<tr>
<td>pilot disc stack</td>
<td>$E_t$</td>
<td>0.77</td>
<td>0.85</td>
<td>0.96</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Figure 5.9.4 (d)</th>
<th>60% v/v homogenised yeast flocculated with 10 g L$^{-1}$ PEI</th>
<th>$t_b = \tau$</th>
<th>Full disc stack</th>
<th>25% disc stack</th>
</tr>
</thead>
<tbody>
<tr>
<td>settling time (s)</td>
<td>7</td>
<td>11</td>
<td>20</td>
<td>35</td>
</tr>
<tr>
<td>laboratory mimic</td>
<td>$E_t$</td>
<td>0.95</td>
<td>0.96</td>
<td>0.96</td>
</tr>
<tr>
<td>pilot disc stack</td>
<td>$E_t$</td>
<td>0.77</td>
<td>0.85</td>
<td>0.96</td>
</tr>
</tbody>
</table>
5.9.3 Dewatering in industrial centrifuges

Table 5.9.5 shows a comparison of sediments from a laboratory spin and the P600 scroll decanter centrifuge under the same conditions of compression (same RCF). The laboratory test results showed that solids content and complex shear modulus tended to increase with residence time as the solids dewatered under compression. The loss angles also decreased as the solids became more elastic.

Table 5.9.5: Comparison of dewatering in laboratory and pilot scale centrifuges using 60% homogenised yeast flocculated with an equal volume of 0.1 M Borax.

20 mL samples of feed material to the P600 scroll decanter centrifuge were spun in a laboratory centrifuge at 3000g for 5, 10, 20 minutes. The dewatering properties of the sediments so formed are shown compared to those discharged from the scroll decanter centrifuge operating at a feed flow rate 60 L/h, conveyor differential 10 rpm and at pond depths of 13.1, 16.2, 19.3 mm.

<table>
<thead>
<tr>
<th>Pond depth (mm)</th>
<th>Residence time for laboratory mimic (s)</th>
<th>P600 sediment</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>300</td>
<td>600</td>
</tr>
<tr>
<td>13.1 Dwt/wt % G* (Pa)</td>
<td>17.21</td>
<td>17.92</td>
</tr>
<tr>
<td></td>
<td>4530</td>
<td>8420</td>
</tr>
<tr>
<td></td>
<td>9.7</td>
<td>9.8</td>
</tr>
<tr>
<td>16.2 Dwt/wt % G* (Pa)</td>
<td>18.29</td>
<td>18.95</td>
</tr>
<tr>
<td></td>
<td>1900</td>
<td>3910</td>
</tr>
<tr>
<td></td>
<td>8.7</td>
<td>9.4</td>
</tr>
<tr>
<td>19.3 Dwt/wt % G* (Pa)</td>
<td>18.02</td>
<td>19</td>
</tr>
<tr>
<td></td>
<td>861</td>
<td>3710</td>
</tr>
<tr>
<td></td>
<td>14.5</td>
<td>11.8</td>
</tr>
<tr>
<td>Feed material</td>
<td></td>
<td></td>
</tr>
<tr>
<td>prepared in the laboratory</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dwt/wt % G* (Pa)</td>
<td>18.97</td>
<td>20.76</td>
</tr>
<tr>
<td></td>
<td>1070</td>
<td>1590</td>
</tr>
<tr>
<td></td>
<td>11.4</td>
<td>13.4</td>
</tr>
</tbody>
</table>
In the deep pond (19.3 mm) mimic, the level of dewatering matched that of the solids discharge from the scroll decanter centrifuge. This indicates that in this case, the solids residence time in the laboratory mimicked that for the sediment in the scroll decanter centrifuge with respect to compression dewatering.

All the laboratory mimic samples could have been spun longer to show that they were approaching their compression equilibrium at 3000 g, particularly in the case of the shallow and intermediate pond mimics which show a degree of further dewatering in the scroll decanter centrifuge. This dewatering is aided by a long residence time inside the scroll decanter centrifuge, which occurs if the scrolling efficiency is low.

Table 5.9.6 shows a comparison of sediments prepared from the same feed material. The sediments formed by compression in the laboratory from the feed flocculated in the stirred tank appeared to be slightly tougher than those from the centrifuge (sample taken from the feed pipe) in the same solids concentration range. This suggests that some conditioning of the flocs occurred in the stirred tank.

The solids concentration of the sediments formed in the laboratory centrifuge is closest to the solids concentrations from the scroll decanter centrifuge after 40 minutes (2400 s). The residence time of solids in the scroll decanter centrifuge must therefore be equivalent to at least 40 minutes with respect to compression dewatering in the laboratory centrifuge.

The complex shear moduli for the sediments taken from the scroll decanter centrifuge were lower than the corresponding shear moduli given by the laboratory mimics in the same solids concentration range, as shown in Figure 5.9.2. This shows that the sediments were softened inside the scroll decanter centrifuge, irrespective of the flocculation method. This is an important consideration for scale down, since it indicates that the shearing and mixing action of the conveyor has an important effect on the sediment properties, and consequently on how the centrifuge is operated.
Table 5.9.6: Comparison of dewatering in laboratory and pilot scale centrifuges for a suspension of 60% homogenised yeast flocculated with an equal volume of 20 g L\(^{-1}\) PEI.

3 x 20 mL samples of feed material to the P600 scroll decanter centrifuge were spun in a laboratory centrifuge for 300, 6000, 1200, 2400 s at 3000 g. The dewatering properties of the resultant sediments are shown compared to those discharged from the scroll decanter centrifuge operating with a total feed flow rate of 60 L h\(^{-1}\), 19.3 mm pond, conveyor differential speeds of 5 and 3 rpm. Feed 1 material was flocculated in a stirring tank for 0.5 h prior to and during the experiment. Feed 2 material was flocculated inside the feed zone of the centrifuge with two feed streams of 30 L h\(^{-1}\).

