



Introduction

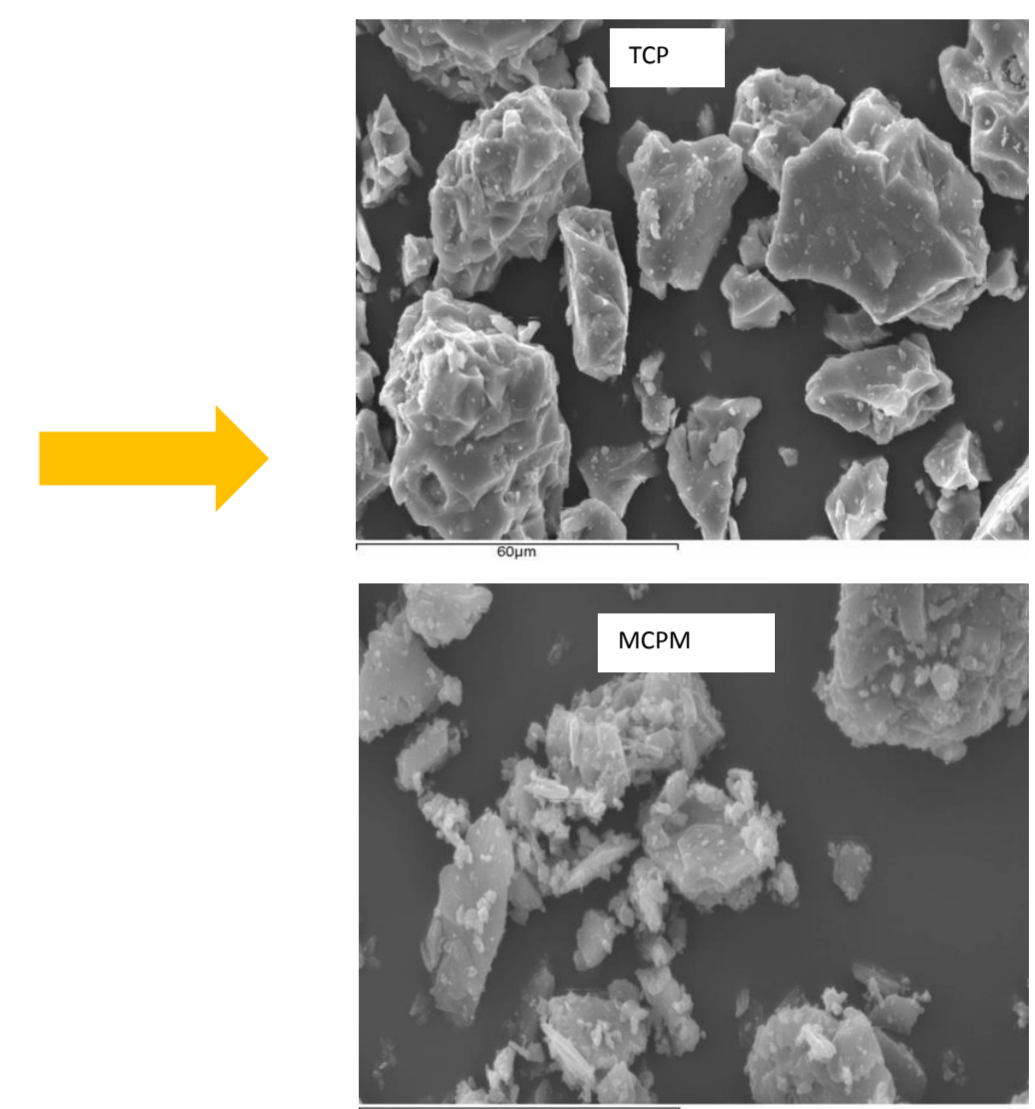
Bacteria cause caries by producing acid that dissolves mineral in teeth. Currently white dental composites are used to just fill the cavities formed in affected teeth. They have higher failure rates than amalgam but the later will soon be phased out due to the Minamata agreement to reduce all mercury use. Dental composite failure occurs as a result of material shrinkage during placement causing de-bonding. Current composites also inhibit the natural tooth repair mechanism of mineral precipitation. This problem may be solved by using re-mineralising dental composites which encourage tooth repair through releasing calcium phosphate and improve bonding between the restorative and dentine.

Objectives

The aims of this study were to understand the effect of reactive calcium phosphate as re-mineralising agents on polymer based composites. Assessments were carried out according to ISO standards with the aid of Raman spectroscopy.

