

Experimental Investigation for the Determination of Characteristics of Nano Particles Using Nano Particle Analyser

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ABSTRACT

Many investigations are being performed for usage of Nano particles in shell and tube heat exchanger for obtaining better results, as a part of determining the characteristics of Nano particles we need to know the size and thermal properties of the Nano particles. In this paper, we are determining the size and thermal properties such as conductivity, viscosity, electrode voltage, zeta potential for copper oxide and aluminum oxide using Horiba Nano Partica SZ -100 Nano particle analyzer. In this experiment 01mg of both the samples are mixed with 10ml of deionized water each and then it is placed in ultrasonic cleaning equipment for an hour and then tested in the Nano particle analyzer for determining the mentioned properties.

Keywords: Nano Particles , Horiba Nano Partica SZ -100 , Ultrasonic Cleaner

I. INTRODUCTION

Nanoparticles are particles between 1 and 100 nanometers in size. In nanotechnology, a particle is defined as a small object that behaves as a whole unit with respect to its transport and properties. Particles are further classified according to diameter Ultrafine particles are the same as nanoparticles and between 1 and 100 nanometers in size, fine particles are sized between 100 and 2,500 nanometers, and coarse particles cover a range between 2,500 and 10,000 nanometers. Scientific research on nanoparticles is intense as they have many potential applications in medicine, physics, optics, and electronics.

A. Definition of Nano Particles:

The term "nano particle" is not usually applied to individual molecules; it usually refers to inorganic materials. A nano particle is defined as the smallest unit that can still behave as a whole entity in terms of properties and transport. In terms of diameter, fine particles cover a range between 100 and 2500 nanometers,[1] while ultrafine particles are sized between 1 and 100 nanometers. Nanoparticles may or may not exhibit size-related properties that are seen in

fine particles. Despite being the size of the ultrafine particles individual molecules are usually not referred to as nanoparticles. Nanoclusters have at least one dimension between 1 and 10 nanometers and a narrow size distribution. Nano powders on the other hand are agglomerates of ultrafine particles, nanoparticles, or nanoclusters. Nano particle sized crystals are called nanocrystals.

II. HISTORY OF NANO PARTICLES

Although generally nanoparticles are considered an invention of modern science, they actually have a very long history. Specifically, nanoparticles were used by artisans as far back as the ninth century Mesopotamia for generating a glittering effect on the surface of pot.[2][3 Even these days, pottery from the middle ages and renaissance often retains a distinct gold or copper colored metallic glitter. This so called luster is caused by a metallic film that was applied to the transparent surface of a glazing. The luster can still be visible if the film has resisted atmospheric oxidation and other weathering. The luster originates within the film itself, which contains silver and copper nanoparticles, dispersed homogeneously in the glassy matrix of the ceramic glaze. These nano particles were created by the

artisans by adding copper and silver salts and oxides, together with vinegar, ochre, and clay on the surface of previously-glazed pottery.[3] The object was then placed to a kiln and heated to about 600°C in a reducing atmosphere. In the heat the glaze would soften, causing the copper and silver ions to migrate into the outer layers of the glaze. There the reducing atmosphere reduced the ions back to metals, which then came together forming the nano particles that give the color and optical effects. Luster technique shows that craftsmen had a rather sophisticated empirical knowledge of materials. The technique originates in the Islamic world. As Muslims were not allowed to use gold in artistic representations, they had to find a way to create a similar effect without using real gold. The solution they found was to use luster. Michael Faraday provided the first description, in scientific terms, of the optical properties of nanometer-scale metals in his classic 1857 paper[4] "Experimental relations of gold (and other metals) to light." Much of the modern day studies of these objects have been conducted at the ESRF laboratory. Several techniques were used to characterize the chemical and physical properties of these lusters, such as Rutherford Backscattering Spectrometry (RBS), optical absorption in the visible ultra violet region, electron microscopy (TEM and SEM).[5]

III. PROPERTIES OF NANO PARTICLES:

Nanoparticles are of great scientific interest as they are, in effect, a bridge between bulk materials and atomic or molecular structures.[6] A bulk material should have constant physical properties regardless of its size, but at the nano-scale size-dependent properties are often observed. Thus, the properties of materials change as their size approaches the nanoscale and as the percentage of the surface in relation to the percentage of the volume of a material becomes significant. For bulk materials larger than one micrometer (or micron), the percentage of the surface is insignificant in relation to the volume in the bulk of the material. The interesting and sometimes unexpected properties[7] of nanoparticles are therefore largely due to the large surface area of the material, which dominates the contributions made by the small bulk of the material.[8]

IV. EXPERIMENTAL PROCEDURE

In this experiment the following process is followed for the determination of the size and thermal properties such as conductivity, viscosity, electrode voltage, zeta potential.

