Experimental Investigation of Thermal Conductivity of Water-Based Fe₃O₄ Nanofluid: An Effect of Ultrasonication Time

Divya P. Barai¹, Bharat A. Bhanvase¹,* and Gaweł Zyła²,*

¹ Department of Chemical Engineering, Laxminarayan Institute of Technology, Rashtrasant Tukadoji Maharaj Nagpur University, Nagpur 440033, MS, India; divyapbarai@yahoo.co.in
² Department of Physics and Medical Engineering, Rzeszów University of Technology, 35-959 Rzeszów, Poland
* Correspondence: bharatbhanvase@gmail.com (B.A.B.); gzyla@prz.edu.pl (G.Z.)

Abstract: Nanofluid preparation is a crucial step in view of their thermophysical properties as well as the intended application. This work investigates the influence of ultrasonication duration on the thermal conductivity of Fe₃O₄ nanofluid. In this work, water-based Fe₃O₄ nanofluids of various volume concentrations (0.01 and 0.025 vol.%) were prepared and the effect of ultrasonication time (10 to 55 min) on their thermal conductivity was investigated. Ultrasonication, up to a time duration of 40 min, was found to raise the thermal conductivity of Fe₃O₄ nanofluids, after which it starts to deteriorate. For a nanofluid with a concentration of 0.025 vol.%, the thermal conductivity increased to 0.782 W m⁻¹K⁻¹ from 0.717 W m⁻¹K⁻¹ as the ultrasonication time increased from 10 min to 40 min; however, it further deteriorated to 0.745 W m⁻¹K⁻¹ after a further 15 min increase (up to a total of 55 min) in ultrasonication duration. Thermal conductivity is a strong function of concentration of the nanofluid; however, the optimum ultrasonication time is the same for different nanofluid concentrations.

Keywords: Fe₃O₄ nanofluid; thermal conductivity; ultrasonication time

1. Introduction

The dispersion of nanoparticles in base fluids is known to alter the various physical, optical, and thermal properties that help make them applicable for different purposes in automobiles [1,2], solar thermal systems [3,4], refrigeration [5,6], electronics cooling [7], industrial heat transfer systems [8,9], environmental remediation [10], medicine [11,12], etc. The properties of base fluids are influenced by the type, size, shape, and composition of the nanomaterial contained in it [13,14]. Such dispersions, commonly known as nanofluids, have gained immense importance as heat transfer fluids. Several studies reveal their intensifying performance on heat transfer systems [15,16]. Nanofluids exhibit high thermal transport because of the high thermal conductivity of the nanoparticles [17]. Various mechanisms have been identified that play a role in imparting high thermal transport properties by the addition of nanoparticles in liquids [18]; however, these mechanisms are also impacted by various factors related to the nanomaterial type and its synthesis process, nanoparticle shape and size, concentration of nanoparticles and surfactants, method of nanofluid preparation, temperature, etc. Nanoparticles differ in materials and so do their thermal conductivities. Moreover, their morphology, size, shape, structure, etc., play an important role in determining their thermal properties. As nanotechnology offers a varying range of synthesis techniques, each technique has its effect on the properties and formation of nanomaterials [19]. Studies have widely demonstrated how concentration and temperature of nanofluids influence their various properties [20]. In addition, the use of surfactants is known to alter the nanofluid properties.
One of the important aspects, which also leaves an impact on the nanofluids properties, is the preparation method. Basically, a nanofluid can be prepared either by a one-step method or a two-step method. In the one-step method, the nanomaterial synthesis and nanofluids preparation are combined, i.e., the nanoparticle synthesis is directly conducted using the base fluid as the medium and is used for further applications. In the two-step method, the nanofluid is prepared after the nanomaterial is synthesized in a separate synthesis protocol, i.e., the dried nanomaterial particles are dispersed in the base fluid using a mixing or homogenizing technique [21]; however, this mixing of nanomaterial into the base fluid is not as straightforward as it seems. As known from the research on nanofluids, the more the nanoparticles are dispersed, the better suspension stability they acquire [22]. Nanoparticles are anticipated to be evenly and uniformly distributed in the nanofluid to ensure better performance. Clustering or settling of nanoparticles deteriorate properties of nanofluids, demeaning them from being known as colloids. At the same time, the thermophysical properties are directly influenced by the nanofluid stability [23]. Nanofluids, for the purpose of heat transfer, have gained importance because of their high thermal conductivity. Thermal conductivity is an important measure of the heat transfer ability of a nanofluid. Furthermore, settling, aggregation, and clustering of nanoparticles within the nanofluids are a threat to its thermal conductivity.

