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The dispersion behavior of Si–C–N nanopowders in organic liquids

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Abstract

The dispersion behavior of laser synthesized Si–C–N nanopowders in 15 pure organic liquids has been studied through particle size analysis and sedimentation test. The Van der Waals potential energy of interaction between the particles and organic liquid molecules, i.e. V_A, is estimated. The results are correlated with the surface composition of powders and the chemical and physical characteristics of the organic liquids. Poor dispersions are found in nonpolar hydrocarbons, whereas in polar organic liquids, the dispersity and stability depend on the functional group, dielectric constant and carbochain length of the organic liquids. After some of the organic groups on the powder surfaces are eliminated by heat-treatment, the dispersity is decreased. The V_A of the dispersions can be adequately used to predicate the stability of the nonpolar hydrocarbon dispersions. However, in polar organic liquids, the contribution coming from the electric double layers and solvation layers should also be considered.

Keywords: A. Suspensions; B. Surfaces; Si–C–N

1. Introduction

In recent years, Si_3N_4/SiC nanocomposites have received increased attention due to their improved strength, toughness, hardness, creep, and oxidation resistance properties [1–3]. Niihara and coworkers have reported the fabrication of the Si_3N_4/SiC nanocomposites from the amorphous Si–C–N powders synthesized by CVD method. The materials exhibit superior mechanical properties [3]. The superplastic behavior was also found in Si_3N_4/SiC nanocomposites [4].

In the forming process, good dispersions are the key roles to achieve microstructure homogenous green, which lead to the improved sinterability and mechanical properties. However, nanoparticles are easy to form agglomerates due to their high surface free energies. One of the solutions is through colloidal process which can allow the particles to be kept in a suspension homogeneously and stably [5]. Several studies have been conducted on the dispersing of Si_3N_4 and SiC powders [6–10], but reports on the dispersion study of Si–C–N nanopowders are rare.

Some problems are encountered in aqueous media. For instance, water are not easily to volatile in the process of drying and different oxides (sintering additives) may have different iso-electric point (IEP) in water, which may cause flocculation. These problems can be drastically reduced by using non-aqueous suspension media [11]. However, owing to the relatively low polarity, it is usually difficult to achieve stabilization in non-aqueous media by the electrical repulsion, which can be easily realized in aqueous media. The physical barriers set up by the adsorbed polymeric molecules can be a good solution to the stabilization in many cases, but it also brings about problems. The removal of polymers by heating the green body at certain temperature is time consuming and it will leave residual contaminants, cracks and pores, which affect the microstructural evolution during sintering and hence the properties of the fabricated body. Bleier's study shows that in certain organic liquids, when Van der Waals potential energy of interaction $|V_A| < -5$ KT, it is possible to achieve stable suspensions by dispersing fine Si_3N_4 powders less than 20 nm without deflocculants [10]. In this study, the dispersion characteristics of Si–C–N nanopowders in various organic liquids were investigated and interpreted. The purpose is to find out some organic liquids for preparing nonaqueous dispersions with small agglomerates

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and good stability, and develop a fundamental understanding of the dispersion behavior in these liquids.

2. Experimental

Nanosized and amorphous Si–C–N powders were synthesized through CO_2 laser irradiation of hexamethyldisilazane (HMDS) and NH_3 mixtures. Details of the preparation method were described in Ref. [12]. Selected powder properties are listed in Table 1. All the organic liquids used in this study were analytic grade. Table 2 lists some physical properties of these liquids, including molar weight, density, refractive index, dielectric constant, and ionization potential (IP).

Table 1
Characteristics of Si–C–N nanopowders

	Parameter	Analysis
Particle size (nm)	30	TEM
Specific surface area (m^2/g)	130	BET
Morphology	Spherical	TEM
Crystal type	Amorphous	XRD
Size distribution	Narrow	PCS [a]
Composition (wt.%)		
C	20.1	Element analysis [b]
N	20.8	Element analysis [c]
Si	53.4	Chemical analysis
H and O	5.7	Calculated as the remainder

[a] Photo Correlation Spectroscopy, Zetasize3000 (Malvern Instrument Ltd, UK).

[b] LECO (Mod.LS-444LS).

[c] LECO(Mod.TC-436).

