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Effects of impurities on particle sizing by acoustic attenuation spectroscopy

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A B S T R A C T

It is important to have correct information regarding particle size in order to interpret, control, and optimize many industrial processes. Prior to the recent advent of acoustic attenuation spectroscopy, it was difficult to study particle size distribution online and under real process conditions in processes involving concentrated dispersions (suspensions or emulsions). The technique still needs improvement because it is less known how and under which conditions to employ the technique when dispersions involve impurities that could be soluble, insoluble, in the form of additives, and so on. This lack of understanding has almost halted the advancement in applications of the technique to various processes that essentially involve dispersions with impurities. This study investigates aqueous suspensions of CaCO₃ at different concentrations (i.e., 5%, 10% and 20% mass/mass) with added impurities of MgCO₃ (insoluble impurity), NaNO₃ (soluble impurity) and sodium polyacrylate (soluble additive) at varying proportions (5%, 10%, 20% and 30% of the weight of CaCO₃). The study characterizes and compares dispersion with and without impurity in order to demonstrate the possible ways in which addition of an impurity change the original acoustic attenuation spectrum of a dispersion. The study brings the conditions in which acoustic attenuation spectroscopy is capable of explaining that addition of an impurity will not change original particle size of the disperse medium.

1. Introduction

It is important to have correct information regarding particle size in many industries such as chemicals, paints, dyes, pharmaceuticals and cosmetics in order to interpret, control, and optimize many processes. In last few decades, many techniques of particle sizing have been developed based on different principles such as laser diffraction, dynamic light scattering, image analysis and acoustic attenuation spectroscopy. Each technique has its advantages and limitations depending upon the application. In above techniques, acoustic attenuation spectroscopy is quite prominent because of many advantages over other techniques of particle sizing. For example, it is non-destructive and non-invasive; it can study dispersions (suspensions and emulsions) without the need for diluting them; it can be employed online, under real process conditions, over a wide range of particle size (10 nm to 1000 μm) and over a wide concentration range (0.5 vol% to 50 vol%), depending upon application [1–5]. Many studies have verified suitability of acoustic attenuation spectroscopy for simple dispersions (i.e. dispersion involving one disperse phase and one continuous phase). However, there is still lack of understanding how and under which conditions to employ the technique when dispersions are not simple. For example,

in many industrial applications, dispersion of CaCO₃ are prepared from raw CaCO₃ that may contain impurities such as silica or MgCO₃ making the dispersion of CaCO₃ complex for particle sizing by means of acoustic attenuation spectroscopy. Similarly, during wet grinding processes, the addition of additives in dispersions makes them complex. The lack of understanding has almost halted the advancement in applications of the technique to various processes that essentially involve dispersions with impurities etc. Pointing out this lack, Franck Babick et al. write that only little experience has been gained for the performance under more real conditions, e.g. for the presence of contamination, for multi-component-systems or for the case that the knowledge of the involved phases is incomplete [6]. Similarly, Povey has emphasized the importance of characterizing the surfactants in the solvent for successful particle sizing by means of acoustic attenuation spectroscopy [7]. This study is an attempt to bridge the gap between particle sizing of the dispersion with and without impurities by means of acoustic attenuation spectroscopy. In particular, the study aims to characterize and compare dispersions with and without impurities at different conditions in order to find possible conditions in which dispersions with impurities may be studied by means of acoustic attenuation spectroscopy.

