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Research Paper

Synthesis, characterization, thermal conductivity and sensitivity of CuO nanofluids

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HIGHLIGHTS

- CuO nanoparticles from two different starting precursors show different properties.
- CuO–distilled water nanofluids give superior thermal conductivity results.
- Thermal conductivity is more sensitive to volume percent change at higher concentration.
- CuO–distilled water nanofluids show higher thermal conductivity sensitivity.

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ABSTRACT

In the present work CuO nanoparticles have been synthesized using wet chemical method and characterized using UV–Vis, PL, DLS, SEM, TEM and XRD. To evaluate effect of precursor salt CuO nanoparticles were synthesized using two different starting precursors viz copper acetate and copper sulphate. It was observed that keeping all other parameters fixed, CuO nanoparticles synthesized from copper sulphate were of regular shape and smaller size as compared to copper acetate based CuO nanoparticles. CuO nanoparticles synthesized from copper sulphate precursor were used for preparation of nanofluids in distilled water, ethylene glycol and engine oil base fluids using two step approach. These prepared nanofluids were examined for their potential of modulation in thermal conductivity. Thermal conductivity was measured using KD2 Pro which is based on transient line heat source method. 40% increment in thermal conductivity was observed for distilled water based nanofluids for change in temperature from 10 to 70 °C and concentration variation from 0 to 2 vol%. In case of ethylene glycol and engine oil based nanofluids thermal conductivity enhancement was 27% and 19%. Sensitivity analysis for thermal conductivity was also performed. Sensitivity analysis shows that at higher concentration sensitivity increases and varies significantly for different base fluids.

1. Introduction

Nanofluids are engineered colloidal suspensions of nanoparticles in fluids that are expected to exhibit superior properties relative to those of conventional heat transfer fluids. Nanoparticles offer extremely large total surface area and therefore have great potential for applications in heat transfer. Among the oxides of transition metals, copper oxide (CuO) nanoparticles (NPs) are of special interest because of their efficiency as nanofluids in heat transfer applications. CuO has been investigated as potential material for nanofluids in heat transfer applications [1], catalysts for the water–gas shift reaction [2], steam reforming [3], CO oxidation of automobile exhaust gases [4], photocathodes for photoelectrochemical water splitting application [5], etc.

CuO have been prepared by sono-chemical method [6,7] sol–gel technique [8], one-step solid state reaction method at room temperature [9], electrochemical method [10], thermal decomposition of precursors [11], wet-chemistry route [12], alkoxide based preparation [13], hydrothermal process [14], solid-state reaction in the presence of a surfactant [15], etc. Haitao et al. developed a wet chemical method to prepare stable CuO nanofluids [16]. Influences

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After preparation

After 10 days

Fig. 1. Images of prepared CuO nanofluids.

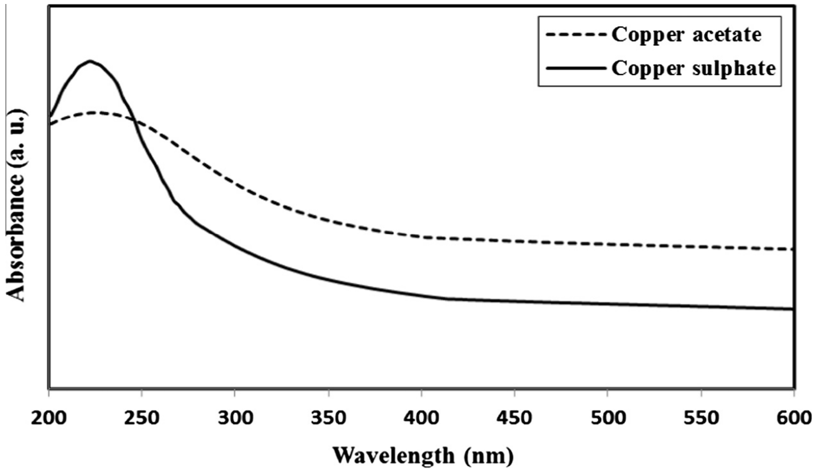

Fig. 2. UV–Vis absorption spectra of synthesized CuO NPs.

of synthesis parameters, such as kinds and amounts of copper salts, reaction time were studied. Concentration of copper acetate and reaction time affected size and shape of clusters of primary nanoparticles. However, effect of different precursor salts on size of the synthesized nanoparticles is not well reported.

Rejith et al. synthesized CuO nanoparticles by microwave assisted solvo-thermal method using copper acetate and urea as precursors [17]. 10–14 nm sized particles having single phase monoclinic structure with spherical morphology were obtained. Synthesis of CuO nanoparticles using different solvents by low cost sol–gel route was also reported [18]. Their XRD analysis indicated that crystallite size and strain are higher for CuO nanoparticles synthesized using propanol as solvent. Swarnkar et al. synthesized copper-oxide nanoparticles by laser ablation of copper metal in aqueous solution of sodium dodecyl sulphate [19]. Zhu et al. synthesized CuO nanofluid by transforming an unstable $Cu(OH)_2$ precursor to CuO in water under ultrasonic vibration, followed by microwave irradiation [20].

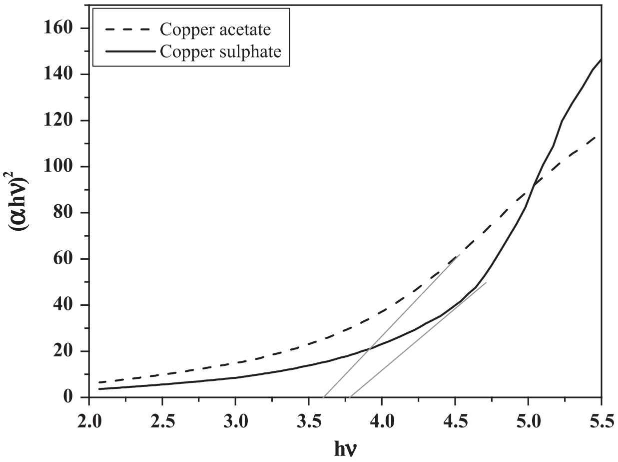

Fig. 3. Direct band gap Tauc relation plot of synthesized CuO NPs.

