Ultrasonic Investigation of Viscoelastic Properties in Silver Nanofluids

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Silver nanofluids have been prepared by a one-step simple, green and cost-effective method using tannic acid as both reducing and stabilizing agent. The physical parameters such as ultrasonic velocity, density and viscosity of silver nanofluids were measured at (298.15 K, 303.15 K, 308.15 K and 313.15 K) and derived parameters like adiabatic compressibility (β), mean free path (L_{mfp}), bulk modulus (E), ultrasonic attenuation coefficient (α/f^2), diffusion coefficient (D) and relaxation time (τ) were computed. The variation of ultrasonic velocity with temperature, concentration and frequency shows anomalous behaviour and the parameters β, L_{mfp}, E too reflected this behaviour. Viscosity variation as a function of temperature and concentration is also studied and correlated with particle size. Ultrasonic attenuation coefficient (α/f^2), diffusion coefficient and relaxation time exhibited a similar pattern as that of viscosity of samples confirming the significant role of viscosity in transport phenomena and flow characteristics of fluids. Results are interpreted in terms of nanoparticle-nanoparticle and nanoparticle-fluid interactions.

Keywords: Nanofluids, Density, Ultrasonic velocity, Viscosity, Attenuation coefficient, Diffusion coefficient, Relaxation time.

INTRODUCTION

Recent innovations in analytical and imaging technologies have led to extensive research activities to design novel nano-materials using a wide variety of manufactured nanoparticles and nanofluids. Nanofluids are a new class of smart fluids engineered by the novel stable suspensions of a very small amount of nanoparticles as guests having size in the range 10-100 nm dispersed uniformly in base fluids such as vegetable oil, water and organic liquids like butanol, ethylene glycol, oil, polymeric solutions [1] or conventional fluid mixtures. The nanoparticles may be metallic (*e.g.* Ag, Cu, Fe, Au), metallic oxide (Al₂O₃, CuO, ZnO) or different forms of carbon (fullerene, graphene). Nanofluids find potential commercial applications as they do not cause erosion to the channels in which they flow and do not produce sedimentation.

The suspended nanoparticles can change the transport and thermal properties of the base fluid in a peculiar way due to their small size. The large specific surface area, less particle momentum and high mobility of added nanoparticles can provide dramatic improvements in the mechanical, optical and thermal properties [2] of host fluids. Fascinating superior thermo-physical properties such as thermal conductivity, thermal diffusivity, viscosity and convective heat transfer coefficients of nanofluids compared to those of base fluids make them sought after in thermal engineering applications [3,4]. The extensive list of applications is mainly due to the transport properties and flow patterns of nanofluids.

Nanosilver is one of the nanomaterials that find applications in a diverse range of consumer products including plastics, soaps, pastes, food thus increasing their market value [5-7]. Due to their distinctive properties, silver nanoparticles are put into therapeutic use in medicine, as an antibacterial, antifungal, antiviral and anti-inflammatory agents [8], incorporated in cotton and silk cloths [9] to reduce infections as well as to prevent bacteria colonization on prostheses, catheters, vascular grafts, dental materials, stainless steel materials and human skin [10] as markers to sense Alzheimer's disease [11] products for wound healing [12] in cancer therapies [13,14] and in single particle assays [15]. Due to its surface plasmon resonance,

silver nanoparticles find remarkable importance in the area of optical sensing [16-18] as imaging agents [19] and in photovoltaics [20-22].

A wide range of potential applications of silver nanoparticles has created a lot of interest among researchers to investigate various properties of silver nanofluids. Extensive research has been taken up to synthesis silver nanoparticles by various methods and their characterization through various techniques. Recently, measurement of thermal diffusivity and conductivity of silver nanofluids are reported using thermal lens technique and transient hot-wire technique respectively [23,24]. Kerosene-based nanofluids prepared using silver nanoparticles have shown enhancement in thermal conductivity [25]. Most of such studies focus on thermal conductivity studies of nanofluids. Few reports are found in the literature devoted to the study of the rheological properties of nanofluids and especially silver nanofluids. The number of articles in which the size dependence of viscoelastic properties of silver dispersed nanofluids is still relatively small. Viscoelastic properties of nanofluids play an extremely crucial role in practical heat transfer systems. Therefore, more attention needs to be paid to this area to understand the complex interaction of nanoparticles with the base fluid molecules, which in turn could also become a powerful additional tool to characterize nanofluids.