2. Theory

Particle sizing by means of acoustic attenuation spectroscopy is based mainly upon ECAH theory, which is presented in terms of differential waves equations derived from fundamental laws of

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conservation of mass, energy, momentum, thermodynamic equations of state and stress-strain relations for isotropic elastic solids (or viscous fluids). It considers case of simple dispersions (one disperse medium in one continuous phase). The details of ECAH theory may be studied in the papers of Epstein and Carhart [8] and Allegra and Hawley [9] who contributed to development of this theory. With the help of ECAH theory, acoustic attenuation spectrum of a dispersion can be determined if particle size distribution and certain physical properties of the dispersion are known. The Fig. 1(a) illustrates a simple schematic of ECAH in terms of input and output. A commercial acoustic attenuation spectrometer for particle sizing (such as Malvern Ultrasizer) makes use of ECAH theory in a manner as illustrated in Fig. 1(b). It measures acoustic attenuation spectrum with the help of transducers; use information of physical properties (mentioned in Table 1) and finally determines particle size distribution by inverse calculations. The process of inverse calculations is carried out with suitable algorithms employed generally with the help of computer program.

When a sound wave of certain frequency is passed through a particulate system, it interacts with the particles (transmission, reflection, absorption and scattering etc.) and as a result it undergoes attenuation (decrease in amplitude) that can be measured in terms of attenuation coefficient [1-5]. It is defined as the decrease in the amplitude of the sound wave per unit distance travelled and may be expressed as [5]:

$$\alpha = -\frac{1}{x} \ln \frac{A_x}{A_0} \quad (1)$$

where A_0 is the initial amplitude of the sound wave ($x=0$) and A_x is the amplitude after it has travelled a distance x as illustrated in Fig. 2.

If a band of sound waves of different frequencies is passed through a dispersion from a certain distance (x), the attenuations in each sound wave of certain frequency may be calculated using Eq. (1). An acoustic attenuation spectrum describes acoustic attenuation constants expressed as a function of respective frequencies of sound waves. In a commercial acoustic attenuation spectrometer such as Malvern Ultrasizer, different values of x and range of frequencies may be selected for determining an acoustic attenuation spectrum. Alba discussed the relationship between frequency, attenuation constant and particle size distribution by means of acoustic attenuation spectroscopy [3]. If the attenuation is measured at a single frequency, there would be four potential monosize distributions, which could have produced that measured attenuation: two in the region of viscous attenuation, one in the Rayleigh scattering region, and a fourth in the diffraction zone. In the absence of either measurement or modeling errors, two frequencies would suffice to identify the correct size from the four possible sizes for each frequency.

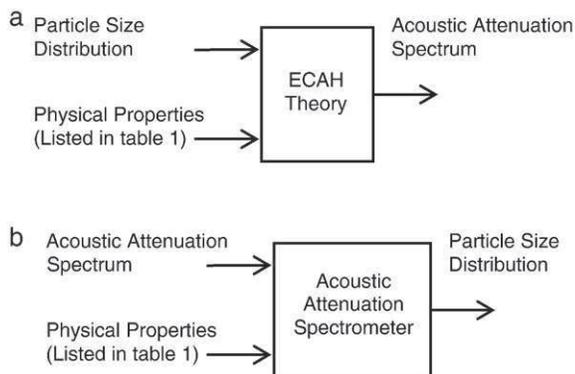


Fig. 1. (a): Schematic of ECAH theory in terms of input-output. (b): Schematic of an acoustic attenuation spectrometer in terms of input-output.

Table 1 Physical properties required for particle sizing by acoustic attenuation spectroscopy.

Disperse medium (particles)	Dispersing or continuous medium
1 – Density (g/cm^3)	1 – Density (g/cm^3)
2 – Sound velocity (cm/s)	2 – Sound velocity (cm/s)
3 – Thermal dilation ($^\circ\text{C}^{-1}$)	3 – Thermal dilation ($^\circ\text{C}^{-1}$)
4 – Thermal conductivity ($\text{erg cm}^{-1} \text{s}^{-1} \text{C}^{-1}$)	4 – Thermal conductivity ($\text{erg cm}^{-1} \text{s}^{-1} \text{C}^{-1}$)
5 – Heat capacity ($\text{erg g}^{-1} \text{C}^{-1}$)	5 – Heat Capacity ($\text{erg g}^{-1} \text{C}^{-1}$)
6 – Shear rigidity (dynes cm^{-2})	6 – Viscosity (Poise)