Lee et al. prepared CuO nanofluid by dispersing commercial CuO nanoparticles in water under ultrasonic vibration [21]. 31.6% enhancement in thermal conductivity with 1% volume fraction of 8 nm CuO in water and 54% enhancement with CuO/EG suspension was shown [22]. Review on heat transfer enhancement through nanofluids is also performed [23]. Nanofluids made of CuO particles of 10–30 nm in length and ethylene glycol were studied for thermal conductivity enhancement [24]. Thermal conductivity measurement shown that substantial enhancement in thermal conductivity with respect to particle concentration is attainable only when particle concentration is below dilute limit. Thermal conductivity enhancement of 3% for 0.1 wt% concentration of copper oxide nanoparticles in engine oil was observed [25]. Nemade and Waghuley reported enhancement of thermal conductivity of CuO/H_2O nanofluids [26]. However, systematic study about effect of different base fluids, temperature and concentration on thermal conductivity is scarce and reported results show lack of consistency. Much more investigations are required to find effects of these parameters, for practical applications of nanofluids.

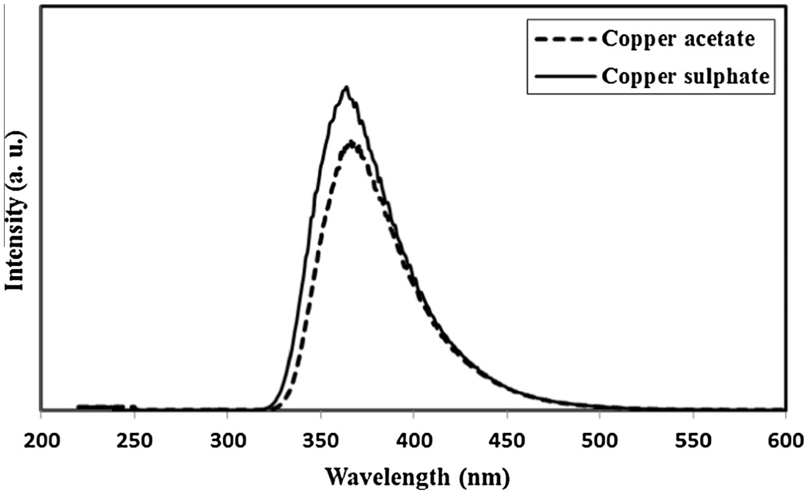

Fig. 4. PL spectra of synthesized CuO NPs.

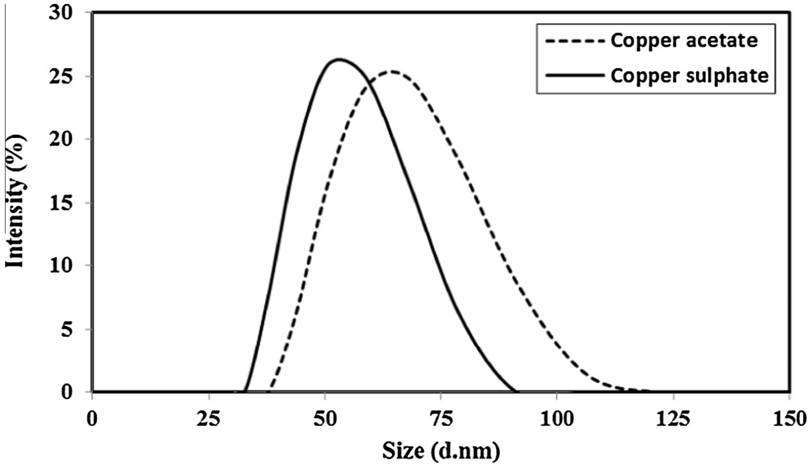

Fig. 5. Particle size distribution of synthesized CuO NPs.

In our study copper oxide nanoparticles have been investigated for their potential in modulation of thermal conductivity. Copper oxide nanoparticles were synthesized by wet chemical method and characterized using UV–Vis, PL, DLS, SEM, TEM and XRD. To evaluate effect of starting precursors on properties of synthesized nanoparticles, CuO nanoparticles have been synthesized using two different precursors. Synthesized copper oxide nanoparticles have been dispersed in distilled water, ethylene glycol and engine oil at different concentration and studied at different temperature values for enhancement in thermal conductivity. Motivation behind the present work was to conduct a systematic study for the effect of temperature, concentration and base fluid on thermal conductivity enhancement of nanofluids, to overcome widespread discrepancies available throughout the literature. Further effects of precursor salts on properties of synthesized nanoparticles were also evaluated.

2. Materials and method

2.1. Synthesis and characterization of CuO nanoparticles

Copper oxide nanoparticles were synthesized using wet chemical method followed by annealing of the samples at high temperature for improving purity. To evaluate the effect of precursor copper oxide nanoparticles were synthesized using two different precursors. Copper acetate, copper sulphate and sodium hydroxide (pellets) used in the synthesis were of analytic reagent grade. To synthesize copper oxide nanoparticles, 1 M aqueous solution (200 ml) of copper acetate/copper sulphate was placed on preheated hot plate and 2 M aqueous solution (200 ml) of sodium hydroxide was added to the above solution drop by drop through vigorous stirring till pH reaches 6–7. Large amount of black precipitate was formed immediately. Precipitate was centrifuged and washed 3–4 times with distilled water. Hence obtained copper oxide nanoparticles were dehydrated and annealed in air furnace at 450 °C for 2 h to improve purity. 14.56 and 14.91 g CuO nanoparticles were obtained using copper acetate and copper sulphate, respectively. Synthesized nanoparticles were characterized using UV–Vis Spectrophotometer (UV–Vis; Shimadzu; UV-1800), Photoluminescence Spectrophotometer (PL; Shimadzu; RF-5301), Dynamic Light Scattering (DLS; Malvern; Nano-ZS), Scanning Electron Microscopy (SEM; Carl Zeiss; EVO-18), Transmission Electron Microscopy (TEM; Tecnai; FEI G2 S-Twin) and X-ray Diffraction (XRD; PANalytical; X'Pert PRO). Characterization results have shown that nanoparticles synthesized from copper sulphate precursor were of small size with narrow size distribution and spherical morphology as compared to nanoparticles synthesized using

