ARPN Journal of Engineering and Applied Sciences

www.arpnjournals.com

SYNTHESIS AND CHARACTERIZATION OF NANO GAMMA ALUMINIUM OXIDE FROM IRAQI BAUXITE USING EXTRACTION METHOD

Hamid S. Meteab, Hadi A. Karem and Watheq K. Salih
Ministry of Science and Technology, Directorate of Materials Research, Baghdad, Iraq
E-Mail: alwathiq316@yahoo.com

ABSTRACT

Aluminium oxide (Al_2O_3) considers one of the important ceramic oxides in present time because of its characteristic properties which are suitable for different industrial applications. The recent developments in nano technology encouraged the scientists to prepare nano alumina using different methods. The present work deals with preparing nano gamma aluminium oxide (γ -Al_2O_3) from Iraqi Bauxite ore using extraction method. The ore was leached with an acidic solution and the aluminium was extraction in the form of aluminium chloride. After extraction, the Aluminium chloride was treated with ammonia solution to obtained aluminium hydroxide which was calcined and milled to get on nano alumina powder (Gamma phase). The resultant powder was characterized by X-Ray diffraction, BET, AFM and FTIR techniques. The work results were clarified the possibility of producing nano gamma alumina with high surface area using this method.

Keywords: Iraqibauxite, nano gamma alumina, XRD, aluminium hydroxide, FTIR, ore extraction.

1. INTRODUCTION

Aluminium oxide is a compound of aluminium and oxygen with chemical formula of Al_2O_3. It is the most commonly occurring of several Aluminium oxides, and specifically identified as Aluminium (III) oxide. It is commonly called alumina. There are a seven polymorphs of transition alumina identified so far, namely, phases γ,α, η, δ, θ, κ and χ. This polymorphism can be classified in terms of the oxygen sub-lattice structure and the distribution into this sub-lattice of aluminium ions in tetrahedral and octahedral interstitial sites. Thus, in α-Al_2O_3, the Oxygen sub-lattice is hexagonal-close-packed(hcp) structured with 2⁄3 of octahedral sites occupied with cations, while γ, δ, η, θ have a face-centered cubic (FCC) arrangement of oxygen atoms and cations present in various proportions in both octahedral and tetrahedral sites[1].

The γ form is one of the most extensively used in industrial catalysis owing to its comparatively large surface area, unique surface characteristics, and exceptional structural stability. γ-alumina contains the same ratio of Al to O atoms as in α- alumina but the only difference is that it has a tetragonal structure where there are 8 cation vacancies for every 160 atoms[2].

The uses of aluminium oxide (all phases) are varied with numerous range like electrical material [3], catalyst [4,5], water treatment [6,7], filler of polymers [8,9], additive for adhesives [10] and optics as transparent film [11].

In recent years, because of the varied applications of γ-Al_2O_3, special attention has been focused on the preparation of γ-Al_2O_3 powders with high purity by various routes such as Precipitation [12], Laser ablation[13], Sol-gel[14,15], Hydrothermal [16,17],Microwave[18],Micro-emulsion[19] and Combustion[20] modes methods.

There are many materials used in recent years to produce nano alumina in all phases specially gamma phase including boehmite and aluminium alkoxide which are classified as expensive materials. So, the efforts were done searching for cheaper materials to reduce the cost of production. Different raw materials like bauxite, kaolin, mica and fly ash were used. Among these materials, Bauxite is the best source to synthesize nano alumina powder because it is a good source of aluminium. It has hard off- white to reddish brown rock colour. aluminiumis the 3rd most abundant element in earth crust.It is a popular one in the modern world due to its corrosion resistance, good electrical conductivity and light weightlessness. It is the 3rd hardest element in nature after diamond and silicon carbide [21]. Bauxite is a naturally occurring, heterogeneous material comprised primarily of one or more aluminium hydroxide minerals plus various mixtures of silica (SiO_2), iron oxide (Fe_2O_3), titanium dioxide (TiO_2), aluminosilicate (clay, etc.) and other impurities in trace amounts. The principal aluminium hydroxide minerals found in varying proportions within bauxite are gibbsite [$Al(OH)_3$] and the polymorphs, boehmite, and diaspore (both $AlO(OH)$) [22,23].