The aim of the current work is to explore the viscoelastic properties of silver nanofluid under various physical situations like variation in concentration, temperature and frequencies and analyze the dependence of particle size on measured viscoelastic properties so as to have the right knowledge of these nanofluids under realistic conditions to deploy them in suitable applications.

EXPERIMENTAL

Reagents of high purity like silver nitrate (AgNO$_3$, purity 99.999%, Sigma-Aldrich), Tannic acid (C$_{76}$H$_{52}$O$_{46}$, Fluka) and K$_2$CO$_3$ from (Merck, India) were used without further purification for the synthesis of silver nanofluids. De-ionized water was obtained from Deionizer Millipore Simplicity UV system.

Preparation of silver nanofluids: There are two methods for the preparation of nanofluids, first method is the one-step method which synthesize nanoparticles directly in the base fluid. This method is suitable particularly for the preparation of nanosuspensions with high thermal conductivity metal nanoparticles, such as Ag, Cu, Al, *etc*. The second method is a two-step method which is the dispersion of NPs in the base fluid. The one-step method is more advantageous than the 'two-step' method as it avoids the problems of hard agglomeration caused in drying, procedures of collection and storage of nanoparticles, which may generate second-level agglomeration or oxidation in the air.

Five samples of silver nanofluids of varying concentrations are prepared by the one-step method through chemical reduction of silver nitrate solution using tannic acid as a reducing agent suggested by Cataldo *et al.* [26]. Fresh working solutions are prepared with deionized water. Weight measurements of samples are made using an electronic balance Citizon CX 285

N with an accuracy of 1×10^{-5} g with readability 0.01/0.1 mg and repeatability ± 0.03/0.1 mg. The alkaline nature (pH = 8.0) of tannic acid before the addition of AgNO$_3$ is measured using the Elico pH meter.

Tannic acid (60 mL) was poured in each of five beakers. Silver nitrate (1.32×10^{-3} M, 1, 2, 3, 4 and 5 mL) were added respectively to five beakers while stirring. Instantaneously a pale yellow colour appeared, indicating the formation of silver nanoparticles at room temperature. Stirring was carried out till no noticeable change observed in the colour. The solution turned gradually darkened with the increase in the amount of silver added. The samples were named S1, S2, S3, S4 and S5 respectively. The prepared nanofluid samples are stable and free from sediments for months together.

Characterization of silver nanofluids: The ultrasonic velocity in the fluids was measured using the continuous wave Nano-fluid ultrasonic interferometer Model NF-12X (Mittal Enterprises, India) working at different frequencies, with cells of 12 mL capacity each and with the accuracy of ± 2 m/s. A digital micrometer screw with the least count of 0.001 mm was used to lower or raise the reflector plate connected to the cell. The specially designed jacketed measuring cell was used to maintain the uniform temperature of the sample. The inlet and outlet of the cell was connected to a constant temperature bath with the accuracy of ± 0.01 K. The interferometer was calibrated with double distilled water and acetone just before put into use.

Data of density measurements of pure components and nanofluids were obtained as an average of triplicate measurements at stable temperature using Rudolph research analytical DDM 2911 automatic density meter with high precision Peltier temperature control of sample which offers 5×10^{-5} g/cm^3 accuracy. All the measurements were made in the temperature range from 298 K to 313 K at an interval of 5 K at atmospheric pressure.

A rotational modular compact rheometer (MCR) 102 (Anton Paar, GmbH, Austria), equipped with a PP 50 parallel plate measuring system was used to study the viscosity of silver nanofluids. Shear stress was imposed on the sample and the related shear rate was recorded at a temperature at an accuracy of ± 0.1 K using a constant temperature bath. A constant shear rate of 50 s^{-1} in a temperature range from 298 K to 313 K with 5 K/60 s step is chosen to study the dependence of dynamic viscosity on temperature. The average size of the particles and their size distribution was measured by dynamic light scattering (DLS) using HORIBA-SZ-100 particle size analyzer at 25 ºC [27].

