

# **Synthesis and characterization of metallic nanofluids**

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**2015**

# **CERTIFICATE**

This is to certify that the thesis entitled “Synthesis and Characterization of Metallic nanofluids” submitted by Sovan Mishra (111MM0369) and Turyansu Subhadarshy (111MM0381) in partial fulfilment of the requirements for the award of Bachelor of Technology in Metallurgical and Materials Engineering at National Institute of Technology, Rourkela is an authentic work which has been carried out by them under my supervision and guidance.

To the best of our knowledge, all the matter embodied in this thesis has not been submitted to any other university/ institute for award of any Degree or Diploma. This work, in our opinion, has reached the standard of fulfilling the requirements for the award of the degree of Bachelor of Technology in accordance with the regulation of the institute.

(Prof. A. Basu)

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# **ABSTRACT**

Nanofluids, which are basically the colloidal fluid suspensions of nanoparticles in a base fluid, have shown varied interesting properties, and the distinctive features they possess unprecedented and highly diversified potential for many applications. The present study deals with the synthesis of copper nanofluid, the copper nanoparticles being achieved by the process of mechanical milling. For evaluating the various properties and comparing the stability of the nanofluid two different base fluids are used, i.e. water and ethylene glycol. The properties of the nanofluid are estimated via several characterization methods such as XRD analysis, SEM analysis, thermal conductivity measurement, *pH* measurement, and Zeta potential measurement. With the characterization done, trends in properties are observed with the change in volume fraction of nanoparticles.

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# **CHAPTER-1: INTRODUCTION**

The particles which have sizes in the nanometer range, basically in the spectrum of 1-100 nm are vividly referred to as nanoparticles. When these particles in the nanometer size range form dilute liquid suspensions with one of the base fluids, they are called as nanofluids. These are fluids which contain nanoparticles which could be either oxides, carbides, or nitrides or nanotubes or even nanometer-sized metal particles. On the basis of foregoing investigations, these have been deduced to possess excellent thermo-physical properties which at times are aptly termed as ‘enhanced’, viz. viscosity, thermal conductivity, diffusivity and a varied other crucial properties, as compared to their base fluid with which the suspension has been prepared.

Possessing a quite distinctly rich history as far as the case of grounded colloidal science goes, and when the particle synthesizing techniques are taken into consideration, these fluids have been very proficiently engineered for a varied and ever increasing spectra of applications. These fluids, basically based on the theory that the Brownian agitation opposes any kind of motion due to gravitational settling, lead to the basic fact that a stable nanofluid is actually possible until the particles are small enough (as already specified i.e. <100 nm). However, maintaining such a miniature size can be a humongous challenge considering the fact that the particles keep coming into contact with each other, rather frequently, thus leading to the obvious formation of large size agglomerates that can settle out of the basic suspension. “Nanofluid”, as the term aptly indicates, is a mixture in which properties of both the base fluid and the nanoparticles rightly contribute to the desired application of the system. Thus, a nanofluid is basically created when the dispersion of nanoparticles into a base fluid takes place under controlled conditions, thus leading to enhancement of its requisite properties.

# **CHAPTER-2: LITERATURE REVIEW**

## **2.1 Coolant and their properties**

A fluid which basically flows through or about a device in order to prevent the overheating, by virtue of transfer of heat produced to other devices which either use the heat or dissipate it is referred to as a coolant. This given fluid can either retain its phase or undergo a phase transition, with the resultant latent heat adding up to increase the overall cooling efficiency. The latter, when used in order to achieve low temperatures, is referred to as a refrigerant. It is widely referred to as a heat transfer fluid in the technical sense, being used in both high and low temperature applications. Solids, gases and liquids can be taken up as coolants, viz. air, hydrogen, inert gases, sulphur hexafluoride, deionized water, oils, etc. provided they have the desired characteristics. Nowadays, a new class of materials, nanofluids are being used extensively because of their superior thermal properties. Thus, it is quite necessary to have an idea about the desired properties in a material in order to use it as a coolant, which is listed below:

1. High thermal capacity- Must have considerable thermal properties, as in high heat retention capacity and has high heat transfer properties.
2. Low viscosity
3. Cost in the affordable range, basically low-cost.
4. Non-toxic and basically inert, in order to prevent reaction with the surrounding material or metal to which it is applied and also serve as an eco-friendly material.
5. Should neither cause nor promote corrosion of the associated cooling system.

## **2.2 Nanoparticle**

The particles which range between 1 to 100 nanometer size spectrum are known as nanoparticles. This field has developed recently as an area where intense research is being carried out all around the globe and it's because of its widespread applications in almost all fields of life, ranging to biomedics to optical and electrical sectors. Even the National Nanotechnology initiative has been taken up in the United States for public funding to carry out high-end research

in the field. These particles are in reality a link between bulk materials and the materials in the atomic or molecular level. The interesting and at times surprising properties of these particles is mainly due to the high surface area these possess, which mainly dominates all the contributions by the smaller bulk of material. These possess superior optical properties because of their ability to confine the electrons, regardless of their small size. Thus, their small size has led them to act as prolific materials for research and the research has led us to get some unexpected results, viz. superior thermal conductivity and other thermal properties, low viscosity, etc. which have led them as a one-step solution for many commercial applications.

### **2.3 Nanofluid**

A fluid containing the nanoparticles in order to form a colloidal suspension with another base-fluid is called nanofluid. These are basically made of oxides, nitrides, etc. and the base fluid most commonly used are water, ethylene glycol, or oil, etc. These fluids have varied novel properties which make them highly essential for heat transfer applications which are described as follows:

### **2.4 Heat Transfer Applications**

Heat transfer technology, as it signifies, stands rightly at the cross roads of two historic developments that have occurred very significantly in the present situation, i.e. miniaturization on one side and an even astronomical rise in heat flux at the other. As is clear, the ever increasing demand for heat removal in some crucial processes, which mainly involve, electronic chips, or similar high energy devices like lasers. The usual limiting factors which are the roadblocks in this regard are manifold. One major part being the vicariously poor thermal characteristics of the usual heat transfer fluids. Being nearly about two orders of magnitude less efficient as compared to their metal counterparts when the case of conductance is taken into consideration, this makes the heat removal mechanism much less effective even with utilization of their highly classic flow properties.