Methods and Materials

Composites were prepared by mixing a liquid and powder in the ratio of 4:1. The powder contained 10 or 20 wt.% calcium phosphate (CaP) (1:1 weight ratio of mono-calcium phosphate (MCPM) and β -tri-calcium phosphate(TCP)) in a standard dental glass filler. The liquid consisted of urethane dimethacrylate : poly(propylene glycol) dimethacrylate 3:1. The depth of cure, monomer conversions at 1mm depth, Polymerisation shrinkage, water sorption/solubility and flexural strength were assessed using ISO 4049:2009, ISO 17304:2013 and FTIR. Re-mineralising capability (ISO 23317;2007), assessed through observation of hydroxyapatite (HA) precipitation on material surfaces in simulated body fluid (SBF), was quantified and confirmed by scanning electron microscopy (SEM) and Raman spectroscopy.



Results and Discussion

The results in table 1 demonstrate that depth of cure, monomer conversion, polymerisation shrinkage and flexural strength all decrease slightly with increasing CaP content. Comparison of water sorption in water and in simulated body fluid (SBF) at 7 days in Fig.1 shows that both formulations absorb more water in SBF than in water. The 10% CaP formulation has a negative value of solubility in both medium (Fig.2). This could be a consequence of some water being bound when fillers react to form brushite. Precipitation of HA on both composite surfaces was confirmed by Raman (Fig. 3).

The 20% CaP composite has greater apparent solubility in water than in SBF. The difference is 2.3 mg/cm³ but only 0.2 mg/cm³ with the 10% CaP composite. This could mean that the precipitation layer of HA with 20% CaP may be thicker and denser. This is consistent with SEM images (Fig.4). Both formulations pass all required ISO standard values.

Table 1: Assessment results

	Depth of cure (mm)	Monomer Conversion (%)	Polymerisation shrinkage (vol.%)	Flexural strength (MPa)	Water Sorption (mg/cm ³)	Solubility (mg/cm ³)
ISO standard	> 1.5	> 50	-----	> 80	< 40	< 7.5
10 wt.% CaP	2.06	90	2.1	110.3	18.2 (H ₂ O) 20.8 (SBF)	-1.3 (H ₂ O) -1.5 (SBF)
20 wt.% CaP	2.00	84	1.9	87.1	30.6 (H ₂ O) 36.5 (SBF)	4.2 (H ₂ O) 1.9 (SBF)

