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# Ultrasonic wave propagation in powders

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**Abstract.** Powder clumps (cakes) has a significant effect on the flowability and stability of powders. Powder caking is mainly caused by moisture migration due to wetting and environmental (temperature and humidity) changes. The process of moisture migration caking involves creating liquid bridges between the particles during condensation which subsequently harden to form solid bridges. Therefore, an effective and reliable technique is required to quantitatively and non-invasively monitor caking kinetics and effective stiffness. This paper describes two ultrasonic instruments (ultrasonic velocity pulse and airborne ultrasound systems) that have been used to monitor the caking phenomenon. Also, it discusses the relationship between the ultrasonic velocity and attenuation measurements and tracking caking kinetics and the effective stiffness of powders.

## 1. Introduction

Powders are the final or intermediate products of many food, chemical and pharmaceutical industries. The processing of powders can pose many challenges. Powder strength can increase significantly in the process equipment and during storage in the pack. This phenomenon is known as caking whereby free flowing powder is transformed into clumps or aggregates [1, 2]. It is mainly caused by moisture migration due to wetting and environmental (temperature and humidity) changes. The moisture migration caking process can be summarized in five steps [2]: (1) wetting and moisture absorption, (2) liquid bridging, (3) drying and moisture desorption, (4) hardening and solid bridging, and (5) compacting and caking. Powder caking can have detrimental effects on flowability, stability, solubility, mixing and dispersions [1]. As a result, it causes loss of product, delays in production, consumer complaints, storage and handling problems. Therefore, in situ monitoring of powder caking is required in many industries to improve their productivity and marketability. The ideal technique should be non-invasive, rapid, robust and effectively related to the caking stiffness and kinetics.

Ultrasonic measurements are easy to make and can be related to the elastic moduli of the bulk solids [3]. Several researchers used ultrasonic techniques to sense different properties of the powder in the past. For instance, Makse et al [4] studied the applicability of elasticity theory to granular materials. They performed acoustic experiments, numerical simulations and theoretical analysis to understand the elastic behavior of a deforming granular assembly of soft glass spheres (beads) under compressional load. Their results show good agreement between simulation and experimental results for the behavior of elastic moduli of the system. However, there was a significant disagreement in a system with shear perturbations. In this case both shear moduli and collective relaxation dynamics that are related to structural disorder and non-affine motion of grains should be included to obtain correct treatments. Coghill and Giang [5] investigated the possibility of using ultrasonic velocity to measure the strength of powder particles formed by agglomeration for two industrial materials: iron ore pellets, and calcined



alumina. They demonstrated that ultrasonic velocity can be related to the packing geometry of coarser powder particles. Nevertheless, the velocity measurements are size independent and not repeatable for particle diameters less than 200  $\mu\text{m}$ . Castellanos et al [6] studied the packing behavior of cohesive powder. They showed that the transmission of ultrasonic signal is determined by the elastic deformation at high consolidation stresses. Akseli and Cetinkaya [7] demonstrated air coupled (non-contact) ultrasonic technique for tablet characterisation. The coating thickness and mechanical properties of drug tablets were determined by this non-contact ultrasonic technique. Saggin and Coupland [8] used an ultrasonic device to monitor the dissolution kinetics of sucrose and lactose powder in water. They found that the reflected signal is sensitive to the amount of dissolved powder in water.

The aim of this paper is to investigate the feasibility of using ultrasonic techniques to monitor powder caking. The theory of ultrasonic wave propagation in powder will be discussed. Two ultrasonic instruments will be described. Experimental results for monitoring caking stiffness and kinetics will be presented that illustrates the relationship between sound velocity and powder caking.

