Asian Journal of Chemistry; Vol. 29, No. 9 (2017), 1981-1984

ASIAN JOURNAL OF CHEMISTRY

<https://doi.org/10.14233/ajchem.2017.20698>

Effects of Milling Parameters on Rheological Behaviours of Silica Nanofluid Prepared by Two-Step Process

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Received: 10 April 2017;

Accepted: 31 May 2017;

Published online: 15 July 2017;

AJC-18479

Nanofluids have garnered the attention of material science and heat transfer fraternity across the world for the reported enhancement of their thermal properties and the amenability for a change of their rheological properties under external influence. Of the various nanofluids that have been studied widely, nano-silica fluids comprise a predominant fraction. The alterations in the morphological characteristics of the nano-silica fluids brought about by mechanical treatment of the samples influence their chemical and rheological behaviours. The present work involves the study of milling parameters on the rheological behaviours of silica particles. The micron size silica is reduced to nano size by ball milling, done by 0-20 h at 300 rpm in Fritsch P5 planetary ball mill. This milled powder will be characterized by XRD and SEM for its crystallite size and morphology. Thus prepared silica on treatment with ethylene glycol and water mixture enhances its dispersion in fluids.

Keywords: Silica nanofluid, Rheological properties, Ball milling, Two-step process.

INTRODUCTION

Nanofluidics is a new class of fluid engineered by the dispersion of high purity of nano-sized material into the base fluid. Also, known that the nano-size colloidal suspension contains condensed nano-size material. Nanofluids are two phase system with one phase of the solid phase and in another liquid phase. It is found to enhance the thermal and physical properties like thermal conductivity, thermal diffusion, viscosity and conductive heat transfer coefficient compared to those of base fluids like water or engine oil. It has huge potential application in the automobile industry, electronic industry and other fields. The two-phase system, few issues should face in day to day life. To achieve desired stability of nanofluid remains a big challenge. Preparation of method of nanofluid mainly classified into different methods like two step method, one step method and another novel method. In two-step method, nano-size powder first produced as a dry powder and then it will be dispersed into base fluid in the second step with help of proper dispersion method. It is a most economic method to produce nanofluid in large industry scale. Some cases surfactants were used to enhance the stability [1-6].

Eastman *et al.* [7] developed a one step physical vapour condensation method for the one-step method. In this method, consist of simultaneously making and dispersing the particles

in the fluid and it is avoiding the process of drying, storage and transportation. This will lead to a reduction of agglomeration and increase the stability. Due to the stability problem, researchers developing a new novel method to produce nanofluids. Wei and Wang [8] developed a continuous flow of microfluidic-microreactor to synthesize copper nanofluids. In this method, copper nanofluids will be continuously synthesized and their microstructure and properties can be varied by adjusting parameters like reactant concentration, flow rate and additive, temperature, acidity (pH), ultrasonic and microwave irradiation, types and concentrations of reactants. One-step chemical solution method (CSM) has been recently developed for creating nanofluids, which have the capable of synthesizing nanofluids of various microstructures. The nanofluids produced by the chemical solution method have both enhancement of higher conductivity and higher stability than those produced by the other methods [9-12].

One of the main problems of the one-step method is can't able to produce in large scale and cost of production is high. The stability is a key issue that influences the properties of nanofluids for application and it is necessary to study the influencing factors of nanofluids. The stability evaluation methods, different ways to enhance the stability and the stability mechanisms are the different parameters. The present work involves the study of milling parameters on the rheological behaviours of silica particles.

EXPERIMENTAL

For the preparation of the nanofluid, two-step methods were using. Micron size silica particle was synthesized following by the mechanical alloying route. It allows the production of a homogenous dispersion of nanopowder. The initial process of mechanical alloying is starting with the right proportion of silica powder and loading the powder sample along with grinding ball in high energy ball mill. The initial precursor is SiO_2 micron size powder with 140 mesh size (99.05 % purity) were mechanical alloying at normal room temperature. Using high energy Fritsch P5 Planetary Ball Mill with WC grinding medium at 300 rpm and 1:10 powder to ball ratio were using. The milling was carried out at in wet medium like toluene, for the desired period of time like 5, 10, 15 and 20 h. After the grinding, the powder will dry in natural room temperature. The prepared powder will be characterized by X-ray diffraction (XRD), field emission scanning electron microscope (FESEM) and energy dispersive spectroscopy (EDS) to identify the phase, particle/crystallite size, shape/morphology and purity.

Mechanical alloying based dried silica powder will be dispersed into the fluid in the second processing step with the help of magnetic force and ultrasonic agitation. Up to 1 vol. % of dry powder of SiO_2 will be dispersed in ethylene glycol and deionized water mixture, by the help of ultrasonic bath and magnetic stirring for effective dispersion into ethylene glycol mixture. The prepared nano silica fluid will be characterized by zeta potential analyzer.

RESULTS AND DISCUSSION

The ultrasonication processing time was set for 30 min for preparing for nanofluid and magnetic stirring was carried out for 60 min. X-ray diffractometer (X-pert Pro, Panalytical) was used to investigate the phase of material and obtained as amorphous state silica. The analysis was performed using a Cu target (voltage and current of 40 kV and 20 mA, respectively), with a scan range of 0 to 100° and speed of 1° for a minute. The XRD intensity shows that the grinding hours increasing with intensity (Fig. 1). The XRD pattern of SiO_2 shows a strong peak at $2\theta = 23.68^\circ$ attributed to the (002) crystalline plane (Fig. 1).

