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Measurements Of Acoustic Parameters Of Clay Suspensions And Granulometric Analysis

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ABSTRACT :*This work is intended to provide elements of response to scientific and technical problems related to the implementation in treatment plant dam water. Several suspensions with different distribution of clay grains were studied. The results obtained show that the attenuation increases as a function of the frequency when the grain size increases, and that the shape of the curves shows the existence of two wave-particle interaction phenomena depending on the particle size and the frequency used. Indeed, at very low frequencies, and for very small particles (<2µm), the diffusion effect predominate (multiple diffusion regime). A linear dependence between attenuation and low frequencies exists, $\alpha_{clay}(f)=k.f^n$ with n=1. At high frequencies and for particle sizes (>10 µm), it is observed that the frequency dependence of the attenuation coefficient is important $\alpha_{clay}(f)=k.f^n$ with n=2, characterizing the effect of absorption. In the latter case, we can assimilate a suspension of clay to a polycrystalline material which caused the increase of the attenuation by the stochastic diffusion of the grains. So we can't establish a linear relationship over the entire frequency range.*

Keywords : *attenuation, clay suspension, granulometric analyzes, ultrasound.*

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I. INTRODUCTION

Drinking water must meet a number of criteria (organoleptic, physicochemical, toxicological and microbiological) making it suitable for human consumption. These characteristics are set at the international level by the World Health Organization recommendations (1994), but each state can define its own regulations. In accordance with the regulations set by the National Office of Drinking Water (ONEP) in Morocco, sanitation systems are subject to self-monitoring. Measuring devices typically installed at the input of the station are composed of flow measurement and turbidity.

II. PROBLEMATIC

During flood periods, the drinking water distributed by ONEP in Agadir is sometimes cloudy and unfit for consumption and daily household use, this has been demonstrated when storing tap water in containers for use domestic, it separates into two phases, a little clarified supernatant and sludge (post-precipitation). Indeed, this presents a danger to the health of the consumer, the waters of the network often have the same turbid appearance as the raw water of the dam. The water clarification phase in the treatment plant seems to be the main cause. The quality of drinking water is not always satisfactory from the point of view of turbidity, which also shows an inappropriate implementation of the clarification step. **Fig. 1.**

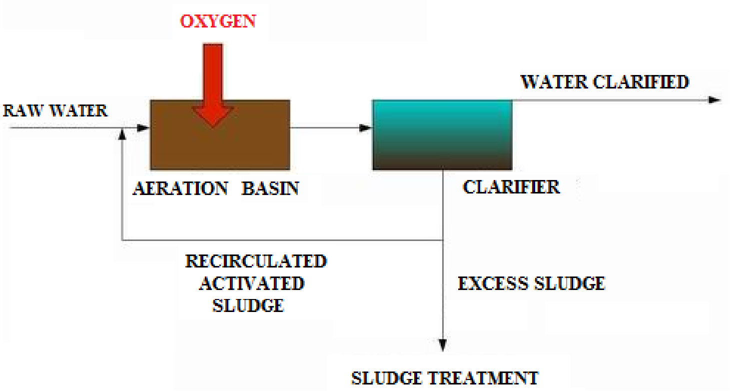

Figure 1: Diagram of the clarification step.

III. NATURE OF THE PARTICLES OF THE DAM WATER

The suspended solids (SS) of the dam water are solid particles like sand, silt and mainly very fine agile. Depending on size, weight and shape, the clay is not decantable. Measurement of (SS) is related to turbidity and gives a first indication of the colloidal content. They limit the penetration of light into the water, decrease the dissolved oxygen content. The study of this kind of biphasic mixture requires the knowledge of the solid phase (clay grains), its hydration and its organization in the liquid phase (water). In these two-phase systems (clay-water grains), ultrasonic methods can also be used to obtain information on the distribution and concentration of clay grains, [1].

IV. PARTICLE SIZE ANALYSIS

The purpose of granulometric analysis is to quantitatively determine the distribution of clay particles by diameter classes. The distribution of the different grain size fractions classifies the soil of the region where the dam is located in a texture class, which defines certain physical behavior parameters, useful water retention, capacity to store nutrients, etc. In principle, all grains can be encountered in dam water, the most common are: pebbles, gravel, sand, silt, and clays. The variability of the grain size extends from the colloidal clay particles, less than 2 µm (difficult to decant), to the blocks that can have a few tens of mm (easily decantable).

4.1 Sieve size analysis

This method consists of using sieving through a series of increasingly smaller sieves. We obtain a distribution by weight range. The particle size analysis is carried out according to the standard method most commonly used AFNOR NF X31/107. It comprises a series of sieves whose openings are based on the 1 mm mesh in logarithmic geometric progression. The granulometric distribution of (SS) and their sizes can be determined using several granulometric methods, such as: sedimentation, for grains of the order of 50 µm and laser diffraction particle size distribution, for sizes less than 10 µm. The conventional analyzes by (sieving and sedimentation) do not allow the development of granulometric distribution curves especially if the sample quantity is small. Moreover, the analysis of a suspension takes only a few minutes at the lazer granulometer or by ultrasound against 6 hours by the method of sedimentation of clay grains.

4.2 Laser diffraction granulometry

Laser scattering granulometry is an indirect measurement technique used in this work. It is generally used to determine the diameter and size distribution of a granular material, which also makes it possible to determine their statistical frequency according to their size. It is therefore a suitable and precise method for checking the particle size of the clay suspensions studied. This method is certainly more accurate than the use of mesh sieves.

4.2.1 Principle

The principle of this method is as follows:

- The optical system of the granulometer records an image of the diffusion (diffraction, reflection, refraction) of a monochromatic radiation by a suspension of particles. **Fig.2**.
- The diffusion images are calculated from a diffusion model, as a function of theoretical particle size distributions.
- The calculated images and the measured image are adjusted by the least squares method.