## 2. Theory of ultrasonic wave propagation in powder

The acoustic properties of granular materials under confining pressure can be extremely nonlinear compared to continuum elastic solids. The simplest theories that describe sound propagation in powder confined by an external stress are the effective medium theory [4, 5]. In principle, these theories apply to uniformly sized spheres where the wavelength is long enough to ignore the effect of scattering from the powder particles. The ratio of sound wavelength to the particle diameter in the wavelength limit is usually greater than 10. The effective medium theories are based on the Hertz-Mindlin contact theory which assumes that the elastic moduli of the medium can be derived from the average particle to particle contact. These theories can be used to calculate the bulk and shear moduli as uniform pressured is applied to the powder particles. The longitudinal (P-wave) sound velocity and the elastic moduli of the isotropic materials are related by [4, 5]

$$V = \left( \frac{K+4/3\mu}{\rho_{dry}} \right)^{1/2} \quad (1)$$

where  $V$  is the longitudinal sound velocity (m/s),  $K$  is the bulk modulus ( $\text{N/m}^2$ ),  $\mu$  is the shear modulus ( $\text{N/m}^2$ ) and  $\rho_{dry}$  is the density of dry particles ( $\text{Kg/m}^3$ ). Makse et al. [4] derived a formula for the sound velocity in an assembly of glass spheres under stress by substituting the values of elastic moduli into equation 1 as follows

$$V = Z^{1/3} \phi^{-1/6} \left( \frac{6\pi P}{K_n} \right)^{1/6} \sqrt{\frac{3k_n}{20\pi\rho_{dry}}} \quad (2)$$

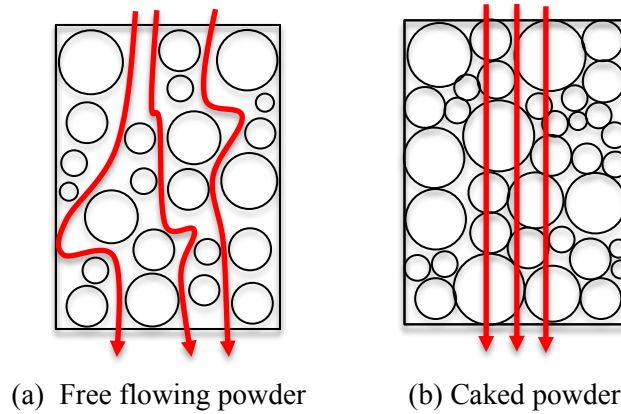
where  $Z$  is the average number of contacts per particle,  $\phi$  is the volume fraction of the sample, and  $k_n = 4\mu/(1-\nu)$  is the effective stiffness ( $\text{N/m}^2$ ) which is defined in terms of shear modulus ( $\mu$ ) of the solid particles and the Poisson ratio ( $\nu$ ) of the solid particle material. Equation 2 shows that the effective stiffness of the caked powder can be calculated from measurements of sound velocity by assuming  $Z$  and  $\phi$  do not significantly vary and determining  $\rho_{dry}$  from the received signal amplitude. The sound velocity in caked powder is higher compared to free flowing powder. This is because of the tortuous path of the propagated ultrasonic signal in the free flowing powder as shown in figure 1.

### 2.1. Relationship between dry and wet density

The reflection and transmission coefficients at the interface between air and powder surface can be calculated as follow

$$R = \frac{Z_{powder} - Z_{air}}{Z_{powder} + Z_{air}} \quad (3)$$

$$T = \frac{2Z_{\text{powder}}}{Z_{\text{powder}} + Z_{\text{air}}} \quad (4)$$



**Figure 1.** Ultrasonic wave propagation in free flowing (a) and caked (b) powder. In comparison with caked powder (b), the ultrasonic signal takes longer to propagate in the free flowing powder (a) due to air gaps between particles.

where  $R$  and  $T$  are the reflection and transmission coefficients, respectively. The acoustic impedance of powder  $Z_{\text{powder}}$  and air  $Z_{\text{air}}$  are given by

$$Z_{\text{powder}} = \rho_{\text{powder}} V_{\text{powder}} \quad (5)$$

The approximate values of the density and sound velocity in air are  $1.225 \text{ Kg/m}^3$  and  $340 \text{ m/s}$ , respectively. Thus, the acoustic impedance of air is

$$Z_{\text{air}} = \rho_{\text{air}} V_{\text{air}} \approx 417 \text{ Kg/m}^2\text{s} \quad (6)$$