- A. Nano Particles Selection
- B. Ultra-Sonic Cleaner
- C. Nano particle analyser Testing

A. Nano Particles Selection

The nano particles of copper oxide and aluminium oxide that are selected for using in the shell and tube heat exchanger are tested on the Nano Particle for determination of size and different Properties. The Properties of the both materials are shown below

- Copper Oxide



Figure 1. Copper oxide powder

| | |
|----------------------|------------------------|
| Appearance | Black colour |
| Molecular Formula | CuO |
| Molecular Mass | 79.55gm/mole |
| Odour | Odourless |
| Density | 6.31gm/cm ³ |
| Melting point | 1201 ⁰ c |
| Solubility in water | Soluble |
| Thermal conductivity | 76W/mk |

Table 1. Properties of Copper oxide

- Aluminium Oxide



Figure 2. Aluminium Powder

| | |
|----------------------|--------------------------------|
| Appearance | White powder |
| Molecular Formula | Al ₂ O ₃ |
| Molar Mass | 101.96 g/mol |
| Odour | Odourless |
| Density | 3.95g/cm ³ |
| Melting point | 2,072°C |
| Boiling Point | 2977°C |
| Specific heat | 880J/KgK |
| Thermal conductivity | 30 W/Mk |

Table 2. Properties of Al₂O₃

B. Ultra-Sonic Cleaner

- As the samples are in the powder form the testing on nano particle analyser cannot be directly carried out. To convert the Nano particles powder to [10] nano fluids, the Nano particles when mixed with deionized water.
- Nano particles of 0.1gm is mixed with 10ml of deionized water and is stirred well with magnetic stirrer



Figure 3. Sedimentation of CuO



Figure 4 Sedimentation of Al₂O₃

- The two solutions which are prepared are to be completely dissolved. To obtain the perfect nanofluid, [11] the prepared sample is kept in the ultra-sonic Cleaning bath for 60 mins to 120 mins. In this experiment we have used Spectralab ultrasonic cleaning bath Model UCB 30.

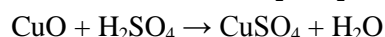
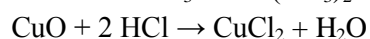
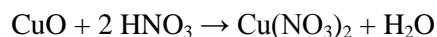


Figure 5. Ultra sonic equipment

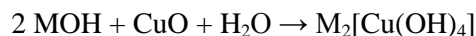
Chemical Reactions of Nano Materials With Deionized Water

- Copper oxide

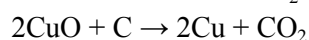
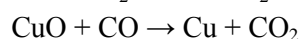
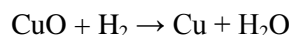
Copper(II) oxide is an amphoteric oxide, so it dissolves in mineral acids such as hydrochloric acid, sulphuric acid, or nitric acid to give the corresponding copper(II) salts:



It reacts with concentrated alkali to form the corresponding cuprate salts:



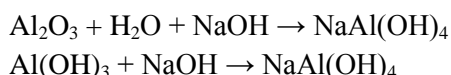
It can also be reduced to copper metal using hydrogen, carbon monoxide or carbon



When cupric oxide is substituted for iron oxide in thermite the resulting mixture is a low explosive, not an incendiary.

- Aluminium Oxide

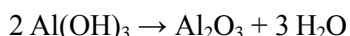
Aluminium hydroxide minerals are the main component of bauxite, the principal ore of aluminium. A mixture of the minerals comprise bauxite ore, including gibbsite ($\text{Al}(\text{OH})_3$), boehmite ($\gamma\text{-AlO}(\text{OH})$), and diaspore ($\alpha\text{-AlO}(\text{OH})$), along with impurities of iron oxides and hydroxides, quartz and clay minerals. Bauxites are found in laterites. Bauxite is purified by the Bayer process



Except for SiO_2 , the other components of bauxite do not dissolve in base. Upon filtering the basic mixture, Fe_2O_3 is removed. When the Bayer liquor is cooled, $\text{Al}(\text{OH})_3$ precipitates, leaving the silicates in solution.



