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Synthesis and Characterisation of Oleic Acid Coated Fe_3O_4 Nanoparticles in Poly Alpha Olefin Oil based Nanofluid for Heat Transfer Applications

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ABSTRACT

Nanofluids are heat transfer liquids with dispersed nanoparticles. Ultrafine nano sized particles, having diameter <100 nm, are suspended in a conventional base fluid to constitute a nanofluid. The present study highlights the synthesis of Fe3O4 nanoparticles (IONPs) and their application in heat transfer phenomenon. For synthesis, a revised version of co-precipitation technique was used to produce pure IONPs and Oleic acid coating was done by physical immobilization method to prevent oxidation of iron and to make the nanoparticles hydrophobic in nature. Poly alpha olefin based nanofluid was prepared in five different volume fractions of 0.1wt %, 0.2wt %, 0.3wt.%, 0.4wt.% and 0.5wt.%. The XRD of the coated and uncoated IONPs was done for phase identification and crystalline structure. The compositional analysis of the coated and uncoated IONPs and confirmation of polymer coating was done by using Fourier-Transform Infrared Spectroscopy (FTIR). The thermal conductivity of the synthesized nanofluid was measured by Thermal Constants analyser technique. The thermal conductivity of the synthesized nanofluid was found to be increased than the pure Poly Alpha Olefin Oil.

Keywords: Nanofluid, Poly Alpha Olefin oil, Thermal conductivity.

1. INTRODUCTION

With the miniaturization of modern technology, the scarcity of effective cooling system has become a preventive factor for further advancement. However, the scarcity of an effective cooling system has become a preventive factor for further advancement. The problem of low thermal conductivity mainly effects the systems using liquid coolants as heat transport fluids (HTFs) [1]. Heat transport fluids are extensively used in different industries such as, transport industry, vehicle and avionics cooling systems, power station, cooling systems in buildings, textile,

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electronics, chemicals industries [2, 3]. Fluids used for heat transport applications are water, engine oil and ethylene glycol. Thermal conductivity of the above-mentioned fluids is lower than the solid phase. Therefore, diverse approaches like surface modification [4] and the use of additives [5] were used by researchers to enhance thermal efficiency of traditional fluids. However, no significant rise in thermal conductivity was observed [6].

Maxwell gave theoretical model of suspending millimeter or micrometer sized solid particles in conventional coolants to boost their active thermal conductivity. However, because of their bulky size and greater density, these particles caused erosion, sedimentation, resistance and required excessive pumping power when used for heat transfer in micro-cooling devices. Therefore, it was not considered as an effective method for heat transfer [7-9]. Choi [10] replaced micro/milli-meter sized particles with nanoparticles due to their unique thermophysical properties and engineered a new class of heat transfer fluids containing nanometer (10^{-9} m) sized particles. These fluids were referred as nanofluids [2]. Nanofluids are heat transport liquids with disseminated nanoparticles. Ultrafine particles, having diameter <100 nm, are suspended in a conventional base fluid to constitute a nanofluid.

The use of nanofluids as HTFs, still has limitations such as low stability [11], particle agglomeration [12], effective viscosity [13], volume fraction [14], nanoparticle size and shape [15], preparation method [16], base fluid type [17], cluster morphology and distribution [1], operating temperature [18], cost [3], effective density [13] and the pH [19, 20]. Therefore, to break through the aforementioned limitations, the field of nanofluids has gained much attention. Choi et al. synthesized carbon nanotube based PAO oil colloids and measured their effective thermal conductivity [21]. Nelson et al. performed experiment on exfoliated graphite nanoparticle fibres suspended in poly alpha olefin at mass concentrations of 0.6 and 0.3% [22]. Narvaez et al. discussed the behaviour of PAO based nanofluids using alumina and CNT as nanoparticles and studied different factors such as clustering, dispersion and stability [1]. Philip et al. dispersed Oleic acid coated Fe_3O_4 nanoparticles in kerosene and showed 300 % enhancement in thermal conductivity. They also proposed that the magnetically polarizable nanofluid can be used as efficient heat transfer fluids [23]. Flow of ferro-fluid can be manipulated by applied magnetic field, because of their superparamagnetic behaviour. Fe_3O_4 nanoparticles have high electrical and thermal conductivity and are cost effective. Hence, iron oxide nanoparticles are judged to be the best choice [24-29].