3. Materials

3.1. CaCO_3

CaCO_3 , procured through Merck KGaA Germany, served as the disperse medium in the study. It was prepared by precipitation and had the following properties: density at $20^\circ\text{C} = 2.93 \text{ g/cm}^3$, bulk density $\sim 400\text{--}700 \text{ kg/m}^3$, solubility in water at $20^\circ\text{C} = 0.014 \text{ g/l}$, and thermal decomposition $>825^\circ\text{C}$. A Mastersizer2000 of Malvern Instruments Limited, U.K., which is based on laser diffraction technique, was used to find the mean particle size of CaCO_3 (in dry form). It was found to be $1.6 \mu\text{m}$.

3.2. Magnesium carbonate

Magnesium Carbonate (anhydrous) was purchased from Fisher Scientific, U.K. It was insoluble in water and its density was 2.958 g/cm^3 at 25°C . Its mean particle size (in dry form) was determined by using the laser diffraction technique with a Mastersizer2000 of Malvern Instruments Limited, U.K. It was found to be $7.3 \mu\text{m}$.

3.3. Sodium nitrate

Sodium nitrate was purchased from Fisher Scientific, U.K. Its density was 2.26 g/cm^3 . It was highly soluble in water with a solubility 921 g/l at 25°C .

3.4. Sodium polyacrylate

Sodium polyacrylate procured through Sigma-Aldrich Co. was used in the study. It had a density of 0.55 g/cm^3 at 25°C and average $M_w \sim 5100$ (determined by gel permeation chromatography (GPC)).

3.5. H_2O

Ultrapure water produced from “Purelab Ultra” of VWS Ltd. (U.K.) was used in the study. The density of water was found to be 0.9981 g/cm^3 at 25°C .

4. Equipment and software

An acoustic attenuation spectrometer (Ultrasizer) of Malvern Instruments Limited U.K. was employed in the study. The system has four main units: (i) instrument, (ii) electronics enclosure, (iii) heater/chiller, and (iv) computer system. The instrument uses transducers to

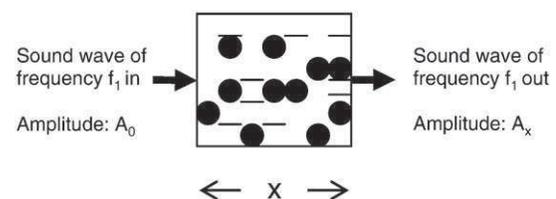


Fig. 2. Passage of a sound wave through a dispersion.

pass ultrasonic waves through a sample of dispersion. The sample may be measured in batch or continuous mode. The electronic enclosure contains the microwave generator and various electronic systems for amplification, for signal processing, for control of movement of transducers and for control of temperature. The heater/chiller was used to control the temperature. The computer system is a standalone computer to run the Malvern software (Ultrasizer MSV) to control the equipment. In Malvern Ultrasizer, selection of an appropriate measurement strategy (selected value of x and frequency range) is an important step. In our study, we wanted to calculate the acoustic contribution of the impurities for which it was important to measure all the combinations of dispersions and impurities with a single measurement strategy. Repeated efforts, finally, led us to a desirable measurement strategy with a frequency range of 1.5–10.0 MHz, transducer position 1 = 0.10 and position 2 = 0.40. In each measurement, a band of 50 different sound waves, as mentioned in Table 2, was passed through the dispersions.

5. Fitness of the measurements

Fig. 1(b) explains that process of determination of particle size depends upon: (i) physical properties of dispersion and (ii) acoustic attenuation spectrum. For right particle sizing, the acoustic attenuation spectrum must be measured with reasonable accuracy. The fitness of an acoustic attenuation spectrum (i.e. attenuation constants vs. frequencies) depends upon accuracy of measurement of attenuation constants. In Malvern Ultrasizer, the fitness of an acoustic attenuation spectrum is measured by a parameter “spectral quality” which depends upon measurement strategy (i.e. selected value of x and frequency range). A spectral quality less than 5 indicates that the attenuation constants have been measured with reasonable accuracy. All measurements reported in this study had a spectral quality less than 5.

