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## **Ultrasonic Technique for the Quality Control of Water Containing Clay**

Adil Hamine, Bouazza Faiz, Driss Izbaim and Ali Moudden

Ibn Zohr University, FS Agadir, 80000 Agadir, Morocco  
driss\_izbaim@yahoo.fr

**Abstract.** In this work we develop a technique based on the ultrasounds for the quality control of dams's water, by measuring the quantities of clay contained in water. This technique permet to control the presence of the clay grains whose dimension is about 10  $\mu\text{m}$ , by measuring in real time the attenuation of the ultrasonic waves using an interface conceived under LabVIEW. For slurries with different Weight percent of clay 1% or less, high sensitivity is gained by analyzing attenuation measurements obtained from multiple paths through the slurry. For slurries with higher concentrations of clay, sufficient sensitivity is obtained by analyzing data from a simple transmission. The experimental results show that the attenuation of sound due to particles varies linearly with mass fraction, and that the proposed theoretical model can be used to predict this attenuation in high concentration.

## 1 Introduction

Using ultrasonic measurements have been performed to characterize liquids, solids, and gases for over 52 years. Most works applies to ultrasonic process measurement, focusing on application for density, flow measurement, and interface sensing. The need for compact, non invasive, real time measurement of density and concentration for waste slurries of clay, compels us to develop an ultrasonic sensing technique based on the reflection and transmission of ultrasonic signal at the sensor-fluid interface. The dams's water contains various sizes of clay grains. The knowledge of the quantity of clay present in water will make possible to optimize the addition of the chemicals products which allow the clay recovery. The physical treatments of water is done in two phases, a preliminary phase is directed towards a natural elimination of the suspended matter by decantation and a secondary phase which is used for elimination of colloids and clays by addition of chemicals products, these two phases are controlled in real time by this ultrasonic technique. The measure of the viscoelastic parameters of the water allows to develop abacuses that will be useful for the quality control of water and to optimize the addition of the chemicals products necessary to the elimination of these clays.

### 1.1 Ultrasonic measurement technique

The distribution of the particles and their size in slurry are connected, with the determination of velocity or/and the coefficient attenuation [1]. Ultrasonic velocity is the distance which the wave traverses through the sample for a unit of time. The attenuation coefficient is the measurement of the reduction in the amplitude of ultrasonic wave for a unit distance. Ultrasonic velocity and the attenuation coefficient of the sample are given with same preceding manner, except we must replace the distance  $d$  by  $2d$ , because the wave made two alleys and return in the container [5].

$$\text{Ultrasonic velocity: } c = \frac{2d}{\Delta t}$$

$$\text{and the attenuation coefficient is: } \alpha = -\frac{1}{2d} \text{Ln}\left(\xi_{\text{réf}} \frac{A_d}{A_0}\right)$$

$A_0$  and  $A_d$  are the ultrasonic amplitudes at  $x=0$  and after having traversed a distance  $x=d$ .

$\xi_{\text{réf}}$ : Ratio of the acoustic impedances of the extreme mediums.

## 2 Slurries clay propreties

Slurry properties include mixture density ( $\rho_m$ ), mass fraction ( $C_m$ ) or volume fraction ( $C_v$ ), particle size ( $a$ ) and bulk density of the solid ( $\rho_c$ ). The mass of the slurry ( $M_m$ ) is equal to the mass of fluid ( $V_f$ ) plus the volume of the particulate of clay ( $M_c$ ). The volume of slurry ( $V_m$ ) is equal the volume of fluid ( $V_f$ ) plus the volume of the particulate ( $V_c$ ). the mass fraction of slurry is determined by the ratio:

$$C_m = \frac{M_c}{(M_c + M_f)} = \frac{\rho_c (\rho_m - \rho_f)}{\rho_m (\rho_c - \rho_f)} \quad (1)$$