The aim of this work is to prepare nano gamma alumina oxide from Iraqi bauxite using extraction method. The resultant powder is characterized using XRD, AFM, SEM and FTIR

2. EXPERIMENTAL

2.1 Materials

All the chemicals and reagents were analytical grade and they were used as supplied without any further purification.

Bauxite was obtained from Iraqi Geological Survey Company and its composition was shown in Table-1. Hydrochloric acid (HCl,37% extra pure, LobaChemie, India),Ethanol (99.8%, Fluka, Germany) and Ammonia solution (32%, BDH, England).

Table-1. The composition of Iraqi Bauxite.

Materials	Percentage %
SiO_2	17.5
Al_2O_3	65.2
TiO_2	3.5
Fe_2O_3	1.3
CaO	1.2
MgO	1.07
others	3.93
L.O.I	6.3

2.2 Experimental procedure

Bauxite sample was milled into fine powder using mortar (PM 400, China).and was passed through a magnetic separator (GYH3, China) to remove the amount of titanium oxide and iron oxide, The Slurry of bauxite was made and passed through a hydro-cyclone (AZVorspin, USA) to remove free silica and then dried to get powder. Then, it was added gradually to the Hydrochloric acid solution (6M HCl), and the solution was refluxed at 90 C̊ for 3 hr. The obtained solution was left to cool and then ethanol- distilled water mixture was added and mixed using ultrasonic probe (Heilscher UP200/400, probe diameter 9 mm and 85% amplitude, Germany) for 1 hr. to get a transparent solution. After that, the ammonia solution was added to the solution drop by drop till the precipitation was appeared. The aluminium hydroxide($Al(OH)_2$) precipitation was filtered under the vacuum .In final step, the precipitated powder was calcined at 900 ◦C for 2 hr. with heating rate of 10 C̊/ min and then milled .The resultant powder (Al_2O_3 gamma phase) was stored in desiccator for characterization tests.

2.3 Analysis

2.3.1XRD

Analysis of X-ray diffraction (XRD) had been Fulfilled by Shimadzu XRD-6000 diffractometer with Cu Kα radiation (A = 1.5418A).The XRD 2θ angles ranged from 10° to 70° at constant scan rate.

2.3.2 AFM

The particle size of prepared γ- Al_2O_3 was conducted using Atomic Force Microscope (NTEGRA Prima model, Netherlands).

2.3.3 BET

The surface area & pore size of synthesized nano gamma Alumina were done using BET method (Horiba SA-9600, Japan).

2.3.4 FTIR

The FTIR spectrum was recorded on Shimadzu-8400S Fourier Transforms-infrared spectrophotometer using KBr disk. The bands selected in the range 400-4000 cm^{-1}

3. RESULTS AND DISCUSSIONS

3.1 Phase characterization using X-ray diffraction Technique:

The prepared samples were (aluminium hydroxide& alumina) irradiated with a beam of monochromatic X-rays over a variable incident angle range. The both samples were characterized at room temperature by X-ray diffraction The XRD pattern of aluminium hydroxide peaks was 18.34,20.6, 26.41, 26.8, 28.1,28.93, 36.6, 36.8,37.2,39.4,40.2,41.2, 44.25,46.1,47.3, 50.2° and 54.3.These peaks values were characterized and matched with ICDD reference data. The prepared aluminium hydroxide is identified with (JCPDS, File # 33-0018).