Table 2
Selected physical properties of organic liquids at 20°C[a]

Liquids	m	ρ(g/cm^3)	ϵ	n	IP (ev)
Methanol	32.04	0.7914	33.62	1.3288	10.85
Ethanol	46.07	0.7893	25.07	1.3611	10.47
n-Propanol	60.11	0.8035	20.79	1.3850	10.22
Iso-propanol	60.11	0.7855	18.96	1.3776	10.12
n-Butanol	74.12	0.8098	17.80	1.3993	10.06
n-Octanol	130.23	0.8270	10.34	1.4295	9.43 [b]
n-Hexane	86.18	0.6603	1.89	1.3751	10.13
Cyclohexane	84.16	0.7785	2.02	1.4266	9.86
Toluene	92.15	0.8669	2.38	1.4961	8.82
Acetone	58.09	0.7899	21.19	1.3588	9.70
Propionic acid	74.08	0.9930	3.35	1.3869	10.52
Acetic acid	60.05	1.0492	6.15	1.3716	10.66
N,N-Dimethylformamide	73.09	0.9080	36.7 [c]	1.4305	9.13
Benzaldehyde	106.13	1.0415	17.80	1.5463	9.49
Acetonitrile	41.05	0.7857	38.8 [c]	1.3442	12.19

[a] Ref. [13].

[b] Using the value of materials with the same molecular formula.

[c] Ref. [14].

Sedimentation tests were performed as follows. The dispersions in various organic liquids were prepared by adding amount of powders in the liquids without any deflocculants and then the mixtures were ultrasonicated for 10 min at the power of 100 W. The concentration of all dispersions was 0.5 vol%. After ultrasonication, the dispersions were poured into a clear 10 ml graduated test tube. The change of flocculated sediment height with time was recorded from 3 h to a week. The particle size analysis was performed by Zetasize 3000 (Malvern Instrument Ltd, UK) at 25°C, using diluted dispersions prepared under the above ultrasonication condition.

The infra-red spectra were obtained by the KBr pellet method using Bruker IFS 55 Fourier transform spectrophotometer. The heat-treatment experiments were conducted in vacuum (10^{-3} torr) at 1200°C for 2 h and in air at 550°C for 35 min, respectively.

3. Results and discussions

3.1. *The dispersity and stability in various pure organic liquids*

Table 3 summarizes the results of particle size analysis. Large agglomerates are found in hydrocarbons, i.e. hexane, cyclohexane and toluene. Medium sized agglomerates are found in benzaldehyde, acetonitrile and most of the alcohols. Propionic acid and acetone dispersed powders have small agglomerates. The best are *N,N*-dimethylformamide (DMF) dispersions. The minim particle size is approximate to the size of a single particle. The valuation of dispersion quality is listed in right column of Table 3, using ☆, assuming that the average particle size below 200 nm is very good, 200–300 nm is good, 300–500 nm is good to poor, and above 500 nm is poor.

Sedimentation tests were conducted to evaluate the stability of dispersions, since sedimentation tests are easily carried out and yield direct information concerning the technological uses of the dispersions [15]. The flocculation rate which can be indicated by the change of the upper level of the flocculated sediment was a good indicator to the stability of dispersions. Fig. 1 shows the change of flocculated sediment height in different organic liquids as function of time. Poor stability was found in hydrocarbons. Stratification took pace in half an hour after ultrasonication, and the sediment height in these dispersions was near the lowest point after 24 h. In organic liquids having polar groups (e.g. alcohol, ketone, carboxylic acid and aldehyde), the settlement rate was slower than hydrocarbons. The best stability of dispersions was found in DMF. The dispersions were constant in a week without any stratification. The second was propionic acid, but stratification took place after four days. Fig. 2 shows the stability of

Table 3
Particle size analysis in various organic liquids at 25°C

Liquids	Average size (nm)	Mode size (nm)	The minimal size (nm)	Valuation of dispersity[a]
Methanol	1034	1023	552	☆
Ethanol	725	850	381	☆
n-Propanol	545	565	210	☆
Iso-propanol	334	368	158	☆☆
n-Butanol	390	405	185	☆☆
n-Octanol	305	295	178	☆☆
n-Hexane	995	1045	410	☆
Cyclohexane	1080	856	424	☆
Toluene	1115	995	550	☆
Acetone	225	215	102	☆☆☆
Propionic acid	194	198	90	☆☆☆
Acetic acid	705	752	278	☆
N,*N*-Dimethylformamide	156	161	46	☆☆☆☆
Benzaldehyde	442	394	197	☆☆
Acetonitrile	360	339	213	☆☆

[a] ☆☆☆☆ very good; ☆☆☆ good; ☆☆ good–poor; ☆ poor.

