Synthesis and Characterization of Silicon carbide nanofluids

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Silicon carbide (SiC) has high thermo-mechanical properties and high resistance for high temperatures. Therefore, it may be recognized for the improvement of the efficiency of electric energy production. SiC nanoparticles shows high surface area hence it may be applicable in power plants. High temperatures are needed for its synthesis. In the present work, SiC nanopowder is synthesize via sol-gel method. The mixture of SiO_2:Mg in the molar ratio 1:2 heated in the furnace at 650°C for 6 hours. For an acid etching of the obtained product for 5 hour a mixture of HF 10% wt and HNO_3 4 M were used. Then the mixture is washed with distilled water and dried at room temperature. The final product is SiC.The synthesized nanoparticles are characterized by X-Ray powder Diffractometer (XRD), Fourier Transform Infra Red Spectroscopy (FTIR). The ultrasonic velocity, density and adiabatic compressibility were analyzed and the results were discussed. Average particle size has been estimated by using Debye-Scherrer formula. It was found to be 30 nm. Thermo acoustic properties of nanomaterials related to the surface of nanoparticles and nanoparticle surfactant interactions. Material characterization of SiC nanofluid was studied by non-destructive technique at various molar concentrations, temperatures and frequencies. The molecular properties of ultrasonic waves in nanofluids undergo changes in highly associated systems and dependent on the cohesive properties of liquids. In the present investigation an attempt is made to study the thermoacoustic behavior of prepared nanofluids in methanol at various concentrations.

Keywords: Nanofluids; Spectroscopic characterization; Ultrasonic velocity: Density; adiabatic compressibility; viscosity, etc.

Introduction:

Silicon carbide has great advantages in solar receptors. It is used for the fabrication of solar receptor due to its high radiative flow, big thermal and mechanical stress, and to work many hours without failure [1]. Due to its high absorptance values, superficial area, porosity and high fusion point [2], and its good thermo-mechanical properties [3-4] at high temperatures, SiC presents great advantages for applications in solar receptors used in a central.

Ultrasonic measurement together with density and viscosity helps in finding out many thermo acoustic parameters and all these are related to the surface of Nanoparticle and nanoparticles surfactant interactions [5-7]. SiC nanoparticles with surface areas 30 nm have been prepared and study its ultrasonic characteristics

in acetone base fluid [8-10]. Nanofluids have attracted greater interest in recent years because of their enhanced thermal conductivity in comparison to that of the base fluids. Therefore, nanofluids can be used as a better heat transfer fluid in the heat exchange systems. Nanoparticles find their wide applications in fields like electronic applications, transportation, industrial cooling application, heating buildings and reducing pollution, nuclear system cooling, space and defense, energy storage, solar absorption, friction reduction, magnetic sealing, antibacterial activity, nondrug delivery, vehicular brake fluids, nanofluids based microbial, fuel cell, and nanofluids based optical filters and sensors. The SiC nanofluids can be synthesized by dispersing a very small amount of Nanoparticles having size in the range about 30 nm in the base fluid acetone. Using the ultrasonication the dispersion of the NPs in the base fluid is made uniform [11]. Different methods have been developed to prepare nanofluids, such as the dispersing method [12-16], physical vapor condensation [17] and one-step chemical method [18], *etc*. In case of the dispersing method, synthesized nanoparticles are dispersed in liquid under stirring or ultrasonic vibration. It is a two-step method, in which the preparation of the nanoparticles and the preparation of the nanofluid are separated. The nanoparticles may agglomerate during the drying, storage, and transportation process, leading to difficulties in the following dispersion stage. Consequently, the stability and thermal conductivity of nanofluid are not ideal. The thermal conductivities behavior of the prepared nanofluids gives a high thermal conductivity than other conventional fluids by changing the precursor and volume fraction. In the present study, SiC nanofluids were prepared and ultrasonicated to get nanofluids by changing the weight percentage of the SiC nanoparticles. The structural, optical and physical properties were experimentally measured for both nanoparticles and nanofluids. In the present work to increase the thermo physical properties to get more heat transfer than other base fluids also by increasing the physical properties like ultrasonic velocity and density is done by increasing the molar concentration of SiC nanoparticles.

