Ultrasonic method to characterize shear wave propagation in micellar fluids

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Viscoelastic micellar fluid characteristics have been measured with mechanically generated shear waves and showed good agreement to shear oscillatory methods. In this paper, shear waves in wormlike micellar fluids using ultrasound were successfully generated and detected. Micellar fluids of different concentrations (100, 200, 300, and 400 mM) were studied with ultrasound-based and conventional rheology methods. The measured micellar fluid complex modulus from oscillatory shear tests between 0.001 and 15.91 Hz was characterized with an extended Maxwell fluid model. The elastic and viscous parameters found using rheological testing were used to estimate shear wave phase velocity over a frequency range from 100 to 500 Hz, and compared to shear wave velocity measured with ultrasound-based methods with a mean absolute error 0.02 m/s. The shear wave frequency content was studied and an increase in shear wave center frequency was found as a function of micellar fluid concentration. Moreover, the bias found in the shear wave group velocity with respect to rheological measurement is attributed to the micellar fluid viscous component. Finally, the shear wave phase velocity evaluated at the center frequency agreed well with the rheological measurements. © 2016 Acoustical Society of America.

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I. INTRODUCTION

Wormlike micellar fluids have received significant attention from researchers in the past decade; their structure, rheology, and applications have been addressed in different articles. Micelles are self-assembling large aggregates that are formed spontaneously from the interaction of surfactant molecules, amphiphilic molecules with a hydrophilic head and a hydrophobic tail, with water and salt.¹ The wormlike micelle is a specific morphology of micelles, as shown in Fig. 1, formed by surfactants with small head groups and/or single long tail end.¹ Although the addition of simple salts such as sodium salicylate (NaSAL) results, generally, in the growth of aggregates,² the length of the wormlike micelles depends on temperature, concentration, size of the surfactant head group, and salinity of the solution.¹

One of the main interests of wormlike micelles exists in their remarkable rheological properties; the wormlike micelles are similar to polymers that exhibit viscoelastic characteristics.²

Experimental evaluation of wormlike micellar fluids' viscoelasticity has been focused on low frequency characterization with conventional rheometers under an oscillatory shear test.² Although a linear viscoelastic Maxwell model often quite accurately represents the wormlike micellar fluid

viscoelastic properties under conventional oscillatory shear test, the micellar fluid loss modulus deviates from the Maxwell model at higher frequencies due to what is thought to be the breakage and reformation process of the micelles undergoing dynamic equilibrium.³ Scaling arguments to characterize micellar fluid dynamic properties at high frequency have been proposed,¹ but a more accurate interpretation of the micellar fluid loss modulus is described with the extended Maxwell model.⁴

In addition to conventional rheology methods, material viscoelastic properties can be quantified by studying shear wave propagation. Shear wave propagation methods are widely used in the medical imaging field because their capabilities of noninvasive estimation of tissue pathology by means of characterization of tissue mechanical properties, a field called elasticity imaging.⁵ Shear wave methods are based on measuring the vertical, or axial, particle motion from shear strain (angular deformation) that is involved in the propagation of transverse waves.⁶ The first proposed elasticity imaging methods either excited tissue externally, as in magnetic resonance elastography (MRE)⁷ and transient elastography (TE),⁸ or use focused ultrasound to produce acoustic radiation force to push tissue as in acoustic radiation force impulse (ARFI) imaging,⁹ shear wave elasticity imaging (SWEI),¹⁰ and supersonic shear imaging (SSI).¹¹ The most common mechanical parameter used in elasticity imaging is the shear modulus, which can vary over six orders of

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FIG. 1. Surfactant solution morphologies including spherical micelles, bilayers, wormlike micelles and entangled wormlike micelles (adapted from Ref. 1).

magnitude within biological tissues.¹⁰ Because biological tissues exhibit viscoelastic behavior, elasticity imaging methods have been developed to study both shear modulus and shear viscosity.^{12,13} The shear wave based elasticity imaging methods capable of characterizing viscoelasticity are usually validated with tissue mimicking materials; however, there are limited materials that exhibit both viscoelastic and acoustic properties similar to biological tissue. Micellar fluids offer a unique material to validate shear wave-based elasticity imaging methods because their acoustic properties are similar to biological tissue and their viscoelastic properties can easily be altered by varying concentration.