RESULTS AND DISCUSSION

The nature of interactions between the nanoparticles and the molecules of base fluid can be obtained from the ultrasonic study. Ultrasonic velocities of silver nanofluid samples were measured experimentally at 2, 4 and 6 MHz in the temperature range from 298 K to 313 K in steps of 5 K at atmospheric pressure and the data is presented in Table-1. The variation of ultrasonic velocity in various samples with temperature and frequency is shown in Fig. 1.

TABLE-1
VARIATION OF ULTRASONIC VELOCITY (m s^{-1}) WITH CONCENTRATION AND
FREQUENCIES (2, 4, 6) MHz AT 298, 303, 308 AND 313 K

| Samples	Frequency = 2 MHz				Frequency = 4 MHz				Frequency = 6 MHz			
	298 K	303 K	308 K	313 K	298 K	303 K	308 K	313 K	298 K	303 K	308 K	313 K
Ag	1494.8	1507.0	1518.3	1472.0	1531.2	1505.4	1510.9	1526.8	1498.1	1501.9	1513.3	1520.8
T	1497.2	1507.3	1518.7	1526.0	1495.3	1502.4	1516.1	1526.8	1495.5	1500.5	1520.5	1522.9
S1	1550.7	1510.3	1518.1	1526.7	1502.8	1508.1	1546	1524	1505.6	1514.4	1518.7	1536.5
S2	1494.1	1558.9	1520.8	1579.3	1508.8	1510.1	1508.3	1522.7	1515.5	1513.9	1524.0	1530.9
S3	1497.6	1507.7	1522.8	1527.6	1508.8	1505.8	1516.1	1527.6	1488.5	1577.1	1547.7	1521.3
S4	1575.2	1446.4	1521.8	1530.1	1499.7	1509.0	1521.1	1529.8	1490.4	1531.5	1526.1	1525.3
S5	1450.0	1510.8	1532.0	1530.1	1503.5	1506.3	1517.0	1523.7	1490.1	1536.8	1521.1	1496.3
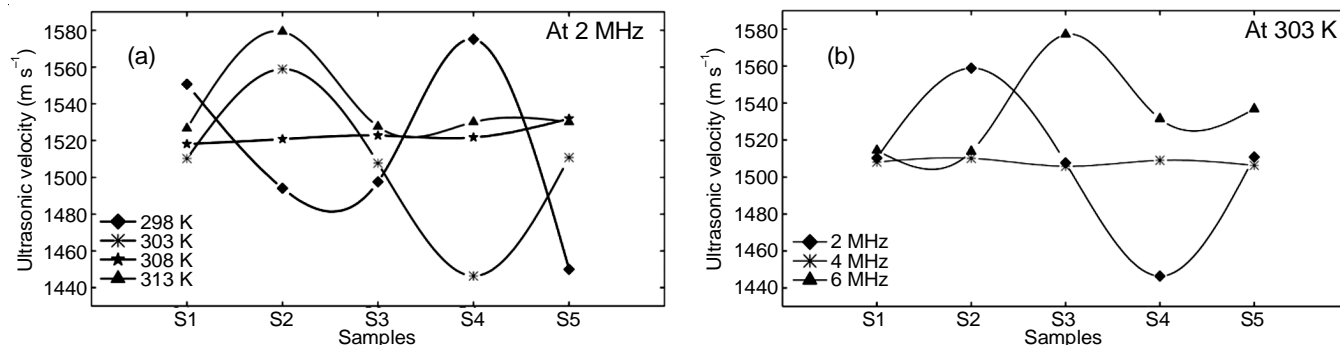
Fig. 1. Variation of ultrasonic velocity with concentration at (a) 2 MHz at different temperatures and (b) 303 K at different frequencies

It is observed that the ultrasonic velocity of silver nanoparticles is temperature-dependent and shows non-linear variation with temperature which may be due to the variation in the size of silver nanoparticles and their distribution in the fluids. Similar observations are reported in the literature [27-29]. The temperature and frequency dependence of the ultrasonic velocity is so anomalous in Fig. 1, which may be due to the presence of various forms of interactions between the incident ultrasonic waves and silver nanoparticles present in the base fluid and is almost not reported in the literature.

