

Acoustic Spectroscopy for Concentrated Polydisperse Colloids with Low Density Contrast

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The mechanisms of sound attenuation are different in dispersions with low and high density contrast. "Viscous losses" are dominant in high density contrast dispersions whereas "thermal losses" predominate in dispersions with low density contrast. The rutile dispersion is chosen as an instance of the high density contrast system. The dispersion of the neoprene latex is an instance of the low density contrast system. The dilution experiment performed with both systems shows that the role of the particle-particle interaction is quite different in these two dispersions. The measured spectra show that attenuation remains a linear function of the volume fraction in the latex dispersion even at 30 vol %. At the same time, attenuation exhibits a nonlinear dependence on the volume fraction for the rutile dispersion even at 10 vol %. This difference means that particle-particle interaction contributes more to the "viscous losses" than to the "thermal losses". We associate this effect with the difference between "viscous depth" and "thermal depth". These parameters characterize the penetration of the shear wave or thermal wave into the liquid. The observed insensitivity of the thermal losses to the particle-particle interaction supports the application of dilute case theory to calculate the particle size distribution in the concentrated (up to 30 vol %) emulsions and latices.

Introduction

The biggest advantage of acoustic spectroscopy over other spectroscopic methods is the ability to characterize intact concentrated dispersed systems. The frequency dependence of the attenuation coefficient is the normal experimental output of the acoustics. This frequency spectrum can be converted to the particle size distribution. There are many successful applications of this technique.⁵⁻⁷

The above-mentioned conversion procedure requires a theory for sound propagation through the dispersed system. This theory has been created by Epstein and Carhart¹ and Allegra and Hawley² (ECAH theory). The validity of the theory is restricted by neglecting the particle-particle interaction. As a result the ECAH theory is strictly limited to dilute systems.

The experimental dilution test performed with the rutile dispersion in our previous work³ confirms that ECAH theory is not adequate for even moderate volume fractions (above 10 vol %) when the density contrast is sufficiently large. It has been shown that attenuation is a nonlinear function of the weight fraction (wfr) (Figure 1), whereas ECAH theory predicts a linear relationship between these parameters.

At the same time there are several works^{2,4-6} showing the validity of the ECAH theory even for concentrated

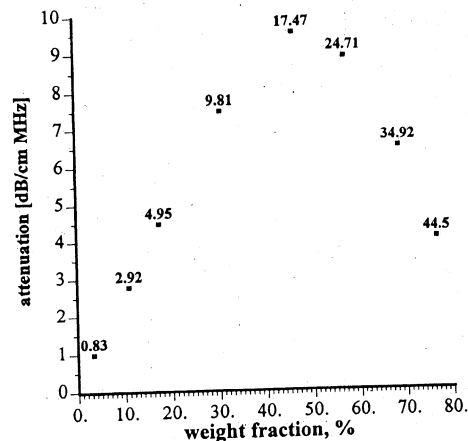


Figure 1. Dependence of the attenuation in the rutile dispersion (rutile R-746 by DuPont) at the frequency 15 MHz on the dispersed system weight fraction. Corresponding volume fractions in percent are shown as the data point labels.

systems (up to 40 vol %) when the density contrast is low. For some unknown reasons particle-particle interaction does not show up in the concentrated latices^{2,6} and emulsions.^{4,5}

The different sensitivity of the sound attenuation to the particle-particle interaction in the systems with high and low density contrast can be associated with the differences in the mechanisms of the sound attenuation in these systems. The "viscous losses" mechanism dominates in the high density contrast systems. The "thermal

