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# Ultrasonic compaction of granular geological materials

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## Abstract

It has been shown that the compaction of granular materials for applications such as pharmaceutical tableting and plastic moulding can be enhanced by ultrasonic vibration of the compaction die. Ultrasonic vibrations can reduce the compaction pressure and increase particle fusion, leading to higher strength products. In this paper, the potential benefits of ultrasonics in the compaction of geological granular materials in downhole applications are explored, to gain insight into the effects of ultrasonic vibrations on compaction of different materials commonly encountered in sub-sea drilling. Ultrasonic vibrations are applied, using a resonant 20kHz compactor, to the compaction of loose sand and drill waste cuttings derived from oolitic limestone, clean quartz sandstone, and slate-phyllite. For each material, a higher strain for a given compaction pressure was achieved, with higher sample density compared to that in the case of an absence of ultrasonics. The relationships between the operational parameters of ultrasonic vibration amplitude and true strain rate are explored and shown to be dependent on the physical characteristics of the compacting materials.

**Keywords:** ultrasonic compaction; granular materials; sub-sea exploration

## 1. Introduction

The need to control the compaction of granular materials has been regarded as a vital element of improving performance in the pharmaceutical and manufacturing industries. For example, the densification or compaction of particles is an essential process in the production of tablet medications such as paracetamol, where a high breaking force limit is vital to prevent fragmentation or disintegration in product life. It has been demonstrated that this breaking force limit can be increased by increasing the density of the particulate, where the compaction process can be enhanced by generating particle motion in the substrate.

One of the earliest accounts of compaction control was published in 1961 [1], and detailed the need for improved densification of amalgam in the production of dental fillings. The motivation for this research was to promote an increase in compressive strength in the amalgam to reduce the likelihood of damage, such as fracture, before the material setting was complete. This was achieved through the regulation of condensation force, which in this case was the force required to compact the amalgam into the space required for the filling. The strength of the amalgam fillings was shown to be dependent on the condensation forces applied, where a general increase in strength was measured for higher condensation forces.

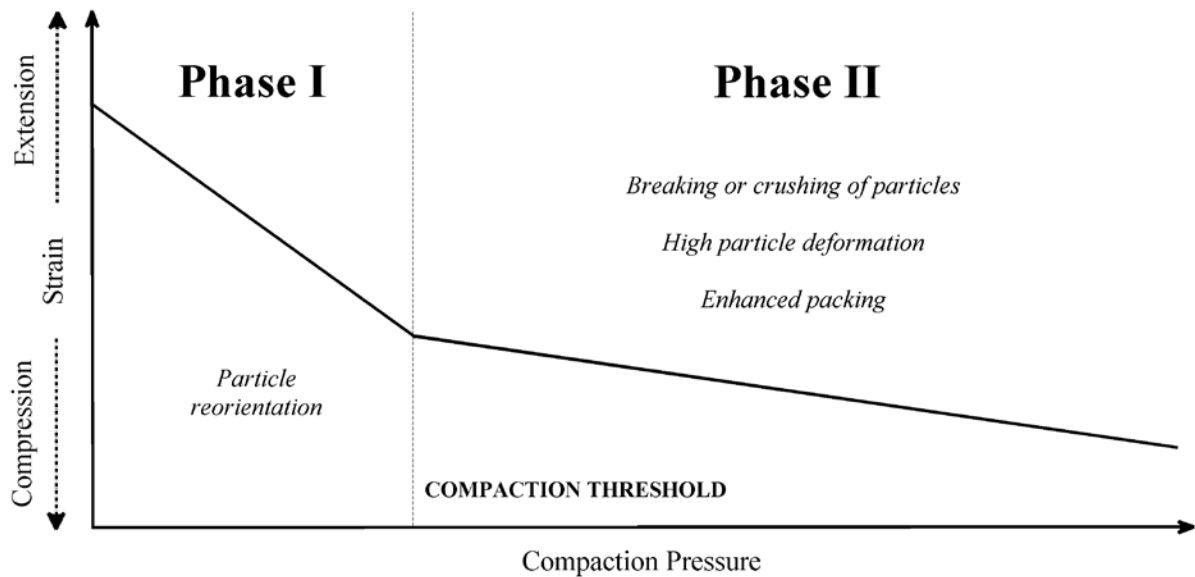
Research conducted in 1969 was an early attempt at incorporating ultrasonics into the amalgam compaction process [2]. However, it was concluded that the strength of the compacted form was independent of the method of compaction, for example pneumatic, ultrasonic or manual, but that the

procedure for amalgam composition must be rigorously controlled. The application of ultrasonic vibrations to the compaction process was then introduced into other disciplines. For example, it was demonstrated in 1981 that ultrasonics could be used to produce high-strength plastic mouldings from polypropylene powder [3]. It was found that ultrasonics was more effective on compaction of smaller particle sizes, where higher strength moulds could be produced, and the pressure applied to the material did not significantly affect the mould strength. An important observation was that the resulting heating of the substrate material promoted increased fusion in the particles, thus improving the mould strength [3]. In 1990 it was shown that ultrasonics only contributes a significant effect to the compaction of substrate material if the compaction pressure is lower than a critical value [4]. By this time, ultrasonics had been utilised to increase Weibull modulus and density, and also to reduce the force required for compaction [4]. However, the effect of compaction pressure on the ability of ultrasonics to confer a notable change to substrate material was not well understood. It was shown that above a critical pressure, ultrasonic vibrations did not affect the green density. The critical pressure was determined to be related to how much the particles within a substrate could move, and was dependent on amplitude of ultrasonic vibration, application time, and the frequency. Further research into the application of ultrasonics in manufacturing processes continued with the production of high-strength ceramics [5,6], where ultrasonic vibrations were shown to reduce porosity, increase ceramic density, and improve grain homogeneity within the fabricated ceramic by eliminating a high proportion of agglomerates and spheroids.