<table>
<thead>
<tr>
<th>60% v/v yeast homogenate flocculated with an equal volume of 20 g L(^{-1}) PEI solution</th>
<th>Residence time (s)</th>
<th>Conveyor differential (rpm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed 1</td>
<td>G(^{'}) (Pa)</td>
<td>Dwt/wwt %</td>
</tr>
<tr>
<td>G(^{'}) (Pa)</td>
<td>1763</td>
<td>20.5</td>
</tr>
<tr>
<td>G(^{'}) (Pa)</td>
<td>10</td>
<td>2400</td>
</tr>
<tr>
<td>Feed 2</td>
<td>G(^{'}) (Pa)</td>
<td>Dwt/wwt %</td>
</tr>
<tr>
<td>G(^{'}) (Pa)</td>
<td>845</td>
<td>20.5</td>
</tr>
<tr>
<td>G(^{'}) (Pa)</td>
<td>9.9</td>
<td>8.8</td>
</tr>
</tbody>
</table>

5.9.3.1 The effect of shear on the dewatering properties of sediments prepared from flocculated material

Figure 5.9.3 shows an attempt to mimic the shearing action of the conveyor in the laboratory. The sediments mounted on the rheometer and left without shearing action tend to increase in shear modulus slightly as they dry out. The sheared samples were mounted for the same drying period, and yet the shear moduli tend to decrease during this time due to the shearing action. Experiments such as this could be developed to enable a laboratory prediction as to whether or not a new process material yields a shear sensitive sediment.
Figure 5.9.2: Comparison of sediments formed in a scroll decanter centrifuge and a laboratory bottle centrifuge from the same feed material. Sediments prepared from 60% homogenised yeast flocculated with an equal volume of 10 g L⁻¹ PEI.

Closed Symbols: Suspension separated at 3000 g in a laboratory centrifuge (Denley)
Open Symbols: Suspension separated at 3000 g in a scroll decanter centrifuge (P600), operating with a deep pond, feed flow rate 60 L h⁻¹, a range of conveyor differential speeds.

- □ Complex shear modulus, G*, (Pa)
- ○ Loss angle, δ, (°)
**Figure 5.9.3:** The effect of continuous shear on the viscoelasticity of a flocculated sediment. Sediments prepared from 60% yeast homogenate flocculated with an equal volume of 10 g L$^{-1}$ PEI and settled in a laboratory centrifuge (Denley) at 3000g for 600 s. 1 ml samples mounted on parallel plates (pp30) in the Bohlin rheometer, and viscoelastic measurements made between intervals of continuous shear. $G^*$ and $\delta$ given at 5 Hz. Closed symbols: not sheared. Open symbols: sheared at 20 s$^{-1}$. 

- $\blacksquare$, $\square$ - Complex shear modulus, $G^*$, (Pa)
- $\bullet$, $\circ$ - Loss angle, $\delta$, (°)
5.10 Discussion of laboratory mimic experiments

Turbulent settling and feed zone shear in a disc stack centrifuge has been successfully scaled down by using a modified version of the pilot machine. However, the results in sections 5.9.1-2 show that a laboratory scale down protocol is required which independently mimics shear damage, the turbulent settling zone and dewatering in industrial centrifuges.

It may be possible to determine shear sensitivity by shearing small samples of material in the laboratory (section 5.9.1.1).

Section 5.9.3 has shown that the dewatering properties of sediments are affected by residence time in the centrifuge, and in the case of the scroll decanter centrifuge this is linked to scrolling efficiency. As sediments resident in the decanter centrifuge dewater, they have less tendency to flow with the turning action of the scroll, and instead are transported axially by the scroll with greater efficiency.

Figure 5.10.1 shows data generated from Records (1974) equations for scrolling efficiency applied to the P600 scroll decanter centrifuge operating at a range of conveyor differential speeds. Sediment cannot travel in a direction axial to the bowl due to the nature of the turning forces acting upon it. Consequently, the maximum scrolling efficiency occurs when the path angle is equal to the screw angle, which in this case results in a maximum 98.9% efficiency.

Scrolling efficiency calculations were compared to the results shown in section 5.9.3. In Table 5.9.6, if the residence time of solids in the centrifuge in the case of the deep 19.3 mm pond is about 1200 seconds as shown by the laboratory mimic, then the scrolling efficiency was about 91%. The scrolling efficiencies of the other solids were lower since they were more highly dewatered. The movement of highly dewatered, tough, elastic sediments is inefficient when compared to wetter, softer, more viscous solids which flow more easily. It is likely that the sediment condition contributes to the rapid solids removal and overall separation efficiency of the pilot scale centrifuge as shown in section 5.5.6.
Figure 5.10.1: Residence time of sediment in a scroll decanter centrifuge.
Residence times calculated for P600 centrifuge for various scrolling efficiencies as given in section 3.3.1 (Records 1974).

Scrolling efficiency:

--- 98.89%, ····· 90%, ····· 70%, ····· 50%
In Table 5.9.7, the laboratory mimic residence time matches the scroll decanter centrifuge discharge solids concentration at a conveyor differential of 5 rpm, hence the scrolling efficiency of solids was again about 91%.

Poor scrolling efficiency indicates that some of the energy imparted by the conveyor is dissipated within the sediment and causes mixing. Munro and van Til (1988) noticed layering of casein curd sludge in laboratory bottle centrifuges, and yet the sediment discharged from a scroll decanter centrifuge is homogenous. Evidence of mixing was also reported by B.Madsen (1993) by comparing solids concentrations from consecutive conveyor slots and finding that the degree of dewatering attained was far higher than could be expected during the time taken for sediment to move from one slot to the next. In this case, mixing of compressible sediment by the shearing action of the conveyor assisted dewatering by overturning saturated pores.
6. **DISCUSSION: Development of predictive methods for centrifugal separation and dewatering.**

Mass balances have illustrated the importance of optimising centrifuge operation for maximum dewatering and so achieving acceptable product yields. In section 2.1, separation of the solids phase was shown as one idealised step carried out in the laboratory centrifuge, hence the product yields shown were the maximum achievable. However, experimental work has shown that separation will not be ideal, i.e. there will be solids carried over into the supernatant on scale up to an industrial centrifuge. Hence a multitude of solids separation steps (including filtration) will be required with an associated drop in yield, particularly as filters and disc stack centrifuges give comparatively low levels of dewatering. The sum of multiple solids separation steps can be regarded as a single stage of the process and as such the overall yield for this stage should be at least 90%. It is unlikely this will be achieved in practice.

Drops in yield drive the process towards more dilute solutions, the working volumes of which are limited by size of existing plant or in terms of capital investment for new plant. This gives us an operating window, shown below in Figure 6.1 in which all the limits can be estimated.

**Figure 6.1: Operating window for dewatering**

![Operating window for dewatering](image_url)
Setting the limits on Figure 6.1 can be based for example, on estimating how much product is planned. Finding the operating window enables decisions to be made as to how to develop the process for future production. For example, experimental work has shown that if planning to scale up to a continuously or intermittently operated solids discharge centrifuge design, then the rheological characteristics of the sediment must be examined on a small scale to ensure that discharge will be effective.

