Synthesis and Characterization of ZrO_2/dH_2O Nanofluids Using Two-Step Techniques

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Researchers have known for decades that nanofluids have superior thermal performance. However, the observation of thermal conductivity (THC) for nanofluids in water has not been thoroughly studied. This study used a two-step technique to prepare zirconium dioxide (ZrO_2) nanofluids. Utilizing scanning electron microscope (SEM) and X-ray diffraction (XRD), the characteristics of synthesized nanoparticles were evaluated. Then, by dispersing the nanoparticles in distilled water (dH_2O), ZrO_2 nanoparticles of the concentrations 0.5, 1.0, and 1.5 % by weight were prepared. A KD2 Pro thermal device measured the thermal conductivity and the temperature ranged from 30 to 50°C. From the experimental findings, the nanofluids' thermal conductivity increased according to the volume concentration of nanoparticles in the base fluid.

Keywords: Nanofluid; zirconium dioxide; SEM; thermal conductivity; XRD

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In recent decades, nanofluids have proved helpful in various applications. If these materials were employed, industrial equipment heat transfer efficiency might increase. Since its relatively recent inception, research on nanofluids has concentrated on enhancing thermal conductivity (THC) and heat transmission rather than viscosity [1–3]. MgO-DW nanofluid was created using ultrasonication treatment periods of 45 to 180 minutes at room temperature and volume concentrations of 0.05 to 0.25 vol. % [4]. Its thermal conductivity was increased by the addition of MgO nanoparticles to a conventional fluid, which was deionized water (DW). SEM (scanning electron microscope) images revealed the produced MgO nano powder's recognizable fluffy or feathery nanostructures. A hybrid solar collector can be used as a working fluid to investigate how different flow rates affect electrical and thermal efficiency. One or more nanoparticles are dissolved in a base fluid to create a nanofluid. Nanofluids are a popular topic for study in materials, physics and chemistry, and have many potential applications throughout the research fields, including chemical engineering, automobile, construction, and power [1, 3, 5, 10, 15]. So far, combinations of numerous metallic oxide nanoparticles, including Al_2O_3, CuO, Fe_3O_4, and TiO_2, have been employed [5–9]. According to a report [10], local bauxite was effectively used to create Al_2O_3 nanoparticles with 4.12 nm crystallite size and 296.7 m^2/gr in specific surface area. It was carefully constructed to test the viscosity of nanofluids. At a concentration of 0.095 vol. %, the critical heat flux enhancement of the nanofluids achieved an ideal value of 54%. Researchers found that magnetic ND (nano-diamond)-Fe_3O_4 nanoparticles have an excellent dispersion in distilled water and included 72% ND and 28% Fe_3O_4 [11]. In comparison to the base fluids, the THC of the nanofluids increased with the concentrations of 20:80, 40:60, and 60:40 EG (ethylene glycol)/Water at 17.8%, 13.43%, 13.6%, and 14.6%, at the temperature of 60°C, respectively. [12] reported that the viscosity of ethylene glycol nanofluids may be measured using the following device, which has been appropriately built: carbon nanotubes and Fe_3O_4 nanoparticles coated with GA (gum arabic) and TMAH (tetramethylammonium hydroxide), respectively. Ultra-sonic homogenization combines CNT (carbon nano tube) nanoparticles with an aqueous ferrofluid solution with a 13 nm average diameter. Experimental research has additionally looked at the THC of the solid volume fractions CuO/EG, water, and others at various temperatures [13]. The findings demonstrate that a nanofluid's conductivity rises as temperature and solid concentration rise. Higher solid concentrations have a more pronounced influence on the temperature than lower solid concentrations. [14] reported that ZrO_2 nanoparticles were successfully produced using the sol-gel technique and sucrose as a chelating agent from local zircon at 800°C calcination temperature. After 20 days, the nanofluid was relatively stable and could be used as a nuclear reactor coolant.