Shear wave propagation in wormlike micellar fluids have been reported. With a mechanical actuator and an optical system, Gladden *et al.*^{14,15} demonstrated the generation and detection of shear waves in wormlike micellar fluids with concentrations from 20 to 500 mM. In Gladden *et al.*, shear waves generated from a 61 Hz excitation had a speed proportional to the square root of the concentration when micellar fluid concentration was lower than 150 mM, on the other hand, a linear relationship was found between the micellar fluids concentration and the measured shear wave speeds for micellar fluids with concentrations higher than 150 mM.¹⁵ Because at very low concentration micellar fluids exhibit weak optical coefficients, an alternative technique to visualize shear wave propagation using microspheres has been recently reported.¹⁶ In Labda *et al.*, shear wave speed measured in a 500 mM micellar fluid using microspheres were in good agreement with optical methods; one of the advantages of using microspheres methods include the ability to characterize micellar fluids of low concentration.¹⁶

To completely characterize viscoelastic materials multiple excitation frequencies should be studied, however, mechanical excitation of shear waves and conventional rheometers can only excite one frequency at a time. Alternative to external mechanical vibration, localized shear waves can be produced by acoustic radiation force of ultrasound.¹⁰ The advantages of using acoustic radiation force to generate shear waves is that anywhere an ultrasound system can focus, a pushing pulse of radiation force can be applied. Moreover, acoustic radiation force is capable of generating shear waves over a certain bandwidth (BW) with a single impulse excitation, reducing the experimental time significantly.

In this paper, we report on the feasibility of using ultrasound to generate and detect shear waves in wormlike micellar fluids. The shear wave propagation is monitored with

conventional pulse echo ultrasound, and the micellar fluids shear wave propagation speed is compared to conventional oscillatory test with a rheometer.

II. MATERIALS AND METHODS

Micellar fluids of different concentrations (100, 200, 300, and 400 mM) were made with a ratio of 5:3 surfactant to salt. Cetrimonium bromide (CTAB, H9151, Sigma-Aldrich, St. Louis, MO) was used as surfactant and NaSAL (S2679, Sigma-Aldrich) was used as salt. Cellulose (S5504, Sigma-Aldrich) was used as ultrasound scatterers. A volume of 200 mL for each of the four samples was prepared as follows: 80 mL of distilled water (HPLC, 34877, Sigma-Aldrich) was distributed in two beakers and heated to 60 °C, NaSAL and CTAB were added to the separate beakers and mixed with the water for 25 min. Cellulose was mixed with 40 mL of distilled water and then added to the beaker containing the NaSAL. Once the solutions in both beakers were homogeneous, the NaSAL/cellulose solution was mixed with the CTAB solution and stirred for 5 h at 60 °C. Finally, the fluids were transferred to containers to cool to room temperature. Table I shows the composition of the micellar fluids.

A. Rheological testing

A strain controlled rheometer (AR 2000, TA Instruments, New Castle, DE) equipped with a concentric cylinder (bob and cup) fixture (Fig. 2) was used to measure the micellar fluids' shear complex modulus $G^*(\omega) = G_s(\omega) + iG_l(\omega)$, where $G_s(\omega)$ is the storage modulus, $G_l(\omega)$ is the loss modulus, and ω is the angular frequency. The conical end bob and cup radii, R_1 and R_2 , were 14 and 15 mm, respectively. The height, H , was 42 mm and a gap of 5000 μm between the end of the conical bob and the bottom of the cup was used. Samples of ~ 16 mL were tested at 23 °C under shear oscillatory at 1% strain from 0.001 Hz to 15.91 Hz.

TABLE I. Micellar fluid (5:3, CTAB:NaSAL ratio).

Solution	CTAB (g)	NaSAL (g)	Cellulose (g)
100 mM	7.29	1.92	2
200 mM	14.58	3.84	2
300 mM	21.87	5.76	2
400 mM	29.16	7.68	2

FIG. 2. Concentric cylinder (bob and cup) scheme. Bob radius, $R1$, height, H , and cup radii, $R2$ (adapted from Ref. 17).