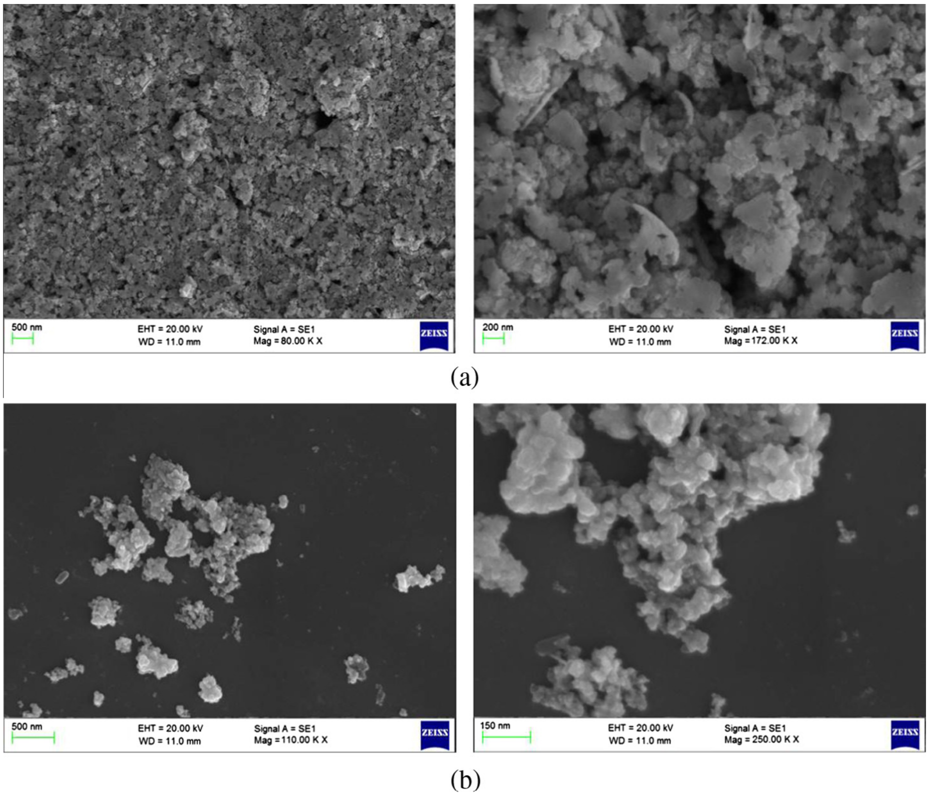

Fig. 6. SEM images of CuO NPs synthesized from (a) copper acetate and (b) copper sulphate.

copper acetate precursor keeping all other synthesis parameter fix. So in the present study nanoparticles synthesized using copper sulphate precursor have been used for preparation of nanofluids throughout the investigation.

2.2. Preparation of nanofluids

Nanofluids from the synthesized nanoparticles were prepared using two step approach. CuO nanoparticles were taken as synthesized from copper sulphate precursor. Ultrapure (type 1) water (Polisher; Biopak), ethylene glycol (Merck; AR) and engine oil (HP; RACER 4) were used as base fluids throughout the investigation. For preparation of different volume percent concentration nanofluids of CuO nanoparticles in base fluids, required amount of synthesized CuO nanoparticles were weighed using sensitive balance (Electronic Balance; Precisa; XB 220A) having resolution of 0.0001 g. The weighed amounts of nanoparticles were mixed in 100 ml base fluid using mortar and pestle. For proper mixing of nanoparticles in base fluid, nanofluids suspension was stirred for 1 h using magnetic stirrer (Tarsons; SPINOT). For increasing stability and removal of agglomeration, nanofluids suspensions were sonicated for 30 min using probe (10 mm diameter) ultrasonic processor (Electrosonic; E1-250 W) at 220 V followed by ultrasonic vibrations for 90 min using water bath ultrasonic cleaner (Toshcon; SW4). Stability of the prepared nanofluids suspension was checked for 10 days and no trace of visible particle sedimentation was observed that shows absence of aggregation and agglomeration (Fig. 1).

2.3. Measurement of thermophysical properties

These prepared nanofluids were investigated for change in thermal conductivity. Measurement of thermal conductivity was done using KD2 Pro Thermal Properties Analyzer of Decagon Devices, Inc. USA using KS-1 sensor which is based on transient line heat source method. For an accurate measurement of thermal properties sample was kept absolutely still, temperature of sample was kept constant during the measurement. Volume, diameter and length of nanofluid container taken were 50 ml, 30 mm and 120 mm, respectively which is sufficiently large to be considered as infinite in comparison to sensor needle (1.3 mm diameter, 60 mm length). The sensor needle was oriented vertically during the measurement to prevent convection as convection or bulk movement of the sample results in error in the thermophysical properties measurement. Error from convective heat exchange is often very large, rendering the thermal properties measurement useless and must be avoided. So for convection free and temperature controlled measurement of thermophysical properties following procedure was adopted for each sample under investigation:

1. Each sample was heated or cooled to achieve desired temperature in refrigerated/heating circulator (Julabo; F30) with sensor needle inserted in sample.
2. Sample temperature was equilibrated to desired temperature for 2 h.
3. Circulator was allowed to become absolutely still for 10 min before measurement was taken.
4. For each sample at fixed temperature and concentration, three different measurements were performed to obtain accuracy in results and then average of these three is reported here. Deviation in three sets of measurement was less than ±5% which shows accuracy of measurements. (After first measurement circulator was turned on for 30 min to equilibrate, then turned off to become absolutely still for 10 min and then second measurement was performed. Similar procedure was adopted for third measurement. This cycle was taken for nullifying effect of temperature increase in vicinity of probe due to transient heat given to probe during measurement and temperature decrease

