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Brief communication

Fabrication of copper oxide nanofluid using submerged arc nanoparticle synthesis system (SANSS)

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Abstract

The optimal parameters are found for preparing nanofluid in our submerged arc nanoparticle synthesis system (SANSS) using a copper electrode. A suspended copper oxide nanofluid is thus produced at the current of 8.5–10 A, voltage of 220 V, pulse duration of 12 μs, and dielectric liquid temperature of 2°C. The CuO nanoparticle are characterized by transmission electron microscopy (TEM), field emission scanning electron microscope (FESEM), X-ray diffraction (XRD), electron diffraction pattern (SAD) and electron spectroscopy for chemical analysis (ESCA). The equality volume spherical diameter of the obtained copper oxide particle is 49.1 nm, regular shape and narrow size distribution.

Introduction

Copper oxide has shown to be an industrially important material that can be widely used in applications such as gas sensors (Frietsch et al., 2000), magnetic storage (Maruyama, 1998), solar energy transformation (Dai et al., 2000), semiconductors and catalysis (Deng et al., 1996).

In the past years, much effort has been made to the synthesis and characterization of oxide nanoparticles metal. So much less study has been devoted to the preparation and characterization of CuO nanoparticles. Gas phase synthesis and sol–gel processing are traditional methods often applied for nanoparticles synthesis. Several synthetic methods for the preparation of CuO nanoparticles have been reported recently such as the sonochemical method (Kumar et al., 2000), sol–gel technique (Eliseev et al., 2000), one-step solid state reaction method (Xu et al., 2000), thermal decomposition of precursors and co-implantation of metal and oxygen ions (Borgohain et al., 2000; Nakao et al., 2000; Xu et al., 2002). Those are make chemical methods, therefore, in order to generate nanoparticle suspension with desired particle properties (smaller size and narrower size distribution) by physics method. A novel nanoparticle synthesis system, called the submerged arc nanoparticle synthesis system (SANSS), was successfully developed to prepare CuO nanoparticles dispersed uniformly in dielectric liquid (deionized water) suspension, namely 'Nanofluid'.

The present work is focused on the synthesis and characterization of CuO nanoparticles suspension prepared by this system. In this paper, we report process analysis is required to fine tune important process parameters, such as applied electric current, voltage, pulse-duration and the dielectric liquid temperature. It describes the experiments

implemented for process analysis on the SANSS and the outcomes of the controlled process. The product has needle-like structure, narrow size distribution and high virtue.

Experiment instrument and principle

Figure 1 shows the schematic diagram of the SANSS. The generation of nanoparticle suspension is briefly detailed here. A pure copper rod, deployed to prepare nanoparticles, is submerged in dielectric liquid (deionized water) in the vacuum chamber. After the parameters of the system are established, the heating source produces an arc with a high temperature between 6000 and 12000°C to melt the metal rod (Patel et al., 1989; Eubank et al., 1993). The rod is melted and vaporized in the region where the arc is generated. In addition, the deionized water is also vaporized rapidly by the arc at the same time. The high-pressure vapor can induce a quick removal of the vaporized metal. Thus, the vaporized metal in the vacuum chamber undergoes nucleation, growth and condensation, finally turning into nanoparticles dispersed in deionized water. The low-temperature deionized water can condense the submerged metal particles immediately and effectively. Because the parameters, such as the pressure of chamber and the temperature of deionized water, can be controlled within a desired level, the submerged arc can be generated steadily.

In the proposed SANSS, the vaporized metal is processed through three stages, according to the nucleation theory namely nucleation, growth and condensation. In addition, the change of free energy when new phase is formed also influences the saturation during material transformation from solid to gaseous state. High saturation is depended on the temperature difference between the arc and deionized water. In other words, high nucleation rate can be obtained when the metal is vaporized in high temperature and condensed rapidly in low temperature. In case of too low the saturation rate, uniform nanoparticles cannot be obtained by the SANSS. Controlling adequate radiuses of condensation nuclei is significantly essential during the nucleation process because the properties of the particles are mainly determined by this factor. In order to obtain desired nanoparticles, decreasing the critical radiuses of condensation nuclei is important.