Experimental work has also shown that dewatering is optimised against clarification in order to maintain overall separation efficiency, particularly in the scroll decanter centrifuge.

The Σ theories originally put forward by Ambler (1952) provide a rigorous approach to scaling down clarification with reasonable success in the disc stack centrifuge. The formulation of grade efficiency curves from particle size analysis data in section 5.7 showed that scale down of solids recovery by reducing the active separation area in a disc stack centrifuge could be achieved for biological systems using Σ theory. With consideration of breakage functions, this method could also be applied to shear sensitive systems.

Scale down of the separation area in a scroll decanter centrifuge poses more difficult practical problems. Reducing centrifuge capacity by widening the scroll flights would cause difficulties with solids conveyance due to "bridging", where solids turn with the scroll and lose axial velocity (Records 1990). Σ theory alone is not generally used to scale up scroll decanter centrifuges because of the dependence of clarification on solids conveyance.

Σ theory applied to scale down from pilot scale to laboratory centrifuges has not been generally successful for either disc stack or scroll decanter centrifuges in this study. The reason for this was primarily due to the lack of a shearing feed zone and/or a turbulent settling zone in the laboratory mimic. In addition, laboratory compression tests are dependent on long residence times, particularly when mimicking dewatering environments experiencing a shear gradient such as in the scroll decanter centrifuge. This can be seen by
comparing the accuracy of the laboratory mimics and pilot centrifuge sediments in Tables 5.9.5 and 5.9.6.

In contrast to the work on clarification, even the most recent literature such as Mahar (1993) and Mackay (1996) provides unsatisfactory procedures for predicting the dewatering of biological systems in centrifuges. The reason for this is primarily due to a lack of understanding about the rheological properties of different sediments as discussed in section 5.6.1. Scale down of dewatering requires constant solids consistency as well as concentration.

6.1 Characteristic dewatering curves

Ward (1989) first used viscoelastic characterisation to explain the behaviour of yeast and other biological systems in dewatering. He chose what he considered to be typical values of complex shear moduli and loss angles and plotted these values against each other. The resultant chart was loosely divided into sections according to the most appropriate centrifuge operation, as shown in section 1.4.10.

A similar chart is shown in Figure 6.1.1 with dewatering curves which are unique to any one system. They were developed by measuring the rheological properties of material sedimented to compression equilibrium under a range of centrifugal pressures. High relative centrifugal forces produce sediments with greater solids concentration. This is associated with a high resistance to dewatering and greater complex shear moduli. Hence dewatering levels increase as one moves upwards along a curve.

The points indicated by arrows show the complex shear modulus and loss angle for various systems sedimented to their compression equilibrium level of dewatering at 3000 g in a laboratory centrifuge. This section of the curve formed the basis for predictions about centrifuge operation for the initial trials with yeast homogenate flocculated with PEI. The points fall close to the boundary of sector (3) and sector (2) of the chart. Sediments falling into sector (3) of the chart require long residence times in the scroll decanter centrifuge. Hence the centrifuge was run with a low conveyor differential speed to achieve maximum dewatering.
Figure 6.1.1: Characteristic dewatering curves for sediments prepared from flocculated suspensions of homogenised yeast. Sediments collected from laboratory centrifuges at various RCF as for Figure 5.3.3.

Closed symbols: sediment prepared at RCF between 10000-45000g in the laboratory (Denley).
Open symbols: sediment discharged from a scroll decanter centrifuge (P600), operating at 3000g, with a feed flow rate of 60 Lh⁻¹, a deep 19.3 mm pond and conveyor differential speed 3 rpm.

- □ 60% yeast homogenate flocculated with an equal volume of 10 gL⁻¹ PEI
- ▲ 60% yeast homogenate flocculated with an equal volume of 0.1 M borax
- ▼ 60% yeast homogenate pre-clarified with an equal volume of 0.1 M borax, and then flocculated with an equal volume of 10 gL⁻¹ PEI.
Compression equilibrium levels of dewatering could be found without the use of a stroboscopic centrifuge by measuring dewatering of the sediment (by rapid microwave dry weight or viscoelastic analysis) until consecutive samples give identical measurements.

Characteristic dewatering curves move through different sectors of the chart, and this is very important in determining appropriate centrifuge and key operating parameters for pilot scale trials. For example, using a disc stack centrifuge for separating yeast homogenate flocculated with PEI would cause problems with solids discharge if the level of solids dewatering achieved in the bowl was in the upper part of the dewatering curve (in sector 2), hence intermittent solids discharge would have to be frequent to avoid excessive dewatering. However, the scroll decanter centrifuge can discharge this sort of material efficiently.

If a curve falls into sector 2 in the laboratory experiments, then the scroll decanter centrifuge can be fitted with a shallow pond so that the sediment discharge moves rapidly up the curve. A deep pond is normally used unless $\delta < 18^\circ$ and $G^* > 1000$ Pa in the laboratory test.

In the case of yeast homogenate pre-clarified with borax and then flocculated with PEI, the curve lies in sector 1. This indicates that a deep pond would be required in the scroll decanter centrifuge operating at 3000 g in order to discharge these viscous solids. However, as the centrifugal pressure increases, the curve moves towards sector 2. Using a high centrifugal pressure such as in a high speed decanter (see Axelsson et al 1992), then it would be possible to produce a tougher, more elastic sediment and discharge it using a shallow pond. A much higher level of dewatering would be achieved in consequence.

6.2 Operating line for the scroll decanter centrifuge

The relationship between dewatering in the scroll decanter centrifuge and torque output shown in Figure 5.6.3 can be used to produce an operating line for process development as shown in Figure 6.2.1 The operating line shows where the resistance of solids to discharge is met by the turning force of the conveyor.
Linear regression through the origin on all given data:

\[ Y = aX \text{ where } a = 8.2473 \]

Standard deviation = 0.4765

Dotted lines show limit for 95% confidence

Figure 6.2.1: Sediment dewatering properties in relation to the selection of operating parameters in a scroll decanter centrifuge.

Data taken from experiments where total mass of solids collected from overflow and underflow streams was not less than 95% of the feed solids.