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Table 1. Physical Properties of the Neoprene Latex, Rutile and Water at 25 °C^a

	neoprene, initial ^a	neoprene, adjusted ^b	rutile	water
density (g cm ⁻³) { ρ_p }	1.23	1.23	4.06	0.997
sound speed (m s ⁻¹) { c_p }	2380	2380	6000	1497
specific heat (J g ⁻¹ K ⁻¹) { C_p }	0.521	0.521	unknown	4.179
thermal conduction (mW cm ⁻¹ K ⁻¹) { τ_p }	2	2	unknown	6.1
thermal expansion (K ⁻¹ 10 ⁴) { β_p }	6	5.3	unknown	2.07
intrinsic losses (dB cm ⁻¹ MHz ⁻²) { α_p/f^2 }	unknown	0.15	0	0.00217

^a Initial values for neoprene were taken from "The Neoprenes".⁹ ^b Adjusted values for neoprene are the result of acoustic characterization.

losses" mechanism causes the sound attenuation in the low density contrast systems.

The theory of "viscous losses" has been modified in our previous paper³ by implementing particle-particle hydrodynamic interactions. The next logical step of our attempts to expand acoustic spectroscopy to the concentrated systems is the analysis of the thermodynamic particle-particle interaction which contributes to the "thermal losses". This analysis is the subject of this paper.

We start with the experimental dilution test performed with the neoprene latex. This test shows that the "thermal losses" mechanism is indeed much less sensitive to particle-particle interaction than "viscous losses". Then we suggest a hypothesis explaining this peculiarity of the sound attenuation in the latices and emulsions.

Preparation of the Neoprene Latex Samples

The low-density dispersion being considered in this paper is a Neoprene Latex 735A designed by DuPont as a wet-end additive to fibrous slurries. The weight fraction of the latex in the initial dispersion is 42.8 wt %, which is equivalent to the volume fraction 37.3 vol %. The pH value at 25 °C is 11.5. The physical properties of the neoprene (slow crystallizing polychloroprene homopolymer) were measured in the DuPont laboratories many years ago. These data (Table 1) are summarized in the monograph "The Neoprenes".⁹

The solution for the dilution is distilled water with the pH adjusted to 11.5 using 1 N potassium hydroxide. The samples with various weight fractions (1.4, 4, 6.6, 13, 19.4, 25.6, 31.6, and 37.5 wt %) were prepared by adding the diluting solution to the initial neoprene latex.

Measurement Technique

The particle size distribution (PSD) of the neoprene latex has been measured using a CHDF Matec Applied Science Instrument employing hydrodynamic chromatography. This instrument characterizes the PSD in the extremely dilute system by pumping it through the capillary. The sample elution time was 300.0, and the pump pressure was 514. This measurement was done at DuPont.

The attenuation spectra of the neoprene latex were measured with Acoustophor PK-8000. This instrument operates in the frequency range from 1 to 100 MHz with an at least 125 mL sample. The variable gap technique makes it possible to exclude calibration procedures. There is no restriction on the volume fraction, but the sample must be fluid in order for the peristaltic pump to pump the sample through the measuring chamber.

The neoprene latex is fluid even at the highest volume fraction. However, it is very shear sensitive when very dilute. Latex clogs pipes while being pumped through the system. Therefore, modified the sample holder in order to avoid pipe clogging. A special flexible rubber container was inserted between the transducer and the receiver of the sound, and latex was poured into this container. The container prevents the sample from contacting the walls of the measuring chamber. At the same time the container

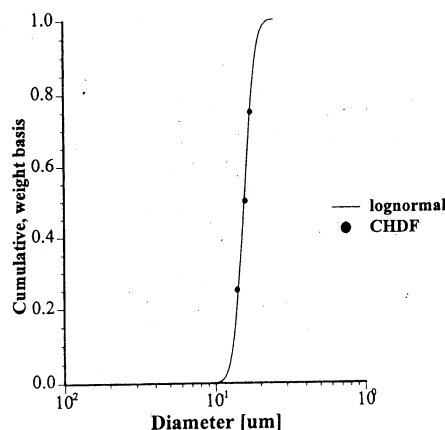


Figure 2. Cumulative particle size distribution (weight basis) of neoprene latex measured with hydrodynamic chromatography (CHDF Matec Applied Science) and approximated using a log-normal assumption. The median size of the log-normal distribution is 0.16 μ m, and the standard deviation is 0.06.