Generally, the colloidal nature of nanofluids by neglecting the interactions between particles will be considered for density measurements. Interactions among the fluid particles assume relevance as the concentration of NPs in the fluids increases. Thus, density and viscosity of the silver nanofluid samples were measured experimentally in the temperature range from 298 K to 313 K in steps of 5 K at atmospheric pressure and the data is presented along with relative viscosity in Table-2. Variation of density and viscosity of experimental samples with temperature is shown in Fig. 2, which show a decreasing trend with

increasing temperature but with concentration, it shows a slight irregular trend which may be due to the size variations of nanoparticles and their non-uniform dispersion in the base fluid and indicates the insignificant dependence on particle size. A similar observation is reported in the literature [30].

Thermal properties of nanofluids are mainly affected by the physical properties of nanoparticles, base fluid and the interaction between them. The flow phenomenon of a base fluid, carrying solid nanoparticles depends upon the hydrodynamic force acting on the surface area of solid nanoparticles suspended in the base fluid. Viscosity describes the fluid internal layer resistance to the flow and so becomes very important for all transport properties of the fluids. Brownian motion of the nanoparticles in the fluid plays an important role in understanding the viscosity of nanofluids. In general, dynamic viscosity for homogeneous and incompressible fluids in isothermal conditions is constant at every point of the fluid. However, this is not exactly true for heterogeneous nanofluids. The viscous behaviour of nanofluid at the solid-liquid interfaces is discontinuous and so the viscosity in each point of nanofluid cannot be accurately

TABLE-2
VARIATION OF DENSITY, VISCOSITY AND RELATIVE VISCOSITY WITH CONCENTRATION AT 298, 303, 308 AND 313 K

| Samples	Density, $\rho \times 10^{-3}$ (Kg m^{-3})				Viscosity, $\eta \times 10^{-3}$ (Pa·s)				$\eta_{re} = \eta_{nf}/\eta_0$			
	298 K	303 K	308 K	313 K	298 K	303 K	308 K	313 K	298 K	303 K	308 K	313 K
Ag	997.07	994.69	992.5	990.09	0.757	0.713	0.554	0.461	1.392	1.498	1.304	1.194
T	997.45	995.54	993.37	991.01	0.544	0.476	0.425	0.386	1.000	1.000	1.000	1.000
S1	997.64	995.73	993.54	991.08	0.862	0.839	0.678	0.614	1.585	1.763	1.595	1.591
S2	998.05	996.14	993.89	991.47	0.732	0.693	0.578	0.504	1.346	1.456	1.360	1.306
S3	997.35	995.59	993.16	990.72	0.525	0.465	0.422	0.376	0.965	0.977	0.993	0.974
S4	997.03	995.1	992.9	990.39	0.522	0.463	0.412	0.369	0.960	0.973	0.969	0.956
S5	997.41	995.49	993.31	990.93	1.470	1.340	1.090	0.912	2.702	2.815	2.565	2.363
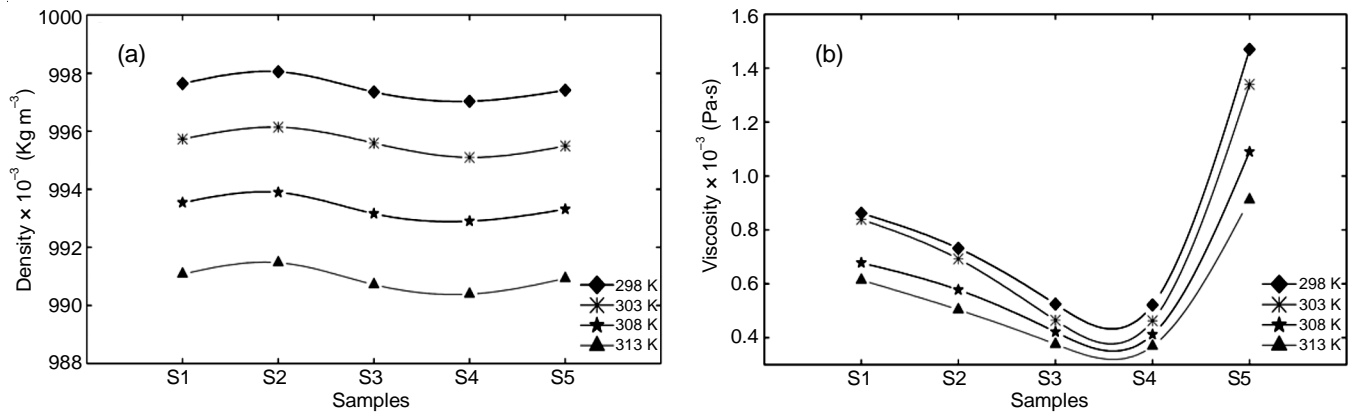
Fig. 2. Variation of density (a) and viscosity (b) with concentration at different temperatures

expressed. Therefore, effective viscosity which refers to the viscosity of the whole heterogeneous nanofluid is only measured for all the samples.