Ultrasonication is one of the widely used techniques for nanomaterial dispersion in a base fluid [24–26]. It involves application of high-frequency sound waves to bring about pressure changes in the fluid. These changes in pressure induce a cavitational effect. Cavitation is a phenomenon in which the pressure differences cause the generation of cavities, which grow in size and collapse after a certain time, releasing a great amount of energy in the form of heat [27]. The temperature and pressure conditions achieved due to cavitation can reach up to 10,000 K and 1000 atm at the micro-level [28]. Such conditions help the breaking of the nanoparticle clusters into tiny nanoparticles and thus dispersing them in the medium [29]; thus, this technique is very useful in nanofluid preparation; however, there are still uncertainties regarding the use of ultrasonication to bring desired changes in the nanofluid properties. Since the wide use of graphene-based nanomaterials for nanofluid application [30], Sandhya et al. [31] presented an extensive review of the effect of ultrasonication for the preparation of graphene nanofluids. Still, the behavior of different nanomaterials under the influence of ultrasonication is not eminent. Ultrasonication involves localized high temperature–pressure conditions, which may alter the physical, as well as chemical, structure of nanomaterials and ultimately influence the nanofluid properties [32]. All of these aspects remain as a gap in the study of nanofluids. Another major aspect is the duration for which ultrasonication must be carried out to achieve an improvement in the thermal properties. It is found that the thermal conductivity of nanofluid increases with an increase in ultrasonication time [33]. The high amplitude of sonication power leads to low aggregate size, high zeta potential, better particle dispersion, and lesser optimum duration [34] (optimum sonication duration for nanofluids is lesser at a high amplitude of sonication power than that achieved for sonication performed at a lower amplitude of sonication power). Mahbubul et al. [35] found that an optimum ultrasonication duration of 150 min is suitable for titania nanofluids. It was also claimed that the average cluster size and ultrasonication time have an interaction effect on zeta potential. In addition, pH is known to be affected by ultrasonication time, thereby affecting the nanofluid stability. Further, Asadi et al. [36] found that multi-walled carbon-nanotube-based nanofluids of different concentrations (0.1, 0.3, and 0.5 vol.%) exhibit maximum thermal conductivity (0.622, 0.638, and 0.66 W m⁻¹K⁻¹) and stability after a ultrasonication period of 60 min. Similarly, the nanofluid also exhibits minimum viscosity due to uniformly distributed nanoparticles [37]. Xian et al. [38] found that an ultrasonication period of 90 min produced highly stable hybrid nanofluid containing mixture of carboxyl-functionalized graphene nanoplatelets and TiO₂ nanoparticles with addition of surfactants. Zheng et al. [39] demonstrated that, for very low concentrations of liquid paraffin-based Fe₃O₄ nanofluid (0.005–0.03 vol.%), the optimum duration of sonication is 3 h. The sum-
mary of studies on the influence of the sonication process on the physical properties can be found in recent review paper presented by Asadi et al. [40].

In this work, the influence of ultrasonication time on thermal conductivity of Fe₃O₄ nanofluid is investigated. For this, water-based nanofluids having different concentrations of Fe₃O₄ nanoparticles were prepared using bath ultrasonication. Further, the thermal conductivity of these nanofluids was measured at ultrasonication duration from 10 to 55 min.

2. Materials and Methods

In this section, a description of the materials used in the study along with the methodology is presented.

2.1. Materials

Anhydrous FeCl₃ (96% purity) and FeSO₄·7H₂O (99% purity) purchased from Merck Specialities Pvt Ltd., Mumbai, India and Loba Chemie Pvt Ltd., Mumbai, India, respectively, were used as precursors to synthesize Fe₃O₄ nanoparticles. In addition, NaOH (98% purity) was used as a precipitating agent, which was procured from Loba Chemie Pvt Ltd., Mumbai, India. Distilled water was used for preparation of solutions, nanofluids, and washing purposes.

2.2. Synthesis of Fe₃O₄ Nanoparticles

Several different methods for the preparation of magnetic nanoparticles have appeared [41,42]; however, in this study, we have used the ultrasound-assisted co-precipitation method to synthesize Fe₃O₄ nanoparticles. For this, 50 mL solutions containing 0.278 g FeSO₄·7H₂O and 0.324 g FeCl₃ were prepared and sonicated for 5 min. Sonication was continued for another 25 min, wherein the precipitation of Fe₃O₄ nanoparticles was accomplished due to rise in pH to 11 by drop-by-drop addition of 1 M NaOH solution. The ultrasonicator used was a bath-type ultrasonicator (Dakshin Ultrasonics) having a fixed frequency of 30 kHz. The product was filtered, washed with distilled water, and dried in an oven for 1 h at 100 °C.