The purpose of this study is to synthesize magnetic nano sized particles and scatter them in Poly Alpha Olefin Oil by controlling size of nanoparticles, added volume fraction, minimizing agglomeration and increased stability of the nano sized particles in the base fluid to enhance thermal conductivity for heat transport applications.

2. MATERIAL AND METHODS

2.1 Chemicals

All the chemicals of Ferric Chloride hexahydrate ($FeCl_3 \cdot 6H_2O$), Ferrous Chloride tetrahydrates ($FeCl_2 \cdot 4H_2O$), hydrochloric acid (HCl), ammonium hydroxide (NH_4OH), Deionized water, oleic acid, o-xylene and poly alpha olefin oil (PAO) were purchased. All these reagents were used without further purification.

2.2 Synthesis of Iron Oxide Nanoparticles

Initially, ferrous Chloride (0.8M) and ferric Chloride (0.5M) were mixed with 40ml of deionized water. This solution has the molar ratio of Fe^{2+}: Fe^{3+} as 1:1.6 and was stirred 800rpm at 40°C for 15 minutes to control the particle size. Then, 20ml of ammonium hydroxide (32% ammonia) was added drop by drop into the prepared solution with constant stirring at 1050 rpm and temperature and to maintain the pH of the solution at 11.5. The black precipitates were observed which indicates the formation of iron oxide nanoparticles. Afterwards, the precipitates were washed three to four times with deionized water and centrifuged at 4000rpm for 5 minutes to completely remove the residuals. The obtained precipitates of nanoparticles were dried at 40°C temperature overnight in the oven.

2.3. Oleic Acid Coating

To prevent oxidation of iron and to make the nanoparticles hydrophobic in nature, the synthesized iron oxide nanoparticles were coated with oleic acid by physical immobilization method. In this technique, the polymer is initially mixed in a solvent and then the particles are added to this solution to obtain a uniform layer.

5% (w/v) of obtained Fe_3O_4 nanoparticles were added in 10% (v/v) of oleic acid solution and was stirred at 1050rpm, at 40°C constantly for 1 hour. O-xylene was used as solvent for oleic acid solution. Then the solution was centrifuged at 4000rpm for 10 minutes. Finally, the oleic acid coated iron oxide nanoparticles were separated and washed with ethanol three to four times. The residual liquid was disposed of via centrifugation at 4000rpm for 10 minutes and dried in vacuum oven (500mbar) at 50°C for 8 hours. Fig. 1 shows the synthesis mechanism of the nanofluid used in this work.