Experimental:

Preparation of Samples

All the reagents used in the experiment were of analytical purity. Silicon carbide nanoparticles have been synthesized by sol-gel method. The mixture of SiO_2:Mg in the molar ratio 1:2 heated in the furnace at 650°C for 6 hours. For an acid etching of the obtained product for 5 hour a mixture of HF 10% wt and HNO_3 4 M were used. Then the mixture is washed with distilled water and dried at room temperature. The final product is SiC. All the prepared nanofluids samples were kept in air tight bottles [19-30]. For comparison, the synthesized nanoparticles are characterized by X-Ray powder Diffractometry (XRD), Fourier Transform Infrared Spectroscopy (FTIR) and analyzed. After ultrasonication ultrasonic velocity, density, adiabatic compressibility and viscosity were analyzed and the results were discussed. There is a good agreement between the data produced by ultrasonic spectroscopy and other measurements.

XRD and FTIR

The X-ray diffraction patterns of the powdered samples of SiC were recorded on a X-ray diffractometer (XRD) with Cu-K (1.5406 *Å*). The average crystalline size (D) has been calculated from the XRD pattern by using Scherrer formula.

$$D = \frac{0.9\lambda}{WCos\theta} \quad \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where λ is the wavelength of X-ray, w is full width of half maximum (FWHM) and θ is Bragg's angle of radians. The results are presented in the Figure 1. FTIR spectra were recorded by using Perkin Elmer FTIR Spectrometer-Spectrum Two in solid phase using the KBr pellets FTIR Spectrometer-Spectrum.

Ultrasonic Measurements

The Ultrasonic velocities for SiC nanofluids of different molar concentration of the samples were measured using multifrequency ultrasonic interferometer operating at 2 MHz frequency. The densities of nanofluids were measured using specific gravity bottle of 10 ml capacity. The time of descent of the liquids between the viscometer marks was measured using an electronic digital timer with least count 0.01 sec. The viscosity was measured in Ostwald's viscometer with an accuracy 0.001 cP.

Results and discussion:

Crystal Structure and FTIR

The prepared SiC nanopowder were characterized for their phase purity and crystallinity by X-ray powder diffraction (XRD) using PAN-analytical diffractometer (Cu-Ka radiation) at a scanning step of 0.01^o, continue time 20 s, in the 2h range from 10^o to 120^o. The typical XRD pattern of the BN nano particles is shown in figure 1. The peak positions of the sample exhibited the monoclinic structure of SiC which was confirmed from the JCPDS data. Further, no other impurity peak was observed in the XRD pattern, showing the single phase sample formation. The crystalline size was calculated using the Scherrer formula, D= 0.9λ/w cosθ, Where 'λ' is the wavelength of X-ray ($1.5460A^0$), 'W' is FWHM (full width at half maximum), 'θ' is the diffraction angle and 'D' is particle diameter. The estimate size of SiC nano particles is found to be 30 nm. It is observed that no other impurity peak was observed in the XRD pattern, showing the single phase sample formation. Lattice parameters and cell volumes were analyzed using unit cell software. These values are in good agreement with the standard values reported by the JCPDS data. Figure 1 exhibits the XRD spectra of SiC nanoparticles. Figure 1 predicts that the intensity of crystalline peaks increases with temperature, indicating an improvement in the samples crystallinity. Simultaneously, the peaks become narrower as the temperature increases resulting in the increase of crystallite size.