The literature has noted that water-based nano-fluids have demonstrated increased THC. Various researchers discovered that a higher surface area to volume ratio in nanofluids caused by smaller nano-particles leads to improved thermal conductivity. Even though ZrO_2 nanoparticles have good thermophysical features, including high heat conductivity and low density, very few nanofluids containing ZrO_2 have been described. Furthermore, ZrO_2/dH_2Obased nano-fluids can offer superior stability. In this work, ZrO_2/dH_2O nano-fluids were synthesized using a two-step technique, and SEM and XRD were employed to characterize the nanoparticles. ZrO_2 nanoparticles with different concentrations of 0.5, 1.0, and 1.5 % by weight were proposed to explore the THC of nanofluids. The prepared nanofluids' THC range of temperatures between 30°C and 50°C were tested.

EXPERIMENTAL

1. Synthesis of ZrO_2 Nanofluids

The technique of producing nanofluids is crucial for long-term stability. As a result, sustained particle dispersion in water can give uniform THC at various nanofluid concentrations. Researchers created stable nanofluids in two steps. Nanoparticle production and nanofluids are generated in a single step. Particles are created in dry form in the two-step technique, and nanofluid dispersions are made by stirring and ultra-sonication. Several methods have been used to fabricate nanoparticles of various sizes [5]. However, the convenience of preparing pre-set concentrations permits the two-step process being extensively adopted by researchers in the manufacture of numerous types of nanofluids. **Figure 1** depicts the preparation process for a ZrO_2 nanofluid.

ZrO_2/dH_2O nanofluids were created using zirconium dioxide nanopowder (Sigma Aldrich, Germany) with the particle size of 100 nm. Initially, 1000 mL of distilled water was placed in a beaker, followed by 0.5 wt. percent of ZrO_2 nanoparticles. The mixture was stirred for 6 hours with a magnetic stirrer. Then, ZrO_2 nanoparticles were needed for 1.0 and 1.5 vol. percent concentration [14]. The amount of nanopowder required for a percentage of volume concentration was calculated using Equation 1. The previously produced nanofluid solution was ultra-sonically agitated with a magnetic stirrer for 180 minutes [11]. The 0.5, 1, and 1.5 percent concentrations were made similarly. The Model-79-1 magnetic stirrer (Labpro, India) and the Model-MTS 750 ultra-sonicator equipment (Mangaldeep Tech Solutions, India) are shown in **Figures 2** and **3**, respectively.

$$\frac{\dfrac{W_{nanoparticle}}{\rho_{nanoparticle}}}{\dfrac{W_{nanoparticle}}{\rho_{nanoparticle}}+\dfrac{W_{basefluid}}{\rho_{basefluid}}} \quad \text{(Equation 1)}$$