Fig. 2 shows the different milling time intervals image of field emission scanning electron microscope (FESEM – CARL ZEISS, Supra 40VP, 2009). It indicates that particle size is reducing by increasing the milling time also the agglomeration chances are getting increasing. The EDAX analysis is confirmed the higher purity of SiO_2 because of the absences of other material (Fig. 3).

Coolants with higher thermal characteristics than water need to be applied for better cooling. Nanofluid is a potential coolant for heat transfer application such as fuel cell thermal management and electronic device cooling and automobile sector [13]. The presence of nanoparticles in the base fluid increase the thermal conductivity behaviour. It is an important property in heat transfer. However, nanofluid also shows higher viscosity and electrical conductivity as the particle concentration is increased across temperature. High viscosity correlated to the pressure drop and pumping power.

Fig. 1. XRD image of SiO_2 micro/nanopowder as synthesized at different time intervals ($2\theta = 23.68$ attributed to (002) crystalline plane)

Fig. 2. FESEM image of SiO_2 nano/micro powder after different milling time intervals, A: 5 h, B: 10 h, C: 15 h, D: 20 h

El	AN	Series	unn. c [wt %]	norm. C [wt %]	Atom. C [at %]	Error	(1 Sigma) [wt %]
O	8	K-series	45.69	64.38	76.04		8.42
Si	14	K-series	25.28	35.62	23.96		1.22
Total			70.97	100.00	100.00

Fig. 3. EDAX image of prepared SiO_2 micron/nano size powder

TABLE-1
PROPERTIES OF NANOPARTICLES AND BASE FLUIDS

Nanoparticle/base fluid	Thermal conductivity (k) (W/m K)	Electrical conductivity (σ) (μ S/cm)	Dielectric constant (ϵ)	Density (ρ) (kg/m^3)	Reference
SiO ₂	1.380	10.00	3.9	2220	[14]
Distilled water	0.615	6.00	80.0	999	[15]
Ethylene glycol	0.252	1.07	38.0	1110	[16]

Among all prepared 4 different set of silica nanofluids, 20 h milled SiO₂ nanofluid shows less particle size [Fig. 5(b)]. Based on the results, if the particle size is reduced the effective thermal conductivity will increase so that further characterization were carried out in 20 h milled SiO₂ nanofluid.

Properties of nanoparticle and base fluid used in the experiment listed in Table-1. The thermal conductivity, electrical conductivity and viscosity are measured at room temperature. The thermal conductivity, viscosity and electrical conductivity are measured using KD2Pro Thermal Analyzer, Zetasizer Nano ZS (Malvern Instruments Ltd., UK), Brookfield DVIII Ultra Programmable Rheometer and Cyberscan PC-10, respectively. The effective thermal conductivity of ethylene glycol and water mixture is shown to be increased up to 30 % by the addition of a nanofluid consisting of approximately 0.35 volume % SiO₂ nanoparticles of mean diameter 78 nm.

First, significant increases in thermal conductivity are seen for SiO₂ nanofluids, with conductivity enhancements of up to 30 % observed for particle loadings well up to one volume percent compared to fresh ethylene glycol and deionized water mixture fluid with nanoparticle addition (Fig. 4). As compared with fluids containing SiO₂ nanoparticle without particles, showed there is an improvement in thermal conductivity.

The sample was milled for 2 h in ethylene glycol and deionized water mixture nanofluid will further characterize because of the thermal conductivity improvement. The milling time is increasing, with respect to the increasing absorption

Fig. 4. Thermal conductivity of prepared SiO₂ nano particle in different medium

[Fig. 5(a)]. Milling time vs. particle size distribution shows the milling time increases with decreasing of the particle size distribution [Fig. 5(b)]. It was determined by using a Zetasizer Nano ZS (Malvern Instruments Ltd., UK). The viscosity of

Fig. 5. (a) Milling hours vs. absorption (nm) of prepared SiO₂ micro/nanofluid; (b) Milling hours vs. particle size distribution SiO₂ micro/nanopowder; (c) Milling hours vs. viscosity of prepared SiO₂ micro/nanofluid; (d) Milling hours vs. conductivity of prepared SiO₂ micro/nanofluid; (e) Milling hours vs. zeta potential of prepared SiO₂ micro/nanofluid

the nanofluid is increasing within increasing milling time, for 20 h milled nanofluid samples viscosity is getting maximum of 0.8875 cp [Fig. 5(c)]. If the temperature increases simultaneously, the viscosity decreases. Zeta potential and conductivity showing the same trend like increasing milling time with increasing conductivity and zeta potential [Fig. 5(d,e)], for 20 h nanofluid sample shows the conductivity of 0.255 ms/cm and zeta potential of -7.65 mv, respectively.

Conclusion

In summary, the 20 h milled silica dispersed ethylene glycol mixed deionized water nanofluid shows better rheological behaviours compared to other samples. Better significant increases in thermal conductivity are seen for SiO₂ nanofluids, with conductivity enhancements of up to 30 % observed for particle loadings well up to one volume percent. As compared with fluids containing SiO₂ nanoparticle without particles, showed there is an improvement in thermal conductivity in the case of SiO₂ nanoparticle addition. The prescribed protocol is very economical compared to the protocol for preparing nano-fluid in large scale. The rheological behaviours strongly depending upon the process parameters like size shape of the particle, viscosity, conductivity and also the nature of dispersion medium. High viscosity shows more chances of aggregations.

ACKNOWLEDGEMENTS

The authors acknowledge to Department of Physics and Nanotechnology, SRM University, Kattankulathur, Chennai, India for providing the research facilities.

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