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Synthesis and Thermodynamic Study of Ceramic Nanoparticles in Organic Based Solvent

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ABSTRACT

Silicon carbide (SiC) is a well-known ceramic type nanoparticle, it has been synthesized via sol-gel method. The size, morphologies and bonding states of synthesized SiC nanoparticles were investigated by X-ray diffraction (XRD), scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR). The results show that the synthesized SiC nanoparticles are high-quality crystals with high aspect ratios. The thermodynamics analysis of SiC nanoparticles in organic base fluids were studied with purpose of applications in various fields. X-ray diffraction (XRD) results indicated the major phases of SiC. Scanning electron microscopy (SEM) images shows that SiC particles mainly composed of crystalline nanoparticles. FTIR provides valuable and practical information about the chemical bond states of the materials. FTIR spectra of the SiC nanoparticles have revealed strong absorption bands with a very small variation. Average particle size of SiC nanoparticle has been estimated by using Debye-Scherrer formula. The thermodynamic properties of nanomaterials related to the surface of nanoparticles and nanoparticle surfactant interactions, these properties of SiC nanosuspension were studied by non-destructive technique.

Keywords : SiC nanoparticles; Sol-gel technique; Nanosuspension, XRD

Introduction

In nanoparticle synthesis it is very important to control not only the particle size but also the particle shape and morphology as well. In the present investigation, the synthesis of SiC Nanoparticles via solgel method and their thermodynamic studies in methanol-based solvent were discussed. Sol-gel technique is an easy, simple, cost effective and convenient route for preparing nanoparticles [1-6]. Silicon carbide is a well-known ceramic type nanoparticle which shows diverse scope due to its exclusive properties like high strength, hardness, chemical, thermal stability, high melting point, oxidation resistance, etc. Therefore, they used in high temperature electronic devices, abrasion and cutting

applications. Silicon Carbide Nanoparticles is known for its stability, refractory properties, wear resistance, thermal conductivity, small thermal expansion co-efficient, and resistance to oxidation at high temperatures. These traits make it ideal for applications across a wide range of domains, with more being uncovered by the research of academics and engineers. It exhibits characteristics like high thermal conductivity, high stability, high purity, good wear resistance and a small thermal expansion co-efficient. These particles are also resistant to oxidation at high temperatures.

Synthesis of Silicon Carbide Nanoparticles:

Silicon carbide nanoparticles have been synthesized by sol-gel method [7-9]. 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 in powder form.

XRD Pattern of Silicon Carbide Nanoparticles:

Figure 1 shows the XRD pattern of Silicon carbide (SiC) nanoparticles. The XRD measurement carried out by using "PAN analytical" X-ray diffractometer keeping the parameter constant at start position [°2Th.]: 10.0154 End Position [°2Th.]: 89.9834, Step Size [°2Th.]: 0.0170, Scan Step Time [s]: 5.7150, Scan Type: Continuous, Measurement Temperature [°C]: 25.00 Anode Material: Cu, K-Alpha1 [Å]: 1.54060. It is seen that the materials are well crystalline in nature and well agreed with standard JCPDS file number 029-1129. The estimate size of SiC nanoparticles using Debye Scherrer formula is found about 50 nm.

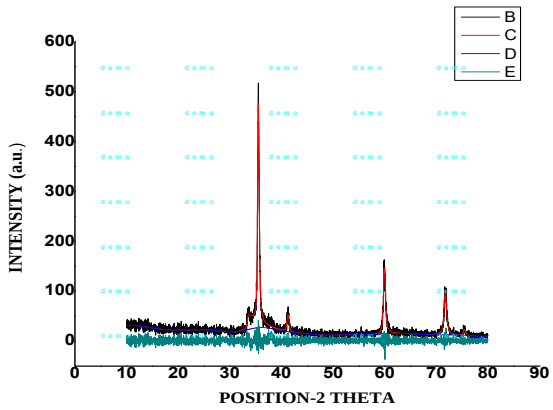

Fig. 1 X- ray diffraction pattern of Silicon carbide (SiC) nanoparticles

Results and Discussion:

The experimentally measured values of ultrasonic velocity, density and viscosity are used to derive thermodynamic parameters such as viscosity, adiabatic compressibility, Acoustic impedance, free length, free Volume, internal Pressure, isothermal compressibility, isothermal bulk modulus, molar compressibility, molar sound velocity, molar volume, Poisson ratio, relaxation time, Van der Wall's Constant, volume expansivity, effective mass and thermal conductivity. These parameters are represented are given in tables 1 (A) and 1 (B). In SiC Methanol based nanosuspension there might be nanoparticle fluid interaction favors in increase in ultrasonic velocity [10]. Rise in ultrasonic velocity may concluded as the strong interaction between nanoparticles of Sicon Carbide (SiC) and microsize molecules of methanol hence there is agglomerisation of SiC nanoparticles due to polar nature of methanol base fluid. Therefore sound will travel faster through the more compact structure by means of longotudinal waves [11-13].