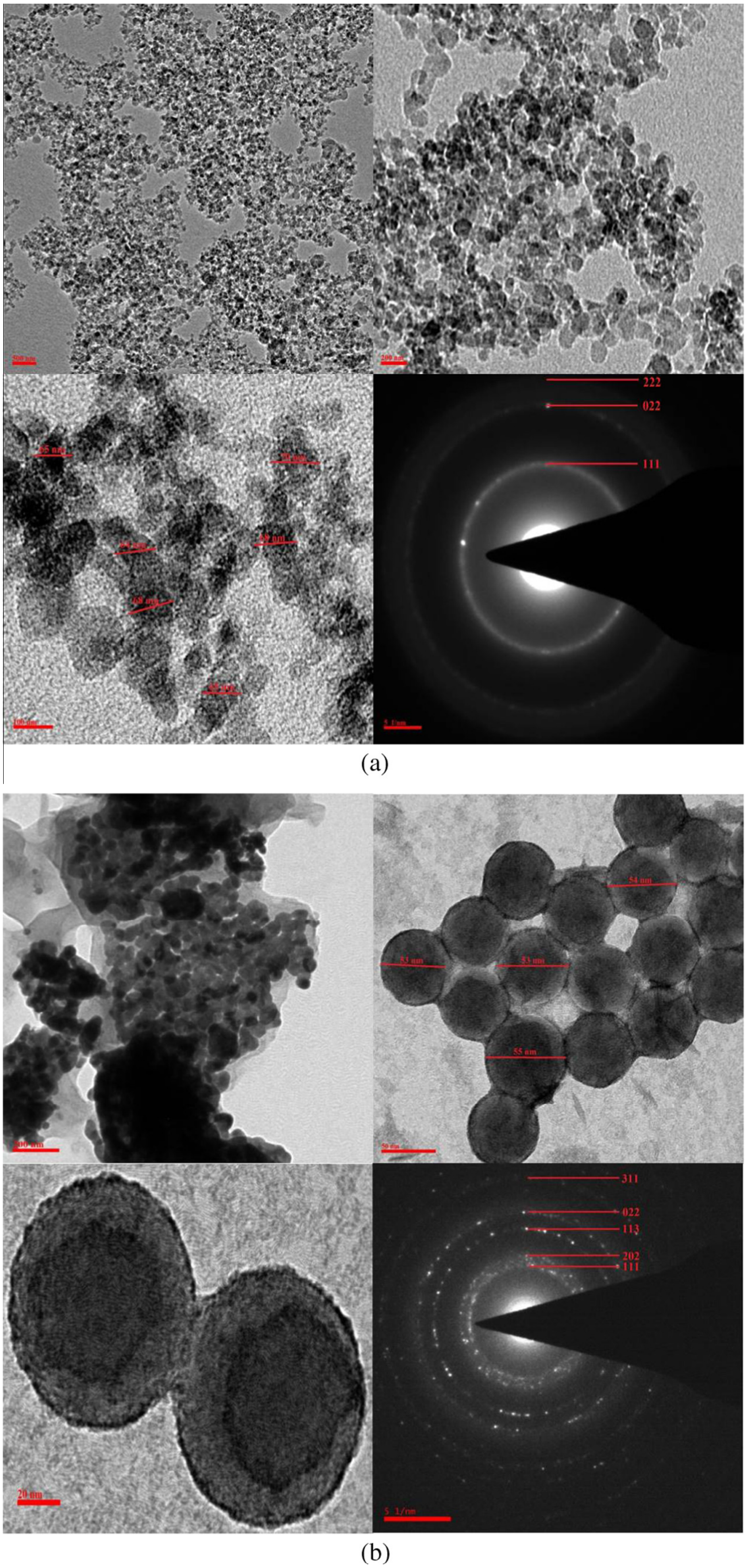

Fig. 7. TEM images of CuO NPs synthesized from (a) copper acetate and (b) copper sulphate.

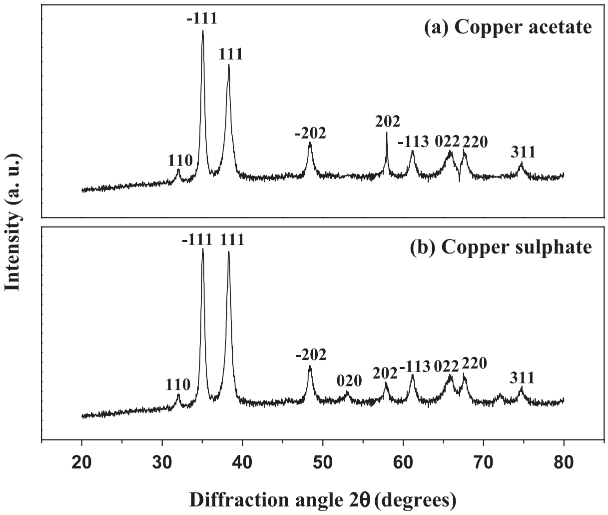

Fig. 8. XRD pattern of synthesized CuO NPs.

Table 1
Summary of characterization results of synthesized CuO nanoparticles.

S. no.	Characterization technique	Parameter investigated	CuO nanoparticles synthesized from		Figure
			Copper acetate	Copper sulphate	
1	UV–Vis	Absorption maximum peak wavelength (nm)	225	222	2
2	UV–Vis	Tauc relation direct band gap (eV)	3.59	3.78	3
3	PL	Emission spectra wavelength (nm)	366	364	4
4	DLS	Weighted average particle size (nm)	66	55	5
5	SEM	Shape	Irregular	Spherical	6
6	TEM	Average particle size (nm)	67 (SD 3)	54 (SD 2)	7
7	XRD	Material identification	CuO monoclinic	CuO monoclinic	8

due to above step and reset it to equilibrium.) To ensure reproducibility of results certain sets of experiment were performed after 10 days. The results hence obtained were again within ±5% of previously obtained results.

Verification of sensor performance was done using glycerin provided with KD2 Pro having thermal conductivity 0.285 W/(m K) at 20 °C as reported by manufacturer. To conduct performance verification, sensor needle was inserted fully into the standard, oriented vertically and centered in the vial without touching side of the vial. Before taking measurement, sample was equilibrated at 20 °C using the procedure mentioned above for our experimental samples. Average thermal conductivity of three sets of reading for the glycerin was 0.279 W/(m K) at 20 °C which falls in the range of ±5% accuracy of standard value as reported by manufacturer.

Samples used in the study were dispersion of synthesized copper oxide nanoparticles, from copper sulphate precursor, in different base fluids (Distilled water, Ethylene glycol and Engine oil) at varying concentration (0, 0.25, 0.50, 0.75, 1.00, 1.25, 1.50, 1.75 and 2.00 vol%). For each such sample thermal conductivity was measured at different temperatures (10, 20, 30, 40, 50, 60 and 70 °C) of nanofluids.

Table 2
Percent increase in thermal conductivity of CuO NPs based nanofluids for different base fluids at different concentrations in the temperature range of 10 to 70 °C.

Concentration (vol%)	Base fluids		
	Distilled water	Ethylene glycol	Engine oil
0	13	5	5
0.25	18	6	5
0.50	19	6	5
0.75	19	7	6
1	19	8	7
1.25	19	8	7
1.50	19	8	7
1.75	19	9	7
2	19	10	8

2.4. Thermal conductivity sensitivity analysis

Sensitivity analysis is performed to determine that how much a quantity is sensitive to change in different parameters [27]. For definite change in volume percent concentration of nanoparticles in base fluid thermal conductivity sensitivity analysis is performed. For 100% increase in volume concentration at different volume concentrations, percent change in thermal conductivity is calculated as base condition for sensitivity analysis.

