

Experimental Study on the Characterisation and Thermo-Physical Properties of Graphene-Agro Based Hybrid Nanofluids

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Abstract

Over the decade, researchers have developed new nanofluids for heat transfer applications. Little or no reports are made with the use of Agro-waste products as nanofluids. The sole purpose of this study is to evaluate the potential of nanoparticles (rice husk ash and graphene) in the thermo-physical enhancement of nanofluid for heat transfer applications. The study also highlights the synthesis of rice husk ash-graphene nanoparticles and the preparation of nanofluids. Particle characterisation was conducted using techniques such as SEM/EDXRF, XRD and TGA/DTA. Thermal conductivity, viscosity and heat capacity of the nanofluid were investigated for different particle concentration (0.1, 0.3 and 0.5wt.%) at temperatures (50, 60 and 70°C) with deionized water as base fluid. The examine nanofluids shows better stability, an average maximum thermal conductivity enhancement of 71% at 70°C and 0.5wt.% concentration for hybrid nanofluid. The excellent thermal properties of the nanofluids make them suitable candidates as nanofluids for heat transfer application.

Keywords: Graphene, Rice husk ash, Nanofluids, Thermal conductivity, Viscosity, specific heat capacity.

1.0 Introduction

Energy is generally known for its significant characteristics in industrial processes that must be transferred from one system to another before a system could perform work. The energy transfer can be performed through work or heat [1]. The science that depicts the rate and manner in which these energies are transferred is referred to as heat transfer. Heat transfer applications can be noticeable in our day-to-day life as in refrigeration [2], transportation [3] and electronic temperature control devices [4], drilling operations [5], and in optimized solar collector [6]. In recent decade, there are a progressive and drastic advancement in the area of technology and its applications which necessitate the development of an efficient heat transfer fluids. One important aim using fluids in operational processes is to overcoming excessive heat generated during operation and to enhance the thermal capability, suitability as well as life expectancy of heat transfer equipment or devices.

Rapid advances in nanotechnology (the compact size of devices has become smaller and thus requires better and efficient thermal management) have significantly results to the emergence of new age cooling media known as “nanofluids”. Nanofluids are new generational class of coolants composed majorly of micro/nano powders suspended in the host fluids [7]. Researchers in their recent studies has proven that nanofluids exhibit better thermal characteristics such as thermal conductivity in comparison with the conventional fluids such as water, mineral and vegetable oils [8] and are considered as a significant material for a holistic thermal transfer. A significant improvement in heat transfer characteristics using nanofluid have aroused researchers’ interests in employing nanofluids as an alternative cooling agent in various areas of applications [9].

Graphene, has shown great potentiality as a thermal transfer fluid and applicable in vast area due to its outstanding material characteristics. Researchers [10] conducted a study on thermal characteristics of graphene nano-powder in an automobile radiator, water-ethylene glycol was employed as the host fluid. Thermal conductivity enhancement of > 40% was measured, giving rise to the possibility of a dynamic and innovative fluid operations [11]. High thermal conductivity of graphene-based fluid makes it a suitable material for better heat transfer management in batteries (Li-ion battery) and fuel cells [12]. An experimentally heat transfer study on an electronic device using graphene-based nanofluid was conducted by [13]. Study measured that with an addition of graphene nanoparticle, convective heat transfer of the fluid increases and discovered to be effective and at elevated heat loading which may increase life-expectancy of the device and further mitigate device failure. Researcher [14]

assess the thermal performance of a compact heat exchanger using graphene in water-ethyl glycol (60:40) nano-suspension with results showing that with an addition of graphene nanoparticles suspension, an enhancement in heat transfer coefficient (HTC) was reported. This enhancement in HTC can be attributed to improvement in the Brownian motion and thermal properties of the nanofluid. Graphene-based nanofluids show better stability and improved thermal performance in heat transfer applications [15-17].