Research continued to investigate the effects of ultrasonic compaction on the properties of substrate materials, but also concentrated on the reduction of measured force on the working tool. For example, it was found that the application of ultrasonics during compaction in the production of paracetamol and ibuprofen tablets significantly reduced the required pressure, thus decreasing the load on the compactor tool [7,8]. More recently, there has been an increased focus on the design of ultrasonic compactor tools, for example by using finite element analysis (FEA), to achieve high compaction performance [9-11]. FEA has enabled the tuned frequency and mode of vibration of the compactor to be controlled, whilst ensuring the mechanical properties of the compactor are sufficient to withstand the compaction loads.

In mechanical compaction, there exist two principal stages [12], summarised generally in Figure 1. The first is reorientation of the particles within the sample, before a critical stress threshold is reached, where very high physical deformation, or a crushing, of the particles takes place. In this secondary compaction phase, the relative motion between particles is very high and particle packing is thereby enhanced [12]. For the compaction processes studied in this paper, the critical stress under different operational conditions of compaction is of interest. However, the influence of the physical characteristics of the granular geological materials is also very important. Although it has been reported that particle mineralogy and shape both affect the performance of an ultrasonic compaction

process [12,13], the influence of geological materials is unclear. There has been conflicting evidence between different studies with respect to the influence of vibrations on densification [14], and it has been proposed that significant differences between the physical properties of the particles, such as grain size and density, are contributory factors. It has also been reported that there is a significantly increased density and particle distribution uniformity within the granular material achieved when ultrasonic excitation is superimposed on the compaction process [9].



**Figure 1: Simplified schematic of mechanical compaction.**

Ultrasonic vibrations have been applied to granular material not just for compaction. For example, ultrasonic penetration into sand has been investigated for planetary drilling [18], where the application of ultrasonic vibrations has been shown to reduce the measured force on the penetrator/drill by generating fluidised behaviour in the granular material. In the vibration of granular material such as sand particles, it is known that there is a stage at which the particles begin to behave more like a fluid than a solid. This can be referred to as the fluidisation transition [14]. Convection motion has been shown to be influential in how the particles move from a solid-like state to fluid. There is also an intermediate region which is produced, and contains material which cannot be classed as either fully compacted or fluidised. A sample of material undergoing compaction could exist as solid, densely-packed material in one region, but fluidised in another. It has been reported that the physical properties of granular materials, such as the particle size and the density, can affect the densification [14], however it remains evident that three zones exist, comprising a lowermost marginally compacted area, a significantly compacted middle layer, and a fluidised top layer resulting from convection. The depth of the fluidised region has been reported to be only weakly linked to the initial sample density [14]. Porosity is a property of a material which is closely related to the density, and reduces as the density increases. It is known that the porosity within a sample of granular material depends on the particle size and shape, the material composition, and also the distribution of the particles [12].

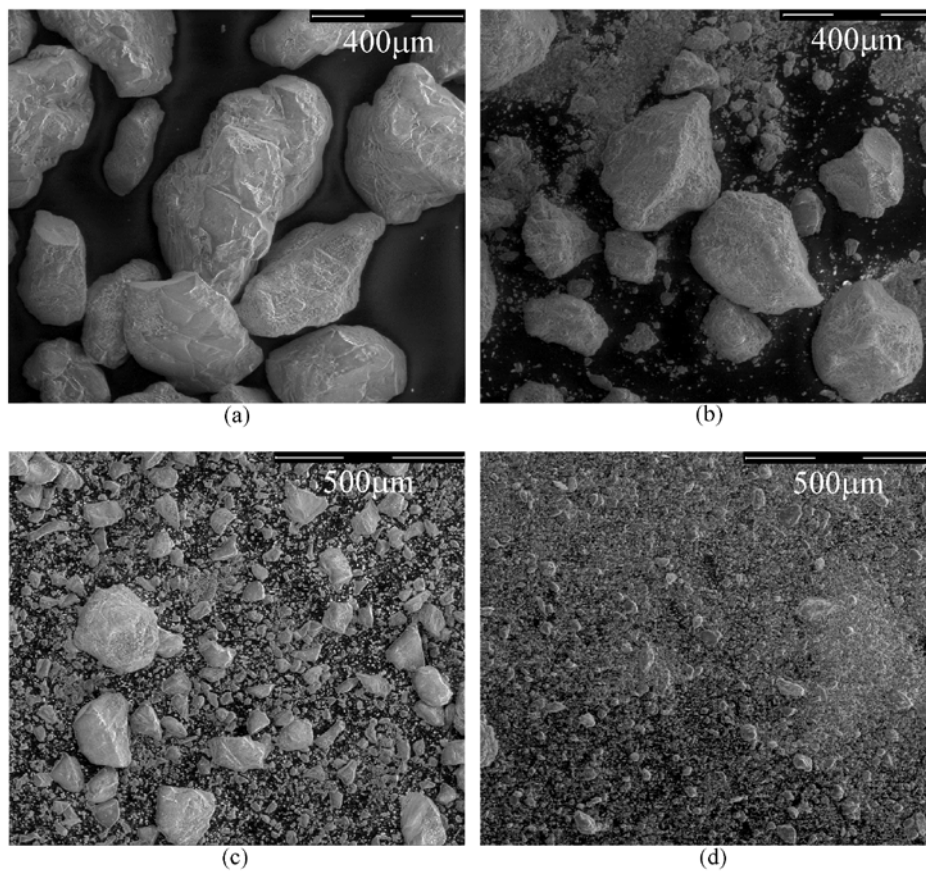
The influence of the direction in which the vibrations are applied to the substrate has also been reported [9,14,19]. In one study, a test setup was manufactured to generate vibrations in the plane of the face of a cylindrical sample (horizontal) and in the axial (vertical) direction [9]. The distinction between the two directions was suggested to be important, because the motion of the particles can change as the granular material is agitated in different ways and settles into voids within the sample. In the case of horizontal vibrations, the rate of compaction increases with respect to ultrasonic vibration acceleration amplitude, however it was reported that these parameters are independent for vertical vibrations [19]. Therefore, horizontal vibrations appear to generate a higher level of compaction than vertical vibrations. In addition, it is known that for horizontal vibrations, the effect of acceleration amplitude decreases as the void ratio, which is a measure of the porosity in the sample, is reduced [19]. Applying ultrasonic vibrations has been found to be successful in reducing the number of voids in the production of white mineral trioxide aggregate (an endodontic cement), and also increases the density [15,16]. It was also suggested that the ultrasonic frequency and the duration of ultrasonic vibration could affect the arrangement of the particles within the endodontic cement sample [16]. It was demonstrated that if ultrasonic vibrations were applied to the material for too long, large voids were produced which could be detected using radiography. Other research into the application of ultrasonic vibrations for dentistry revealed that for root canal treatment, a filling of greater density could be produced by using ultrasonics in the procedure, and again a lower number of voids were produced than using cold lateral compaction [17]. However, it was also stated that there remains a concern of the effect of heating on the substrate material which requires further investigation.

