

Mechanical behavior of a hardened oil-silica sand composite

Ke Chen¹, Hongjie Lin², Deyun Liu³, Manman Hu⁴, Béatrice A. Baudet⁵, Sérgio D.N. Lourenço⁶

ABSTRACT

To evaluate the mechanical behavior of a potential soil stabilizer (a hardening Tung oil) with a coarse silica sand, the strength characteristics at both peak and critical state, and the stress-dilatancy relationship were investigated on both natural sand and the hardened oil-sand composite. Tung oil concentration and heating duration varied from 3 to 5% and 1 to 3 days, respectively. Triaxial compression tests were performed under effective confining pressures ranging from 50 to 800 kPa, in both drained and undrained conditions. The results revealed a stress-dependent behavior. Compared to natural sand, the peak strength of the hardened oil-sand composite was enhanced at a confining pressure of 50-200 kPa, decreasing at higher confining pressures (400-800 kPa). The stress ratio at critical state of the composite decreased at low confining pressures (50-200 kPa), followed by an increase at high confining pressures (200-800 kPa). The peak strength envelope exhibited a similar stress-dependent trend. SEM-EDS and TGA were used to identify the mechanisms controlling the behavior, by measuring the ratio of silicon (Si) to carbon (C) on particle surface, and the weight loss of Tung oil, respectively. Under high confining pressures (>200 kPa), coating abrasion was associated with increasing ratio of Si to C and decreasing weight loss of Tung oil. This peculiar stress-

¹ PhD Student, Department of Civil Engineering, The University of Hong Kong, Haking Wong Building LG04, Hong Kong S.A.R., China.

² Assistant Professor, School of Civil Engineering, Sun Yat-sen University, Guangzhou, Guangzhou 510275, China.

³ Research Assistant Professor, Department of Civil & Environmental Engineering, The Hong Kong University of Science and Technology, Clear Water Bay, Hong Kong S.A.R., China.

⁴ Assistant Professor, Department of Civil Engineering, The University of Hong Kong, Haking Wong Building 621, Hong Kong S.A.R., China.

⁵ Professor, Department of Civil, Environmental and Geomatic Engineering, University College London, Chadwick Building, Gower Street, London, WC1E 6BT, UK.

⁶ Associate Professor, Department of Civil Engineering, The University of Hong Kong, Haking Wong Building 621, Hong Kong S.A.R., China. (corresponding author).

dependent behavior of the hardened oil-sand composite was consistent with the recovery of the stress ratio at critical state.

Keywords: sand; Tung oil; mechanical behaviour.

INTRODUCTION

Soil stabilization is a process of improving the shear strength parameters of a soil, thus improving the bearing capacity of a subgrade to support earth structures, to cope with the increasing scarcity of land and demand for natural resources due to rapid urbanization (Ingles and Metcalf, 1972; Makusa, 2013). Among a variety of soil stabilization techniques, are mechanical stabilization, such as the densification of loose sandy soil or drainage of soft clay to avoid liquefaction and excessive or differential settlement (Indraratna *et al.*, 2005; Hejazi *et al.*, 2012) and chemical stabilization, such as stabilization by cement and lime (Gu and Chen, 2020). However, conventional stabilizers are often associated with high greenhouse gas emissions and high energy cost and potential contamination (Gowthaman *et al.*, 2018), and there is a strong incentive to develop new environmentally friendly methods to stabilize weak soil. More environmentally friendly alternatives include grouting materials such as sodium silicate and colloidal silica (Latifi *et al.*, 2014; Wong *et al.*, 2018; Vranna *et al.*, 2022), microbiologically induced calcium carbonate precipitation (Mitchell and Santamarina, 2005; Ivanov and Chu, 2008; Botusharova *et al.*, 2020), self-healing microcapsules-based materials (Cao *et al.*, 2023; Chen *et al.*, 2023, Qi *et al.*, 2024), and nanomaterials such as multi-walled carbon nanotubes, plastic fines and nano-SiO₂ particles (Zuber *et al.*, 2013; Figueiredo *et al.*, 2015; Lv *et al.*, 2018; Correia *et al.*, 2021). A different method using Tung oil is proposed here. Tung oil has been the object of recent research carried out to resolve the strength and water-related problems simultaneously, where it was found that Tung oil could not only induce hydrophobicity (water repellency), but also enhance the UCS (Unconfined Compressive Strength) and aggregate tensile strength of soil (Lin *et al.*, 2019, 2021).

Tung oil is a traditional Chinese vegetable oil, obtained by pressing the seed from the nut of the Tung tree (*Vernicia fordii*). It is cost-effective (7 US\$/liter) and non-toxic. It has conventionally been used as drying oil on wood for centuries due to its high durability (Carter *et*

al., 1989; Zhang *et al.*, 2016). With over 80% of crystalline unsaturated fatty acid, Tung oil can dry in approximately 5 days owing to its reaction with oxygen and polymerize into a glossy solid film. As Tung oil dries in air, the oil molecules (unsaturated fatty acids) will cross-link in a tight complex formation by reacting with oxygen. This oxy-polymerization process will increase the viscosity of Tung oil and form solid films, which also make the surface waterproof. The hardening film or bonding is capable of withstanding tear or abrasion (Samadzadeh *et al.*, 2011). These properties make Tung oil especially useful in paints, wood preservatives and construction (Yang *et al.*, 2015). As a coating material, Tung oil has been applied as an autonomous repairing agent for self-healing epoxy, and a self-healing agent for reinforced concrete (Samadzadeh *et al.*, 2011; Chen *et al.*, 2017).

