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Synthesis and Surface Area Determination of Alumina Nanoparticles by Chemical Combustion Method

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Abstract: In this present work a new technique employed for synthesing Al_2O_3 nanoparticles using chemical combustion method. Apart from adsorption method, BET-adsorption Isotherm process is used to find the surface area of particle size. The surface area of nano particle is arrived by Bet (Brunauer, Paul Hugh Emmett and Edward Teller) by physical adsorption of gas molecules on solid surface for evaluation of surface area of particle per unit weight of nano particles.
Keywords: Alumina nanoparticles.BET curve, Chemical Combustion Method, Adsorption Method, Sorptomatic Instrument.

1. Introduction

Materials for base fluids and nanoparticles are diverse. Stable and highly conductive nanofluids are produced by one and two-step production methods. Both approaches to creating nanoparticles suspensions suffer from agglomeration of nanoparticles, which is a key issue in all technology involving nanopowders. Therefore, synthesis and suspension of nearly nonaggloromerated or monodispersed nanoparticles in liquids is the key to significant enhancement in the thermal properties of nanofluids. Nanostructure or nanophase materials are made up of nanometer-sized substances engineered on the atomic or molecular scale to produce either new or enhanced physical properties not exhibited by conventional bulk solids. The noble properties of nanophase materials come from relatively high surface area/volume ratio, which is due to the high proportion of constituent atoms residing at the grain boundaries. Nanoparticles used in nanofluids have been maybe of various materials, such as oxide ceramics (Al_2O_3 and CuO), nitride ceramics (AIN, SiN), metals (Cu, Ag, Au), Semiconductors (Tio_2, SiC), carbon nanotubes and composites materials such as alloyed nanoparticles or nanoparticles core-polymer shell composites. In addition to nonmetallic, metallic and other materials for nanoparticles, completely new materials and structures, such as materials doped with molecules in their solid-liquid interface structure, may also have desirable characteristics. Akoh et al. [1] devopled a promising technique for producing nonagglomerating nanoparticles involves condensing nanophase powders from the vapour phase directly into flowing low-vapour-pressure fliud.Eastmanet.al [2] developed direct evaporation system that overcomes the difficulties of making stable and well dispersed nanofluids. Zhu et al [3] developed one step chemical method for producing stable Cu-in–ethylene glycol nanofluids by reducing copper sulfate pentalhydrate with sodium hypophosphite in ethylelne glycol under microwave irradiation. They claim that this one-step method chemical method is faster and cheaper than one step physical method .The thermal conductivity enhancement approaches that of Cu nanofluids prepared by one-step physical method developed by Eastman et.al[4]. For thermal conductivity containing high conductivity metals, it is clear that the single-step technique is preferable to the two-step method. The first-ever nanofluids with carbon, nanotubes-in -synthetic oil(PAOs) were produced by a two-step method by Chopkar.et al.[5] Multiwall carbon nanotubes (MWCNTs) were produced in a CVD reactor with xylene as the primary carbon source and ferrocene to provide the iron catalyst. Chon et al.[6] Used ball milling to produce $Al_{20}Cu_{30}$ nanoparticles and dispersed their alloyed nanoparticles in ethylene glycol. Gransqvist and Buhrman [7] developed a physical methods include inert-gas condensation (IGC) and mechanical grinding. Suslick et al. [8] a sonochemical method has been developed to make suspensions of iron nanoparticles stabilized by oleic acid. Murshed et al [9] used oleic acid and cetyl trimethyl ammonium bromide (CTAB) surfactants to ensure proper dispersion and better stability of TiO_2-water nanofluids. Hwang et al [10] used sodium dodecyl sulfate (SDS) during the preparation of water-based multiwall carbon nanotubes dispersed nanofluids, since the fibers are entangled in the aqueous suspension. However, the heat transfer performance of the nanofluids can be affected by the addition of dispersions in fluids, especially at high temperature i.e. in the convective heat transfer and two-phase heat transfer regime. Wang et .al [11]investigated Al_2O_3 and CuO nanopowder through inert gas condensation process, because they were easy to produce and chemically stable in solution that produced 2-200 nm sized particles. The major limitation in this method is its tendency to agglomerate and its unsuitability to produce pure metallic nanopowder. But it can overcome the agglomeration by using a direct evaporation condensation method. Keblinski et al.[12] proposed four possible microscopic mechanisms for the anomalous increase in the thermal conductivity of nanofluids ,which include Brownian motion of the particles, molecular-level layering of the liquid at the liquid particle interface, the ballistic rather than the diffusive nature of heat conduction in the nanoparticles ,and the effects of nanoparticles clustering. Mohammad Ali Karimi et al. [13] was intended to use a new and easy method for preparation of homogeneous porous nano-sized ZnO and MgO particles. For this purpose, single frequency ultrasonic waves were applied to prevent growth and help formation of nanoparticles. In addition, polyvinyl pyrrolidone (PVP) was used as a structure director additive. This is the first report on application of ZnO and MgO nano-powders and ZnO/MgO nanocompositeto the preparation of zinc polycarboxylate dental cement. The mechanical strength property of this cement is more than two commercially available polycarboxylate cements.Sanjay Srivastava et al.[14]synthesized CuO nanoparticles using a sol gel combustion route. XRD spectra confirmed the formation of single phase CuO nanoparticles. Crystallite size was found to increase with the increase in annealing temperature. Minimum crystallite size of 16 ± 1.26 nm was observed in the case of CuO nanoparticles annealed at 300°C. TEM results corroborate well with XRD results. FTIR spectra also validated the purity of CuO nanoparticles. In this paper a new method is employed for synthesis. The method adopted because of its capability to produce particles of very low size involving least expenditure.

