

RESEARCH ARTICLE

PREPARATION AND CHARACTERIZATION OF WATER-BASE AL₂O₃ NANOFLUIDS FOR HIGH VOLTAGE APPLICATIONS COOLING.

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Abstract

..... In this study, the water-base Al₂O₃ nanofluids were synthesized for the high voltage application cooling enhancement. Firstly, the alumina (Al₂O₃) was prepared by the sol-gel technique to make alumina nanparticles. The synthesized Al₂O₃ was inspected by the X-Ray Diffractometer and the Transmission Electron Microscopy. The inspected Al₂O₃ was a single phase material, which had average particles sizes \approx 8nm: 12 nm with cubic shapes. The water-base Al₂O₃ nanofluids were synthesized by the two-step method with different weight concentrations of alumina (0.2%, 0.4%, 0.6%, 0.8%, 1%, and 1.2%). The alumina was used in the nanofluids synthesizing with small concentrations to reduce particles agglomeration chance. The thermal conductivity of the synthesized nanofluids increased with increasing of Al₂O₃weight concentration as well as with temperature increasing, which gave higher thermal conductivity by about 11% at room temperature and about 25% at T=41°C for 1.2% Al₂O₃ weight concentration. Increasing of Al2O3 concentration increased the viscosity of prepared nanofluid, but its viscosity decreased with The water-base Al₂O₃ nanofluid electrical temperature rising. conductivity increased with Al2O3 concentration increasing, and increased with temperature ascending; however the synthesized nanofluids electrical conductivities were still low values. So the waterbase Al₂O₃ nanofluids could enhance the cooling of high voltage applications because of the significant thermal conductivity increasing at low nanoparticles concentration, and these nanofluids could be safe for the high voltage applications due to their low electrical conductivity.

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Introduction:-

The cooling in the high voltage applications is one of the important factors which affects directly on the high voltage instrument performance. Some of these applications use the double distilled water (DDW) as a heat transfer fluid. The cyclotron accelerator (Model: MGC-20) is one of the high voltage applications which is used for the radioisotopes production. The MGC-20 cyclotron accelerator has a primary cooling system, which is used for

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cooling of the high temperature cyclotron parts. The heat transfer fluid of the primary cooling system is cooled by a secondary cooling system (central cooling system) in a shell and tube heat exchanger.

The double distilled water (DDW) is essentially used as a heat transfer fluid in the primary cyclotron cooling system. It is preferred to use the DDW because of its low electrical conductivity which reduces the probable electrical sparks in the cyclotron components. In some cases, these electrical sparks cause damages in the cyclotron accelerator and that causes the machine shut down (Alekseev A.G. et al., 1969 & Galchuk A.Y et al., 1985). In some times during the cyclotron working for the production, the secondary cooling system chillers suddenly stop and that leads to the temperature rise of the cooling DDW which causes to the failure of the radioisotope production operation. So this study will concern about improving the thermal performance of the cyclotron accelerator cooling by modernizing the heat transfer fluid. One of the modern techniques for the heat transfer enhancement is using the nanofluid as a heat transfer fluid. The nanofluids are a new class of solid-liquid composite materials consisting of solid nanoparticles (in the range of 1-100 nm), scattered in a heat transfer fluid. Nanofluid also takes care about some issues such as sedimentation and corrosion which conventionally happened in non-homogenous solid/liquid mixture with particles in larger scales (Li X. et al., 2008 & Lee S. et al., 1999). The challenge is synthesizing a nanofluid with a high a thermal conductivity and low electrical conductivity because of adding the nanoparticles to the liquids may affect on the electrical conductivity of the nanofluid (Brien R.W.O., 1980). In this study, the aluminum oxide (Al₂O₃) was used as a nanoparticle material in this study.