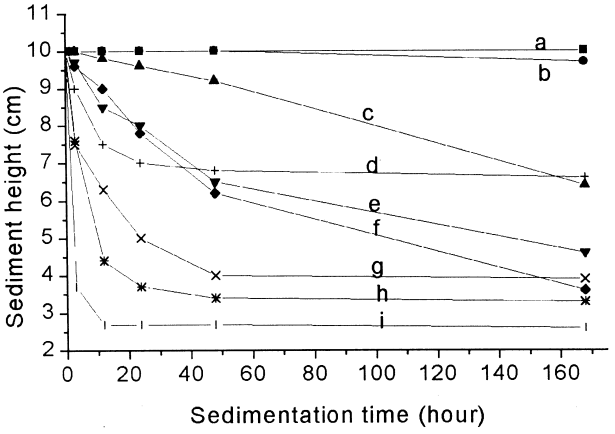

Fig. 1. The change of sediment height as function of time in different organic liquids: (a) DMF, (b) propionic acid, (c) acetone, (d) benzaldehyde, (e) acetonitrile, (f) ethanol, (g) acetic acid, (h) cyclohexane, (i) toluene.

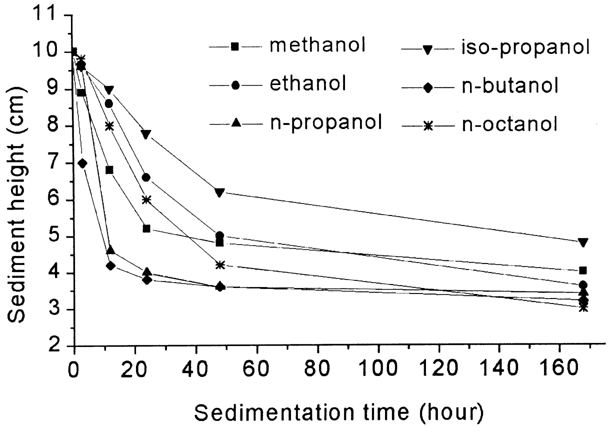

Fig. 2. The change of sediment height as function of time in alcohols.

dispersions in the same kind of organic liquids, i.e. alcohols. Dispersions of iso-propanol, ethanol and *n*-octanol have a slower settlement rate, whereas in methanol, *n*-propanol and *n*-butanol, the flocculated sediments settle fast. The stability is basically accordant with the particle size analysis, i.e. the more stable dispersions, and the smaller average particle size in the dispersions. With regard to the wetting ability, Si–C–N nanopowders have a good wetting ability to most of organic liquids. After a short time of ultrasonication, the agglomerates were separated by the liquids and formed constant dispersions. This can attribute to the organic groups that were produced from the laser synthesis processing on the powder surfaces (see curve a in Fig. 3: $-CH_3$ is at bands 1255 cm^{-1}, –CH is at bands 2919 cm^{-1}, the peak at 1633 cm^{-1} is the overlap of $-NH_2$ and –OH and the peak at 3443 cm^{-1} is due to $-NH_2$). While in the aqueous media, the powders cannot be wetted by the water, since the organic groups on the powder surfaces are hydrophobic.

Although it is expected that organic liquids with high dielectric constants can ensure good dispersions, it is not always the case. For instance, the dielectric constant of propionic acid is low (3.15), but the dispersity and stability in propionic acid are far better than in methanol and acetonitrile that have high dielectric constants. Therefore, the composition of powder surfaces and the functional groups of organic liquids should also be considered.

In dispersions of DMF, the $-NH_2$ on the Si–C-N powder surfaces and the functional group $-CONH_2$ of DMF have like structures so that a good compatibility is ensured. In addition, the powders can adsorb a layer of DMF molecules through the hydrogen bonds between the oxygen atoms on the –C=O of DMF and the hydrogen atoms on the $-NH_2$ of Si–C–N powder surfaces, which may curb the growing of agglomerates.