The sound velocity is approximately inversely proportional to the powder density [5] as follows

$$V_{\text{powder}} \approx \frac{1}{\sqrt{\rho_{\text{powder}}}} \quad (7)$$

Thus the powder acoustic impedance in equation 5 is equal to  $\sqrt{\rho_{\text{powder}}}$ . Substituting equation 5 into equations 3 and 4 yields

$$R = \frac{\sqrt{\rho_{\text{powder}}} - 417}{\sqrt{\rho_{\text{powder}}} + 417} \quad (8)$$

$$T = \frac{2(\sqrt{\rho_{\text{powder}}})}{\sqrt{\rho_{\text{powder}}} + 417} \quad (9)$$

Equations 8 and 9 can be used to calculate the reflection and transmission coefficient of dry and wet powder as follows

$$R_{\text{dry}} = \frac{\sqrt{\rho_{\text{dry}}} - 417}{\sqrt{\rho_{\text{dry}}} + 417} \quad (10)$$

$$T_{\text{dry}} = \frac{2(\sqrt{\rho_{\text{dry}}})}{\sqrt{\rho_{\text{dry}}} + 417} \quad (11)$$

$$R_{wet} = \frac{\sqrt{\rho_{wet}-417}}{\sqrt{\rho_{wet}+417}} \quad (12)$$

$$T_{wet} = \frac{2(\sqrt{\rho_{wet}})}{\sqrt{\rho_{wet}+417}} \quad (13)$$

The powder particles agglomerate as the powder is wetted. A larger portion of ultrasonic signal is reflected from the wet powder due to formation of liquid bridges which increase the reflection coefficient relative to air. Thus, comparing the peak to peak amplitude ( $V_{p-p}$ ) and the reflection and transmission coefficients of the wet and dry powder yields

$$\frac{V_{p-p \text{ dry}}}{V_{P-P \text{ wet}}} = \frac{R_{dry}}{R_{wet}} \quad (14)$$

$$\frac{V_{p-p \text{ dry}}}{V_{P-P \text{ wet}}} = \frac{T_{dry}}{T_{wet}} \quad (15)$$

The relationship between wet and dry powder density can be obtained by comparing equations 14 and 15 with equations 10 to 13 and using Taylor series approximation as follows,

$$\rho_{wet} = \left( \frac{V_{p-p \text{ dry}}}{V_{p-p \text{ wet}}} \right)^2 \rho_{dry} \quad (16)$$

### 3. Ultrasonic measurement instruments

Two different instruments were used to perform the caking effective stiffness and kinetics measurements. The instruments are ultrasonic velocity pulse and airborne ultrasound systems. Both systems are operated in through-transmission (pitch-catch) mode. This approach is based on transmitting ultrasonic compression wave pulses between two piezoelectric transducers. However, each system has different setup and transducers as illustrated in sections 3.1 and 3.2.

#### 3.1. Ultrasonic velocity pulse system

This system was used to perform the caking effective stiffness measurements. It consists of off-the-shelf instrument (Pundit Lab, Proceq Group, Switzerland) connected to a pair of transducers. The transducer pairs are placed in contact with the powder sample as shown in figure 2. Two different pairs of transducers were used in these measurements. Their centre frequencies are 24 and 50 KHz. five different thickness of powder samples were used to separate the transducer as shown table 1. A Lecroy oscilloscope (WaveRunner 44Xi, Teledyne LeCroy SA, Geneva - Switzerland) was connected to the instrument to monitor the received signals from the receiver transducer.

The system was calibrated by aligning the transducers and measuring the speed of sound in air. The powder sample was placed between the transducer pairs and wetted with water. Then it was left for some times to dry. The speed of sound and attenuation were measured during the drying time. This procedure was repeated for each sample in table 1.