Table 2. Moments of the Neoprene Latex Particle Size Distribution Measured with a CHDF Matec Applied Science Hydrodynamic Chromatograph

PSD parameter	number basis	weight basis
median diameter (nm)	137	158.7
standard deviation (nm)	39.7	23.9
25% diameter (nm)	82.6	145
75% diameter (nm)	158.7	170.4

contribution to the sound attenuation is not significant because the container walls are very thin. This modification of the sample holder allows us to measure attenuation spectra for all latex samples.

Results

The moments of the particle size distribution measured with hydrodynamic chromatography are shown in Table 2.

Interpretation of the attenuation spectra requires information about the entire particle size spectra. We use a log-normal approximation of the measured PSD. Figure 2 shows that a log-normal particle size distribution with the median size 0.16 μ m and the standard deviation 6% gives a perfect fit to the three experimentally measured points from hydrodynamic chromatography. This log-normal PSD will be used to calculate theoretical attenuation spectra.

The attenuation spectra measured with Acoustophor PK-8000 are shown in Figure 3 ($w_{fr} > 20\%$) and Figure 4 ($w_{fr} < 20\%$). Each sample was measured three times. As a result there are three curves for each weight fraction in Figure 3. It is seen that the reproducibility for the concentrated samples of Figure 3 is very good in the frequency range between 2 and 60 MHz. The reproducibility for the moderate weight fractions 18.12% and 12.2% is satisfying at the frequencies from 4 to 50 MHz. Dilute samples with weight fractions below 10% show good

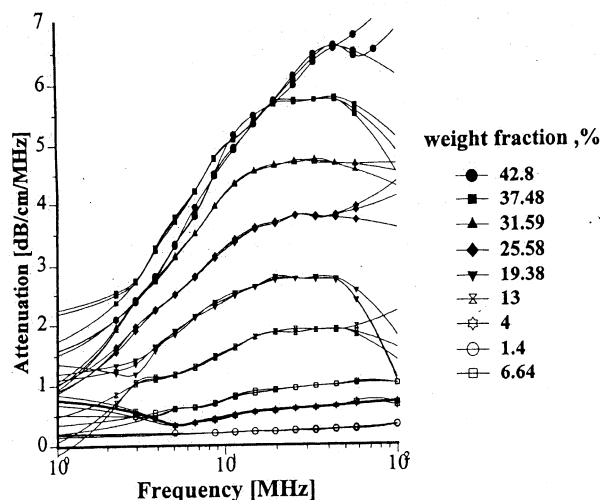


Figure 3. Attenuation spectra for neoprene latex.

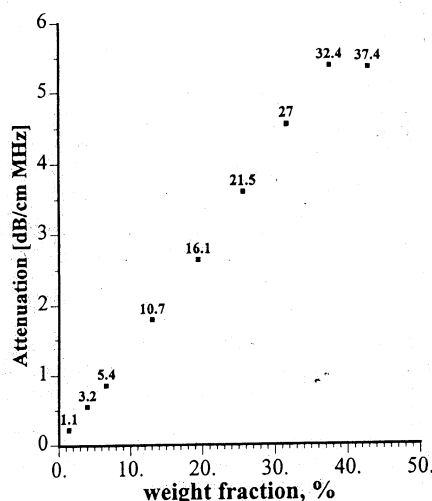


Figure 4. Dependence of the attenuation in neoprene latex at the frequency 15 MHz on the dispersed system weight fraction. Corresponding volume fractions in percent are shown as the data points labels

reproducibility for the frequencies from 5 to 100 MHz. Our experience with the rutile dispersion described in ref 3 indicates that all frequency ranges are wide enough to get reliable information about the PSD.

The attenuation coefficient at the frequency 15 MHz exhibits a linear dependence on the weight fraction up to 37.5 wt %, as shown in Figure 4. This peculiarity of the neoprene latex attenuation spectra is the same as that observed by Allegra and Hawley² for the polystyrene latex. This linearity allows us to assume that dilute case ECAH theory is valid sometimes for unknown systems yet reasonable even for concentrated systems.