### **2.4.1 Obstacles**

The extremely high thermal conductivity of solids can be used for increasing the thermal properties, viz. thermal conductivity of a fluid, mostly by addition of small solid particles to it. The investigations by various researchers, concerning the usage of such solid particles suspensions, with varying sizes, nearly on the order of 2 millimetres or micrometres, led to the following crucially significant drawbacks (Das et al., 2006):

1. The rapid settling of particles, mostly leading to the formation of a defining layer on the surface itself and subsequent reduction of the fluid's heat transfer capacity.
2. Reduction of sedimentation, in case the fluid circulation rate is rapidly increased, nevertheless leading to the erosion of the heat transfer devices, viz. pipelines etc. quite rapidly.
3. The clogging of the flow channels due to the extremely large particle size, particularly when the cooling channels tend to be narrow.
4. Considerable increase of the pressure drop in the fluid.
5. Finally, considerable enhancement in conductivity based on particle concentration (i.e., greater the enhancement in case of higher volume fraction of the particle, leading to greater intensity of the problems mentioned above.)

Thus, even if well-known, the process route of suspending particles was a rejected option as far as transfer applications are considered.

However, the wholesome emergence of materials technology proved as a saviour, providing the opportunity of producing particles in the nanometer range, significantly different from the parent material in terms of mechanical, electrical, optical, thermal and other physico-chemical properties.

### **2.4.2 Advantages**

Nanofluids, over all these years have been rightly considered, provided the stark prominence they possess, because of their vividly varied properties. Most of all, they have been utilised as advanced heat transfer fluids, almost about two decades now. But, the wide variety and the

extreme complexities of the nanofluid systems, have led to no agreement regarding the extent of potential benefits in using these fluids for the concerned heat transfer applications.

But even though being surrounded by such complexities, these systems possess some very lavish advantages, when compared to their solid-liquid suspension counterparts, when heat transfer is taken into consideration:

1. They possess an extremely large specific surface area and thus, heat transfer surface is more between the particles and the fluids.
2. Extremely high dispersion stability along with a highly predominant motion of particles, rightly called Brownian motion.
3. Reduction in pumping power compared to the pure liquid systems, in order to achieve nearly equivalent heat transfer operations.
4. Reduced particle clogging as compared to conventional slurries, thus leading to widespread system miniaturization.
5. The crucial properties, viz. thermal conductivity and wettability, can be very proficiently adjusted by varying the particle concentrations in order to suit the varied spectrum of applications.

## **2.5 Recent studies on nano fluid**

**Siva V. Manoja** *et al.* (1) performed an extensive evaluation of the electrical conductivity of nanoparticles made up of metallic and ceramic nanoparticles (Cu, Al<sub>2</sub>O<sub>3</sub>, CuO) with volume fractions in different regimes. Thus, it was observed that the electrical conductivity increases with both particle concentration and particle size reduction, in case of water and ethylene-glycol based nanofluids. It is also argued that the electrical conductivity enhancement is higher in case of ceramic nanofluids than metallic ones because of the effective dielectric constant and density. It is also emphasized that the use of surfactant increases the stability, which in turn decreases the conductivity due to its higher viscosity in turn. Also, it is observed that the conductivity increases at lower concentrations of electrolyte than at higher concentrations where it shows an

in turn lower value. These observations are then compared with the ones proposed in the model of O'Brien for electrical conductivity of suspensions. Also, it was eventually observed that there was no significant influence of fluid temperature on the conductivity.

**Amy Teja et al.** (2) presented a one-parameter model concerning the electrical conductivity of dispersed metallic nanofluids. It takes the decreasing size of metallic nanoparticles and the consequent decrease in electrical conductivity into account. But, although all of this was done, still the effect of size of nanoparticles on thermal conductivity could not be elucidated precisely. Thus, the thermal conductivity of six silver nanofluids were reported with varying volume fractions. As a result of this, it was deduced that the decreasing thermal conductivity had to be taken into brief account for the development of further thermal conductivity models.

**Daniel Oliva et al.** (3) presented a brief study on the varied applications of nanofluids, as in, coolant in the diesel electric generator (DEG). The specific heat of aluminium oxide with varied particle sizes have been measured, indicating their decrease with increase in temperature and concentration. Widespread experiments were performed to assess the co-generation efficiency of the DEG. Thus, there showed a decrease in its value on applying nanofluids. This resulted due to a decrease in the specific heat, which influenced the waste heat recovery from the system, which in turn increased in the effect of the nanofluids, as a result of its superior convective heat and radiative heat properties.

**Somnath Basu et al.** (4) performed an experiment for the effective electrical conductivity measurement of aluminium oxide nanoparticles suspensions. These were performed as a function of volume fraction as well as temperature to examine their respective effects. Thus, the results that were reported indicated considerable enhancement of the value with increase in both of the fractions. But, it was observed that the effect of temperature was much more pronounced than that of volume fraction.

**M.N. Pantzali et al.** (5) investigated the efficacy of nanofluids as coolants. It indicated that the thermo-physical properties were considerably affected by nanoparticle addition to the base-fluid. In this regard, a 4% CuO suspension in water is selected. Then its performance on a plate heat exchanger (PHE) was studied. The studies gave rise to the fact that the flow in the exchanger

also affects the coolant efficacy. Thus, the fluid viscosity comes out to be a crucial factor. Thus, it was concluded that for large volume fractions, nanofluids are not the suitable replacement.

**John Philip** *et al.* (6) reviewed the recent advancements in nanofluids, with special emphasis on the associated material properties and its corresponding effect on thermal conductivity and other thermal properties and the novel approaches to achieving a high value of the associated properties. This task was taken up by him in order to resolve the high degree of ambiguity surrounding the high promise as was expected in case of nanofluids. The high scale miniaturization of the particles which led to the high-scale properties have been quite precisely described in the study. The associated problems of low conductivity, both thermal and electrical, led to this study being of high-scale prominence.