Recent research has applied ultrasonics in the compaction of polytetrafluoroethylene (PTFE) [20,21], which is anticipated to be relevant to the production of bearings and hydraulic components. Considerable improvements in the mechanical properties of the substrate, such as increased tensile strength and elastic modulus, were measured.

There are evident benefits of applying ultrasonic excitation of compaction processes, for both industrial and medical applications. However, further understanding of the influence of ultrasonics on the compaction of geological materials is required to enable the benefits to be realised for sub-sea exploration technology. Towards this aim, this paper investigates the ultrasonic compaction, at 20kHz, of four different granular materials three of which are representative of waste materials derived from the drilling of natural rocks, namely oolitic limestone (OL, Somerset, UK), clean quartz sandstone (CQS, Provodin, Czech Republic), and phyllitic slate (PS, Rogaland, Norway), and the fourth being a commercially available quartz block paving sand (QBPS). The compaction performance is assessed by measuring the relationship between compressive strain in the sample and compaction pressure, and also by the densification of the materials with respect to ultrasonic vibration amplitude.

## 2. Geological material characterisation

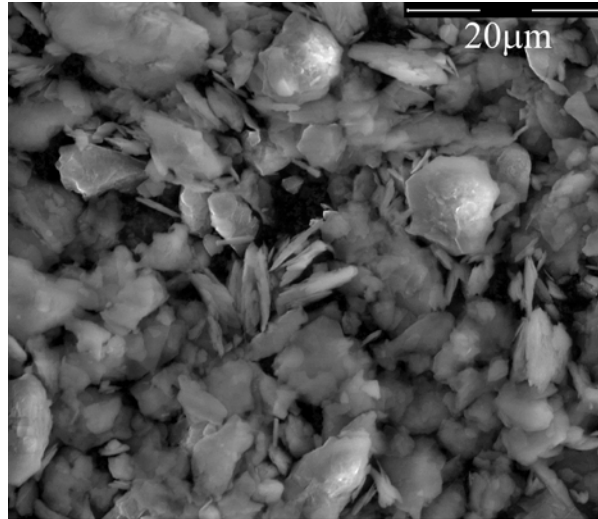
First, understanding the physical characteristics of the granular geological materials is critical in order, subsequently, to assess the influence of various ultrasonic operational parameters on their compaction. Two experimental techniques were adopted to characterise the granular materials. Scanning electron microscopy (SEM), (FEI Quanta 200F) was used to inspect both the particle sizes and compositions in a small sample of each material, and laser diffraction analysis (LDA), (Beckman Coulter LS230) was used to measure the particle size distribution for the different materials, before the compaction experiments were conducted.



**Figure 2: SEM of the granular materials, showing (a) QBPS, (b) OL, (c) CQS, and (d) PS.**

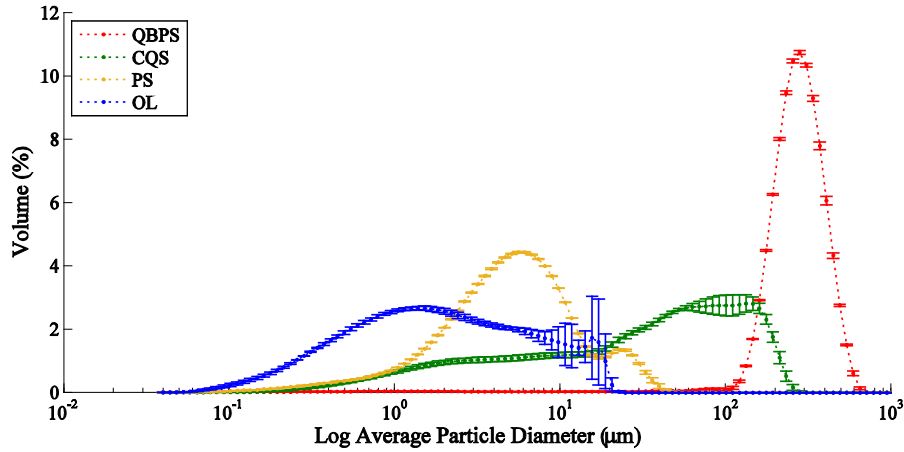
Micrographs from SEM for each material are shown in Figure 2. The QBPS particles are significantly larger than the other granular materials, and there appears to be a higher level of particle size homogeneity for both the QBPS and the PS particles compared to those of the OL and CQS. The QBPS particles, shown in Figure 2(a), are non-spherical and primarily quartz-based, with clear overgrowths. These overgrowths will create voids between the particles which will permit a high level of particle motion under oscillation, for example from ultrasonic vibrations. The OL particles, displayed in Figure 2(b), are principally composed of calcite and quartz, where there is a mixture of rounded quartz and angular-edged quartz. The CQS particles comprise both clays and quartz, as