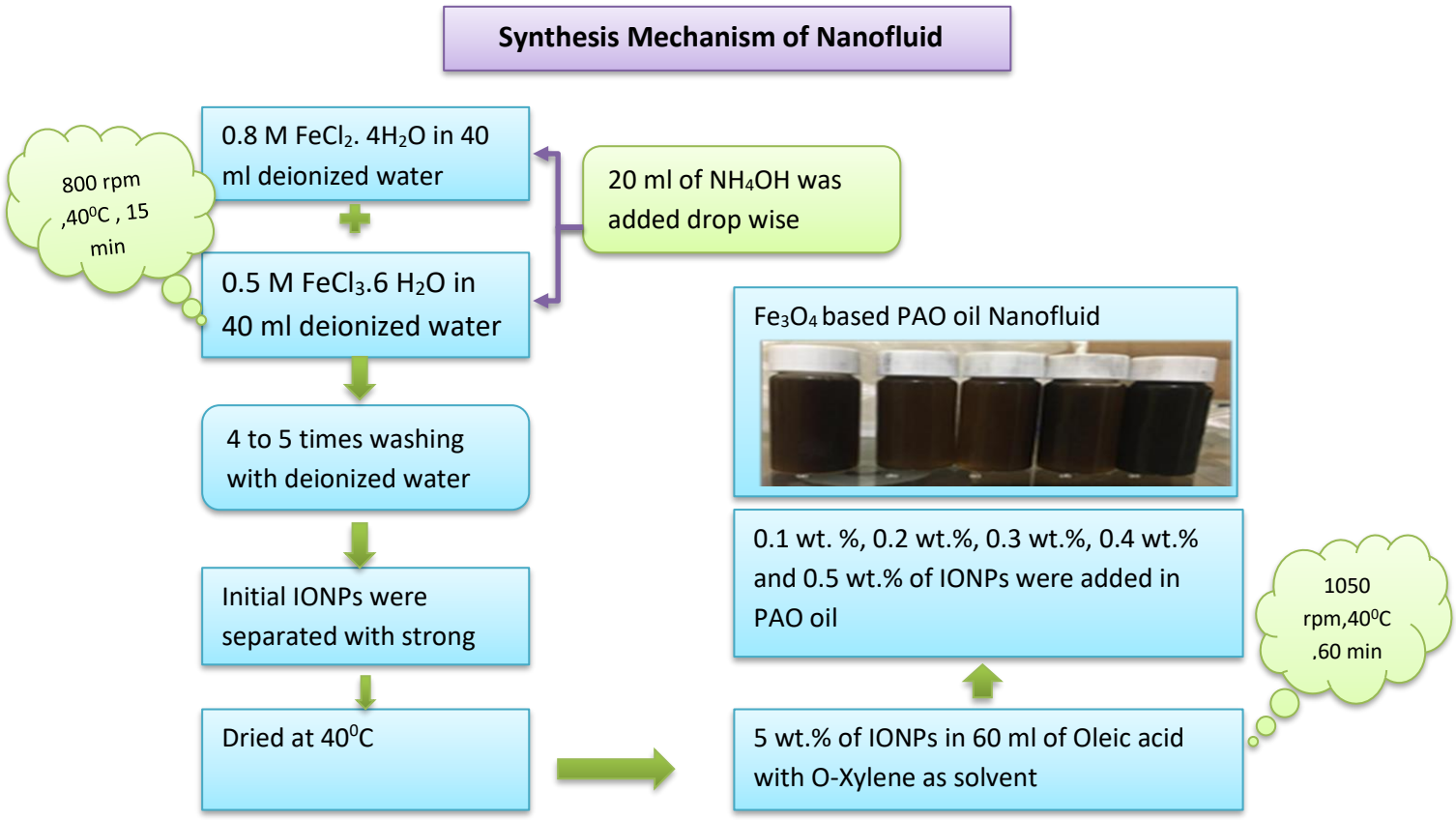

Figure 1. Synthesis Mechanism of Nanofluid

2.4. Synthesis of Nanofluid

Synthesized Oleic acid coated Fe_3O_4 nanoparticles were used to create Poly alpha olefin based nanofluid. Nanofluid was prepared in five different volume fractions of 0.1wt %, 0.2wt %, 0.3wt.%, 0.4wt.% and 0.5wt.%. Then, the nanocomposites were dispersed in 14ml of poly alpha olefin oil through sonication of 2 hours at 70°C to obtain a stable suspension.

3. CHARACTERIZATION

X-ray diffraction was employed to examine the phase, crystal structure, d-spacing and particle size of the sample. X-ray diffraction analysis (XRD) of iron oxide nanoparticles and coated nanoparticles was examined. The diffractometer used for this analysis was Bruker D8 Advance Diffractometer prepared with copper tube ($\lambda = 1.5406A$) and average range was set from 20° to 80° along with the step size of 0.02°. Fourier Transform Infrared Spectroscopy was used to detect compositional analysis. The spectrometer used for analysis was Perkin-Elmer double beam spectrophotometer in combination with the KBr disc. Range of the spectral series was taken from 4000-400 cm^{-1} at room temperature. The KBr disc technique was used to prepare samples for coated and uncoated IONPs. 200 mg of ground KBr was mixed with 4mg of the nanoparticles, which were than pressed (10 $tonnes/cm^2$) to form pallets. Scanning electron microscopy was employed to examine the surface morphology of the nanocomposite. VEGA 3 TESCAN (Czech Republic) 10 nanometers at 30 kV was used to take the SEM micrographs and the double carbon coated sticky tapes were used to analyse the sample and evade charging. The thermal conductivity of the synthesized nanofluid was evaluated by Transient Plane Source (TPS) method. The device used was TPS 2500 Thermal Constants analyser. It can measure the thermal conductivity of a material over a series of 0.005 to 1800 W/m/K.

4. RESULTS AND DISCUSSION

In this section, the results of synthesis and characterisation of uncoated Iron Oxide nanoparticles, 5 wt.% Oleic acid coated IONPs and Poly Alpha Olefin oil based nanofluid with 0.1wt %, 0.2wt %, 0.3wt.%, 0.4wt.% and 0.5wt.% are discussed. The crystalline structure, composition, dispersion stability and thermal conductivity measurement of the synthesized nanoparticles and nanofluid was studied by using XRD, FTIR and Thermal Constants analyser technique, respectively.