**Hai-tao Zhu** *et al.* (7) presented a novel one-stop method for the preparation of copper nanofluids from its associated sulphide,  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  using  $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$  in ethylene glycol under very irradiation in the microwave scale. Highly non-agglomerated and highly stable nanofluids were obtained. The effects of the different reacting agents, i.e., the complexes and the microwave irradiation were observed precisely under a transmission electron microscope (TEM) and associated infrared and sedimentary analysis. It was ultimately found to be one of the most effective and rapid one-step solution for preparing Cu nanofluids.

**Dongsheng Wen** *et al.* (8) worked ahead in regards of determining the varied limiting factors in order to push ahead the development of nanofluids. It was taken up in order to fasten the stagnating scope of research in the field, since the time it had been formulated regarding enhanced electrical conductivity.

**Nader Nikkam** *et al.* (9), investigated the fabrication, the associated thermal conductivity and the varied rheological properties of nanofluids comprising of copper nanoparticles in diethylene glycol base. The fabricated fluids exhibited enhanced thermal conductivity. This was formed by directly forming the nanoparticles in the ethylene base fluid, by means of assisted microwave heating, which accelerated the clusters of metal to monodispersed nanostructures. The particles exhibited an average size of  $75 \pm 25$  nm for SEM micrographs, which aggregated to form spherical agglomerates within the size range of 300 nm. The various physicochemical properties, thermal conductivity and viscosity included, were measured in the size range of 0.4-1.6 %

weight fraction of nanoparticles within the range of 20-5- Celsius. The correlational models, appropriate to the system, was applied to compare the associated properties, which were rather found to be higher in case of thermal conductivity enhancement than the viscosity counterpart. Thus, it showed that with changing concentrations, the thermal properties display more pronounced variation than in the case of viscosity.

**G. Paul** *et al.* (10) reported the detailed analysis regarding the synthesis of very low concentration comprising nanofluids, the most common ways being one-step and two-step methods. While, stable nanofluids can be produced with wide concentrations in case of the one-step method, the two-step provides a means to produce nanofluids with widespread dispersity. In spite of all this, the central point lies the thermal conductivity of the fluids, which is the most researched aspect all around the globe. In this study, an insight has been presented into the different factors which affect this really crucial property of the nanofluids. Along with it, has been presented how the theories that existed explain the anomaly regarding the enhancement of the thermal conductivity of the fluids. Thus, it provides a widely varying view of the different aspects that lead to the sole concept of nanofluids, may it be with regards to physical or the associated theoretical and experimental regards which support the whole background of experiments and developments that led to its wholesome evolution.

**X.J. Wang** *et al.* (11), investigated the thermal storage characteristics of Cu-H<sub>2</sub>O nanofluids as phase changing material (PCM) in case of cooling systems. Varied experiments were carried out to study the influence of the sole nanoparticle agent in the supercooling of PCM which are highly water based. The associated temperature shape and ice shape were observed over the web using infrared heat camera and color digital camera. A mechanism to improve the thermal characteristics was devised by measuring the contact angle and their thermal conductivity. Thus, the experimental results showed that the Cu-H<sub>2</sub>O system showed a considerably lower degree of supercooling than the water PCMs. On addition of 0.1 wt. % of nanoparticles, the degree of supercooling was reduced by about 20.5%, whereas the freezing time got reduced by about 19.2 %. Thus, these results showed the highly promising and widely diverse applications of nanofluids, when their thermal distributions are taken into account.

**Zhen-Hua Liu** *et al.* (12), investigated the thermal performance of a flat CPL, using water based and ethanol based Cu nanofluids under several steady sub-atmospheric working

systems. This was done with the evaporator of the CPL horizontally placed and bottom-heated. Thus, the results showed that on addition of Cu the evaporating heat coefficient gets highly enhanced along with the maximum heat removing capacity of the system. But, there is an optimal concentration of the particles at which we observe the highest coefficient and it decreases with further increase in the concentration. Along with, the coefficient increases drastically with the operating temperature of the system. The coefficient and the heat removal capacity consequently can be increased upto levels of 45% and 16% respectively when substituted with ethanol-bas fluids.

**Enrique J. Lavernia** *et al.* (13), summarized the recent researches with the trends of synthesis, their associated thermo-physical properties, their reated heat transfer properties and their consequential pressure-drop characteristics. They showed that with proper hybridization, the properties of the hybrid nanofluids could be so tailored as to become promising enough for heat transfer enhancement and associated thermal properties along with it. This study was taken up in order to overcome the problems and limitations faced by the conventional nanofluids and to find a mid-way in order to enhance their properties without losing their base characters. In spite of all the ups and downs in its research type, it has highly emerged as a very promising fluid which is very effective when it comes to heat transfer and the same goes for acting as a coolant in different systems under highly varying condition.