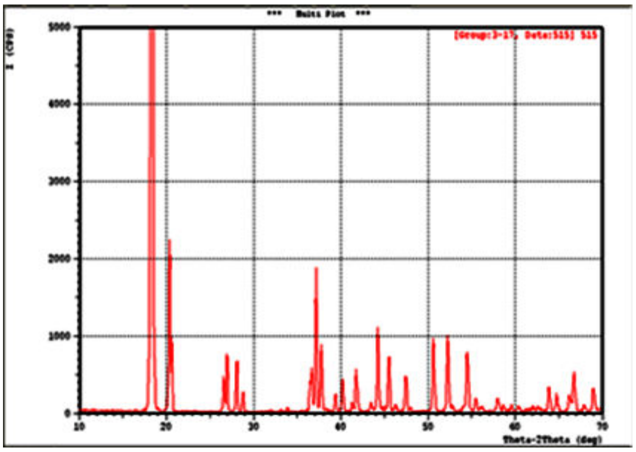

Figure-1. XRDpatternof aluminium hydroxide.

While, the synthesized nano γ-alumina is shown in Figure-3 that the three peaks at 2θ =37.6º, 2θ =45.5º, and 2θ =67.0º are assigned to (311), (400) and (440) reflections of γ- Al_2O_3. The peaks of γ- Al_2O_3 is agree with (JCPDS, File # 29-0063) peaks.

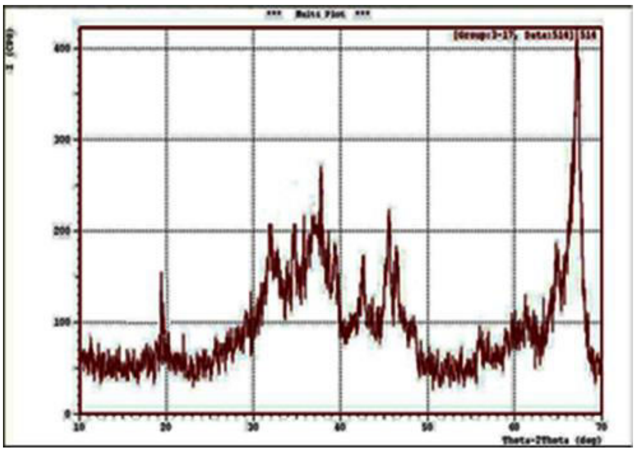

Figure-2. XRD Pattern of γ -aluminium oxide.

ARPN Journal of Engineering and Applied Sciences

3.2 Particle size and diameter characterization

The average diameter of the particles is about 55.5 nm, while the particle size distribution from the smallest to largest size is in 10 to 155 nm range as shown in Figure-3 while the morphology is shown in Figure-4.

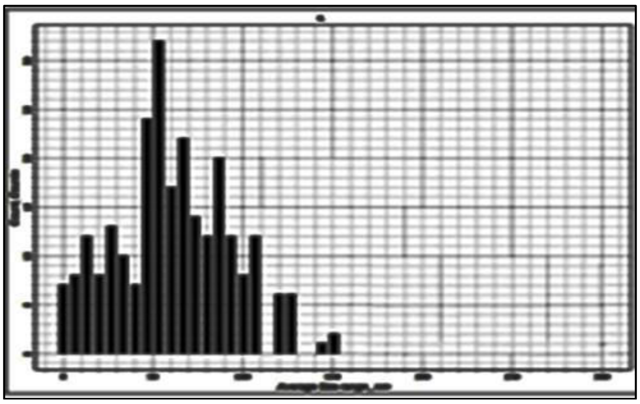

Figure-3. Particle size distribution of γ -aluminium oxide.