4.1 Phase Identification

XRD of the coated and uncoated IONPs was done for phase identification and crystalline structure. The interactions of the sample with X-rays give different diffraction patterns belonging to a specific crystal.

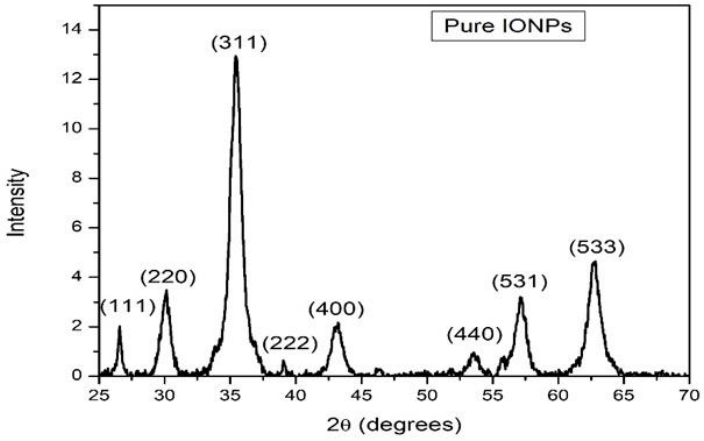

Figure 2. XRD patterns of Pure IONPs

The XRD investigation of pure IONPs is shown in Fig. 2, peaks at (220), (222), (311), (400), (440), (531) and (533) lattice planes which match to the standard pattern of face centred cubic spinel Fe_3O_4 (JCPDS card No. 82-1533). The broad peaks indicate the polycrystalline and ultra-fine nature of the samples. The results show that this is the pattern for Fe_3O_4 because the planes

(210), (300), (320) did not appear which gives peaks for Fe_2O_3. The size was found using Scherrer Equation and found to be 6.45 nm.

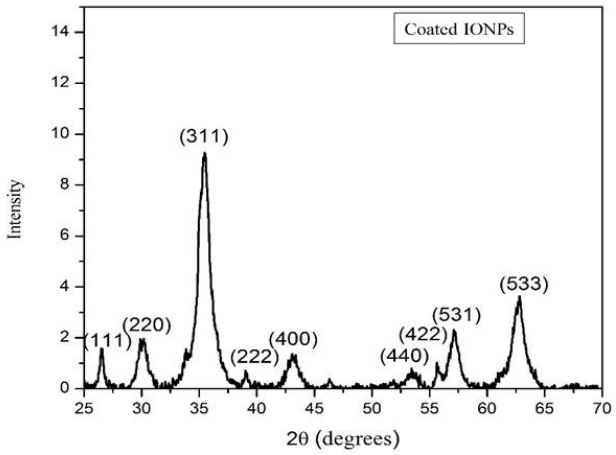

Figure 3. XRD patterns of Oleic acid coated IONPs

The pattern of Oleic acid coated IONPs is shown in Fig. 3 which shows a decrease in the intensity resulting in broadening of the peak due to the coating of oleic acid. The size was found to be 6.48 nm.

4.2 Compositional Analysis

The compositional analysis of the coated and uncoated IONPs was done by using Fourier-Transform Infrared Spectroscopy (FTIR). The interaction of infrared rays with the particle gives information about presence of elements in a compound.

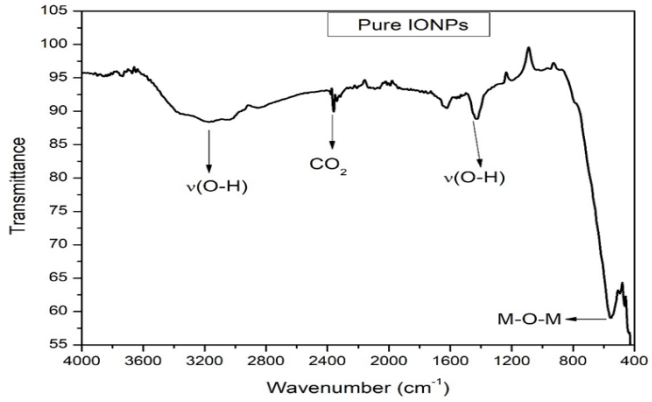

Figure 4. FTIR Spectra of pure IONPs

The FITR of pure IONPs is shown in Fig. 4. The bands in the region of 600 cm^{-1} and 571 cm^{-1} is the characteristics of Fe-O group and is due to the stretching vibrations tetrahedral and octahedral positions of the metallic particles (M_{Th}–O–M_{Oh}) [30]. The peak at 3400 cm^{-3} is due to the stretching vibrations of O-H group and the peak due to absorbed water in the nanoparticles is shown at 1650 cm^{-3}.