- ○ 60% homogenised yeast flocculated with an equal volume of 10 gL\(^{-1}\) PEI,
- ■ 60% homogenised yeast flocculated with an equal volume of 0.1M borax,
- △ 30% homogenised yeast.
In development studies, an appropriate level of dewatering can be found from laboratory compression tests at the same centrifugal pressure as that experienced in the pilot scale centrifuge. A small volume of sediment (1 mL) is required to find $G^*$ at this level of dewatering. The mass discharge rate of solids is calculated from the sediment solids concentration in the test sample and a mass balance against the feed flow rate (found from $\Sigma$ theory) and a knowledge of the feed solids concentration. Hence $G^*M_{\text{sed}}$ is obtained.

The operating line is used to estimate the appropriate combination of scrolling torque and conveyor differential to which the centrifuge must be controlled on the first pilot scale trial. The scrolling torque will be of a high value for maximum dewatering. In general, high torques will be obtained at low conveyor differentials for compressible sediments such as the yeast cell debris flocculated with PEI in this study. However, it is expected that high torques will be obtained together with high conveyor differentials for close packed sediments such as the protein precipitates examined by Devereux et al (1984) and Ward (1989). Hence the operating line may extend to higher values on the abscissa.

A window for scroll decanter centrifuge operation can be superimposed onto Figure 6.2.1. The lower limits are constrained by economic conditions with respect to poor solids conveyance on the abscissa and poor solids recovery on the ordinate. The upper limits are constrained by the size of machine and its specific torque output on the abscissa, and equilibrium dewatering at the specified RCF on the ordinate. Optimal operation will aim to achieve the highest point on the operating line within these limits.

6.3 Problems associated with scale down using laboratory centrifuges

It is difficult to mimic the effect of shearing of the sediment which occurs in a scroll decanter centrifuge by dewatering to equilibrium compression levels in laboratory centrifuges. If the scrolling efficiency is low then solids mixing occurs. Here the compression equilibrium is unstable, and solids release water into the pond from overturned pores. This kind of shear-assisted dewatering has a major impact on highly compressible sediments such as flocculated yeast debris. In the scroll decanter centrifuge, sediments of yeast...
homogenate flocculated with PEI discharged at low conveyor differentials with long residence times (sections 5.5.5 and 5.5.6) were not as close to the laboratory dewatering tests at 3000 g (section 5.9.3) as sediments discharged at higher conveyor differentials (sections 5.5.3 and 5.5.6).

This was due to the poor scrolling efficiency of this material at low conveyor differential speeds, and hence the greater degree of shear-assisted dewatering.

Another important consideration is to examine if the sediment is shear sensitive. In this case, dewatering levels may increase, but progress up and along the characteristic dewatering curve in the pilot centrifuge is hindered since the solids remain soft. This is illustrated in Figure 6.1, where the sediment shown discharged from the scroll decanter centrifuge has a solids concentration 28.4 % dwt/wwt, whereas the adjacent sample of the same material prepared in the laboratory has a solids concentration 20.6 % dwt/wwt. This behaviour can be predicted by exposing sediment to shear in the laboratory as described in section 5.9.3.

7. FUTURE WORK

7.1 Scale down of the disc stack centrifuge

PVAc provides a robust, spherically shaped particle suspension with predictable settling characteristics. This makes it ideal for eliminating machine variables in the scale down process. In contrast to PVAc, biological particle suspensions are often shear-sensitive, and often have ill-defined shape and density factors. Nevertheless, useful information about separation performance of these suspensions can still be found from scale down experiments. Future work might include development of appropriate adjustment procedures to allow for problems such as shear breakage, and extension to more concentrated feed streams where hindered settling occurs.

Further development of this work where the solids holding volume is taken into account is essential. This part of the centrifuge should also be "blanked off" to reduce the active volume required to reach the assumed steady state. Repositioning of the active stack location might be required if the flow
pattern is affected by the solids blanking inserts. If this is successful, then it should be possible to predict the separation performance of a pilot scale machine using laboratory scale volumes of process material.

7.2 Scale down of the scroll decanter centrifuge

The $G'M_{\text{sed}}$ vs $\Delta n(T-T_o)$ relationship requires further exploration and extension using tough, compact systems such as protein precipitates. This should be possible using the close control system now available on the P600 decanter centrifuge at UCL.

The overall recovery of homogenised yeast flocculated with PEI in the scroll decanter centrifuge needs to be improved. Supernatants from scale down tests matched maximum potential clarification tests. Hence shear damage in the centrifuge is not so excessive that good clarification is impossible. In addition, small samples of supernatant exiting the scroll decanter centrifuge in the first 60 s of operation also matched maximum potential clarification tests. This indicates that solids build up contributed to a turbulent settling zone and poor clarification. Hence solids recovery could be improved by using more dilute feed streams and very low throughputs to maximise solids conveyance and reduce its effect on clarification.

A "slender" design of scroll decanter centrifuge as described by Madsen and Madsen (1989) could enable good clarification to be achieved without reducing the flow throughput to impracticable limits.

The disc stack centrifuge currently operates on a much smaller scale than the scroll decanter centrifuge. It would be useful if feed zone breakage of shear sensitive suspensions in the disc stack could be used as a mimic of similar breakage in the scroll decanter centrifuge.
## APPENDIX I:

### MASS BALANCES FOR DEWATERING

**KEY:**
- An input variable which can be easily altered for re-calculation of the spreadsheet.

### Composition of yeast cells:

<table>
<thead>
<tr>
<th>Component</th>
<th>Wild type wt.%</th>
<th>Value used here wt.%</th>
</tr>
</thead>
<tbody>
<tr>
<td>total protein</td>
<td>35-45</td>
<td>40</td>
</tr>
<tr>
<td>80% soluble</td>
<td></td>
<td>32</td>
</tr>
<tr>
<td>20% colloidal</td>
<td></td>
<td>8</td>
</tr>
<tr>
<td>lipid</td>
<td>5-10</td>
<td>8</td>
</tr>
<tr>
<td>DNA</td>
<td>0.3-0.4</td>
<td>0.4</td>
</tr>
<tr>
<td>RNA</td>
<td>5-10</td>
<td>7.6</td>
</tr>
<tr>
<td>stored carbohydrate</td>
<td>30-45</td>
<td>38</td>
</tr>
<tr>
<td>mineral ash (assume insoluble)</td>
<td>4-10</td>
<td>6</td>
</tr>
<tr>
<td>total</td>
<td></td>
<td>100</td>
</tr>
</tbody>
</table>