# **CHAPTER-3: Experimental Procedure**

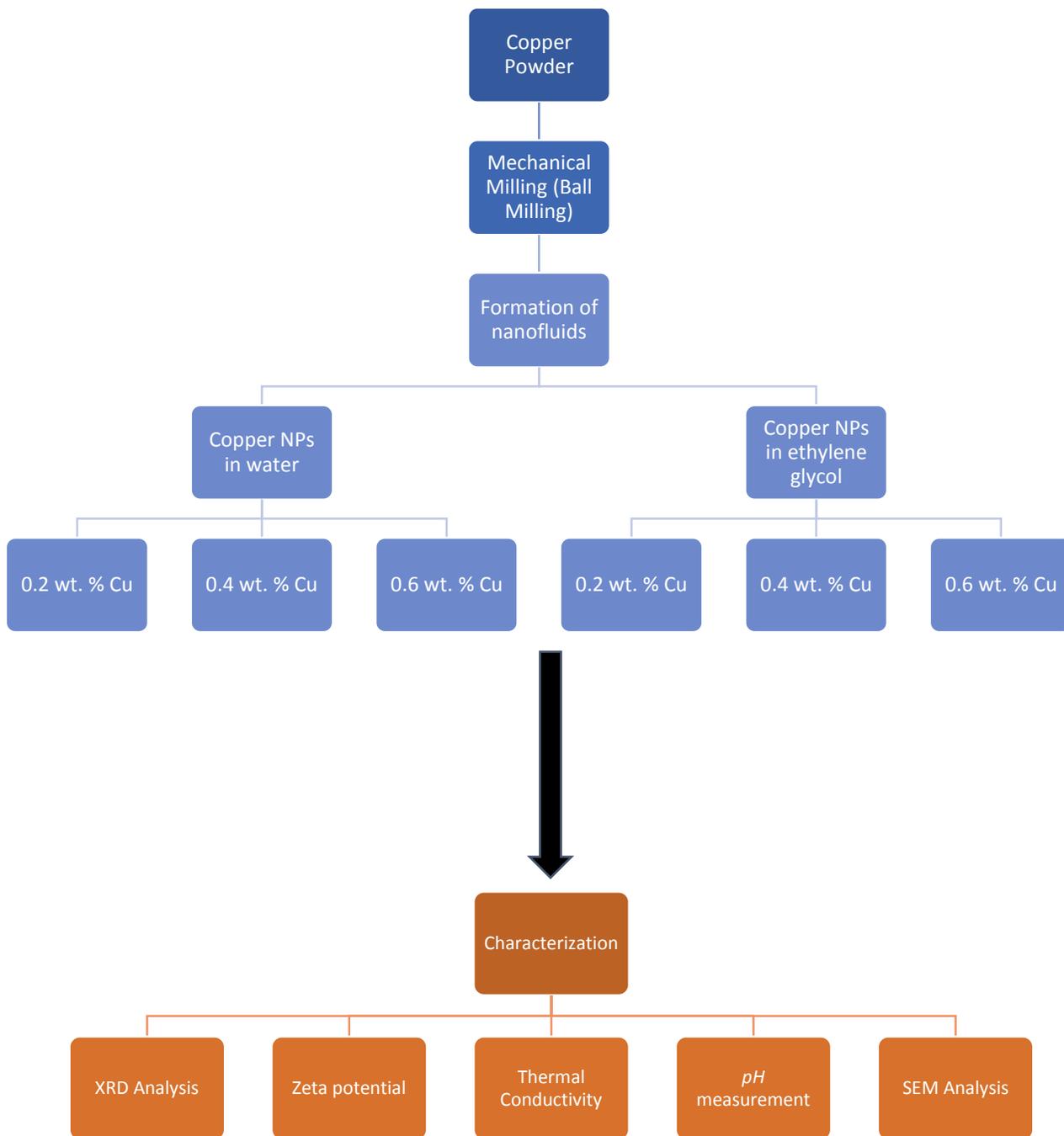
## **3.1 MATERIALS**

The following materials were used in the synthesis and characterization methods:

- Copper powder
- De Ionized water
- Toluene
- Ethylene Glycol
- Oleic Acid

## **3.2 SYNTHESIS**

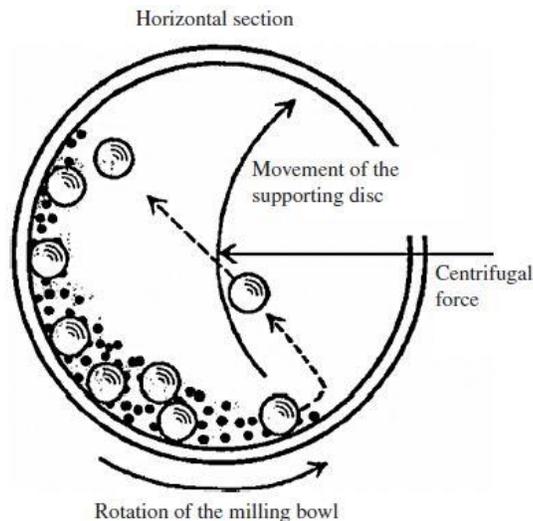
Preparation of nanofluid and its characterization was carried out in different steps. The same has been shown in flow chart displayed as Fig. 1.



**Fig. 1** Flowchart representing the experimental procedure

### 3.2.1 Preparation of Copper nanoparticle

The method of nanoparticle preparation chosen was Ball milling. For the purposes of achieving near nano-sized particles, wet milling process was used which has been seen to produce a faster rate of reduction in size of particles than dry milling. About 20g of powdered Copper was taken in a metallic vial and approximately half the volume of the vial was filled with toluene. Copper particles being volatile, toluene was chosen as the fluid during the milling process, and not water. Then metallic balls varying in their size, made of chrome steel were added to the mixture in the vial so that a weight ratio of 1:10 is maintained between the Copper powder and the metallic balls. Thus around metallic balls weighing about 200g were added to the mixture mentioned above. Then 2 such metallic viles were carefully placed in a High Energy Planetary Mill. Parameters like rotational speed and time duration of milling could be chosen as per necessity. A speed of 300 rpm was maintained in the mill in order to get a significant reduction in the particle size. The milling was done for a total of 10 hours in cycles of 30 minutes each with cooling interval of 30 minutes in order to control the temperature inside the mill (i.e. to avoid overheating of the arrangement). After the completion of a total 10 hours of milling, the viles were taken out of the mill and the powder of Copper nanoparticles were separated from the metallic balls and toluene within the viles. Since nanoparticles are very prone to agglomeration, the obtained sample was kept in an air tight container so as to prevent any agglomeration.



**Fig. 2 Horizontal section of a ball milling bowl**

### **3.2.2 Preparation of Copper nanofluid**

A nanofluid is a solution of nano-sized particles immersed in a base fluid. For analysing the effect of a base fluid on the produced solution, 2 different base fluids were used, i.e. DI (deionised) water and Ethylene Glycol. The nanofluids were produced in test-tubes of approximately 30 ml volume. Different weight fractions of Copper nanoparticles were used in different solutions. Solution in DI water and Ethylene Glycol were taken in test-tubes and 0.2 wt. %, 0.4 wt. %, and 0.6 wt. % of Copper nanoparticles respectively were added to the fluids. These solutions were then placed in an ultrasonicator where the ultrasonic frequencies (i.e. > 20 kHz) agitated the nanoparticles for a time duration of about 2 hours in cycles of 45 minutes each. Before the sonication process, the solutions were seen to have the nanoparticles agglomerate and sediment at the bottom of the test-tube leaving the solution clear and transparent. After the sonication process, the nanoparticles were dispersed in the solution which was confirmed by the change in colour and transparency of the solution.