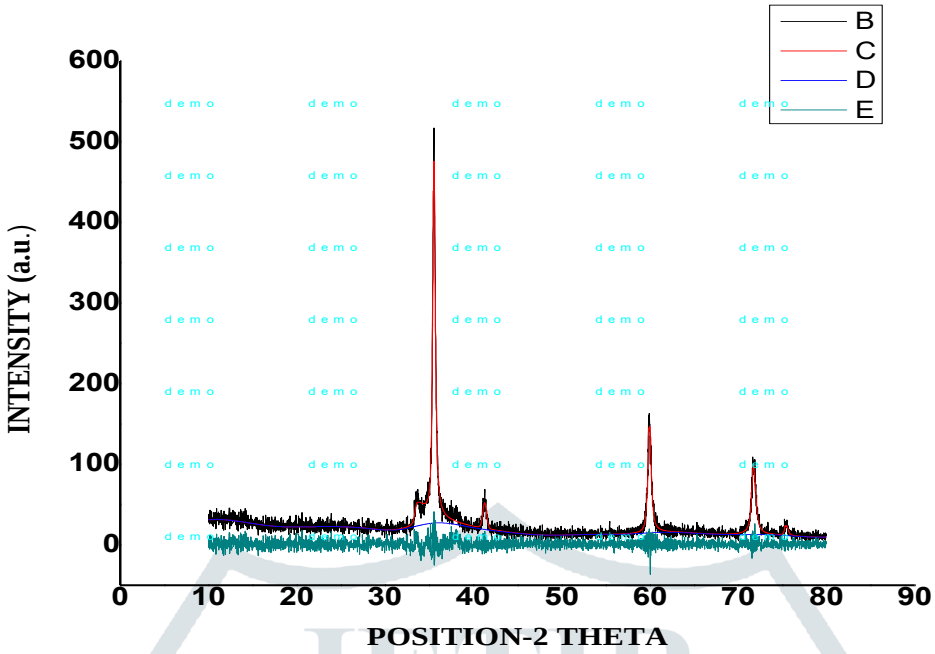

Fig.1 XRD pattern of Silicon carbide nanoparticles

Fig. 2 represents the FTIR spectra recorded in solid phase using the KBr pellets technique in the range of 4000- 500 cm^{-1} with resolution of 1 cm^{-1}. The broad absorption peak around 500 cm^{-1} to 1300 cm^{-1} is caused by OH starching of absorbed water molecules. The peaks from 500 cm^{-1} to 850 cm^{-1} indicates the Si-C stretching and 850 cm^{-1} to 1300 cm^{-1} indicates the SiC-O-H compounds. FTIR spectra exhibit only two vibrations occurring at approximately 604.72 cm^{-1} and 1078.95 cm^{-1} for the sample, which can be attributed to the vibrations of BN, confirming the formation of highly pure SiC nano particles [19].

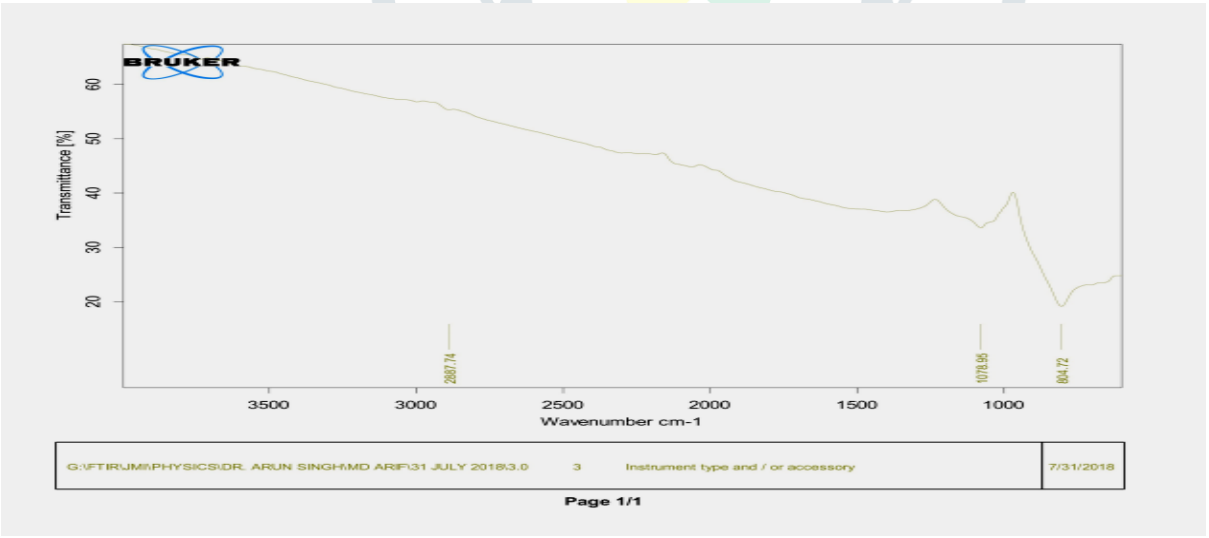

Fig. 2 FTIR analysis boron nitride nanoparticles

Ultrasonic velocity gets increases with increasing the molar concentration of the samples SiC in methanol this shows that the physical parameters of the sample changes by increasing the molar concentration. For SiC nanoparticle the velocity of the nanofluid is higher than methanol and also by increasing the molar

concentration of the sample SiC the velocity get increases and it is shows in the figure 3. The cause behind this increase of ultrasonic velocity with increase in molar concentration (x) is due to weakening of interaction between nanosize particle and micro sized fluid molecule. Ultrasonic velocity can be interpreted as the nanosize SiC particles have more surfaces to volume ratio and formation of hydrogen bonds with methanol molecules can absorb more methanol molecules on its surface, hence making the transport easy from point to another point, which enhances the velocity. The decrease of ultrasonic velocity with increase in concentration is due weakening of interaction between nano sized particle and micro sized fluid molecule. Peak at molar concentration 0.6 indicates strong aggregation of nano suspension. Non linear variation of ultrasonic velocity may due to the Brownian motion of BN nanoparticles.