Firstly, it is required to get the aluminum oxide in the nano-scale, and also this material should be single phase material (pure). The aluminum oxide was found in the Egyptian market, but by analyzing the Al_2O_3 wasn't a single phase material in the nano-scale. So it is better to prepare the aluminum oxide experimentally in the nano-scale. The sol-gel technique is one of the most valuable methods for the nano-sized materials production because it produces ultra fine powder with narrow size distribution in a moderately short preparing time at a low temperature (**Jiang L. et al., 2005**).

Materials and Experimental Details:-

Materials:-

Aluminum Sulphate $[Al_2 (SO_4)_3.16H_2O]$ and Anhydrous Citric Acid $[C_6H_8O_7]$ were obtained from ADWIC company (analytical grade). Double Distilled water (DDW) has been filtrated by **Fistreem Cyclon distiller (model:** WSC044) **and it** was used for all experiments. Oleic Acid was acquired from Polskie Odczynniki chemiczne).

Preparation of the alumina powder:-

In this research, it is required to prepare water base Al_2O_3 nanofluids, and that needs pure nanoparticles of alumina. So first, it is needed to prepare the alumina nanoparticles. The sol-gel technique is one of the most moderate methods used in the metal oxide nanoparticles preparation. So the sol-gel method was used for alumina powder fabrication to get it in the nanorange. In the sol-gel technique experiment, the used primary materials were the aluminum sulphate $[Al_2 (SO_4)_3.16H_2O]$ and the anhydrous citric acid. The aluminum sulphate was used as an aluminum source material and the anhydrous citric acid was used as a reaction fuel. The mole ratio of the aluminum sulphate to the anhydrous citric acid was 1: 1 in the aqueous solution. Firstly 0.1 mole of the anhydrous citric acid was added to 500 ml of DDW and the solution was stirred for 10 min till it became clear, then 0.1 mole of aluminum sulphate was added to the solution. The resultant gel was dried at 200 $^{\circ}$ C in a drying oven (Genlab/model: OV/100) for 24 hours, and then the resultant product was grinded in a ceramic mortar till it became a fine powder.

This powder was annealed at 900 0 C in a tubular furnace (Carbolite/model: CTF 12/75/700) for 3 hours till it transformed to a white alumina powder. Finally, the morphology of the Al₂O₃ powder should be analyzed by the X-ray diffractometer (XRD) and the Transmission Electron Microscopy (TEM) to get enough information about the alumina prepared particles.

Water Base Al₂O₃ (WBA) Nanofluids Preparation:-

In this study, WBA nanofluids were synthesized using the two-step preparation method because the two-step method is more suitable to synthesize nanofluids containing oxide nanoparticles (Eastman J.A. et al., 2001). The two-step preparation method is widely used in nanofluids synthesizing by mixing the nanoparticles with the base fluid. First, nanoparticles and the base fluid were previously prepared, and then nanoparticles were dispersed in the base fluid with direct mixing methods. An ultrasonic vibrator and a magnetic stirrer are used for nanopowders

dispersion in the base fluid to reduce particle agglomeration (**Mukherjeel S. & Paria S., 2013**). In these experiments, a 500 ml glass beaker and 50 ml graduated cylinder were used in the solution samples measurements. Hot plate stirrer (Torrey pines /model: HS10) and ultrasonic vibrator (COXO /model: CD-4820) were used for nanopartilces propagation. (AE ADAM /model: AEP-450G) weighing balance is used for samples weighing. The base fluid was prepared by adding the DDW to the oleic acid. The Double distilled water (DDW) was used as the main percentage in the base fluid with 90% of volume, and the Oleic acid was 10% of volume, which used as a surfactant. The surfactant could help in nanoparticles suspension in the nanofluid (stabilizing agent) (**Thrush S. J., 2012**).The WBA nanofluids were synthesized by suspending Al₂O₃ nanoparticles with different weight percentages (0.2%, 0.4%, 0.6%, 0.8%, 1%, and 1.2% wt.) in the base fluid was prepared with a volume 50 ml, and then the sample was weighted. The required weight percentage of alumina nanoparticles was added to each sample and mixed by the stirrer for 30 minutes, and then the sample was vibrated by the ultrasonic vibrator for another 30 minute to break particles agglomeration and give good nanopowder dispersion.