In soils, mixing Tung oil on its own with CDG (Completely Decomposed Granite) and silica sand was shown to enhance their UCS, and research has focused on the effect of Tung oil concentration, curing temperature and curing time on the degree of UCS enhancement of the soil (Lin *et al.*, 2019, 2021; Chen and Lourenço, 2023). Tang *et al.* (2020) attempted to mix both Tung oil and sticky rice juice to increase the shear strength of silt by means of direct shear box tests. The ecotoxicity of Fujian sand and CDG mixed with Tung oil has also been assessed, showing negligible total concentrations of heavy metals measured by leachate tests, and minimal influence on soil microorganisms, and therefore demonstrating a negligible environmental impact (Lin *et al.*, 2022). Although a potential candidate as green stabilizer in soils, many issues have still not been explored, such as (1) the characterization of the shear behaviour (strength and dilatancy) of the hardened Tung oil-soil composite in saturated condition; (2) the effect of the effective confining pressure on the soil stabilization efficiency (stress ratio and dilatancy at peak) by hardened Tung oil; (3) and whether the CSSM (Critical State Soil Mechanics) framework which is able to describe the behaviour of natural sand can be extended to describe that of Tung oil treated soil, for ease of modelling and numerical analysis in engineering practice (Schofield and Wroth, 1968; Wood, 1990). Tung oil provides

both bonds and coatings to soil particles. As a comparison, Bardet *et al.* (2011) found that coated petroleum wax can improve the shear strength of sand at low pressure via direct shear box in dry condition. The mechanical behavior of polydimethylsiloxane-coated coarse Fujian sand was also investigated by triaxial compression tests, revealing a decreased stress ratio at critical state and peak (Liu *et al.*, 2019; Liu and Lourenço, 2021).

In this paper, the strength and dilatancy characteristics of a coarse silica sand before and after mixing with Tung oil are presented. The effect of heating duration (1-3 days), Tung oil concentration (3-5%) and confining pressure (50-800 kPa) on the mechanical behaviour of both materials is examined. It was found that 3-5% of Tung oil and 1-3 days of heating are effective to induce high and persistent hydrophobicity of both CDG soil (Lin *et al.*, 2021) and coarse silica sand, and significant improvement of UCS (200-400 kPa) of coarse silica sand (Chen and Lourenço, 2023). The efficiency of the Tung oil addition as reinforcing agent is shown through a comparison of the stress-strain curves, dilatancy paths, critical state lines (CSLs) and peak strength envelopes of both natural sand and Tung oil-sand mixtures under different conditions, while complementary analyses using SEM-EDS and thermogravimetric analyses (TGA) shed light on phenomena such as bond breakage or coating abrasion occurring at the particle and microscale.

EXPERIMENTAL METHODS

Materials

A coarse uniform silica sand (0.6 to 1.18 mm; $D_{50}=0.99$ mm) and Tung oil from Jogel Co. (China) were used in the tests. The physical properties of the sand are given in Table 1. The detailed features and chemical composition of this Tung oil have been given by others (Samadzadeh *et al.*, 2011; Yang *et al.*, 2015; Zhang *et al.*, 2016; Lin *et al.*, 2021). Specifically,

Tung oil, which is transparent with amber colour, contains 80% alpha-eleostearic acid and 20% fatty acids. The density of Tung oil is around 0.94 g/cm³ at room temperature (25°C).

The Tung oil-sand mixtures were prepared by mixing Tung oil with the sand at Tung oil concentrations of 3% and 5% by mass of air-dried sand, based on data showing that the UCS can be improved by adding 5% of Tung oil and curing the mixtures for 1-14 days at controlled temperature and relative humidity (Chen and Lourenço, 2023). The specific gravity (G_s) of the natural sand and Tung-oil sand mixtures were determined by the pycnometer method (BS 1377-2:1990) with de-aired water as the wetting agent. The void ratios, e , of the mixtures were calculated using the volume-mass relation of the soil ($e = G_s \rho_w / \rho_d - 1$), with ρ_w the density of water (0.997g/cm³ at 25°C), and ρ_d the dry density of soil. The results showed G_s to be 2.60 and 2.57 for the hardened oil-sand composite with Tung oil concentrations of 3% and 5%, respectively, slightly lower than the G_s of the sand (Table 1), as has also been found for other chemically treated hydrophobic soil such as dimethyldichlorosilane-treated Toyoura sand (Liu *et al.*, 2019) and Zycosoil treated kaolin clay (Choi *et al.*, 2016). Using the same procedures as those used to determine the extreme void ratios of a natural sand (BS 1377-4:1990), values of $e_{\max}=1.11$ and $e_{\min}=0.62$ were determined for the oil-sand mixtures with 3% Tung oil concentration, and $e_{\max}=1.13$ and $e_{\min}=0.59$ for 5% concentration. The smaller $e_{\min}$ (see Table 1) could be attributed to decreasing G_s and lubrication effect caused by the presence of oil, which facilitated compaction (Al-Sanad *et al.*, 1995; Lin *et al.*, 2021). The larger $e_{\max}$ agreed with previous study on oil contaminated soil that soil particles were weakly bonded by the oil to form larger sized aggregates, which created larger voids (Ijimdiya, 2013).

Sample preparation

The triaxial specimens (38 mm in diameter and 76 mm in height) were prepared to obtain a relative density of 70.0±3.1% (or post-consolidation void ratio of 0.791±0.014, 0.767±0.020 and

0.752±0.017 for natural sand, oil-sand mixture with 3% Tung oil concentration and oil-sand mixture with 5% Tung oil concentration, respectively) before shear as both materials manifested a wide range of densities or void ratios. The natural sand specimens were prepared by the moist tamping method with the under compaction-technique so that homogeneity and the desired density could be achieved (Ladd, 1978; Yang and Wei, 2012; Sze and Yang, 2014). In detail, the average mass of each layer was calculated based on the target initial density, with each successive lower and upper layer at 1% more or less to avoid the denser lower portion due to more compaction. The sand was saturated by flushing the specimen with carbon dioxide and de-aired water using a vacuum of 5-10 kPa.