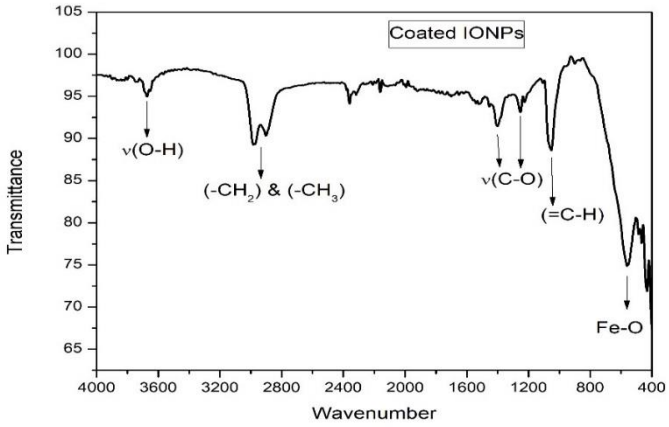

Figure 5. FTIR Spectra of Oleic acid coated IONPs

The C-O-C group present at 1185 cm^{-1} and stretching of -COO- at 1435 cm^{-1} and 1500 cm^{-1} refers to the presence of Oleic acid, as shown in Fig. 5. The peaks at 2935 cm^{-1} and 2850 cm^{-1} are referred to as asymmetric and symmetric stretching of ($-CH_2$) and ($-CH_3$) group present in the oleic acid. Hence, the compositional analysis confirmed coating of polymer on nano particles.

4.3 Surface Morphology

The surface morphology and agglomeration behaviour of the pure and oleic acid coated IONPs is examined through SEM. Fig. 6 shows the SEM photographs and histogram for mean diameter (µm) of pure IONPs. It was observed that the particles are agglomerated for high concentration of nanoparticles after 2 hours of sonication in ethanol (solvent). The aggregation of nanoparticles is due to their high magnetic properties and the interaction among them. The high surface charge and dipolar interactions caused these particles to form large clusters. The mean size of IONPs was calculated carefully using ImageJ software and was obtained to be 6.4nm which is consistent with the crystallite size.

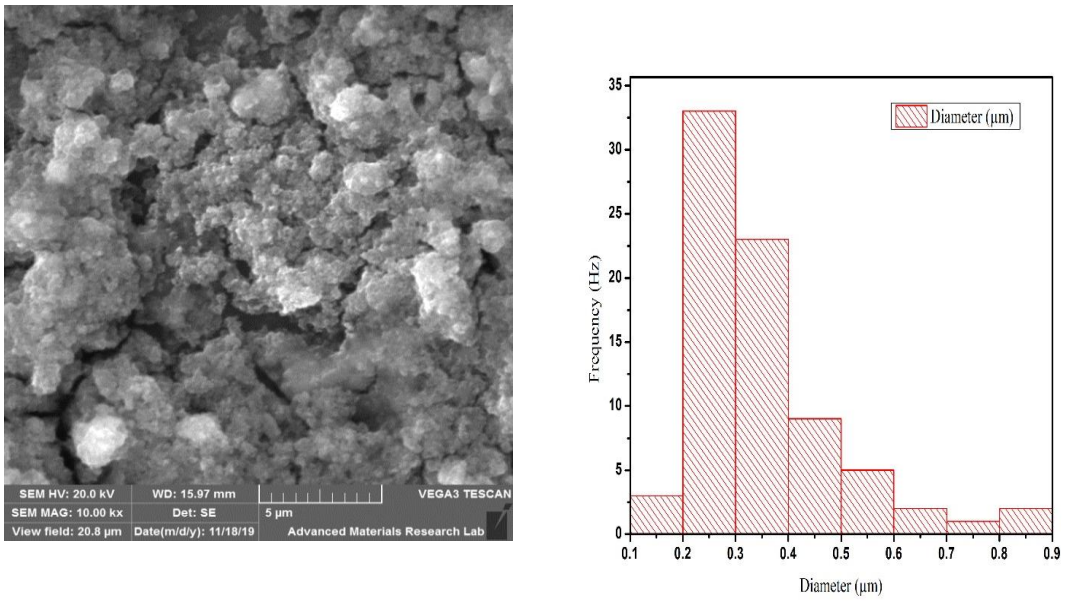

Figure 6. SEM Analysis of pure IONPs

Fig. 7 shows the SEM examination of coated IONPs. The nano sized particles show slight cluster formation due to the magnetic interaction among particles. The high surface energy and dipolar attraction forces are reduced due to coating of polymer and the size was enhanced which is evident from the histogram of mean diameter. The size was reported as 6.43nm consistent with crystallite size.