6. Experiments

The experiments were designed to sequentially study the effects of three different impurities (magnesium carbonate (insoluble), sodium nitrate (soluble) and sodium polyacrylate (additive)) added to different aqueous dispersions of CaCO_3 (5% m/m, 10% m/m and 20% m/m) in the proportions 5%, 10%, 20% and 30% of the weight (mass) of the disperse medium (CaCO_3). The notation m/m represents mass of CaCO_3 divided by sum of masses of CaCO_3 and water.

In all experiments, dispersions of CaCO_3 in water were prepared directly in the batch measurement cell of the acoustic equipment (Malvern Ultrasizer) at 25 °C and at an agitator speed of 1800 rpm. Mixtures of impurities in dispersions were evaluated at the same conditions of temperature and agitation speed.

The following is a detailed description of one case for elaboration of the procedure. Consider the case of 5% m/m dispersion of CaCO_3 in water that was contaminated with different concentrations of

magnesium carbonate. First, 500.0 ± 0.1 g water was added to the batch measurement cell of the acoustic instrument (Malvern Ultrasizer), followed by 26.32 ± 0.1 g of CaCO_3 . Both were mixed for 20 min in the measurement cell at 25 °C and at 1800 rpm, followed by the first acoustic measurement. After 10 min, another acoustic measurement was taken under the same conditions. The second acoustic measurement was compared with the previous one. The similarity in the results of the two acoustic measurements convinced us of the homogeneity of the dispersion. In the cases where two measurements were not found to be similar, we considered the system to be inhomogeneous. It was further mixed until achievement of homogeneity. After confirmation of homogeneity of the dispersion of CaCO_3 , 1.32 ± 0.1 g of magnesium carbonate (i.e., 5% of the weight of the disperse medium) was added to the dispersion. The system was further mixed until it again became stable and homogeneous. The stability and homogeneity were confirmed by the procedure described above. The concentration of magnesium carbonate was increased to 10% of the weight of the disperse medium by a further addition of 1.32 ± 0.1 g of magnesium carbonate into the system. Acoustic measurement was repeated upon achievement of stable and homogenized state. Similarly, the concentration of magnesium carbonate was increased to 20% of the weight of the disperse medium by adding 2.63 ± 0.1 g magnesium carbonate into the system. Acoustic measurement was repeated during the stable and homogenized phase as mentioned above. The process was repeated by increasing the concentration of magnesium carbonate to 30% of the weight of the disperse medium. The same procedure was adopted in all other experiments.

7. Results and discussions

7.1. Effects of concentration of dispersions on acoustic attenuation spectra

A comparative approach was used to study the effects of concentration of the dispersion of CaCO_3 on acoustic spectra in the absence and in the presence of certain proportions of impurities.

7.1.1. Dispersions without impurities, contaminations or additives

Our experiments revealed that in this case, acoustic attenuation spectra rise with increase in concentration of dispersion, as shown in an example in Fig. 3(a). In order to confirm whether the two factors (i.e., attenuation and concentration of dispersion) are directly proportional, the attenuation values were divided by the respective values of concentrations. All three attenuation spectra were almost superimposed, indicating a proportional relationship between attenuation and concentration of dispersion, as shown in Fig. 3(b).

A slight irregularity in the first portion of the graphs in Fig. 3(a) and (b) may be due to operational incapability (higher signal to noise ratio) of the equipment that usually occurs at the beginning of an acoustic measurement. A possible explanation of the phenomenon of

Table 2
Frequency band employed in the measurements.