Theoretical Background

Loss Mechanisms. There are six known loss mechanisms of the sound interaction with a dispersed system: (1) viscous; (2) thermal; (3) scattering; (4) intrinsic; (5) structural; and (6) electrokinetic.

(1) The viscous losses of the acoustic energy occur due to the shear wave generated by the particle oscillating in the acoustic pressure field. These shear waves appear because of the difference in the densities of the particles and the medium. This density contrast causes the particle motion with respect to the medium. As a result the liquid layers in the particle vicinity slide relative to each other. This sliding nonstationary motion of the liquid near the particle is referred to as "shear wave".

(2) The reason for the thermal losses is the temperature gradients generated near the particle surface. These temperature gradients are due to the thermodynamic coupling between pressure and temperature.

(3) The mechanism of the scattering losses is quite different for the viscous and thermal losses. The acoustic scattering mechanism is similar to light scattering. Acoustic scattering does not produce the dissipation of acoustic energy. Particles simply redirect the acoustic part of the energy flow, and as a result this portion of the sound does not reach the sound transducer.

(4) The intrinsic losses of the acoustic energy occur due to the interaction of the sound wave with the materials of the particles and the medium as homogeneous phases.

(5) The oscillation of the network of interparticle links in the structured dispersed system causes structural losses. Thus, this mechanism is specific for the structured systems.

(6) Oscillation of the charged particles in the acoustic field leads to the generation of an alternating electrical field and, consequently, an alternating electric current. As a result, a part of the acoustic energy transforms to electric energy and then irreversibly to heat.

Only the first four loss mechanisms (viscous, thermal, scattering, and intrinsic) make a significant contribution to the overall attenuation spectra in most cases. Structural losses are significant only in structured systems, which requires a quite different theoretical framework. Finally, the contribution of electrokinetic losses to the total sound attenuation is almost always negligibly small⁷ and will be neglected.

The general theory taking into account all four main mechanisms exists only for dilute systems when particle-particle interaction is not important. It is called the ECAH theory.

Only three mechanisms (viscous, thermal, and intrinsic) are important for neoprene latex. The contribution of the scattering losses is negligibly small because of the small size of the neoprene particles. It is known²⁻⁴ that scattering losses become important only for large particles with diameters above 1 μm and high frequencies above 50 MHz.

The viscous and intrinsic losses were calculated using the theory developed in our previous paper³ (preceding paper in this issue). This theory takes into account hydrodynamic particle-particle interaction and is valid for concentrated systems.

The contribution of the thermal losses will be calculated using ECAH theory.

Existing ECAH Theory. The dilute case theory for the four most important mechanisms (viscous, thermal, scattering, and intrinsic) was developed by Epstein and Carhart¹ and Allegra and Hawley.² It is the so-called ECAH theory. This theory describes the attenuation for the dilute monodisperse system of spherical particles.

The term "monodisperse" is normally used to suggest that all of the particles are assumed to have the same diameter. Extensions of the ECAH theory to include polydispersity have typically assumed a simple linear superposition of the attenuation for each size fraction.

The term "spherical" is used to denote that all calculations are performed assuming that each particle can be adequately represented as a sphere. We use the same model for the particles.

Most importantly, the term "dilute" is used to indicate that there is no consideration of particle-particle interactions. This fundamental limitation normally restricts the application of the resultant theory to dispersions with a volume fraction less than a few volume percent. However, there is some evidence that the ECAH theory, in some

very specific situations, does nevertheless provide a correct interpretation of experimental data, even for volume fractions as large as 30%.