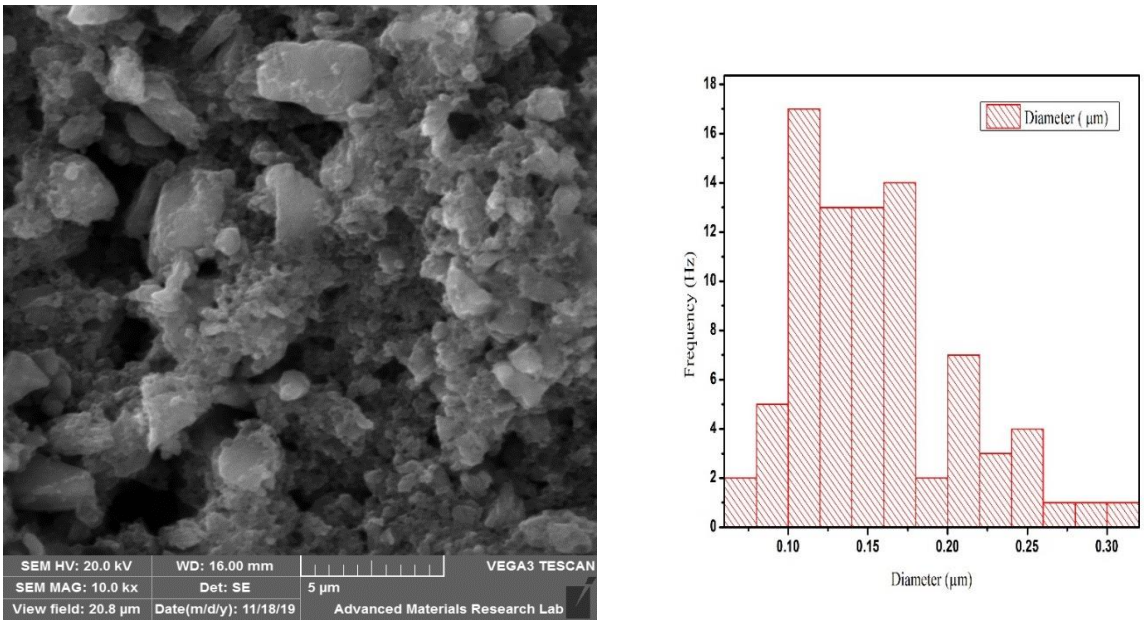

Figure 7. SEM Analysis of coated IONPs

4.4 Thermal Conductivity of the Synthesized Nanofluid

The thermal conductivity of the synthesized nanofluid was measured by Transient Plane Source (TPS) method. The main working of TPS method is the transiently heating of plane sensor thus, it is referred as Hot Disk Thermal Constants Analyzer.

Fig. 8 shows that in each of the above curves, the thermal conductivity reduces with increment in temperature. This inverse relation is due to the formation of aggregates over a long period of time, providing new pathways for conduction but when the quantity of cluster is very high, it decreases the thermal conductivity values [31]. The slope of the curve depends upon the volume fraction, as shown in the graph.

High thermal conductivity value was achieved for 0.2 wt. % at 50^{o} C and the lowest value was observed at 0.5 wt. % at 97^{o} C. Curve (a) shows a rise at 73^{o} C but this is a negligible rise. Curves (b) and (c) also show rise at some point.