## **3.3 CHARACTERIZATION**

### **3.3.1 X-Ray Diffraction Analysis**

X-ray diffraction of the Copper powder before and after milling was done using PHILIPS X-ray diffractometer. 2 theta ( $2\theta$ ) values scanned was from a range of 20 to 100 degrees. Continuous scanning was done with generator settings of 35kV and 30mA. The data thus obtained was analysed using X-pert analyser and the respective interplanar lattice spacing (“d-spacing”) and full width at half maximum (FWHM) were calculated for the desired peaks. Then, the crystal size was calculated using Debye-Scherrer equation.

### 3.3.2 Zeta Potential Measurement

First, the milled Copper powder was mixed with DI water and was sonicated till a proper dispersion was achieved. Since a fine dispersion couldn't be achieved in the initial sonication cycles, the cycles were continued for about 10 times till a fine enough dispersion was obtained. Then the zeta potential and particle size of the nanofluid was measured using a Zetasizer. The  $pH$  of the nanofluids were varied by adding drops of dilute Nitric Acid solution. A plot of  $pH$  vs. zeta potential values was obtained by the observed values and iso-electric point was extrapolated thereafter.

### 3.3.3 Particle size analysis

From the zetasizer, particle size distribution and average particle size was directly obtained. For this measurement dispersed powder in de ionized water was used.

### 3.3.4 SEM Analysis

A ICON field emission scanning electron microscope was used to characterise the particle size, morphology and microstructures of un-milled and milled powder specimens. Micrographs were obtained at a suitable accelerating voltage to achieve the best possible resolution by the secondary electron imaging device.

### 3.3.5 Thermal Conductivity Measurement

The thermal conductivity of nanofluids was evaluated using KD2 pro table top thermal property analyzer.