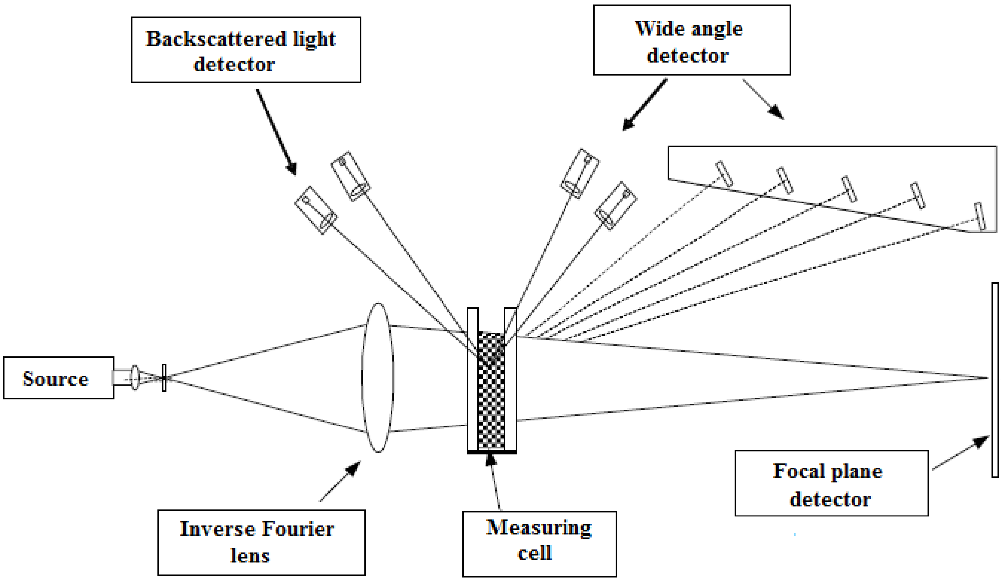

Figure 2: Optical diagram of a laser granulometer.

Commercial software by laser granulometry usually integrates two mathematical models. The most complete model is based on the diffusion theory of Mie. It takes into consideration all optical phenomena related to particle diffusion (diffraction, refraction, reflection).

V. PRINCIPLE OF THE PROPAGATION OF ULTRASONIC WAVES

The causes of attenuation of ultrasonic waves in clay suspensions are:

- The loss of energy by thermal effect. Indeed, any vibration causes a loss of heat.
- The density and size of the grains facilitate more or less the passage of energy.
- The size of the grains will create dispersion.

The attenuation of ultrasonic waves is mainly due to two superimposed phenomena: absorption and diffusion.

$$\alpha = \alpha_a + \alpha_d \qquad (1)$$

Where α_a and α_s are respectively the absorption coefficient and the diffusion coefficient.

5.1 Diffusion and absorption

When an ultrasonic wave encounters a clay grain of small size relative to its wavelength, a multidirectional diffusion is observed, instead of a partial reflection and transmission, (**Fig.3**). The microstructure of the clay suspensions studied is in the form of a medium with multiple sources of diffusion. The irregular shape of the clay grains is also at the origin of diffusion phenomena. Instead of directing the reflected wave in the form of a monodirectional beam, the clay grains re-emit it in all directions.

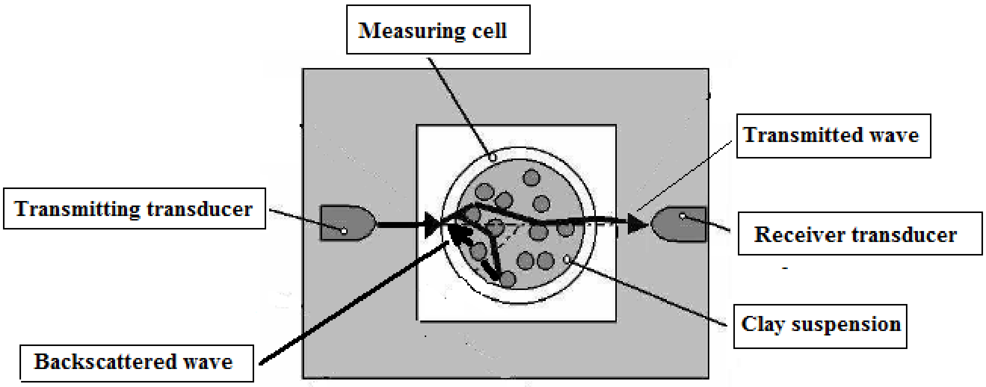

Figure 3: Diagram of an ultrasound wave meeting grains of clay.

The backscattered fraction returns to the transducer, while the scattered waves make significant interferences between them, some constructive, others destructive, having a random character. For this purpose, the backscattered waves contain little information because the backscattered signal is of low intensity so it does not provide information on the size and position of the diffusers.

5.1.1 Absorption

Part of the mechanical energy of the acoustic wave is absorbed by transformation into heat. The ultrasonic wave undergoes successive reflections, and gradually decreases in depth under the effect of diffusion and absorption. The attenuation increases linearly with the frequency of the transducer; the depth of exploration is thus limited with the high frequency transducers. The order of magnitude of the attenuation is 1dB / MHz / m.