The critical state void ratio e_{cs} of each specimen was initially determined from the initial void ratio during sample preparation and the volumetric change during consolidation and shearing. An alternative method by measuring the final water content at the end of the test to calculate the critical state void ratio e_{cs} was proposed (Verdugo and Ishihara, 1996; Yang and Wei, 2012), to eliminate the likely error caused by CO₂ and de-aired water percolation, and back pressure saturation. An average value based on these two void ratios was adopted to determine the CSLs in e - p' plane.

For the mixtures, the Tung oil and sand were mixed thoroughly until a uniform color was observed, then they were put into a 38 mm diameter by 76 mm height cylindrical split mould. The specimen was prepared following the same 4-layer compaction method but without the 2% water content to avoid potential water and oil incompatibility. The compacted specimens inside the mould, where they were sealed by a porous stone at bottom end, were placed in a temperature-controlled oven and cured at 60°C for 1 or 3 days, in order to investigate the effect of heating duration on the behavior of Tung oil-sand mixture. Mixing soil with Tung oil can lead to aggregation, since Tung oil is able to oxidize and polymerize to semi-solid films (Mustapha *et al.*, 2022) that provide bonding between particles. This phase change can be accelerated by

heating, during which the Tung oil undergoes a rapid crystallization process, producing a hardening effect. In practice, air drying at ambient temperature (25°C) could still achieve a similar hardening efficacy of Tung oil with more time of curation (Qi *et al.*, 2024). In this study, heating was employed to shorten the time of Tung oil hardening, and not as a process used in a practical solution. By choosing an appropriate heating temperature (60°C), the loss of Tung oil due to evaporation of volatile organic compounds is negligible (Lin *et al.*, 2019). After curing, the stabilized composite specimens (Fig. 1(a)) were removed from the mould, and their mass and dimensions were measured in order to compute the void ratios. Two-dimensional (2D) computed tomography (CT) scans using TOSCANER-31300 (Voltage: 130 kV, Focus size: 5 μm , Toshiba Co., Japan) were conducted to observe the spatial distribution of bonding in the porous structure of Tung oil-sand mixtures (5%3d) as shown in Fig. 1(b). The brighter (white) regions correspond to high density areas (sand particles), while the darker (black) regions refer to low density areas (pores). As the initial dry specimen only contains three phases (sand, air and hardened Tung oil), the bonds (grey regions) could be distinguished from the other two, as they primarily occur between grain contacts.

After curing, the specimens were immersed inside a vacuum container filled with de-aired water under a negative pressure of 20 kPa for 48 hours prior to testing, to ensure that the hydrophobicity induced by the Tung oil does not influence the degree of saturation. The specimens were then covered with a membrane enclosed in the membrane stretcher by way of suction and placed in the triaxial cell. De-aired water was percolated to saturate the sample without prior circulation of carbon dioxide. The cell and back pressures were increased consecutively at an effective stress difference of 10kPa until the natural sand and Tung oil-sand mixtures reached a target Skempton B value ($B > 0.95$), indicative of saturation. This was typically achieved for a back pressure of 300 to 500 kPa. After saturation, both types of

specimens were isotropically consolidated under an effective stress, p'_0 . A period equal to double the consolidation time was sustained to ensure equilibrium before shearing.

Testing programme

The testing programme included both drained and undrained triaxial compression tests, under confining pressures of 50 to 800 kPa. Both natural sand and Tung oil-sand mixtures were subjected to a constant axial displacement rate of 0.1 mm/min in drained condition and 0.5 mm/min in undrained condition. The repeatability of the tests on both natural sand and Tung oil-sand mixtures was guaranteed by reproducing the same drained and undrained tests with approximately the same post-consolidation void ratio (see Appendix). The similar soil response indicated that the above-mentioned procedures were suitable to produce consistent specimens. The effect of the shearing rate was checked by varying it between 0.1 and 0.2 mm/min in drained condition, and 0.5 and 1.0 mm/min in undrained condition. It was found that the results were not affected by the rate chosen. Note that although the back pressure was comparatively high at 500 kPa, results were found to be repeatable in the range of 200-500 kPa, indicating that the Tung oil bonding was not affected before shearing. Four isotropic compression tests were performed on all materials at effective confining pressure, p'_0 up to 1400 kPa. Another four oedometer tests were conducted on all materials at effective vertical stress, σ'_v up to around 12 MPa to provide a reference normal compression line in one-dimensional compression condition (1D NCL).

The microstructure of both natural sand and Tung oil-sand mixtures after test was observed in a scanning electron microscope (SEM, Hitachi S-3400N) equipped with EDS (Energy Dispersive X-ray Spectroscopy) detector. Immediately after shearing the specimens were oven dried at 100°C for 1 day, then observed by performing the micro-analysis. The EDS allowed running elemental mapping to capture the distribution and mass content of various

chemical elements on the particle surface. The thermal stability of both dried sand and Tung oil-sand mixtures was evaluated by TGA.

EXPERIMENTAL RESULTS AND ANALYSIS

Isotropic compression of natural sand and hardened oil-sand composites

The void ratio, e versus mean effective stress, p' curves from isotropic compression tests on both natural sand and Tung-oil sand mixtures are shown in Fig. 2(a). Tung oil-sand mixtures show a more compressible behaviour than the sand. For instance, the compressibility index, C_c is 0.0769 for natural sand, which is smaller than 0.0827, 0.0813 and 0.0854, which are the C_c values of Tung oil-sand mixtures (3%1d, 3%3d and 5%3d, respectively). This response closely resembles that of gasoline or diesel oil-contaminated soil (Al-Sanad, *et al.*, 1995; Singh *et al.*, 2008; Taqieddin, 2017; Askarbioki *et al.*, 2019), and may be attributed to a lubricating effect of the Tung oil, though aged and hardened, facilitating the sliding of particles over each other. For another, the low hardness and stiffness of aged Tung oil coating in comparison with sand might also cause more volumetric change (Wang and Erhan, 1999; Yun and Santamarina, 2005).