Rice husk ash (RHA) generally known to be composed essentially of carbon and high level of silica has shown outstanding heat transfer properties. Certain authors [18] measured and optimised the thermal characteristics of paraffin-wax material incorporating Rice husk-derived graphene nanoparticles (RGP) to enhance solar plate collector's energy storage capabilities and efficiency. Study shows, thermal conductivity of 0.59W/mK was measured for RGP and Maximum efficiencies of 63.9% were noted for 1.5wt.% of Rice husk-derived graphene at a 3L/min flow rate. A good heating value and moisture content of rice husk ash indicates its potentiality for use in energy generation [19]. Experimentally, rice husk ash (RHA) has been shown to exhibit high degree of emissivity, which makes it a suitable material for radiative cooling applications - microwave absorbing properties [20], thermal insulating material in building [21]. In another view, rice husk ash has been utilised to develop an efficient, cost-effective heat exchanger for vary industrial applications [22-24].

Based on literature survey, it can be inferred that the thermal-performance of heat transfer fluids are majorly dependant on their individual thermal-physical properties. In this study, an experimental study on graphene/rice husk ash nanofluid suspension was critically analysed to evaluate the thermal properties as potential candidate for heat transfer fluids.

2.0. Experimental Procedures

2.1 Materials

The Chemical vapour deposition (CVD) method was adopted in the production of graphene nanoparticle (GNP) and the CVD reactor was used to achieve graphene growth. The growth was done under mixture of Argon (Ar), Hydrogen (H₂) and acetylene gases. The Ni-foil was used as a substrate material for synthesizing graphene. Metal foil of dimensions 20 × 5 × 0.2mm was sliced from a larger sheet. The CVD reactor chamber was pre-heated to a desired temperature with ramping rate of 750°C/min under flow of 1000sccm H₂ and Ar gases, this was followed by annealing the nickel (Ni) foil prior to the graphene growth under flow of Ar, H₂ and acetylene gases for 15min at 1000°C. The annealed Ni foil was then inserted into the reactor chamber. Acetylene gases were further introduced into the chamber, followed by the H₂ gas. Acetylene gas was set for 15sccm flow while argon and hydrogen gases was set for 60sccm. Pressure of 5, 3 and 1mbar was maintained for Ar, H₂ and acetylene gas respectively. The particle growth time was set for only 10min. Chamber was made to cool down under H₂ to room temperature, sample was afterward collected. An average particle size of 18 – 35nm was measured for the study.

To produce rice husk ash nanoparticle (RHA) the husks were separated from the seed with the use of a mini rice milling machine. Samples of rice husks were collected, washed and dried under room temperature. A 1500gram of rice husk samples were manually grounded to a powder form, and sieved with 250µm size mesh. Husks samples were calcinated at 700°C in laboratory muffle furnace for 6 hours and then allowed to cool in the furnace. A nano-sized RHA powders were prepared by mechanical milling with the use of a high energy planetary ball mill at 250 rpm for 3 hours and were further sonicated for 4 hours. An average sized-particle of 2 - 6nm was measured for the study. Table 1 shows the properties of water, graphene and rice husk ash nanoparticle.

2.2. Experimental Procedures

Dynamic light scattering (DLS) method was employed to measure the particle size distribution. This method uses a photon correlation spectrometer to measure the un-organised motion of particles in the fluids, consequently determine the hydrodynamic diameter of the particles.

Scanning Electron Microscopy (SEM) attached with Energy Dispersive X-Ray Florescence (EDXRF) - SEM/EDXRF analyser was used in the study to measure the various morphology and elemental compositions in the developed nanoparticles. SEM analysis was performed using FEG-HR Zeiss-Ultra 55 SEM system while EDXRF analysis using Hitachi-S-4800 system. A collection of highly accelerated parallel ray pierce through the sample resulting to re-emitted particles. The EDXRF maximises the use of diffracting power of a single crystal to divide the polychromatic rays from the sample into two separate wavelengths.

Particles characterisations were carried out to determine crystal structure or phase composition using Rigaku SmartLab with high-resolution X-ray diffractometer. The crystallography patterns of the particles were obtained by producing X-rays diffractions current and voltage of 35mA and 40kV.