A linear viscoelastic extended Maxwell model was fit to the measured shear complex modulus $G^*(\omega)$. The extended Maxwell model has been shown to accurately represent the wormlike micellar fluid mechanical properties.⁴ The extended Maxwell model storage modulus, $GM_s(\omega)$, and loss modulus, $GM_l(\omega)$, are described as follows:⁴

$$GM_s(\omega) = \frac{\mu\omega^2\tau^2}{1 + \omega^2\tau^2}, \quad (1)$$

$$GM_l(\omega) = \frac{\mu^2\omega\tau}{1 + \omega^2\tau^2} + \eta_s\omega, \quad (2)$$

where μ is the shear modulus, τ is the relaxation time, η_s is the steady-state viscosity, and ω is the angular frequency. The data from the rheology instrument were fit to Eqs. (1) and (2). The fits were evaluated by computing the mean absolute error (MAE) between the magnitude of the measured complex modulus, $|G_{\text{measured}}^*(\omega)| = \sqrt{G_s^2(\omega) + G_l^2(\omega)}$, and the magnitude of the theoretical complex modulus, $|G_{\text{fit}}^*(\omega)| = \sqrt{GM_s^2(\omega) + GM_l^2(\omega)}$, at frequencies between 0.001 and 15.91 Hz. The MAE is defined as

$$\text{MAE} = \text{mean}(|G_{\text{measured}}^*(\omega)| - |G_{\text{fit}}^*(\omega)|). \quad (3)$$

FIG. 3. (Color online) Micellar fluid complex moduli measured with rheometer. (a) Storage modulus, (b) loss modulus.

B. Acoustic radiation force shear wave elastography

A Verasonics V-1 ultrasound system (Verasonics, Inc., Kirkland, WA) equipped with a linear array transducer (L7-4, Philips Healthcare, Andover, MA) was used for the experiments. Three repeated measurements were made in each phantom. A 4.09 MHz, 200 μ s duration push beam was transmitted and focused 20 mm from the micellar fluids surface to generate shear waves. The shear wave propagation was measured with the same transducer with plane wave compounding imaging technique.¹⁸ A set of three plane waves with different emission angles (-4° , 0° , $+4^\circ$) were transmitted at 10 kHz pulse repetition frequency (PRF). By coherently compounding each set of three plane waves, a compound image PRF of 3.33 kHz was produced. The spatial resolution in x - and z -directions was 0.31 mm and 0.31 mm, respectively. Local displacement was estimated using an autocorrelation method.¹⁹ The same shear wave generation and detection parameters were used with all micellar fluids.

1. Shear wave speed measurement and frequency analysis

The shear wave group velocity (c_g), phase velocity (c_p), center frequency (f_c), and BW were measured. The shear wave displacement data within the focal zone were averaged along the axial direction (focal depth ± 1.5 mm) to create spatiotemporal maps. Then, the shear wave group velocity (c_g) was estimated using lateral time-to-peak (TTP)²⁰ and random sample consensus (RANSAC)²¹ algorithms. A spatiotemporal interpolation factor of 10 was used (interpolation was performed both on temporal and spatial dimensions by a factor of 10 in each direction). The RANSAC method was used to find the c_g from the spatiotemporal data points for each phantom. The RANSAC method parameters were selected from preliminary phantom studies. The parameter σ , which is a standard deviation (SD) of the shear wave time delay estimation was set to 0.20 ms, the number of iterations was set to 5000, and the inlier probability was 99%.

Shear wave phase velocity was estimated by a previously described two-dimensional Fourier transform method (2D FFT).^{22,23} The shear wave center frequency (f_c), a parameter that describes the frequency content of shear

wave, in the frequency-domain was calculated from the peak energy in the 2D FFT of shear wave particle velocity. Similarly, the BW was estimated from the upper -12 dB frequency relative to the center frequency on the 2D FFT space (k -space).²⁴ Shear wave phase velocity, c_p , written as a function of the shear complex modulus yields²⁵

$$c_p(\omega) = \sqrt{\frac{2(G_s^2(\omega) + G_l^2(\omega))}{\rho(G_s(\omega) + \sqrt{G_s^2(\omega) + G_l^2(\omega)})}}. \quad (4)$$

The phase velocities measured with acoustic radiation force and the rheological test were compared by computing the MAE between the ultrasound measured phase velocities, $c_{p_ultrasound}(\omega)$, and the rheological test extended Maxwell fit in combination with Eq. (4), $c_{p_rheometer}(\omega)$;

$MAE = \text{mean}(|c_{p_ultrasound}(\omega) - c_{p_rheometer}(\omega)|)$, at frequencies between 100 and 500 Hz.

III. RESULTS

The measured micellar fluid shear complex moduli (storage and loss moduli) from the rheological shear oscillatory test are illustrated in Fig. 3.

Superimposed upon the measured micellar fluid complex moduli in Fig. 4 are the extended Maxwell model fits, Eqs. (1) and (2). The extended Maxwell model shear modulus, μ , relaxation time, τ , steady-state viscosity, η_s , and the computed MAE of the fits, Eq. (4), evaluated from 0.001 Hz to 15.91 Hz of the micellar fluids are summarized in Table II.