The tangent bulk modulus, K , plotted against the mean effective stress, p' in Fig. 2(b), initially has a slower rate of increase with stress level for the treated sand, until about 200 kPa, after which there is a sudden and marked increase until the K -value of the treated sand reaches that of the untreated sand around 1 MPa. This may be due to gradual but limited bond breakage, slightly different from what was found by others where the increase in K past a given stress level was more marked and accompanied by bond breakage (e.g., Yun and Santamarina, 2005; Porcino *et al.*, 2012). This bond breakage indicated by isotropic compression data was minor as careful observation of the specimens after testing did not show any significant bond breakage. The effects of heating duration and Tung oil concentration on the compression curve and bulk modulus development appear to be negligible.

Critical state of natural sand and hardened oil-sand composites

Fig. 3 and Fig. 4 show the soil response to drained and undrained shearing of the sand and Tung oil-sand mixture prepared at 5% Tung oil concentration and heated for 3 days (5%3d). Hollow symbols and dashed lines are used to represent the natural sand, while solid symbols and solid lines represent the Tung oil-sand mixtures. Only the peak state and end of test points are shown on the curves. The testing conditions and selected key results are summarized in Table 2. The Tung oil-sand mixtures are stiffer than the sand at low confining stress level, with higher peak stress ratio, M_p , varying from a value of 2.33 at 50 kPa confining stress to 1.31 at 800 kPa confining stress, compared with the M_p values of the sand which vary between 1.68-1.40 (Fig. 3(a), (b)). Both natural sand and Tung oil-sand mixtures show comparable pore water pressure response at the low confining stress of 50 kPa, after which the mixture tends to be more dilative, eventually showing a positive pore pressure response at the confining stress of 800 kPa (Fig. 3(c)). The Tung oil-sand mixture, however, exhibits a less contractive behaviour than the natural sand, possibly due to the phase change of Tung oil. The hardened Tung oil bond could resist the volume change of pore spaces during shearing, contributing to less positive excess pore pressure at the end of shearing under relatively high confining pressure, which is in a manner similar to the effect of densification (Porcino *et al.*, 2012; Georgiannou *et al.*, 2017; Triantafyllos *et al.*, 2022; Vranna *et al.*, 2022).

The results from the drained tests show that the Tung oil-sand mixtures are softer but with higher peak strengths in the 50-200 kPa confining stress region (Fig. 4(b)), and the mixtures develop faster rates of dilation than the natural sand after peak (Fig. 4(c)). The ultimate strengths reached around 25% axial strain are similar for natural sand and the mixture up to large stress levels, although the volumetric behaviour of the Tung oil-sand mixture remains dilative even at large stresses, when the natural sand would be contractive. The lower

efficiency of soil stabilization at high confining pressures, when cementation or bonds might break down and the soil response is dominated by friction has been observed in other bonded geomaterials (e.g. Cuccovillo and Coop, 1999; Consoli *et al.*, 2012; Georgiannou *et al.*, 2017; Ali Rahman *et al.*, 2018).

Fig. 5(a) shows the one-dimensional normal compression line (1D NCL) of both sand and Tung oil-sand mixtures. The 1D NCL in $e-p'$ plane could be obtained by the original compression data in $e-\sigma_v'$ plane and using the equation ($p' = \sigma_v'(3 - 2\sin\phi_{cs})/3$), where the critical state friction angle, ϕ_{cs} could be known from Fig. 5(b). The NCL of natural or bonded sand was curved at lower stresses (Santos *et al.*, 2010). The straight portion of the NCL could be determined by fitting the post-yield compression data. The critical state lines of all materials are presented in $q-p'$ plane (Fig. 5(b)) and $e-p'$ plane (Fig. 5(c)-(f)). The end-of-test points were plotted on a unique straight critical state line of slope of $M_{cs}=1.30$ ($\phi_{cs}=32.3^\circ$) in Fig. 5(b). The end-of-test points for Tung oil-sand mixtures tended to plot on a different line with a lower slope $M_{cs}=1.17-1.18$ ($\phi_{cs}=29.3^\circ-29.5^\circ$), irrespective of the Tung oil concentration or heating duration. Note that only selected tests were shown in Fig. 3 and Fig. 4 for clarity, and that Fig. 5 shows all test results, and where a specimen was still showing signs of volumetric variations at the end of the tests, an arrow on the graphs indicates the direction of its path, as shown in Fig. 5(c)-(f).

Different critical state lines can be identified for the sand and the Tung oil-sand mixtures prepared at different Tung oil concentration and heating duration in the volumetric plane (Fig. 5(c)-(f)). Similar to NCL, the CSL of sand or bonded sand is curved at lower stresses and becomes a straight line at higher stresses in $e-p'$ plane (Hachey *et al.*, 1991; Klotz and Coop, 2002; Triantafyllos *et al.*, 2022). Since the straight portion of 1D NCL is parallel to that of CSL, the slope of 1D NCL obtained from Fig. 5(a) could be used to define a straight line after the CSL curvature. The critical state parameters, *i.e.* the intercept Γ and the slope λ determined

from the straight portion of the curve, are summarized in Table 3. The CSLs of the Tung oil-sand mixtures locate to the left and below the CSL of the natural sand, with a tendency to converge at high mean effective stress. This convergence of the stabilized soil with the natural soil behaviour at larger stresses in $e-p'$ plane is often observed, e.g. in sand stabilized with colloidal silica gel, fibre-reinforced sand and sand treated with polydimethylsiloxane (Santos *et al.*, 2010; Liu *et al.*, 2019; Triantafyllos *et al.*, 2022), and attributed to a breakage of the bond between particles or fibres. However, the convergence of the CSLs in both $e-p'$ plane and $q-p'$ plane may require a higher stress level (*i.e.*, with effective confining pressure higher than 800 kPa).