5.1.2 Absorption attenuation

When the propagation medium dissipates energy (so it is not purely elastic), we can say that it is absorbing. The intensity of the wave decreases proportionally to the distance traveled dx:

$$dI = -I\alpha\, dx \tag{2}$$

By integrating the equation (2) we obtain:

$$I(x) = I\, e^{-\alpha x} \tag{3}$$

Where I is the intensity of the incident wave and α is the attenuation coefficient expressed in Np/m. This relation makes it possible to estimate:

$$\alpha = -\frac{10}{x}\mathrm{Log}\left(\frac{I}{I_0}\right) \tag{4}$$

The conversion to decimal logarithm gives the usual unit in $dB.m^{-1}$.

$$\alpha_{dB.m^{-1}} = -\frac{10}{x}\mathrm{Log}\left(\frac{I}{I_0}\right) \tag{5}$$

The relationship between the two expressions is naturally:

$$\alpha_{dB.m^{-1}} = -\frac{10}{x}\mathrm{Log}\left(\frac{I}{I_0}\right) = -\frac{10}{x}\frac{\mathrm{Ln}\left(\frac{I}{I_0}\right)}{\mathrm{Ln}\,(10)} = \frac{10}{\mathrm{Ln}\,(10)}\alpha = 4{,}342\,\alpha_{Np.m^{-1}} \tag{6}$$

In fact that α depends on the frequency of the wave. As a first approximation:

$$\alpha = \beta f^{n} \tag{7}$$

Where β and n are numbers that characterize the medium traversed by the wave. In the suspensions, the causes of attenuation are essentially related to the viscosity and physico-chemical changes in the structure of the propagation medium. Viscosity forces exerted between neighboring molecules are an important cause of sound wave absorption in solids and liquids. It is shown that n = 2 for most liquids. The attenuation in water alone was measured at room temperature T = 25°C, α_{water}=0.22 $dB.m^{-1}$ with a 1MHz center frequency transducer, while with a frequency transducer f=100MHz, the attenuation α_{dB} = 2,5$dB.m^{-1}$. So we have interest in limiting the path of the wave to a few micron-meters if we want to avoid significant attenuation. The attenuation measurements can be affected by the insertion of solid particles (clay grains), especially that the density of the clays ρ_{clay} =2500 kg/m^3. The absorption is due to a conversion of vibratory mechanical energy into heat, through different interaction processes between the sound wave and the grains. The absorption coefficient α_a is defined as a function of the frequency, [2].

5.1.3 Diffusion

In the case of diffusion, a fraction of the wave is deflected or reflected during the meeting of suspensions of clay grains larger than 50µm. This causes the reflection of part of the transmitted energy. This reflection results in a dissipation of energy in the form of radiation in all directions of space and the diffusion is more important. In general, three frequency domains corresponding to the ratio between the grain diameter D and the wavelength λ are considered: the high frequency domain, the intermediate domain (stochastic domain) and the Rayleigh domain. The **Fig.4**, describes the amplitude of the backscattered waves as a function of the grain diameter ratio at the wavelength [3].

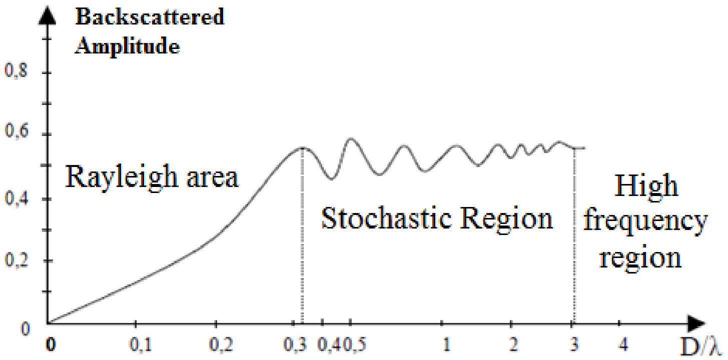

Figure 4: Ultrasound diffusion according to the size of the grains.

5.1.3.1 High frequency domain

We have a so called «diffuse» diffusion, when the size of the grains is large in front of the wavelength ($\lambda << D$). The diffusion is produced by a reflection of the incident ultrasonic waves on the grains. This diffusion is proportional to the frequency and average grain size: $\alpha_s = C_d f$ (8)

5.1.3.2 Stochastic domain

When the average diameter of the grains is of the order of magnitude of the wavelength ($\lambda \approx 2\pi D$), we have a stochastic diffusion. This diffusion increases with the square of the frequency:

$$\alpha_s = C_s f^2 \quad (9)$$

C_s is the diffusion coefficient for waves corresponding to the stochastic domain. The following relations respectively give the expression of the diffusion coefficient for a longitudinal wave. [4]:

$$C_S^L = \frac{3.10^{-2} \pi^2 V A^2}{\rho_0^2 V_L^6} \quad (10)$$

Where: *A* is a factor of anisotropy depending on the elastic constants (constants realives in the meduim), ρ_0 the density of the medium, V the mean volume of the grains, V_L is the velocity of the longitudinal waves.

5.1.3.3 Rayleigh Domain

When the average diameter of the grains D is very small than the ultrasonic wavelength λ, we obtain the Rayleigh scattering ($\lambda >> 2\pi D$). In this field, each grain can be considered as a particle scattering the wave following a diffraction mechanism. The total energy dispersed per unit volume can then be calculated as being proportional to the sum of the grain cross sections contained in the unit volume. It has been shown that this diffusion coefficient is of the form, [4] $\alpha_s = C_r f^4$ (11)

$$C_r^L = \frac{2{,}1\ 10^{-2} \pi^3 V A^2}{\rho_0^2 V_L^8} [2 + 3(V_L)^5] \quad (12)$$

V_L is the velocity of the longitudinal waves.

VI. TEXTURE CLASS OF THE STUDIED SAMPLES

The most significant textural classification of our sample is to locate it in the triangle of texture classes (G.E.P.P.A), study group of applied soil problems, **Fig.5**. [5]. This triangle contains 16 classes grouped into three predominantly groups of clay, silt or sand. The textural class of the studied sample is determined according to the positioning of the values in clays and total silts. According to the microscopic analysis of a 1.5cm^2 sample spread on a slide, we have estimated that the studied sample Ei, consist of 27.58% clays and 29.82% silts. The horizontal line passing through the level of the clays and the vertical line passing through by the level of the silts intersect at the point located in the area of silt and clay, which shows that the sample studied are described as silt-clay.