Where  $\rho_f$  is the fluid density. The corresponding volume fraction relationship is:

$$C_v = \frac{C_m}{[C_m + S(1 - C_m)]} \quad (2)$$

Where  $S$  is the solids relative density ( $\rho_p/\rho_f$ ). The volume fraction is proportional to the number of particles per unit volume ( $n$ ). Suppose there are  $N$  particles in the total volume ( $V_T$ ) and each particle occupies a given volume ( $V_p$ ). The volume fraction is given by

$$C_v = NV_p / V_T = nV_p.$$

The density of the slurry was obtained also by weighing a known volume of the sample. The density of the slurry was also obtained from the known weight percentage of the slurry, the density of clay (2600 kg/m<sup>3</sup>), and the density of water. When the particulate is insoluble in the liquid, the weight percentage of solids in the slurry is calculated by using Eq.(3). For process control for a given type of particulate in the slurry, the instrument calibration provides the attenuation as a function of weight percentage. When the instrument operates in-line, the attenuation in turn is related to the weight percentage of solids in the slurry. An example of an in-line sensor for process control is shown in Fig. 1.

$$Wt\% = \frac{\rho_w (\rho_s - \rho_w)}{\rho_s (\rho_c - \rho_w)} \times 100\% \quad (3)$$

Where  $\rho_w$  is the density of water,  $\rho_c$  is the density of the clay, and  $\rho_s$  is the density of the slurry. Some slurries are very attenuative.

## 3 Description of the experimental device

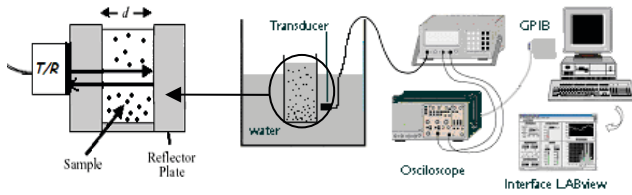


Fig.1 Technique by reflexion

The experimental study is led in a parallelepipedic container (20x8x1) cm<sup>3</sup>. To obtain exploitable signals, it is necessary to be not obstructed by reflexions on the walls of the container. This last is equipped with system of precise positioning for the piezoelectric samples and sensors. This container is filled with water whose density is  $\rho_{eau}=1000$  kg/m<sup>3</sup> and in which the velocity of sound  $c_{eau}=1470$  m/s. was measured at the ambient temperature of 20°C by [5]. The experimental device consists of a generator of pulse SOFRANEL 5052pr which plays the part of receiving and transmitter which sends a very short pulse on the piezoelectric transducer Parametric type to broad band. The pulse generated is dispatched on the container of Plexiglas, containing water and the various quantities of clays. The received signal is amplified and digitized by means of a numerical oscilloscope HP, and is sent towards a microcomputer, by an acquisition card GPIB (IEEE-488.2). The acquisition card is compatible with the graphic language LabVIEW; they are both built by National Instruments [6].

### 3.1 Control in real time under Labview interface of water containing clay

We begin control with the acquisition of the signals illustrated on the screen of the oscilloscope by an application under the graphic programming language LabVIEW. The developed interface makes it possible to specify the number of acquisitions wanted during the experiment and time separating two successive acquisitions. The LabVIEW program collects 50 signals each time and carries out an average to neutralize the interfering signals. The resulting signal represents for the user only one acquisition. An example of an experimental signal retrodiffused by the container which contains the sample is shown in fig.2. This signal is composed of two echoes A<sub>2</sub> and A<sub>4</sub> shown successively from left to right on the figure. This signal is captured starting from the oscilloscope by the LabVIEW program developed. We noted that water is characterized only by the echoes A<sub>2</sub> (reflexion on the interface between the second face of plate 1 and water/clay locked up) and A<sub>4</sub> (reflexion with the interface between water and clay and the first face of plate 2).

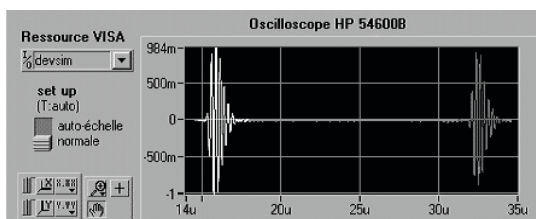


Fig. 2 Experimental signal retrodiffused by the container containing water &amp; clay.

Then, the program separates the echoes by temporal windows by taking only the echo (A<sub>2</sub> and A<sub>4</sub>) however the sites of echoes compared to time (X-coordinate) remain unchanged. The user has the possibility of choosing the first time manually the echo which will take a different color thereafter. The continuous spectra of phase of the echoes A<sub>2</sub> and A<sub>4</sub> are presented on the fig. 3.