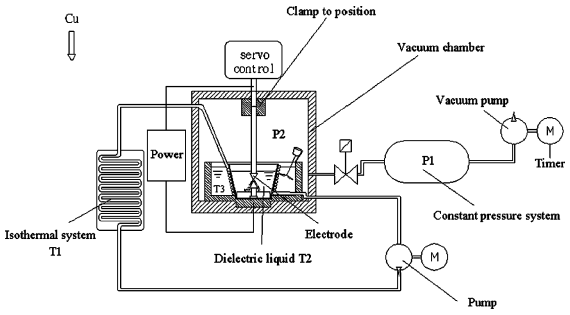

Figure 1. Schematic diagram of SANSS.

Experimental design

The CuO nanofluid is prepared using the SANSS, which consists of a heating source, a cooling unit, and a state control system, wherein the state control system includes an isobaric control system and an isothermal control system. First, a pure copper rod is heated by the submerged arc from the heating source. The state control system is deployed to regulate various process parameters. The governing parameters of these instruments, such as applied electric current, voltage, pulse-duration and the dielectric liquid temperature, are crucial determinants of the nanoparticle suspension. To conduct an effective process analysis, the modulated range of the process parameters was designed to include their full variation range, as shown in Table 1.

After the particles had been vaporized and condensed for a period of time, the nanoparticle suspension was sampled to a collector for inspection and analysis to determine the characteristics of suspension CuO nanofluids, such as particle size and structure. The particles dispersed were safe and pure because they were submerged in deionized water, and thus not contaminated. When the particles were analyzed by transmission electron microscopy (TEM, JEOL2000FX), electron diffraction pattern (SAD, JEOL2000FX), field emission scanning electron microscope (FESEM, JEOL JSM-6700F), the mean of secondary particle size and size distribution obtained from a dynamic light scattering particle-size analyzer (PSA, HORIBA LB-500, Japan) the particle suspension

Table 1. Modulated range of the process parameters applied in SANSS

Electric current I (A)	Pulse duration, t_{on} (μs)	Pulse duration, t_{off} (μs)	breakdown voltage V (V)
0.5–25	2–2400	2–2400	90–220
Volume of chamber (mm^3)	Pressure of chamber (Torr)	Temperature of dielectric liquid (°C)	Volume of dielectric liquid (cc)
1.52×10^7	20–760	2–25	150

could be directly used as the sample for examination without involving complex handling required by other synthesis methods. For the particle X-ray diffraction (XRD, Japan MAC Sience, MXP18) inspection in determining the nanoparticle structure and electron spectroscopy for chemical analysis (ESCA, UK VG Scientific ESCALAB 250) is characterize the purity and the composition of the CuO nanoparticle.

Experimental results and discussion

The nanoparticle suspension is obtained by the SANSS under high temperature and low pressure, in which only Cu, O and H are present. The CuO nanoparticles, instead of pure Cu are generated because coppers react with the oxygen existing in dielectric liquid (deionized water). Figure 2 illustrates the XRD results of the sampled particles. It shows that the prepared nanoparticles are CuO.

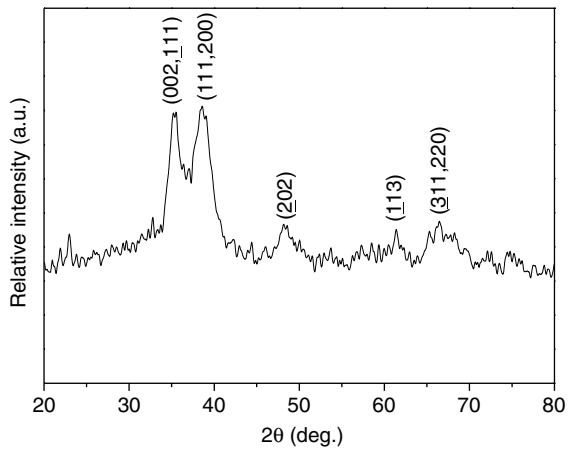

Figure 2. Results of X-ray diffraction of CuO nanoparticle prepared in deionized water.