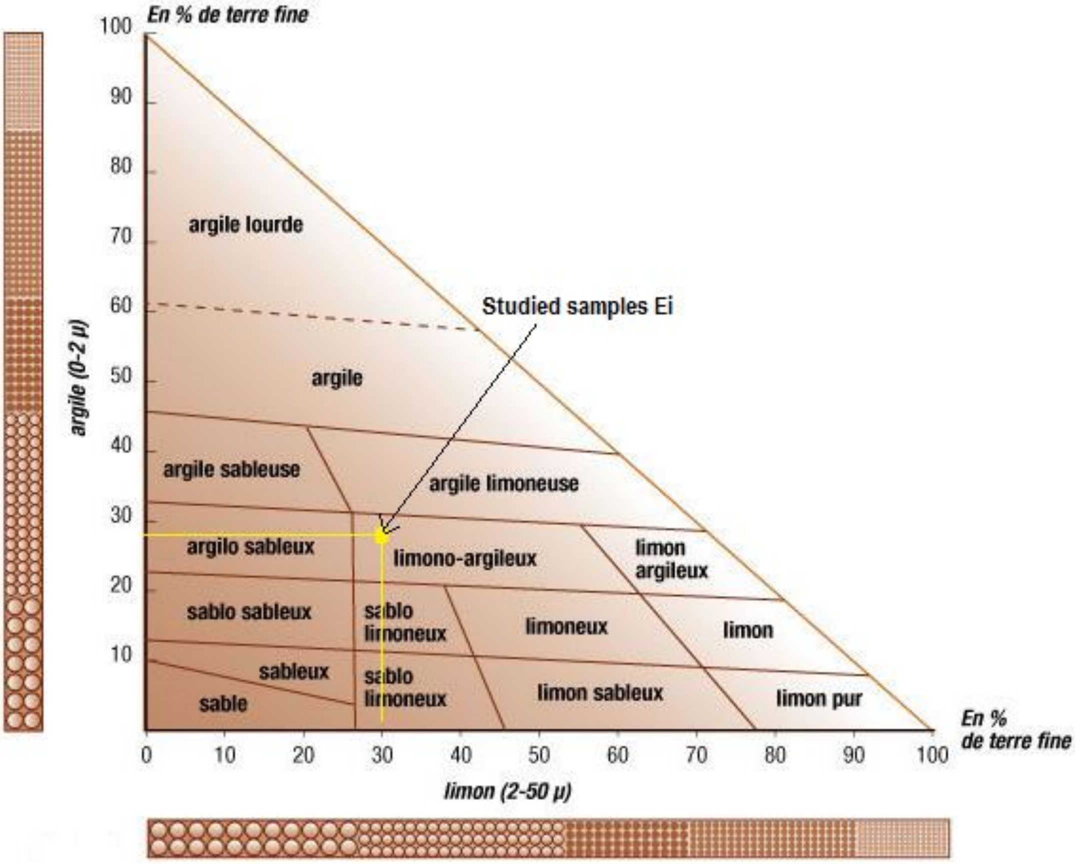

Figure 5: Texture diagram according to GEPPA classifications. [5].

VII. METHODS AND TECHNIQUES OF MEASUREMENT

The clay used in this study is taken directly from the dam water treatment plant. Samples of sludge and water were collected at the entrance of the station during floods. After, the upper part consisting of fine particles (less than 2 µm), coarse elements (gravel, pebbles, blocks) is separated, fine clay suspensions are obtained, which will be studied later in a lazer granulometer. The coarser fractions, sands and silts are determined by sieving. The sum of the mineral granulometric fractions is equal to 100%. In order to determine the particle size of the sample taken, a quantity of 500 g of sludge is taken from the bottom of the test tube, and placed in an oven at 105°C for 6 hours, at the end there is a quantity of 300g of dry clay, it is placed there after in a SASSUOLOLAB vibrator type SE-7 equipped with several sieves vertically classified from top to bottom as follows: (600µm, 425µm, 300µm, 250µm, 180µm, 125µm, 63µm). The time of this second operation is half an hour. At the end, we weigh the rejects in order to deduce the percentage of sieve.

VIII. SIEVE RESULTS BY SIEVING

8.1 Particle size curve

The granulometric curve gives the distribution of grains by size classes, it is essentially known by the values of cuts between the classes of particles. The values of cuts between coarse and fine grains are conventional. The curve in **Fig.6**, illustrates the different cuts between grains of our sample studied. This curve decreases exponentially, shows that the screen at 2µm corresponds to the cut between fine and coarse clays.

- The 2 mm sieve separates the sands from the gravel;
- The 0.05 mm sieve corresponds to the separation between coarse and fine silts.

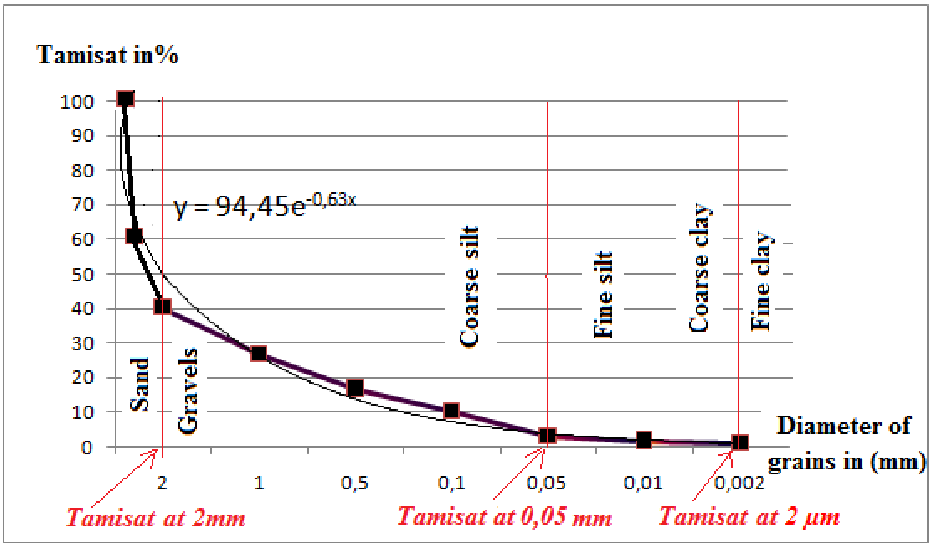

Figure 6: Granulometric curve of the sample studied.