No.	Frequency (MHz)								
1	1.5	11	2.2092	21	3.2537	31	4.7921	41	7.0578
2	1.5592	12	2.2964	22	3.3822	32	4.9813	42	7.3364
3	1.6208	13	2.3871	23	3.5157	33	5.1779	43	7.626
4	1.6847	14	2.4813	24	3.6545	34	5.3823	44	7.9271
5	1.7513	15	2.5793	25	3.7987	35	5.5948	45	8.24
6	1.8204	16	2.6811	26	3.9487	36	5.8156	46	8.5653
7	1.8922	17	2.7869	27	4.1046	37	6.0452	47	8.9034
8	1.9669	18	2.8969	28	4.2666	38	6.2839	48	9.2549
9	2.0446	19	3.0113	29	4.435	39	6.5319	49	9.6202
10	2.1253	20	3.1302	30	4.6101	40	6.7898	50	10

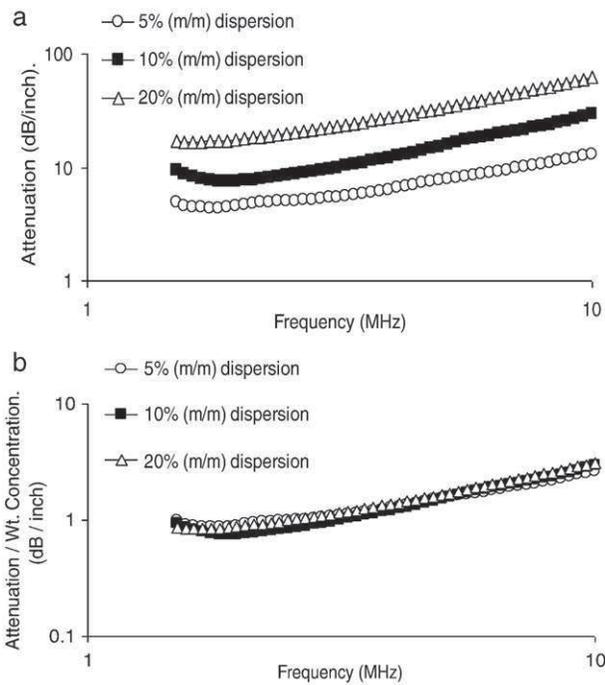


Fig. 3. (a): Attenuation spectra of dispersions of calcium carbonate without addition of impurities. (b): Attenuation spectra of dispersions of calcium carbonate (without impurity) divided by respective weight concentrations.

superimposition, as observed in Fig. 3(b), could be that at a given concentration of the dispersion, each particle (or aggregate) had a certain average size and a certain acoustic attenuation corresponding to that size. Total acoustic attenuation of the dispersion was the sum of acoustic attenuation of all particles (or aggregates). Every increase in the concentration of the dispersion added more particles (or aggregates) of same average size. These particles (or aggregates) retained their size; in other words, they did not interact to form bigger particles (flocs or aggregates). Therefore, the total acoustic attenuation increased proportionally to the number of particles (or aggregates), or in other words, proportionally to the concentration of the dispersion. The situation leads to the following conclusions:

- the particles of the same average size are added to the dispersion,
- the addition of particles (i.e. increase in concentration) does not lead to change in average particle size of the dispersion (absence of beginning of aggregation or de-aggregation etc.) and
- the addition of particles does not cause to start multiple scattering.

7.1.2. Dispersion with impurities, contaminations or additives

As in Section 7.1.1, the effects of concentration of dispersions of CaCO_3 (5%, 10% and 20% m/m) on acoustic spectra were studied by adding the impurity MgCO_3 in the amounts of 5%, 10%, 20% and 30% of the CaCO_3 used. Similar experiments were performed using the impurity sodium polyacrylate, and later on impurity NaNO_3 . Overall, three trends are found:

- Acoustic attenuation spectra rise proportionally with increasing concentrations of the dispersion (the same trend as in Section 7.1.1). Please see Fig. 4(a) and (b) as an example.
- Acoustic attenuation spectra rise, but non-proportionally, with increasing concentrations of the dispersion. Fig. 5(a) and (b) demonstrate this.
- Acoustic attenuation spectra do not show a definable relationship with concentrations of the dispersion. Fig. 6(a) and (b) demonstrate this.