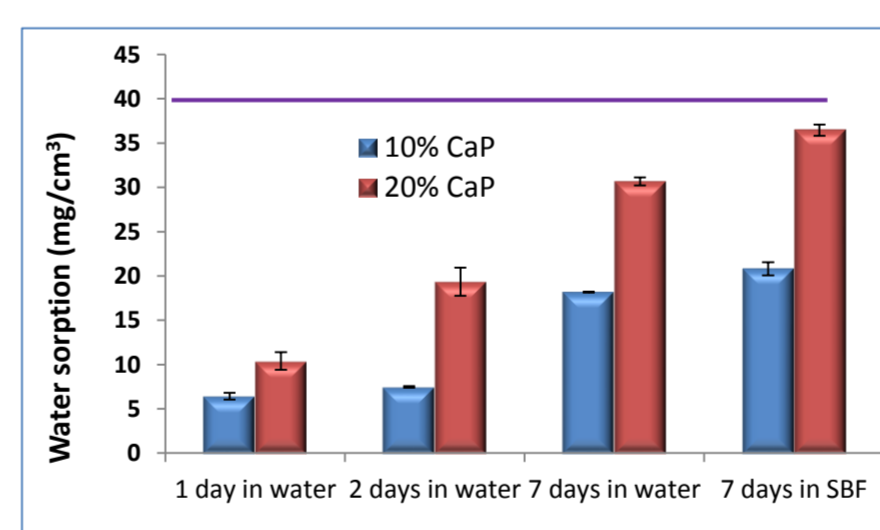


Figure 1: Water sorption

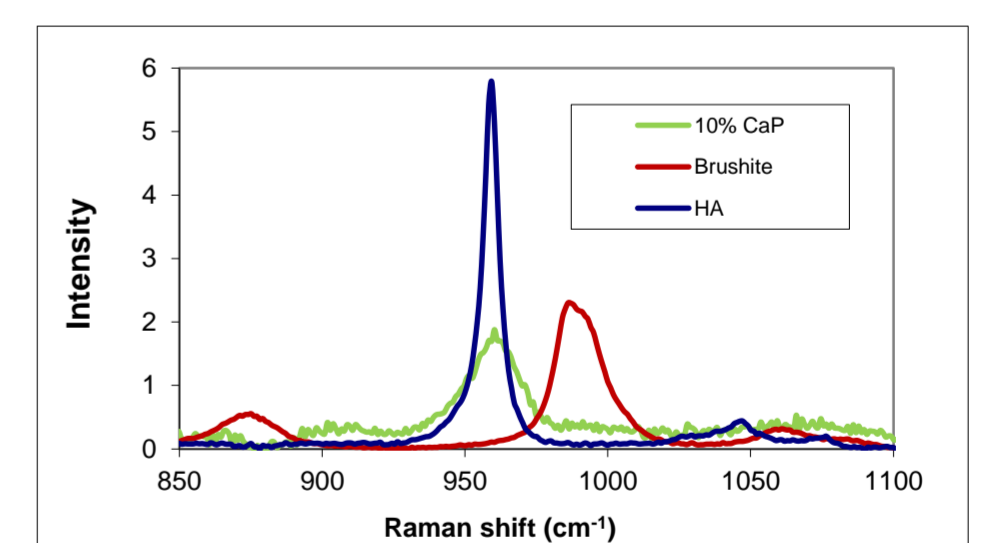


Figure 3: Raman spectroscopy

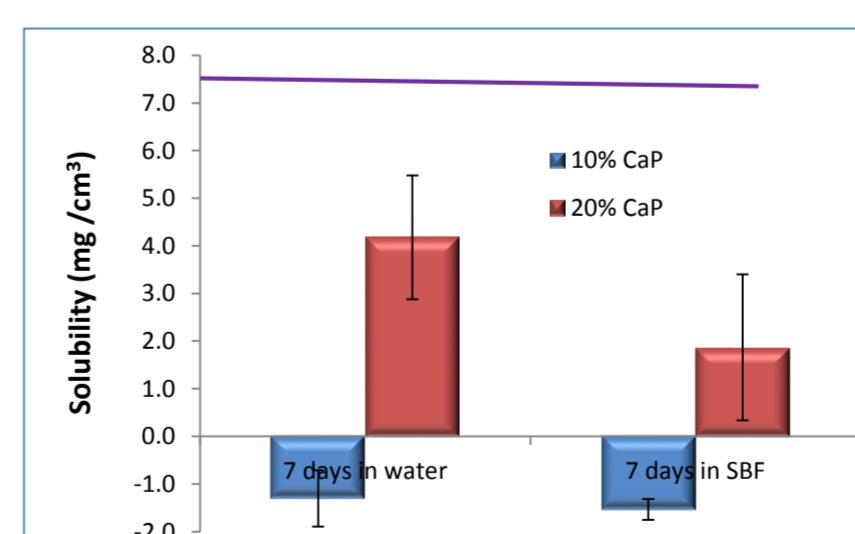


Figure 2: Solubility

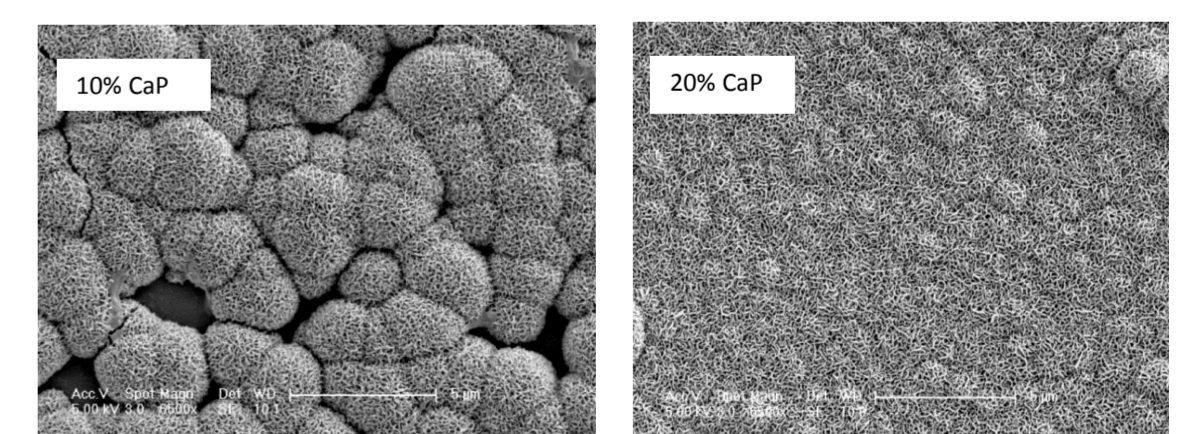


Figure 4: SEM images of HA layer

Conclusions

Incorporation of reactive calcium phosphate into dental composites enhances HA precipitation on the material surfaces in SBF. Raman spectroscopy proved to be effective at distinguishing HA from other forms of calcium phosphate on the composite surfaces. The formation of HA was enhanced with increase of CaP content. This study confirmed that CaP filled composites can have this advantageous feature whilst still passing ISO standard tests.

Acknowledgments

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