Thermogravimetric analysis/Differential Thermal Analysis (TGA/DTA) analyser (USA model TGA Q500) was used to measure the thermal degradation phases that occur under elevated temperature. About 10.523 and 10.933 mg of graphene and rice husk ash samples were subjected to a heating process in nitrogen atmosphere at a range of 30 - 950°C at 10°C/min heating rate. The initial decomposition temperature and weight loss of the samples were measured from the TGA and DTG graphs as a measure of their individual thermal stability.

Furthermore, nanofluid was prepared by employing two-step technique with a nanoparticle concentration of 0.1, 0.3 and 0.5% respectively. Due to the nature of graphene (hydrophobic characteristics), the use of a surfactant (Sodium Lauryl Sulfate - SLS) was adopted to stabilise the nanofluids. The choice for these surfactants was due to their high affinity to absorb into nanoparticle surfaces of the substance and enable proper wettability [25], with the base fluids and its directed related to the steric stabilization mechanism [26]. 1000ml of distilled water (DW), vary weight concentration of nanoparticles were thoroughly mixed for one hour at vary temperatures (25, 50, 60 and 70°C respectively). To obtain an homogeneous suspension, the nanofluids were thoroughly stirred for 30 minutes afterward the surfactant was introduced into the mixture. The mixture was further stirred further another 30 minutes. The composition specifications in the preparation of both individual and hybrid nanofluid is depicted in Table 1.

Table 1: Nanofluids composition

Sample/wt.%	GNP (0.1)	GNP (0.3)	GNP (0.5)	RHA (0.1)	RHA (0.3)	RHA (0.5)	GNP/RHA (0.1)	GNP/RHA (0.3)	GNP/RH A (0.5)
GNP (wt. %)	0.1	0.3	0.5	0.00	0.00	0.00	0.05	0.15	0.25
RHA (wt. %)	0.00	0.00	0.00	0.1	0.3	0.5	0.05	0.15	0.25

Thermal conductivities of developed nanofluids were measured using the KHCT-143 equipment at vary temperatures and weight concentrations. An average of five (5) readings were taken at each temperature and particle concentration.

NDJ-8S rotational viscometer was used to measure the rheological behaviour of the developed nanofluids at vary temperatures and particle concentrations. It was measured in accordance to ASTM D-44504 standard.

Specific heat capacities measurement of the developed nanofluids was measured employing the ASTM method of E 1269-05 (DSC/PerkinElmer, DSC4000, Waltham, MA, USA).

3.0 Results and Discussion

3.1 Materials Characterisation

Table 2 shows the thermo-physical properties of nanoparticles under investigation. Moreso, morphology of developed nanoparticles was characterised by SEM/EDXRF, as depicted in Figure 1. EDXRF spectrum shows 45% carbon and 20.2% Oxygen for GNP and lower carbon presence of 4.22% for RHA nanoparticles as depicted in Figure 1(a,b). GNP nanoparticles are nanoplates that have 18 - 35nm particle thickness with a 126 μ m average particle diameter. The RHA nanoparticles have 2 - 8nm outside diameter with an average of 9 - 11 μ m.

Table 2: Thermo-physical characteristics of GNP and RHA nanoparticles

Characteristics	Water	GNP	RHA
Colour	Colourless	Black	Grayish-black
Molecular mass (mass/g mol ⁻¹)	17.91	16.64	35.20
Morphology	NA	Spherical	spherical
Density (Kg/m ³)	986	123.4	2115.1
Thermal conductivity (W/mK)	0.632	0.699	0.681
Specific heat capacity at 20 °C (KJ/KgK)	4.167	0.683	0.760

Figure 1 also shows the presence of various elemental compositions for each developed nanoparticle. SEM characterization patterns at high magnifications of the developed graphene and rice husk ash powders shows that the nanomaterial used has spherical shape with closely stacked nanoplatelets very visible. This may be due to the materials large surface to volume ratio. However, the apparent diameter was measured to range from ~1 - 50 μ m and such variation in diameter in all the nano-powders is normal and has been recorded by [17, 19, 27-28]. The elemental spectrum and mapping using EDX analysis (a,b) show that the sample was majorly silica and carbon elements which are pivot elemental composition in rice husk ash and graphene nanoparticles. Figure 1 depicts the SEM and EDX pattern of GNP and RHA while Figure 2 shows the XRD pattern of graphene and Rice husk ash nanoparticles.

