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Investigation on Copper Nanofluid obtained through Micro Electrical Discharge Machining for Dispersion Stability and Thermal Conductivity

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Abstract

Nanoparticles show a practical way of exhibiting enhanced size-dependent properties compared to extremely fine or larger particles of the same material. Nanofluids consist of nanoparticles dispersed in base fluid and are usually used for enhancing thermal conductivity in various systems. This study investigates the dispersion stability and thermal conductivity of copper based nanofluids obtained by generating copper nanoparticles using micro electrical discharge machining (micro-EDM) process in both, de-ionized (DI) water and de-ionized water with polyethylene glycol (PEG) stabilizing agent. Micro-EDM was performed with various operating parameters such as the voltage, current, pulse width and duty factor. The generated nanoparticles in the base fluid were examined by energy dispersive analysis by X-rays (EDAX) and transmission electron microscope (TEM) to evaluate their chemical nature, size and morphology. The size distributions of the copper particles in PEG-based nanofluid were determined resulting in average particle size of around 6 nm. The experimental results show that the dispersion stability of PEG-based copper nanofluid as compared to DI water-based copper nanofluid is improved as discussed in the following sections of the paper. It was also found that the thermal conductivity of copper-water nanofluid and copper-water with PEG nanofluid are higher than that of pure DI water.

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1. Introduction

In many industrial processes there is a need for efficient heat transfer systems, typically provided in the form of flowing fluid. Recently, heat transfer fluids such as water, ethylene glycol, air and oil are extensively used as the cooling media in automobiles, electronics, power generation, refrigeration and air conditioning systems and chemical processing industries. However, these fluids generally have very low thermal conductivities compared to solid materials, resulting in low heat removal efficiencies. Therefore, fluids containing suspended solid particles can be fairly anticipated to have higher thermal conductivities than pure fluids. In the past few decades, rapid advance in nanotechnology have lead to emerging of new generation of nanofluids. Nanofluids refer to a new kind of fluids in which nanoparticles are suspended in a base fluid [1]. In most of the studies the base fluid has mainly been either water or ethylene glycol or suitable mixture of both. The nanoparticles have usually been metals or oxides. Choi [2] proposed that nanofluids consisting of metallic nanoparticles have attracted significant attention by researchers due to their peculiar properties, different from those of atomic or molecular species. It has been shown that if nanoparticles are dispersed in fluid, the thermal conductivity of nanofluid can be significantly increased [3]. This enhancement can improve the efficiency of fluids in heat transfer applications, as well as allow a reduction in size on such systems.

Nomenclature

μ	dynamic viscosity of the base fluid (centiPoise)
g	acceleration due to gravity (m/s^2)
D	diameter of the spherical ball (mm)
ρ	density of spherical ball (kg/m^3)
ρ_f	density of base fluid (kg/m^3)
V	terminal velocity (m/s)
$R_{\text{DI water}}$	average radius of copper nanoparticle dispersed in de-ionized water (nm)
R_{PEG}	average radius of copper nanoparticle dispersed in polyethylene glycol (nm)
$V_{\text{DI water}}$	sedimentation velocity of copper particles in de-ionized water based nanofluid (m/s)
V_{PEG}	sedimentation velocity of copper particles in polyethylene glycol based nanofluid (m/s)

Among various metal particles, copper nano-particles have attracted considerable attention because of their low cost and novel catalytic, thermal, mechanical, and electrical properties which are different from those of bulk metals [4-6]. Copper based nanofluids are widely used in industrial applications as coolants [7]. A wide variety of methods have been used for the preparation of copper nanoparticles, such as microemulsions [8], high energy ball milling [9], reduction of aqueous copper salts [10], ultraviolet light irradiation [11] and polyol process [12]. Lee et al. [3] prepared copper nanofluids using a two-step method by fabricating copper oxide nanoparticles first and then redispersed in base fluids-water and ethylene glycol. They found that the aggregation of nanoparticles resulted from separation process usually caused poor stability of nanofluids after redispersion. All these methods require control on the particle size produced and size distribution, costly precursors and high yield. There is a specific need to find a cost effective method for generating copper nanoparticles. To our knowledge, micro electrical discharge machining (micro-EDM) method has not been used to obtain copper nanofluids.