Table: 1 Ultrasonic velocity of SiC nanofluids in methanol at various temperatures

x	293 K	298 K	303 K	308 K	313 K
0	1410	1420	1429	1437	1440
0.1	1429	1439	1438	1446	1453
0.2	1515	1525	1534	1542	1549
0.3	1480	1490	1499	1507	1514
0.4	1458	1468	1477	1485	1492
0.5	1510	1520	1529	1537	1544
0.6	1468	1478	1487	1495	1502
0.7	1464	1474	1483	1491	1498
0.8	1470	1480	1489	1497	1504
0.9	1480	1490	1499	1507	1514
1	1410	1420	1429	1437	1440

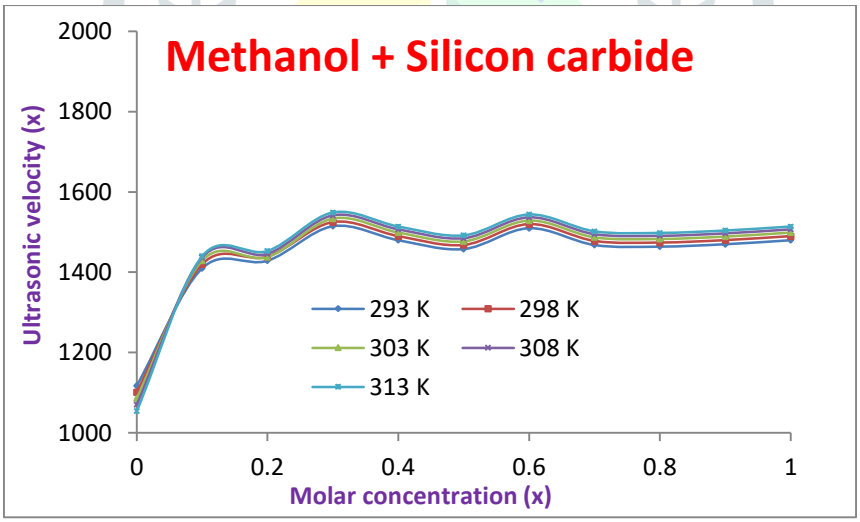

Fig. 3 Ultrasonic velocity versus molar concentration of SiC nanoparticle in methanol base fluid

Figure 4 shows the variation of density with molar concentration of SiC nanoparticles in methanol. Densities of the nanosuspension are calculated by measuring the weight of the nanofluid using 25 ml of specific gravity bottle and also by using the standard value of density of water. Increase in density indicates the close packing between the SiC nanoparticles in methanol base fluid. Non linear variation of density may be due to Brownian motion of SiC nanoparticle in methanol base fluid.

Table: 2 Density of SiC nanofluids in methanol at various temperatures

x	293 K	298 K	303 K	308 K	313 K
0	784	779.25	774.5	769.5	765
0.1	799.16	796.92	793.77	790.62	787.38
0.2	805.94	802.43	799.74	796.17	793.68
0.3	804.45	801.36	798.11	795.37	792.22
0.4	813.51	810.34	807.68	804.09	801.35
0.5	812.37	809.37	806.39	803.09	800.06
0.6	808.98	805.83	802.31	799.06	796.55
0.7	822.89	819.21	816.58	813.52	810.08
0.8	839.13	836.03	833.57	830.42	827.64
0.9	851.39	848.59	845.45	842.36	839.96
1	858.19	855.16	852.68	849.81	846.53