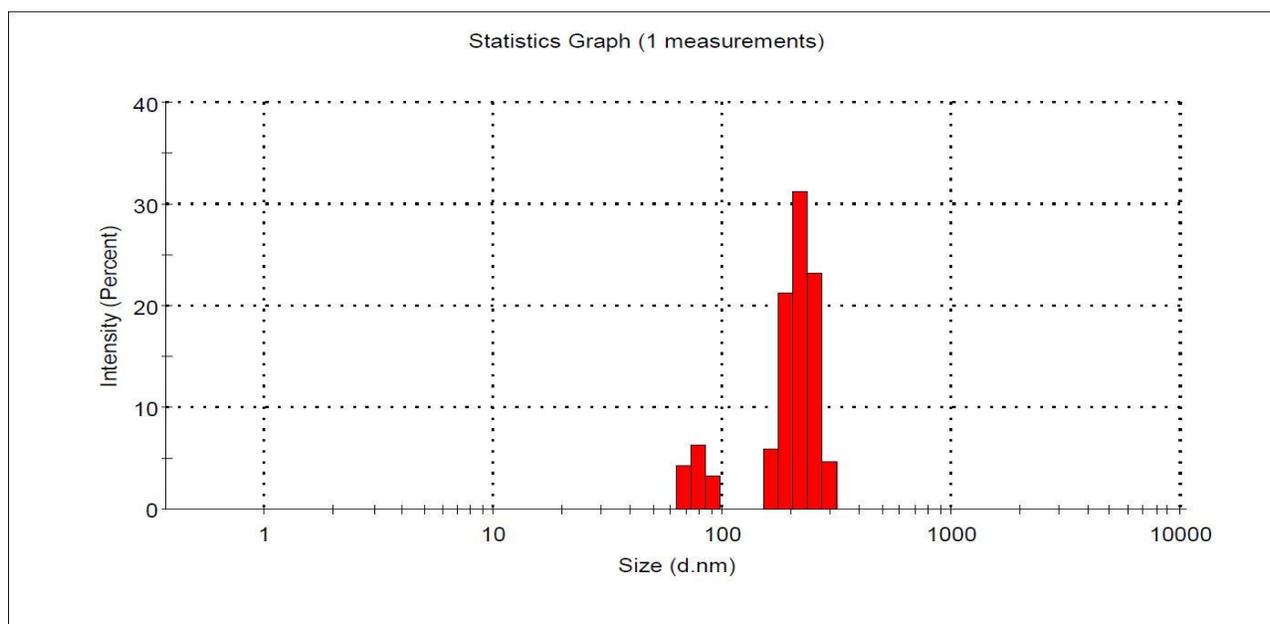


**Fig. 3 KD2 pro table top thermal property analyser**

# **CHAPTER-4 RESULTS AND DISCUSSION**

## **3.4 Particle Size Analysis**

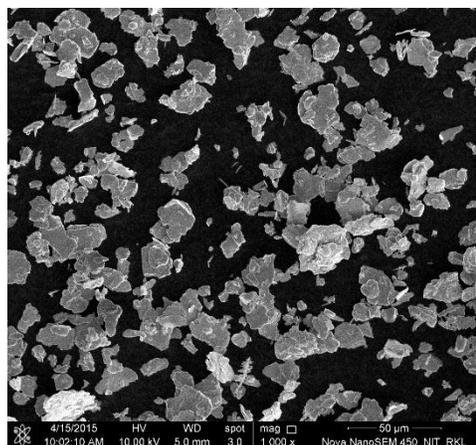
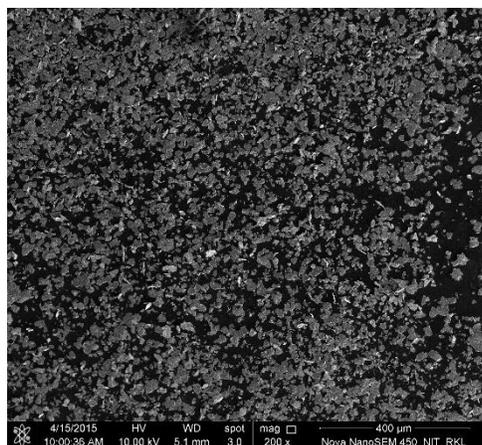
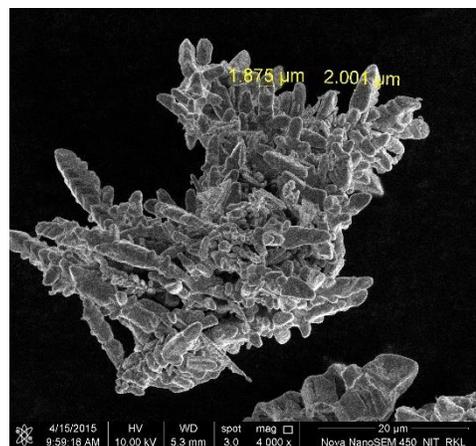
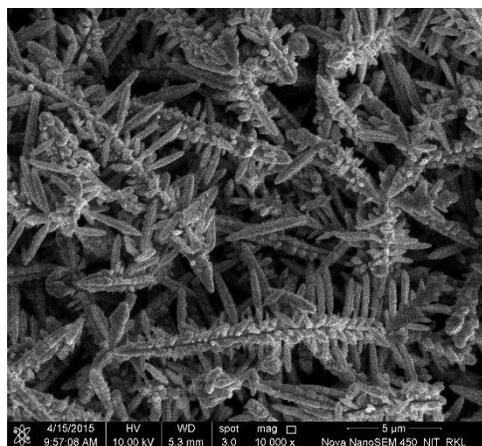
### **3.4.1 By Zeta Sizer (particle size analyser)**



**Fig. 4 Plot of size (in nm) vs. Intensity (%) for Cu nanoparticles as determined from the zeta sizer, showing the overall size distribution of the particles throughout the system.**

From the figure, it can be seen that the overall size distribution can be seen scattered throughout the plot. This bimodal distribution consists of particles size range of 60-100 nm (minor population) and 100-400 nm (major population). Thus, we get to have a clear picture regarding the major size fraction of the particles from the above plot. The average particle size was found to be 300 nm.

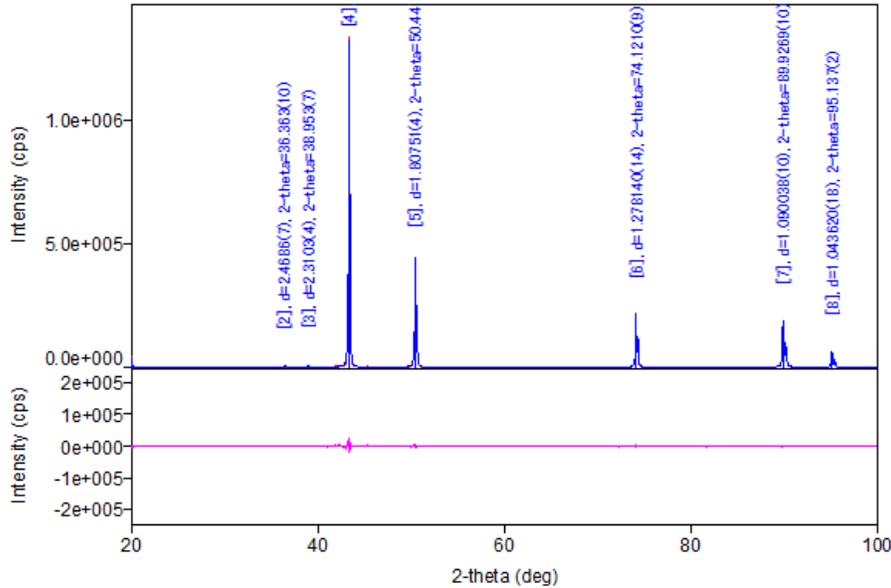
### 3.4.2 By SEM study



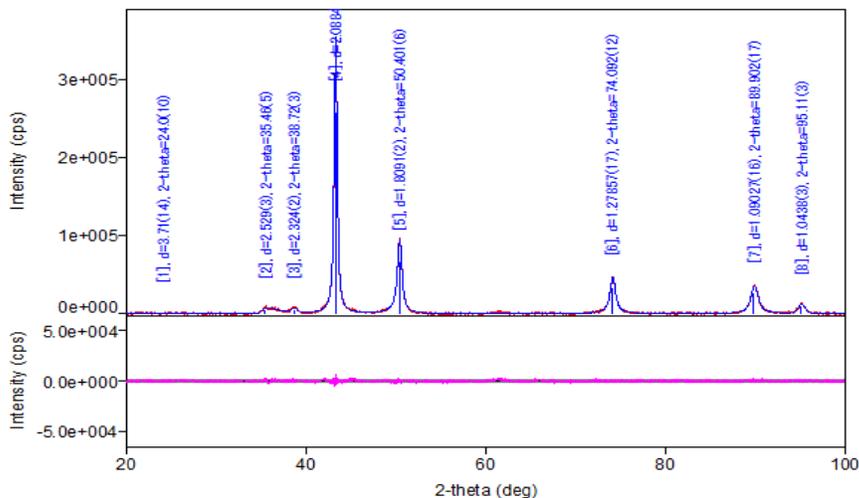
**Fig. 5 SEM image of Cu particles (a, b) before milling (at 10000X and 4000 X respectively) and (c, d), after milling (at 200X and 1000X respectively).**

Figure 5 shows the SEM micrograph of the Cu powder before and after milling. The as received Cu powder shows typical morphology of electrolytic powder. The as received unmilled powder shows a size range of 50-100  $\mu\text{m}$  when observed at 1000X magnification, but after milling they have been converted to size range of 1-20  $\mu\text{m}$  at 2000X magnification. Thus, we see a corresponding reduction in size after the milling stage is completed. As can be seen from the images corresponding to both before and after milling, it is quite clear that there was an obvious reduction in size when the images are compared with respect to both the stages. From Fig. 4 and 5 it can be understood that after milling there was size reduction. But, the data obtained from zetasizer and SEM micrograph are different. Higher size obtained in SEM may be due to clogging of particle during spraying them on the adhesive tape.