Thermal conductivity (TC) Measurement :-

There are transient and steady state methods for the thermal properties measurements. Although steady-state methods are simple theoretically, these are complicated in experimental procedures, one of them the electronic control systems which enable condition stability for the steady state conditions during the experiment. Transient methods allow fast measurement and reduce unwanted processes of heat transfer. Most nanofluids thermal properties were measured using transient techniques of measurement. The thermal conductivity is one of the most important thermal properties, which affect directly on the thermal behavior of the heat transfer machines. In this study the thermal conductivity was measured using the transient hot wire method (Jwo C. & Teng T., 2005).

Transient Hot-Wire (THW) Method:-

The Transient Hot-Wire (THW) method is considered one of the simplest and most accurate techniques used to measure the fluid thermal conductivity. A THW arrangement includes a metal wire submerged symmetrically in a cylindrical cell, which is filled with the test fluid. The metal wire is used as a heating source, so the wire should have a high electrical resistance (see Fig.1.a).



Fig.1:-a) THW schematic view. b) Transient Hot-wire system used for thermal conductivity measurements.

The THW technique depends on recognition of the transient temperature ascend in a thin metal wire in a submerged in the test fluid. The ideal theoretical model of the (THW) technique assumes that the hot wire radius (r) is so small $r \rightarrow 0$ and the wire length (l) is too long $l \rightarrow \infty$, and this hot wire is immersed in an infinite homogeneous isotropic fluid with constant initial temperature. If the heat flux q is a constant value of heat production per unit length of the wire and per unit time (W.m⁻¹), started at the time t=0, the radial heat flow happens around the wire. Then the temperature rise formula ΔT (r,t) at a radial position r from the heat source is shown in Eq.1. (Nagasaka Y. & Nagashima A., 1981). Where

$$\Delta T(r,t) = \frac{q}{4\pi k} \ln \frac{4at}{r^2 c} \tag{1}$$

k: the thermal conductivity (W.m⁻¹.K⁻¹), *a*: thermal diffusivity (m².s⁻¹), *C*: the exponential of the Euler's constant = $e^{0.57721}$

, and by plotting the temperature T(r, t) with time t on a logarithmic scale, the resulted slope (s) of the graph will be linear $s = (q/4\pi k) = d (\Delta T)/d (ln(t))$ so the thermal conductivity can be calculated from this formula:

$$k = \frac{q}{4\pi s} = \frac{q \cdot d(\ln(t))}{4\pi \cdot d(\Delta T)}$$
(2)

Therefore, if the fluid temperature is measured as a function of time at any certain radial situation, the fluid thermal conductivity k, is proportional to the wire heat flux and inversely proportional to the temperature difference gradient with regard to the natural logarithm of time, see Eq. (2).

The THW system consists of a thin metal wire made of Ni-Cr alloy with diameter (\emptyset) 50 µm and 2 cm length (l). This Ni-Cr wire is immersed in a cylindrical cell made of teflon with volume about 50 ml, filled with the test fluid as shown in Fig.1.a. The Ni-Cr wire was soldered with tin at each end to be connected to 1 mm \emptyset copper wire.

Since the resistance of the Ni-Cr wire is so higher than the resistance of the copper wire and the two tin soldered end points of the Ni-Cr wire, only the Ni-Cr wire resistance was taken into account (R_{HW} \approx R_{total}). The Ni-Cr wire is heated by an electric power supply (EZ Digital /Model: GP-4303D). The K type thermocouple is immersed in the fluid cell in touch with the Ni-Cr wire to measure its temperature change. The thermocouple is connected to data logger (Pico/Model: TC-08) to record the temperature variation with time (see Fig.1.b), and these data were analyzed by Pico data logger software on the PC. In order to get the thermal conductivity, the temperature rise with time data was recorded by the data logger while heating the Ni-Cr wire with a constant current. The heating power can be calculated from the heating current and voltage of the power supply.