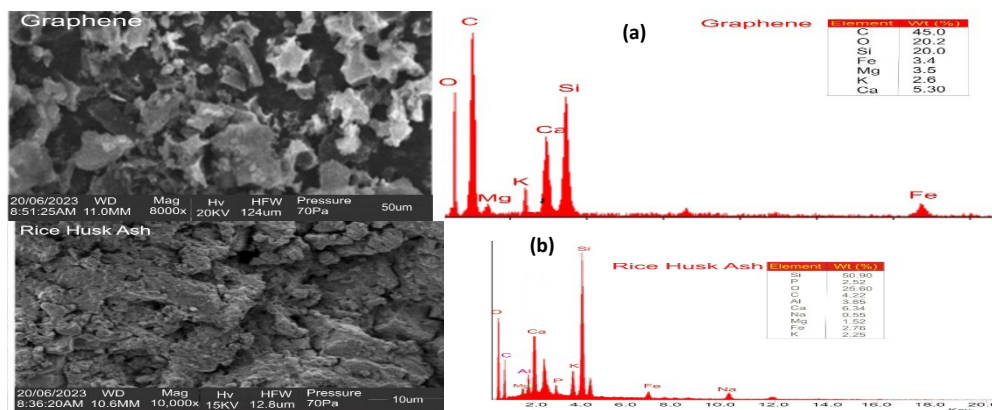


Figure 1: XRD, SEM and EDXRF characterisation analysis of (a) GNP and (b) RHA nanoparticles

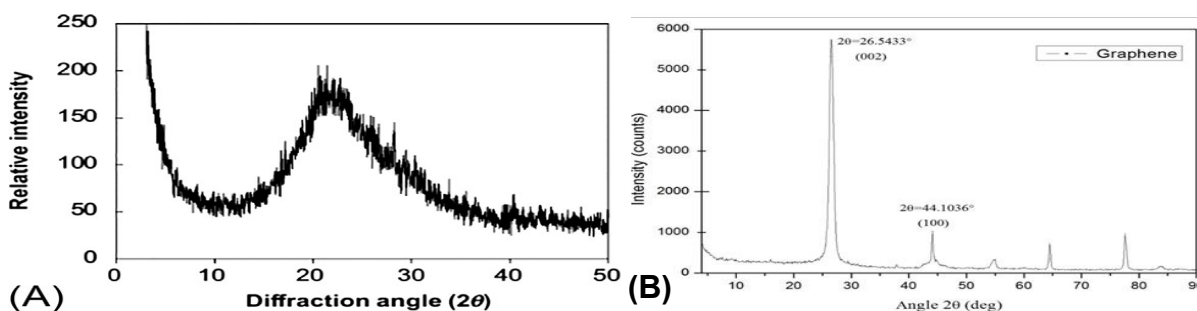


Figure 2: XRD characterisation analysis of (a) RHA and (b) GNP nanoparticles

XRD pattern of graphene (Figure 2) shows a crystalline index of 0.876563 with few-layer graphene, consisting of 2 – 5 graphene layers, with a high degree of crystallinity and a well-defined hexagonal lattice. The crystallinity lattice may exhibit highly ordered crystal planes, with low presence of defects, vacancies, or impurities, contributing to the high crystalline index value. This also explains the high thermal conductivity and mechanical strength. The graphene nanoparticle exhibits an extremely small crystalline domain size of 0.006884nm which is approximately 6.884 Å (angstroms) of roughly few atomic layers. With such a small crystalline size, the surface area of the nanoparticles is expected to be very high, potentially leading to unique properties. At this scale, quantum confinement effects may become significant, thereby influencing the particles’ optical, electrical and magnetic properties resulting in high reactivity due to the large surface area and potential defects.