Effect of applied electric currents on nanofluid

In order to investigate how the magnitude of current affects the CuO particle size, the parameters such as the breakdown voltage, pulse-dura tion, temperature of dielectric liquid and pressure are maintained constant while the current is set at 2, 5, 8 and 15 A. It was found that the particles prepared by the applied electric current at 2 and 15 A can easily precipitate at the bottom of the flask and the nanoparticle suspension becomes transparent in a few hours.

In contrast, the nanoparticle suspension prepared by the applied current equal or more than 8 A, appeared brown. In addition, particles of the sampled suspension did not precipitate onto the bottom of the flask. In other words, the prepared particles are dispersed in the dielectric liquid (deionized water). This phenomenon can be attributed to the fact that surface energy of the prepared particles can generate the effect of Brownian motion that is greater than the gravity caused by sedimentation.

Figure 3 shows the results that the current set at 2 A obtained 231.0 nm, set at 5 A obtained 115.0 nm, set at 8 A obtained 67.6 nm, and set at 15 A obtained 278.0 nm. The standard deviations were 389.3, 200.2, 32.7, and 335.3 nm, respectively (within $\pm\sigma$ measurement range). It is important to clarify that the particles did not precipitate if the applied current about 8 A. Furthermore, the experimental results also suggest that the current about 8 A is applied in SANSS, the smaller and concentrate distribution the particle obtained.

Effect of applied breakdown voltages on nanofluid

The mean of secondary particle size prepared by different breakdown voltages with other process parameters maintained constant, shows in Figure 4. The means of secondary particle size were

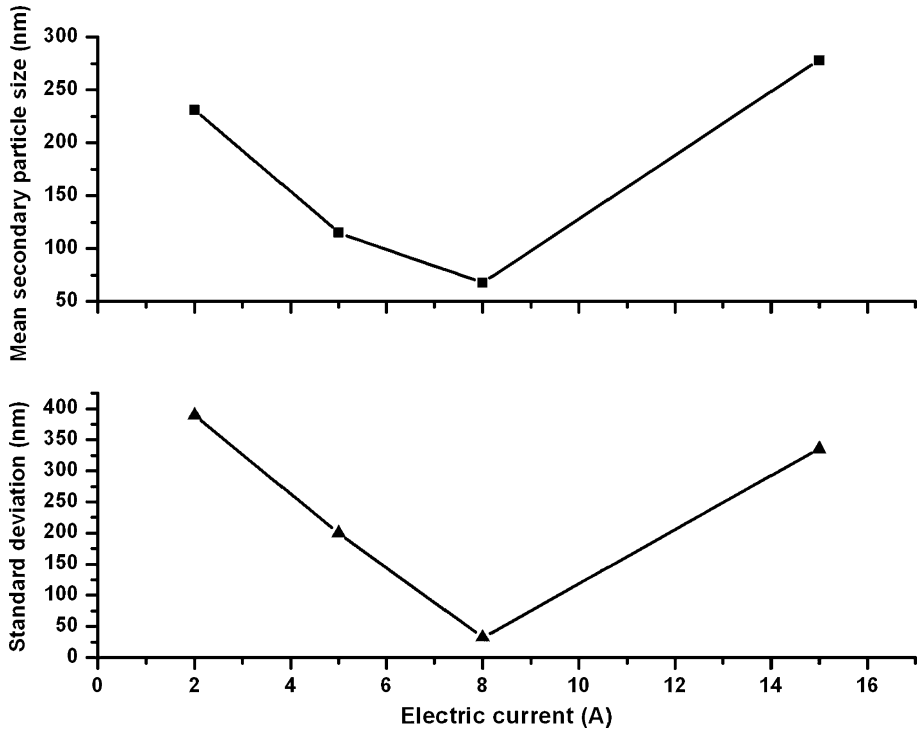

Figure 3. Effect of electric current on the mean secondary particle size and the standard deviations.