Stress dilatancy of natural sand and hardened oil-sand composites

An effective way to assess the effect of the Tung oil hardening on both the dilatancy and the strength mobilization of the soil is by examining the stress-dilatancy behaviour (Rowe, 1962) of the stabilized sand with that of the natural sand (Fig. 6). The dilatancy D was estimated in terms of total strain ratio instead of plastic strain ratio ($D = d\varepsilon_v^p / d\varepsilon_q^p \approx d\varepsilon_v / d\varepsilon_q$) due to the small elastic region of sand (<0.01% shear strain; e.g., Oztoprak and Botton, 2013), therefore assuming that the elastic strain components are negligible (Jefferies and Been, 2015; Liu and Lourenço, 2021). Though the initial response of Tung oil-sand mixtures appears to be more elastic, at the larger strains, plastic strains dominate for both materials. The critical state stress ratio obtained at zero dilatancy and the maximum rate of dilation D_p are extracted for analysis.

Consistent with the value of M_{cs} determined from the $q-p'$ plot and given in Table 2, a value of 1.30 is found for the natural sand. The dilative response noted in Fig. 4 is observed, with higher peak stress ratio M_p and maximum dilatancy D_p at the lower confining stresses for Tung oil-sand mixtures. The effect of the bonding provided by the hardened Tung oil is the increase of dilatancy at peak stress ratio (plateau seen in Fig. 6(b)-(d)), and a gradual breakage of the

bonds has the effect of reducing the rate of dilation while the soil turns to a more frictional response, eventually reaching critical state. Similar behaviour has been seen on sands stabilized by other means (Cuccovillo and Coop 1999; Lo and Wardani, 2002; Coop, 2005; Wang and Leung, 2008; Porcino *et al.*, 2012). At low confining pressure, the peak stress ratio increases for 5%3d compared with 3%3d and 3%1d, but this enhancement diminishes at high confining pressure, while the effect of the Tung oil heating duration seems less expressive (Fig. 6(b)-(d)). A marked decrease in both peak strength and maximum rate of dilation occurs with increasing stress level, in all Tung oil-sand mixtures. The stress ratio at critical state is lower than that for the sand and almost coincident for all Tung oil-sand mixtures at confining stress larger than 50 kPa, consistent with values in Table 2.

The variations of M_{cs} and D_p of both natural sand and Tung oil-sand mixtures against a wide range of confining pressures are shown in Fig. 7. The M_{cs} and D_p of natural sand are less sensitive to confining pressure, and the differences in D_p disappear and approach 0 when the confining pressure reaches 800 kPa. At confining stress of 50-200 kPa, the contribution of hardened Tung oil to strength increase becomes smoother, and the competitive lubrication effect will dominate the frictional behaviour of the mixtures, thus decreasing the M_{cs} . At higher confining pressure of 200-800 kPa, the M_{cs} of Tung oil-sand mixture increases moderately. This peculiar feature agrees with Liu *et al.* (2019)'s triaxial results on polymer-coated sand under relatively high confining pressure (500 kPa), which was attributed to the damage of polymer-coatings under high stresses. In this study, the damage or abrasion of aged Tung oil coatings occurs after the bond breakage at high stresses. The coating abrasion event will diminish the lubrication by Tung oil and make the M_{cs} of the mixtures rise close to that of natural sand.

The aforementioned mechanism to account for the initial decrease and subsequent recovery of M_{cs} of Tung oil-sand mixtures is depicted in Fig. 8. Specifically, since the weak

bonding effect given by aged Tung oil degrades with increasing confining pressure, the M_{cs} will decrease at first. Within the confining pressure of 200-800 kPa, the aged Tung oil coatings will be scratched, thus weakening the lubrication effect on interparticle friction of sand grains. Consequently, the M_{cs} will restore and even tend towards that of natural sand with the rise of confining pressure. As shown in Fig. 8, the M_{cs} of Tung oil-sand mixtures increases from 1.09 to 1.24 (3%1d), 1.08 to 1.20 (3%3d) and 1.15 to 1.17 (5%3d) under a confining pressure of 200-800 kPa.

Particle surface characterization via SEM-EDS analysis

The elemental compositions of the natural sand and the Tung oil-sand mixtures subjected to different confining stresses were analyzed using a scanning electron microscope equipped with energy dispersive X-ray spectrometry (SEM-EDS). The aim was to examine whether effect observed at the macroscale in terms of stress-strain behaviour, critical state stress ratio and dilatancy can be explained at the microscale. The predominant elements making silica sand are silicon and oxygen which form silicon dioxide SiO_2 , and account for 97.2-99.7% (Taxiarchou *et al.*, 1997; Nor *et al.*, 2012; Alfayez *et al.*, 2019). Tung oil is composed exclusively of the elements C and H (Oyman *et al.*, 2005; Samadzadeh *et al.*, 2011; Zhang *et al.*, 2016; Lin and Lourenço, 2020), but only the former could be detected by EDS. Hence, the amount of Si reflects the contribution of sand particles whereas the amount of C reveals the amount of aged Tung oil on the sand surface. A higher Si/C ratio (mass ratio of Si to C) implies less Tung oil coating on sand surface or higher surface roughness (Liu *et al.*, 2019) attributed to natural sand, whereas a lower Si/C ratio indicates a more dominant role of Tung oil coating, or lubrication effect. The Si/C ratio could therefore be used to quantify the degree of coating abrasion, assumed in Fig. 8 at high confining pressure, which implies a high Si/C ratio. The sand grains were collected after shearing and drying for elemental analysis and mapping to