Experimental data on the thermal conductivity of nanofluids widely varies and recent reviews have tried to assess the large amount of data to help in understanding the mechanism of thermal conductivity enhancement of nanofluids. Mechanisms proposed to explain thermal conductivity enhancement include Brownian motion of nanoparticles [13, 14], layering of fluid around nanoparticles [15, 16] and near field radiative heat transfer [17]. However, there is limited data available on the thermal conductivity enhancement of PEG-based copper nanofluids.

Vadasz [18] studied the effect of surface area to volume ratio of nano-particle suspended in the fluid on thermal conductivity of the fluid. Copper nano-particles in ethylene glycol and carbon nanotubes in oil are used as samples for the experiment. The specific area was shown to impact substantially and the results show that there is enhancement in the thermal conductivity of the fluid. Jang and Choi [19] studied the role of Brownian motion in the enhanced thermal conductivity of nano fluids. This study led to the realization that Brownian motion of nano particles at the molecular and nano scale levels is a key mechanism governing their thermal behavior. Eastman et al. [20] reported an almost 40% improvement in thermal conductivity through the dispersion of 0.3 vol % copper nanoparticles in ethylene glycol. They used direct condensation of metallic vapor into nanoparticles by contact with a flowing low vapor pressure liquid. Zhu et al. [21] produced copper in ethylene glycol nanofluid by reducing a mixture of copper sulphate pentahydrate in ethylene glycol with sodium hypophospite monohydrate. They used polyvinylpyrrolidone as a surfactant and reported an almost nine percent increase in thermal conductivity with 0.1 vol % loading of copper nanoparticles in ethylene glycol. Liu et al. [22] synthesized copper nanoparticles in water using the chemical reduction method. Samples of volume fractions ranging from 0.05% to 0.2% of copper nanoparticles in water were prepared. They reported a maximum increase in thermal conductivity of water of about 23.8% with a volume fraction of 0.1 vol % copper nanoparticles in water. Xuan and Roetzel [23] have concluded that the heat transfer enhancement by copper nanoparticles was due the suspended particles which increase the thermal conductivity of the two phase mixtures and the chaotic movement of ultrafine particles which accelerates energy exchange process in the fluid. Garg et al. [24] have studied the thermal conductivity and viscosity of copper nanoparticles in ethylene glycol. They found that the measured thermal conductivity enhancement was twice the value predicted by the Maxwell effective medium theory and viscosity increase was almost four times of that predicted by the Einstein law of viscosity. Xuan and Li [25] investigated the heat transfer enhancement of copper nanofluids. They prepared copper nanofluids using step-by-step method by dispersing copper nanoparticles into base liquids. They found that the sedimentation of nanoparticles was reduced by employing laurate salt as surfactant with water and thermal conductivity of the fluid was greatly enhanced. Ibrahim et al. [26] prepared stable propylene glycol-based nanofluids by prolonged ultrasonication without causing particles aggregation. They found that the thermal conductivity of nanofluids increases non-linearly with particle concentration. The enhancement of thermal conductivity was temperature independent and parallels the behaviour of pure propylene glycol.

In this article, we obtain copper nanofluids by generating copper nanoparticles using micro-EDM process in both, de-ionized (DI) water and DI water with polyethylene glycol (PEG) stabilizing agent. Micro-EDM technique is basically a vapor phase synthesis technique, where debris is formed by repetitive spark discharge between the electrodes. PEG prevents agglomeration and coagulation of copper nanofluid during micro-EDM process. The nano sized copper particles generated in the solution were characterized using energy dispersive analysis by X-rays (EDAX), transmission electron microscope (TEM) and selected area electron diffraction (SAED). Further, an experimental investigation has been carried out to understand the stability and thermal conductivity of copper based nanofluids.

2. Experimental details

The schematic diagram of micro-EDM is shown in Fig.1 (a) and micro-EDM prototype developed by us is shown in Fig. 1 (b). It consists of tool (cathode) and work piece (anode) separated by a small gap, surrounded by dielectric fluid. The piezoactuator is considered for tool feed control in a micro-EDM setup. It consists of two piezostacks arranged in series. Each piezostack consists of 196 piezo wafers of 100 μm thick and 25 μm^2 cross-sectional areas. These two piezostacks are housed inside a flexural link to amplify the displacement of the actuator. The upward and downward movement of the piezoactuator is caused by the flexural link. In micro-EDM setup a transistor type pulse generator is used because high material removal rate and uniform discharge energy are possible. The frequency and the duty cycle of the discharge can be controlled by changing the resistance values of the potentiometers. The pulse control circuit consists of pulse width modulator, which produces rectangular pulses. The voltage drops when the spark occurs and correspondingly some amount of material is removed making it an open circuit. Whenever a short circuit occurs voltage drops to zero. The feedback circuit senses the average gap

voltage and sends it to the linear amplifier, which amplifies the voltage and makes it fall between safe operating range of the actuator. This amplified signal is directly sent to the actuator which feeds the tool and controls the spark