### Basis: volume of cell suspension entering homogeniser

- Scale of fermentation required = 10 1
- Fermenter volume = 12.00 1

- Cell concn at end of fermentation = 60 g/l
- Harvest concn factor = 10 ×
- Initial cell concn = 600 g/l
- Initial cell concn (dcw) = 180 g dcw/l

### HOMOGENISATION:

\[
\log \left( \frac{R_{m}}{(R_{m}-R)} \right) = KNP^{2.9}
\]

- Here: \( K = 3.5E-09 \) for yeast disrupted at 5 oC
- \( N = 5 \)
- \( P = 550 \) kgf/cm²

- \( R = 0.9718 \) Rm

<table>
<thead>
<tr>
<th>Component</th>
<th>IN Mass (g)</th>
<th>IN concn. (g/l)</th>
<th>OUT Mass (g)</th>
<th>OUT concn. (g/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>whole cells dw</td>
<td>180.00</td>
<td>180.00</td>
<td>5.08</td>
<td>5.08</td>
</tr>
<tr>
<td>soluble protein</td>
<td>57.60</td>
<td>57.60</td>
<td>55.97</td>
<td>55.97</td>
</tr>
<tr>
<td>DNA</td>
<td>0.72</td>
<td>0.72</td>
<td>0.70</td>
<td>0.70</td>
</tr>
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<td>RNA</td>
<td>13.68</td>
<td>13.68</td>
<td>13.29</td>
<td>13.29</td>
</tr>
<tr>
<td>carbohydrate</td>
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<td>66.47</td>
<td>66.47</td>
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<td>10.80</td>
<td>10.80</td>
<td>10.50</td>
<td>10.50</td>
</tr>
<tr>
<td>PEI</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>extracellular water</td>
<td>400.00</td>
<td>808.14</td>
<td></td>
<td></td>
</tr>
<tr>
<td>intracellular water</td>
<td>420.00</td>
<td>11.86</td>
<td></td>
<td></td>
</tr>
<tr>
<td>total</td>
<td>1000.00</td>
<td>1000.00</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

- soluble protein step yield = 97%
**PEI FLOCCULATION:**

0.075% PEI flocculates an equal volume of 225 gdcw/1 cell homogenate.

<table>
<thead>
<tr>
<th>Volume mixing ratio</th>
<th>Volume mixing ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1 : 1</td>
<td></td>
</tr>
</tbody>
</table>

**Cell concn for this mass balance:**

- PEI = 0.060 g
- PEI = 0.060 g
- PEI concn = 0.060 g/l

**% remaining in soln when crude homogenate is treated with PEI on a laboratory scale:**

<table>
<thead>
<tr>
<th>Component</th>
<th>% Remaining in Soln</th>
</tr>
</thead>
<tbody>
<tr>
<td>Whole cells</td>
<td>0.0</td>
</tr>
<tr>
<td>Soluble protein</td>
<td>100.0</td>
</tr>
<tr>
<td>Colloidal protein</td>
<td>16.0</td>
</tr>
<tr>
<td>Lipid</td>
<td>2.3</td>
</tr>
<tr>
<td>DNA</td>
<td>10.0</td>
</tr>
<tr>
<td>RNA</td>
<td>10.0</td>
</tr>
<tr>
<td>Carbohydrate</td>
<td>50.0</td>
</tr>
<tr>
<td>Ash</td>
<td>0.0</td>
</tr>
<tr>
<td>PEI</td>
<td>10.0</td>
</tr>
<tr>
<td>Extracellular water</td>
<td>95.6</td>
</tr>
<tr>
<td>Intracellular water</td>
<td>0.0</td>
</tr>
</tbody>
</table>

**IN OUT**

<table>
<thead>
<tr>
<th>Component</th>
<th>Mass Concn (g)</th>
<th>PEI Mass Concn (g)</th>
<th>Solids Mass Concn (g)</th>
<th>Liquor Mass Concn (g/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Whole cells</td>
<td>5.08</td>
<td>0.00</td>
<td>5.08</td>
<td>0.00</td>
</tr>
<tr>
<td>Soluble Protein</td>
<td>55.97</td>
<td>0.00</td>
<td>0.00</td>
<td>55.97</td>
</tr>
<tr>
<td>Colloidal Protein</td>
<td>13.99</td>
<td>0.00</td>
<td>0.00</td>
<td>13.99</td>
</tr>
<tr>
<td>Lipid</td>
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<td>0.00</td>
<td>0.00</td>
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</tr>
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<td>0.00</td>
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</tr>
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<td>0.00</td>
<td>0.00</td>
<td>1.33</td>
</tr>
<tr>
<td>Carbohydrate</td>
<td>66.47</td>
<td>0.00</td>
<td>0.00</td>
<td>34.57</td>
</tr>
<tr>
<td>Ash</td>
<td>10.50</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>PEI</td>
<td>0.00</td>
<td>0.06</td>
<td>0.60</td>
<td>0.01</td>
</tr>
<tr>
<td>Extracellular Water</td>
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<td>0.00</td>
<td>868.18</td>
</tr>
<tr>
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<td>0.00</td>
</tr>
<tr>
<td>Total</td>
<td>1006.00</td>
<td>100.06</td>
<td>138.65</td>
<td>961.35</td>
</tr>
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</table>

**Soluble protein step yield:**

100 %

**IDEAL SEPARATION**

<table>
<thead>
<tr>
<th>Component</th>
<th>Mass Concn (g)</th>
<th>Solids Concn (g)</th>
<th>Liquor Concn (g/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Whole cells</td>
<td>5.08</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Soluble Protein</td>
<td>55.97</td>
<td>0.00</td>
<td>55.97</td>
</tr>
<tr>
<td>Colloidal Protein</td>
<td>13.99</td>
<td>0.00</td>
<td>13.99</td>
</tr>
<tr>
<td>Lipid</td>
<td>13.99</td>
<td>0.00</td>
<td>2.24</td>
</tr>
<tr>
<td>DNA</td>
<td>0.70</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>RNA</td>
<td>13.99</td>
<td>0.00</td>
<td>1.33</td>
</tr>
<tr>
<td>Carbohydrate</td>
<td>66.47</td>
<td>0.00</td>
<td>34.57</td>
</tr>
<tr>
<td>Ash</td>
<td>10.50</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>PEI</td>
<td>0.00</td>
<td>0.06</td>
<td>0.01</td>
</tr>
<tr>
<td>Extracellular Water</td>
<td>808.14</td>
<td>0.06</td>
<td>868.18</td>
</tr>
<tr>
<td>Intracellular Water</td>
<td>11.86</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Total</td>
<td>138.65</td>
<td>961.35</td>
<td>637.84</td>
</tr>
</tbody>
</table>

**Soluble protein step yield:**

66 %
MASS BALANCES FOR SCALE-DOWN

KEY:
- An input variable which can be easily altered for re-calculation of the spreadsheet.