The slope of the temperature versus natural logarithmic of the time is directly inversely proportional to the thermal conductivity (see Eq. 2). In order to find the thermal conductivity, the THW setup should be calibrated using materials with known thermal conductivity. At the beginning, ethanol and acetone were tested on the THW setup to assure about the measured thermal conductivity results is dependable or not. The measured thermal conductivity of the Ethanol is 0.175 W/m.K and the theoretical value is 0.171 W/m.K with a deviation about 2% it and the measured thermal conductivity was calculated using the recorded data from the THW system and compared with the theoretical value .Finally; the THW system is trusted and gives a good accuracy with about 2% deviation between theoretical and experimental values of the thermal conductivity.

Results and Discussion:-

Characterization of the Alumina Powder:-

Alumina powder was analyzed by the XRD (X-Ray Diffractometer) for phase identification, crystallite size and determination of the crystal structure. The XRD spectra for alumina has been performed by the X-ray diffraction system (X'Pert-Pro PANalytical's). The X-Ray Diffractometer has a copper anode with radiation ($\lambda = 1.54$ Å), and the diffractometer operated in the $\theta/2\theta$ mode primarily in the 30-90° (2 θ) range with a step scan $\Delta 2\theta = 0.05^{\circ}$. The average particle size has been determined from the XRD peaks, employing Scherrer's equation:

$$D = \frac{\kappa \lambda}{\beta \cos \theta} \tag{3}$$

Where *D* is the crystallite size (nm), *K* is the Scherrer constant (=0.94), θ is the Bragg angle (rad), λ is the wavelength of X-ray radiation of the copper anode (0.154 nm) and β is the full width at half maximum (FWHM) of the most intense diffraction peaks (rad). The XRD pattern of the Al₂O₃ particles is shown in Fig.2.



The XRD pattern shows the formation of a single phase of Al_2O_3 nanostructure (cubic structure) with characteristic peaks at diffraction angles (2 θ) equal 19.52°, 32.75°, 37.802°, 39.519°, 42.288°, 45.672°, 47.976°, 55.571°, 60.848° and 66.79°.

In the XRD pattern, the diffraction angles and relative intensity of the characteristic peaks are well consistent with those of the standard JCPDS 010-070-9085. the XRD spectra results states that the crystal shape of the alumina is the cubic shape, and the average alumina crystal size was about ≈ 8 nm, which it was calculated using the Scherrer's equation at the greatest peak. The XRD results states that the prepared alumina powder is a single phase material (Al₂O₃) with a cubic crystal size in nano-scale. So it was confirmed that the prepared alumina particles were nanoparticles, but the XRD pattern didn't state any exact information about the sample surface morphology. Further the sample needs to be characterized by Transmission electron microscopy (TEM) to get the exact particle sizes and morphology.

Transmission Electron Microscopy (TEM) Analysis :-

Transmission Electron Microscopy (TEM) is utilized to get the size, dispersion and the morphology of the synthesized nanoparticles. In the TEM measurements, The Al_2O_3 sample was prepared by adding 0.1 gm of a the Al_2O_3 nanopowder to 10 ml of DDW and mixing them well using ultrasonic probe for 15 minute, then a drop of the mixed sample has been set on a Formvar covered Cu grid and dried at room temperature. A TEM instrument (JEOL /Model: 100CXII) has been used for TEM scanning. The TEM test has been done by scanning the prepared drop sample and taking some proper pictures of this sample. These pictures show different particle sizes in the sample with the scanning scale (see Fig.3). The TEM result picture was analyzed to get the particle size distribution. The ImageJ software is one of the easiest facilities used to analyze this type of pictures to get the particle size distribution.