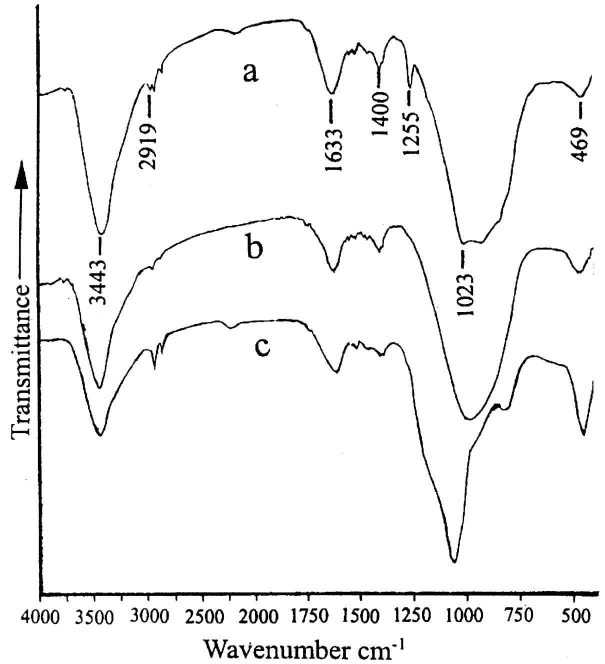

Fig. 3. The infra-red spectra of Si–C–N nano powders: (a) as-received, (b) heat-treated in vacuum at 1200°C, (c) heat-treated in air at 550°C for 35 min.

Thus, good dispersity and stability are expected in DMF dispersions.

Good dispersions in propionic acid can be explained in two points. On is that carboxy is the combination of carbxide and hydroxy, it is easier to form hydrogen bonds than alcohol and ketone. The other is that it has a proper length of carbochain and a low dielectric constant. It is not easy to hydrogen bond to itself, which decrease the stability of the dispersions. The poor dispersions in acetic acid may be due to the strong forming tendency of self hydrogen bonds, since the acidity of acetic acid is stronger than that of propionic acid.

Although acetonitrile has a high dielectric constant, it is not better than propionic acid and acetone. This may attribute to the poor ability to form hydrogen bonds of N compared with O, and the short chain of acetonitrile molecules that can not form effective steric hindrances.

In alcohols, with increasing carbochain length, the dielectric constant decreases, the viscosity increases and the average particle size decreases. The smallest average particle size was found in *n*-octanol. The fact that larger agglomerates existed in high dielectric constant liquids may be due to their stronger self hydrogen bonds forming abilities that decrease the interaction with the organic groups on powder surfaces. While in alcohols having a long carbochain, i.e. *n*-octanol, the high viscosity and the long carbochain can be good hindrances to the forming of large agglomerates after they are broken down into small agglomerates by ultrasonication. In alcohols with the same carbochain length, i.e. *n*-propanol and iso-propanol, they have a similar dielectric constant, surface tension and viscosity, but powders dispersed with iso-propanol have a slower settlement rate and smaller average particle size than *n*-propanol. This suggests that the position of the functional groups (–OH) in the carbochain also has effects on the dispersity and stability of the dispersions.

3.2. Effects of the surface composition change on the dispersity

Surface compositions greatly influenced the dispersion behavior of powders. For instance, the IEP of Si_3N_4 has a close relationship with the number of amino and silanol groups on the powder surfaces [16]. Hoffman et al. have also reported that the dispersion quality could be improved by preoxidating Si_3N_4 powders in air, which enhanced the specific absorption of ammonium polyacrylate [17]. To investigate the effects of surface composition change on the dispersity, Si–C–N nanopowders were heat-treated in vacuum at 1200°C for 2 h (sample b) and in air at 550°C for 35 min (sample c) respectively.

The infra-red spectra (Fig. 3) illustrate the composition change of powder surfaces after being heat-treated. In sample b, the $-CH_3$ at 1255 cm^{-1} and –CH at 2919 cm^{-1} all disappeared, which indicates that $-CH_3$ groups have escaped from the powder surfaces, therefore the H contents in the powder surfaces were greatly reduced. In sample c, The broad peak which is the combination of Si–C(820 cm^{-1}), Si–N (950 cm^{-1})and Si–O (1080–1100 cm^{-1}) has divided into two peak and shift to the Si–O absorption band. This indicates that more Si–O bonds exist on the powder surfaces. The intensified C–H vibration peak at bands 2919 cm^{-1} can also attribute to the formation of Si–O–C bonds, which improved the electronegativity of carbon, thus the interactions between C and H are intensified.