2. Need of Nanofluid

The conventional way to enhance heat transfer in thermal systems is to increase the heat transfer surface area of cooling devices and the flow velocity or to disperse solid particles in heat transfer fluids. However a new approach to enhancing heat transfer to meet the cooling challenge is necessary because of the increasing need for more efficient heat transfer fluids in many industries. The major problems with suspensions containing millimeter- or micrometer-sized particles are the rapid settling of these particles. If the fluid is kept circulating to prevent particle settling, millimeter- or micrometer-sized particles would wear out pipes, pumps, and bearings. Furthermore, such particles are not applicable to Microsystems because they can clog micro channels. These conventional solid fluid suspensions are not practical because they require the addition of a large number of particles (usually,>10 vol %), resulting in significantly greater pressure drop and pumping power. Heat transfer is one of the most important processes in many industrial and consumer products. The inherently poor thermal conductivity of conventional fluids puts a fundamental limit on heat transfer. Therefore, for more than a century since Maxwell (1873), scientists and engineers have made great efforts to break this fundamental limit by dispersing millimeter- or micrometer-sized particles in liquids. The concept and emergence of nanofluid is related directly to trends in miniaturization and nanotechnology. Maxwell's concept is old, but what is new and innovative in the concept of nanofluid is the idea that particle size is of primary importance in developing stable and highly conductive nanofluid. This conventional approach has two major technical problems*:* (1) conventional millimeter- or micrometer-sized particles settle rapidly in fluids, and (2) the conductivities of these suspensions are low at low particle concentrations. Furthermore, these conventional suspensions do not work with the emerging "miniaturized" devices because they can clog the tiny channels of such devices. Modern nanotechnology has enabled the production of metallic or non-metallic nanoparticles with average crystallite sizes below 100 nm. The mechanical, optical, electrical, magnetic, and thermal properties of nanoparticles are superior to those of conventional bulk materials with coarse grain structures. Recognizing an excellent opportunity to apply nanotechnology to thermal engineering, Choi conceived the novel concept of nanofluid by hypothesizing that it is possible to break down these century-old technical barriers by exploiting the unique properties of nanoparticles. Nanofluids are a new class of nanotechnology-based heat transfer fluids engineered by dispersing nanometre-sized particles with typical length scales on the order of 1 to 100 nm (preferably, smaller than 10 nm in diameter) in traditional heat transfer fluids.

3. Experimental

3.1methodlogy for Synthesis Procedure

The required stoichiometric quantity of Urea (Fuel) and nitrate in the molar ratio of 1:1 is measured with the help of Physical balance.

Table 1.Raw materials proportion

Nano-particle	Fuel	Salt	Molar Ratio	Colour of salt
Alumina	Urea	Aluminium Nitrate	1:1	white

Prepared salt mixture is dissolved in 100 ml distilled water to get homogeneous solution. The solution is kept in hot oven at 90^{o}C to remove all the water in the solution gets evaporated. The resultant paste like masse is removed from the container and transferred in to ceramic crucible.

3.2. Drying Of Mass:

The wetted mass is dried in a muffle furnace at 200^{o}C to ensure the complete removal of moisture. This process takes place at about 1-1.5 hours in hot oven. The final mixture is taken out and tested for moisture content and colour change.