68.9, 122.2 and 286.3 nm. The standard deviations were 35.6, 92.2, and 110.6 nm, respectively (within $\pm\sigma$ measurement range). The results indicate that a set electric current accompanied with a breakdown voltage with an alternative-mixing (high and low) pattern can produce particles smaller and concentrate distribution than those with single high- or low-mode of breakdown voltages.

Effect of pulse-duration on nanofluid

The length of pulse-duration of applied electric energy discharged determines the heating pattern (magnitude and frequency) of the SANSS. The melted and vaporized depth zone is deeper when the electrode is machined under longer pulse-duration. Shorter pulse-duration can generally create shallower vaporized zone than longer ones. Thus, to investigate the effect of pulse-duration on particle size, the pulse-duration was set at 12, 25 and 100 μs. From our experimental results, it was identified that the particle size changes with the pulse-duration. As shown in Figure 5, the means of secondary particle size were 73.7, 143.1, and 267.3 nm, respectively, and the standard deviations were 47.6, 76.4, and 141.6 nm, respectively (within $\pm\sigma$ measurement range). The experimental results also suggest that the pulse-duration about 12 μs is applied in SANSS, the smaller and concentrate distribution the particle obtained.

Effect of dielectric liquid temperature on nanofluid

In order to realize how the temperature of dielectric liquid effects the preparation of the nanofluids, the other process parameters maintained constant and the temperature of dielectric liquid is set as 2, 10, and 25°C respectively. The results are shown in Figure 6. The means of secondary particle size were decreased from 84.4 nm to 148.4 and 386.6 nm. The standard deviations were 64.4, 86.0, and 156.0 nm, respectively (within $\pm\sigma$ measurement range). It is proved that the particle size changers with the temperature of dielectric liquid, and the lower temperature is 2°C, the smaller and concentrate distribution particle size is obtained.

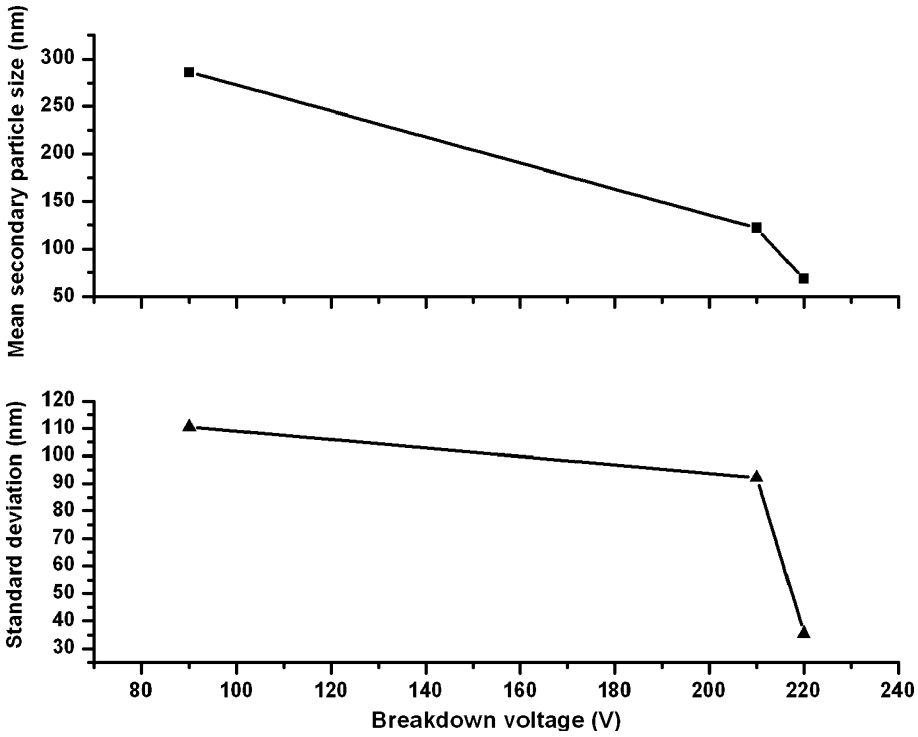

Figure 4. Effect of breakdown voltage on the mean secondary particle size and the standard deviations.