Where, W = weight and ρ = density

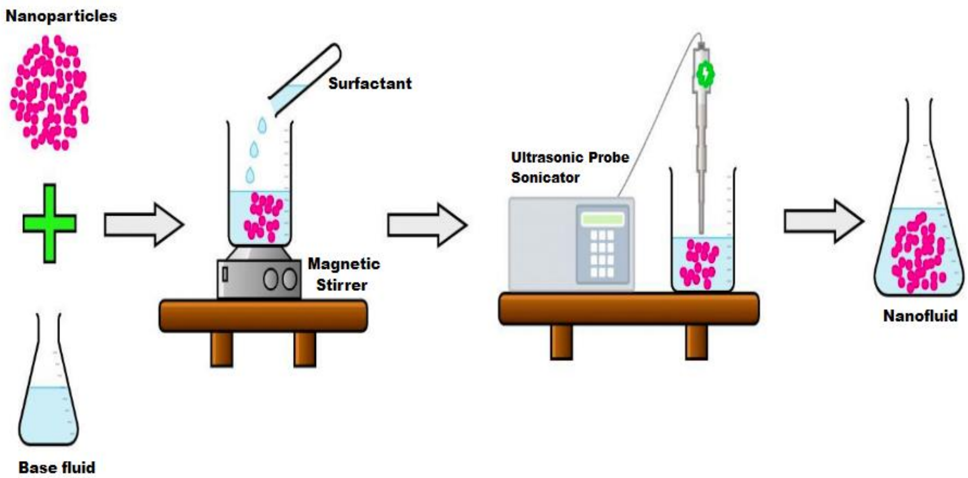

Figure 1. Preparation process of ZrO_2 nanofluid

Figure 2. Magnetic stirrer

Figure 3. Ultrasonicator apparatus

2. Characterization Methods

The Model-D8 Advance ECO XRD equipment (Bruker, United States) and the Model-EVO 18 SEM equipment (Zeiss, India) were used to evaluate the morphology of the nanoparticle sample. An X-ray Diffractometer device was used for the analysis of the diffraction patterns of the nanoparticles. According to the JCPDS file No. 32-1498, the reflections in the XRD results corresponded to the tetragonal phase for ZrO_2 nano-particles. The ZrO_2 average particle size was determined using the Debye Scherrer formula [15].

3. Testing Method

The KD2 Pro thermal device (Decagon Devices, USA) was used to test the THC of nanofluids. The Transient hot wire method (THWM) approach is utilized in this industrial device. A thin metal wire that functions as a heat source and temperature sensor was inserted into the test liquid. THWM detects electrical pulses fast and monitors wire temperature. The test sample's THC may be developed by changing the hot wire temperature for a predetermined time. The sensor was inserted vertically into a 45 mL sealed glass tube containing the sample. The temperature of the thermostatic bath in which the tube was submerged was controlled. Three measurements were made [16].

RESULTS AND DISCUSSION

1. X-ray Diffraction

The Debye-Scherrer equation can be used to calculate the size of particles smaller than 100nm, i.e., $(D) = (k\,\lambda / \beta \cos\theta)$, where D is the particle size, k is the Scherer's constant (k=0.94), λ is the X-ray wavelength (1.54178), β is the full width at half maximum (FWHM) of the diffraction peak, and θ is the angle of diffraction. As observed in the XRD pattern, the result was zirconium dioxide, and the small size of the crystallite widened the diffraction peaks [15]. **Figure 4** shows the ZrO_2 nano-particle diffraction pattern. The usual crystal size of ZrO_2 nanoparticles, according to the Debye-Scherrer formula, is 28 nm.

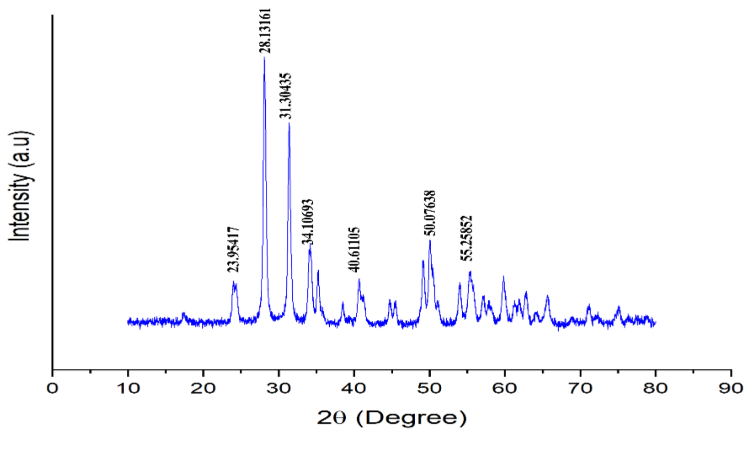

Figure 4. XRD pattern of pure ZrO_2 nanoparticles.