Ultrasound shear wave generation and detection is illustrated in Fig. 5 by shear wave displacement maps shown

FIG. 4. (Color online) Measured complex modulus with rheometer and extended Maxwell model fits.

TABLE II. Extended Maxwell model fit parameters (shear modulus, μ , relaxation time, τ , steady-state viscosity, η_s) and MAE of the fit.

	100 mM	200 mM	300 mM	400 mM
μ (Pa)	36.25	149.42	262.97	422.55
τ (s)	48.88	25.62	21.83	9.41
η_s (Pa s)	0.33	0.48	0.83	1.34
MAE (Pa)	0.92	1.18	3.47	7.34

12 ms after the acoustic radiation force excitation in the micellar fluids.

The mean shear wave displacement in the different fluids at the focal area (3 mm window in z axis) as a function of lateral distance (x) and time (t) are shown in Fig. 6.

The mean shear wave group velocity measurements as a function of micellar fluid concentration is illustrated in Fig. 7.

Shear wave center frequencies and BW computed from the 2D FFT of shear wave particle velocity illustrated in Fig. 8 are summarized in Table III.

Measured mean shear wave phase velocity as a function of frequency (100 Hz–500 Hz) for each micellar fluid is shown in Fig. 9. The dashed line represents the phase velocities computed from the extended Maxwell model fits of the complex modulus measured by a rheometer [refer to Table II and Eqs. (1), (2), and (4)]. The computed MAE of shear wave phase velocities were < 0.02 m/s for all micellar fluids.

IV. DISCUSSION

In this study, we demonstrate the feasibility of an ultrasound-based method to generate and detect shear waves in micellar fluids of different concentration. Although the shear wave group velocity correlated with micellar fluid concentration, the shear wave group velocity does not account for viscous component of the materials. A more accurate characterization of micellar fluid viscoelastic properties is found with the shear wave phase velocity.

Rheological studies of micellar fluid suggest micellar fluid complex shear modulus can be accurately modeled by the extended Maxwell model.⁴ In this study, the measured shear complex modulus of micellar fluids shows remarkable agreement with the extended Maxwell model (Fig. 4). Regardless of some discrepancies at higher frequencies observed in the 100 nM micellar fluid, the MAE of measured complex modulus from shear oscillatory test and the extended Maxwell model fit were considerably small. Sources of error when performing the shear oscillatory test are attributed to highly sensitive phase angle measurement at high frequencies due to instrument limits.²⁶ As summarized in Table II, the results of the extended Maxwell model fit of elastic, μ , and viscous, η_s , components increased proportional to micellar fluid concentration over the studied frequency range of 0.001–15.9 Hz.

Although shear wave propagation in micellar fluids has been reported previously in studies by Gladden *et al.*,^{14–16} the feasibility of using ultrasound-based shear wave methods have not been studied. Successful ultrasound shear wave generation and detection in micellar fluids is shown in

Fig. 5. The shear wave displacement maps illustrate shear wave fronts traveling further and decreasing their amplitude as the micellar fluid concentration increases (Fig. 6). One of the most common methods to characterize shear wave

FIG. 5. (Color online) Shear wave displacement maps in micellar fluids of different concentrations at $t = 12$ ms.

FIG. 6. (Color online) Shear wave displacements at focal area as a function of time (t) and lateral distance (x) of micellar fluids.

propagation is by measuring shear wave group velocity c_g . A Pearson's correlation coefficient of 0.99 was found between micellar fluid concentration and shear wave group velocity (Fig. 7). As expected, in micellar fluids with concentrations larger than 150 mM, a linear correlation exists between micellar fluid concentration and shear wave group velocity.¹⁵

The shear wave propagation frequency content in the micellar fluids was characterized by the shear wave center frequency (f_c) and shear wave BW. Although the shear wave center frequency depends on the acoustic radiation force temporal duration and spatial distribution,²⁴ in this study the same acoustic radiation force pulse parameters were used to study all micellar fluids. The decrease in center frequency as a function of micellar fluid concentration found in this study is attributed to the micellar fluid viscoelastic properties (refer to Table III). This latter statement agrees with the increase in steady-state viscosity, η_s , as a function of micellar fluid concentration found from the extended Maxwell model fit. The measured BW of micellar fluids did not vary considerably as a function of micellar fluid concentrations (refer to Table III).