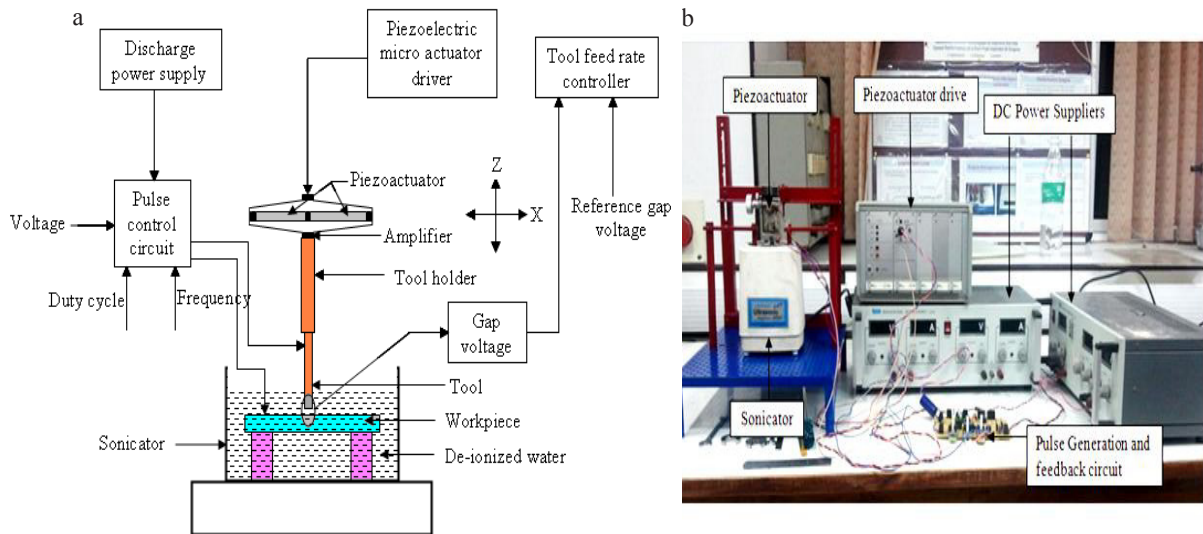


Fig. 1. (a) Schematic diagram of micro-EDM; (b) Developed micro-EDM setup.

gap. In the current setup a sonicator is introduced to produce ultrasonic vibrations with frequency 55 kHz during the experiment to avoid the agglomeration of the particles generated. Also, mechanical and electrical accessories such as DC power supply, oscilloscope, etc. are included in the present setup to monitor the process parameters.

The process parameters for the experiments conducted were pulsed dc voltage, pulse width and input current. The operating voltage can be varied from 0 to 96 V. The pulse-on time could be set by varying the frequency and the duty cycle. In this work, workpiece material is taken as copper (300 μm thickness), tool material is also copper (900 μm diameter) and DI water as dielectric fluid. The parameters used for the experimentation are provided in Table 1.

Table 1. Process parameters for experimentation

Process parameters	Values
Pulsed dc voltage	30 V
Input current	2 A
Frequency	5 kHz
Duty cycle	30 %
Pulse width	60 μs

The copper workpiece was first cleaned with acetone and then with dilute nitric acid which would remove all the organic and inorganic impurities present on the surface. This ensures the formation of pure metal copper particles during the experiment. In this study, 200 ml of DI water was taken in a Sonicator chamber. The required voltage is set for the experiment. The current is limited to 2 A to prevent any damage to the power supply when a short circuit takes place. The feedback voltage should lie between 0 – 7.5 V to prevent any damage to the piezoactuator driver. The workpiece and the tool were brought close to each other and machining at each point is done for about 20-30

minutes. Copper nanofluid obtained was collected in glass vials. Initially experiment was conducted using only DI water under input conditions as given in Table 1. Characterization was done to the particles in the sample collected using only DI water and the results are presented in section 3.