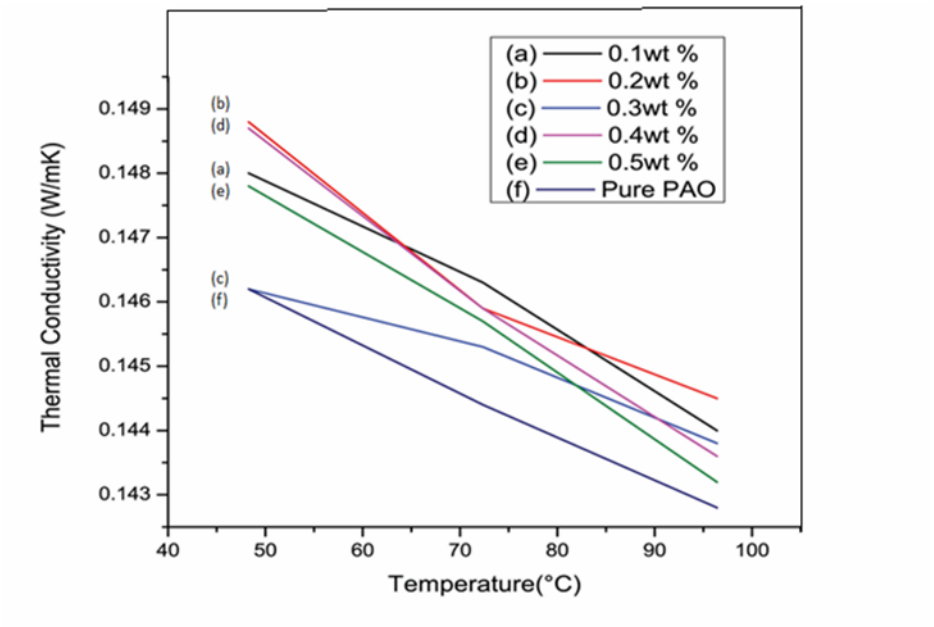

Figure 8. Thermal Conductivity VS Temperature graph

The thermal conductivity of nanofluids increases with increase in temperature, but oils have a trend of reducing thermal conductivity with the increase in temperature because the viscosity of oils decreases due to intermolecular collisions. This is because the conduction pathways are less available due to the increased distance between the molecules [32]. On comparing all of the curves, it was observed that the thermal conductivity increases with the increase in volume fraction of the nanoparticles.

4.5 Stability of Nanofluid

Stability of the nanofluids is one of the most important characteristics to be controlled while synthesizing a nanofluid. The shelf life of synthesized Poly Alpha Olefin oil (PAO) based nanofluid was examined over time to check the aggregation behaviour. This involves photograph capturing of the PAO nanofluid of 0.1wt %, 0.2wt %, 0.3wt.%, 0.4wt.% and 0.5wt.% volume fraction stored in bottles, over a period of one month. Fig. 9 shows the digital images of five different concentrations of nanofluids after one day of synthesis. High stability of the PAO nanofluid can be observed.

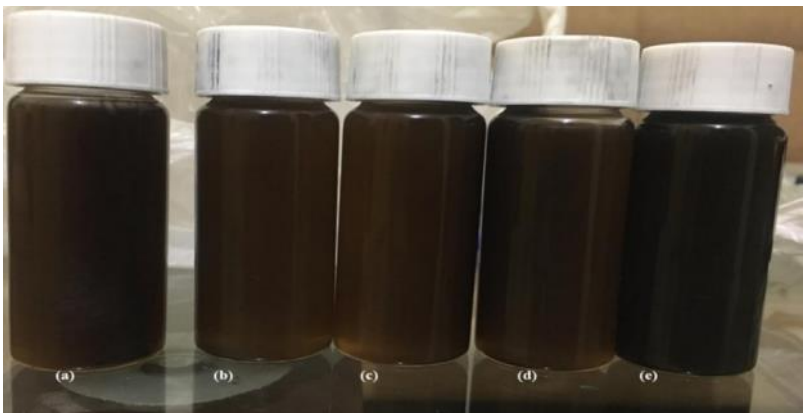

Figure 9. Photograph of (a) 0.1wt % (b) 0.2wt % (c)0.3wt.% (d) 0.4wt.% (e) 0.5wt.% of nanofluid after one day.

After 14 days, the nanoparticles suspended in PAO based nanofluid started to settle down at the bottom of bottle. However, this sedimentation was restored by sonication of 5 minutes. This is shown in Fig. 10.