An early demonstration of the ability of the dilute ECAH theory to describe some concentrates was in fact provided by Allegra and Hawley. They observed almost perfect correlation between experiment and dilute case ECAH theory for several systems: a 20 vol % toluene emulsion; a 10 vol % hexadecane emulsion; and a 10 vol % polystyrene latex. Similar work with emulsions by McClements^{4,5} has provided similar results. The recent work by Holmes, Challis, and Wedlock⁶ shows the good agreement between ECAH theory and experiment even for a 30 vol % polystyrene latex.

It is important to note that the surprising validity of the dilute ECAH theory for moderately concentrated systems has only been demonstrated in systems where the "thermal losses" were dominant. The neoprene latex considered in this paper is an example of that kind of dispersion. Therefore, the "thermal losses" contribution to the total measured attenuation will be calculated using ECAH theory.

Long-Wavelength Requirement. We would like to keep the theory as general as possible. Nevertheless, one important simplification of the theory will be employed, the so-called "long-wave requirement",² which requires the wavelength of the sound wave λ to be larger than the particle radius a . This "long-wave requirement" restricts the sound frequency to be above some certain critical frequency ω_{lw} , which is reciprocally proportional to the particle size according to the equation

$$\omega_{lw} = \frac{2\pi c_0}{a} \quad (1)$$

where c_0 is the sound speed in the medium.

The long-wave requirement is valid for the considered neoprene latex within the entire frequency range from 1 to 100 MHz because of the small particle size.

Invoking this long-wave requirement also permits us to simplify the analysis by neglecting scattering losses.³

Thermal Losses. By restricting frequency and particle size with the long-wave requirement, we can use the simpler explicit expression for the thermal losses obtained initially by Isakovich⁸ and confirmed later by Epstein and Carhart¹ and Allegra and Hawley.²

$$\alpha_{the} = \frac{3\varphi T c_0 \pi_0 \tau_0}{2a^2} \Delta^2 \text{Re } H \quad (2)$$

where

$$\Delta = \frac{\beta_0}{\rho_0 X_\pi} - \frac{\beta_\pi}{\rho_\pi X_\pi} \quad (3)$$

$$H = \frac{1}{1 - iz_0} - \frac{\tau_0 \tanh z_p}{\tau_p \tanh z_p} \quad (4)$$

$$z = (1 + i)a\sqrt{\frac{\omega\rho C_p}{2\tau}}$$

where φ is the volume fraction, T is the absolute temperature, ω is the angular frequency, c is the sound speed, ρ is the density, τ is the thermal conduction, β is the thermal expansion, and C_p is the specific heat at constant pressure. The index 0 corresponds to the medium whereas the index p indicates particles.

Intrinsic Losses. Viscous, thermal, and scattering losses exist due to the heterogeneity of the dispersed system. There are no mechanisms like those in the homogeneous system. However, it does not mean that the sound wave does not attenuate in pure water, for instance. It does, but this attenuation is very low, about 20 dB/cm at 100 MHz. Same "partial attenuation" α_0 exists for any material. This attenuation α_0 is frequency dependent. It is approximately proportional to the frequency squared.

These "partial attenuations" contribute to the total attenuation not only in the homogeneous system but also in the heterogeneous one as well. The contribution of each phase "partial attenuation" is proportional to the phase volume fraction. The total contribution is referred to as "intrinsic losses" following Allegra and Hawley² or "lossless scatters" following McClements.⁴

The expression for the "intrinsic losses" α_{int} has been derived by Allegra and Hawley for the dilute case. The equation for the complex wavenumber given by McClements⁴ gives the way to generalize the "intrinsic losses" expression for the concentrated system. Extracting imaginary part of the complex wavenumber, we obtain the following expression:

$$\alpha_{int} = \frac{(1 - \varphi)\alpha_0\rho_\pi\chi_\pi + \varphi\alpha_\pi\rho_0\chi_0}{\sqrt{(1 - \varphi)\rho_\pi^2\chi_\pi^2 + \varphi\rho_\pi\rho_0\chi_0^2}} \sqrt{\frac{\rho^*}{\rho_0}} \quad (5)$$

where α_0 and α_p are "partial attenuations" in the dispersion medium and the dispersed phase.