shown in Figure 2(c), and the PS particles, shown in Figure 2(d), contain a significant proportion of clays. The SEM resolution for the analysis of PS was significantly increased to provide a clearer image of the particles, displayed in Figure 3, clarifying that the PS particles are primarily disc-like in shape.



**Figure 3: Higher resolution SEM of PS.**

The SEM results illustrate the significant differences between the geological materials under consideration with respect to particle size. However, there are similarities in particle shape, with the exception of PS. The physical characteristics are important, as it has been reported that particles within a sample are close to a fragility limit prior to loading [22]. This means that the application of any loading, including ultrasonic vibrations, can instantaneously and significantly change the properties of the sample, and influence the force chains. In the first stage of the compaction process, a reorientation of the particles takes place, and so particle shape is not specified to be as critical [22]. However, the size of the particles and their deformability clearly affect their compaction. Furthermore, particles form directionally-dependent force chains in compression, which constitutes a jamming of the material as the loading is increased. It has been reported that only a marginal change in the direction of applied force can cause instability such that new force chains are formed [22]. As such, the particle dimensions are one set of parameters critical to the stability of the granular materials under compression, and therefore critical to the compaction performance. To characterise the particle size distribution, LDA was conducted, where the experimental preparation required a small amount of each geological material to be mixed with 6ml of calgon dispersant and 25ml of water. The results of this experiment are shown in Figure 4.



**Figure 4: Average particle size distribution of the granular materials from LDA.**

QBPS exhibits low particle size distribution compared to CQS and OL, and PS was also relatively low. This means that there are likely to be a significantly greater number of voids in the QBPS sample before compaction compared to the other materials, but also a higher level of homogeneity. The low particle size distribution of the QBPS shown in Figure 4 correlates well with the inspection of the particles using SEM, shown in Figure 2(a). The results of the LDA and SEM, shown in Figure 2(d), for the PS are also broadly correlated, particularly in relation to the average particle diameter. The results for the CQS with respect to average particle size were also as expected from LDA compared to SEM, shown in Figure 2(c), where the wider range of particle sizes has been detected using both techniques. It is evident that combining LDA and SEM is a powerful aid to the analysis of the particle sizes, distributions and compositions of the granular samples.

The effectiveness of ultrasonics in the compaction process is dependent on the mean absolute particle size, the particle size distribution, the particle size relative to the void size. For example, smaller particles are able to move in small voids, and there can also be a high number of voids in a sample of granular material containing relatively narrow particle size distributions. The application of ultrasonic vibrations to a uniform, homogeneously distributed sample can cause extensive fluidised activity in the uppermost sample layers, where the void space permits a high level of particle motion. Conversely, if ultrasonic vibrations are applied to a sample containing a wider distribution of particle sizes, then the reduced void space restricts the extent to which the particles can move.

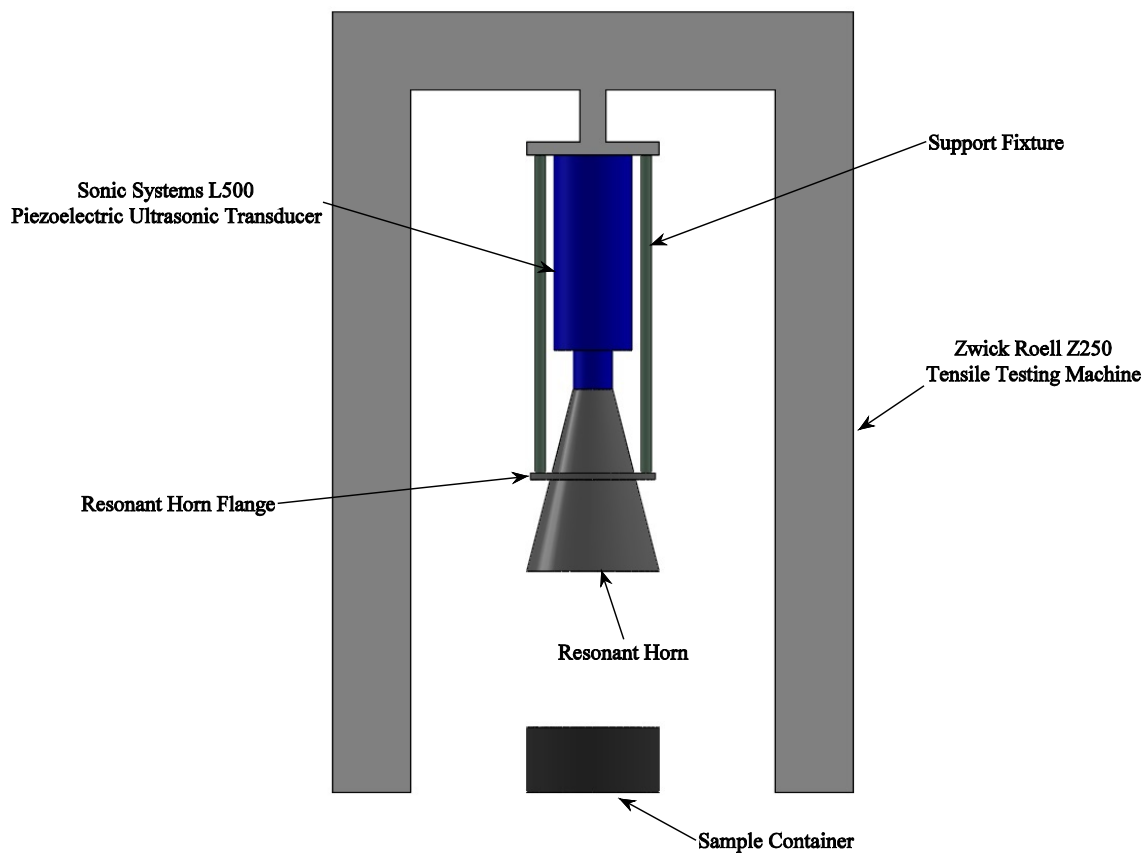
### **3. Compaction of granular geological materials**