The solid $\text{Al}(\text{OH})_3$ Gibbsite is then calcined (heated to over 1100C) to give aluminium oxide:



C. Nano particle analyser Testing

The prepared nano fluids of the samples are suitable for the testing on the nano particle analyser. In this experiment Horiba Nano Partica SZ -100 Nano particle analyser[12] is used for the testing. The SZ-100 Series measures particle diameter and particle distribution width, realizing this wide dynamic range by means of photon correlation spectroscopy. Analysis across a wide range of sample concentrations: Measurement of samples ranging from low ppm-order concentrations to high-concentration samples in double-digit percentages is possible. Accepts commercially available sampling cells. Analysis of small-volume samples is also possible.



Figure 6. Nano particle analyser

Key features and specifications of Horiba Nano Partica SZ -100 Nano particle analyzer

- Since the SZ-100 Series analyzer covers a wide sample concentration measurement range, sample dilution and other preprocessing[13] is nearly eliminated. The use of a dual optical system enables measurement of high-concentration samples such as slurry and ink pigments as well as low-concentration proteins and polymers.

- A single device analyzes the three parameters that characterize nanoparticles: particle diameter, zeta potential, and molecular weight

- HORIBA-developed disposable cells for zeta potential measurement prevent sample contamination. Simple analysis by means of ultra micro-volume dedicated cells (volume as low as 100µL). Suitable for analysis of dilute samples.

| | |
|---|--|
| Measuring unit optical system | Light source: Semiconductor laser excitation solid laser (532 nm, 10 mW) Detectors: Photomultiplier tubes (PMT) |
| Laser classification | Class 1 |
| Operating temperature and humidity | 15-35°C, RH85% or less (no condensation) |
| Holder temperature control temperature settings | 1-90°C (up to 70°C for cells with electrodes and plastic cells) |
| Purging | Nitrogen tube connection is possible. |
| Power supply | AC 100-240 V, 50/60 Hz, 1.50 VA |
| Dimensions | 385 (D) x 528 (W) x 273 (H) mm (excluding protrusions) |
| Weight | 25 kg |
| Personal computer | Windows XP or Vista compatible IBM PC/AT type computer |
| Interface | USB 2.0 (between measuring unit and PC) |
| OS | Windows® XP or Vista™ (Japanese Windows® XP or Vista™ required for Japanese-language display) |
| Printer | Windows® XP or Vista™ compatible printer |
| Operation method | Input using mouse and keyboard in the Windows® XP or Vista™ environment |

Figure 7. Key features of horiba

Testing Details

A Small amount of the nano fluid of the both the samples are taken into the small test holder and are placed in the nano particle analyser device. The test holder is then inserted in the device and the test is carried out. The required properties and size of the nano particles are obtained.



Fig 8 Sedimentation



Fig 9 inner view of particle analyser

The device is connected to the computer where the results and graph for the test are generated.



Figure 10. PC Interfacing Set up

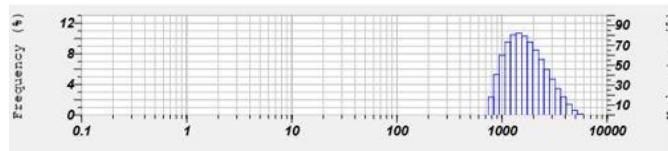


Figure 12. graph for frequency and nano particle size

IV. RESULTS AND DISCUSSION

A. Particle Size and Zeta testing results for Copper Oxide

| | |
|------------------------------------|----------------------------|
| Measurement Type | Particle Size |
| Sample Name | Cu-Size |
| Scattering Angle | 173 |
| Temperature of the holder | 25.0 deg.C |
| T% before meas. | 7812 |
| Viscosity of the dispersion medium | 2.035 mPa.s |
| Form of Distribution | Standard |
| Representation of result | Scattering Light Intensity |
| Count Rate | 1420 kCPS |

Table 3. Properties of CuO from nano particle analyser

| Peak No | S.P Ratio | Mean | S.D | Mode |
|---------|-----------|-----------|----------|-----------|
| 1 | 1.00 | 1835.6 nm | 834.7 nm | 1448.6 nm |