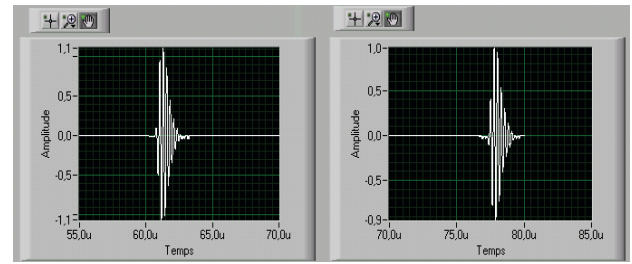


Fig. 3 Filtered signal of the A2 echo, &amp; the A4 echo.

The spectral amplitudes, of the echoes A<sub>2</sub> and A<sub>4</sub>, are determined by the application of the Fast Transform of Furrier to the signals of fig.3. The spectra of amplitudes of the two echoes A<sub>2</sub> and A<sub>4</sub> are respectively presented on fig4.

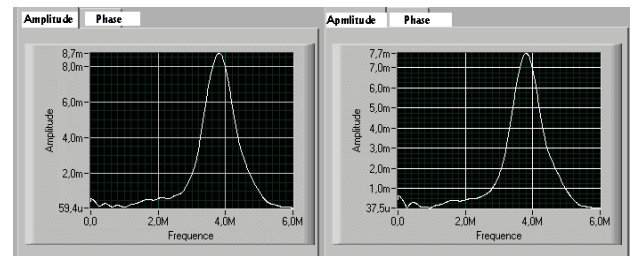


Fig. 4 Spectral amplitude of the A2 echo and spectral Amplitude of the A4 echo

### 3.2 Speed of sound in water and clay mixture

The speed of phase in water is given by the following expression [5,7]

$$V_{produit}(v) = \frac{\omega 2L}{\phi_{produit/pg} - \phi_{pg/produit}} \quad (4)$$

L is the thickness of the container,  $\omega=2\pi f$ ,

$\phi_{pg/produit}$  is the phase of the echo A<sub>2</sub> et  $\phi_{produit/pg}$  is the phase of the echo A<sub>4</sub>.

The transformed Furrier of the echo A<sub>2</sub> insolated by the program, gives the real part R<sub>2</sub>( $\omega$ ) and imaginary part I<sub>2</sub>( $\omega$ ), making possibility to calculate the phase according to the traditional relation:

$$\phi_{pg/produit} = \arctg\left(\frac{I_2(\omega)}{R_2(\omega)}\right) \quad (5)$$

Whereas the transform of Furrier of the A<sub>4</sub> echo insolated by the program developed in our laboratory, gives the real part R<sub>4</sub>( $\omega$ ) and imaginary part I<sub>4</sub>( $\omega$ ), making possibility to calculate the phase  $\phi_{produit/pg}$ :

$$\phi_{produit/pg} = \arctg\left(\frac{I_4(\omega)}{R_4(\omega)}\right) \quad (6)$$

In the calculation of the two expressions (5) and (6) we obtain two spectra whose phase varies between  $-\pi/2$  and

$\pi/2$ . We conceived a LabVIEW interface with an algorithm allowing to unroll the phases in continuous form. Using the algorithm of the phases, and the LabVIEW interface developed, we determined the speed of sound in the product locked up according to the frequency.

### 4 Attenuation waves in mixture

The attenuation of sound in clay and water mixture is given by the following equation: [5,8].

$$\alpha(\nu) = -\frac{1}{2L} \ln\left(\frac{A_4}{A_2} \xi_{ref}\right) \text{ With } \xi_{ref} = \frac{(Z_{pg} + Z_{eau+arg})^2}{4Z_{pg}Z_{eau+arg}}$$

$$Z = \rho_{pg} \cdot C_{pg} \text{ and } Z_{eau+arg} = \rho_{eau+arg} \cdot C_{eau+arg}$$

$Z_{pg}$  is the acoustic impedance in the Plexiglas

$Z_{eau+arg}$  is the acoustic impedance in the mixture of water and clay.

### 5. Results and discussion

We send pulses to the container containing a mixture of water and clay with various concentrations, after a sufficient agitation, which ensures the average distribution of the particles by element of volume, we let the suspension sedimented.