identify chemical components (Si, O, C, Al) quantitatively (Fig. 9). For each Tung oil concentration, heating duration and confining pressure, approximately 10 grains were randomly selected, and their Si/C ratios quantified. The mean value as well as the standard deviation of the Si/C ratio of these selected grains are used to represent the degree of abrasion on aged Tung oil. The Si/C ratio and the critical state stress ratio M_{cs} are then plotted against the confining pressure in Fig. 10: for all mixtures, the value M_{cs} steadily decreases under confining pressure of 50-200 kPa, which is attributed to bond breakage. For a Tung oil concentration of 3%, the aged Tung oil bonding appears to be weak, as can be observed from the continuing upward trend of the Si/C ratio. This increase becomes more significant at confining pressures of 200-800 kPa, due to the conversion from bond breakage to coating abrasion, as shown in terms of the recovery of the M_{cs} (Fig. 10(a-b)). With the same heating duration of 3 days, the Si/C ratio fluctuates (0.27, 0.24 and 0.25) under confining pressure of 50, 100 and 200 kPa for specimen with a Tung oil concentration of 5%, because the stress is not high enough to damage the aged Tung oil bonds (Fig. 10(c)). Under confining pressures of 200-400 kPa, though the Si/C ratio increases, the M_{cs} still decreases, indicating that the Si/C ratio is not sufficiently high to result in the upswing of the M_{cs} . Specifically, for specimens with Tung oil concentration of 3%, the threshold value of the Si/C ratios are 0.410 ± 0.016 and 0.369 ± 0.014 under confining pressure of 400 kPa for heating duration of 1 day and 3 days, respectively, which causes transition of the M_{cs} . However, the corresponding Si/C ratio is only 0.322 ± 0.012 for specimens with Tung oil concentration of 5%. Under confining pressure of 400-800 kPa, a slight increase of the Si/C ratio is captured, although with relatively large error bars. In the meanwhile, the M_{cs} also increases slightly or remains constant ($M_{cs}=1.13, 1.17$ and 1.17 under confining pressures of 400, 600 and 800 kPa). Note that the Si/C ratio is 0.341 ± 0.022 and 0.363 ± 0.038 under confining pressure of 600 kPa and 800 kPa respectively.

Particle surface characterization via TGA analysis

Thermogravimetric analysis (TGA) can be further used to assess the degree of abrasion, quantified as degree of loss in Tung oil mass. Tung oil completely decomposes when subjected to temperatures as high as 800 °C (He et al., 2019). In contrast, silica sand is thermally stable. The TGA profiles for all specimens are shown in Fig. 11. In Fig. 11(a), while the mass of sand remains the same throughout heating to 800 °C, the mass of Tung oil-sand mixtures (5%3d) decreasing with heating: specimens tested under a confining pressure of 100 kPa, lost 4.9% mass on reaching 800 °C while specimens tested under 800 kPa lost 3.2% mass, implying that there was less aged Tung oil on the sand grains after testing at high pressure. A similar pattern also applies to the Tung oil-sand mixtures (3%3d) as shown in Fig. 11(b). Note that only single sand grains were picked up by a tweezer for SEM-EDS and TGA analysis, thus only the aged Tung oil still coated on the sand grains was considered, regardless of the scraped aged Tung oil during shearing.

Peak and state dependence of natural sand and hardened Tung oil-sand composites

Fig. 12 shows the peak envelopes of both natural sand and Tung oil-sand mixtures. A straight line of slope $M_p = 1.43$ can be fit to the sand data, while for the Tung oil-sand mixtures, a clear effect of the bonding and its degradation with confining stress level are seen. For the mixtures at the same Tung oil concentration of 3% the value of M_p is initially around 2.11-2.17, for low stress levels, after which a gradual decrease of M_p is observed, until a value of about 1.24-1.29 at higher stress levels (Fig. 12(a), (b)). The heating duration does not seem to have a strong effect. On the other hand, if the Tung oil concentration is increased to 5% (Fig. 12(c)), the value of M_p reaches 2.33, decreasing to 1.31 at larger stress. The initial bonding effect of the aged Tung oil is evident from the increased peak stress ratio M_p , but as it degrades the

Tung oil-sand mixtures behave like a coated sand where the coating seems to have a lubricating effect.

The shear strength parameters, stress-dilatancy relationship and strains obtained from tests are significantly affected by the mode of failure (Coop and Atkinson, 1993; Marri *et al.*, 2012; Kutanaei and Choobbasti, 2016; Shakeri *et al.*, 2018). The natural sand specimens tested predominantly failed in barreling under all confining pressures of 50-800 kPa. The Tung oil-sand mixtures, however, saw the development of a significant shear band at lower confining pressure (50-100 kPa), while their failure at higher confining pressures (200-800 kPa) was accompanied by barreling. This apparent change from a more brittle to a more ductile behaviour can be linked back to the transition from sharp to smooth peak deviator stress in the stress-strain curves (Fig. 4(b)). The same failure modes were observed in all Tung oil-sand mixtures (3%1d, 3%3d, 5%3d) under various confining pressures.

The peak stress ratio M_p is plotted against the state parameter (Been and Jefferies, 1985) at the peak dilatancy, ψ_p in Fig. 13. Two distinct relationships can be drawn for the sand and for the Tung oil-sand mixtures (5%3d) in both drained and undrained loading mode, and the D_p - ψ_p relationship in drained loading mode at peak for both natural sand and Tung oil-sand mixtures. ψ_p is defined as the difference between the void ratio at peak e_p and the critical state void ratio e_{cs} for a given effective mean stress p' . In the case of natural sand, M_p decreased linearly with ψ_p , which agrees with previous studies for natural sands (Been and Jefferies, 1985; Huang *et al.*, 2014; Liu and Lourenço; 2021; Triantafyllos *et al.*, 2022). The M_p in drained condition appears to be more dependent on ψ_p than in undrained loading mode (Fig. 13(a)). In other words, dilation contributes more to shear resistance compared with dilative constant-volume remolding at a given ψ_p . The dilatancy ratio D_p at peak in drained condition increases linearly with ψ_p for natural sand. For Tung-sand mixtures, a similar but potentially stronger

state-dependent behavior could be observed, though much more scattered in terms of both M_p - Ψ_p and D_p - Ψ_p relationship.