The shape of the granulometric curve is also extremely important, we speak of the uniform particle size, if the grains are almost all of the same size, of the discountinue granulometry, if the grain sizes are different, and of the spread particle size, the one between the first two [6]. From this study we concluded that our sample contains a variety of grain sizes, ranging from 2mm up to 2µm, and has a spread particle size. The results of measurements concerning dry particle size analysis are given in **Tab.1.**

Tab1e 1: Different types of grains and percentages by mass in each sieve.

Grain type	Pebbles M1	Gravel M2	Coarse sands M3	Fine sand M4	Coarse silt M5	Fine silt M6	Clay M7	--
Grain size in (mm)	≥ 2	≥ 1	≥ 0,5	≥ 0,1	≥ 0,05	≥ 0,01	< 0,002	Total
Mass retained (g)	120,5	80,3	50,4	30,9	9,2	5,4	3,3	300

Data from the particle size analysis can also be represented using a plot of the percentage of the sieve (grains smaller than a given size) as a function of sieve size or the logarithm of sieve size, called granulometric curve. **Fig.6**, **Tab.2**.

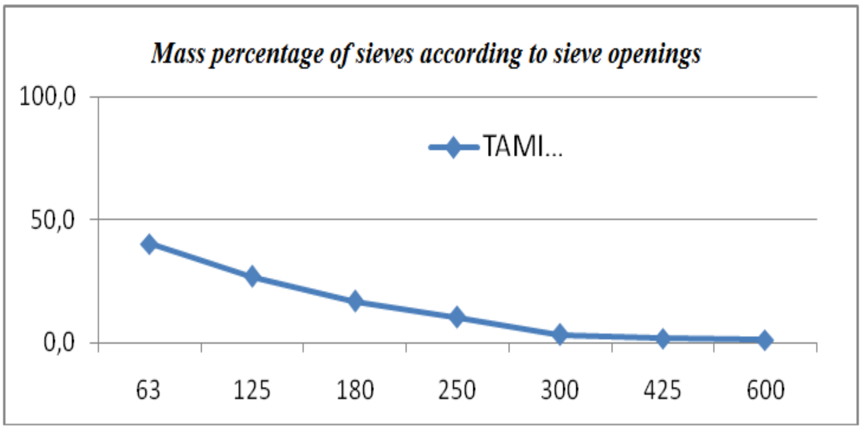

Figure 6: Representation of the mass percentage of the different sieves as a function of the nominal sieve opening size.

Table 2: Weight of rejects and their mass percentages in each sieve.

Sieve diameter (µm)	63	125	180	250	300	425	600
Weight retained in (g)	120,5	80,3	50,4	30,9	9,2	5,4	3,3
Tamisat %	40,2	26,8	16,8	10,3	3,1	1,8	1,1

According to the granulometric curve and the table 3, we have:

- From 63 µm to 180 µm the mass percentage of restraints equal to 83.7%;
- From 180 µm to 600 µm the weight percentage of retainers equal to 16.3%.

8.2 Results of the Lazer granulometry

8.2.1 Measurement method

To make a lazer granulometry of a sample of clay grain size less than 50 µm, it is first necessary to dilute it, so that the granulometer is not out of adjustment. Then a background measurement is made at room temperature in order to record the diffraction phenomena generated by the water which serves as diluent. Then a quantity of clay of the M7 sample is added to this water, in order to prepare a diluted suspension of 1/10 (tenth). The clay suspension thus prepared is injected into the clean measuring cell, and each clay particle which passes in front of the light beam deflects the light which is then analyzed by detectors. From **Fig.7**, we see that the average diameter of the clay particles is of the order of 2 µm, this value is identical to the value given by the dry sieving method. Subsequently, this sample will be studied by ultrasound.

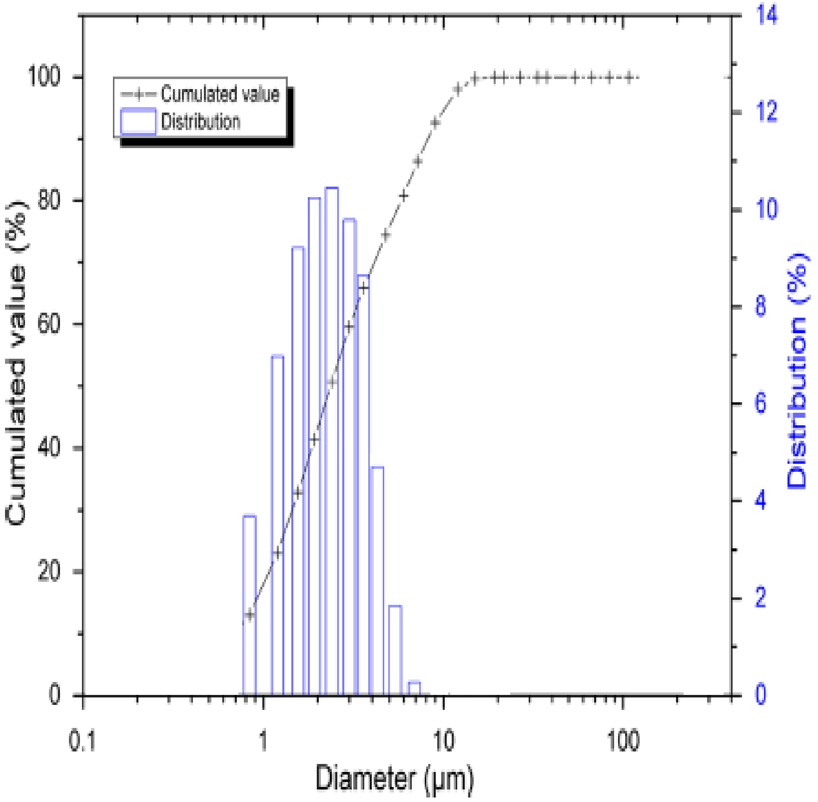

Figure 7: Granulometric curves of the clays obtained. (Averaging 10 measurements).