The preparation of the geological samples was rigorously controlled to ensure consistency and repeatability between experiments, and also to eliminate voids or collections of grains as much as possible, which could influence the accuracy of the results. In each case, the granular material was deposited from a funnel at a height of 0.48m above the target container, which was 50mm in depth.



Before the compaction experiments were performed, the OL, CQS, and PS were observed to contain sporadic agglomerations during particle deposition, where small proportions of the particles clustered together. Any collection of material within a sample reduces the homogeneity, and this agglomeration was mitigated where possible by repeating the deposition process.

After sample preparation, the compaction of each material at different amplitudes of ultrasonic vibration was performed. The compressive true strain rate of compaction was controlled using a compactor system consisting of a 20kHz piezoelectric transducer (Sonic Systems L500) and a resonant steel horn connected to a tensile testing machine (Zwick Roell Z250). The setup for the compaction experiments is shown in Figure 5.



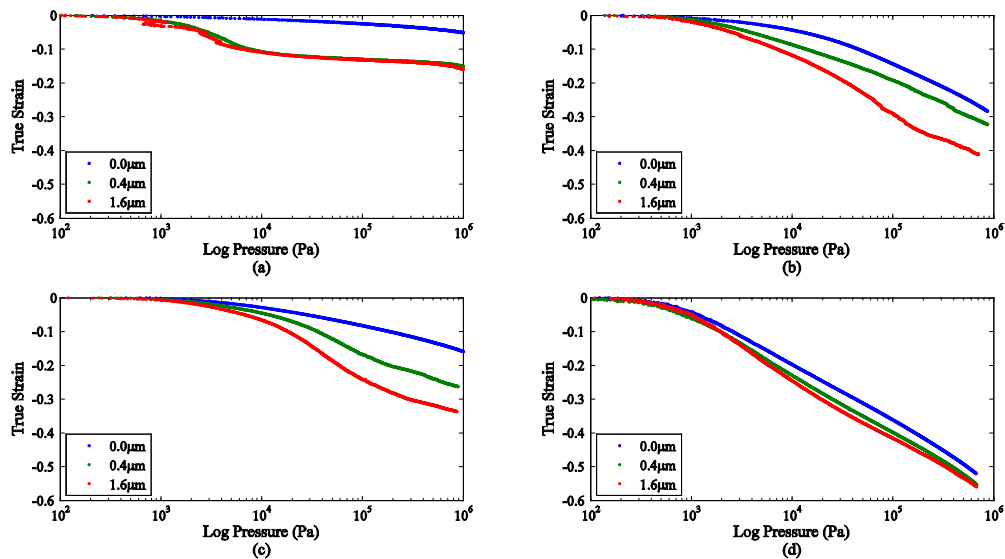
**Figure 5: Schematic of the compaction experimental setup.**

The resonant steel horn was tuned to 20kHz in the longitudinal mode of vibration. A flange was incorporated on the horn, positioned at the nodal region. A support fixture was used to interface the transducer and resonant horn with the tensile testing machine, by connection to the flange. This configuration protected the transducer from, potentially damaging, high loads. During each compaction cycle, the transducer was activated, generating the selected ultrasonic vibration amplitude at the output face of the resonant horn. The compactor system was then used to compress geological

material in a sample container, where the pressure on the compactor system was measured for two different amplitudes of ultrasonic vibration, set and monitored using the transducer power generator, as a function of compressive true strain.

### 3.1 Relationship between compressive true strain and compaction pressure

In each experiment, the ultrasonic compactor system was positioned above the sample container, where compaction of material was induced up to a maximum pressure of 1.23MPa. Although this has been reported to be a relatively low compaction pressure [3], this pressure limit was set to avoid mechanical failure of the compactor. The pressure was measured for three true strain rates, comprising  $0.03\pm 0.01\text{s}^{-1}$ ,  $0.05\pm 0.01\text{s}^{-1}$  and  $0.22\pm 0.06\text{s}^{-1}$ , and for two amplitudes of ultrasonic vibration, comprising  $0.4\mu\text{m}$  and  $1.6\mu\text{m}$ . The compaction pressure as a function of strain in the absence of ultrasonics was also measured. The results are shown in Figures 6-8.