2.3. Characterization

The ultraviolet-visible (UV-vis) spectrum of the synthesized Fe₃O₄ nanoparticles was recorded on UV-vis spectrophotometer (LABINDIA Analytical UV3200 model). The transmission electron microscope (TEM) image of the sample was taken using JEOL JEM-1400 Flash Electron Microscope. Fourier transform infrared spectroscopy (FTIR) spectrum of the Fe₃O₄ nanoparticles was obtained using the Bruker Alpha II instrument. Finally, the X-ray diffraction (XRD) pattern of the Fe₃O₄ nanoparticles was obtained using Rigaku’s Miniflex 1800 diffractometer.

2.4. Synthesis of the Fe₃O₄ Nanofluids

Water-based nanofluids containing different concentrations of Fe₃O₄ nanoparticles (in two fractions of nanoparticles: 0.01, 0.025 vol.%) were prepared by dispersing the nanoparticles using ultrasonication in a bath ultrasonicator (Dakshin Ultrasonics) working at a fixed frequency and power of 30 kHz and 500 W, respectively. The bath had a capacity of 30 L. The nanofluids were initially ultrasonicated for 10 min in strictly controlled temperature.

2.5. Thermal Conductivity Measurement

The thermal conductivity of the Fe₃O₄ nanofluid in the examined concentrations was measured using the KD2 Pro thermal property analyzer (Decagon Devices Inc., Pullman, Washington, DC, USA). To determine the effect of ultrasonication time, the thermal conductivity of the nanofluid was measured at intervals of 5–10 min within the total ultrasonication time of 55 min conducted in the bath ultrasonicator. Temperature was maintained at 303.15 K for all the measurements.
Uncertainty Determination

The uncertainty of the results obtained with the above-described measuring device was determined by performing a series of measurements of the thermal conductivity of water and then determining the standard deviation of the results. Ten consecutive measurements of the thermal conductivity of water were performed at 303.15 K. The results of these measurements are summarized in Figure 1. The value of the thermal conductivity of water determined with this measurement was \( k = 0.6337 \text{ W m}^{-1}\text{K}^{-1} \) with the standard deviation \( u(k) = 0.0067 \text{ W m}^{-1}\text{K}^{-1} \), which is 1.1%. Taking into account this result, the expended relative uncertainty (with \( K = 2 \)) was determined to be 3% of the experimental value, which corresponds with the value presented in the literature [43–45]. Finally, the obtained result of the thermal conductivity of water \( k = (0.634 \pm 0.020) \text{ W m}^{-1}\text{K}^{-1} \) is in good agreement with the literature data presented by Shokouhi et al. [46] as \( k = (0.613 \pm 0.002) \text{ W m}^{-1}\text{K}^{-1} \).

![Figure 1](image1.png)

**Figure 1.** Results of the ten consecutive measurements of the thermal conductivity of water performed at 303.15 K. Points refers to experimental data; solid lines present average value of performed series of experiments; dotted lines show the standard deviation of the average and relative uncertainty of 3% of the average.

3. Results and Discussion

In this section, the characterization of developed nanofluids is presented. The results of the study are summarized along with the discussion of mechanisms that leads to observed behavior.

3.1. Formation of Fe₃O₄ Nanoparticles and Its Characterization

The formation of Fe₃O₄ nanoparticles was confirmed using various characterization techniques. Figure 2 shows the UV-visible spectra of synthesized Fe₃O₄ nanoparticles. A small peak at 370 nm and the absorption edge between 375 and 650 nm confirms the successful formation of Fe₃O₄ nanoparticles [47]. The origin of the decaying absorption tail after 200 nm up to 900 nm is attributed to the fact that the material does not absorb any radiation in the visible range of the electromagnetic spectrum.

Figure 3 shows the TEM image of synthesized Fe₃O₄ nanoparticles. The image shows that the Fe₃O₄ nanoparticles have a spherical shape and polydisperse nature. The particle size as observed from the TEM image ranges between 6 to 21 nm; however, the average particle size of the Fe₃O₄ nanoparticles is estimated to be around 11.7 nm.

Figure 4 depicts the FTIR spectra of synthesized Fe₂O₃ nanoparticles. It shows strong absorption between 550 cm⁻¹ to 680 cm⁻¹. The most intense peak at 564 cm⁻¹ represents the Fe-O bond within the Fe₂O₃ structure [48,49]. Further, the band at 1643 cm⁻¹ is attributed to the OH-bending and the one at 3429 cm⁻¹ is attributed to the OH-stretching. These bands occur due to the presence of hydroxyl groups.
The XRD pattern of the synthesized Fe₃O₄ nanoparticles is depicted in Figure 5. The peaks at 18.6°, 30.1°, 35.6°, 43°, 57.3°, 62.9°, and 74.3° represent the planes of Fe₃O₄ at (111), (220), (311), (400), (511), (440), and (533), respectively, confirming its cubic spinel phase [50]. The average crystallite size obtained from Debye–Scherrer equation based on the most intense XRD peak is estimated to be 25.97 nm. In addition, the lattice constant for the Fe₃O₄
spinel nanoparticles is found to be 8.34(3) Å, which is in accordance with that available in literature [51].