It was observed that sub-micron size particles were formed due to coagulation and agglomeration. The agglomeration will not only result in settlement and clogging of particles, but also decreases the stability and thermal conductivity of fluids. To avoid this problem, PEG was used in the present study. PEG was added to the DI water. The concentration of the solution was 2 gm/200 ml. Characterization was done to the particles in the sample collected from the experiment conducted under same input conditions as given in Table 1 and the results are presented in section 3.

The composition of the nanoparticles was analyzed by EDAX. The particle size measurements were carried out through TEM (Phillips CM-12 with EDAX attached) studies. The particle size was evaluated based on the TEM bright and dark field images. The size distribution of the copper nanoparticles was obtained. To do TEM measurement, sample is first dripped onto a 50-mesh carbon-coated copper grid and then dried at ambient atmosphere. Element of the nanoparticles was confirmed by doing SAED measurement on carbon-coated copper grid.

The viscosity of the base fluids was measured using a Brookfield falling ball type viscometer (Gardco, Inc, Model: KF20, operating temperature range: -60°C to $+150^{\circ}\text{C}$, viscosity range: 0.5 cP to 70,000 cP, accuracy: 0.5% - 2.0%). In this instrument a steel ball was allowed to fall in the tube containing fluid samples. The time taken for the ball to reach the bottom of the tube was measured, depending on which the viscosity of fluids was calculated according to Stokes law of viscosity as given in Equation 1.

$$\mu = \frac{gD^2(\rho - \rho_f)}{18V} \quad (1)$$

The viscosity measurement enables to determine the relation between the sedimentation velocities of copper based nanofluids, thus determining the dispersion stability.

The thermal conductivity of the nanofluid samples was measured using a KD2 Pro device (Size-15.5 cm×9.5 cm×3.5 cm; operating environment: controller- 0 to 50 $^{\circ}\text{C}$, sensor- -50 to 150 $^{\circ}\text{C}$) soon after two hour of sonication. This device consists of a long and thin heating source (large needle probe: size- 100 mm length, 2.4 mm diameter, accuracy $\pm 10\%$, thermal conductivity range 0.1 to 4 W/m K) coated with an electrically insulating layer. The needle probe is immersed in the samples and heated with constant power, which allows us to measure the thermal conductivity of nanofluids based on transient line heat-source method. This method precisely measures the thermal conductivity in minimal time under low heating rates.

3. Results and discussion

3.1. Copper nanofluid characteristics

Fig. 2 shows the TEM micrograph of copper nanoparticles generated by the micro-EDM process using pure DI water. The TEM images of copper nanoparticles show that the particles are nearly spherical in shape. The particle size lies in the range of 600 nm to 1100 nm. Such large particles could be the possible outcome of the coagulation and then agglomeration of the particles. Fig. 2(a) shows particle with dense structure throughout. This indicates that nucleated nanoparticles collide with each other or coagulate to form larger size particles. However, Fig. 2(b) and Fig. 2(c) shows sub-micron particles with not so dense structure at the periphery indicating that smaller particles are agglomerated.

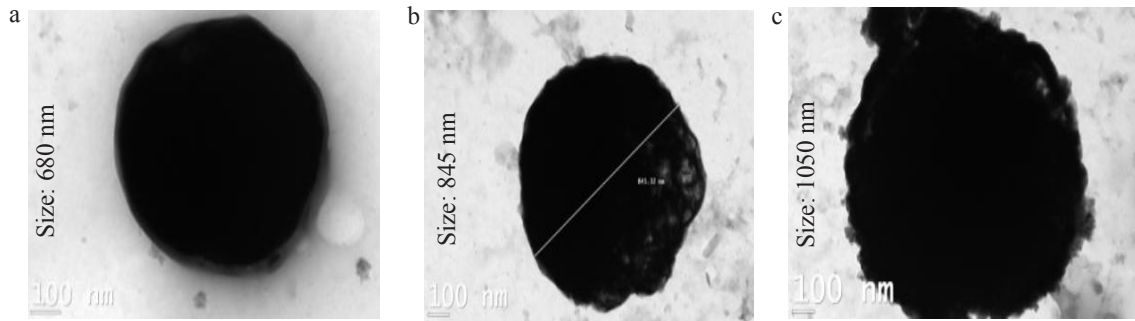


Fig. 2. TEM bright field micrograph showing different sized copper nanoparticles

The composition of the copper nanofluid obtained by micro-EDM was determined by EDAX. The typical EDAX pattern for the copper nanoparticle dispersed in DI water is shown in Fig. 3 (a) and the SAED micrograph for copper nanoparticle is shown in Fig. 3 (b).