**Figure 6: True strain and compaction pressure relationship at  $0.03\pm 0.01\text{s}^{-1}$  for (a) QBPS, (b) OL, (c) CQS, and (d) PS.**

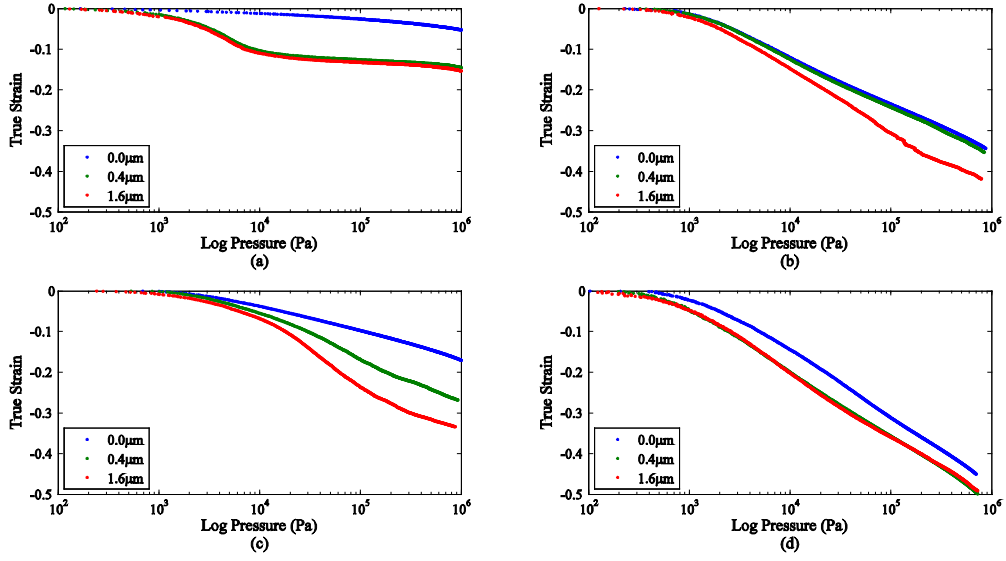


Figure 7: True strain and compaction pressure relationship at  $0.05\pm 0.01s^{-1}$  for (a) QBPS, (b) OL, (c) CQS, and (d) PS.

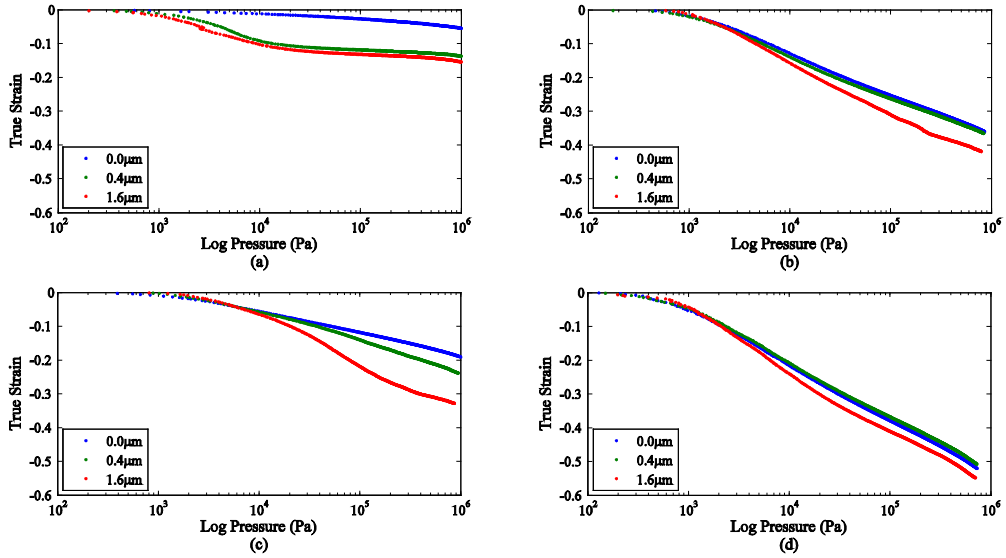


Figure 8: True strain and compaction pressure relationship at  $0.22\pm 0.06s^{-1}$  for (a) QBPS, (b) OL, (c) CQS, and (d) PS.

In each case, the pressure increases with compressive true strain, and this trend is consistent for the different true strain rates. The data trends can be compared with the schematic shown in Figure 1. It is clear that the transition between the first and second phase of compaction has not occurred for all material types or amplitudes of vibration. In the first phase of compaction, where the grains are reoriented and compacted together, there is no particle jamming. However, this phenomenon appears in the second phase, where grains can experience physical deformation. The two-phase compaction behaviour is clearly evident for the QBPS samples with ultrasonics applied, and the stage where

jamming begins is distinguishable as a distinct change in gradient of the data, as shown in Figures 6-8(a). However, jamming is not detected for the other granular materials. The most likely explanation for this is that the pressure is insufficient to produce the secondary compaction phase. For the QBPS, the ultrasonic vibrations generate a fluidised zone of particle motion near the uppermost surface of the sample. The agitation of the particles creates a reduction in resistance to the advance of the compactor in the material, and hence a higher level of compressive strain can be achieved, where particle reorientation is completed at a lower compaction pressure. Consequently, the compaction threshold is reached at a lower pressure compared to the case comprising an absence of ultrasonic vibrations.

The QBPS results at each true strain rate are consistent, where the compressive true strain up to a pressure of 1MPa is significantly greater for those with ultrasonic vibrations applied rather than without. There is a notable change in compressive true strain when there is ultrasonic excitation, even at these low levels of ultrasonic amplitude. However, there is little difference between the compressive true strain at an ultrasonic amplitude of 0.4 $\mu\text{m}$  and that of 1.6 $\mu\text{m}$ , over the measured pressure range.