The SEM images of the specimens were taken after shearing (Fig. 14) to support the existence of Tung oil bonding, bonding breakage, and bonding abrasion at the particle scale. As can be seen in Fig. 14(a), the bonds are absent for the cohesionless sand, and no particle breakage is observed, at least up to the maximum confining pressure of 800 kPa used here. The close-up in Fig. 14(a) confirms the integrity of sand particles after shearing, along with the rough surface characteristics for natural sand. Fig. 14(b) shows micro-features of Tung oil-sand mixtures at low confining pressure (100 kPa), where the aged Tung oil appears to act as bonding between separate sand particles. The close-up reveals a smoother surface topology, which is attributed to the presence of aged Tung oil films. The bond contributes to shear strength enhancement at low confining pressure while the smooth sand surface decreases the friction between the grains. Fig. 14(c) examines the bond abrasion of Tung oil-sand mixtures under high confining pressure (800 kPa). The close-up shows that the aged Tung oil on the sand surface is damaged or abraded, with scattered debris on the surface. The rough nature of sand particles is observed again to some extent, which explains the recovery of stress ratios at high confining pressures.

CONCLUSIONS

Laboratory experiments have been performed to study the impact of aged Tung oil on the mechanical behavior of poorly-graded coarse silica sand. Herein, Tung oil concentration, heating duration and confining pressure are acting as three variables. Important changes in the sand's response were observed as follows:

(1) Aged Tung oil could increase the compressibility of sand in isotropic compression tests. No significant bond breakage was detected from the e - p' curve and K - p' curves, nor in the

specimens observed after shearing. The effect of Tung oil concentration and heating duration on the compression behavior of the sand seems relatively minor.

(2) The comparison of the behavior of Tung oil-sand mixtures and natural sand at the same relative density before shear shows that the competitive influence of bonding and lubrication by aged Tung oil affects the soil behavior alternatively, causing increased strength and stress ratio at both peak and critical state at low confining pressure (<200 kPa), changing to decreased strength or stress ratio under high confining pressure (>200 kPa), when compared with the natural sand. The enhanced dilatancy and brittleness of Tung oil-sand mixtures is noted.

Increasing Tung oil concentration has a positive effect on strength enhancement but could not change the aforementioned trend. The effect of heating duration on strength appears minor.

(3) The CSL of Tung oil-sand mixtures in both q - p' plane and e - p' plane locates below that of the untreated sand, although, at high stresses, the lines appear to converge or crosslink in the volumetric plane. Increasing the heating duration and Tung oil concentration had the effect of slightly lifting up the position of the CSL, which was accompanied by enhanced dilatancy.

(4) The behavior of Tung oil-sand mixtures is more stress-dependent, seen from M_{cs} , M_p and D_p variations against confining pressure. For Tung oil-sand mixtures (3%1d and 3%3d), the enhanced M_{cs} and M_p decreases under low confining pressure (50-200 kPa), while recovering under high confining pressure (200-800 kPa), which can be demonstrated by bond breakage and coating abrasion in light of SEM-EDS and TGA analysis. For Tung oil-sand mixtures (5%3d), the M_{cs} keeps decreasing and then starts to increase under confining pressure of 400 kPa, possibly due to thicker Tung oil coating, which may require higher stress to be damaged.

(5) Unlike the behavior of silica sand, which is state-dependent, for Tung oil-sand mixtures, the state parameter at peak ψ_p was found to lead to a greater variation in the dilatancy ratio and stress ratio.

488 In general, these results suggest optimal conditions for aged Tung oil to stabilize soils.
489 Confining pressures up to 200 kPa provide an increase of peak shear strength and peak
490 dilatancy ratio.

491

492 **DATA AVAILABILITY STATEMENT**

493 Some or all data, models, or code that support the findings of this study are available from the
494 corresponding author upon reasonable request.

495

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500

501 **APPENDIX. REPEATABILITY**

502 See Fig. 15.

503 The potential effects of sample variability, equipment aging, calibration shifts and lab
504 temperature on the stress-strain curve, volume change and excess pore pressure change of
505 both natural sand and Tung oil-sand mixtures are evaluated by repeating several tests under
506 the same sample preparation method and effective confining pressure and similar post-
507 consolidation void ratio.

508

509 **NOTATION**

510 G_s = specific gravity;

511 C_u = uniformity coefficient;

- 512 C_c = curvature coefficient;
- 513 D_{50} = mean diameter;
- 514 e = void ratio;
- 515 $e_{\min}$ = minimum void ratio;
- 516 $e_{\max}$ = maximum void ratio;
- 517 e_0 = initial void ratio;
- 518 e_c = post-consolidation void ratio;
- 519 e_{cs} = near constant volume void ratio or critical state void ratio;
- 520 p' = mean effective stress;
- 521 p'_0 = effective isotropic confining pressure prior to shearing;
- 522 q = deviator stress;
- 523 $q_{\max}$ = deviator stress at peak;
- 524 q_{cs} = deviator stress at critical state or near constant volume state;
- 525 M_p = peak stress ratio;
- 526 M_{cs} = stress ratio at critical state or near constant volume state;
- 527 λ = slope of critical state lines;
- 528 Γ = intercept of critical state lines;
- 529 Δu = excess pore pressure;
- 530 K = bulk modulus;
- 531 D = dilatancy
- 532 D_p = dilatancy at peak;
- 533 ψ_p = state parameter at peak dilatancy;
- 534 ε_a = axial strain;

535 ρ_d = dry density of soil;
 536 ρ_w = density of water;
 537 $d\varepsilon_v^p$ = plastic volumetric strain increment;
 538 $d\varepsilon_q^p$ = plastic deviator strain increment;
 539 $d\varepsilon_v$ = total volumetric strain increment;
 540 $d\varepsilon_q$ = total deviator strain increment.
 541 σ_v' = effective vertical stress
 542 ϕ_{cs} = critical state friction angle

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Table 1. Physical properties of silica sand.