3. Results and discussion

3.1. Characterization of CuO nanoparticles

3.1.1. UV–Vis

Fig. 2 shows absorption spectra of synthesized copper oxide nanoparticles recorded by UV–Vis Spectrophotometer. Two different graphs correspond to synthesized copper oxide nanoparticles from two different precursors i.e. copper acetate and copper sulphate. Nanoparticles synthesized from copper acetate shown absorption maximum at 225 nm and absorption peak at 222 nm was obtained for copper oxide nanoparticles synthesized from copper sulphate that shows blue shift in absorption maximum. These peaks correspond to inter band transition from deep level electrons of valance band. The small blue shift in absorption maximum peak wavelength refers to different size of synthesized nanoparticles from two precursors which is in agreement of shifting of energy levels with nanoparticle size.

Calculated direct band gap energy using Tauc relation plots (Fig. 3) are 3.59 eV and 3.78 eV for samples synthesized from copper acetate and copper sulphate respectively. Both the samples shown blue shift in direct band gap as compared to bulk value (3.25 eV) [18] that may be attributed to the quantum confinement effect [28].

3.1.2. PL

Fig. 4 shows emission spectra of copper oxide nanoparticles synthesized from two different precursors recorded by PL Spectrophotometer. Samples were excited at their absorption peak maximum obtained from UV–Vis spectra which is 225 nm and 222 nm for samples prepared form copper acetate and copper sulphate respectively. Emission peaks in PL spectra were obtained at 366 nm and 364 nm for the two samples prepared from copper acetate and copper sulphate respectively which corresponds to band-edge emission [29]. The difference in band-edge emission for the two samples is due to shifting of levels which may be attributed to different size of the synthesized nanoparticles using two precursors.

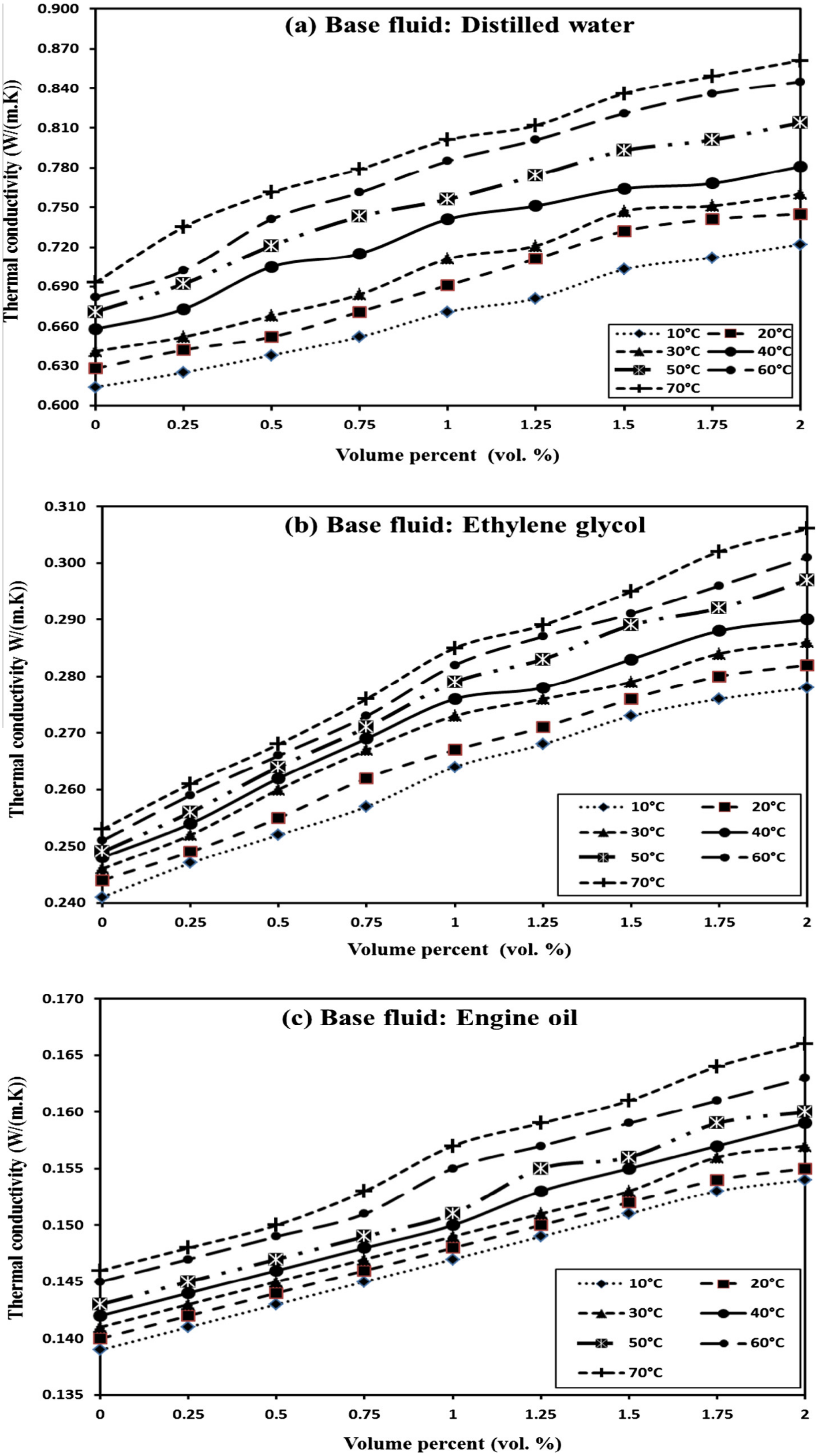

Fig. 9. Enhancement in thermal conductivity of nanofluids with increase in concentration of CuO NPs in different base fluids at different temperatures.

3.1.3. DLS

Size and zeta potential measurement was done using DLS. Particle size distribution shows (Fig. 5) weighted average particle size of 66 and 55 nm for samples prepared from copper acetate and copper sulphate precursors respectively, which confirms that synthesized particles are of nano size.