Figure 5. X-ray diffraction (XRD) pattern of synthesized Fe$_3$O$_4$ nanoparticles.

3.2. The Effect of Ultrasonication Time on Thermal Conductivity

Figure 6 shows the trend of thermal conductivity of water and different concentrations of Fe$_3$O$_4$ nanofluid with respect to the ultrasonication time. As usual, the thermal conductivity of water is almost constant with respect to time even after ultrasonicating for the previously mentioned duration.

The thermal conductivity of the nanofluid depends on the ultrasonication time. The term ‘agglomerate’ refers to the weak interaction of the nanoparticles, thereby forming clusters that can be broken by physical forces similar to those of ultrasonication; however, the aggregates composed of particles that are connected together (welded) with solid necks are difficult to break [52,53]. Initially, the nanoparticles added to the base fluid, here water, are in an agglomerated state. The thermal performance of the nanofluid is lesser at
higher agglomeration conditions [31]. For the Fe$_3$O$_4$ nanofluid concentrations, i.e., 0.01 and 0.025 vol.%, the thermal conductivity increases with ultrasonication time until 40 min of duration, after which it starts to deteriorate. At a lesser ultrasonication duration, there is insufficient cavitation, which is not able to break the nanoparticle clusters (Stage 1); this causes the fluid to remain unstable and thereby exhibit lesser thermal conductivity. As the ultrasonication duration increases, more and more nanoparticle agglomerates or clusters break, releasing tiny nanoparticles into the nanofluid (Stage 2) [54]. Firstly, the larger clusters are broken down into smaller clusters and then into individual nanoparticles [55]. Further increase in ultrasonication duration not only contributes to the breaking down of the clusters, but also to disperse the tiny nanoparticles uniformly throughout the nanofluid body (Stage 3) [55]. This process continues until the ultrasonication duration of 40 min ends. The evenly dispersed nanoparticles aid in imparting high thermal conductivity to the fluid due to the participation of the maximum number of nanoparticles in various heat transfer mechanisms (Stage 4); however, the decrease in thermal conductivity after 40 min is an indication of the reagglomeration of the nanoparticles into clusters, thereby again decreasing the nanofluid stability. This is caused by the excessive Brownian motion and enhanced contact between individual nanoparticles that lead to their interaction and thus increased clustering [56]. The clustering of nanoparticles gives rise to settling and thus the deterioration of the nanofluid stability. As stability is directly related to the nanofluid thermal conductivity, unstable nanofluids exhibit worse thermal conductivity [36]. All these stages are graphically represented in Figure 7. The linear increase and decrease in the thermal conductivity value during the sonication time can be modeled with simple functions as presented in Figure 6.

**Figure 7.** Various stages of nanofluid during ultrasonication.

Furthermore, thermal conductivity of 0.025 vol.% Fe$_3$O$_4$ nanofluid was recorded to be 0.717 W m$^{-1}$K$^{-1}$, which was more than that of 0.01 vol.% Fe$_3$O$_4$ nanofluid (0.672 W m$^{-1}$K$^{-1}$). This is consistent at all instances of ultrasonication time. This is due to the larger number of nanoparticles, as heat carriers, taking part in thermal transport [20]. At higher concentrations, a greater number of nanoparticles, having their characteristic Brownian motion, efficiently transfer heat energy due to the induced micro-movement.

4. Conclusions

This work demonstrates the effect of ultrasonication at various durations (10 to 55 min) on the thermal conductivity of the Fe$_3$O$_4$ nanofluid. It was found that the optimum duration of ultrasonication for water-based Fe$_3$O$_4$ nanofluid is 40 min, at which it shows maximum thermal conductivity. After 40 min of ultrasonication, the thermal conductivity starts to deteriorate. For a 0.025 vol.% of Fe$_3$O$_4$ nanoparticles in the nanofluid, the thermal conductivity increased from 0.717 W m$^{-1}$K$^{-1}$ to 0.782 W m$^{-1}$K$^{-1}$ with an increase in ultrasonication time from 10 min to 40 min. This is due to the breaking of the clusters...
of the nanoparticles due to ultrasonication; however, the thermal conductivity further deteriorated to 0.745 W m$^{-1}$K$^{-1}$ after an ultrasonication time of 40 min up to 55 min. This is due to the excessive Brownian-motion-induced contact and clustering, which further lead to settling and the decrease in the stability of the nanofluid.