The measured spectrum of rice husk ash nanoparticles as shown in Figure 2a broad diffraction peaks confirmed a completely amorphous structure and matched well with the predominant diffraction patterns of the bulk silicon dioxide (SiO₂) with other elements.

The measurement of thermal stability of the nanoparticles in respect to particle weight loss and temperature of rice husk ash and graphene nanoparticles using TGA/DTA analysis is depicted in Figure 3.

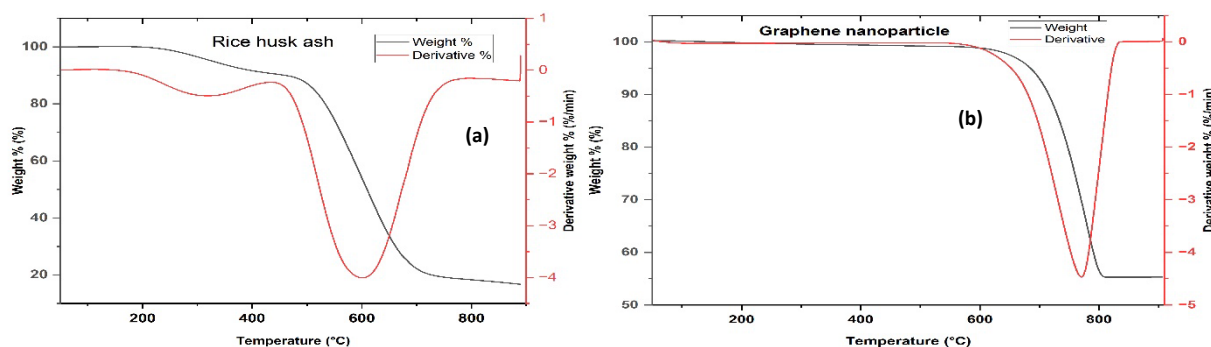


Figure 3: TGA/DTA analysis of (a) rice husk ash and (b) graphene nanoparticles

Figure 3(a, b) depicts the decomposition pattern obtained for the developed nanoparticles. A total of 72.1wt.% loss at 210 °C, 440 °C and 745 °C was measured for rice husk ash (RHA) nanoparticle while a total of 43.5wt.% loss was measured for graphene (GNP) nanoparticle at 570°C respectively. Decomposition temperature in the decarbonisation profile differs in the various nanomaterials. The oxides of decomposition of RHA nanoparticles occurs at about 240 °C earlier than GNP which show late decomposition at about 620°C. There is a downward shift in the thermal stability of RHA nanoparticle compared to GNP nanoparticles. No residue was observed in

the decomposition pattern which implies that the samples were free of metallic impurities. Thermal stability of the developed nanoparticles follow this pattern.

GNP nanoparticles > RHA nanoparticles

3.2 Nanofluids Properties

3.2.1 Thermal Conductivity

Results from Figure 4 indicates that high thermal conductivity can be traced down to a corresponding increase in nanoparticle concentration and temperature. These results are in accordance with the researchers [29-35]. One other possible explanation related to this development is the fact that with increase in particle concentration, an increase in the number of fluid particles is observed, resulting to clustering of these particles. In this condition, the transfer of heat from solid phase is rapider than from liquid phase, resulting in an enhancement in thermal conductivity [36]. Moreso, nanoparticle movement increases with increasing temperature due to higher collision between surface atoms and fluid molecules, relative thermal conductivity increases [37]. However, researchers [38-39] argued that the relative thermal conductivity is not a function of temperature. This implies that an increase in thermal conductivity of nanofluids with temperature were due mainly on the base fluids (water and ethyl glycol) rather than the nanoparticles. Researchers [40] in a bid explain the theoretical inconsistency of the nanofluids enhancement in thermal conductivity, cited possible causes as a result of incomplete characterisation of nanofluids samples; broad range of experimental approaches adopted and the differences in synthesis processes used for sample preparation, even for nominally similar nanofluids.