This expression for the "intrinsic attenuation" transfers to the Allegra-Hawley expression for the low volume fraction. The corresponding real part of the complex wavenumber gives the Wood expression for the sound speed in the dispersed system.

Discussion

The experimental data collected with the neoprene latex give an opportunity to check the validity of the ECAH theory when thermal losses are the dominant mechanism of the sound attenuation. In order to calculate the theoretical attenuation spectra, one should have information about the particle size, the thermodynamic properties of the dispersed phase and dispersion medium materials, as well as the "partial intrinsic attenuations". Fortunately, we have almost everything we need in this particular case. The approximate thermodynamic properties of neoprene are known from the independent investigation performed at DuPont (first column in Table 1). The neoprene PSD is also provided by DuPont (Table 2). The thermodynamic properties and intrinsic attenuation of water are known from the literature.⁴ Therefore, only the neoprene intrinsic attenuation is unknown. We assume from the beginning that this parameter is zero.

The theoretical attenuation is calculated as the sum of the thermal, intrinsic, and viscous losses. Thermal losses are calculated according to expressions 2-4. Expression 5 gives the intrinsic losses. Viscous losses are calculated following our previous work.³ The relative contribution of each mechanism is shown in Figure 5 for the volume fraction 10 vol %. It is seen that thermal losses are definitely dominant.

The results of the calculations based on this assumption are shown in Figure 6. It is seen that theory describes in general the peculiarities of the experimental spectra. However, there are obvious discrepancies between theory and experiment. It looks like theory overestimates attenuation around 10 MHz and underestimates it at high frequency.

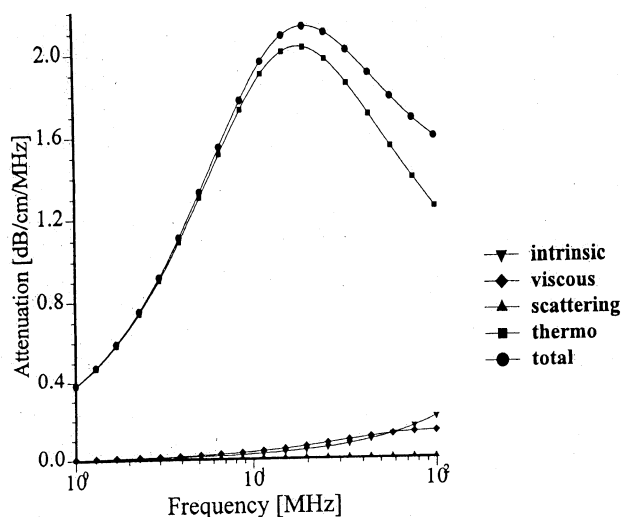


Figure 5. Theoretical attenuation spectra for the various mechanisms of acoustic energy losses. The neoprene latex properties correspond to the first column of Table 1. The volume fraction is 10 vol %.

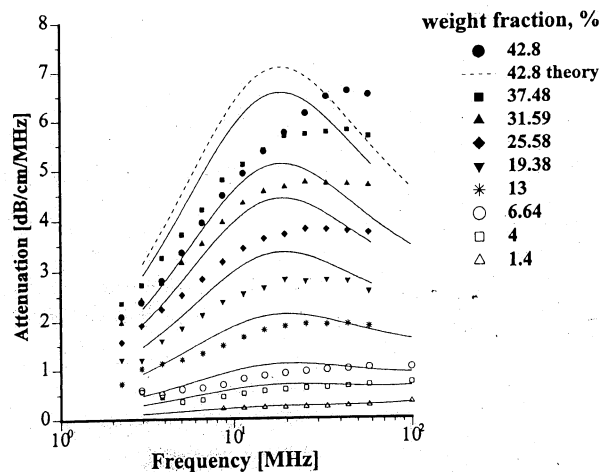


Figure 6. Experimental and theoretical attenuation spectra for the weight fractions indicated in the legend. The neoprene latex particle size distribution log-normal is shown in Figure 2. The neoprene latex properties correspond to the first column of Table 1.