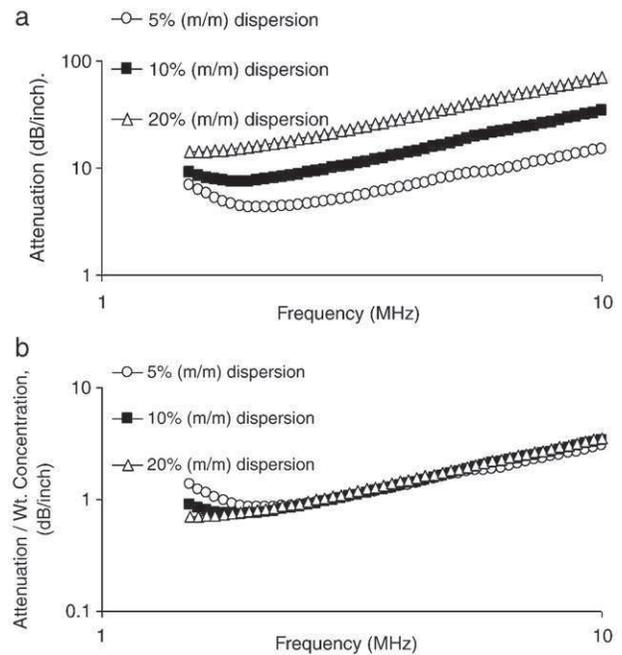


Fig. 4. (a): Acoustic attenuation spectra of dispersions of CaCO_3 at different concentrations. In each case, 10% MgCO_3 of the weight of CaCO_3 is present as impurity. (b): The graph shows attenuation per unit concentration vs. frequency for dispersions of CaCO_3 at different concentrations. In each case, 10% MgCO_3 of the weight of CaCO_3 is present as impurity.

The conditions leading to the three trends are summarized as follows.

Trend 1 The dispersions of CaCO_3 follow trend 1 in the presence of the impurity magnesium carbonate (in all proportions: 5%, 10%, 20% and 30% by weight of the disperse medium). Similarly,

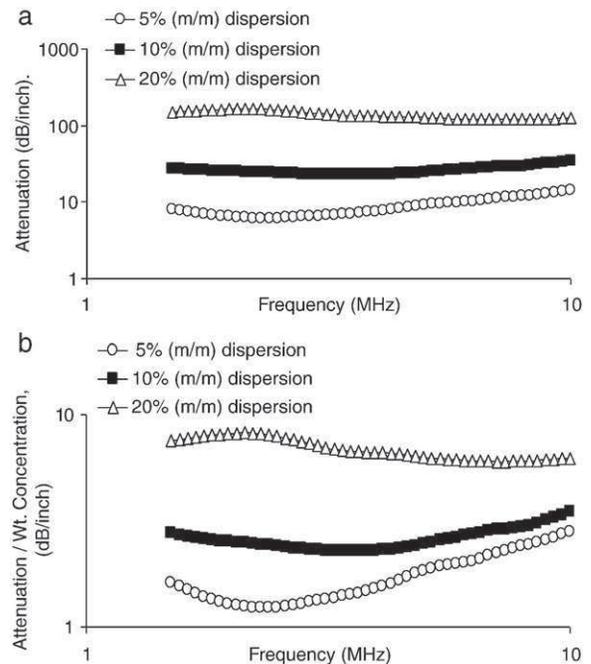


Fig. 5. (a): Acoustic attenuation spectra of dispersions of CaCO_3 at different concentrations. In each case, 30% NaNO_3 of the weight of CaCO_3 is present as impurity. (b): The graph shows attenuation per unit concentration vs. frequency for dispersions of CaCO_3 at different concentrations. In each case, 30% NaNO_3 of the weight of CaCO_3 is present as impurity.