The temperature dependence of the viscosity of a nanofluid is the most important thermophysical characteristic. Generally in liquids, the viscosity coefficient decreases with increasing temperature. It is observed (Fig. 2) that the viscosity decreases with the increase in temperature for all samples, which can be attributed to the weakening of interparticle and intermolecular adhesive forces between the nanoparticles and their base fluids [31]. It is found that at lower concentrations, the nanofluid remained Newtonian. With a further increase in the concentration, the nanofluid behaves as a shear thinning non-Newtonian fluid [32]. Non-Newtonian behaviour obviously affects the heat transfer performance of the fluid. More efficient mixing is therefore needed and increasing shear rates cause the nanofluid to behave in a more Newtonian manner.

The effect of silver nitrate concentration on the formation of silver nanoparticles of varying size [33] and the distribution for samples S4 and S5 is shown in Fig. 3 to support the findings of the present study. Viscosity decreases with increasing concentration and shows minimum for sample S4 and becomes maximum for sample S5 indicating the dependence of viscosity on the particle size. Particle size has been observed to have a significant impact on the potential enhancement of viscosity of nanofluids [34-38]. Another report in the literature reveals that alumina particles with 47 nm size have enhanced viscosity more than that of 36 nm size [39]. Sample S5 in the present study has maximum viscosity at all temperatures implying that the size of the nanoparticles formed in that sample may be bigger whereas the viscosity of sample S4 is the minimum at all temperatures indicating the smaller particle sizes formed, which can be verified from Table-3. This may be due to the fact that bigger particle size leads to smaller surface area and so the contact area with the base fluid decreases which favours enhancement in viscosity in sample S5 whereas the reverse is observed with small size

TABLE-3
PARTICLE SIZE DATA FROM DLS
STUDY OF SILVER NANOFLUIDS

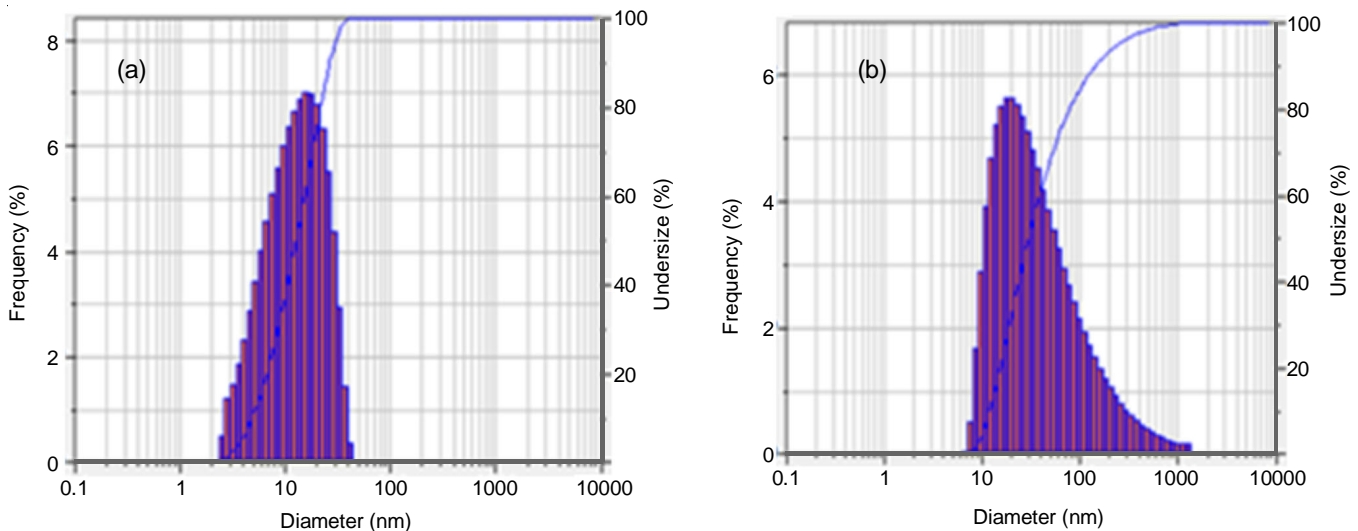| Samples	Concentration ratio of tannic acid to $AgNO_3$ (approx.)	Mean particle size ± standard deviation (nm)
S1	2:1	20.6 ± 6.1
S2	1:1	23.6 ± 8.0
S3	0.7:1	21.2 ± 5.8
S4	1:2	14.5 ± 8.4
S5	1:2.2	68.4 ± 6.1