2. Scanning Electron Microscopy

The pure ZrO_2 nanoparticles' SEM pictures are shown in **Figure 5**. The scanning electron microscopy technique investigated the ZrO_2 nanoparticles' surface morphology. Based on the SEM pictures, the nano-particle aggregation increased the amount of ZrO_2 nanoparticles and had a consistent spherical shape, as shown in **Figure 6**. Elemental mapping using EDS (energy dispersive X-ray spectroscope) analysis indicated that the nano-powder sample was made entirely of zirconium and oxygen, the main ingredient of ZrO_2 nanoparticles [4]. The EDS spectrum of the ZrO_2 is shown in **Figure 7**. The spectrum identified the chemical components of the sample. For all elements, the EDS spectrum displayed an impurity peak; however, most of the sample comprised of ZrO_2, as shown in **Table 1**.

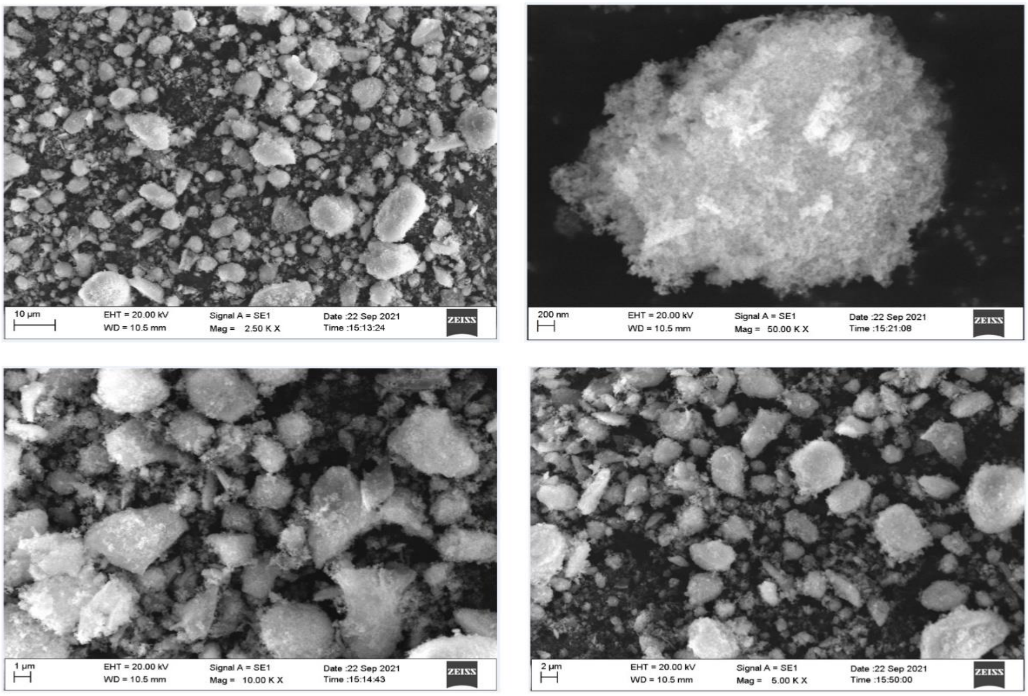

Figure 5. SEM photos of pure ZrO_2 nanoparticles at various magnifications

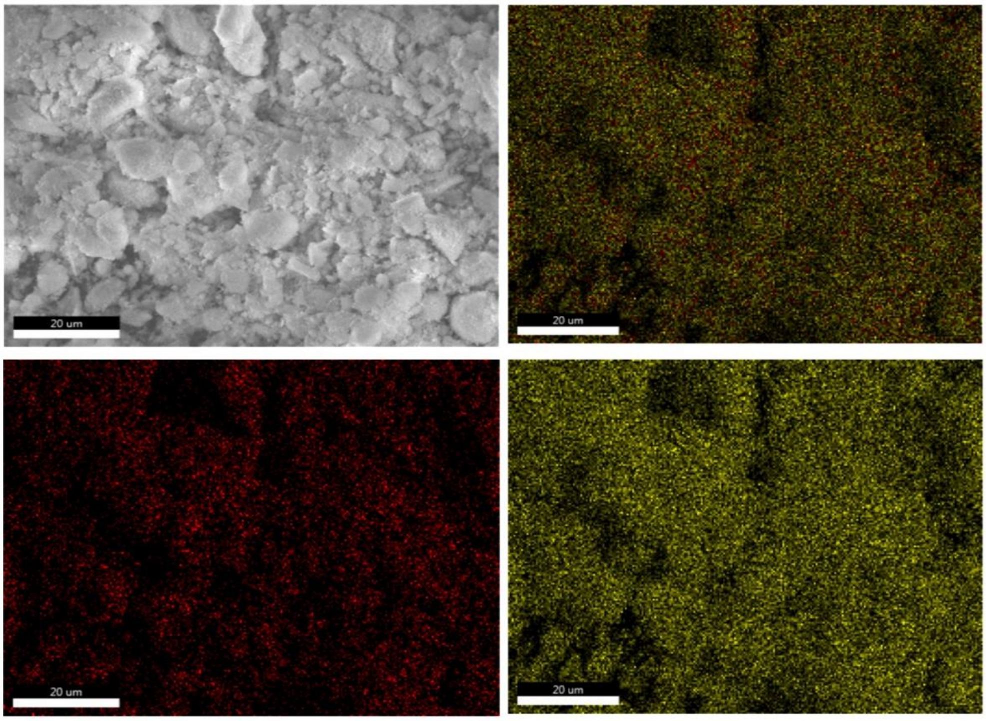

Figure 6. Elemental mapping of ZrO_2 nanoparticles

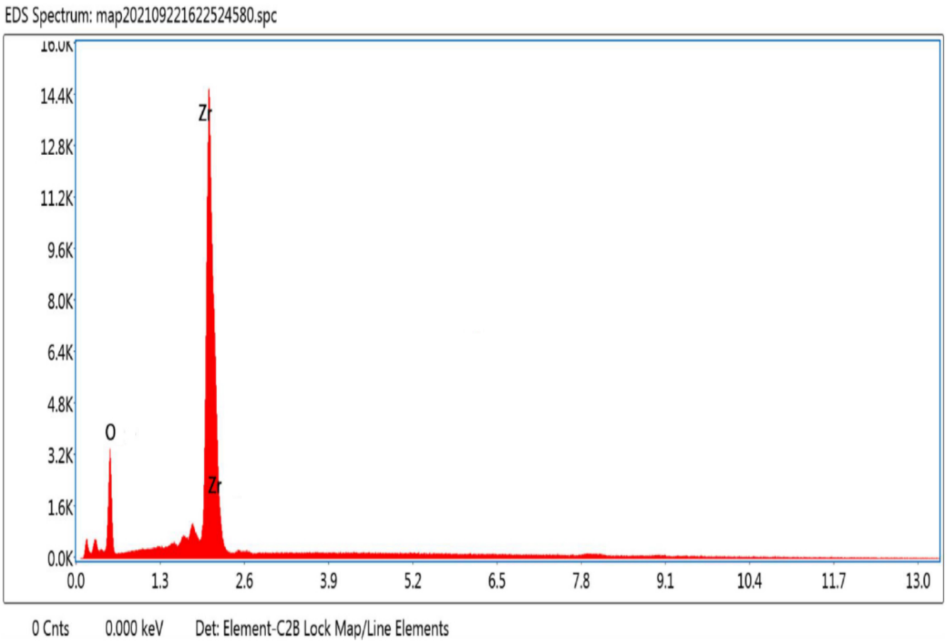

Figure 7. EDS result of ZrO_2 nanoparticles.

Table 1. Chemical composition of ZrO_2 nanoparticles.

Element	Weight %	Atomic %	Error %	K ratio
O	30.8	71.7	10.4	0.0552
Zr	69.2	28.3	1.4	0.6233

3. Analysis of THC

In this study, the THC of nanofluids made of ZrO_2/dH_2O was experimentally investigated. The Bruggeman equation [17] was employed to apply Equation 2 to find the sample nanofluids' actual THC.