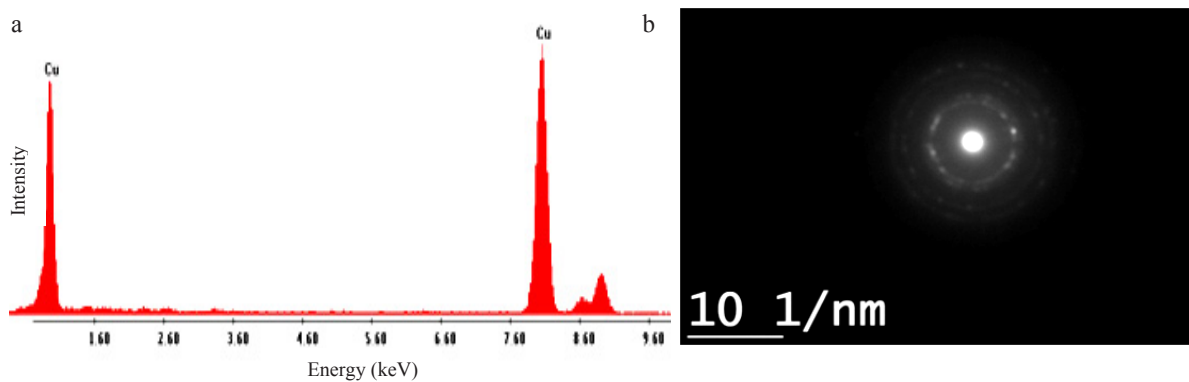


Fig. 3. (a) EDAX pattern of copper nanoparticle; (b) SAED image of copper nanoparticle

Fig. 3 (a) clearly shows two peaks of copper. Here the first peak corresponds to the excitation of electrons to the L-shell in copper atom, known as L-peak and the second peak (K-peak) is suspected to have risen from the carbon-coated copper grids. The EDAX pattern shows the presence of copper particles generated in the dielectric medium. Also, it is confirmed from EDAX that oxides of copper are not observed. Fig. 3 (b) shows a series of spots, each spot corresponding to a satisfied diffraction condition of the sample's crystal structure. The diffraction pattern shows concentric rings, the distance from the centre to each concentric ring was measured. It was found that these distances are characteristic of the polycrystalline metal copper. It is confirmed from EDAX and SAED results that the generated particles were of pure metallic copper.

The particles generated in the experiment are of sub-micron size rather than nano-size (order <100nm). Ultrasonic vibration was provided but still large size particles were formed. This indicates that ultrasonic vibration was not so effective to prevent coagulation and agglomeration. Further, in order to obtain stable DI water based copper nanofluid and reduce agglomeration, sonication was applied for 2 hours using a digital Sonicator. The size of the particles was measured after sonication and the average diameter of the particle was found to be 880 nm. The DI water based copper nanofluid obtained shows poor dispersion stability with visible sedimentation after a period of time. To overcome this problem, PEG was used as a stabilizing agent in the present study. This stabilizer when added to the dielectric possibly reduces interaction between the nucleated particles, therefore reducing the average

size of the particles generated and also improves the stability of nanofluid.

Fig. 4 shows the TEM structure of copper nanoparticles generated by the micro-EDM process dispersed in PEG and DI water mixture. The TEM image of copper nanoparticles shows that the particles are nearly spherical in shape. The particle size lies in the range of 4 nm to 10 nm. The TEM micrograph showed a very fine dispersion of particles in the dielectric. The particles which are represented by the bright spots exhibit crystalline structure indicating the presence of copper in the sample.

