

Fabrication Al₂O₃ Nano Powder and study some of physical properties

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ABSTRACT

In this research we used Mie scattering method to analyzing the particle size of nano-powders that prepared by spray pyrolysis method , also to find out the effect of some important variables such as the vertical distance of spraying and the molarity on particle size of producer powder . As the use of this technology gives us the opportunity appropriate to detect the particle size. alumina powder was prepared from aluminum nitrate suspended in a chemical spray pyrolysis and in different molarity (0.001, 0.01 and 0,1 M) for each spray distance was chosen.

Some tests were conducted such as X-ray diffraction and scanning electron microscope examination in addition to particle size in a manner in a Mie scattering models and implementing the programs .

The results showed that the sizes of particles formed was 100, 120 and 150 nm approximately and SEM results showed that the particles homogeneous and irregularly shape.

Keyword Mie scattering, (CSP) chemical spray pyrolysis, nano powder, alumina, particle size, XRD, SEM, laser application, flow rate. Laser scattering angle , polarizer, He-Ne Laser, Laser Detector .

INTRODUCTION

Nanoparticles size are the key to many modern technologies and their applications are rapidly increasing, examples are pigments in paints and cosmetics, pharmaceuticals, self-cleaning surface coatings, dressed particles in high throughput genomic screening and advanced dispersion materials[1]. Optics are based on three phenomena taking place during a microscopic particle's interaction with light: refraction, reflection, and diffraction[2]. Mie-scattering ellipsometry is applicable to in situ analysis of nanoparticles under high gas pressures and in liquids[1]. MACKINNON ET AL.[3] used laser scattering and other method to calculate particle sizes for kaolinite Clay using Mie theory analyses of separated size fractions showed that in the <2µm range. G. GEBAUER and J WINTER [1] discuss the method of multiple-wavelength

Rayleigh– Mie scattering ellipsometry for the in situ analysis of nanoparticles of analysis of etching of melamine–formaldehyde nanoparticles suspended in oxygen plasma. Results demonstrating that the size distribution and the complex refractive index can be determined. SZYMANSKI and WAGNER [4] also used optical methods to measured concentration and size distribution and refractive index of airborne particle. BIERI ET AL . [5] study particle size to a liquid solvent is employed as the carrier of gold nanoparticles possessing a low melting temperature compared to that of bulk gold. KUSTOV[2] also study optical methods for microscopic particle size measurement. Al₂O₃ was used in many application, O'KEEFE ET AL. [6] study γ-Al₂O₃ and ZnO nano-powders oxides (to form ZnO-Al₂O₃) as nano-composites material to fabrication micro-electromechanical systems (MEMS) device .They find that by using a nanopowder, a 27% increase in theoretical density and conservation of nanoscaled features were obtained. STEINBORN ET AL. [7] study Particle size determination at different concentrated Al₂O₃ and SiO₂ nanopowder suspensions with the ultrasonic spectrometer at different percentage have mean value of 150 nm.

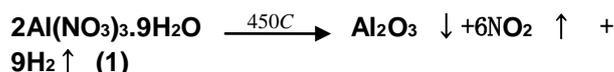
The aim of our research is to determine the particle size of Al₂O₃ powder prepared by spray pyrolysis using Laser Scattering and Mie Theory.

EXPERIMENTAL WORK

Experimental work includes powder preparation and Measurement.

Powder preparation

Chemical Spray Pyrolysis[13] (CSP) (the technique adopted in this study) involves spraying of a solution, usually aqueous, containing soluble salts of the constituent atoms of the desired compounds onto heated substrates. An aqueous solution of Aluminum nitrate [Al(NO₃)₃.9H₂O] has been used to prepare Al₂O₃ nanopowder. The concentrations that used was (0.001, 0.01 and 0.1M). The acidity was maintained to be ~6 pH during spraying.



The spraying apparatus used in this study was manufactured locally in the university laboratories with inexpensive equipment. Air was used as a carrier gas and to atomize the spray. The temperature was maintained to be 450 °C during spraying with ±10 °C and 2.5 ml/min of flow rate. To deposit films of uniform thickness the distance between the substrate and spray nozzle was kept at (28±1 cm.).