The deviations at high frequencies are associated with our assumption about zero neoprene intrinsic attenuation. We can find this parameter using one of the attenuation spectra. All other attenuation spectra would be the test for this procedure. In addition to the neoprene intrinsic attenuation, we can adjust the neoprene thermodynamic properties as well. There is definitely some uncertainty in these parameter values. It allows us to eliminate the deviations at the intermediate frequencies.

All these adjustments were done with the 13 wt % sample. The new adjusted properties of neoprene are shown in the second column of Table 1. It is seen that the adjustment of the thermodynamic properties is small. Nevertheless, the correlation between theory and experiment for the new values of the neoprene properties is much better for all volume fractions (see Figure 7). The theoretical fit is very good up to 37.5 wt % (32.4 vol %). This improvement is related mostly to the increase of the intrinsic losses. The changes in the relative contribution of the various mechanisms corresponding to the new neoprene properties are shown in Figure 8.

Thus, we can conclude that ECAH theory is able to provide a very good theoretical fit to the experimental measured attenuation spectra even up to 32.4 vol %. This

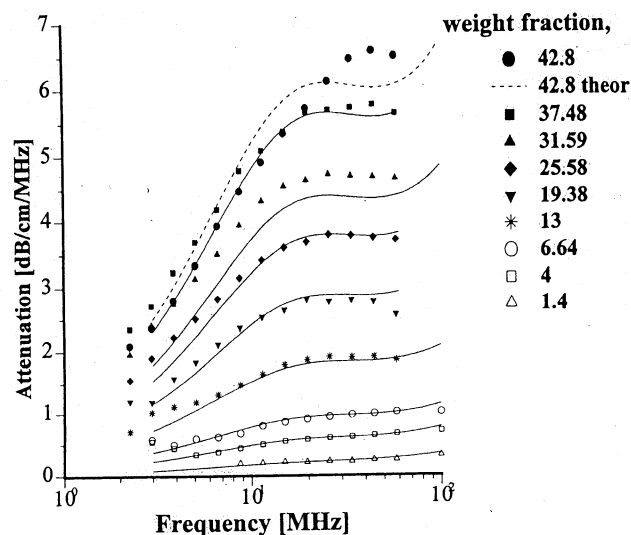


Figure 7. Experimental and theoretical attenuation spectra for the weight fractions indicated in the legend. The neoprene latex particle size distribution log-normal is shown in Figure 2. The neoprene latex properties are adjusted according to the second column of Table 1.

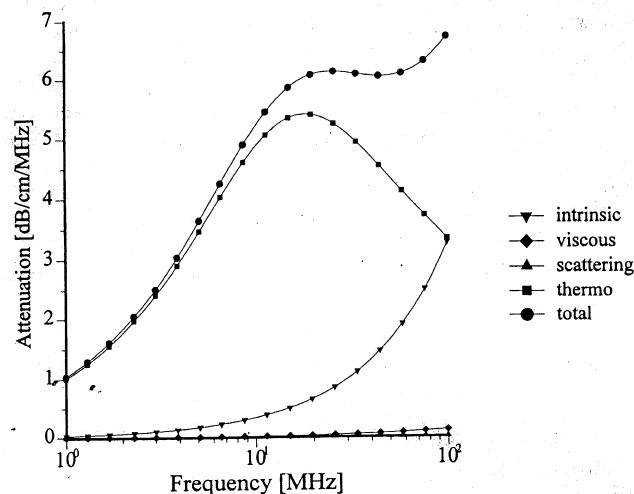


Figure 8. Theoretical attenuation spectra for the various mechanisms of acoustic energy losses. The neoprene latex properties are adjusted according to the second column of Table 1. The volume fraction is 10 vol %.

statement gives rise to the question why particle-particle interaction does not show up for such concentrated systems.