The results of the compaction experiments performed on OL and CQS, shown in Figures 6-8(b,c), indicate that compaction is more sensitive to amplitude of vibration than for the QBPS, since there is a prominent difference between the results for the two ultrasonic amplitudes. For the OL, the introduction of ultrasonics at an amplitude of 0.4 $\mu\text{m}$  to the compaction process produced a discernible increase in the compressive true strain for the true strain rate of  $0.03\pm 0.01\text{s}^{-1}$  compared to compaction with no ultrasonics. This increase in compressive strain was not as prominent between the experiments conducted using no ultrasonics and an amplitude of 0.4 $\mu\text{m}$  at the higher true strain rates. However, a considerable increase in compaction was measured up to 1.23MPa when the amplitude of vibration was increased to 1.6 $\mu\text{m}$  for a true strain rate of  $0.05\pm 0.01\text{s}^{-1}$ . The results with the most substantial difference in compaction for each amplitude of vibration were for the CQS, the particle size distribution of which was measured to be generally broader than that of both QBPS and PS. This suggests that the particle size distribution alone does not account for the fluidised activity caused by the ultrasonic vibrations, and the subsequent reduction of measured pressure on the compactor system for the same true strain. In an attempt to quantify the compaction performance of each material, compaction moduli were extracted from the data shown in Figures 6-8. For each experiment, the gradient of the first compaction phase was calculated. A higher gradient, or compaction modulus, indicates a more expeditious transition between the compaction phases, as displayed in Figure 1, and is therefore indicative of better compaction performance. This data is summarised in Table I.

Of the four materials, the PS samples did not exhibit any significant increase in compaction resulting from the introduction of ultrasonic vibrations to the compaction process. It may be the case that the disc-type shape of the PS particles limits the occurrence of fluidised behaviour near the uppermost

surface of the sample, since a disc-type particle shape permits fewer voids between particles. It is also likely that the measured pressure is not sufficient to induce the secondary phase of compaction, and that differences in the strain-pressure relationship between different amplitudes of vibration would be clearer for a higher pressure limit. During the sample preparation process, it was noticed during deposition that the PS particles tended to exhibit significantly higher inclination to agglomerate than the other materials. A resultant lower fluidisation level for this material would suggest a higher compaction pressure is required to complete the transition to the second compaction phase. This is a further indicator that the material type greatly affects the compaction performance.

**Table I: Compaction moduli derived from the strain-pressure relationships.**

Material	Ultrasonic amplitude ( $\mu\text{m}$ )	Compaction modulus magnitude ( $\text{Pa}^{-1}$ )		
		Strain rate $0.03\pm 0.01\text{s}^{-1}$	Strain rate $0.05\pm 0.01\text{s}^{-1}$	Strain rate $0.22\pm 0.06\text{s}^{-1}$
QBPS	0.0	0.07	0.07	0.07
	0.4	0.42	0.51	0.38
	1.6	0.42	0.51	0.32
OL	0.0	0.45	0.47	0.40
	0.4	0.42	0.47	0.40
	1.6	0.55	0.62	0.51
CQS	0.0	0.21	0.25	0.21
	0.4	0.49	0.51	0.29
	1.6	0.62	0.73	0.60
PS	0.0	0.55	0.62	0.53
	0.4	0.55	0.62	0.53
	1.6	0.55	0.62	0.53

In the compaction process, the level of particle agitation from the ultrasonic vibrations is important to consider, especially in relation to particle size. This is because the energy to fluidise or induce high velocities of smaller particles will be different to that required for larger particles. Consequently, both the particle size, as well as sample homogeneity, are critical to the understanding of how ultrasonics affects different granular geological materials. If there are fewer voids within a sample, then there is less available space for particles to become fluidised in the uppermost regions of the sample, especially as compaction pressure is increased. The LDA results of particle size distribution in Figure 4 show that the particle sizes of each material type are not the same, and so the particle vibrations induced by the application of ultrasonics will also be different for each material sample. As well as the physical characteristics of the particles, the compaction pressure must be sufficiently high so that both stages of compaction can be detected. This compaction pressure level has been shown to be dependent on the ultrasonic amplitude, and is important in order to acquire a comprehensive understanding of the compaction process for each geological material.

### 3.2 Densification of the geological materials

The percentage increase in granular material sample density, from zero to approximately 1MPa of compaction pressure, is referred to as the densification for the purposes of this study. The densification results for samples of QBPS, OL, CQS and PS for compaction using different ultrasonic amplitudes, and at three strain rates, are shown in Figure 9.