Where,
K = Thermal conductivity
ϕ = Volume fraction

$$k_{effective} = \frac{k_{basefluid}}{4}\left[(3\phi-1)\frac{k_{nanoparticle}}{k_{basefluid}} + (2-3\phi) + \frac{k_{basefluid}}{4}\sqrt{\Delta}\right] \quad \text{(Equation 2)}$$

$$\Delta = \left[(3\phi-1)^2\left(\frac{k_{nanoparticle}}{k_{basefluid}}\right) + (2-3\phi)^2 + 2(2+9\phi^2)\left(\frac{k_{nanoparticle}}{k_{basefluid}}\right)\right]$$

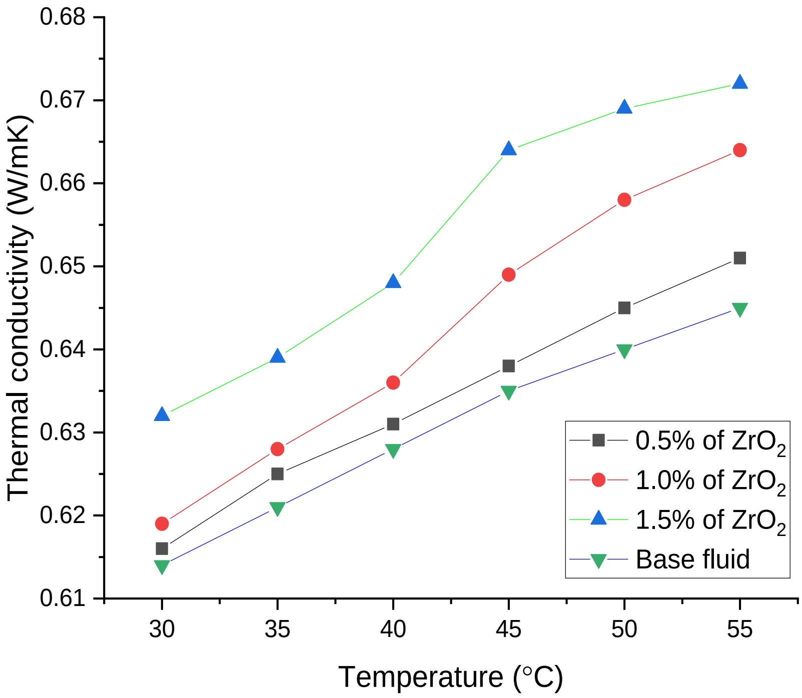

Figure 8. THC values vary with temperature

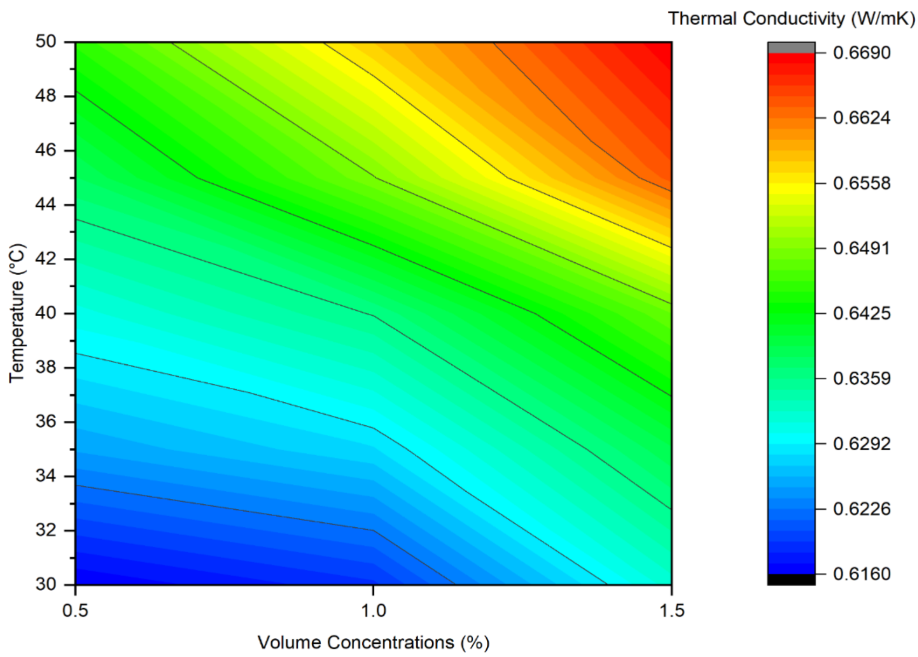

Figure 9. THC varies according to temperature and nanoparticle volume concentration.

The THC of ZrO_2/dH_2O nanofluids in relation to temperature is shown in **Figure 8** for various volume fractions. The improvement in THC was comparatively more remarkable at low volume fractions than at other fractions. Depending on the temperature, values indicated THC augmentation. The effect of temperature on THC was more noticeable at high volume fractions of solids than at low volume fractions. The experimental findings demonstrated a considerable increase in the generated nanofluids' effective THC vs. the base fluid. The contour plot in **Figure 9** shows how THC varies with temperature and nanoparticle volume concentration [5].

The percentage of Enhanced Thermal conductivity (ETHC) of ZrO_2 in the three-volume concentrations is shown in **Figure 10**, computed using Equation 3 to increase the THC of ZrO_2 nanofluids. It showed that the percentage enhancement increment increases by employing a more significant percentage of 1.5 percent ZrO_2 nanofluid; given that nanoparticles move in a Brownian motion in all directions pushing molecules in front of them along each temperature differential increases the THC of the ZrO_2 nanofluid [6].

$$ETHC\ (\%) = \left[\frac{K_{nanofluid} - K_{basefluid}}{K_{basefluid}}\right] \times 100 \qquad \text{(Equation 3)}$$