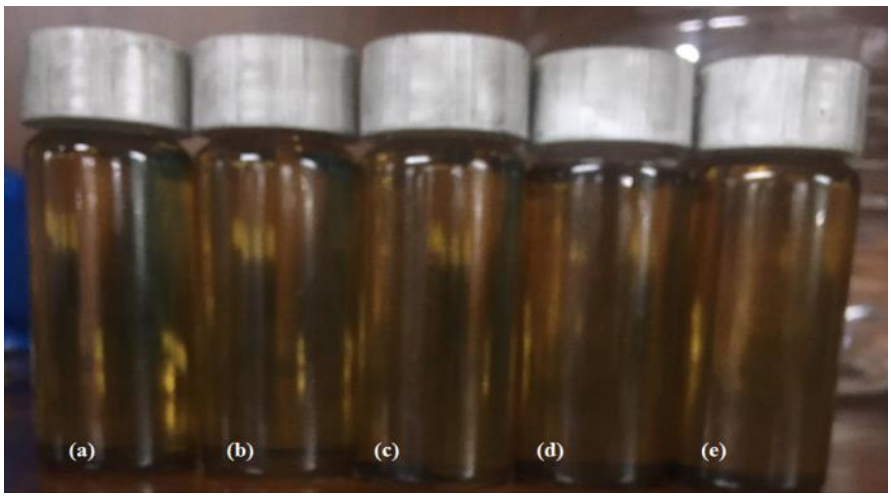

Figure 10. Photograph of (a) 0.1wt % (b) 0.2wt % (c)0.3wt.% (d) 0.4wt.% (e) 0.5wt.% of nanofluid after 14 days.

The complete aggregation of the nanoparticles was observed after 30 days. This indicates that the shelf life of PAO based nanofluids is 30 days, after this period, the nanoparticles were completely settled down in the base fluid. This can be observed in Fig. 11.

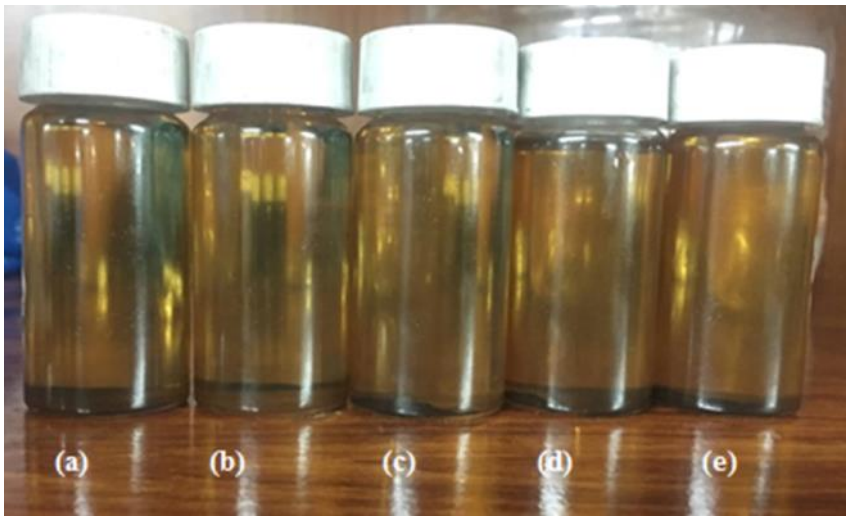

Figure 11. Photograph of (a) 0.1wt % (b) 0.2wt % (c)0.3wt.% (d) 0.4wt.% (e) 0.5wt.% of nanofluid after 30 days.

5. CONCLUSION

Synthesis of uncoated IONPs was carried out by a simple yet effective method by slight changes in the chemical co-precipitation method. Initial concentration ratio used for Fe^{+2} and Fe^{+3} is 1:1.6. This type of preparation technique is very significant as it can have entirely different outcome in thermophysical properties, settle down behaviour, particles clustering and pH values. The surfactant employed for the stability of synthesized nanoparticles was Oleic acid. The carboxylic acid forms a strong bond with Fe_3O_4 nanoparticles. The prepared nanoparticles and nanofluid were characterised to study the crystal structure, morphology, composition and thermal

conductivity of the synthesized nanofluid. The XRD analysis of the coated and uncoated IONPs shows that the synthesized particles are polycrystalline with a face centred cubic spinel structure. The FTIR analysis reveals the successful spread over Oleic acid on the synthesized Fe_3O_4 nanoparticles. The photograph capturing method to estimate the stability of the synthesized nanofluid shows that the nanofluid is partially stable after two weeks of synthesis while the complete settling of the Fe_3O_4 nanoparticles occurred after one month of the synthesis. The thermal conductivity of the nanofluid was found to increase than that of the pure Poly Alpha Olefin oil. High thermal conductivity value was achieved for 0.2 wt. % at 50^{o} C.

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