### 3.5 XRD Analysis



(a)

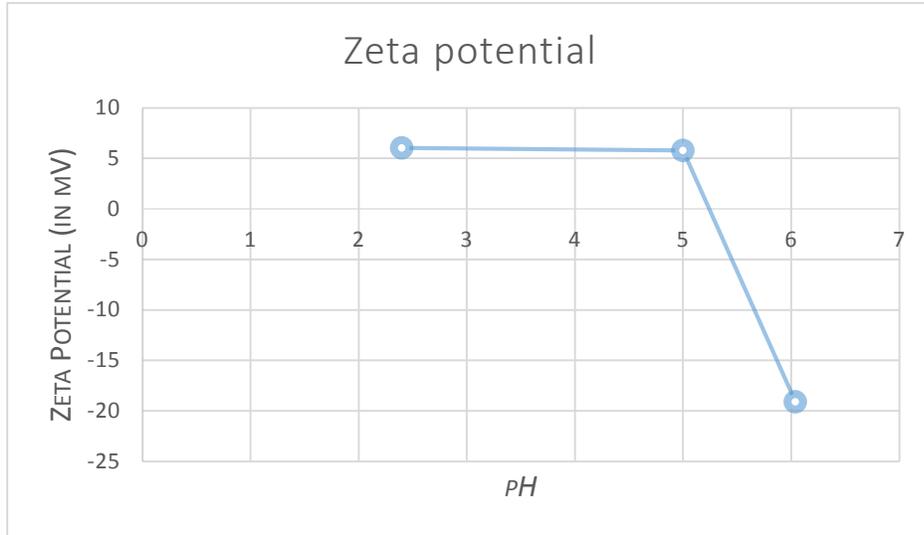


(b)

**Fig. 6 X-ray diffraction plots of Cu nanoparticles as a function of Intensity (cps) vs. 2-theta (deg): (a) before milling and (b) after milling.**

Fig. 6 shows the XRD plots of Cu powder before and after milling. Certain distinct maxima which are called ‘peaks’ corresponding to certain values of 2-theta (diffraction angle) were observed. From the Fig. 6(a), it was seen that there is a maximum peak at a diffraction value of 40 degrees and then the peaks slowly decrease in intensity as the diffraction angle goes on increasing. Thus, a trend of increase in diffraction angle with decrease in the peak intensity was seen. Similarly, in the Fig. 6(b) a maxima peak at diffraction angle of 40 degrees was observed after milling which was then seen to decrease gradually. Thus, from the plots, the intensity distribution throughout the crystal as a function of diffraction angle was evaluated. The corresponding values of 2-theta was used in the Debye-Scherrer equation in order to determine the corresponding lattice spacing( $d$ ), which was subsequently used to determine the crystal size (a) for a given diffraction angle. The average crystallite size after milling was found to be 0.43nm.

### 3.6 Zeta Potential Measurement



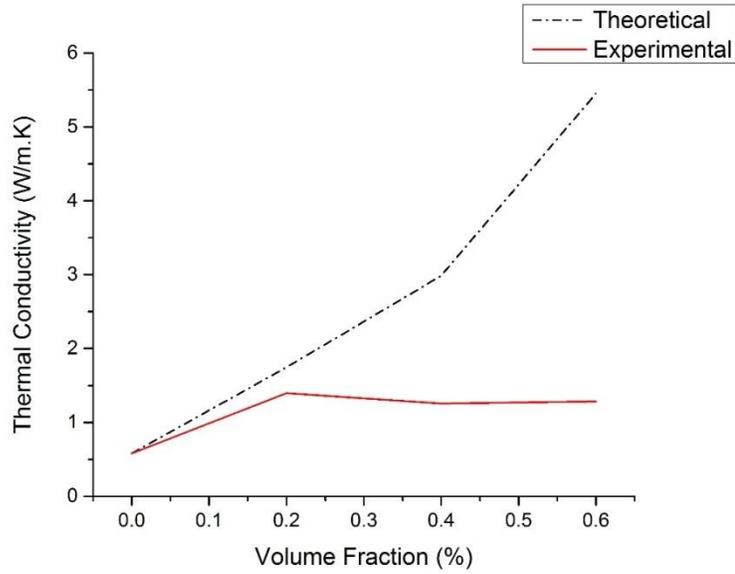
**Fig. 7 Plot of Zeta Potential (in MV) vs. *pH* for Cu- Nanoparticle after milling**

The plot for the variation of Zeta Potential with corresponding change in *pH* of the nanofluid with addition of dilute HNO<sub>3</sub> was obtained. Thus, the zeta potential at various concentrations of the acid when added to the fluid was observed. From the plot the corresponding isoelectric point was found out to be around 5.4. Thus, we see that there was a varying trend of Zeta Potential with variation of *pH*, i.e. the potential remained constant with increase of *pH* to around 5, followed up by a steep decrease when it was increased beyond 5. Thus for stable dispersion, the solution *pH* should be away from 5.4.

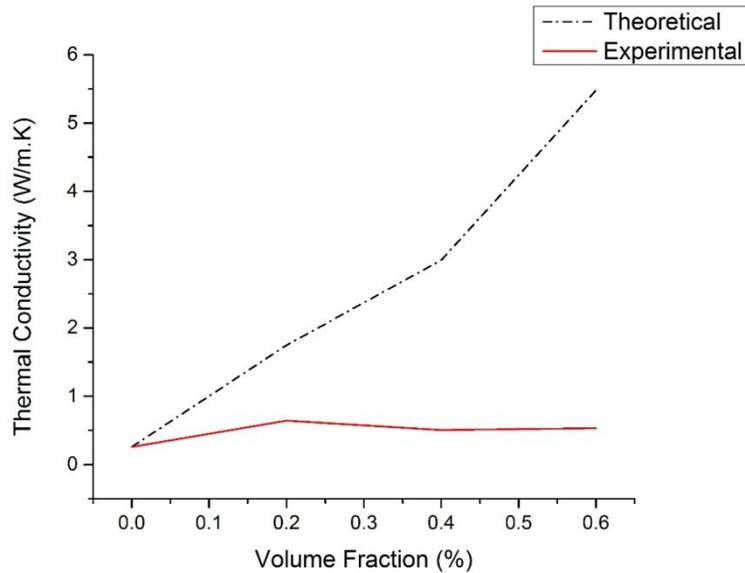
### 3.7 Thermal Conductivity

| Base Fluid      | Cu vol. fraction (%) | Thermal Conductivity (W/mK) | <i>pH</i> |
|-----------------|----------------------|-----------------------------|-----------|
| Water           | 0                    | 0.58                        | 7         |
| Water           | 0.2                  | 1.397                       | 9         |
| Water           | 0.4                  | 1.257                       | 9.7       |
| Water           | 0.6                  | 1.282                       | 9.4       |
| Ethylene Glycol | 0                    | 0.258                       | 7.8       |
| Ethylene Glycol | 0.2                  | 0.642                       | 7.2       |
| Ethylene Glycol | 0.4                  | 0.505                       | 7.2       |
| Ethylene Glycol | 0.6                  | 0.534                       | 7.1       |