Where,
K = Thermal conductivity

CONCLUSION

In this study, ZrO_2 nanoparticles were characterized using XRD and SEM investigation, and nanofluids were prepared using a two-step technique. The nanopowder was dissolved in distilled water to prepare ZrO_2 nanofluids of the concentrations 0.5, 1.0, and 1.5 % by weight. The temperature ranged from 30 to 50°C, and a KD2 Pro device with a THWM was used to determine the THC. The experimental findings show that the produced nanofluids' effective THC significantly improved compared to the base fluid. The THC of nanofluids substantially intensifis with increasing concentration.

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REFERENCES

1. Pastoriza-Gallego, M. J., Lugo, L., Legido, J. L., Piñeiro, M. M. (2011) Thermal conductivity and viscosity measurements of ethylene glycol-based al2o3 nanofluids. *Nanoscale Res. Lett.*, **6**, 1–11.

2. Anandakumar, J., Palaniradja, K. (2021) Assessment on Performance advancement utilizing nanofluid as additional fluid in a Refrigeration System. *Int. J. Emerg. Trends Eng. Res.*, **9**, 1205–1210.

3. Babar, H., Ali, H. M. (2019) Towards hybrid nanofluids: Preparation, thermophysical properties, applications, and challenges. *J. Mol. Liq.*, **281**, 598–633.

4. Judran, H. K., Tuaamah Al-Hasnawi, A. G., Al Zubaidi, F. N., Al-Maliki, W. A. K., Alobaid, F., Epple, B. (2022) A High Thermal Conductivity of MgO-H2O Nanofluid Prepared by Two-Step Technique. *Appl. Sci.*, **12**, 1–18.

5. Ali, H. M., Babar, H., Shah, T. R., Sajid, M. U., Qasim, M. A., Javed, S. (2018) Preparation techniques of TiO2 nanofluids and challenges: A review. *Appl. Sci.*, **8**.

6. Ouikhalfan, M., Labihi, A., Belaqziz, M., Chehouani, H., Benhamou, B., Sarı, A., et al. (2020) stability and thermal conductivity enhancement of aqueous nanofluid based on surfactant-modified TiO2. *J. Dispers. Sci. Technol.*, **41**, 374–382.