The f_c value is also important to note when calculating the group velocity in a viscoelastic medium because dispersion causes the shear wave speed to depend on frequency. Bias in shear wave group velocity estimation from ultrasound-based methods has been previously reported by

our group.²⁷ Usually these discrepancies are due to the fact that shear wave velocity will vary as a function of frequency (a phenomenon known as shear wave velocity dispersion that results from viscoelasticity) and the TTP method used to estimate shear wave group velocity assumes the material is pure elastic. To illustrate this, for a pure elastic material Eq. (4) can be approximated to

$$c_p(\omega) = \sqrt{\frac{G_s(\omega)}{\rho}}, \quad (5)$$

by replacing the extended Maxwell model shear modulus, μ , from the rheological test (refer to Table II) in Eq. (5), the shear wave velocities are 0.19, 0.39, 0.52, and 0.66 m/s compared to ultrasound-based shear wave group velocities of 0.24, 0.44, 0.56, and 0.71 m/s for 100, 200, 300, and 400 mM micellar fluids, respectively. On the other hand the shear wave phase velocities at the center frequency were 0.19, 0.39, 0.51, and 0.65 m/s for 100, 200, 300, and 400 mM micellar fluids, respectively. Hence, a more accurate estimation of viscoelasticity is achieved by the shear wave phase velocity.

Shear wave phase velocities as a function of frequency (100 Hz–500 Hz) are computed from the 2D FFT of shear wave particle velocity. Superimposed to measure shear wave phase velocities in Fig. 9 are the computed phase velocities from extended Maxwell model fits [refer to Table II and Eqs. (1), (2), and (4)], over the frequency range of 100–500 Hz. An excellent agreement was found between ultrasound-based and rheology test phase velocities (computed MAE < 0.02 m/s) over 100–500 Hz.

Compared to external single frequency excitation, ultrasound-based shear wave elasticity methods offer a localized and faster approach to study shear wave propagation in micellar fluids. However, there are some limitations to studying micellar fluids with ultrasound-based shear wave elasticity methods. For instance, to detect shear wave propagation in micellar fluids with ultrasound imaging methods, scatterers need to be added to the micellar fluids and the effects of the scatterers in micellar fluids viscoelastic properties would need to be evaluated. However, the results from the same fluids with scatterers were used in the oscillatory shear tests and ultrasound-based shear wave elasticity method and good agreement was found, which indicates that the scatterers did not adversely affect the mechanical properties of the micellar fluids. Another limitation is that the frequency BW of generated shear waves with ultrasound radiation force (>100 Hz) is considerably different to the conventional shear oscillatory test (<16 Hz) where micellar

FIG. 7. (Color online) Mean shear wave group velocity as a function of micellar fluid concentration. The error bars represents one standard deviation of six measurements (three repeated measured containing right and left propagating shear waves).

FIG. 8. (Color online) 2D FFT of shear wave propagation in micellar fluids. The color scale is fixed from -12 dB to 0 dB.

TABLE III. Shear wave center frequency (f_c) and BW. Mean $\pm$ SD, $n = 6$.

	100 mM	200 mM	300 mM	400 mM
f_c (Hz)	167.27 ± 12.86	153.69 ± 3.37	143.01 ± 6.01	142.01 ± 14.59
BW (Hz)	478.44 ± 9.59	466.50 ± 9.70	464.05 ± 9.85	482.78 ± 24.64

fluids are highly viscous.²⁸ Modulated ultrasound methods could be employed to generate acoustic radiation force to investigate other frequencies, but would require a more involved experimental setup. Nonetheless, micellar fluids offer a unique material to model semi-solid biological tissue such as cyst or fat, thus, its advantages to validating and developing new ultrasound shear wave imaging methods are valuable. Future studies include modulating ultrasound radiation force to explore other frequency ranges.

FIG. 9. (Color online) Measured mean shear wave velocity and computed shear wave phase velocity from extended Maxwell model fit to complex modulus measurements with rheometer as a function of frequency of all micellar fluids. The error bars represent one standard deviation of six measurements (three repeated measured containing right and left propagating shear waves).

V. CONCLUSION

This paper presents an alternative method to study micellar fluid viscoelastic properties by using ultrasound to generate and detect shear waves in micellar fluids. The result demonstrates that shear wave phase velocities measured by the ultrasound method are in good agreement with the conventional shear oscillatory test. Viscoelastic micellar fluids have the potential to be used as a model of biological tissues and benefit the development of non-invasive shear wave methods.

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