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Histogram Operations
Size (Median)      : 1616.1 nm
Mode               : 1448.6 nm
% Cumulative (1)  : 10.0 (%) - 982.0 (nm)
% Cumulative (2)  : 50.0 (%) - 1616.1 (nm)
% Cumulative (3)  : 90.0 (%) - 3032.7 (nm)
% Cumulative (4)  : 30.0 (%) - 1281.4 (nm)
% Cumulative (5)  : 40.0 (%) - 1438.7 (nm)
% Cumulative (6)  : 50.0 (%) - 1616.1 (nm)
% Cumulative (7)  : 20.0 (%) - 1133.6 (nm)
% Cumulative (8)  : 70.0 (%) - 2090.0 (nm)
% Cumulative (9)  : 95.0 (%) - 3549.6 (nm)
% Cumulative (10) : 100.0 (%) - 8510.6 (nm)
Cumulant Operations
Z-Average         : 1718.7 nm
PI                : 0.279
    
```

Figure 11. Calculations result

| | |
|------------------------------------|----------------|
| Measurement Type | Zeta Potential |
| Sample Name | Cu-zeta |
| Temperture of the holder | 25.0 deg.C |
| Viscosity of the dispersion medium | 0.895 mPa.s |
| Conductivity | 0.119 mS/cm |
| Electrode Voltage | 3.4 V |

Table 4. properties of CuO obtained from Zeta Potential measurement

| Peak No | Zeta Potential | Electrophoretic Mobility |
|---------|----------------|-------------------------------|
| 1 | -40.2 mV | -0.000311 cm ² /Vs |

B. Particle Size and Zeta testing results for Aluminium Oxide

| | |
|------------------------------------|----------------------------|
| Measurement Type | Particle Size |
| Sample Name | Al oxide -Size |
| Scattering Angle | 90 |
| Temperature of the holder | 25.0 deg.C |
| T% before meas. | 10769 |
| Viscosity of the dispersion medium | 2.036 mPa.s |
| Form of Distribution | Standard |
| Representation of result | Scattering Light Intensity |
| Count Rate | 2342 kCPS |

Table 5. Properties of Al₂O₃ from nano analyser

| Peak No | S.P Ratio | Mean | S.D | Mode |
|---------|-----------|----------|----------|----------|
| 1 | 0.65 | 220.5 nm | 57.9 nm | 184.3 nm |
| 2 | 0.35 | 676.7 nm | 196.8 nm | 619.7 nm |

Histogram Operations
 Size (Median) : 269.0 nm
 Mode : 184.3 nm
 % Cumulative (1) : 10.0 (%) - 164.1 (nm)
 % Cumulative (2) : 50.0 (%) - 259.0 (nm)
 % Cumulative (3) : 90.0 (%) - 782.9 (nm)
 % Cumulative (4) : 30.0 (%) - 202.4 (nm)
 % Cumulative (5) : 40.0 (%) - 225.5 (nm)
 % Cumulative (6) : 50.0 (%) - 259.0 (nm)
 % Cumulative (7) : 20.0 (%) - 183.0 (nm)
 % Cumulative (8) : 70.0 (%) - 466.1 (nm)
 % Cumulative (9) : 95.0 (%) - 910.0 (nm)
 % Cumulative (10) : 100.0 (%) - 8510.6 (nm)

Cumulant Operations
 Z-Average : 293.4 nm
 PI : 0.293

Figure 13. Calculations results

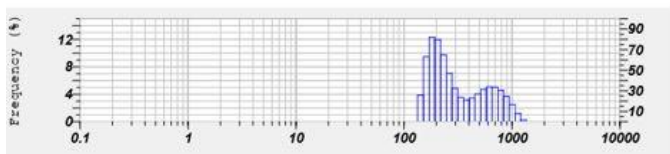


Figure 14. graph for frequency and nano particle size

| | |
|------------------------------------|----------------|
| Measurement Type | Zeta Potential |
| Sample Name | Al-oxide-zeta |
| Temperature of the holder | 25.0 deg.C |
| Viscosity of the dispersion medium | 0.892 mPa.s |
| Conductivity | 0.081 mS/cm |
| Electrode Voltage | 3.9 V |

Table 6. Properties of Al₂O₃ obtained from Zeta Potential measurement

| Peak No | Zeta Potential | Electrophoretic Mobility |
|---------|----------------|------------------------------|
| 1 | 6.0 mV | 0.000046 cm ² /Vs |

V.CONCLUSION

- The experiment proved that the both the materials are nano materials and the nano material sizes are determined.
- The zeta potential is found out for the both materials.
- The determination of nano particles will be helpful to determine the variation of the thermal conductivity with the size of the particles when used in the shell and tube heat exchanger.

VII. ACKNOWLEDGEMENTS

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