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**Abbreviations**
The following abbreviations are used in this manuscript:

- UV-vis  Ultraviolet-visible
- TEM     Transmission electron microscope
- FTIR    Fourier transform infrared spectroscopy
- XRD     X-ray diffraction

**References**

1. Chougule, S.S.; Sahu, S.K. Comparative study of cooling performance of automobile radiator using Al$_2$O$_3$-water and carbon nanotube-water nanofluid. *J. Nanotechnol. Eng. Med.* 2014, 5, 010901. [CrossRef]

2. Chaurasia, P.; Kumar, A.; Yadav, A.; Rai, P.K.; Kumar, V.; Prasad, L. Heat transfer augmentation in automobile radiator using Al$_2$O$_3$–water based nanofluid. *SN Appl. Sci.* 2019, 1, 257. [CrossRef]

3. Chichghare, K.K.; Barai, D.P.; Bhanvase, B.A. Applications of nanofluids in solar thermal systems. In *Nanofluids and Their Engineering Applications*; CRC Press: Amsterdam, The Netherlands, 2019; pp. 275–314.

4. Tembhare, S.P.; Barai, D.P.; Bhanvase, B.A. Performance evaluation of nanofluids in solar thermal and solar photovoltaic systems: A comprehensive review. *Renew. Sustain. Energy Rev.* 2022, 153, 111738. [CrossRef]

5. Ajayi, O.O.; Ukasoanya, D.E.; Ogbonnaya, M.; Salawu, E.Y.; Okokpujie, I.P.; Akinlabi, S.A.; Akinlabi, E.T.; Owoeye, E.T. Investigation of the effect of R134a/Al$_2$O$_3$–nanofluid on the performance of a domestic vapour compression refrigeration system. *Procedia Manuf.* 2019, 35, 112–117. [CrossRef]

6. Sheikholeslami, M.; Rezaiehjouybari, B.; Darzi, M.; Shafee, A.; Li, Z.; Nguyen, T.K. Application of nano-refrigerant for boiling heat transfer enhancement employing an experimental study. *Int. J. Heat Mass Transf.* 2019, 141, 974–980. [CrossRef]

7. Tharayil, T.; Asirvatham, L.G.; Ravindran, V.; Wongwises, S. Thermal performance of miniature loop heat pipe with graphene–water nanofluid. *Int. J. Heat Mass Transf.* 2016, 93, 957–968. [CrossRef]

8. Xie, X.; Zhang, Y.; He, C.; Xu, T.; Zhang, B.; Chen, Q. Bench-scale experimental study on the heat transfer intensification of a closed wet cooling tower using aluminum oxide nanofluids. *Ind. Eng. Chem. Res.* 2017, 56, 6022–6034. [CrossRef]

9. Imani-Mofrad, P.; Saeed, Z.H.; Shanbedi, M. Experimental investigation of filled bed effect on the thermal performance of a wet cooling tower by using ZnO/water nanofluid. *Energy Convers. Manag.* 2016, 127, 199–207. [CrossRef]

10. Zhao, D.; Cai, L.; Sun, Z.; Zhang, A.; Héraux, P.; Kim, H.; Ye, W.; Liu, Y. Efficient degradation of tetracycline by RGO@ black titanium dioxide nanofluid via enhanced catalysis and photothermal conversion. *Sci. Total Environ.* 2021, 787, 147536. [CrossRef]

11. Tripathi, D.; Bég, O.A. A study on peristaltic flow of nanofluids: Application in drug delivery systems. *Int. J. Heat Mass Transf.* 2014, 70, 61–70. [CrossRef]

12. Chahregh, H.S.; Dinarvand, S. TiO$_2$-Ag/blood hybrid nanofluid flow through an artery with applications of drug delivery and blood circulation in the respiratory system. *Int. J. Numer. Methods Heat Fluid Flow* 2020, 30, 4775–4796. [CrossRef]

13. Bhanvase, B.A.; Barai, D.P.; Sonawane, S.H.; Kumar, N.; Sonawane, S.S. Intensified heat transfer rate with the use of nanofluids. In *Handbook of Nanomaterials for Industrial Applications*; Elsevier: Amsterdam, The Netherlands, 2018; pp. 739–790.
14. Qiu, L.; Zhu, N.; Feng, Y.; Michaelides, E.E.; Zyla, G.; Jing, D.; Zhang, X.; Norris, P.M.; Markides, C.N.; Mahian, O. A review of recent advances in thermophysical properties at the nanoscale: From solid state to colloids. *Phys. Rep.* **2020**, *843*, 1–81. [CrossRef]