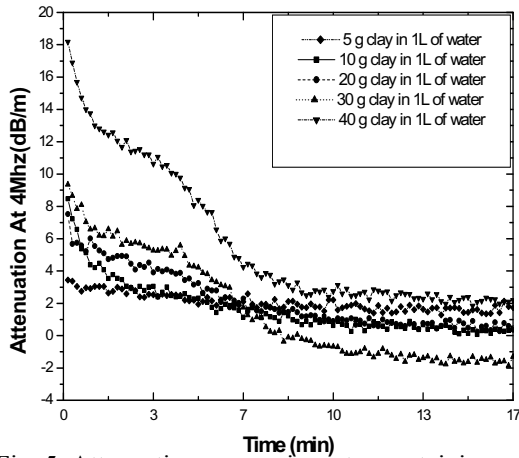


Fig. 5 Attenuation curves in water containing various concentrations of (Transducer used 4 MHz)

The analysis of the curves in fig.5 obtained with a transducer 4 MHz, shows that there is a difference in attenuation for each concentration of clay grains (5g/L-10g/L, 20g/L, 30g/L and 40g/L), marked by an attenuation fall especially for the first minutes of the decantation, after there is a stabilization of attenuation around (0 dB / cm). The analysis of the curves obtained in fig.6 with a transducer 5MHz, shows that the attenuation increases when the clay concentration increases. After eleven minutes from the beginning of settling there is also an attenuation fall increasingly important that the concentration of clay is more important, due to the effect of fast decantation. Data, shown in Table 1, were obtained for the attenuation of ultrasound through clay slurries, having a weight percentage of 1%, 2%, 10%, and 20%.

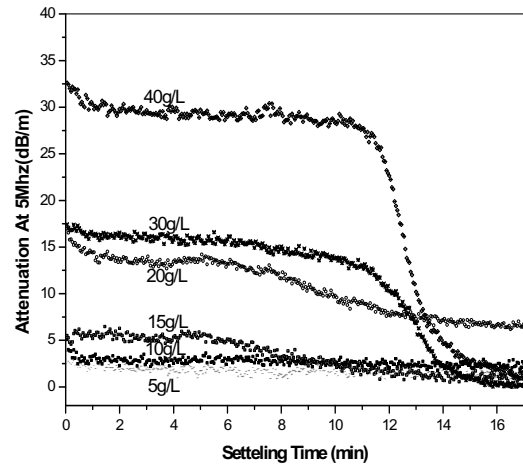


Fig. 6 Attenuation curves in water containing various concentrations of clay (Transducer used 5 MHz)

| Slurry   | Velocity (m/s) | Independent density Measurement | Sensor density | Density obtained from Weight% Kg/m <sup>3</sup> | Z <sub>liq</sub> (Kg/m <sup>2</sup> s) |
|----------|----------------|---------------------------------|----------------|---|--|
| Water    | 1489           | 998                             | 993            | -----   | 1486022                                |
| 1% clay  | 1482           | 1003                            | 1016           | 1004  | 1486446                                |
| 2% clay  | 1487           | 1009                            | 1032           | 1010  | 1500383                                |
| 10% clay | 1484           | 1061                            | 1035           | 1064  | 1574524                                |
| 20% clay | 1489           | 1087                            | 1087           | 1098  | 1618543                                |

Table1: Data obtained for clay slurries

### 5. Theory

The goal with this section is to derive an expression for the attenuation of sound that passes through a suspension of small solid particles of clay and water. For a sound wave that travels in pure water we can consider the water to be lossless. If, however, an object such as a small particle obstructs the wave, the wave will lose energy, and the most dominant loss process is scattering. In a suspension with many particles the scattering will repeat itself many times. When the incoming plane wave interacts with a particle, the sound is scattered in all directions. Assume that all sound energy scattered away from the receiving transducer is lost.

Then, if all particles are of equal size and they all have the same mechanical properties the energy loss,  $d\xi$ , caused by N particles can be written as

$$d\xi = - N \xi_{réfléchi} \tag{7}$$

Where  $d\xi$  is the backscattered energy from each particle and N is the number of particles in the path of the wave. In order to proceed we must determine the number of particles in the path of the wave, and the scattered energy from each

particle. The mass fraction,  $C_m$ , of particles in the suspension is

$$C_m = \frac{N m_{particle}}{m_{liquid} + N m_{particle}} \approx \frac{N m_{particle}}{m_{liquid}} \quad (8)$$

Where  $m_{particle}$  is the mass of one particle and  $m_{liquid}$  is the mass of the liquid phase of the suspension. For a control volume with geometry as shown in Fig.8.