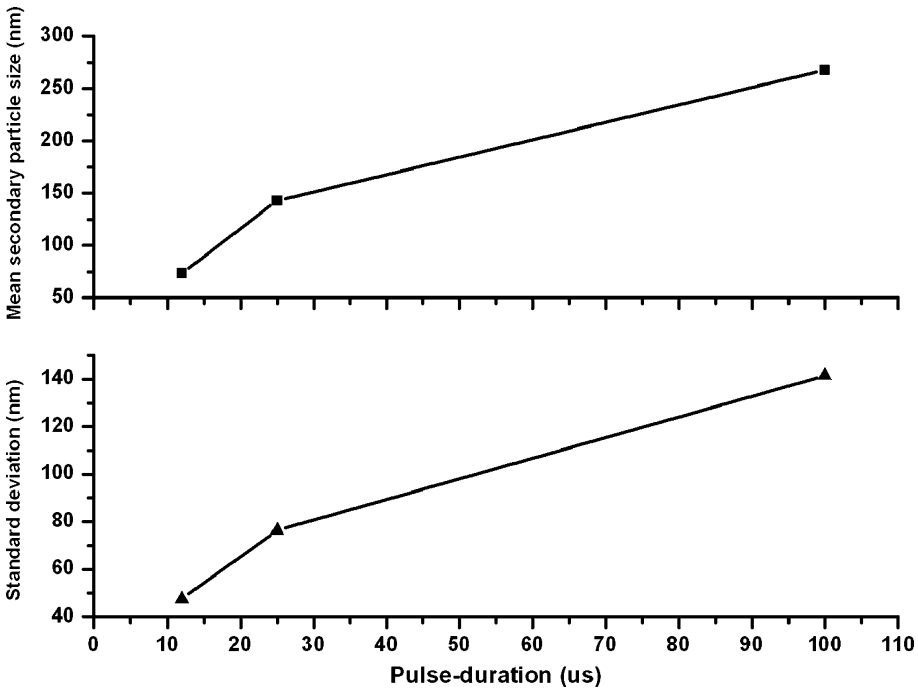

Figure 5. Effect of pulse-duration on the mean secondary particle size and the standard deviations.