#### 3.2. Airborne ultrasound system

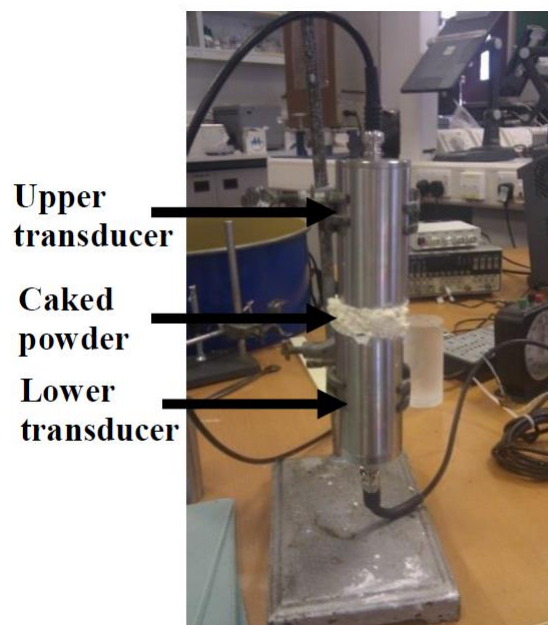
The interaction between the coating liquid and powder was monitored by using a bespoke airborne ultrasound system. This system is based on sending and receiving ultrasonic signal through air by a pair of non-contact transducer as shown in figure 3. The transducer centre frequency was 140 KHz and the path length between the transducers was 76 mm. This value was selected to be identical to the transducer focal distance. A high power amplifier was used to drive the transmitter transducer to transmit the signal through air and an automatic gain compensation amplifier was used to amplify the received signal from the receiver transducer. The transmitted and received signals were monitored via a handy scope HS3

USB oscilloscope (TiePie Engineering, Sneek, Netherlands) connected to the transducers. The sample was scanned automatically by connecting the transducers to a movable stage (Prior Scientific Instruments Ltd, Cambridge, UK). A high precision motor was embedded in this stage and controlled by ProScan III controller. LABVIEW software (National Instruments Corporation (U.K.) Ltd., Newbury, UK) was used for automation control.

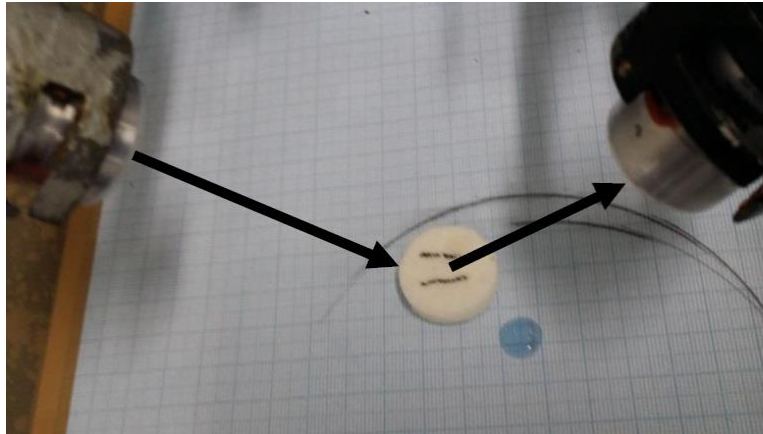
The powder sample shown in figure 3 was non-ionic surfactant linear alkylbenzene sulfonates (LAS) flake powder which is one of major chemicals used in manufacturing detergent powder. The powder was tableted to obtain a single surface particle to ignore the effects of changing particle shapes and sizes on studying particle-liquid interaction. The calibration was made by sending and receiving the signal from the stage without the tablet. This signal is used as a reference to compare with the waveform obtained from placing the tablet between the transducers as shown in figure 4. It can be seen that the signal amplitude of the reflected signal from the tablet surface has been decreased significantly. In addition, the time of flight of the reflected signal is less than the reference time. This is because part of ultrasonic signal is reflected from the top surface to form the first echo. The other portion of this signal penetrates into tablet and reflects from its bottom surface to form the second echo.

The reduction in the signal amplitude triggers the gain compensation amplifier to increase gain to amplify the received signal. This indicates that the peak to peak signal amplitude is inversely proportional to the instrument gain. Thus, using dry and wet powder gain ( $G_{dry}$ ,  $G_{wet}$ ) in equation 16 instead of the corresponding amplitude ( $V_{p-p\ dry}$ ,  $V_{p-p\ wet}$ ) yields