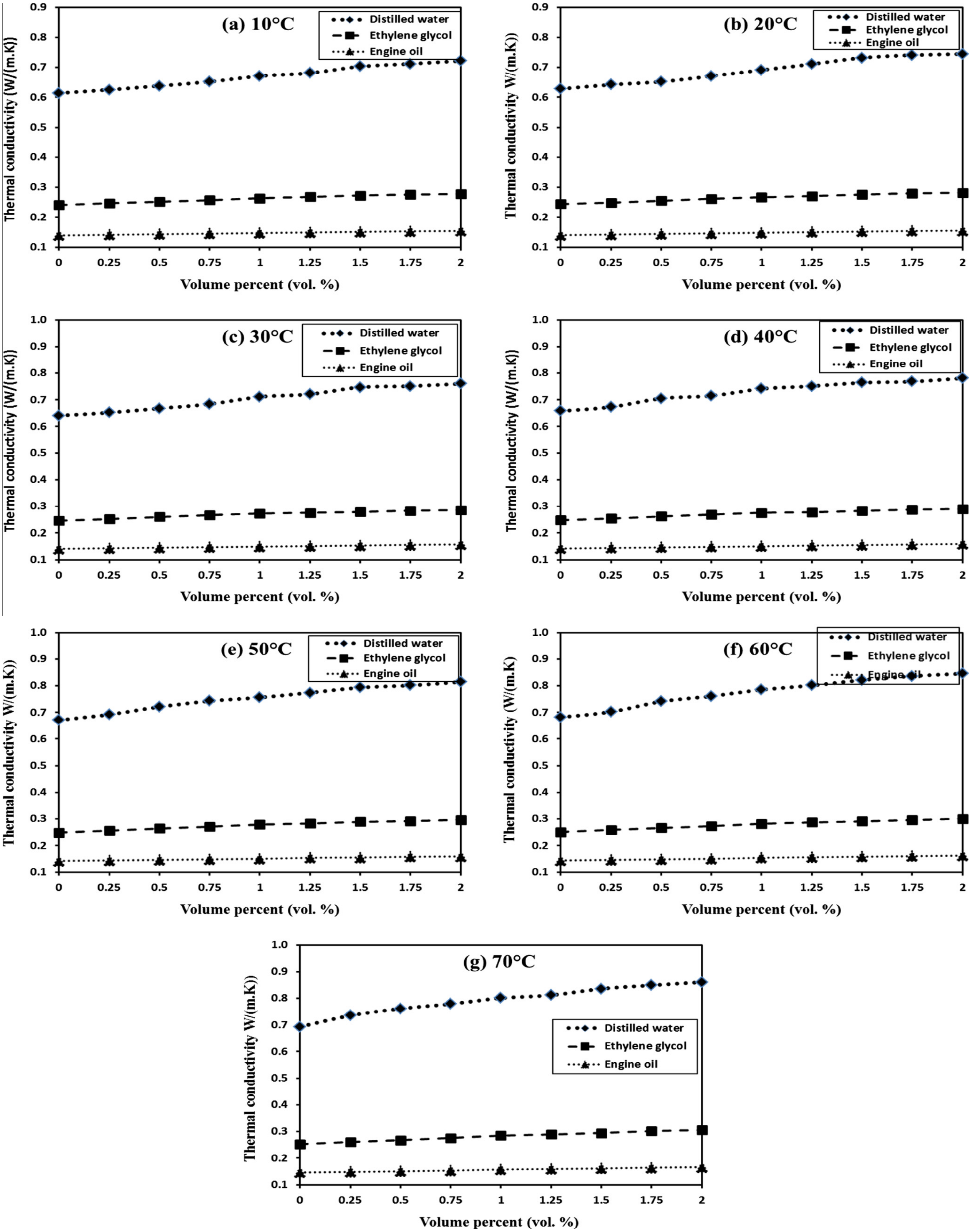

Fig. 10. Enhancement in thermal conductivity of nanofluids with increase in concentration of CuO NPs at different temperatures in different base fluids.

Table 3
Percent increase in thermal conductivity of CuO NPs based nanofluids for different base fluids at different temperature in the concentration range of 0–2 vol%.

Temperature (°C)	Base fluids		
	Distilled water	Ethylene glycol	Engine oil
10	18	15	11
20	19	16	11
30	19	16	11
40	19	17	12
50	21	19	12
60	24	20	12
70	24	21	14

3.1.4. SEM

SEM images of two samples prepared from different precursors are shown in fig. 6. Images show nano size distribution of synthesized nanoparticles along with formation of few clumps.

3.1.5. TEM

Fig. 7 shows TEM images of samples synthesized from two different precursors. Fig. 7(a) indicates average particle size 67 nm and standard deviation of 3 nm for samples synthesized from copper acetate. For copper sulphate precursors, average particle size of 54 nm and standard deviation of 2 nm were obtained (Fig. 7(b)). Copper oxide nanoparticles synthesized from copper sulphate precursor were smaller in size. Images also confirm no aggregation among synthesized nanoparticles as individual particle boundary can easily be identified. Electron diffraction pattern of copper oxide nanoparticles is also shown. Small spots forming rings confirms the presence of polynanocrystalline nature of synthesized nanoparticles. Each spot arises out due to Bragg's reflection.

3.1.6. XRD

Fig. 8 shows XRD pattern of synthesized CuO nanoparticles using two different precursors. All the obtained peaks are well matched for both precursors and also consistent with the JCPDS card (048–1548) with no impurity peak. Results confirm synthesis of single phase CuO nanoparticles with monoclinic structure. The obtained results are well consistent with previously reported literature [18,28].

Table 1 lists summary of characterization results obtained for synthesized CuO nanoparticles from copper acetate and copper sulphate precursors.

Characterization results show that CuO nanoparticles synthesized from copper sulphate precursor is smaller in size and more uniform in shape as compared to CuO nanoparticles synthesized from copper acetate precursor. This variation in size and shape is due to the effect of anions on growth orientation and process of nanoparticles formation by adsorption or coordination interaction of anions with special crystal face of particles [16]. Brownian motion of nanoparticles in nanofluids are conjectured to play key roles on determining effects of the temperature on thermal conductivity enhancement of nanofluids [30]. According to Einstein diffusion theory [31], Brownian velocity of a nanoparticle increases greatly with decrease of diameter of nanoparticles that leads to enhanced heat transfer. So in the present study copper oxide nanoparticles synthesized from copper sulphate precursor (smaller in size), having average particle size 55 nm, have been used for modulation of thermophysical properties.

3.2. Measurements of thermophysical properties of CuO nanofluids

Significant variation in thermal conductivity was obtained for CuO based nanofluids. Fig. 9 shows variation in thermal conductivity of CuO based nanofluids with different base fluid namely distilled water, ethylene glycol and engine oil. Fig. 9(a) shows that keeping the base fluid distilled water, increase in concentration of CuO nanoparticles in base fluid enhances thermal conductivity that further enhances with increase of temperature. Higher thermal conductivity of CuO nanofluids can be attributed to enhanced heat transfer that mainly depends on available surface area and heat transfer characteristics of material. Both water and CuO nanoparticles [32] shows good heat carrying capacity and nanoparticles in nanofluids possess relatively larger surface area that leads to enhanced heat transfer. Increase in concentration of nanoparticles provides increased surface area for heat transfer that leads to enhanced thermal conductivity. Brownian motion of particles in fluids also affects heat transfer [33]. Smaller the particles size and higher the temperature more will be Brownian motion that in turn enhances thermal conductivity through increased heat transfer. Highest increment in this case was of 40% between sample at 10 °C having 0% CuO nanoparticles (i.e. base fluid distilled water) and sample at 70 °C having 2 vol% CuO nanoparticles in distilled water which is also a significant value. Similar trend of results have also been reported earlier [16].