Parameter	Value
Specific gravity, G_s	2.65
Uniformity coefficient, C_u	1.40
Curvature coefficient, C_c	1.03
Mean diameter, D_{50} : mm	0.99
Minimum void ratio, e_{min}	0.68
Maximum void ratio, e_{max}	1.05

Table 2. Test series and key results.

Test ID	e_0	e_c	e_{cs}	p'_0 : kPa	q_{max} : kPa	q_{cs} : kPa	M_p	M_{cs}
SD1	0.812	0.801	0.882	50	192	118	1.68	1.31
SD2	0.817	0.796	0.876	100	338	238	1.58	1.33
SD3	0.833	0.795	0.848	200	608	401	1.51	1.27
SD4	0.836	0.795	0.815	400	1167	824	1.49	1.27
SD5	0.833	0.780	0.767	600	1678	1330	1.45	1.28
SD6	0.838	0.787	0.747	800	2098	1755	1.40	1.27
SU1	0.817	0.805	0.805	50	657	629	1.40	1.32
SU2	0.826	0.801	0.801	100	873	785	1.42	1.30
SU3	0.838	0.799	0.799	200	999	882	1.45	1.30
SU4	0.844	0.782	0.782	400	1374	1298	1.40	1.33
SU5	0.850	0.781	0.781	800	1851	1695	1.43	1.32
T31D1	0.795	0.773	0.843	50	398	129	2.17	1.38
T31D2	0.810	0.773	0.806	100	367	218	1.64	1.25
T31D3	0.837	0.787	0.822	200	528	349	1.41	1.09
T31D4	0.840	0.752	0.789	400	1003	881	1.36	1.26
T31D5	0.857	0.755	0.751	600	1368	1030	1.30	1.25
T31D6	0.867	0.754	0.704	800	1698	1480	1.24	1.24
T31U1	0.795	0.771	0.771	50	556	537	2.29	1.24
T31U2	0.838	0.778	0.778	100	490	455	1.89	1.08
T31U3	0.802	0.759	0.759	200	801	719	1.57	1.13
T31U4	0.830	0.758	0.758	400	847	780	1.23	1.11
T31U5	0.876	0.753	0.753	800	1383	1157	1.28	1.17
T33D1	0.803	0.774	0.821	50	348	126	2.11	1.35
T33D2	0.801	0.765	0.815	100	502	222	2.11	1.27
T33D3	0.819	0.763	0.814	200	627	342	1.52	1.08
T33D4	0.828	0.757	0.784	400	978	825	1.36	1.23
T33D5	0.834	0.750	0.766	600	1402	1163	1.31	1.21
T33D6	0.848	0.751	0.742	800	1805	1567	1.29	1.21
T33U1	0.802	0.777	0.777	50	565	540	1.29	1.16
T33U2	0.803	0.772	0.772	100	751	718	1.30	1.22
T33U3	0.810	0.763	0.763	200	962	909	1.28	1.19
T33U4	0.807	0.759	0.759	400	1147	1028	1.26	1.17
T33U5	0.796	0.752	0.752	800	1715	1580	1.27	1.18
T53D1	0.755	0.736	0.821	50	466	159	2.33	1.58
T53D2	0.792	0.754	0.821	100	579	194	1.96	1.18
T53D3	0.813	0.750	0.800	200	694	383	1.65	1.15
T53D4	0.804	0.746	0.798	400	1073	719	1.42	1.13
T53D5	0.818	0.736	0.774	600	1470	1136	1.35	1.17
T53D6	0.826	0.738	0.756	800	1850	1496	1.31	1.17
T53U1	0.801	0.769	0.769	50	751	658	1.30	1.16
T53U2	0.782	0.771	0.771	100	880	697	1.41	1.09
T53U3	0.799	0.750	0.750	200	1043	860	1.31	1.09
T53U4	0.818	0.749	0.749	400	1227	1064	1.39	1.16
T53U5	0.840	0.735	0.735	800	1690	1521	1.30	1.22

Note: S, natural silica sand; T, Tung oil-sand mixtures; The first number after T, Tung oil concentration; The second number after T, heating duration; D, consolidated drained test; U, consolidated undrained test; The third number after D or U, the serial number; e_0 , initial void ratio; e_c , void ratio prior to shearing; e_{cs} , near constant volume void ratio or critical state void ratio; p'_0 , effective isotropic confining pressure prior to shearing; q_{max} , deviator stress at peak; q_{cs} , deviator stress at critical state or near constant volume state; M_p , peak stress ratio; M_{cs} , stress ratio at critical state or near constant volume state.

Table 3. Critical state parameters of both natural sand and Tung oil-sand mixtures.

Materials	M_{cs}	λ	Γ
Natural sand	1.30	0.2547	2.5391
Tung oil-sand mixtures (3%1d)	1.17	0.2006	2.1479
Tung oil-sand mixtures (3%3d)	1.18	0.2173	2.2633
Tung oil-sand mixtures (5%3d)	1.17	0.2170	2.2680

Note: M_{cs} , stress ratio at critical state; λ , slope of critical state lines; Γ , intercept of critical state lines.

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802 Figure 2. (a) Isotropic compression of the sand and Tung oil-sand mixtures prepared with
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804 mean effective stress.

805 Figure 3. Undrained response: (a) effective stress paths, (b) stress-strain curves and (c)
806 excess pore water pressure (S sand; T Tung oil-sand mixture at 5%3d; U undrained; 1, 3, 4, 5
807 denote stress levels 50, 200, 400, 800 kPa respectively).

808 Figure 4. Drained response: (a) effective stress paths, (b) stress-strain curves and (c)
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810 3, 4, 5, 6 denote stress levels 50, 100, 200, 400, 600, 800 kPa respectively).

811 Figure 5. (a) One-dimensional compression data and normal compression lines in $e-p'$ plane;
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