In the study, overall thermal conductivity enhancement for RHA nanofluid at 0.1wt.%, 0.3wt.% and 0.5wt.% particle concentration are recorded as 20, 25 and 27% respectively while graphene shows higher overall thermal conductivity enhancement of 38, 41 and 51% compare with the deionize water only. Hybrid nanofluid (RHA+GNP) measured better thermal conductivity enhancement of 52, 54 and 71% at vary particle concentration compared with the use of deionize water alone. The significant thermal conductivity increase using graphene-based nanofluids as compared to rice husk ash nanofluids can be attributed to its exceptional thermal characteristics as compared to the other nanoparticles. Thermal conductivity of both individual and hybrid nanofluid shows better enhancement of 27, 51 % and 71% for rice husk ash, rice husk ash/graphene nanofluid at 70°C and 0.5wt.% particle concentration. Figure 5 depicts the influence of temperature on viscosity of the developed nanofluids.

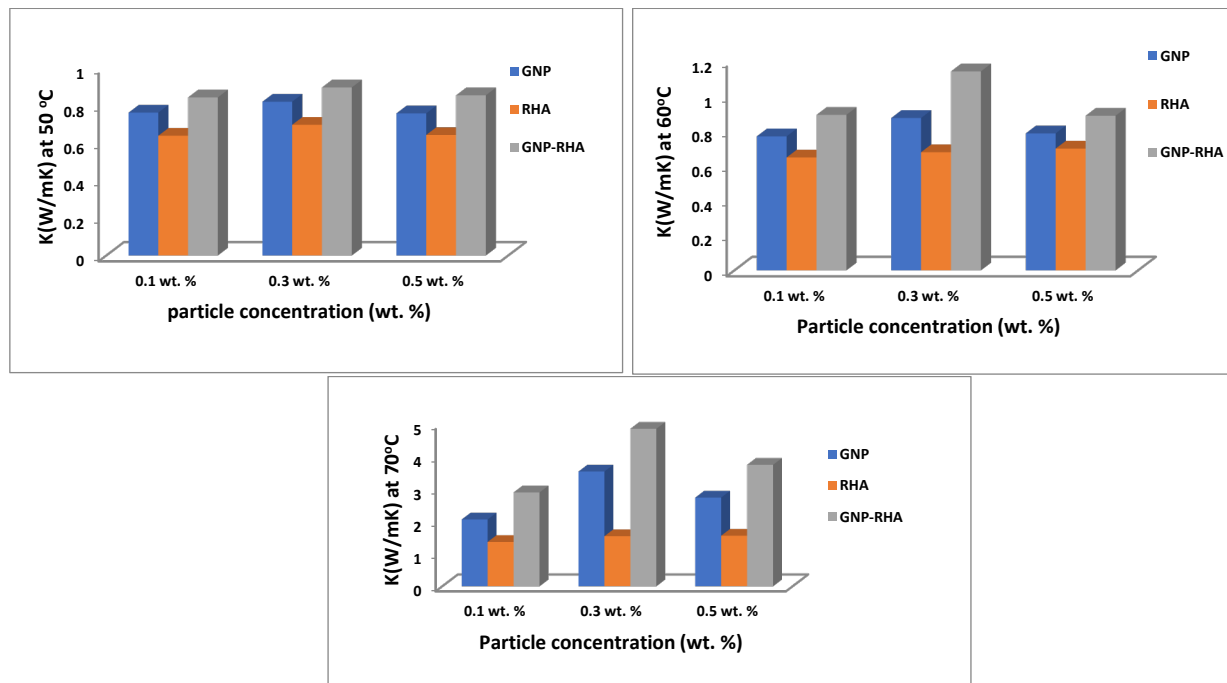


Figure 4: Thermal conductivity of the developed nanofluids at varies temperature

3.2.2 Viscosity

Figure 5 depicts the relationship between particle concentration and viscosity of nanofluid. Rice husk ash nanofluid shows viscosity decreases with increase in temperature but show incremental with particle