Fig. 9(b) shows thermal conductivity variation of nanofluids for ethylene glycol as base fluid and CuO as nanoparticles. Increment in thermal conductivity of CuO nanoparticles based nanofluids for base fluid ethylene glycol at 10 °C and 2 vol% nanofluids at 70 °C was 27% which is also smaller but significant value as obtained in case of distilled water.

Variation in thermal conductivity of CuO/engine oil based nanofluids is shown in fig. 9(c). Simultaneous increase of temperature and volume percent from base fluid engine oil at 10 °C to 2 vol% CuO nanoparticles based nanofluids at 70 °C gives 19% increment in thermal conductivity which is good enough but still half the value for distilled water (40%) and even less than ethylene glycol (27%) based nanofluids. The reason for such large deviation in rate of enhancement is that different base fluids have different thermal conductivity and heat carrying capacity. Rate of enhancement of thermal conductivity depends on strength of the solid like interfacial layer [34] that directly depends on interaction between nanoparticles and base fluid. That is why the rate of enhancement in thermal conductivity is different for the nanofluids with three different base fluids for similar nanoparticles, concentration and temperature. Higher rate of thermal conductivity enhancement for distilled water shows the strong interaction between CuO nanoparticles and base fluid as compared to ethylene glycol and engine oil base fluids. The results are also in close agreement with reported results of experimental evaluation of engine oil properties containing CuO nanoparticles [25]. Results are summarized in Table 2.

Fig. 10 shows a comparative account of thermal conductivity variation of nanofluids for different base fluids with increasing concentration at different temperatures. Results indicate that engine oil based nanofluids shows inferior performance with respect to volume percent increase in concentration, compared to distilled water and ethylene glycol based nanofluids. So for engine oil based nanofluids higher concentration may be required for efficient use in heat transfer applications. Results are summarized in Table 3.

3.3. Sensitivity analysis of CuO nanofluids thermal conductivity

Fig. 11 show results of thermal conductivity sensitivity analysis for three base fluids (Distilled water, Ethylene glycol and Engine oil) at temperatures (10, 20, 30, 40, 50, 60 and 70 °C) for volume concentration (0.25%, 0.50%. 0.75% and 1%). Results show that thermal conductivity for CuO nanofluids is sensitive to change in temperature and volume concentration. No definite trend was

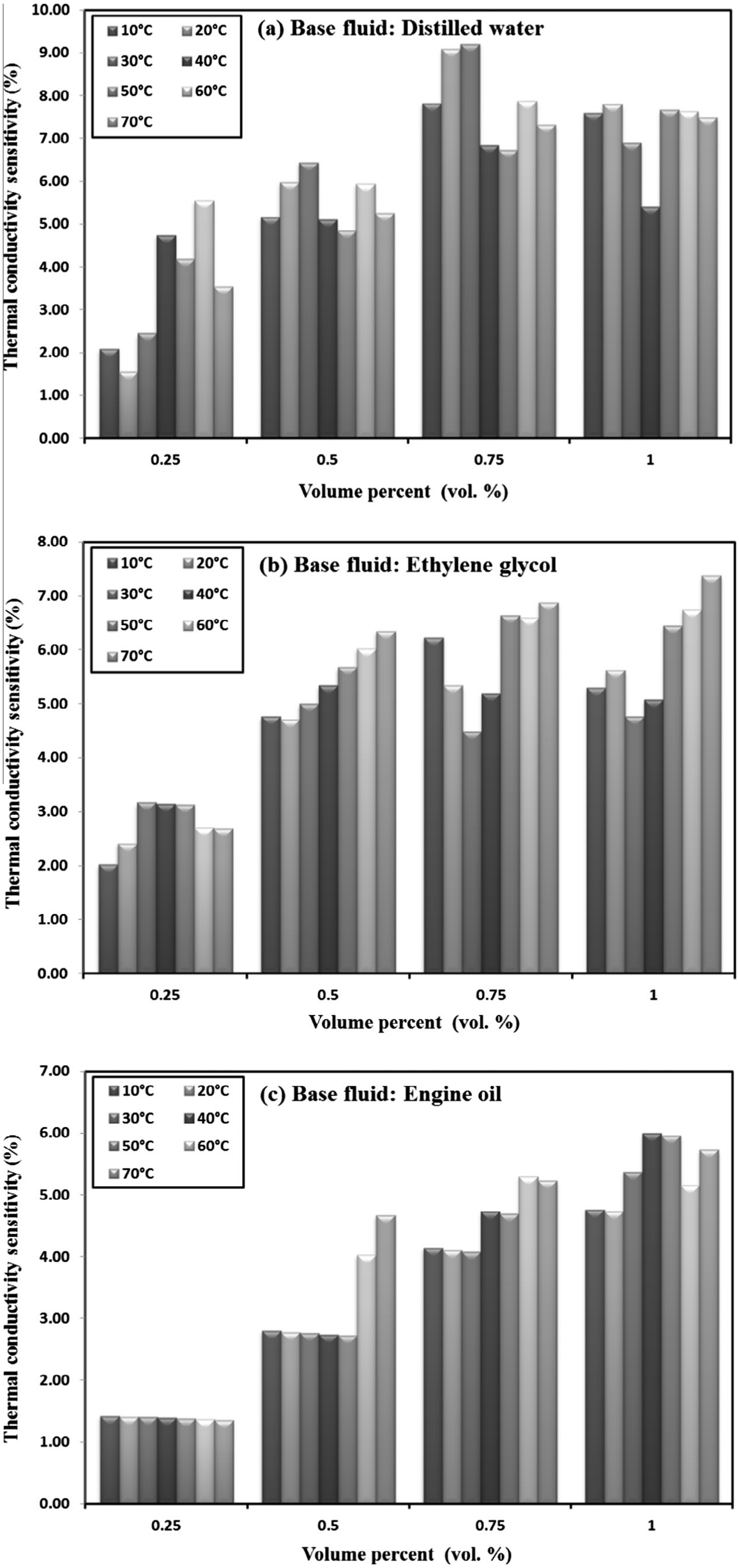

Fig. 11. Sensitivity analysis for thermal conductivity of nanofluids with increase in concentration of CuO NPs in different base fluids at different temperatures and concentrations.