We believe that the difference between the "viscous depth" and the "thermal depth" gives an answer to the formulated question. These parameters characterize the penetration of the shear wave and the thermal wave, respectively, into the liquid. A particle oscillating in a sound wave generates these waves, which damp exponentially in the particle vicinity. The distance for the shear wave amplitude to decay by a factor e is the "viscous depth" δ_v . The corresponding distance for the thermal wave is the "thermal depth" δ_t . The following expressions give these parameter values in the dilute systems:

$$\delta_v = \sqrt{\frac{2\nu}{\omega}} \quad (6)$$

$$\delta_t = \sqrt{\frac{2\tau_0}{\omega\rho_0 C_p}} \quad (7)$$

where ν is the kinematic viscosity.

The relationship between δ_v and δ_t has been considered before. For instance, McClements plots "thermal depth" and "viscous depth" versus frequency.⁴ It is easy to show using Table 1 data that the "viscous depth" is 2.6 times more than the "thermal depth" in aqueous dispersions. As a result, particle viscous layers overlap at a lower volume fraction than particle thermal layers. The overlap of the boundary layers is the measure of the corresponding particle-particle interaction. There is no particle interaction when corresponding boundary layers do not overlap.

Thus, an increase of the volume fraction for the given frequency first leads to the overlap of the viscous layers because they extend further into the liquid. Thermal layers overlap at higher volume fractions. This means that particle hydrodynamic interaction becomes more important at lower volume fractions than particle thermodynamic interaction.

The 2.6 times difference between δ_v and δ_t leads to the big difference in the volume fractions corresponding to the beginning of the boundary layers' overlap. The dilute case theory is valid for the volume fractions smaller than these critical volume fractions φ_v and φ_t . These critical volume fractions φ_v and φ_t are functions of the frequency and particle size. We can conventionally define these parameters from the condition that the shortest distance between particle surfaces equals double δ_v or double δ_t . This conventional definition gives the following expression for the ratio of the critical volume fractions of aqueous dispersions:

$$\frac{\varphi_v}{\varphi_t} = \left(\frac{a\sqrt{\pi f} + 2.6^{-1}}{a\sqrt{\pi f} + 1} \right)^3 \quad (8)$$

where a is in microns and f is in megahertz.

The ratio of the critical volume fractions depends on the frequency. For instance for neoprene latex, the critical "thermal" volume fraction is 10 times higher than the "viscous" one for 1 MHz and only 3 times higher for 100 MHz.

Conclusions

Attenuation of sound in dispersions with low density contrast is associated primarily with temperature gradients in the vicinity of the particle interface. That part of the acoustic energy transforms to heat within these boundary thermodynamic layers. This describes the "thermal losses" mechanism of the sound attenuation, which is dominant in the low density contrast systems like latices and emulsions.

Attenuation spectra measured for neoprene latex show a linear dependence on volume fraction up to 30 vol %. This means that the "thermal losses" mechanism is not sensitive to particle-particle thermodynamic interaction. This fortunate peculiarity of the "thermal losses" mechanism of sound attenuation makes it possible to apply a simple dilute case theory to convert attenuation spectra into the particle size distribution. This conversion requires information about thermodynamic properties of the dispersed phase. This information is often unavailable. Acoustic spectroscopy gives an opportunity to adjust these properties when the PSD is known. It is shown that the set of thermodynamic properties adjusted for one neoprene latex attenuation spectrum (10 vol %) provides a perfect theoretical fit for the nine other attenuation spectra with different volume fractions. It confirms also the validity of the dilute case ECAH (Epstein-Carhart-Allegra-Hawley) theory for the concentrated systems with low density contrast.

The weakness of the thermodynamic particle-particle interaction means that there is no overlap of the thermodynamic layers at high volume fraction. The overlap does not occur because the thickness of the thermodynamic layer is much smaller than the thickness of the viscous boundary layer. Therefore, thermodynamic layers overlap at much higher volume fractions than viscous layers.

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