15. Radkar, R.; Bhanvase, B.; Barai, D.; Sonawane, S. Intensiﬁed convective heat transfer using ZnO nanofluids in heat exchanger with helical coiled geometry at constant wall temperature. *Mater. Sci. Energy Technol.* **2019**, *2*, 161–170. [CrossRef]

16. Lanjewar, A.; Bhanvase, B.; Barai, D.; Chawhan, S.; Sonawane, S. Intensiﬁed Thermal Conductivity and Convective Heat Transfer of Ultrasonically Prepared CuO–Polyaniline Nanocomposite Based Nanofluids in Helical Coil Heat Exchanger. *Period. Polytech. Chem. Eng.* **2020**, *64*, 271–282. [CrossRef]

17. Barai, D.P.; Chichghare, K.K.; Chawhan, S.S.; Bhanvase, B.A. Synthesis and Characterization of Nanofluids: Thermal Conductivity, Electrical Conductivity and Particle Size Distribution. In *Nanotechnology for Energy and Environmental Engineering*; Springer: Berlin/Heidelberg, Germany, 2020; pp. 1–49.

18. Bhanvase, B.A.; Barai, D. *Nanofluids for Heat and Mass Transfer: Fundamentals, Sustainable Manufacturing and Applications*; Thermophysical Properties of Nanofluids; Academic Press: Cambridge, MA, USA, 2021; pp. 101–106.

19. Ganachari, S.V.; Banapurmath, N.R.; Salimath, B.; Yaradoddi, J.S.; Shettar, A.S.; Hunashyal, A.M.; Venkataraman, A.; Patil, P.; Shoba, H.; Hiremath, G.B. Synthesis techniques for preparation of nanomaterials. *Handb. Ecomater.* **2017**, *2083*, 2103.

20. Sarode, H.; Barai, D.; Bhanvase, B.; Ugwekar, R.; Saharan, V. Investigation on preparation of graphene oxide-CuO nanocomposite based nanofluids with the aid of ultrasound assisted method for intensiﬁed heat transfer properties. *Mater. Chem. Phys.* **2020**, *251*, 123102. [CrossRef]

21. Bhanvase, B.A.; Barai, D. *Nanofluids for Heat and Mass Transfer: Fundamentals, Sustainable Manufacturing and Applications*; Lab-Scale Synthesis and Scale up Challenges; Academic Press: Cambridge, MA, USA, 2021; pp. 43–68.

22. Bhanvase, B.A.; Barai, D. *Nanofluids for Heat and Mass Transfer: Fundamentals, Sustainable Manufacturing and Applications*; Stability of Nanofluids; Academic Press: Cambridge, MA, USA, 2021; pp. 69–97.

23. Martinez, V.A.; Vasco, D.A.; Garcia-Herrera, C.M. Transient measurement of the thermal conductivity as a tool for the evaluation of the stability of nanofluids subjected to a pressure temperature. *Int. Commun. Heat Mass Transf.* **2018**, *91*, 234–238. [CrossRef]

24. Chawhan, S.S.; Barai, D.P.; Bhanvase, B.A. Sonochemical preparation of rGO-SnO nanocomposite and its nanofluids: Characterization, thermal conductivity, rheological and convective heat transfer investigation. *Mater. Today Commun.* **2020**, *23*, 101148. [CrossRef]

25. Chawhan, S.S.; Barai, D.P.; Bhanvase, B.A. Investigation on thermophysical properties, convective heat transfer and performance evaluation of ultrasonically synthesized Ag-doped TiO2 hybrid nanoparticles based highly stable nanofluid in a minichannel. *Therm. Sci. Eng. Prog.* **2021**, *25*, 100928. [CrossRef]

26. Singh, K.; Barai, D.P.; Chawhan, S.S.; Bhanvase, B.A.; Saharan, V.K. Synthesis, characterization and heat transfer study of reduced graphene oxide-Al2O3 nanocomposite based nanofluids: Investigation on thermal conductivity and rheology. *Mater. Today Commun.* **2021**, *26*, 101986. [CrossRef]

27. Barai, D.P.; Bhanvase, B.A.; Saharan, V.K. Reduced graphene oxide-Fe3O4 nanocomposite based nanofluids: study on ultrasonic assisted synthesis, thermal conductivity, rheology, and convective heat transfer. *Ind. Eng. Chem. Res.* **2019**, *58*, 8349–8369. [CrossRef]