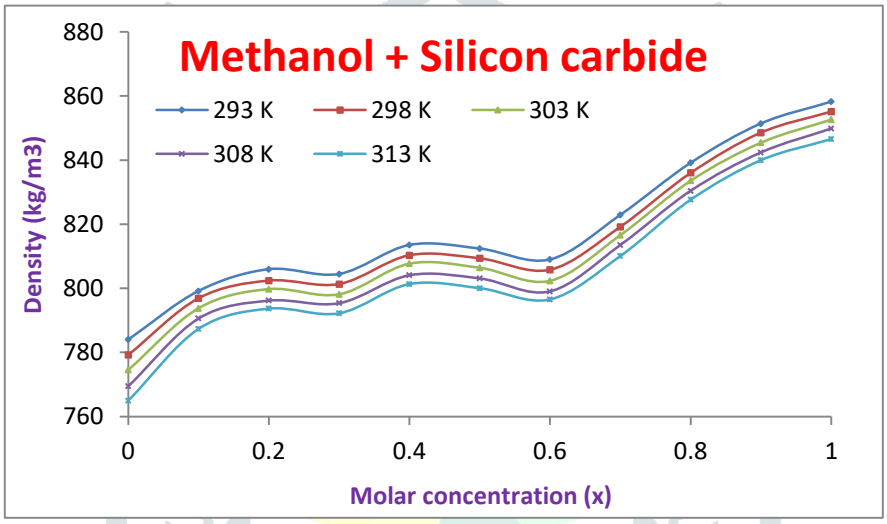

Fig. 4 Density versus molar concentration of SiC nanoparticle in methanol base fluid

Fig.5 represents the variation of adiabatic compressibility with molar concentration of SiC nanoparticles in methanol. The adiabatic compressibility of nanofluids enables direct access to the nanoparticle structure in terms of the particle packing density and the inter particle forces. It is related to density and ultrasonic velocity of nanosuspension and shows the reverse trends as that of ultrasonic velocity which is theoretically accepted. The non-linear variation of adiabatic compressibility with concentration of SiC indicates the presence of phase separation of SiC nano suspension in methanol base fluids. It is also observed from the plot that the ultrasonic velocity of SiC nanofluid in methanol increases slightly with molar concentration in the measured range of entire molar concentration indicating that the nanofluids with small amount of NPs are less compressible [31-34].

Table: 3 adiabatic compressibility of SiC nanofluids in methanol at various temperatures

x	293 K	298 K	303 K	308 K	313 K
0	1.02E-09	1.06E-09	1.09E-09	1.13E-09	1.18E-09
0.1	6.29E-10	6.22E-10	6.14E-10	6.13E-10	6.12E-10
0.2	6.08E-10	6.02E-10	6.03E-10	6.01E-10	5.97E-10
0.3	5.42E-10	5.37E-10	5.30E-10	5.29E-10	5.26E-10

0.4	5.61E-10	5.56E-10	5.49E-10	5.48E-10	5.44E-10
0.5	5.79E-10	5.73E-10	5.66E-10	5.65E-10	5.61E-10
0.6	5.42E-10	5.37E-10	5.31E-10	5.30E-10	5.27E-10
0.7	5.64E-10	5.59E-10	5.52E-10	5.50E-10	5.47E-10
0.8	5.56E-10	5.51E-10	5.44E-10	5.42E-10	5.38E-10
0.9	5.44E-10	5.38E-10	5.32E-10	5.30E-10	5.26E-10
1	5.32E-10	5.27E-10	5.20E-10	5.18E-10	5.15E-10

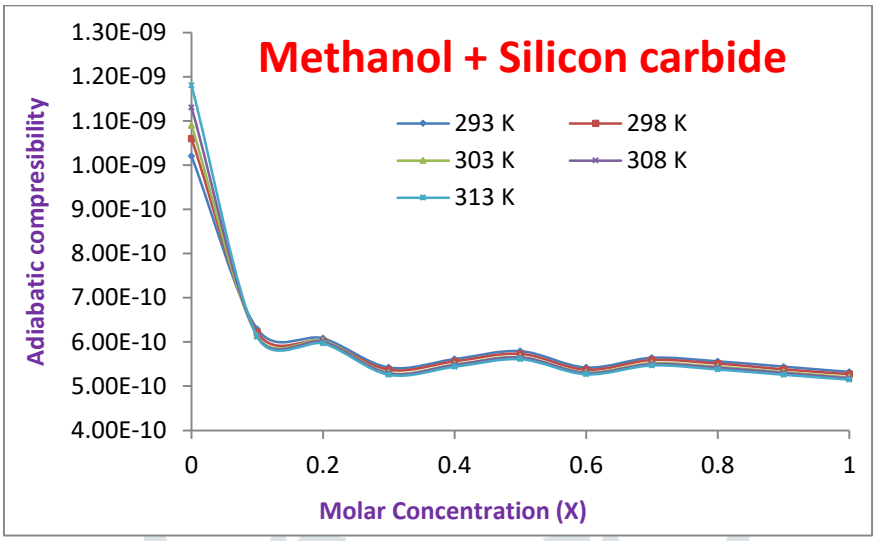

Fig. 5 Adiabatic compressibility versus molar conc. of SiC nanoparticle in methanol base fluid