Composition of yeast cells:

<table>
<thead>
<tr>
<th>Wild type</th>
<th>Value used here</th>
</tr>
</thead>
<tbody>
<tr>
<td>wt.%</td>
<td>wt.%</td>
</tr>
<tr>
<td>total protein</td>
<td>35-45</td>
</tr>
<tr>
<td>80% soluble</td>
<td>32</td>
</tr>
<tr>
<td>20% colloidal</td>
<td>8</td>
</tr>
<tr>
<td>lipid</td>
<td>5-10</td>
</tr>
<tr>
<td>DNA</td>
<td>0.3-0.4</td>
</tr>
<tr>
<td>RNA</td>
<td>5-10</td>
</tr>
<tr>
<td>stored carbohydrate</td>
<td>30-45</td>
</tr>
<tr>
<td>mineral ash (assume insoluble)</td>
<td>4-10</td>
</tr>
<tr>
<td>total</td>
<td>100</td>
</tr>
</tbody>
</table>

Basis: volume of cell suspension entering homogeniser = 0.04 l

Scale of fermentation required = 0.138 l
Fermenter volume = 0.17 l
Cell concn at end of fermentation = 60 g/l
Harvest concn factor = 3.45 x
Initial cell concn = 207 g/l
Initial cell concn (dcw) = 62 g dcw/l
ADH activity in units (umoles/min) = 570 units/g
ADH concentrations given in units/ml

HOMOGENISATION

\[ \log \left( \frac{R}{Rm - R}\right) = KNPA^{2.9} \]

Here: K = 3.3E-09 for yeast disrupted at 5 oC
N = 5
P = 550 kg/cm²

Now
\[ R = 0.9718 Rm \]

<table>
<thead>
<tr>
<th>IN</th>
<th>Mass (g)</th>
<th>concn (g/l)</th>
<th>OUT</th>
<th>Mass (g)</th>
<th>concn (g/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>whole cells dcw</td>
<td>2.48</td>
<td>62.10</td>
<td>0.07</td>
<td>1.75</td>
<td></td>
</tr>
<tr>
<td>soluble protein</td>
<td>0.79</td>
<td>19.87</td>
<td>0.77</td>
<td>19.31</td>
<td></td>
</tr>
<tr>
<td>Colloidal protein</td>
<td>0.20</td>
<td>4.97</td>
<td>0.19</td>
<td>4.83</td>
<td></td>
</tr>
<tr>
<td>lipid</td>
<td>0.20</td>
<td>4.97</td>
<td>0.19</td>
<td>4.83</td>
<td></td>
</tr>
<tr>
<td>DNA</td>
<td>0.01</td>
<td>0.25</td>
<td>0.01</td>
<td>0.24</td>
<td></td>
</tr>
<tr>
<td>RNA</td>
<td>0.19</td>
<td>4.72</td>
<td>0.18</td>
<td>4.59</td>
<td></td>
</tr>
<tr>
<td>carbohydrate</td>
<td>0.94</td>
<td>23.60</td>
<td>0.92</td>
<td>22.93</td>
<td></td>
</tr>
<tr>
<td>ash</td>
<td>0.15</td>
<td>3.73</td>
<td>0.14</td>
<td>3.62</td>
<td></td>
</tr>
<tr>
<td>PEI</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td></td>
</tr>
<tr>
<td>extracellular water</td>
<td>31.72</td>
<td>37.35</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>intracellular water</td>
<td>5.80</td>
<td>0.16</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>total</td>
<td>40.00</td>
<td>40.00</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>ADH activity units</td>
<td>22156</td>
<td>554 (units/ml)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Step soluble protein yield</td>
<td>97 %</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
PEI FLOCCULATION

volume mixing ratio = \[ \frac{\text{PEI}}{\text{homogenate}} = \frac{1}{1} \]

0.075% PEI flocculates an equal volume of 225 gdcw/l cell homogenate.

Cell concn for this mass balance = 62.100 gdcw/l

PEI for 1 l homogenate = 0.021 g
PEI for 0.04 l homogenate = 0.001 g
PEI concn = 0.021 g/l

% remaining in soln when crude homogenate is treated with PEI on a laboratory scale:

<table>
<thead>
<tr>
<th>Component</th>
<th>% Remaining in Soln</th>
</tr>
</thead>
<tbody>
<tr>
<td>Whole cells</td>
<td>0.0</td>
</tr>
<tr>
<td>Soluble protein</td>
<td>100.0</td>
</tr>
<tr>
<td>Colloidal protein</td>
<td>16.0</td>
</tr>
<tr>
<td>Lipid</td>
<td>2.3</td>
</tr>
<tr>
<td>DNA</td>
<td>10.0</td>
</tr>
<tr>
<td>RNA</td>
<td>10.0</td>
</tr>
<tr>
<td>Carbohydrate</td>
<td>50.0</td>
</tr>
<tr>
<td>Ash</td>
<td>0.0</td>
</tr>
<tr>
<td>PEI</td>
<td>10.0</td>
</tr>
<tr>
<td>Extracellular water</td>
<td>95.6</td>
</tr>
<tr>
<td>Intracellular water</td>
<td>0.0</td>
</tr>
<tr>
<td>ADH activity</td>
<td>145</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Component</th>
<th>Mass Conc (g/l)</th>
<th>Mass Conc (g/l)</th>
<th>Mass Conc (g)</th>
<th>Mass Conc (g/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Whole cells dew</td>
<td>0.07</td>
<td>1.75</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Soluble protein</td>
<td>0.77</td>
<td>19.31</td>
<td>0.00</td>
<td>10.27</td>
</tr>
<tr>
<td>Colloidal protein</td>
<td>0.19</td>
<td>4.83</td>
<td>0.00</td>
<td>0.77</td>
</tr>
<tr>
<td>Lipid</td>
<td>0.19</td>
<td>4.83</td>
<td>0.00</td>
<td>0.77</td>
</tr>
<tr>
<td>DNA</td>
<td>0.01</td>
<td>0.24</td>
<td>0.00</td>
<td>0.01</td>
</tr>
<tr>
<td>RNA</td>
<td>0.18</td>
<td>4.59</td>
<td>0.00</td>
<td>0.17</td>
</tr>
<tr>
<td>Carbohydrate</td>
<td>0.92</td>
<td>22.93</td>
<td>0.00</td>
<td>6.10</td>
</tr>
<tr>
<td>Ash</td>
<td>0.14</td>
<td>3.62</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>PEI</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Extracellular water</td>
<td>37.35</td>
<td>40.00</td>
<td>3.40</td>
<td>73.95</td>
</tr>
<tr>
<td>Intracellular water</td>
<td>0.16</td>
<td>0.00</td>
<td>0.16</td>
<td>0.00</td>
</tr>
<tr>
<td>Total</td>
<td>40.00</td>
<td>40.00</td>
<td>4.77</td>
<td>75.23</td>
</tr>
<tr>
<td>ADH activity units</td>
<td>22156</td>
<td>554</td>
<td>0</td>
<td>0.00</td>
</tr>
</tbody>
</table>