Table 4 summarizes the average particle size of samples b and c in some of the organic liquids. Compared with the powders without heat-treatment (Table 3), the average particle size of sample b in nonpolar organic liquids, i.e. cyclohexane, neither decreased nor increased much. However, in the polar organic liquids, i.e. propionic acid, DMF and acetone, the average particle size greatly increased, especially in propionic acid. Although the average particle size in methanol and ethanol decreased, the agglomerates were still above 300 nm. Sample c shows poor dispersity in all selected organic liquids.

The decreased dispersity of sample b in most of the organic liquids may due to two reasons. One is that the organic groups on the powder surfaces were eliminated by the heat-treatment, so the powders can not interact effectively with the polar functional groups of organic liquids and form adsorbed solvent layers to prevent the growing of the agglomerates. The other reason is that the powders have relatively high content of carbon (20.1

Table 4
The average particle size of heat-treated powders in selected organic liquids at 25°C

Liquids	Average particle size of sample b (nm)	Average particle size of sample c (nm)
Methanol	378	432
Ethanol	489	536
Iso-propanol	373	395
Cyclohexane	1196	1234
Propionic acid	704	948
N,N-Dimethylformamide	346	628
Acetone	422	813

wt.%). After being heat-treated, some of the free carbons were produced on the powder surfaces. The layer of carbon can be a hindrance to the separation of agglomerates by the liquids. The decreased stability of the dispersions due to excess of free carbon has also been reported elsewhere [18]. The poor dispersity in dispersions of sample c can attribute to the excess of Si–O bonds on the powder surfaces, which increased the polarity of powders. Therefore, the powders are incompatible with most of the organic liquids, especially those with low dielectric constants.

3.3. Estimation of Van der Waals potential energy of interaction V_A

London–Van der Waals interaction V_A, is always present and always attractive between like particles [15] and is an important parameter in colloidal suspensions. In this section, the values of V_A between Si–C–N and various organic liquids are estimated using Hamaker's expression for two particles with the same radius [19]:

$$V_A = -\frac{A_{131}}{6}\left[\frac{2a^2}{H^2+4aH}+\frac{2a^2}{H^2+4aH+4a^2}+\ell n\left(\frac{H^2+4aH}{H^2+4aH+4a^2}\right)\right]$$

H is the distance between the surface of two particles, a is the radius of the particle and A_{131} is the effective Hamaker constant for two particles of materials 1 (Si–C–N) through a medium of materials 3 (organic liquid).

Hamaker's original treatment is also called microscopic approach [20]. An alternative approach has been derived by Lifshitz. This approach treats each body as a continuum with certain dielectric properties and incorporates many-body effects. But the expression of A_{131} is complex and the approach is restricted to those materials for which optical data are available. Gregory [21] related the Hamaker constant directly to the static dielectric constant of two bodies of materials i:

$$A_{ii} = 0.23h\nu_\nu\left[\frac{(\varepsilon_{0i}-1)^2}{(\varepsilon_{0i}+1)^{3/2}(\varepsilon_{0i}+2)^{1/2}}\right] \tag{2}$$

ϵ_{0i} is the static dielectric constant of materials i, h is the Planck's constant and υ_υ is the characteristic frequency of molecules comprising materials i. The $h\upsilon_\upsilon$ can be substituted by the ionization potential [20]. The ionization potential of various organic liquids used to estimate the A_{33} are listed in Table 2. The expression for A_{131} is given by a approximate approach [20]:

$$A_{131} = A_{11} + A_{33} + 2(A_{11}A_{33})^{1/2} \tag{3}$$

The problem encountered here is that the υ_υ and ϵ_{0i} of Si–C–N are not available in the literatures. However, we can use the values between Si_3N_4 and SiC in Ref. [22] to estimate those of Si–C–N, since the final sintered body are consist of Si_3N_4 and SiC. Here the characteristic frequency of Si–C–N is taken as 1.4×10^{16}rad/s and the static dielectric constant is 9.0. The calculated A_{11} of Si–C–N is 51.22 kT at 20°C. The diameter of the particle used to calculate V_A is 30nm.

Table 5 summarizes the values of A_{33}, A_{131} and V_A. The distance between two particle surfaces, i.e. H, is taken as the twice the diameters of the organic liquid molecules, which is a critical position prior to the encounter of two particles. The diameters of organic liquid molecules were calculated from the molecule volume using the parameters m and ρ listed in Table 2 under the assume that the molecule is spherical.