Fig.1. Muffle Furnance

3.3 Reaction Zone:

The prepared dried mass is then transferred into another ceramic crucible and kept in Muffle furnace at 500 ^{o}C. At this temperature, the entire nitrate is evaporated and the fuel supplied is sufficient for combustion reaction at this temperature, Nitrate salt becomes oxide at this temperature, the resulting complex mixture contains only oxides of metals in nano meter size. In this method, there is no controlling of size. For better particle size distribution of nano particle, the molar ratio of Nitrate and salt can be increased. The size is controlled by reaction zone temperature. But, due to economical consideration, the reaction temperature is limited to 500^{o}C. Under this operating condition and for the same molar ratio of different nitrate salt and fuel, different particle size is arrived and tabulated as given below

Table 2. Nano particles product size

Products	Raw Material	Fuel	Reaction Temperature (^{o}C)	Molar Ratio	Colour of the product
Alumina	Aluminium Nitrate	Urea	500	1:1	Pale yellow

The prepared chemicals nano particles are dried to ambient condition and mixed in the required volume fraction with distilled water. The product solution is so called Nano- fluid and then given for Ultrasonic treatment for better suspension of particle in base fluid

3.4. Synthesis of Nanoparticles:

In the present study chemical combustion method was adopted for synthesis of nanoparticles because of its capability to produce particles of very low size involving least expenditure. The procedure employed for synthesis is listed below

1. Stoichiometric quantity of raw material in the molar ratio of 1:1 (Salts of nitrate : Urea) was dissolved in distilled water to get a homogeneous solution
2. The solution was dried in a furnace and heated to a temperature of 110^{o}C so as to remove all the volatiles / moisture present in it.
3. The resulting paste was heated in a muffle furnace between 250-500^{o}C depending on the particle size requirement.
4. Particle size mainly depends on the molar ratio of raw material and operating temperature. As the temperature increases the surface area per unit volume increases and particle size reduces.
5. The generated powder was then annealed to ambient condition very slowly
6. The annealed powder was dissolved in distilled water based on the requirement of volume fraction and was then subjected to ultrasonification to get continuous dispersion of nano-particle in base fluid.
7. The synthesized nanofluid was tested for settling in a settling chamber for 10 days.
8. If settling was observed, as in the case of CuO, surfactant was added to improve better suspension of nanoparticles.

Finally magnetic stirring was carried out to avoid agglomeration before loading the nano particles in to the collector.

3.5. Characterisation of Nano Particle

Apart from adsorption method, BET-adsorption Isotherm method is used to find the surface area of particle size. An experiment was conducted by using Sorptomatic Instrument (fig.2) to measure the surface area by using suitable gas absorbing agent. N_2 gas is used as adsorbent based on the particle pressure in vapour phase and vapour pressure in solid phase determines density value at isothermal condition (77k) from1mm to 760mm of Hg. From the surface area the particle size is calculated by using the Sphericity method for spherical particle.

4. Results and Discussions

4.1. Evaluation of Surface Area of Nano Particle

The surface area of nano particle is arrived by BET (BRUNAUER, PAUL HUGH EMMETT AND EDWARD TELLER) by physical adsorption of gas molecules on solid surface for evaluation of surface area of particle per unit weight of nano particle. This is done by Sorptomatic Instrument.

Fig.2. Sorptomatic Instrument

The concept of the theory is an extension of the Langmuir theory, which is a theory for monolayer molecular adsorption, to multilayer adsorption with the following hypotheses: (a) gas molecules physically adsorb on a solid in layers infinitely; (b) there is no interaction between each adsorption layer; and (c) the Langmuir theory can be applied to each layer. The resulting **BET equation** is expressed

$$\frac{1}{V\left\{\frac{P_o}{P}-1\right\}} = \frac{C-1}{V_m C}\left[\frac{P}{P_o}\right] + \left[\frac{1}{V_m C}\right] \qquad (1)$$

A straight line plotted with $1 / \{V [P_0 / P - 1]\}$ on (Y –axis) (2) and P/Po on the x-axis.

This plot is called a **BET plot**.

A total surface area and a specific surface area evaluated by the following equations

$$S = V_m N_s / V \qquad (2)$$

The required stoichiometric quantity of Urea (Fuel) and nitrate in the molar ratio of 1:1 is measured with the help of Physical balance. Since the collector area ($0.5m^2$) is small so, a lesser quantity nano particle is required for synthesisation hence, we employ chemical combustion method for preparing the nanoparticles and also it is very economical and high purity of nano particle is obtained which is difficult in other synthesis process. The prepared chemicals/nano particles are dried to ambient condition and mixed in the required volume fraction with distilled water.