Fig. 3. DLS spectra of samples S4 (a) and S5 (b)

nanoparticles in S4. The effect depends significantly on temperature and was greater at a lower temperature of the nanofluid.

The increase in viscosity in S5 may also be due to density and velocity micro fluctuations in the base fluid induced by moving nanoparticles [40]. These nanoparticles interact with these micro-fluctuations that determine the main mechanism of relaxation of the nanoparticles velocity [41,42]. The energy spent in producing such fluctuations accelerates the nanoparticles and which in turn experience increasing fluid layer resistance leading to an increase in the viscosity. Another possible reason for the enhancement of viscosity may be the probability of structure formation with short-range order in nanofluids, which increases with the particle concentration and favours momentum transfer among the nanoparticles and accounts for enhanced viscosity. The large value of viscosity depending on particle size must be taken into account for any practical application particularly in a heat transfer system, where the engine performance gets affected by the changes in viscosity of the fuel.

In contrast, some studies available in literature [35,43,44] show a reduction in the viscosity of nanofluids with increasing particle size. Another study reported that the viscosity of nanofluids is virtually independent of the size of nanoparticle [45]. The report of such contradicting results may be due to the difference in the choice of nanoparticle material. Conflicting information on the influence of nanoparticle size on viscosity is thus existing in the literature only indicate that the specific properties of the nanoparticles such as shape, size, structure, nature of the material, temperature, concentration of nanoparticles, elasticity of particles and the interaction between the base fluid and nanoparticles can play an essential role in determining the rheological behaviour of nanofluids. The temp-

erature dependence of the relative viscosity of nanofluids η_{re} from Table-2 also is useful to understand the particle fluid interactions. A few studies claim that the relative viscosity changes as a function of temperature; both decrease [46] and increase [47]. The present study however reveals the anomalous behaviour of relative viscosity with increasing temperature. Relative viscosity in sample S5 is almost three times that of the base fluid.

Using the data of ultrasonic velocity, density and viscosity, derived parameters like adiabatic compressibility (β), mean free path (L_{mfp}), bulk modulus (E), ultrasonic attenuation coefficient (α/f^2), diffusion coefficient (D), relaxation time (τ) were computed and reported in Tables 4-6, respectively. Variation in β, L_{mfp} and E with temperature and concentration from Table-4 show an anomalous behaviour as these are derived from ultrasonic velocity, which also shows similar inconsistent nature. This may be due to varying size and the uneven distribution of synthesized nanoparticles that perturbs the cohesive interaction forces that act among the nanoparticles as well as the density of the nanofluid from point to point.

The attenuation spectra of the samples (Fig. 4) show that the attenuation of the sound wave increases non-linearly with increasing concentration and shows minimum for S4 and maximum for S5 samples, respectively at all frequencies. In general, nanoparticles move from a region of higher concentration to a region of lower concentration and is known as diffusion and the diffusion coefficient tells the number of particles crossing per unit area per second per unit concentration gradient. Sample S5 at all the experimental temperatures and frequencies from Tables 4-6 indicate enhanced viscosity accompanied by high attenuation coefficient, high diffusion coefficient and higher relaxation time. The reason for such behaviours may be due to the cluster formation at a higher concentration.