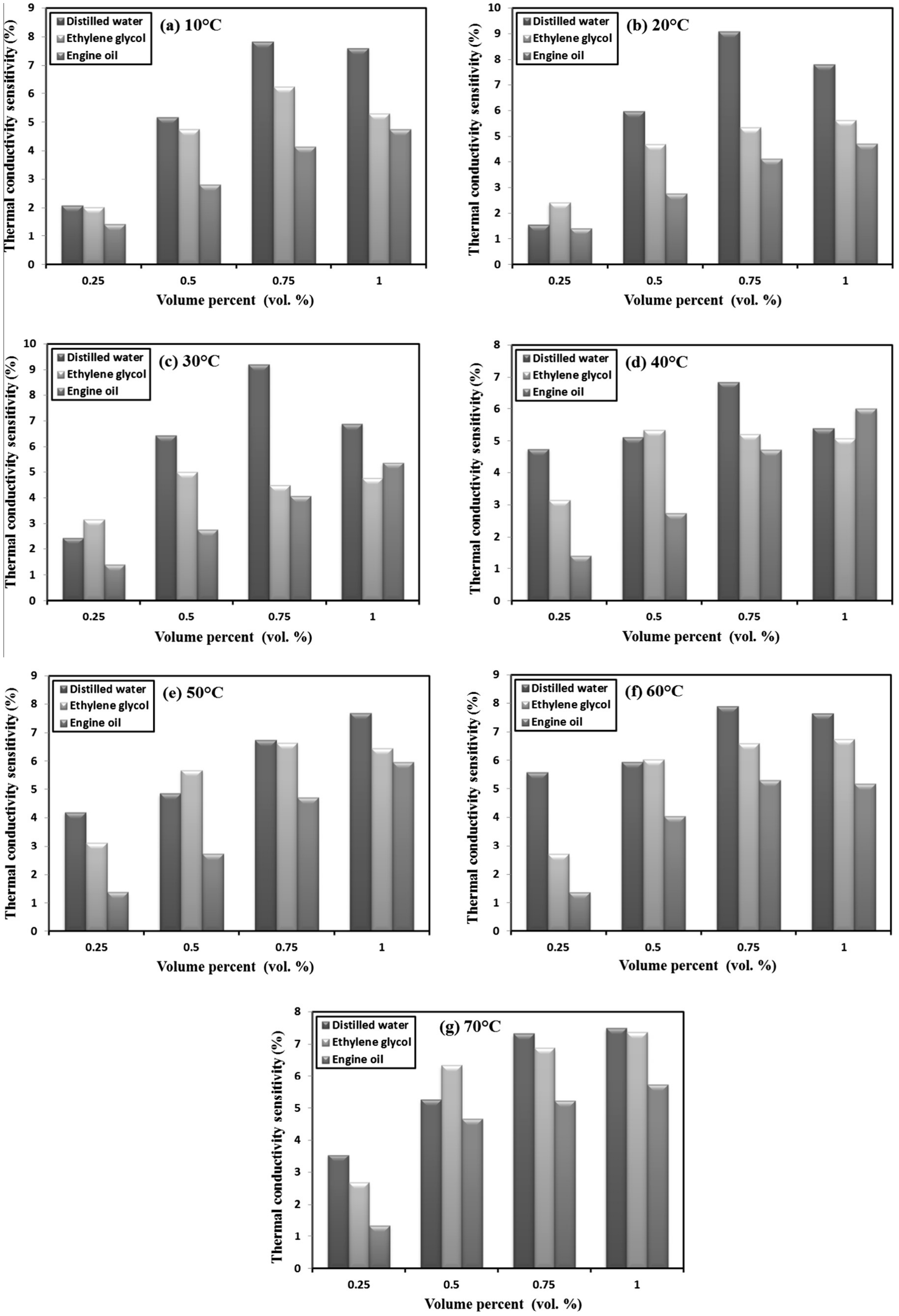

Fig. 12. Sensitivity analysis for thermal conductivity of nanofluids with increase in concentration of CuO NPs at different temperatures in different base fluids for different volume concentrations.

observed but from figure it can be stated that change in thermal conductivity is more sensitive for increase in volume percent at higher concentration.

A comparative account of change in sensitivity for different base fluids at a particular temperature is shown in Fig 12. Figure shows that at the same concentration and temperature, thermal conductivity of distilled water based nanofluids are more sensitive to change in concentration as compared to ethylene glycol and engine oil based nanofluids. Engine oil based nanofluids have shown minimum sensitivity for the change in volume concentration. Reason of relatively higher sensitivity for distilled water based CuO nanofluids may be that CuO nanoparticles form better interfacial layer with distilled water base fluid as compared to ethylene glycol and engine oil base fluid that plays important role in heat transfer capacity.

4. Conclusions

CuO nanoparticles synthesized using copper sulphate precursor were of smaller size and regular spherical shape as compared to copper acetate precursors where nanoparticles obtained were relatively larger and of irregular shape. Weighted particle size distribution using DLS shown size of 66 and 55 nm for CuO nanoparticles synthesized using copper acetate and copper sulphate precursor. Although the synthesized CuO nanoparticles were in nanometer range for both the precursors, still difference in their properties clearly establish effect of precursor salt on nucleation and growth mechanism. It can be stated that different precursor salts leads to differing growth process and orientation that in turn gives nanoparticles with different size and shape. In the present work aim was to enhance thermal conductivity so CuO nanoparticles synthesized from copper sulphate precursor were used due to their small size. It has been already established that smaller particles gives higher rate of thermal conductivity enhancement due to large surface area available for heat transfer.

Dispersion of CuO nanoparticles in different base fluids enhances thermal conductivity. Results show that rate of increase in thermal conductivity with increase in temperature or concentration of CuO nanoparticles for ethylene glycol base fluid was lower than distilled water based nanofluids. Thus in case of CuO nanoparticles, distilled water based nanofluids would be more efficient as compared to ethylene glycol based nanofluids for heat transfer applications with respect to rate of increment in thermal conductivity. Results also show poor performance of engine oil based nanofluids as compared to distilled water and ethylene glycol based nanofluids at low volume concentration indicating that for better performance of engine oil based nanofluids, higher volume percent concentration of CuO nanoparticles may be required and even it can work more efficiently at higher temperature. In summary, the rate of increase in thermal conductivity varies significantly with a particular base fluid at different temperatures or concentrations and different base fluids shows different rate of increase. The sensitivity analysis reveals that sensitivity increases with increasing concentration and different base fluids shows varying sensitivity.

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