28. Kale, A.R.; Barai, D.P.; Bhanvase, B.A.; Sonawane, S.H. An Ultrasound-Assisted Minireactor System for Continuous Production of TiO2 Nanoparticles in a Water-in-Oil Emulsion. *Ind. Eng. Chem. Res.* **2021**, *60*, 14747–14757. [CrossRef]

29. Mandhare, H.; Barai, D.P.; A. Bhanvase, B.; Saharan, V.K. Preparation and thermal conductivity investigation of reduced graphene oxide-ZnO nanocomposite-based nanofluids: study on ultrasonic-assisted method. *Mater. Res. Innov.* **2020**, *24*, 433–441. [CrossRef]

30. Barai, D.P.; Bhanvase, B.A.; Sonawane, S.H. A review on graphene derivatives-based nanofluids: investigation on properties and heat transfer characteristics. *Ind. Eng. Chem. Res.* **2020**, *59*, 10231–10277. [CrossRef]

31. Sandhya, M.; Ramasamy, D.; Sudhakar, K.; Kadingama, K.; Harun, W. Ultrasonication an intensifying tool for preparation of stable nanofluids and study the time inﬂuence on distinct properties of graphene nanofluids–A systematic overview. *Ultrason. Sonochem.* **2021**, *73*, 105479. [CrossRef] [PubMed]

32. Arrigo, R.; Teresi, R.; Gamberotti, C.; Parisi, F.; Lazzara, G.; Dintcheva, N.T. Sonication-induced modification of carbon nanotubes: Effect on the rheological and thermo-oxidative behaviour of polymer-based nanocomposites. *Materials* **2018**, *11*, 383. [CrossRef] [PubMed]

33. Sonawane, S.S.; Juwar, V. Optimization of conditions for an enhancement of thermal conductivity and minimization of viscosity of ethylene glycol based Fe3O4 nanofluid. *Appl. Therm. Eng.* **2016**, *109*, 121–129. [CrossRef]

34. Mahbubul, I.; Saidur, R.; Amalina, M.; Eclioglu, E.; Okutucu-Ozyurt, T. Effective ultrasonication process for better colloidal dispersion of nanofluid. *Ultrason. Sonochem.* **2015**, *26*, 361–369. [CrossRef]

35. Mahbubul, I.; Eclioglu, E.B.; Saidur, R.; Amalina, M. Optimization of ultrasonication period for better dispersion and stability of TiO2–water nanofluid. *Ultrason. Sonochem.* **2017**, *37*, 360–367. [CrossRef]

36. Asadi, A.; Alarifi, I.M.; Ali, V.; Nguyen, H.M. An experimental investigation on the effects of ultrasonication time on stability and thermal conductivity of MWCNT-water nanofluid: Finding the optimum ultrasonication time. *Ultrason. Sonochem.* **2019**, *58*, 104639. [CrossRef] [PubMed]

37. Asadi, A.; Alarifi, I.M. Effects of ultrasonication time on stability, dynamic viscosity, and pumping power management of MWCNT-water nanofluid: An experimental study. *Sci. Rep.* **2020**, *10*, 15182. [CrossRef] [PubMed]
10. Xian, H.W.; Sidik, N.A.C.; Saidur, R. Impact of different surfactants and ultrasonication time on the stability and thermophysical properties of hybrid nanofluids. *Int. Commun. Heat Mass Transf.* 2020, 110, 104389. [CrossRef]

11. Zheng, Y.; Shahsavari, A.; Afrand, M. Sonication time efficacy on Fe$_3$O$_4$-liquid paraffin magnetic nanofluid thermal conductivity: An experimental evaluation. *Ultrason. Sonochem.* 2020, 64, 105004. [CrossRef] [PubMed]

12. Asadi, A.; Pourfattah, F.; Szilágyi, I.M.; Afrand, M.; Zyla, G.; Ahn, H.S.; Wongwises, S.; Nguyen, H.M.; Arabkoohsar, A.; Mahian, O. Effect of sonication characteristics on stability, thermophysical properties, and heat transfer of nanofluids: A comprehensive review. *Ultrason. Sonochem.* 2019, 58, 104701. [CrossRef]

13. Serga, V.; Burve, R.; Maiorov, M.; Krumina, A.; Skaudžius, R.; Zarkov, A.; Kareiva, A.; Popov, A.I. Impact of gadolinium on the structure and magnetic properties of nanocrystalline powders of iron oxides produced by the extraction-pyrolytic method. *Materials* 2020, 13, 4147. [CrossRef]

14. Li, Y.; Wang, Z.; Liu, R. Superparamagnetic α-Fe$_2$O$_3$/Fe$_3$O$_4$ heterogeneous nanoparticles with enhanced biocompatibility. *Nanomaterials* 2021, 11, 834. [CrossRef]