sieving method. Subsequently, this sample will be studied by ultrasound.

In the same way, we take a quantity of the silts of the sample M6, mixed with the distilled water, (concentration of 1/10) is the injected in the cell of measurement. **Fig.8** shows that the average distribution of the grain size is of the order of 10 µm. This value is identical to the value given by the dry sieving method. Subsequently, this sample will be studied by ultrasound.

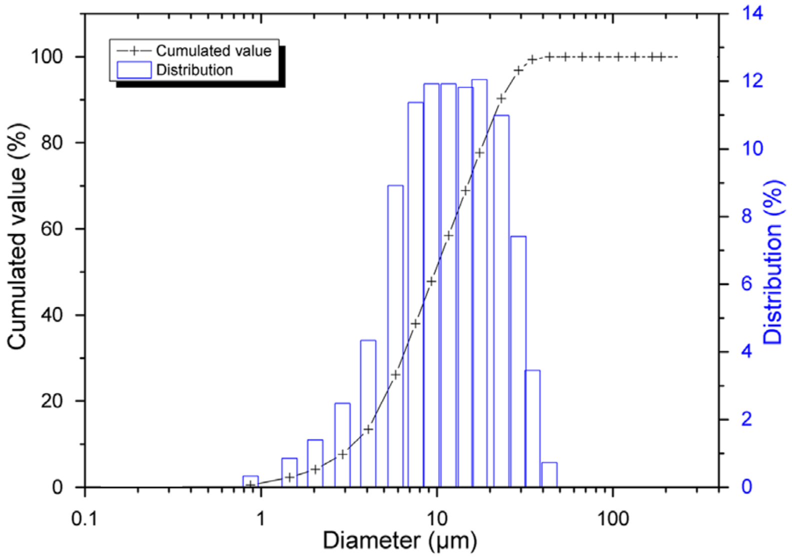

Figure 8: Granulometric curves of the fine silt obtained. (Averaging 10 measurements).

In the same way, that the first two analyzes, we take a quantity of the sample M5 is injected into the measuring cell. **Fig.9** shows that the average distribution of the grain size is of the order of 50 µm. This value is identical to the value given by the dry sieving method. Subsequently, this sample will be studied by ultrasound.

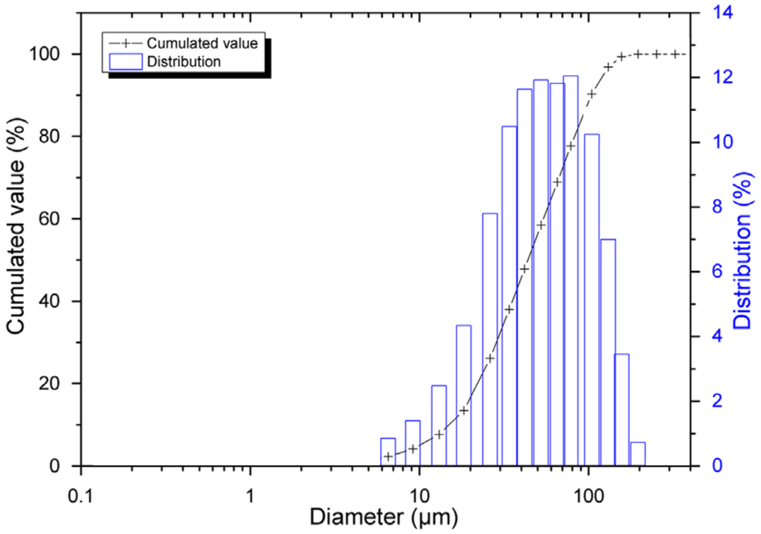

Figure 9: Granulometric curves of the coarse silt obtained. (Averaging 10 measurements).

IX. MEASUREMENT OF ULTRASONIC PARAMETERS OF SUSPENSIONS

The transmission method is used for measuring the ultrasonic parameters of the suspensions. This method consists of placing two transducers on either side of the container containing the suspension studied. The transmitting translator transmits an ultrasonic wave through the container, if the suspension is concentrated in clay grains of large sizes, the ultrasonic wave is reflected. If the suspension is diluted in clay grains of very small size, the sound wave reaches the second translator opposite the transmitter which serves only as a receiver on the other side of the container, and a lower energy will be collected. This loss of energy is all the more important as the grain size and the density are large. **Fig.10.**

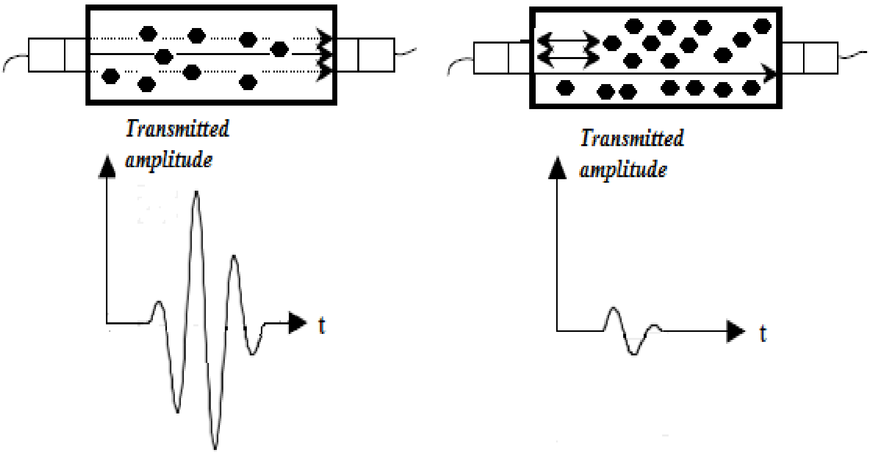

Figure 10: Simplified diagram and example of ultrasonic signal by the transmission method.