In most cases, the total potential energy of interaction V_T, can consist of three components, i.e. (1) V_A [19], (2) V_{DL} which comes from the interactions of the electrical double layers between two suspended particles [23], and (3) V_S which comes from the "steric effects" due to the adsorbed surfactant layers [24]. In our discussed dispersions, since no surfactants or deflocculants are added, we can assume that V_S is absent.

In nonpolar dispersions, it is difficult to develop surface charges on the particles. Therefore, the contribution of V_{DL} is very small and V_A equals approximately to the V_T. In other words, V_A is dominant in the stability determining factors and it can be used to predict the stability of the dispersions without considering other items. If we assume $V_T < -5$ kT is favorable for the forming of agglomerates, poor stability is expected in these dispersions. The calculated V_A is accordant with the poor stability in these dispersions discussed previously.

However, the case is complex in polar organic liquids. In dispersions of DMF and acetone, the $|V_A|$ is small, and the stability is also good, whereas in dispersions of propionic acid, the V_A is more negative than that of ethanol, but the stability of former is far better than that of latter. Therefore, using V_A to predict the stability of

Table 5
London–Van der Waals Hamaker constants and potential energy of interaction in the unit of kT between Si–C–N particles at 20°C

Liquids	A_{33}	A_{131}	V_A
Methanol	86.37	4.57	−3.83
Ethanol	79.65	3.13	−2.24
n-Propanol	74.96	2.25	−1.44
Iso-propanol	72.71	1.88	−1.19
n-Butanol	71.21	1.64	−0.96
n-Octanol	55.78	0.10	−0.05
n-Hexane	7.53	19.47	−9.68
Cyclohexane	8.90	17.42	−9.44
Toluene	11.74	13.92	−7.60
Acetone	71.49	1.69	−1.09
Propionic acid	25.19	4.57	−2.93
Acetic acid	47.24	0.08	−0.06
N,N-Dimethylformamide	73.50	2.01	−1.24
Benzaldehyde	67.15	1.08	−0.60
Acetonitrile	98.80	7.74	−5.81

these dispersions is not adequate, and V_{DL} should be considered. At least two approaches for generating surface charge are involved in discussed dispersions. One is the adsorbing the H^+ and OH^- dissociated by little amount of water existing in the organic liquids, since our solvents did not undergo drying treatments. The other is through surface reaction. For instance, the −COOH of propionic acid can react with the $–NH_2$ of Si–C–N through Bronsted and Lewis mechanisms. Thus the V_{DL} produced from the electric double layers may overcome the V_A and make the V_T positive.

The other factor should be considered is the interactions between particle surfaces and functional groups of organic liquids. Because the strong mutual interactions can make the particles adsorb a layer of solvent molecules. Provided no desorption occurs, the two particles will be kept at approximately twice the thickness of the adsorbed layer so that V_A will not further increases. The forces produced by the solvate layers are also called the solvation forces. Some attempts have been made to offer theoretical explanations for experimental effects about these forces [25], but there is no general theory of such forces, since they depend on specific features of particular surface and solvent involved [26].

4. Conclusions

1. Si–C–N nanopowders dispersed with nonpolar hydrocarbons show poor dispersity and stability, whereas in polar organic liquids, the dispersion behavior is determined by the physical and chemical properties of the liquids, i.e. dielectric constants, functional groups, carbochain length and etc. The most stable dispersions and the minimal average particle size was found in powders dispersed with DMF. Good dispersions are also found in propionic acid that has low dielectric constant.
2. The dispersity was decreased in most of the polar organic liquids after some of the organic groups on powder surfaces were eliminated by heat-treatment. This indicates that these organic groups on the powder surfaces play an important role in the dispersion behavior of Si–C–N nanopowders. Excess of Si–O bonds on the powder surfaces lead to a poor dispersity in almost all the organic liquids, although it is slightly better in alcohols.
3. In absence of deflocculants, it is adequate to use Van der Waals potential energy of interaction, V_A, to predicate the stability of dispersions in nonpolar hydrocarbons. However in polar organic liquids, the potential energy of interaction comes from the electrical double layer and solvation layer should be considered, since strong interactions can exist between powder surfaces and functional groups of organic liquids.

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