TABLE-4
VARIATION OF ADIABATIC COMPRESSIBILITY, MEAN FREE PATH AND BULK MODULUS
WITH CONCENTRATION AND TEMPERATURES (298, 303, 308 AND 313 K) AT 2 MHz

Samples	$10^{-10} \times \beta$ (P a^{-1})				$10^{-11} \times L_{mfp}$ (m)				E $\times 10^{10}$ (Pa)			
	298 K	303 K	308 K	313 K	298 K	303 K	308 K	313 K	298 K	303 K	308 K	313 K
Ag	4.49	4.43	4.37	4.66	4.167	4.175	4.184	4.358	0.223	0.226	0.229	0.215
T	4.47	4.42	4.36	4.33	4.160	4.172	4.181	4.202	0.224	0.226	0.229	0.231
S1	4.17	4.40	4.37	4.33	4.016	4.163	4.182	4.200	0.240	0.227	0.229	0.231
S2	4.49	4.13	4.35	4.04	4.167	4.033	4.174	4.059	0.223	0.242	0.230	0.247
S3	4.47	4.42	4.34	4.33	4.159	4.171	4.170	4.199	0.224	0.226	0.230	0.231
S4	3.57	4.80	4.35	4.31	3.718	4.349	4.174	4.192	0.247	0.208	0.230	0.232
S5	4.77	4.40	4.29	4.31	4.295	4.162	4.145	4.191	0.210	0.227	0.233	0.232

TABLE-5
VARIATION OF ULTRASONIC ATTENUATION COEFFICIENT WITH CONCENTRATION
AT FREQUENCIES (2, 4, 6) MHz AND TEMPERATURES (298, 303, 308 AND 313 K)

Samples	$10^{-15} \times \alpha/f^2$ at 2 MHz				$10^{-15} \times \alpha/f^2$ at 4 MHz				$10^{-15} \times \alpha/f^2$ at 6 MHz			
	298 K	303 K	308 K	313 K	298 K	303 K	308 K	313 K	298 K	303 K	308 K	313 K
Ag	5.98	5.51	4.19	3.84	5.56	5.52	4.25	3.44	5.94	5.56	4.23	3.48
T	4.27	3.67	3.21	2.88	4.29	3.71	3.23	2.88	4.29	3.72	3.20	2.90
S1	6.09	6.43	5.13	4.58	6.69	6.46	4.86	4.60	6.66	6.38	5.12	4.49
S2	5.78	4.83	4.35	3.39	5.61	5.31	4.46	3.79	5.54	5.27	4.32	3.72
S3	4.12	3.58	3.16	2.80	4.03	3.60	3.21	2.80	4.20	3.13	3.01	2.83
S4	3.52	4.04	3.10	2.73	4.08	3.56	3.10	2.74	4.16	3.41	3.07	2.76
S5	12.71	10.26	8.02	6.75	11.40	10.36	8.26	6.84	11.71	9.75	8.20	7.22

TABLE-6
VARIATION OF DIFFUSION COEFFICIENT AND RELAXATION TIME WITH
CONCENTRATION AND TEMPERATURES (298, 303, 308 AND 313 K) AT 2 MHz