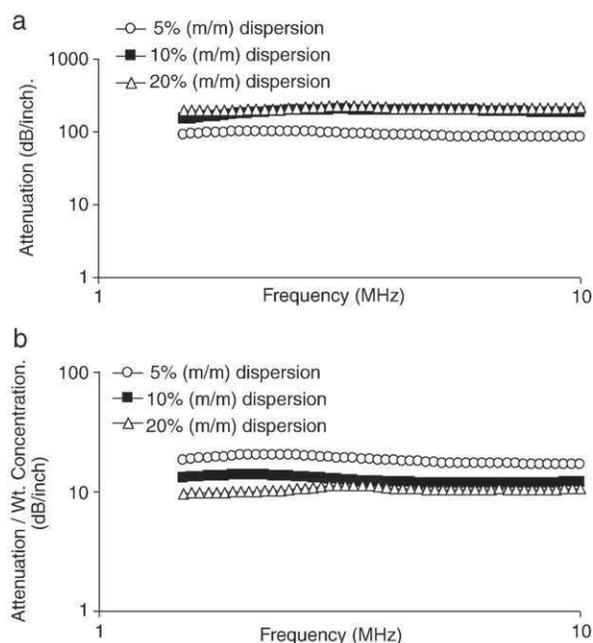


Fig. 6. (a): Acoustic attenuation spectra of dispersions of CaCO_3 at different concentrations. In each case, 30% sodium polyacrylate of the weight of CaCO_3 is present as impurity. (b): The graph shows attenuation per unit concentration vs. frequency for dispersions of CaCO_3 at different concentrations. In each case, 30% sodium polyacrylate of the weight of CaCO_3 is present as impurity.

the dispersions of CaCO_3 follow the trend 1 in the presence of impurity NaNO_3 (only in proportions of 5% and 10%). Trend 1 is similar to the trend observed in Section 7.1.1; hence, the conclusions drawn in Section 7.1.1 may be equally applied to all combinations of the dispersions and impurities falling under this trend. The approach opens a way to study dispersions with impurities using acoustic attenuation spectroscopy.

- Trend 2** The dispersions of CaCO_3 follow trend 2 in the presence of the impurity NaNO_3 (in proportions of 20% and 30%). Similarly, trend 2 is observed in case of the impurity sodium polyacrylate (in proportions of 5%, 10% and 20%). Trend 2 is characterized by a non-proportional increase in acoustic spectra with concentration. There are many possible explanations for this tendency, such as the beginning of formation of aggregates or flocs between particles, between particles and the impurity or between the impurity, or the beginning of multiple scattering, etc.
- Trend 3** This was observed in presence of the impurity sodium polyacrylate in the proportion of 30%. The trend was found to be complex in nature. The likely causes could be de-aggregation or multiple scattering that appear at a certain combination of concentrations of dispersion and dispersant.

7.2. Effects of concentration of impurities on acoustic attenuation spectra

This section analyzes the effect of an increase in concentration of an impurity on attenuation spectra of a dispersion at a given concentration.

Table 3
Conditions in which the effect of an impurity may be predicted.

Impurity	5% (m/m) dispersion of CaCO_3	10% (m/m) dispersion of CaCO_3	20% (m/m) dispersion of CaCO_3
MaCO_3	Predictable effect	Predictable effect	Predictable effect
NaNO_3	Predictable effect	Predictable effect	Unpredictable effect
Sodium polyacrylate	Unpredictable effect	Unpredictable effect	Unpredictable effect