9.1 Measurements of transmission attenuation

Measuring the attenuation coefficient, α_{clay}(dB/m), is the measure of the energy loss along the path of the acoustic wave in the suspension. Part of the energy is scattered outside the reception beam. The ultrasonic attenuation spectrum of five suspensions of clay of the same concentration 20 g/L is measured over a maximum frequency range of 1 to 100 MHz. Each of them is formed by a mean grain diameter: (2 µm, 10 µm, 50 µm, 100 µm and 500 µm). In general, it is noted that the attenuation in a suspension increases as grain size and frequency increases. At low frequencies, between 1 MHz and 20 MHz, relatively large propagation distances must be used to achieve measurable attenuation. At high frequencies, between 20MHz and 100MHz, there are strong attenuations, the propagation distances must be shorter so that the overall signal is not absorbed by the suspension. **Fig.11.**

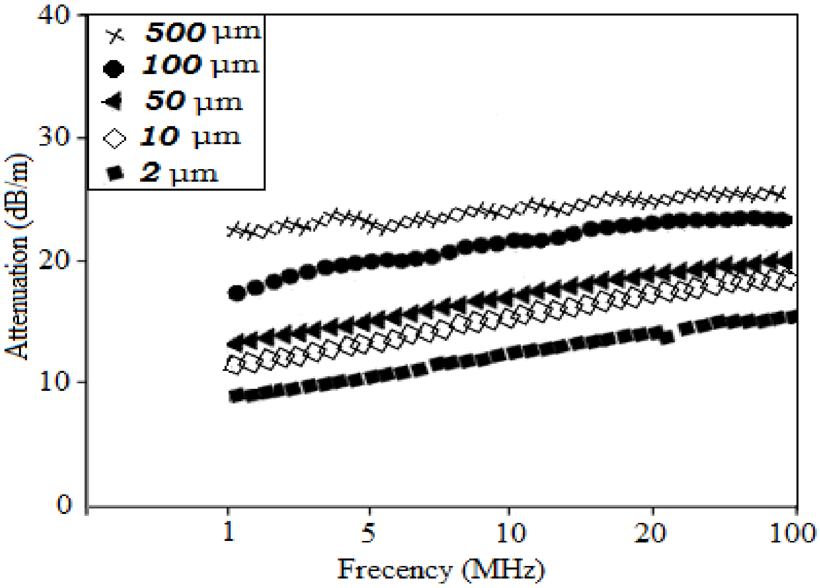

Figure 11: Attenuation according to the central frequencies of the transducers, for a concentration of 20 g/L.

Fig.12, shows the results relating to the attenuation measurement of the ultrasonic waves through several suspensions for different frequencies and grain sizes. We note that the attenuation increases with frequency as grain size increases. The appearance of the spectra shows the existence of two wave-particle interaction phenomena depending on the particle size and the frequency.

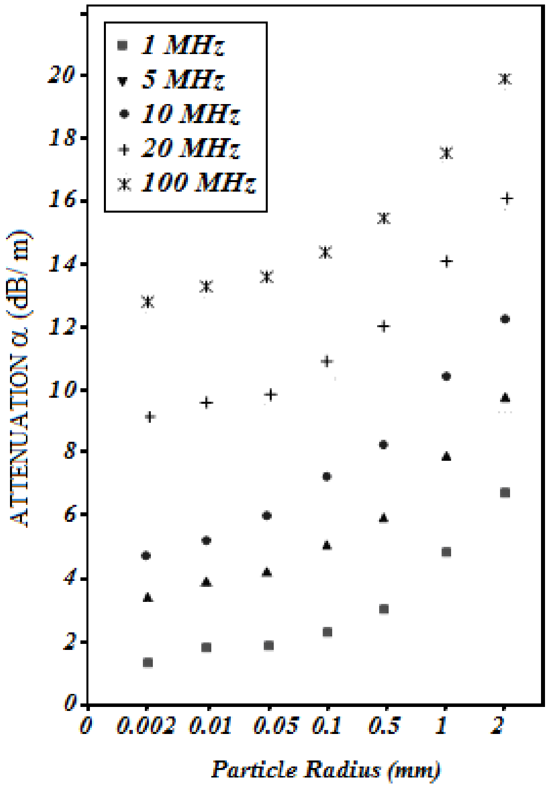

Figure 12: Attenuation as a function of the average particle diameter. Concentration 5g/L.