Ultrasonic velocity gets increases with increasing the molar concentration of the SiC nanoparticles in methanol this shows that the physical parameters of the sample changes by increasing the molar concentration. Nanoparticles suspensions do not settle which provides a long self- life which imparts

ultrasonic velocity to them. For SiC nanoparticle the velocity of the nanofluid is higher than methanol and also by increasing the molar concentration of the SiC nanoparticle the velocity gets high values at 0.3 and 0.6. This represents strong aggregation of SiC nano suspension in methanol-based fluid at these molar concentrations. The cause behind this increase of ultrasonic velocity with increase in molar concentration (x) is due to strong interaction between nanosized particle and micro sized fluid molecule and also due to increase in density of nanofluid with increase of molar concentration. Ultrasonic velocity can be interpreted as the nanosized SiC particles have more surfaces to volume ratio and which can absorb more methanol molecules on its surface, which enhances the ultrasonic velocity. The decrease of ultrasonic velocity with increase in concentration is due weakening of interaction between SiC nano sized particles and micro sized fluid molecules of methanol.
Densities of the nanosuspension were 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. Nanosuspension has more density than methanol. Increase in density indicates the close packing between the SiC nanoparticles in methanol-based fluids.

When SiC nanoparticles suspended in methanol fluid, motion of particle becomes more rapid, lighter the particles, faster the motion and denser the particles slower the motion. Also suspended nanoparticles do not settle and they have long self-life. Hence, they can be easily suspended despite high solid density.
The densities of SiC nanoparticles in methanol based nanosuspension shows that the density steeply increases with increase in concentration. This increase in density decreases the volume indicating association of SiC nanoparticles in nanosuspension. It may be increase due to structural reorganization. The viscosity slightly increases with increase in molar concentration of SiC nanoparticles in methanol base nanosuspension. As the motion of nanoparticles becomes more rapid when the temperature of the medium was raised which lowers the viscosity of the medium as the size of the particles was reduced. Hence viscosity of nanosuspension decreases with increase in temperature.

The viscosity of SiC nanoparticle strongly depends on structure of SiC nanoparticles and consequently interactions between the SiC nanoparticles and molecules of the fluid. Thus, the viscosity depends on interactions between components of nanosuspension as well as on the size and shape of the SiC nanoparticles. Measurements of viscosity of nanofluids yield some reliable information in the study of nano cluster. The viscosity gives the strength of interaction between the interacting SiC nanoparticles and molecules of methanol. The interactions between SiC nanoparticles and molecules of methanol increase the viscosity of nanosuspension.
The adiabatic compressibility (β_a) decreases with increase in molar concentration indicating strong interaction between SiC nanoparticles and molecules of methanol showing agreegation of nanoparticles. The surface area of the material is increased by the reduction in particle size. Due to this higher percentage of the SiC Nanoparticles can interact with surrounding fluids. It may due to decrease in interspacing of SiC nanoparticles in nanosuspension with increase in molar concentration. It is a measure of association or dissociation or repulsion. It also determines the orientation of the nanoparticles in nanosuspension. Kiyohara and Benson [14] suggest that adiabatic compressibility is the result of several opposing effects like strong interactions between nanoparticles and molecules of the fluids also interstitial accommodation leads to a more compact structure and decreases adiabatic compressibility. The magnitudes of the various contributions depend mainly on the relative size of the SiC nanoparticles. Isothermal compressibility (β_i) and adiabatic compressibility (β_a) exhibits similar trend and both

decreases with increase in molar concentration of SiC nanoparticles in nanosuspension.

The relaxation time slightly decreases with increase in molar concentration of SiC nanoparticles indicating less stability of SiC nanoparticles in nanosuspension. It has high value at molar concentrations 0.3 and 0.6. For the nanoparticles in nanosuspension, the gravitational pull is not stronger than the random thermal motion of the particles hence nanoparticles do not settle which provides long self life at these molar concentrations which increases the relaxation time. Stability of SiC nanoparticles in nanosuspension is totally depends on its surface energy. Less surface energy more stable will be the SiC nanoparticles. The relaxation is caused by the energy transfer between translational and vibrational degrees of freedom and all these degrees take part in the process observed [15]. Free length is the distance between the surfaces of the neighboring SiC nanoparticles. The free length slightly decreases with increase in molar concentration up to 0.7 and then increases. The free length (L_f) of a nanosuspension is a measure of attraction between SiC nanoparticles and molecules of the methanol in nanosuspension. Decrease in free length is a result of increase in surface to volume ratio of SiC nanoparticles with methanol molecules, association through interactions between nanoparticles and molecules of the constituents of the nanosuspension.