$$\rho_{wet} = \left( \frac{G_{wet}}{G_{dry}} \right)^2 \rho_{dry} \quad (17)$$



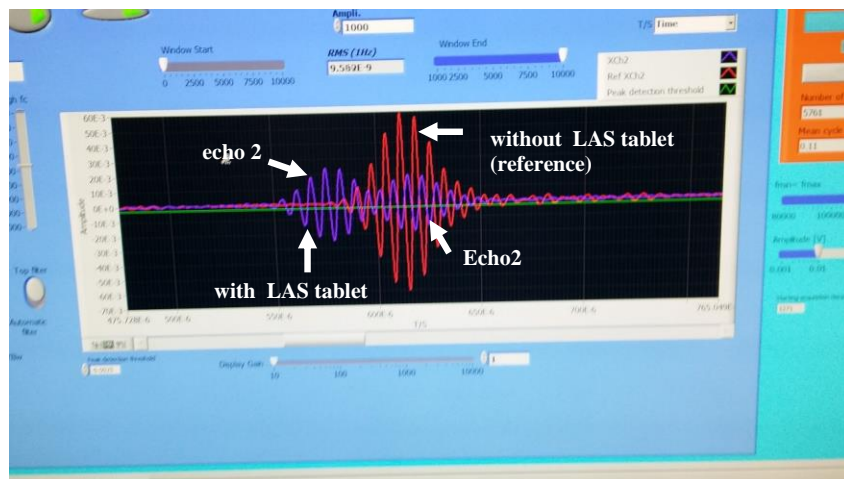
**Figure 2.** Ultrasonic transducers in contact with caked powder to measure sound velocity and attenuation.



**Figure 3.** Non-contact airborne ultrasound transducers transmitting and receiving ultrasonic signal from the surface of LAS flake powder surface

**Table 1.** Different combination of ultrasonic transducers and powder thickness used in the Ultrasonic velocity pulse system measurements shown in figure 2.

Sample	Transducer center frequency (kHz)	Powder thickness (mm)
1	24	16
2	24	21.6
3	24	20
4	50	19
5	50	24



**Figure 4.** Comparison of the reflected signals that received by the airborne ultrasound transducers shown in figure 3. Echo 1 (red color) corresponds to the signal reflected from the stage surface without placing LAS flake tablet. However, echo 2 (violet color) represents the reflected signal from the LAS tablet.

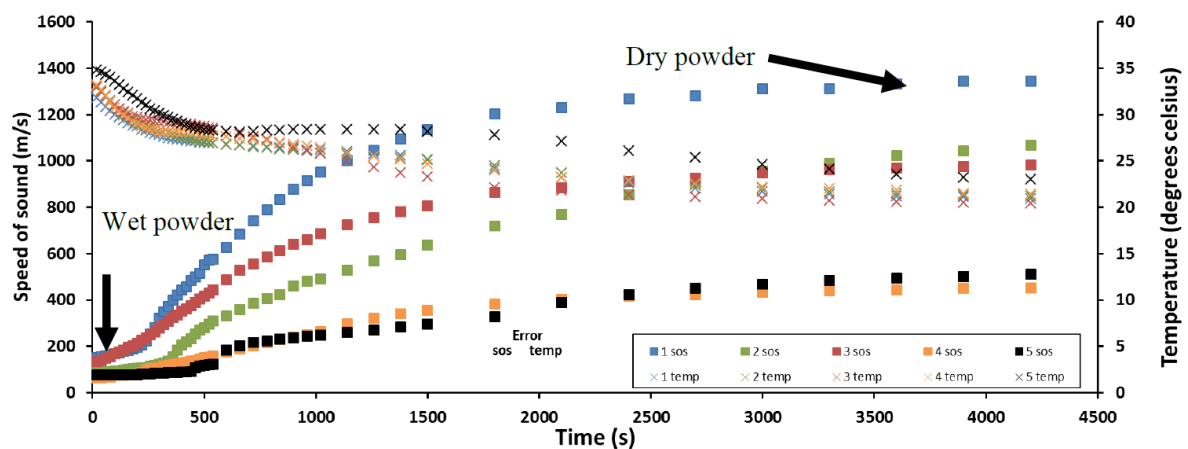
## 4. Results and discussion

### 4.1. Cakes stiffness monitoring

The particles are agglomerated as the powder was wetted due to formation of liquid bridges between these particles. The speed of sound in the wetted powder is low as shown in figure 5. This is because these liquid bridges do not readily transmit sound due to their very small shear modulus and hence acoustic mismatch with the solid particles (High shear modulus) which they connect. However, solid bridges are formed between the particles as the sample consistently dries from 35°C to 22°C. Therefore, the speed of sound significantly increases by an order of magnitude as shown in figure 5. This increase in sound speed depends on the transducer centre frequency. The speed of sound is lower in the higher frequency transducers.