concentration. An average percentage decrease in viscosity at vary temperature with 0.1, 0.3 and 0.5wt.% particle concentration are 7.5, 3.5 and 4% respectively while graphene nanofluid shows percentage decrease in viscosity of 6, 5.5 and 5.5% only. Hybrid nanofluid (RHA+GNP) measured an average decrease of 7 and 2.6% with 0.1, and 0.3% but with further increase in temperature above 60°C, an increase of 4% in viscosity was measured.

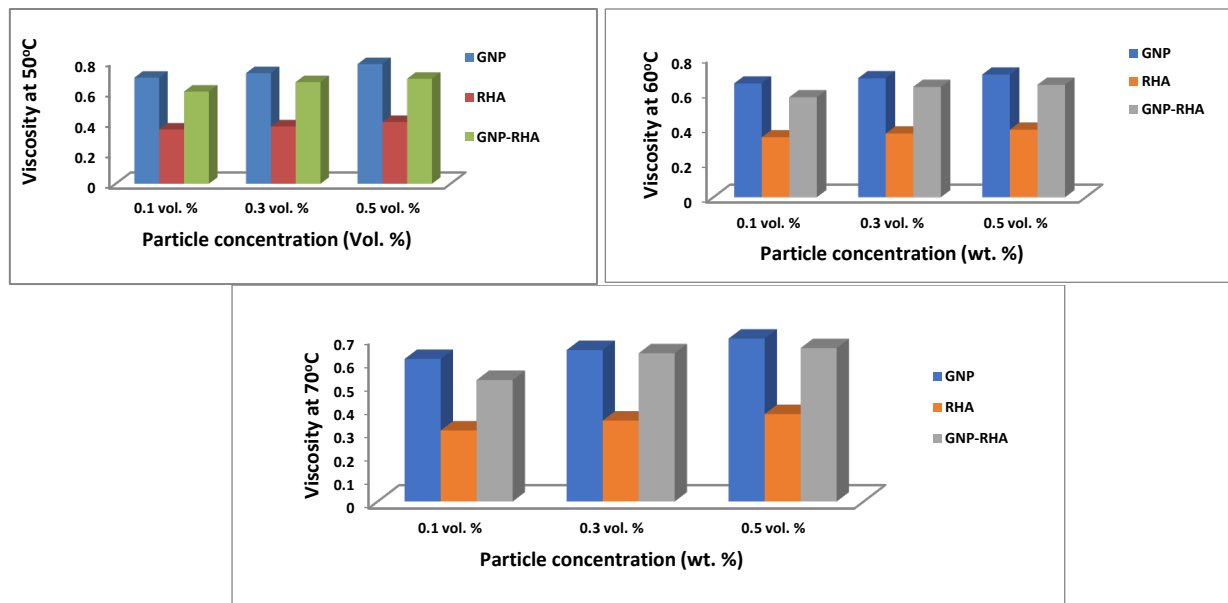


Figure 5: Viscosity of developed nanofluid at varies temperature

The both individual, hybrid nanofluid samples show increase in viscosity with corresponding increase of particle concentration but a decline with rise in temperatures as depicted in Figure 4, which is generally accepted. This thermal behaviour may be traced weakening of molecular cohesive forces with in-creasing temperature, resulting to a reduced the shear stress and then the viscosity [41]. Moreso, the declination of viscosity is attributed to nanoparticle’s micro-conventional motion in base fluid which consequently decreases the inter-molecular forces between base fluid molecules [42]. Furthermore, as temperature increases, the velocity of nanoparticle increases due to Brownian motion [43]. Viscosity of the various nanofluids showed contradictive characteristics with report by [44]. A decrease in viscosity with increasing temperature with graphene-based nanofluids was also recorded by researchers [41,45-46].