The internal pressure (π_i) and free volume (V_f) versus molar concentration of SiC nanoparticles in nanosuspension represented that the internal pressure as well as free volume in nanosuspension is a measure of attraction between their constituents. In this nanosuspension, free volume decreases and internal pressure increases. Further, the increase in free volume and decrease in internal pressure with rise in concentration clearly show the increasing magnitude of interactions [16]. Such behavior of internal pressure and free volume generally indicates the association through interactions between SiC nanoparticles and molecules of methanol.

Acoustic impedance is found reciprocal to that of adiabatic compressibility [17-20]. It is in good agreement with the theoretical requirements. In this case, acoustic impedance increases at molar concentration 0.3 and 0.6. As the size of the material decreases, the percentage of surface atoms increases, hence large amount of substance comes in contact with surrounding materials. This results in increase in acoustic impedance. The increase in acoustic impedance (Z) with molar concentration can be explained on the basis of the interactions between components of the nanosuspension, which decreases the distance of their components.

The molar volume (Vm) of SiC nanoparticles depends on the structural arrangement and interactions between the components of the nanosuspension medium. The structural arrangement may be decided by the interaction forces in the nanosuspension medium. It is decreases with increase in molar concentration because the molecular weight is directly proportional to the molar volume. The molar volume is found to have similar type of trend as that of molar compressibiliry [21-30]. The change in Vander Waal's constant (b) would be due to a change in geometry of the SiC nanoparticles.

The sound velocity (R) increases due to increase in surface to volume ratio. It shows similar treands as that of ultrasonic velocity as it depends on it. The molar compressibility (W) decreases with increase in molar concentration of SiC nanoparticles in methanol based nanosuspension due to aggration of SiC nanoparticles in the medium. As it is function of adiabatic compressibility, its variation is similar as that of adiabatic compressibility. The isothermal Bulk modulus (B_i) increases with increase in molar concentration, it exhibits similar trends as that of ultrasonic velocity and inverse trend as that of

isothermal compressibility. The volume expansivity decreases with increase in molar concentration due to aggration of SiC nanoparticles in nanosuspension. As it is depends on internal pressure and isothermal compressibility it shows their resultant effects.

The effective thermal conductivity of nanosuspension increases with temperature. It has substantially higher values at 0.3 and 0.6. The thermal conductivity enhancements are highly dependent on specific surface area of nanoparticle, with an optimal surface area for the highest thermal conductivity. There is strong relationship between Brownian motion and temperature of nanoparticles. Furthermore, the effect of temperature on thermal conductivity is not very well understood and documented. It is reported that it shows the similar behavior as that of ultrasonic velocity.

Poisson's ratio σ depends upon adiabatic compressibility and isothermal Compressibility. It is ratio of lateral strain to longitudinal strain and its limiting values are in between 1 and 0.5.