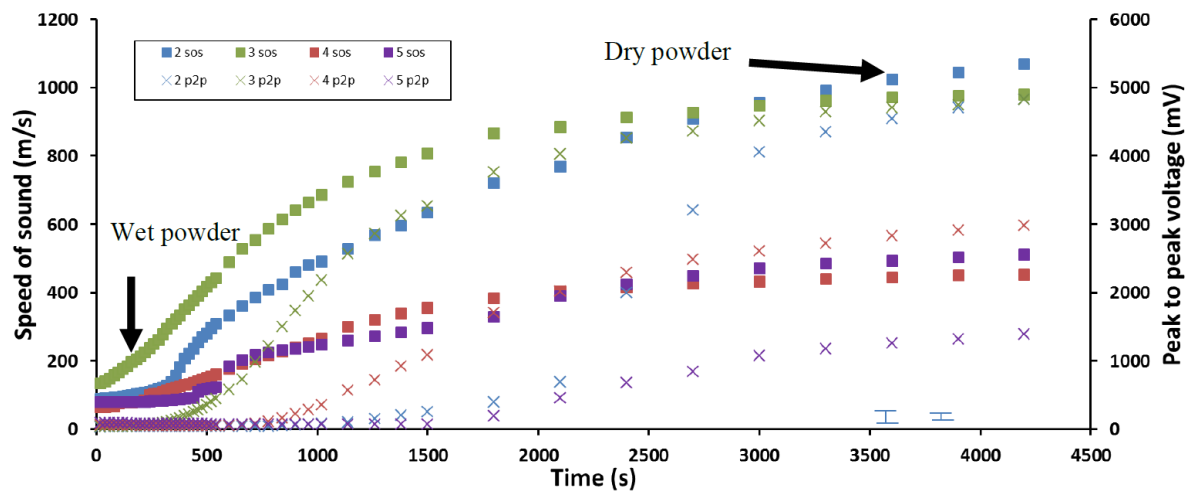
The peak to peak voltage was also measured with the drying time as shown in figure 6. The initial portion of the curves is similar to the velocity curves due to liquid bridges. As the sample dries, the peak to peak voltage of the signal increases due to formation of solid bridges. This indicates that there is a reduction in attenuation across the sample.

The minimum size of the detected caked regions is of the order of a wavelength ( $\lambda$ ) of sound ( $v=f\lambda$ ). The wavelength is 3 mm in the dry powder at 24 KHz frequency ( $f$ ). However, it increases up to 175 mm in the wet caked powder. The transition from dry to caked powder occurs at a wavelength of around 9 mm.



**Figure 5.** Ultrasonic velocity measurements obtained by the system shown in figure 2. The sound speed is low in wetted powder whilst it dramatically rises in the dried powder. The symbol sos corresponds to the speed of sound for the samples shown in table 1 and the symbol temp corresponds to the temperature.