Table.3 Preparation of Nanoparticles

Products	**Raw Material**	**Fuel**	**Reaction Temperature (°C)**	**Molar Ratio**	**Product Size (nm)**	**Colour of the product**
Alumina	Aluminum Nitrate	Urea	500	1:1	40	Pale yellow

4.2. Surface Area Determination

From the analysis, The BET curve for Alumina has been prepared with the help of Sorptomatic Instrument. The Fig. (3.1 & 3.2) shows the Adsorption Isotherms and BET Line for Al_2O_3 Nanoparticles.

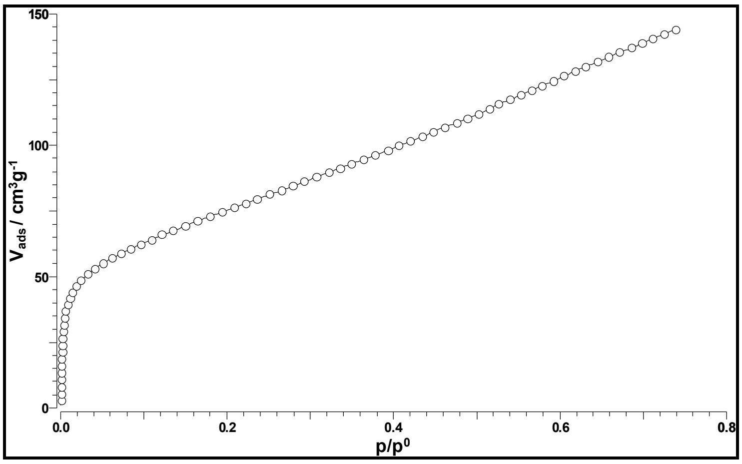

Fig.3 Adsorption Isotherms Al_2O_3

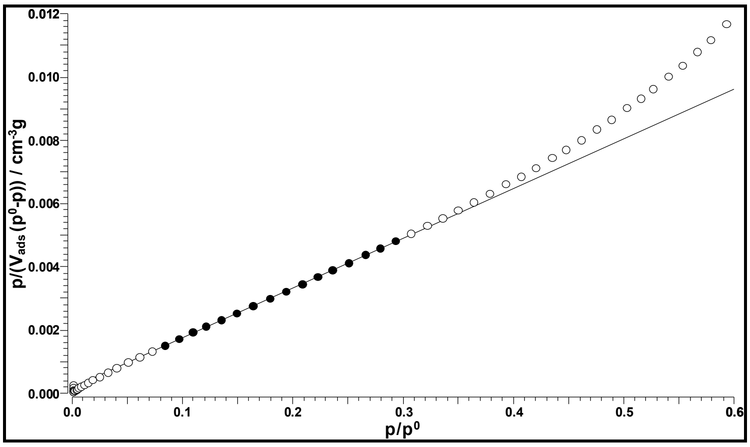

Fig.4 Al_2O_3 BET Line

From the result it was noted that, Surface area of Alumina nano particle is 378.78 m^2/gm. SEM images samples are shown in the Fig.3.3.

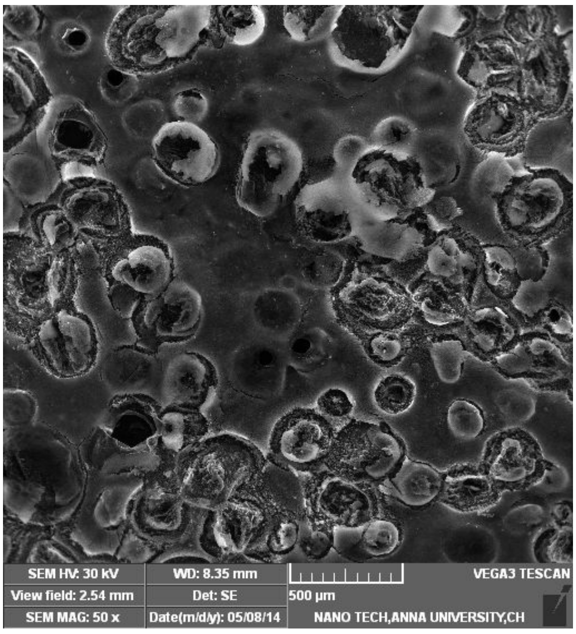

Fig.5.SEM image of Al_2O_3

5. Conclusion

Aluminium Oxide nanoparticles were successfully prepared by synthesizing using chemical combustion method .The surface area of alumina nanoparticles by BET method is 274.01(m^2/gm) and its particle size is 5.52nm.

6. References

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