Fig.6 represents the variation of viscosity with molar concentration of SiC nanoparticles in methanol base fluid. From the figure it is predict that viscosity diminishes as the sample fluid temperature increases. Furthermore, it shows that with higher nanoparticle concentrations, nanofluids possess higher viscosity. The shape of each curves for various temperatures are similar indicating the consistency of trend of the experimental measurements. The viscosity of nanofluids is an important transport property for applications of nanofluids as a new class of heating or cooling medium in thermal devices such as heat exchangers or cooling systems. As the temperature increases, the intermolecular attraction between the nanoparticles and their base fluids decreases. Hence the viscosity of nanofluids decreases with increase in temperature.

Table: 4 Viscosity of SiC nanofluids in methanol at various temperatures

x	293 K	298 K	303 K	308 K	313 K
0	0.5901	0.5471	0.5199	0.4849	0.4562
0.1	0.6749	0.6567	0.6386	0.6206	0.6027
0.2	0.6886	0.6703	0.6522	0.6341	0.6161
0.3	0.6829	0.6646	0.6464	0.6283	0.6103
0.4	0.7307	0.7123	0.6941	0.6758	0.6577
0.5	0.7319	0.7134	0.6952	0.6767	0.6585
0.6	0.7181	0.6995	0.6812	0.6626	0.6443
0.7	0.7601	0.7416	0.7232	0.7049	0.6867
0.8	0.7814	0.7632	0.7447	0.7265	0.7084
0.9	0.8053	0.7868	0.7684	0.7501	0.7319
1	0.8178	0.7993	0.7809	0.7626	0.7444

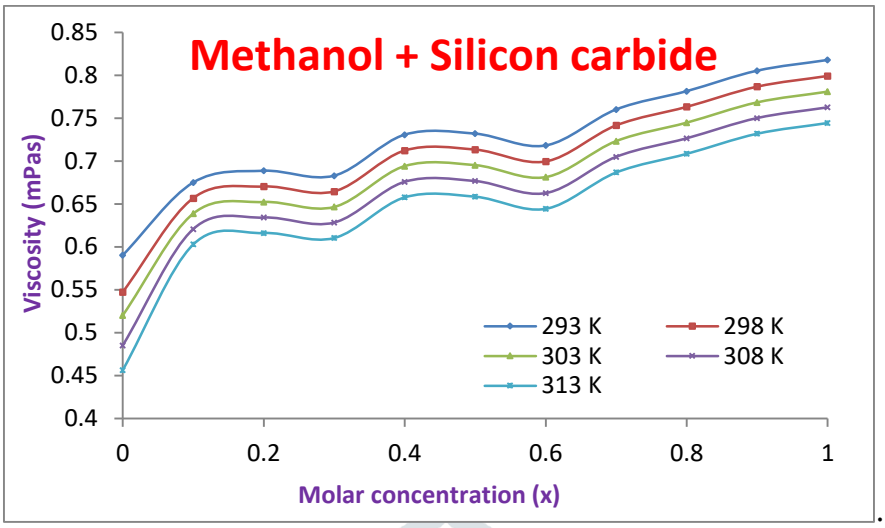

Fig. 6 Viscosity versus molar conc. of SiC nanoparticle in methanol base fluid

Conclusions

1. The structural, optical and ultrasonic properties of SiC nanoparticles and nanofluids are characterized by XRD, FTIR, ultrasonic velocity, density, adiabatic compressibility, and viscosity.
1. FTIR confirms that the SiC nanoparticles present around 604.72 $cm^{-1}$and 1078.95 cm^{-1}.
2. The ultrasonic velocity increases with increase in the concentration of SiC nanoparticles due to aggregation of boron SiC nanoparticles in methanol base fluid.
3. Ultrasonic velocity decreases with increase in temperature due to Brownian motion of nanoparticles in methanol based nano fluid and thermal agitation.
4. The non-linear variation of density, adiabatic compressibility and viscosity indicates the presence of phase separation in nano fluids.
5. It is confirmed that aggregation of nano particles in nano fluids plays a key role for association and hence enhancement of ultrasonic velocity.
6. These nanofluid samples can be used successfully for any heat transfer management systems in industrial applications.

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