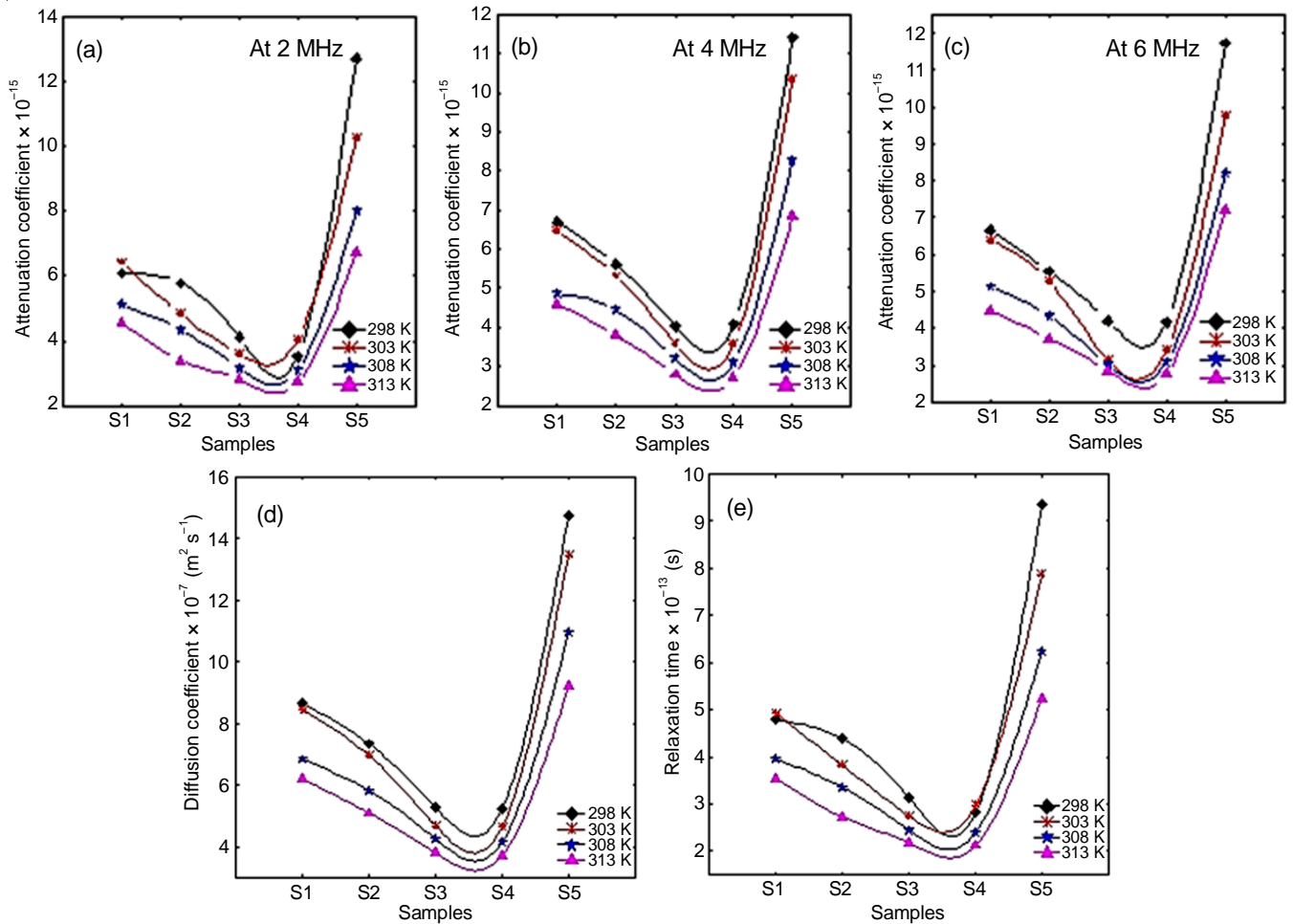| Samples	$10^{-7} \times D$ (m² s⁻¹)				$10^{-13} \times \tau$ (s)			
	298 K	303 K	308 K	313 K	298 K	303 K	308 K	313 K
Ag	7.59	7.17	5.58	4.66	4.53	4.21	3.23	2.87
T	5.45	4.78	4.28	3.90	3.24	2.81	2.47	2.23
S1	8.64	8.43	6.82	6.20	4.79	4.93	3.95	3.54
S2	7.33	6.96	5.82	5.08	4.38	3.82	3.35	2.72
S3	5.26	4.67	4.25	3.80	3.13	2.74	2.44	2.17
S4	5.24	4.65	4.15	3.73	2.81	2.97	2.39	2.12
S5	14.74	13.46	10.97	9.20	9.35	7.86	6.23	5.24

Fig. 4. Variation of attenuation coefficient with concentration at (a) 2 MHz, (b) 4 MHz, (c) 6 MHz, (d) diffusion coefficient and (e) relaxation time with concentration at 2 MHz at different temperatures

Conclusion

The present study reports the effect of concentration, temperature and frequency on the ultrasonic and viscoelastic properties of silver nanofluids prepared by the one-step method. Ultrasonic velocity and its derived acoustic parameters exhibit anomalous behaviour for all samples under study at all the experimental variables. Variation of density with concentration at different temperatures is least but not negligible. A non-linear variation of viscosity as a function of concentration indicates its sensitivity to particle size. The viscosity and other acoustic parameters that directly depend on viscosity exhibited similar behaviour and their sensitivity on the smallest and largest size of silver nanoparticles in the nanofluids is compared. Results were interpreted in terms of nanoparticle–nanoparticle and nanoparticle–base fluid interactions and ascertain the significant role of viscosity in transport phenomena and flow characteristics in nanofluids.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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