Having width  $w=8\text{cm}$ , height  $h=20\text{cm}$ , and length  $dx=1\text{cm}$  the mass of fluid inside the control volume is given by

$$m_{liquid} = \rho_{liquid} V_{liquid} = \rho_{liquid} \times (w \times h \times dx) \quad (9)$$

and the mass of a particle is

$$m_{particle} = \rho_p \frac{4}{3} \pi \langle a \rangle^3 \quad (10)$$

Where  $\rho_{liquid}$  and  $\rho_{particles}$  is the densities of the liquid and the particles, respectively, and  $\langle a \rangle$  is the mean particle radius. From this we can estimate the number of particles as

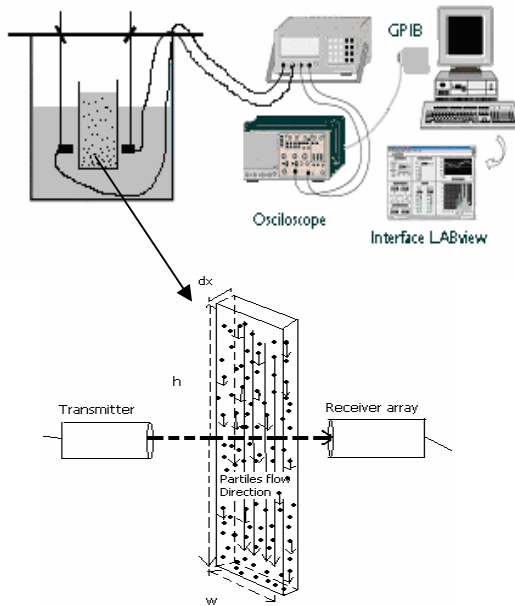


Fig. 8 Transmitter and receiver array used in the measurements, with notations used in the derivation of the attenuation coefficient. (Technique by transmission).

Now, when we have an estimate of the number of particles inside the control volume, we need to determine how each of these clay particles scatters the incoming sound wave. It should be noted, however, that if particles conglomerate, this will change the number of particles. It can be shown [9] that when a plane wave of intensity  $I$  encounters an incompressible rigid particles, the scattered intensity  $I_s$  is given by

$$\frac{I_s}{I} = \frac{16 \pi^4 f^4}{9 C^4 r^2} \langle a \rangle^6 (1 - 3 \cos \theta)^2 \quad (11)$$

Where  $f$  is the frequency,  $c$  is the speed of sound in the surrounding medium;  $r$  and  $h$  are the radius and angle coordinates of a polar coordinate system, respectively. This expression is valid for wavelengths such that  $ka \ll 1$ , where  $k$  is the wave number. Fig.9 shows the pattern of the scattered intensity. Even though Eq. (11) is given in polar coordinates  $r$  and  $h$  it is valid in three dimensions, because

of symmetry around the  $x$ -axis. As expected and as Fig.9 shows, most of the energy is backscattered, i.e. scattered in the opposite direction of the incoming sound wave. In fact only 1/8 of the energy is scattered forward (to the right in Fig.9). The same ratio is true as the long wavelength approximation holds ( $ka \ll 1$ ). The total backscattered power is found by integration of Eq. (11) over a half sphere with origin at the same position as the particle in question. [9]

$$N \approx \frac{m_{eau}}{m_{particle}} = C_m \frac{3}{4} \frac{\rho_{eau} w h}{\rho_{particle} \pi \langle a \rangle^3} \quad (12)$$

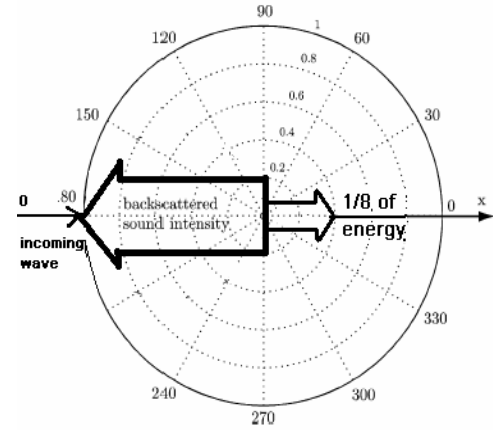


Fig. 9 Scattering from a rigid particle when a plane wave is incident from the left in the picture ( $ka=0,3$ ).