Table: -1(A) Experimental values of thermodynamic Parameters at 5 MHz

X	U (m/s)	ρ (Kg/m^3)	$\eta*10^{-3}$ (NS/m^2)	$\beta a*10^{-10}$ (m^2/N)	$\beta i*10^{-10}$ (ms^2/kg)	$\tau*10^{-10}$ (s)	L_f*10^{-11} (m)	π_i*10^6 (N/m^2)	V_f*10^{-8} (m^3/mol)
293 K									
0.1	1410	799.16	0.6749	6.29	7.55	5.66	4.89	10.2164	6.42
0.2	1429	805.94	0.6886	6.08	7.31	5.58	4.81	10.0211	6.59
0.3	1515	804.45	0.6829	5.42	6.51	4.94	4.54	9.4166	7.55
0.4	1480	813.51	0.7307	5.61	6.73	5.47	4.62	9.6647	6.82
0.5	1458	812.37	0.7319	5.79	6.95	5.65	4.69	9.4829	6.88
0.6	1510	808.98	0.7181	5.42	6.51	5.19	4.54	8.9699	7.71
0.7	1468	822.89	0.7601	5.64	6.77	5.72	4.63	9.2308	7.01
0.8	1464	839.13	0.7814	5.56	6.67	5.79	4.60	9.2633	6.92
0.9	1470	851.39	0.8053	5.44	6.53	5.84	4.55	9.2495	6.86
298 K									
0.1	1420	796.92	0.6567	6.22	7.46	5.45	4.91	10.1944	6.76
0.2	1439	802.43	0.6703	6.02	7.22	5.38	4.83	9.9917	6.93
0.3	1525	801.36	0.6646	5.37	6.44	4.76	4.56	9.3929	7.94
0.4	1490	810.34	0.7123	5.56	6.67	5.28	4.64	9.6473	7.16
0.5	1468	809.37	0.7134	5.73	6.87	5.45	4.71	9.4662	7.22
0.6	1520	805.83	0.6995	5.37	6.44	5.01	4.56	8.9511	8.11
0.7	1478	819.21	0.7416	5.59	6.71	5.53	4.65	9.2143	7.35
0.8	1474	836.03	0.7632	5.51	6.61	5.61	4.62	9.2565	7.24
0.9	1480	848.59	0.7868	5.38	6.45	5.64	4.56	9.2469	7.18
303 K									
0.1	1429	793.77	0.6386	6.14	7.37	5.23	4.92	10.1625	7.12
0.2	1438	799.74	0.6522	6.03	7.24	5.24	4.87	10.0023	7.22
0.3	1534	798.11	0.6464	5.30	6.36	4.57	4.57	9.3658	8.35
0.4	1499	807.68	0.6941	5.49	6.59	5.08	4.65	9.6328	7.51
0.5	1477	806.39	0.6952	5.66	6.79	5.25	4.72	9.4492	7.57
0.6	1529	802.31	0.6812	5.31	6.37	4.82	4.57	8.9289	8.51
0.7	1487	816.58	0.7232	5.52	6.62	5.32	4.66	9.2042	7.71
0.8	1483	833.57	0.7447	5.44	6.53	5.40	4.63	9.2506	7.57
0.9	1489	845.45	0.7684	5.32	6.38	5.45	4.58	9.2405	7.51
308 K									
0.1	1437	790.62	0.6206	6.13	7.36	5.07	4.96	10.1283	7.49

0.2	1446	796.17	0.6341	6.01	7.21	5.08	4.91	9.9677	7.59
0.3	1542	795.37	0.6283	5.29	6.35	4.43	4.61	9.3403	8.78
0.4	1507	804.09	0.6758	5.48	6.58	4.94	4.69	9.6075	7.88
0.5	1485	803.09	0.6767	5.65	6.78	5.10	4.76	9.4251	7.95
0.6	1537	799.06	0.6626	5.30	6.36	4.68	4.61	8.9040	8.93
0.7	1495	813.52	0.7049	5.50	6.61	5.17	4.69	9.1892	8.07
0.8	1491	830.42	0.7265	5.42	6.51	5.25	4.66	9.2394	7.93
0.9	1497	842.36	0.7501	5.30	6.36	5.30	4.61	9.2330	7.84
313 K									
0.1	1440	787.38	0.6027	6.12	7.34	4.92	5.01	10.1049	7.85
0.2	1453	793.68	0.6161	5.97	7.16	4.90	4.94	9.9399	7.98
0.3	1549	792.22	0.6103	5.26	6.31	4.28	4.63	9.3091	9.24
0.4	1514	801.35	0.6577	5.44	6.53	4.77	4.71	9.5877	8.25
0.5	1492	800.06	0.6585	5.61	6.73	4.93	4.78	9.4025	8.34
0.6	1544	796.55	0.6443	5.27	6.32	4.53	4.64	8.8838	9.38
0.7	1502	810.08	0.6867	5.47	6.56	5.01	4.72	9.1696	8.45
0.8	1498	827.64	0.7084	5.38	6.46	5.08	4.69	9.2293	8.29
0.9	1504	839.96	0.7319	5.26	6.31	5.13	4.63	9.2292	8.19