Step soluble protein yield = 100 %
### DEWATERING: SCROLL DECANTER CENTRIFUGE

**solids separation**
- 40% solids total carried over in liquor
- 30% dry/wet

<table>
<thead>
<tr>
<th>IN</th>
<th>OUT</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>solids liquor</strong></td>
<td><strong>Solid sludge liquor</strong></td>
</tr>
<tr>
<td>(g)</td>
<td>(g/dL)</td>
</tr>
<tr>
<td>whole cells dw</td>
<td>0.07</td>
</tr>
<tr>
<td>soluble protein</td>
<td>0.00</td>
</tr>
<tr>
<td>Colloidal protein</td>
<td>0.16</td>
</tr>
<tr>
<td>lipid</td>
<td>0.19</td>
</tr>
<tr>
<td>DNA</td>
<td>0.01</td>
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<tr>
<td>RNA</td>
<td>0.17</td>
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<tr>
<td>carbohydrate</td>
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<tr>
<td>ash</td>
<td>0.14</td>
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<tr>
<td>PEI</td>
<td>0.00</td>
</tr>
<tr>
<td>extracellular water</td>
<td>3.40</td>
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<tr>
<td>intracellular water</td>
<td>0.16</td>
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<tr>
<td>total</td>
<td>4.77</td>
</tr>
<tr>
<td>ADH activity units</td>
<td>32126</td>
</tr>
</tbody>
</table>

Step soluble protein yield: 91%
Approx. solid sludge sample volume: 10 ml

### CLARIFICATION: DISC-STACK CENTRIFUGE

**solids separation**
- 10% solids total carried over in liquor
- 14% dry/wet

<table>
<thead>
<tr>
<th>IN</th>
<th>OUT</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>solids liquor</strong></td>
<td><strong>Solid sludge liquor</strong></td>
</tr>
<tr>
<td>(g)</td>
<td>(g/dL)</td>
</tr>
<tr>
<td>whole cells dw</td>
<td>0.03</td>
</tr>
<tr>
<td>soluble protein</td>
<td>0.00</td>
</tr>
<tr>
<td>Colloidal protein</td>
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<tr>
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<tr>
<td>DNA</td>
<td>0.00</td>
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<tr>
<td>RNA</td>
<td>0.07</td>
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<tr>
<td>carbohydrate</td>
<td>0.18</td>
</tr>
<tr>
<td>ash</td>
<td>0.06</td>
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<tr>
<td>extracellular water</td>
<td>1.36</td>
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<tr>
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<tr>
<td>total</td>
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<tr>
<td>ADH activity units</td>
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</tr>
</tbody>
</table>

Soluble protein yield: 85%
Approx. solid sludge sample volume: 12 ml
### CLARIFICATION: FILTER POLISH

**solids separation**
- 0 % solids carried over in liquor
- 5 % dry/wt

<table>
<thead>
<tr>
<th></th>
<th>IN</th>
<th>OUT</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>solids</strong></td>
<td>Mass (g)</td>
<td>Mass (g)</td>
</tr>
<tr>
<td>whole cells</td>
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<td>0.00</td>
</tr>
<tr>
<td>dry</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>soluble protein</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Colloidal</td>
<td>0.01</td>
<td>0.01</td>
</tr>
<tr>
<td>protein</td>
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<td>0.00</td>
</tr>
<tr>
<td>DNA</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>RNA</td>
<td>0.01</td>
<td>0.01</td>
</tr>
<tr>
<td>carbohydrate</td>
<td>0.02</td>
<td>0.02</td>
</tr>
<tr>
<td>ash</td>
<td>0.01</td>
<td>0.01</td>
</tr>
<tr>
<td>PEI</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>extracellular</td>
<td>0.14</td>
<td>0.14</td>
</tr>
<tr>
<td>water</td>
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<td>0.00</td>
</tr>
<tr>
<td><strong>total</strong></td>
<td>0.19</td>
<td>0.19</td>
</tr>
<tr>
<td><strong>ADH activity units</strong></td>
<td>115.6</td>
<td>42.7</td>
</tr>
</tbody>
</table>

**Solids sludge**
- liquor concn (g/l)
- solid overflow concn (g/l)

<table>
<thead>
<tr>
<th></th>
<th>IN</th>
<th>OUT</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>solids</strong></td>
<td>Mass (g)</td>
<td>Mass (g)</td>
</tr>
<tr>
<td>whole cells</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>dry</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>soluble protein</td>
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<td>0.00</td>
</tr>
<tr>
<td>Colloidal</td>
<td>0.01</td>
<td>0.01</td>
</tr>
<tr>
<td>protein</td>
<td>0.01</td>
<td>0.01</td>
</tr>
<tr>
<td>DNA</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>RNA</td>
<td>0.01</td>
<td>0.01</td>
</tr>
<tr>
<td>carbohydrate</td>
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<td>0.02</td>
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<tr>
<td>ash</td>
<td>0.01</td>
<td>0.01</td>
</tr>
<tr>
<td>PEI</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>extracellular</td>
<td>0.14</td>
<td>0.14</td>
</tr>
<tr>
<td>water</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td><strong>total</strong></td>
<td>0.19</td>
<td>0.19</td>
</tr>
<tr>
<td><strong>ADH activity units</strong></td>
<td>115.6</td>
<td>42.7</td>
</tr>
</tbody>
</table>