**Table 1 Variation of  $pH$  and thermal conductance with volume fraction**



**Fig. 8 Plot of thermal conductivity (W/m.K) vs. Volume fraction (%) of Cu nanofluid when water is used as the base fluid.**

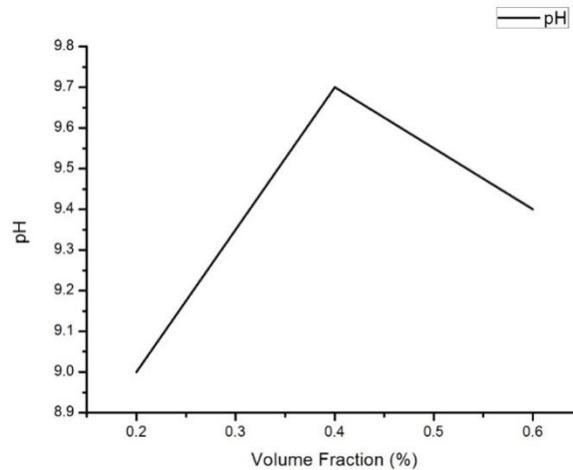


**Fig. 9 Plot of thermal conductivity (W/m.K) vs. Volume fraction (%) of Cu nanofluid when ethylene glycol is used as base fluid.**

Figure 8 and 9 show the variation of Cu content in nano fluid and related change in thermal conductivity of the fluid tested with water and ethylene glycol as base fluid. Moreover, they also display theoretical conductivity value calculated by Maxwell equation. From the above plots (Fig. 8 and Fig. 9), a comparison between theoretical and experimental values of thermal conductivity as a function of volume fraction of copper particles was made. In the water based as well as the ethylene glycol based nanofluid, an increase in the volume fraction showed a temporary increase in measured conductivity value (up to 0.2%) followed by decrease in thermal conductivity which is contradictory to the theoretically expected trend. A probable explanation for this anomaly could be the agglomeration of nanoparticles with increase in the volume fraction. Addition of more particles in a fixed volume of liquid can be considered as congestion in particle movement imposed externally. These can lead to clogging or agglomeration which in turn reduces the particle-fluid interface and some fraction may precipitate out. Both this mechanism will ultimately play against the nano-fluid's working mechanism. Moreover, the theoretical conductivity values shows higher trend than the measured one. This can also be explained by the agglomeration/instability of the nano fluid.

Apart from these common trends in both the fluid, it was also observed that the water based nanofluid showed a higher thermal conductivity than the ethylene glycol based nanofluid. This was due to the higher thermal conductivity of the base fluid, i.e. water.

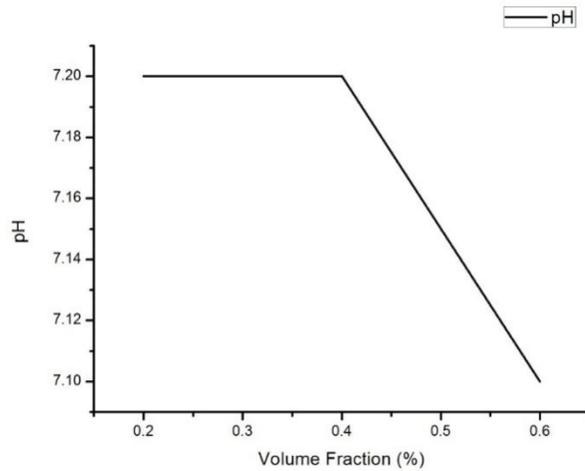
### 3.7 pH



**Fig. 10 Plot of *pH* vs. Volume fraction (%) of Cu nanofluid when water is the base fluid.**

Fig. 10 shows the  $pH$  vs. Cu volume fraction (in water) variation and from the plot it is clear that with increase in Cu % there was an increase in  $pH$  value, followed by a decrease in the same. Theoretically a  $pH$  value far greater or smaller than 5.4 (IEP value) signify a stable dispersion of nanoparticles and the dispersion can be maintained for a long period. Here, with increase in Cu % there was increase in  $pH$  up to 0.4% of Cu. Thus the stability of the nano fluid would poor in this range and the same was observed in Fig. 8 where best conductivity was observed at 0.2%. After 0.4% Cu the  $pH$  value increases (away from IEP value) but still there was no improvement in conductivity/stability as more amount of powder plays the main role in agglomeration.

Fig. 11 displays the plot of Cu vol. % (in ethylene glycol) vs.  $pH$  and it depicts similar trend of Fig. 10.



**Fig. 11 Plot of  $pH$  vs. Volume fraction (%) of Cu nanofluid with ethylene glycol as base fluid.**

## **CHAPTER-5: CONCLUSION**

1. The as-received copper powder was successfully ball milled to reduce its size to an average value of 300 nm as observed by particle size measurement. Moreover isoelectric point of the powder was found out to be 5.4.
2. Due to milling there was also a reduction of grain size to a value of 0.43nm.
3. Nanofluids, with water and ethylene glycol base were prepared by dispersing different volume fractions of the milled powder in them.
4. Increase in the amount of copper nanoparticles increases the thermal conductivity of the fluid up to a certain value of volume fraction of Cu due to agglomeration/instability with further addition.
5. The change in volume fraction of the nanofluid affects the  $pH$  value, which in turn affects the stability of the dispersion.

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