$$\xi_{\text{rétrodiffusé}} = \int_{\frac{\pi}{2}}^{\pi} I_s(r, \theta) 2\pi r^2 \sin \theta d\theta \quad (13)$$

Where the frequency has been replaced by wavelength  $\lambda = c/f$ . For the control volume in Fig.8, the intensity of the incoming wave is related to the energy by Eq. (14), Where  $\xi$  is the incoming plane wave, at position  $x$ , and  $D=16\text{mm}$  is the diameter of the transmitter transducer.

$$I = \frac{\xi}{\pi \left(\frac{D}{2}\right)^2} = \frac{4\xi}{\pi D^2} \quad (14)$$

The backscattered energy now becomes

$$\xi_{\text{rétrodiffusé}} = \frac{114}{9} \frac{\pi^5 \langle a \rangle^6}{\lambda^4} I = \frac{152}{3} \frac{\pi^4 \langle a \rangle^6}{\lambda^4 D^2} \xi \quad (16)$$

The use of Eq. (16) together with Eq. (12) in Eq. (7) leads to an ordinal differential equation for the energy loss of the sound wave.

$$\frac{d\xi_{\text{rét}}}{\xi} = -\frac{152}{3} \frac{\pi^4 \langle a \rangle^6}{\lambda^4 D^2} \times C_m \frac{3}{4} \frac{\rho_{eau} w h}{\rho_{particle} \pi \langle a \rangle^3} \quad (17)$$

$$\frac{d\xi_{\text{rét}}}{\xi} = -A \frac{\langle a \rangle^3}{\lambda^4} \frac{\rho_{eau}}{\rho_{particle}} C_m dx \quad (18)$$

Where the constant  $A = 38\pi^3 \frac{wh}{D^2}$  Integration of both sides

$$\text{gives } \int_{\xi_0}^{\xi} \frac{d\xi_{\text{rét}}}{\xi} = -\int_0^x A \frac{\langle a \rangle^3}{\lambda^4} \frac{\rho_{eau}}{\rho_{particle}} C_m dx \quad (19)$$

Where  $\xi$  and  $\chi$  are integration variables, and  $\xi_0$  is transmitted energy at position  $x = 0$ . After integration we have the attenuation of sound in the suspension is

$$\alpha = C_m A \frac{\langle a \rangle^3}{\lambda^4} \frac{\rho_{eau}}{\rho_{particle}} \quad (21)$$

is the attenuation coefficient due to backscattering. As expected, the attenuation depends on the mass fraction of particles and the propagation distance of the wave. For a measurement system where the distance between the transducers is constant (see Fig.8) the measured attenuation coefficient,  $\alpha$ , will be proportional to the mass fraction,  $C_m$ , alone. The expression for  $\alpha$  in Eq. (21) is valid as long as the mass fraction of particles is low so that we can assume that there is no multiple scattering. Furthermore, in the model we assume a constant mean particle radius,  $\langle a \rangle$ , and constant wavelength of the sound.

## 6. Conclusion

At high concentrations of clay, there is a fall of attenuation at certain moment of the decantation, due to the effect of fast decantation. And there are appearances of plates at the beginning of experiment. It shows that the behavior is not the same for the concentrated slurries and no concentrated in clay. Each concentration is characterized by a clean acoustic signature. The experimental results show that the attenuation of sound due to the particles of clay varies almost linearly with mass fraction of clay. Fig.10. The addition of 0.9% of CaCl<sub>2</sub> each mixture of water and clay makes it possible to accelerate the sedimentation test. We have presented a simple theoretical model for the coefficient of excess attenuation,  $\alpha$ , of pulsed ultrasound due to the presence of particles.

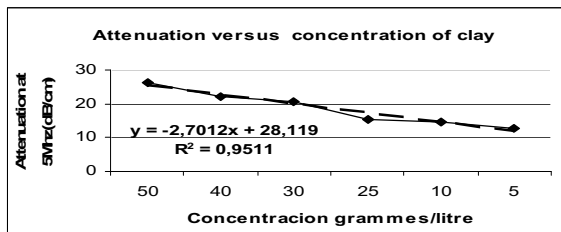


Fig.10 The attenuation coefficient varies linearly with particle mass fraction. Dotted: experiment; line: model

The model predicts that the attenuation coefficient varies linearly with particle mass fraction. The ultrasounds waves are sensitive to the small quantities of clays. We can also control the addition of the chemicals components used to recover clays in suspension in the dams basins.

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