3.2.3 Specific heat capacity (SHC)

The fluid heat capacity plays a significant role by ascertaining the cooling or heating capacity of the heat transfer fluid per unit increase or decrease in temperature. Table 3 shows the specific heat capacity of nanofluids with temperature (°C) and particle concentration (wt. %).

Table 3: Specific heat capacity of developed nanofluids

Samples (wt.%)/Temp(°C)	GNP (0.1)	GNP (0.3)	GNP (0.5)	RHA (0.1)	RHA (0.3)	RHA (0.5)	GNP/RHA (0.1)	GNP/RHA (0.3)	GNP/RHA (0.5)
50	2.900	2.827	2.784	3.558	3.479	3.446	3.432	3.410	3.403
60	2.904	2.831	2.788	3.563	3.481	3.453	3.438	3.415	3.406
70	2.907	2.835	2.791	3.576	3.483	3.456	3.440	3.416	3.407

Table 3 illustrates the specific heat capacity with particle concentration variation at vary temperatures (50 – 70 °C). The overall specific heat of both individual and hybrid nanofluids shows an incremental in temperature but decreases with addition of particle [47]. This may be attributed to the fact that particle excitement remains on ground level unless there is a temperature rise. Hence, the higher degree of stored thermal energy, the higher the degree of particle that is unconstrained [47]. Rice husk ash nanofluid shows an average decrement in heat capacity of 1.6, 1.6 and 1.7% at 0.1, 0.3 and 0.5wt.%, graphene nanofluid show higher average percentage decrease of 1.4, 2.0 and 2.1% while hybrid nanofluid measured lower average decrease in heat capacity of 0.4, 0.5 and 0.5% respectively. An average of 0.1% increase in heat capacity was measured with temperature rise for all nanofluids samples. An average heat capacity of 15.7, 31 and 18% was measured for rice husk ash, graphene and hybrid nanofluid compared to base fluid. However, most studies show similar reports but contrary results were observed [48].

4.0 Conclusion

The experimental study is carried out to evaluate the suitability of rice husk ash, graphene and rice husk ash/graphene nanofluid for heat transfer application by measuring the thermal properties of the nanofluids under study. In the present study, a water-based rice husk ash/graphene nanofluid is prepared using two-step technique. The thermal-physical properties were measured experimentally at vary nanofluid concentrations of 0 – 0.5% and temperatures of 50 – 70°C, the following conclusion can be deduced;

The maximum thermal conductivity is enhanced by 27 and 51% at 70°C and 0.5wt.% concentrations for rice husk ash and graphene nanofluids compare to base fluid. Graphene/rice husk ash nanofluid show higher thermal conductivity of 71% at temperature of 70°C and 0.5wt.% concentration respectively.

The sensitivity of thermal conductivity to nanofluid concentration increases by the addition of nanoparticles and temperature rise. The temperature variation shows its lowest increasing effect on the viscosity for suspension with 0.3% concentration. Rice husk ash, graphene and rice husk ash/graphene nanofluids show a viscosity decrease of 4%, 5.5% and 2.6% with increasing temperature and particle loading.

Overall heat capacity of the developed nanofluids increases in temperature with decreased particle loading and more significant at temperature above 50°C. Moreso, heat capacity of individual nanofluids is higher than the hybrid nanofluids, it can be deduced that hybrid nanofluid will gain and loose heat faster rate, and therefore present a better fluid for heat transfer purpose.

Though, rice husk ash shows amorphous spectrum but its considerable thermal conductivity characteristic makes it a potential material for heat transfer application.

In consideration on the various thermal properties of graphene and rice husk ash, these materials hold great potential as candidate as heat transfer fluids. Graphene, rice husk ash based nanofluid can be introduced as an alternative working fluid with high heat transfer potentials for use in the relevant heat related applications in heat pipes, automobile sectors (heat exchangers), solar collectors, electronics (transistors, sensors), energy storage and conversion (batteries, solar cells), catalysis and chemical reactions.

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