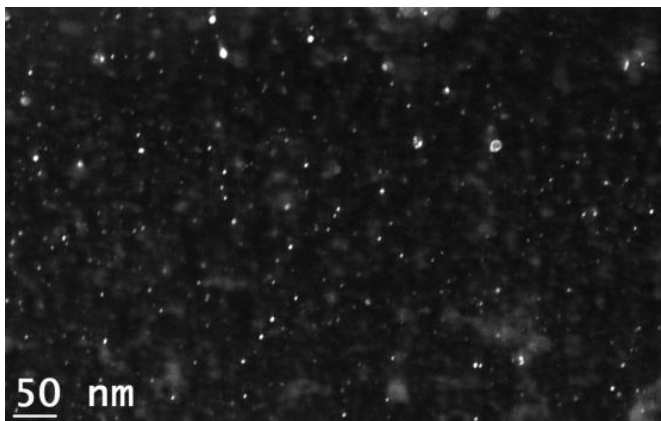


Fig. 4. TEM dark field micrograph of copper nanoparticles with PEG sample

The size of the particles were measured and adopted for the analysis of particle size distribution. Fig. 5 shows the particle size distribution of generated copper nanoparticles.

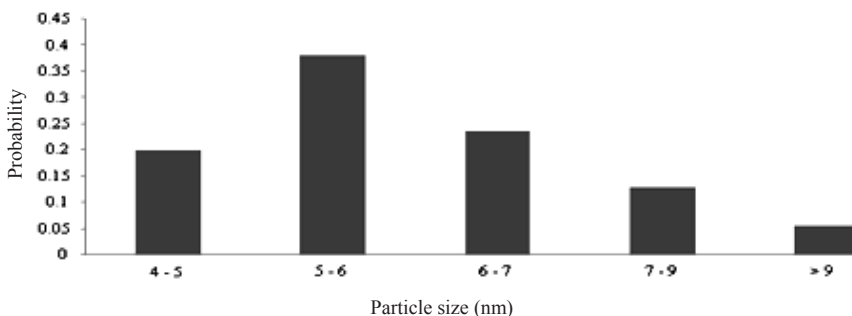


Fig. 5. Size distribution of generated copper nanoparticle with PEG sample

The average diameter of the particle was observed to be 6.04 nm. A reduction on the mean diameter of the particle is observed with the change of the stabilizer PEG. It is observed that particle size measurement follows normal distribution. This result is consistent with the particle size distribution (range: 22 nm to 48 nm) of the synthesized copper nanoparticles in ethylene glycol [27].The particle size distribution narrowed its range which indicates that approximate uniformity in the size of the particles is obtained.

Fig. 6 (a) shows the EDAX pattern for PEG based copper nanofluid obtained through micro-EDM process and Fig. 6 (b) shows the SAED micrograph for copper nanoparticles (PEG sample). The EDAX analysis shows a distinct L-peak for copper. Certain impurities have also peaked because here analysis was done over a small area

rather than a single particle. The carbon and oxygen peak in the EDAX analysis are from the polymer PEG present in the dielectric. From the Fig. 6 (b), it is evident that the diffraction pattern looks like a superposition of single crystal spot patterns as seen in Fig. 3 (b). By comparing the lattice spacing based on rings in the SAED micrograph with the standard atomic spacing along their characteristic hkl index documented in the powder diffraction file, nanoparticles generated by micro-EDM process in this study were verified as poly crystalline metallic copper. It is confirmed that from EDAX and SAED results, the generated particles were of pure metallic copper.

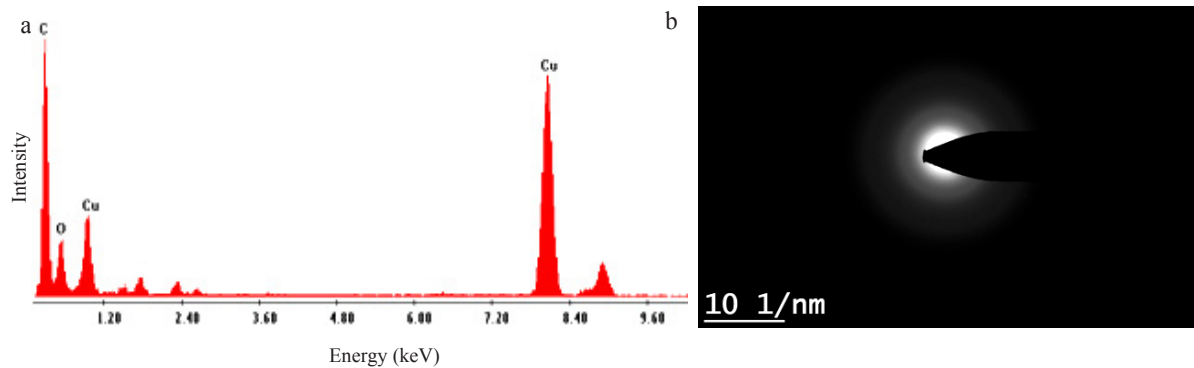


Fig. 6. (a) EDAX pattern of copper nanoparticles with PEG sample; (b) SAED image of copper nanoparticles with PEG sample