Fig.3:- TEM micrograph of Al₂O₃ nanostructures

The selected picture was analyzed by the ImageJ program (image processing software used for the scientific pictures analysis) and the analysis result was presented in the particle sizes histogram (Fig.4). It is noted that the particle sizes in the sample varied from (4nm: 18 nm), and (8nm:12nm) particle sizes were the most counting of the alumina particle size in the histogram. The imageJ analysis result has a good agreement with the average calculated particle size from XRD results.



Fig.4:- Particle size distribution of Al₂O₃ nanoparticles powder

The thermal conductivity (TC) Results:-

In the thermal conductivity (*TC*) measurement procedures, the WBA nanofluids samples (each sample vol.=50ml) were prepared with different alumina weight concentrations (0.2%, 0.4%, 0.6%, 0.8%, 1%, and 1.2% wt.). Each sample was tested into the THW cell. A current of 0.33A and a voltage of 10V were applied from the power supply. The data logger record the variation in the temperature at each one second during the sample temperature changed from *T*= 25 °C up to 41 °C. The *TC* of samples was calculated by Eq.2. using the recorded data by the logger. These results were plotted in Fig.5 and Fig.6. It is noted that the *TC* of the alumina nanofluids increases with increasing in

the sample temperature, as well as with increase in Al_2O_3 weight concentration in the nanofluid (see Fig.5.). Also the TC of WBA nanofluid is bigger than the base fluid thermal conductivity.



Fig.5:- Thermal conductivity of the WBA nanofluid at different weight concentrations% of Al₂O₃ versus sample temperature

Adding of the alumina nanoparticles with average sizes (8nm: 12nm) to the base fluid enhances the thermal conductivity of it by about 11% (see Fig.7) in the room temperature at 1.2% wt., and this enhancement increase with the temperature rising till it reaches to about 25% at Temperature = 41° C (as shown in Fig. 6.)



Fig. 6:- Thermal conductivity enhancement $((K_{nf}-K_{bf})/K_{bf})$ of WBA nanofluid at different weight concentration of Al_2O_3 and temperature.



Fig.7:-Thermal conductivity enhancement ($(K_{nf} K_{bf})/K_{bf}$) % of WBA nanofluid vs. different alumina weight fraction% at Temperature 25 ${}^{0}C$

Viscosity Measurements:-

Viscosity is another critical property of nanofluids for every thermal application including fluid flows since it describes the inertial resistance when fluid streams in the thermal system. Quantitative data of nanofluids viscosity is fundamental for fluid and heat transfer applications to generate the suitable pumping power as well as the convective heat transfer coefficient (Kole M. & Dey T.K., 2010). It is approved that viscosity is important as thermal conductivity in industrial systems because the nanofluid enhances the thermal conductivity without pressure drop, so that is affected by fluid viscosity. The Cone/Plate Viscometer (Gardco /Model: CPA-42Z) was used to nanofluids viscosity measurements. The viscometer has an accurate torque meter which is driven at separated rotational speeds. The measuring system of torque has a calibrated spring which connecting the drive mechanism to a rotating cone, this spring senses the rotation resistance caused by the sample fluid. This resistance produces a proportional torque to the shear stress of nanofluids viscosity results are shown in Fig.8. The WBA nanofluid viscosity increases with increasing of the alumina weight concentration in the nanofluid, although it decreases with the sample temperature rising (see Fig.8).



Fig.8:- influence of temperature on the viscosity of WBA nanofluid at different weight concentrations %