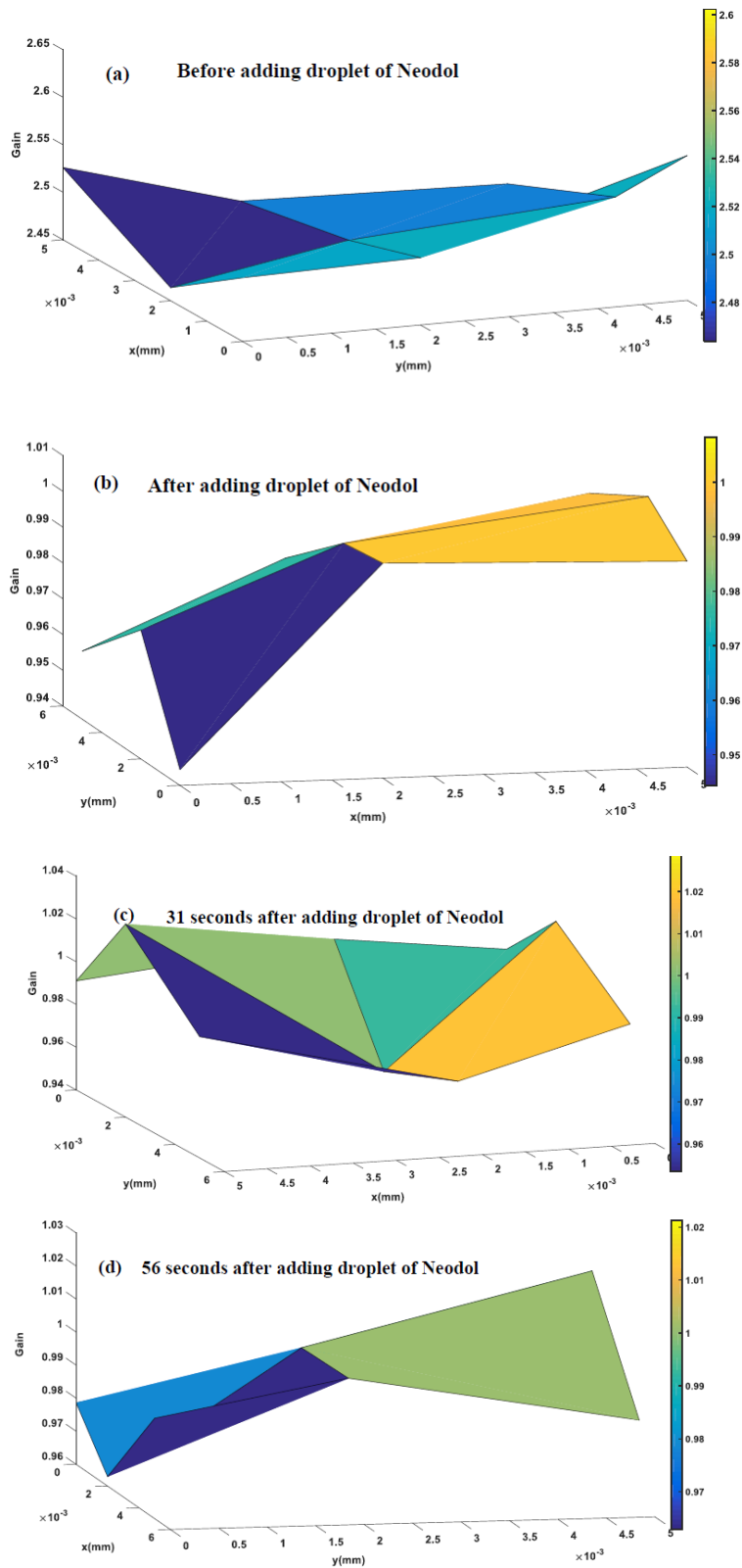




**Figure 6.** Ultrasonic attenuation measurements obtained by the system shown in figure 2. The attenuation is high in wetted powder whilst it greatly improves in the dried powder. The symbol sos corresponds to the speed of sound for the samples shown in table 1 and the symbol p2p corresponds to the peak to peak signal.

#### 4.2. Cakes kinetics monitoring

The interaction between non-ionic surfactant (Neodol) with ionic surfactant was monitored by adding a droplet of Neodol to the surface of LAS tablet. The tablet surface was scanned to measure the pulse time of flight and signal amplitude. Figure 7 shows 3D image of the powder surface before and after adding Neodol droplet. The gain is high before adding the droplet. This is because the signal is highly attenuated as it penetrates through the tablet. This triggers the gain compensation amplifier of the instrument to increase the gain automatically to compensate this reduction in signal amplitude. However, liquid bridges are formed between the particles after adding the liquid droplet. As a result, large portion of the signal is reflected from the surface and therefore the gain value is small. Nevertheless, the gain starts to increase again as the liquid droplet dries at the surface and ultrasonic signal penetrates through the sample.



**Figure 7.** 3D images of LAS tablet obtained: (a) before adding Neodol droplet, (b) directly after adding Neodol droplet, (c) 31 seconds after adding Neodol droplet, and (d) 56 seconds after adding Neodol droplet.

## 5. Conclusions

The goal of this paper was to provide an initial assessment of the feasibility of using sound speed and attenuation measurements to monitor powder caking. The results show that sound speed and attenuation are sensitive to cake effective stiffness and the kinetics of caking. We conclude that ultrasound can be used to monitor powder caking.

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