Measurement

The scattering of light because of the redirection of light that takes place when an electromagnetic (EM) wave (i.e. an incident light ray) encounters an obstacle or non homogeneity, in our case the scattering particle as the EM wave interacts with each discrete particle, the electron orbits within the particle's constituent molecules are perturbed periodically with the same frequency (ν_0) as the electric field of the incident wave.[8]

MIE THEORY

Mie theory based on the theory of Mie, the differential scattering cross sections are defined in terms of the angular intensity functions i_1 and i_2 , as given by the total extinction and scattering cross sections are expressed as:[9]

$$\sigma_{ext} = \frac{\lambda^2}{2\pi} \sum_{n=0}^{\infty} (2n+1) \text{Re}\{a_n + b_n\} \quad (2)$$

$$\sigma_{scat} = \frac{\lambda^2}{2\pi} \sum_{n=0}^{\infty} (2n+1) (|a_n|^2 + |b_n|^2) \quad (3)$$

Where:

σ_{ext} : The total extinction which is equal to summation of absorption and total scattering cross sections(cm^2) .

σ_{scat} : The scattering cross sections .

n : integer-order Bessel function.

λ : the wavelength of Laser .

a_n , b_n : are parameters in terms of Ricatti-Bessel functions .The equations above were incorporated in the program of (Mie plot).

Mie Plot was originally designed to provide a simple interface (for PCs using Microsoft Windows) to the classic BHMIE algorithm for Mie scattering from a sphere - as published by Bohren and Huffman in "Absorption and scattering of light by small particles". In addition to calculations of Mie scattering for

single wavelengths, MiePlot offers calculations for scattering of sunlight - and simulations of

atmospheric optical effects (such as rainbows, coronas and glories). These simulations can be superimposed on digital images of actual optical effects. [10] By using the setup of optical system as illustrated in (fig.1), the size of nanoparticles was calculated .[11]

EXPERIMENTAL SETUP

1) Devices that have been used are:

a) He-Ne Laser source with (632.8 μm) wavelength, and power of 5 mw .

b) Reflector mirror.

c) Two polarizer.

d) An aperture was used to maintain the angular alignment of the wave front beam .Silicon detector: It is used to detect and

convert the scattering rays of the laser to voltage signals.

e) Sample of (Al_2O_3 Nano powder).

f) Mounted arm revolves ($0^\circ - 360^\circ$).

2)The above devices are arranged as in figure (1),the laser source was operated .Polarizer(1) and polarizer(2) has been placed at(90° , 0°) respectively, Where the laser beam did not emerge from the second polarizer. The first and second polarizer are placed at (90°) to be the laser with vertical Polarization .The sample and detector been

placed on the degree of (0°) with respect to the laser beam. When the laser beam collision with the sample, they are scattered in all directions. Light intensity was recorded at the scattering angle of (0°).Detector was moved through different angles ($0^\circ-160^\circ$), The intensity of the beam was recorder relative to scattering angle. Data have been

introduced to the (Mie-plot) program to calculate the radius of nano-particles .The curve of this program as in figure (2). To determine the grain size of Al_2O_3 powder, X-ray diffraction was used from diffractometer type $\text{CuK}\alpha$ with ($\lambda = 1.54056 \text{ \AA}$), the scanning speed was 3%.The data was compared with that ASTM (46-1212) card, the size of Al_2O_3 powder was also investigated by SEM (S-4160).Finally we examined apparent density of nano-powder.

Table(1)Result of XRD of AL_2O_3

M	g.s(nm)
0.001	80
0.01	100
0.1	120

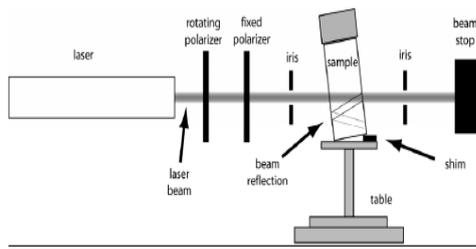


Figure.1 : The optical system setup of Mie scattering[12]

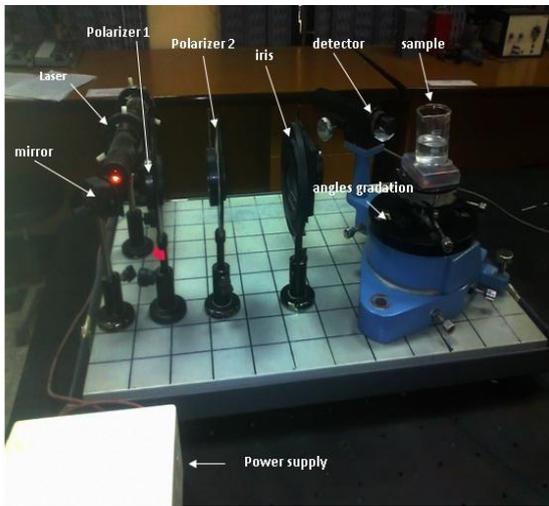
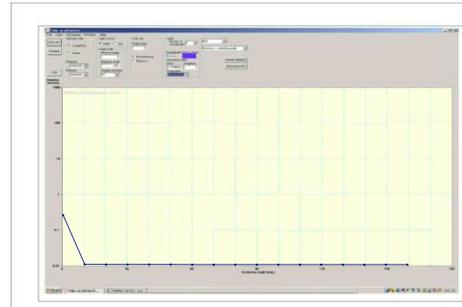
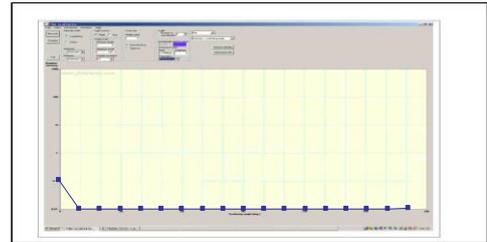