Electrical conductivity (EC) measurement:-

Since, the major research field is focusing on measuring the thermal and physical properties of the nanofluids especially the thermal conductivity, some little studies concentrate on studying the electrical conductivity of the nanofluid and its importance. So this study will concern on studying the electrical conductivity of the prepared nanofluids for high voltage applications, especially the applications which use the DDW as a heat transfer fluid because of its low electrical conductivity. The main advantage of DDW using is the electric insulation, which reduces sparks chance in the high voltage applications heat transfer systems. So it is necessary to study the effect of the nanoparticles addition to the heat transfer fluid on the fluid electrical conductivity, and then the alumina particles suspension in the base fluid may have an effect on the electrical conductivity of the prepared nanofluids. The fluid electrical conductivity is measured by estimation the nanofluid resistance between two flat electrodes separated by a fixed distance (Azimi H. R. & Taheri R., 2015). Since, the electrical conductivity of the DDW is very low, and the prepared nanofluid majority is of the DDW, so it is expected that the prepared nanofluids EC is low also. For accurate measuring of the electrical conductivity, the EC measuring setup was installed. The setup consists mainly of two flat electrodes immersed in a beaker filled with the prepared nanofluid sample, and the electrodes connected the DC power supply and standard resistance $(S.R) = (10M\Omega)$ (see Fig.9.a & Fig.9.b.) A multi-meter (BK PERCISION/ Model: 5492) was used to measure the voltages, then the sample resistance can be calculated from Eq.4.



Fig.9:- a) Schematic of the EC circuit diagram b) The EC measuring setup

$$R_{sample} = \frac{V_{sample}}{I_{sample}} = \frac{(V_{total} - V_{S,R})}{(V_{S,R}/R_{S,R})}$$
(4)

Since, the electrical conductivity is the inverse of the resistance, so the samples *EC* can be calculated from $(EC_{sample}=1 / R_{sample})$. A hot plate was used to heat the nanofluids to measure the *EC* at different temperatures, and a digital thermometer (BK PERCISION/model: 710) recorded the sample temperature during EC measurements. The WBA nanofluids electrical conductivities were measured at different temperatures and different alumina weight concentrations, and the results was plotted in the graph of Fig.10.



Fig.10:- Influence of temperature on the EC of WBA nanofluid at different weight concentrations% of Al₂O₃

It is noted that the electrical conductivity of the WBA nanofluid increases with increasing of the alumina weight concentrations (0.2%, 0.4%, 0.6%, 0.8%, 1%, and 1.2% wt.), as well as with increasing of the nanofluid temperature. Although, the WBA nanofluid EC is more than the base fluid EC, but it is still low in μ s/cm unit range and close to the distilled water EC values.

Conclusion:-

This study was performed to research the availability of improving of the high voltage applications cooling by using the nanofluids as a heat transfer fluid, which can offer good thermal properties. The challenge was preparing a nanofluid which has a higher thermal conductivity with a low electrical conductivity, this advantage was found in the nanofluids prepared by the metal oxides such as the Al_2O_3 .

- The water-base Al₂O₃ nanofluids was chosen to be tested as a nanofluid for the high voltage parts cooling system in the MGC-20 cyclotron
- > The Al₂O₃ was fabricated by the sol-gel technique, and it was inspected by the XRD and the TEM
- ➤ The alumina XRD and TEM analysis indicated that the Al₂O₃particles were in the nano-scale with cubic crystal shapes in average sizes ≈ 8nm: 12 nm
- The water-base Al₂O₃ nanofluids was prepared by the two-step methods, which consisted of 90% DDW, 10% oleic acid and different small weight percentages of alumina (0.2%, 0.4%, 0.6%, 0.8%, 1%, and 1.2% wt.) for producing stable nanofluids and reducing nanoparticles agglomeration.
- > The thermal conductivity of the prepared WBA nanofluids increased with increasing of Al_2O_3 weight concentration% as well as with temperature increasing, which offers about 11% enhancement in *TC* at room temperature and about 25% enhancement in *TC* at T=41°C for 1.2% wt. of Al_2O_3
- > The WBA nanofluids viscosity increased with increasing of Al_2O_3 weight concentration % but it decreased with the temperature increasing.
- > The *EC* of the WBA nanofluid increased with increasing of the Al_2O_3 weight concentration %, and also with increasing of the temperature. Although, the *EC* of the WBA increased by 133% of the base fluid *EC* at the room temperature, but it is still low and safe for the high voltage applications.

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