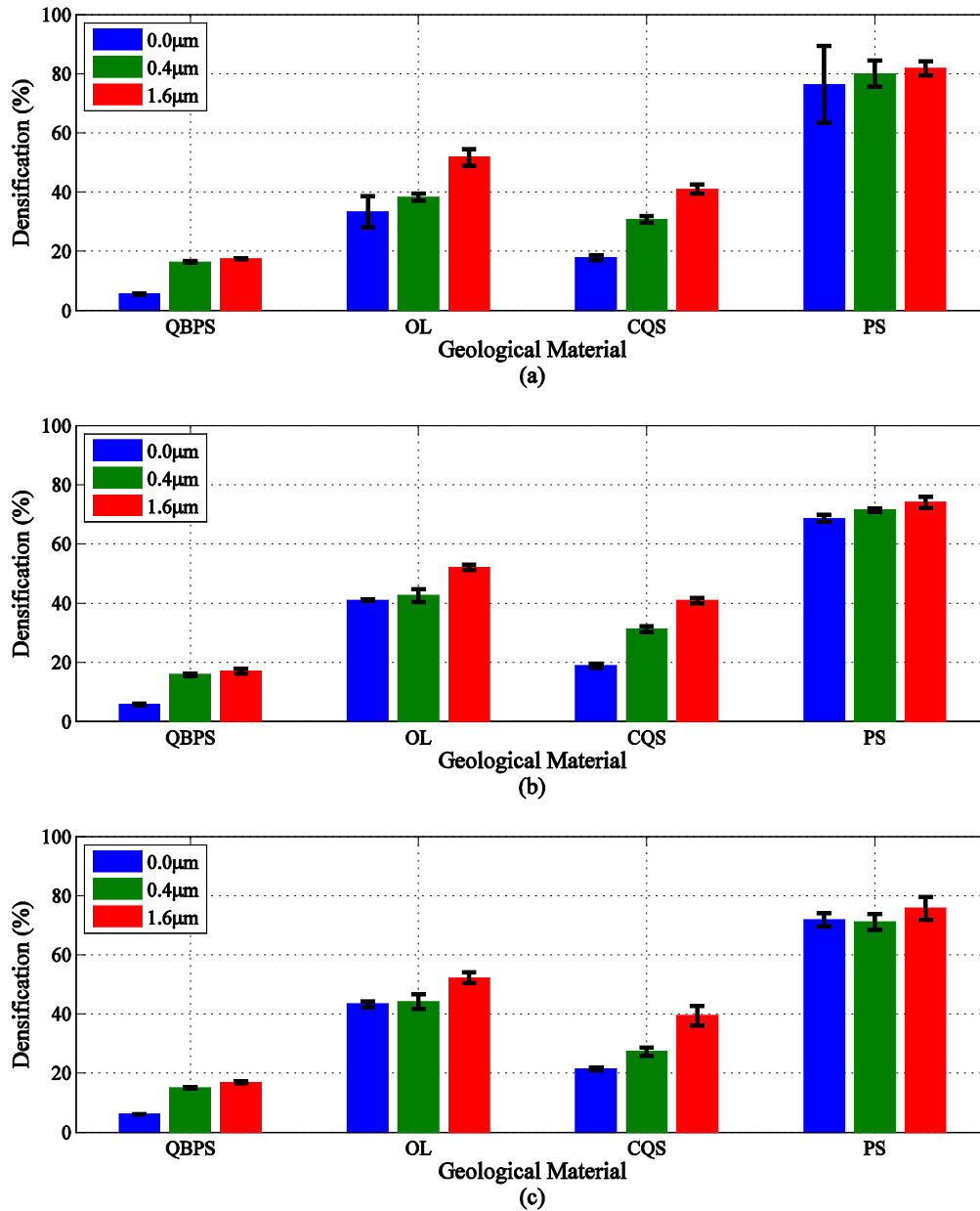


Figure 9: Average densification at true strain rates of (a)  $0.03 \pm 0.01 \text{ s}^{-1}$ , (b)  $0.05 \pm 0.01 \text{ s}^{-1}$ , and (c)  $0.22 \pm 0.06 \text{ s}^{-1}$ .

The application of even a very low level of ultrasonic amplitude to QBPS causes a significantly higher level of densification, consistent for the three strain rates. For the OL, the densification for an ultrasonic amplitude of 0.4 $\mu$ m exhibits little difference from no ultrasonic excitation, however there is a notable increase in densification for an ultrasonic amplitude of 1.6 $\mu$ m. The results for CQS show about 10% increase in densification between no ultrasonics and 0.4 $\mu$ m, and a further 10% between 0.4 $\mu$ m and 1.6 $\mu$ m. For PS the change in densification is much smaller than for the other materials for the selected range of amplitudes. The trends for differences in true strain for each amplitude and each material, in Figures 6-8, are consistent with the corresponding trends for change in densification.

It is clear that over the designated compaction pressure range, the two ultrasonic excitation amplitudes, although both relatively small, have improved the compaction of the four geological materials. The extent of the improvement, however, depends on the physical characteristics of the granular materials. For example, QBPS exhibited a much narrower distribution of particle size and much larger particle sizes than the other materials. Despite this, compaction of PS was the least affected by ultrasonic excitation of the compactor, suggesting that LDA measurements alone cannot provide an indication of the effectiveness of ultrasonics in compaction performance. By employing SEM in the analysis of PS, as shown in Figures 2(d) and 3, it was determined that the general physical shape of the particles was principally disc-type, different to QBPS, OL and CQS. The PS also exhibited a high degree of agglomeration during the deposition process, unlike the other materials. In general, it is evident that a range of experimental techniques are required to assess the influence of ultrasonics on different geological materials, and this investigation has demonstrated that by incorporating material characterisation techniques such as SEM and LDA, a greater understanding of the ultrasonic compaction of geological materials can be achieved.

#### **4. Conclusions**

This investigation has shown that ultrasonic vibrations can be used to enhance the compaction of granular geological materials, for example by increasing compressive strain in a sample for a given compaction pressure. However, the process is constrained by the physical properties of the compacting materials, and also the operational parameters of the compactor system, such as strain rate. It is shown that the strain-pressure relationship and the level of densification achievable are closely related for a particular geological material, for the ultrasonic amplitudes in this study. It is also demonstrated that the compaction modulus is a useful indicator of ultrasonic compaction performance.

Ultrasonic amplitude and true strain rate influenced the four geological materials differently. Also, the four materials exhibited different levels of compaction improvement under ultrasonic compaction, but

these could not be directly correlated with the results of a single characterisation technique. A combination of experimental methods, in this case comprising LDA and SEM, provides a more comprehensive understanding of the effect of ultrasonics on granular geological material compaction.

The results of this study are being used to aid the design of an ultrasonic compactor for sub-sea exploration, and to evaluate the potential for effective ultrasonic compaction of the geological materials typically encountered.

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