In fact, at very low frequencies between 1MHz and 10MHz, and for very small particles (<50 μm), the diffusion effect predominates (multiple scattering regime) and the attenuation values are low. For particle sizes (> 50 μm), there is an exponential increase characterizing the effect of absorption. At high frequencies between 10 MHz and 100 MHz, the curves are similar in appearance to low frequencies, except that the attenuation values are higher, characterizing the scattering effect (multicast regime). A linear dependence between attenuation and low frequencies exists, $\alpha_{clay}(f) = k.f^{n}$ with n = 1. At high frequencies and for particle sizes (> 50 μm), it is observed that the frequency dependence of the attenuation coefficient is important $\alpha_{clay}(f) = k.f^{n}$ with n = 2, characterizing the effect of absorption. This frequency dependence of the attenuation coefficient has been proposed by Hamilton, [7-8], who defends the theory of a linear relationship between the frequency and the attenuation coefficient. While Kibblewhite et al. [9], argues from a series of measurements that we can't establish a linear relationship over the entire frequency range. The same approach was taken up by Xeridat [10], who developed a model describing the attenuation dominated by the diffusion phenomenon. Thus, the diffusion coefficient has been modeled as a function of the frequency f^{γ}. This γ parameter reflects the interaction between the ultrasonic signal and the scattering grains.

9.2 Effect of increased concentration

Fig.13, shows that attenuation varies linearly with frequency as grain concentration and size increase. This can be explained by the fact that the attenuation α is proportional to the number of particles and also to their sizes. So we can say that the attenuation is composed of two terms, we related to the absorption processes (α_a), and the other to the diffusion (α_d) :

$$\alpha = \alpha_a + \alpha_d \tag{13}$$

Absorption attenuation is associated with dissipative losses of energy both by the particle and by the external medium, while diffusion attenuation results from energy losses due to reflections of the incident wave front on the grains clay.

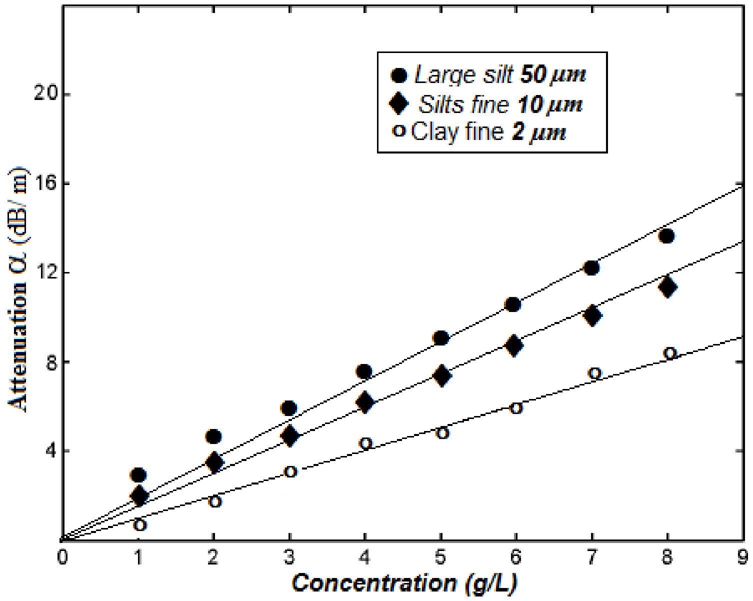

Figure 13: Attenuation as a function of grain concentration; With a 5 MHz transducer.

9.3 Ultrasonic velocity measurements by transmission

Fig.14 shows that ultrasonic velocity increases slightly as grain concentration and size increases. We also note that when the size of the grains is d = 10 µm which is close to the order of the wavelength λ of the frequency used, we found that the ultrasonic velocity varies in a linear and fast way depending on the concentration. As a result, the ultrasonic velocity can't be a relevant parameter for the characterization of clay suspensions unless the frequency of the transducer used is of the order of the size of the clay grains.

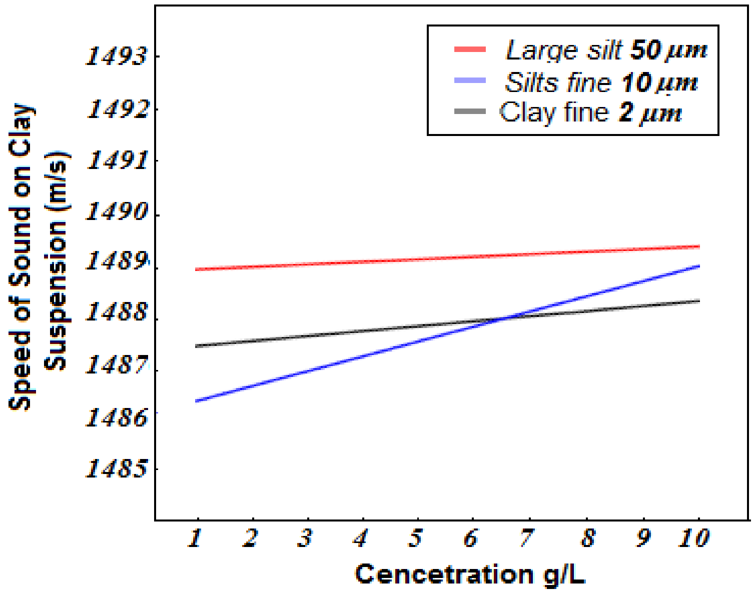

Figure 14: Ultrasonic velocity as a function of grain concentration. (With a 5 MHz transducer).

X. CONCLUSION

From the results obtained, we observe that the attenuation increases as a function of frequency when the grain size increases, and that the shape of the curves shows the existence of two wave-particle interaction phenomena depending on the size particles and frequency. Indeed at very low frequencies, and for very small particles, the scattering effect predominates whereas at high frequencies and for particle sizes, there is a significant frequency dependence of the attenuation coefficient $\alpha_{Clay}(f) = k.f^{n}$ with n=2, characterizing the effect of absorption. In this case, we can assimilate a suspension of clay to a polycrystalline material which caused the increase of the attenuation by the stochastic diffusion of the grains. This diffusion increases with the square of the frequency. As a result, a linear relationship can't be established over the entire frequency range. The results made it possible to construct frequency charts that will be used to know the size and distribution of suspended solids of any suspension.

Ultrasonic velocity can't be a relevant parameter for the characterization of clay suspensions unless the frequency of the transducer used is of the order of the size of the clay grains.

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