Table-1(B) Experimental values of thermoacoustic Parameters at 5 MHz

X	$Z*10^{6}$ (kg/m^2s)	$Vm*10^{-2}$ (m^3/mol)	$b*10^{-5}$ (m^3/mol)	$R*10^{-4}$(m3/mol) $(m/s)^{1/3}$	$W*10^{-4}$ ($m^{19/7}/S^1$)	$Bi*10^{9}$ (N/m^2)	$\alpha*10^{-5}$ (1/K)	$T*10^{-3}$ (W/mK)	Meff (mol
293 K									
0.1	1.1759	4.1101	1.9929	486.59	8.48	1.32	2.63	3.50	32.846
0.2	1.2024	4.1755	1.9931	495.75	8.66	1.37	2.50	3.51	33.652
0.3	1.2149	4.2834	1.9935	530.08	9.03	1.54	2.09	3.65	34.458
0.4	1.2361	4.3348	1.9935	519.89	9.09	1.49	2.22	3.54	35.264
0.5	1.2597	4.4401	1.9934	516.28	9.27	1.44	2.25	3.43	36.070
0.6	1.2880	4.5583	1.9937	539.40	9.61	1.54	1.99	3.49	36.876
0.7	1.2990	4.5792	1.9936	525.19	9.59	1.48	2.13	3.39	37.682
0.8	1.3145	4.5867	1.9937	524.05	9.63	1.50	2.11	3.37	38.488
0.9	1.2987	4.6153	1.9938	527.29	9.72	1.53	2.06	3.37	39.294
298 K									
0.1	1.1797	4.1216	1.9929	490.51	8.52	1.34	2.55	3.51	32.846
0.2	1.2056	41938	1.9931	499.95	8.71	1.38	2.42	3.52	33.652
0.3	1.2165	4.2999	1.9935	534.26	9.07	1.55	2.03	3.67	34.458
0.4	1.2390	4.3518	1.9935	524.09	9.14	1.50	2.16	3.56	35.264
0.5	1.2622	4.4566	1.9934	520.46	9.32	1.45	2.18	3.45	36.070
0.6	1.2908	4.5762	1.9937	543.68	9.66	1.55	1.94	3.51	36.876
0.7	1.3010	4.5998	1.9936	529.56	9.65	1.49	2.07	3.40	37.682
0.8	1.3165	4.6037	1.9937	528.28	9.68	1.51	2.05	3.39	38.488
0.9	1.3015	4.6305	1.9938	531.46	9.77	1.55	2.00	3.39	39.294
..303 K									
0.1	1.1820	4.1380	1.9929	494.26	8.57	1.36	2.47	3.53	32.846
0.2	1.2068	4.2079	1.9930	500.16	8.73	1.38	2.39	3.51	33.652

0.3	1.2194	4.3174	1.9935	538.14	9.13	1.57	1.97	3.68	34.458
0.4	1.2406	4.3661	1.9934	527.83	9.18	1.52	2.10	3.57	35.264
0.5	1.2637	4.4730	1.9934	524.30	9.37	1.47	2.12	3.46	36.070
0.6	1.2929	4.5962	1.9937	547.69	9.71	1.57	1.88	3.52	36.876
0.7	1.3018	4.6146	1.9936	533.36	9.70	1.51	2.01	3.41	37.682
0.8	1.3196	4.6172	1.9937	532.02	9.72	1.53	1.99	3.40	38.488
0.9	1.3041	4.6477	1.9938	535.35	9.82	1.57	1.95	3.40	39.294
308 K									
0.1	1.1829	4.1545	1.9928	497.69	8.60	1.36	2.46	3.54	32.846
0.2	1.2037	4.2267	1.9929	503.69	8.78	1.39	2.37	3.52	33.652
0.3	1.2240	4.3323	1.9935	541.57	9.16	1.57	1.96	3.69	34.458
0.4	1.2422	4.3856	1.9934	531.44	9.23	1.52	2.09	3.58	35.264
0.5	1.2656	4.4914	1.9934	527.86	9.41	1.47	2.11	3.47	36.070
0.6	1.2946	4.6149	1.9937	551.30	9.76	1.57	1.87	3.53	36.876
0.7	1.3028	4.6320	1.9935	536.90	9.74	1.52	2.00	3.42	37.682
0.8	1.3230	4.6348	1.9936	535.57	9.77	1.54	1.98	3.41	38.488
0.9	1.3062	4.6648	1.9937	538.88	9.86	1.57	1.94	3.41	39.294
313 K									
0.1	1.1845	4.1716	1.9928	499.41	8.64	1.36	2.37	3.54	32.846
0.2	1.2057	4.2401	1.9930	506.66	8.81	1.40	2.27	3.53	33.652
0.3	1.2255	4.3495	1.9935	544.75	9.20	1.58	1.88	3.70	34.458
0.4	1.2422	4.4006	1.9934	534.51	9.27	1.53	2.00	3.59	35.264
0.5	1.2655	4.5084	1.9934	531.01	9.45	1.49	2.02	3.48	36.070
0.6	1.2944	4.6295	1.9937	554.40	9.79	1.58	1.79	3.54	36.876
0.7	1.3027	4.6516	1.9936	540.17	9.79	1.52	1.92	3.43	37.682
0.8	1.3236	4.6503	1.9936	538.68	9.81	1.55	1.90	3.42	38.488
0.9	1.3067	4.6781	1.9937	541.92	9.90	1.58	1.86	3.42	39.294

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