**Liquor**
- liquor concn (g/l)

<table>
<thead>
<tr>
<th></th>
<th>IN</th>
<th>OUT</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>solids</strong></td>
<td>Mass (g)</td>
<td>Mass (g)</td>
</tr>
<tr>
<td>whole cells</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
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<tr>
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<tr>
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<td>protein</td>
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<td>RNA</td>
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<tr>
<td><strong>ADH activity units</strong></td>
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**Liquid overflow**
- liquor concn (g/l)

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<tbody>
<tr>
<td><strong>solids</strong></td>
<td>Mass (g)</td>
<td>Mass (g)</td>
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<td>ash</td>
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<td>0.01</td>
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<tr>
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<tr>
<td><strong>total</strong></td>
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<tr>
<td><strong>ADH activity units</strong></td>
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<td>42.7</td>
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</tbody>
</table>

**CHROMATOGRAPHIC PURIFICATION**

- Soluble protein yield = 75.0%
- Purification factor = 8 x
- DNA clearance = 1.3E-05 x
- Q sepharose loading capacity = 6.5 mg protein/ml gel
- Column volume required = 86 ml

If viscosity of protein stream exceeds 4cp, then dilution is required.

**Dilution factor used here** = 4 x

<table>
<thead>
<tr>
<th></th>
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<th>OUT</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Product stream</strong></td>
<td>liquor concn (g/l)</td>
<td>Elution buffer liquor concn (g/l)</td>
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<tr>
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<td>42.7</td>
</tr>
</tbody>
</table>

**Step soluble protein yield** = 94%

**Approx. solid sludge sample volume** = 4 ml

<table>
<thead>
<tr>
<th></th>
<th>IN</th>
<th>OUT</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Product stream</strong></td>
<td>liquor concn (g/l)</td>
<td>Elution buffer liquor concn (g/l)</td>
</tr>
<tr>
<td><strong>ADH activity units</strong></td>
<td>115.6</td>
<td>42.7</td>
</tr>
</tbody>
</table>

**Step soluble protein yield** = 75%

**Overall soluble protein yield** = 53%

**Approx. liquor sample volume** = 118 ml

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9. **APPENDIX II: Linear viscoelastic theory**

A steady dynamic stress condition is obtained after the start of oscillation in a sinusoidal strain experiment as illustrated in Figure 1.4.3 (a). In practice the steady sinusoidal stress is approached gradually when an elastic liquid is suddenly exposed to a sinusoidal strain, hence measurement commences after a delay period amounting to a number of periods of oscillation frequency.

Consider the situation:

\[
\begin{align*}
\dot{y} &= 0 \quad \text{t}<0 \\
\gamma &= \gamma_o \sin \omega t \quad \text{t}\geq0 \\
\gamma &= 0 \quad \text{t}<0 \\
\dot{\gamma} &= \gamma_o \omega \cos \omega t \quad \text{t}\geq0
\end{align*}
\]

where \(\omega\) = wave frequency

\(\gamma_o\) = maximum strain amplitude

The stress alternates sinusoidally but is out of phase with the strain. The constitutive equation for linear viscoelastic theory in simple shear (Boltzmann superposition principle) is derived from matrices which describe the three-dimensional stress tensors experienced by the sample (Ferry 1980, Whorlow 1980):

\[
\tau(t) = \int_0^\infty G(t-t')\dot{y}(t') \, d't
\]

Inserting \(\dot{y}\):

\[
\tau(t) = \int_0^\infty G(t-t')\gamma_o \omega \cos \omega t' \, d't
\]

As \(t \to \infty\):

\[
\tau = \gamma_o G'(\omega) \sin \omega t + \gamma_o G''(\omega) \cos \omega t
\]

where \(G'(\omega)\) and \(G''(\omega)\) are the storage and loss dynamic moduli respectively, and the time lag \((t-t')\) is abbreviated to \(u\):

\[
\begin{align*}
G' &= \omega \int_0^\infty G(u) \sin \omega u \, du \\
G'' &= \omega \int_0^\infty G(u) \cos \omega u \, du
\end{align*}
\]
G' and G'' are functions of frequency, but not of elapsed time, and so equation (99) can be conveniently written:

\[ \tau = \gamma_o (G' \sin \omega t + G'' \cos \omega t) \quad 102 \]

The maximum amplitude of the stress \( \tau_0(\omega) \) and the phase angle \( \delta(\omega) \) between stress and strain can be expressed in terms of trigonometric relations:

\[ \tau = \tau_0 \sin(\omega t + \delta) = \tau_0 \cos \delta \sin \omega t + \tau_0 \sin \delta \cos \omega t \quad 103 \]

Comparing equations (102) and (103) shows that:

\[ G' = \left( \frac{\tau_0}{\gamma_o} \right) \cos \delta \quad G'' = \left( \frac{\tau_0}{\gamma_o} \right) \sin \delta \]

\[ \text{and} \quad \frac{G''}{G'} = \tan \delta \quad 104 \]

Hence each periodic or dynamic measurement at a given frequency provides simultaneously two independent quantities: either \( G' \) and \( G'' \) or else \( \tan \delta \) and \( \left( \tau_0/\gamma_o \right) \). Sometimes it is more convenient to express the sinusoidally varying stress as a complex quantity. For this purpose, the sinusoidally varying strain is first expressed showing a phase shift of \( \pi/2 \):

\[ \gamma = \gamma_o \cos \omega t \quad 105 \]

Hence the shear stress for linear viscoelastic behaviour is:

\[ \tau = \gamma_o (G' \cos \omega t - G'' \sin \omega t) = \gamma_o \sqrt{G'^2 + G''^2} \cos(\omega t + \delta) \quad 106 \]

Complex, time independent strain and stress can be defined as:

\[ \gamma^* = \gamma_e e^{i \omega t} \quad \text{and} \quad \tau^* = \tau_e e^{i(\omega t + \delta)} \quad 107 \]
A complex shear modulus may now be defined:

\[ G^* = \frac{\tau}{\gamma} = \left( \frac{\tau_o}{\gamma_o} \right) e^{i\delta} \]  

\[ G' + iG'' = \left( \frac{\tau_o}{\gamma_o} \right) (\cos\delta + i \sin\delta) \] 

Taking real and imaginary parts:

\[ G' = \left( \frac{\tau_o}{\gamma_o} \right) \cos\delta \quad G'' = \left( \frac{\tau_o}{\gamma_o} \right) \sin\delta \]

as stated earlier. Complex notation enables the use of the complex shear modulus to describe linear viscoelastic behaviour, which unlike \((\tau/\gamma)\) is time invariant.
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