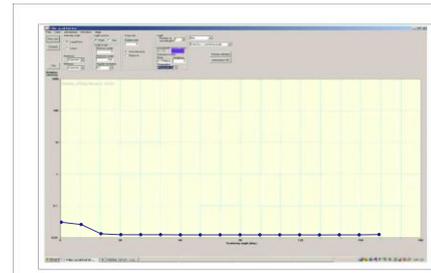
Figure (2): The setup of Nano-Particles



(a)



(b)



(c)

Figure (3) Results of Mie scattering

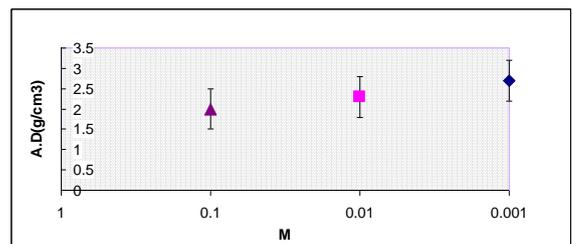
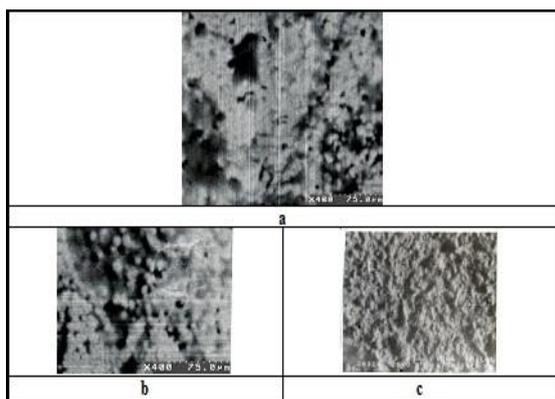


Figure (4) Apparent density with molarity



Figure(5)

RESULTS AND DISCUSSTION

Figure (3) shows the results of the program Mie plot(Mie scattering method) for nano powder of alumina prepared by spraying pyrolysis method where we find it in (a) the size was 100 nm , in (b) 120 nm and in the (c) was 150 nm, this difference in grain size of alumina nano powder can mostly be attributed and linked with the molarity (i.e., the primary concentration the user, taking into consideration other factors of spraying was constant) the rapid feeding (i.e., high rate) led to some granular growth, which led to the largest particle in size. Crystallite Size Measurements from Crystallite Obtained from XRD Measurements was shown in table (1), Crystallite Size (grain size) increased with molarity .Figure (4) shown apparent density with molarity ,we can see that apparent density was decreased as molarity increased we believe that high grain size lead to low density. The results of scanning electron microscope also shown other hand that there is a difference in the .Grain Size of the powder, the record in addition to the differences in simple form, which is shown in figure (5), where we find in the A and B and c images of the form of powder, the record at (0.001, 0.01,0.1M)respectively. That the shape of the powder in the whole is similar to the shape of the record of other ways [14] (where it is generally irregular shape).

CONCLUSIONS

- 1-The powder that resulted from spray pyrolysis method is within the Nano-scale.
- 2- The Nano particles of alumina powder that produced are homogeneous but irregularly shaped in general.

3- Results of Mie-scattering are dependable and its matched well with the other tests results.

ACKNOWLEDGEMENT

We would like to thank everyone who helped us in completing this paper.

NOMENCLATURE

n : integer-order Bessel function.

a_n , b_n : are parameters in terms of Ricatti-Bessel

Greek Letters

σ_{ext} : The total extinction which is equal to summation of absorption and total scattering cross sections(cm^2) .

σ_{scat} : The scattering cross sections .

λ : the wavelength of Laser .

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