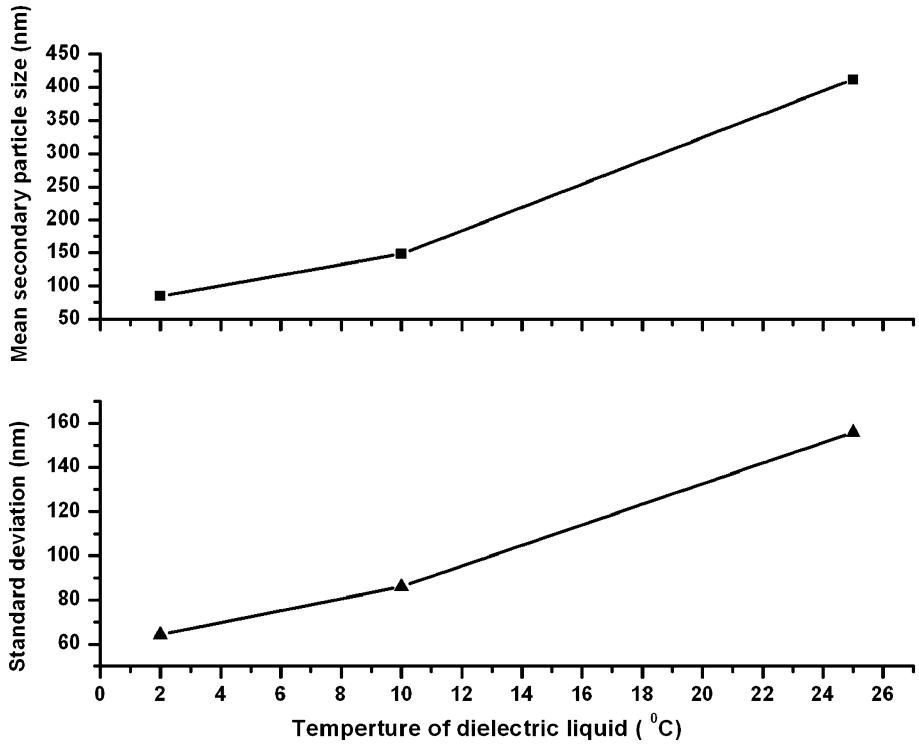

Figure 6. Effect of dielectric liquid temperature on the mean secondary particle size and the standard deviations.

A process example of the SANSS

Taking into consideration all the process parameters and their possible influences on the particle properties, we chose a set of optimal parameters of the SANSS as a process-testing example (shown in Table 2). As seen in Figure 7, the mean of secondary particle size was 49.1 nm and the standard deviation was 38.9 nm (within $\pm\sigma$ measurement range). Figure 8(a) and (b) shows that the TEM image of the nanoparticle prepared by the SANSS, with an average width of 20 nm and length of 80 nm. The SAD image (Figure 8(c)) shows that the particles are well crystallized. The above results demonstrate that the size of CuO particles prepared by the SANSS can be effectively controlled

Table 2. Selected optimal parameters used in the SANSS preparing CuO nanofluid

Process parameters	Description
Working breakdown voltage (V)	220 V
Electric current (I)	8–10 A
On-time pulse duration (t_{on})	25 μs
Off-time pulse duration (t_{off})	25 μs
Pressure of chamber (P)	30 Torr
Temperature of dielectric liquid (T)	2°C
Electrode diameter (D)	12 mm
Dielectric liquid	Deionized water

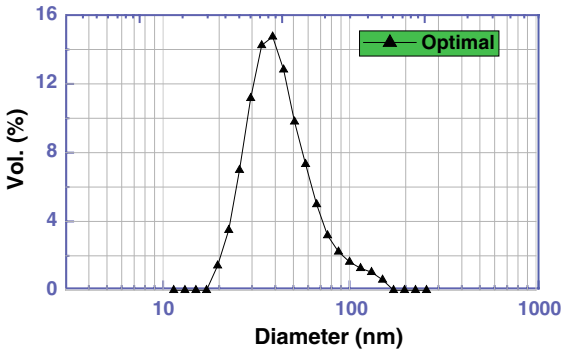

Figure 7. Size distribution of CuO nanofluid prepared by the optimal parameters, the mean of secondary particle size was 49.1 nm and the standard deviation was 38.9 nm (within $\pm\sigma$ measurement range).

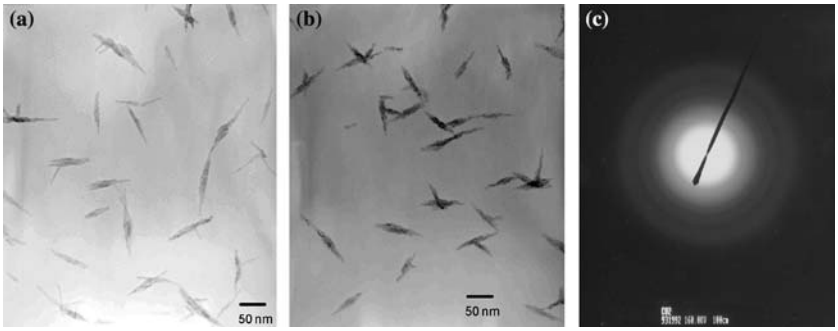

Figure 8. (a, b) TEM image and (c) SAD pattern of the preparing CuO nanoparticle.

within a nanoscale range. The pressure is considered as an important process parameter to be controlled for obtaining the desired nucleation and growth of the nanoparticles in the system (SANSS). Similar ideas have been formulated by Tshih and co-workers by the experimental results indicate that the nanoparticles can be prepared when the operating pressure is kept constant at 30 Torr (Tshih et al., 2003). However the morphology of particles remains unchanged whether the pressure varies or not. The pressure control techniques used in the SANSS have been successfully developed with the developed balance-operating vacuum chamber as a working reservoir for nanoparticle fabrication by integrating direction flow valves (Type: two-ways-two-positions) and the feedback control algorithm. It has been identified that the pressure in the vacuum chamber can be maintained at a stable condition, which is crucial to successful fabrication of nanoparticles.