15. Zyla, G. Thermophysical properties of ethylene glycol based yttrium aluminum garnet (Y$_3$Al$_5$O$_{12}$–EG) nanofluids. *Int. J. Heat Mass Transf.* 2016, 92, 751–756. [CrossRef]

16. Pastoriza-Gallego, M.J.; Lugo, L.; Legido, J.L.; Piñeiro, M.M. Thermal conductivity and viscosity measurements of ethylene glycol-based Al$_2$O$_3$ nanofluids. *Nanoscale Res. Lett.* 2011, 6, 221. [CrossRef]

17. Pastoriza-Gallego, M.; Lugo, L.; Cabaleiro, D.; Legido, J.; Piñeiro, M. Thermophysical profile of ethylene glycol-based ZnO nanofluids. *J. Chem. Thermodyn.* 2014, 73, 23–30. [CrossRef]

18. Shokouhi, M.; Jalili, A.H.; Mohammadian, A.H.; Hosseini-Jenab, M.; Nouri, S.S. Heat capacity, thermal conductivity and thermal diffusivity of aqueous sulfolane solutions. *Thermochim. Acta* 2013, 560, 63–70. [CrossRef]

19. Chaki, S.; Malek, T.J.; Chaudhary, M.; Tailor, J.; Deshpande, M. Magnetite Fe$_3$O$_4$ nanoparticles synthesis by wet chemical reduction and their characterization. *Adv. Nat. Sci. Nanosci. Nanotechnol.* 2015, 6, 035009. [CrossRef]

20. Srivastava, S.; Awasthi, R.; Gajbihiye, N.S.; Singh, A.; Yadav, A.; Gupta, R.K. Innovative synthesis of citrate-coated superparamagnetic Fe$_3$O$_4$ nanoparticles and its preliminary applications. *J. Colloid Interface Sci.* 2011, 359, 104–111. [CrossRef] [PubMed]

21. Ozkaya, T.; Toprak, M.S.; Baykal, A.; Kavas, H.; Köseoglu, Y.; Aktaş, B. Synthesis of Fe$_3$O$_4$ nanoparticles at 100°C and its magnetic characterization. *J. Alloy Compd.* 2009, 472, 18–23. [CrossRef]

22. Zhang, X.; Feng, Z.B.; Liu, L.; Zhang, X.M.; Wang, Z.R.; Lu, P.; Sun, Q. One-pot synthesis of Ag-decorated pomegranate seed-like Fe$_3$O$_4$ composite for high-performance lithium-ion battery. *Ionics* 2019, 25, 4099–4107. [CrossRef]

23. Aksimentyeva, O.; Savchyn, V.; Dyakonov, V.; Piechota, S.; Horbenko, Y.Y.; Opainych, I.Y.; Demchenko, P.Y.; Popov, A.; Szymczak, H. Modification of polymer-magnetic nanoparticles by luminescent and conducting substances. *Mol. Cryst. Liq. Cryst.* 2014, 590, 35–42. [CrossRef]

24. Nichols, G.; Byard, S.; Bloxham, M.J.; Botterill, J.; Dawson, N.J.; Dennis, A.; Diart, V.; North, N.C.; Sherwood, J.D. A review of the terms agglomerate and aggregate with a recommendation for nomenclature used in powder and particle characterization. *J. Pharm. Sci.* 2002, 91, 2103–2109. [CrossRef]

25. Teleki, A.; Wengeler, R.; Wengeler, L.; Nirschl, H.; Pratsinis, S. Distinguishing between aggregates and agglomerates of flame-made TiO$_2$ by high-pressure dispersion. *Powder Technol.* 2008, 181, 292–300. [CrossRef]

26. Ghadimi, A.; Metsealaar, I.H. The influence of surfactant and ultrasonic processing on improvement of stability, thermal conductivity and viscosity of titania nanofluid. *Exp. Therm. Fluid Sci.* 2013, 51, 1–9. [CrossRef]

27. Sadri, R.; Ahmadi, G.; Togun, H.; Dahari, M.; Kazi, S.N.; Sadeghinezhad, E.; Zubir, N. An experimental study on thermal conductivity and viscosity of nanofluids containing carbon nanotubes. *Nanoscale Res. Lett.* 2014, 9, 151. [CrossRef]

28. Sonawane, S.S.; Khedkar, R.S.; Walsewar, K.L. Effect of sonication time on enhancement of effective thermal conductivity of nano TiO$_2$–water, ethylene glycol, and paraffin oil nanofluids and models comparisons. *J. Exp. Nanosci.* 2015, 10, 310–322. [CrossRef]