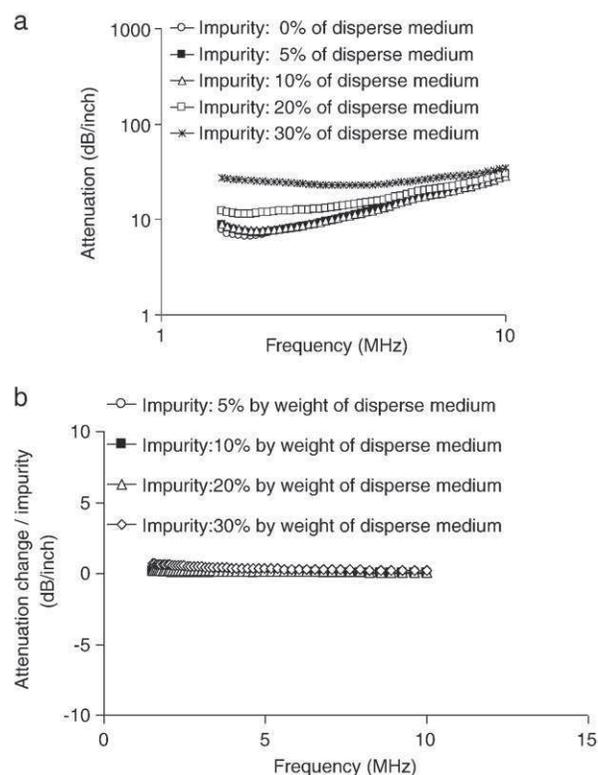


Fig. 7. (a): Effects of concentration of impurity of NaNO_3 on 10% (m/m) dispersion of CaCO_3 . (b): Effects of concentration of impurity of NaNO_3 on 10% (m/m) dispersion of CaCO_3 .

Several combinations were studied. It was found that under some conditions, the rise of attenuation spectra was proportional to the amount of the added impurity. Fig. 7(a) shows the effects of different concentrations (proportions) of the impurity NaNO_3 on a 10% (m/m) dispersion of CaCO_3 . In order to study the relationship between the change in spectra and the concentration of the impurity, the following method was adopted. In all experiments, attenuation values of a pure dispersion were subtracted from attenuation values of the relevant impure dispersion to obtain the attenuation change due to the total quantity (%age) of impurity. This value was divided by the total quantity (%age) of impurity, and the results were plotted against corresponding frequencies to get attenuation spectra based on unit impurity. If attenuation spectra per unit impurity superimpose, then we can quantify the effects contributed by that impurity to the total acoustic attenuation spectra under given conditions. Fig. 7(b) presents an example.

The method was applied to the entire series of the experiments in order to determine the conditions at which the effects of impurities can be predicted. Table 3 presents the results.

8. Conclusions

The addition of an impurity into a dispersion may or may not change original particle (or aggregate) size of the disperse medium, depending on the conditions. With the help of acoustic attenuation spectroscopy, it is possible to predict the conditions (i – concentration of dispersion;

ii – proportion of impurity) at which the particle (or aggregate) size of the disperse medium is not influenced by the addition of the impurity. In such cases, we can calculate the acoustic contribution of the impurity in the total acoustic attenuation spectrum of the dispersion. This contribution may be subtracted from the total attenuation spectrum of the dispersion in order to get more realistic information of the particle size of the disperse medium. The cases in which the size of the disperse medium seems to be influenced by the addition of certain quantities of impurities may be explained based on the existence of multiple scattering (a complex phenomenon that disturbs attenuation in acoustic measurement), interaction between particles of the disperse medium or interaction between particles of the impurity and those of the disperse medium. In such cases, it becomes more difficult to get exact particle size information of the disperse medium by using acoustic attenuation spectroscopy. In the study, higher quantities of impurities were considered in order to emphasize their effect on acoustic response. However, under usual conditions, when the proportion of an impurity is less than 20% of the weight of the disperse medium, it may be assumed that presence of the impurities in a dispersion may lead to predictable effects, except for the impurities (such as sodium polyacrylate) that may influence electrostatic and steric stabilization of the dispersions.

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