The morphology of the CuO nanoparticle prepared in deionized water reveals a needle-like structure, as reported in Figure 9 FESEM image. Figure 10 is shown the ESCA result of the prepared CuO nanoparticle. The pattern used AlKα X-ray as the excitation source and choosing C 1s (284.6 eV) as the reference line. All the peaks are corrected accordingly. No peaks of other elements expect Cu, O and C were observed in the picture, indicating the high purity of the product.

The effective thermal conductivity of the CuO nanofluid can be much higher than the normally used industrial heat transfer fluid. Carrying an excellent thermal conductivity, CuO nanofluid can be used in machine tool is in motion, the magnetic filed created by the power source and dynamic systems would affect the material properties of the circulation fluid. Chang and co-workers, develops a magnetic environment system to simulate an environment having additional magnetic field

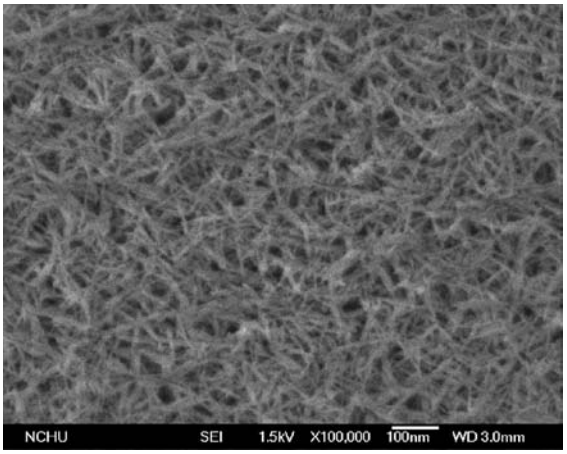

Figure 9. FESEM image of the preparing CuO nanoparticle.

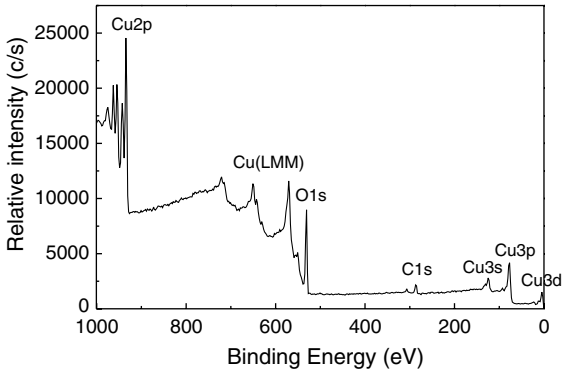

Figure 10. Electron spectroscopy for chemical analysis of the preparing CuO nanoparticle.

imposed onto the fluid (Chang et al., 2004). The results of affected by additional magnetic field, the repulsion force between the particles decreases, causing the electric potential to drop and allowing the nanoparticles to aggregate. It is very important to investigate the effect of magnetic field on the nanofluid.

Conclusions

This article introduced a novel system design of the SANSS for preparation of copper dioxide nanoparticle suspension (nanofluid). The experimental results indicate that the CuO nanofluid with the means of secondary particle size of the obtained copper oxide particle is 49.1 nm and the standard deviation was 38.9 nm can be successfully prepared. Most important, the established SANSS is demonstrated to be effective in avoiding particle aggregation and producing uniformly distributed and well-controlled size of CuO nanoparticles dispersed in deionised water suspension. The production of CuO nanofluid can be wide applications in various fields.

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