3.2. Stability improvement of copper nanofluids

The measured viscosity of fluid samples is shown in Table 2. According to “Equation (1),” the sedimentation velocity follows by,

$$\frac{V_{PEG}}{V_{DIwater}} = \frac{R_{PEG}^2 \times \mu_{DIwater}}{R_{DIwater}^2 \times \mu_{PEG}} \times \frac{V_{PEG}}{V_{DIwater}} = 0.0135 \quad (2)$$

Thus the ratio between V_{PEG} and $V_{DIwater}$ could be determined using the viscosity values (Table 2) and the average diameter values of copper nanoparticles found in both the experiments.

Table 2. Viscosity measurement of base fluids

Sample	Viscosity (centiPoise)
DI Water	11.02
DI Water + PEG	15.405

From “Equation (2),” it was found that there is a reduction of 99% in the sedimentation velocity of copper nanoparticles in PEG nanofluid when compared to DI water based copper nanofluid. It can be seen that there is excellent decrease in the sedimentation of copper nano-particles when PEG is added to the dielectric. Visual inspection of the nanofluid samples confirms the above result. Thus, the dispersion stability of PEG based copper nanofluid as compared to DI water based copper nano fluid is greatly improved.

3.3. Thermal conductivity

The measured thermal conductivity of nanofluid samples are presented in Table 3. Similar working conditions

were maintained for all the experiments so as to keep the concentration of nanoparticles in the different samples constant.

Table 3. Thermal conductivity of base fluids and nanofluids

Sample	Thermal Conductivity (W/m K)
DI water	0.572
DI water + PEG	0.560
Sample 1	0.606
Sample 2	0.656

From the Table 3, it can be seen that the thermal conductivity of copper-water nanofluid (sample1) and copper-water with PEG nanofluid (sample 2) are almost 6 % and 15 % higher than that of pure DI water. It is known that PEG absorb water when exposed to atmosphere, since water has a higher thermal conductivity than that of water-PEG fluid, therefore presence of water in PEG based copper nanofluids could conclude that the measured enhancement is due to the presence of copper nanoparticles and this result is consistent with [23].

As seen from Table 3, the thermal conductivity decreases on the addition of polymer, but after the machining process the copper nanoparticles suspended in the fluid enhance the thermal conductivity. Thermodynamic penalty has to be paid with the use of stabilizers, as there was a reduction in the thermal conductivity on their addition to DI water.

4. Conclusions

In summary, micro-EDM method has been developed to generate copper nanoparticles in both, DI water and water-PEG stabilizing agent. The TEM study shows that the generated copper particles are of spherical shape. The size of copper nanoparticles dispersed in pure DI water lies in the range of 600 nm-1100 nm and in PEG sample lies in the range of 4 nm-10 nm. The size distribution study of generated copper particles indicates that the measurement follows normal distribution. It is observed that when PEG was added to the dielectric, the copper particles in PEG nanofluid have an average size of about 6.0 nm. The EDAX and SAED results show that the generated particles are of pure metallic copper. Experimental results reveal that by using PEG agglomeration of copper nanoparticles is prevented, size of the particles has certainly reduced and excellent dispersion stability of copper particles in PEG nanofluid was obtained. Limited data exist for the thermal conductivity of PEG based copper nanofluid. Thermal conductivity of copper nanofluid was measured using a transient heat line source method. The data show that the thermal conductivity of copper-water nanofluid and copper-water with PEG nanofluid are almost 6 % and 15 % higher than that of pure DI water. This reveals that smaller the particles dispersed in the fluid, better is the thermal conductivity of the fluid as smaller particles would have a larger surface area for better conduction of the heat. Further research on the generation of other nanoparticles by micro-EDM method in the base fluid is on the way.

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