7. Anandakumar, J. (2015) To Conduct the Performance Test on Chiller Unit By Using Nanofluid Cooled Condenser*Int. J. Mech. Eng. Robot. Res.* **4**, 279–285.

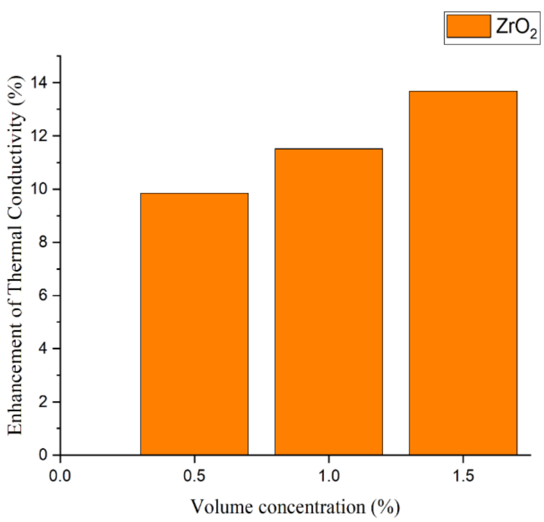

Figure 10. ETHC of ZrO_2 nanofluids

8. Agarwal, R., Verma, K., Agrawal, N. K., Duchaniya, R. K., Singh, R. (2016) Synthesis, characterization, thermal conductivity and sensitivity of CuO nanofluids. *Appl. Therm. Eng.*, **102**, 1024–1036.

9. Yang, S., Tan, Y., Yin, X., Chen, S., Chen, D., Wang, L., et al. (2017) Preparation and characterization of monodisperse solvent-free silica nanofluids. *J. Dispers. Sci. Technol.* **38**, 425–431.

10. Syarif, D. G. (2016) Characteristics of ethylene glycol-Al2O3 nanofluids prepared by utilizing Al2O3 nanoparticles synthesized from local bauxite. *J. Phys. Conf. Ser.*, **776**.

11. Sundar, L. S., Venkata Ramana, E., Graça, M. P. F., Singh, M. K., Sousa, A. C. M. (2016) Nano-diamond-Fe3O4 nanofluids: Preparation and measurement of viscosity, electrical and thermal conductivities. *Int. Commun. Heat Mass Transf.*, **73**, 62–74.

12. Shahsavar, A., Salimpour, M. R., Saghafian, M., Shafii, M. B. (2015) An experimental study on the effect of ultrasonication on thermal conductivity of ferrofluid loaded with carbon nanotubes. *Thermochim. Acta*, **617**, 102–110.

13. Hemmat Esfe, M., Saedodin, S., Akbari, M., Karimipour, A., Afrand, M., Wongwises, S., et al. (2015) Experimental investigation and development of new correlations for thermal conductivity of CuO/EG-water nanofluid. *Int. Commun. Heat Mass Transf.*, **65**, 47–51.

14. Dani Gustaman Syarif, Djoko Hadi Prajitno (2013) Characteristics of Water-ZrO2 Nanofluid Made from Solgel Synthesized ZrO2 Nano-particle Utilizing Local Zircon. *J. Mater. Sci. Eng. B.*, **3**, 124–129.

15. Manimaran, R., Palaniradja, K., Alagumurthi, N., Sendhilnathan, S., Hussain J. (2014) Preparation and characterization of copper oxide nanofluid for heat transfer applications. *Appl. Nanosci.*, **4**, 163–167.

16. Balaji, V., Arulprakasajothi, M., Logesh, K., Tharunpillai, B. (2018) Assessment of heat transfer behavior of water based alumina nanofluid. *Mater. Today Proc.*, **5**, 20641–20646.

17. Bruggeman (2009) Berechnung verschiedener physikalisher konstanten von heterogenen substanzen. *Ann. Der Phys.* **5**, 160–178.