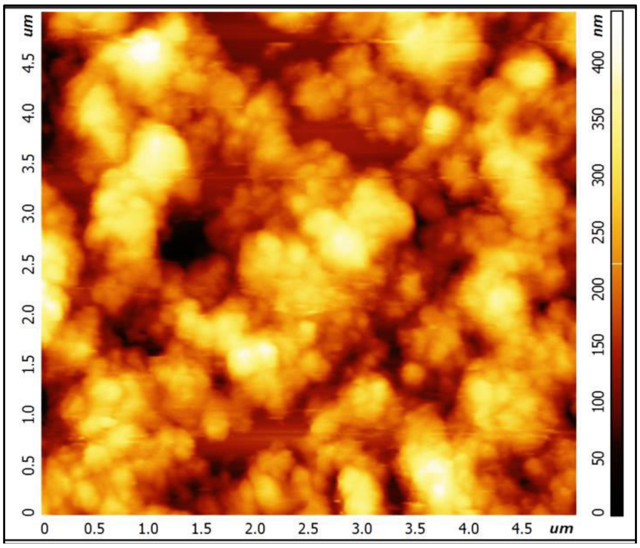

Figure-4. Morphology of γ -aluminium oxide particles.

3.3 Surface area and pore size

The specific surface area of a powder is estimated from the quantity of nitrogen adsorbed in relationship with its pressure at the boiling temperature of liquid Nitrogen under normal atmospheric pressure. The surface area was about (240 m^2/g) while pore volume was about (0.37 cm^3/g). This value of surface area is greater than 125 m^2/g and it indicates that synthetic nano gamma alumina is very stable [24].

3.4 4Molecular bonding characterization

The molecular bonding of nano gamma alumina molecules was investigated via FTIR test. The bands selected in the range 400-4000 cm^{-1}.The band around 3520 cm^{-1}can be attributed to the stretching vibration of (-OH) from Al-OH framework while the other around 1610 cm^{-1} are assigned to (O-H) stretching and bending modes of adsorbed water respectively [25]. The wide band is appeared between 400-800cm^{-1} corresponds to the vibrational frequencies of co-ordinate (O-Al-O) bond. This wide band can be divided in two clear banding, the first around 560 cm^{-1} was assigned to ν-AlO_6 and the other at 760-800 cm^{-1} was assigned to ν-AlO_4 in nano Al_2O_3[26,27].

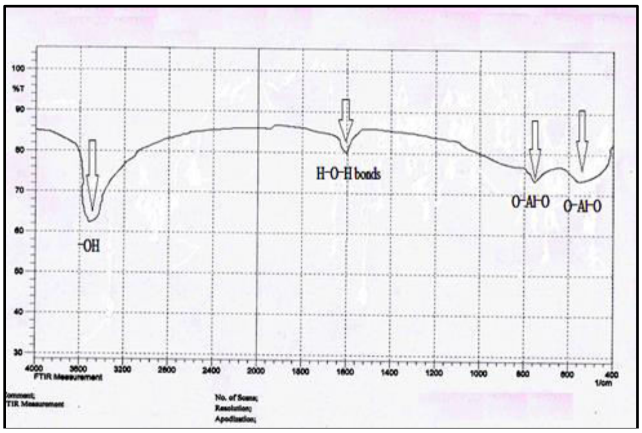

Figure-5. FTIR spectrum of synthesized aluminium oxide.

4. CONCLUSIONS

The results have successfully demonstrated that γ-Al_2O_3 nanoparticles can be synthesized from Bauxite ore by used chemical and physical treatments involving ultrasonic mixing with high surface area and pore volume which is suitable to be used for different applications as catalyst and filler in polymer nanocomposites, water treatments and other uses.

REFERENCES

[1] Boumaza A., Favaro L., Lédion J., Sattonnay G., Brubach J. B., Berthet P., Huntz A. M., Roy P.Tétot R. 2009. Transition alumina phases induced by heat treatment of boehmite: An X-ray diffraction and infrared spectroscopy study, J. Solid State Chem. 182:1171-1176.

[2] Kaya C., HeJ. Y., Gu X., Butler E.G. 2002. Nanostructured Ceramic Powders by Hydrothermal Synthesis and Their Applications. Microporous Mesoporous Mater. 54:37-49.

[3] Bandara T. M. W. J., Karunathilaka D. G. N., Ratnasekera J. L., De Silva L. A., Herath A. C., Mellander B. E.2017. Electrical and complex dielectric behaviour of composite polymer electrolyte based on PEO, alumina and tetrapropylammonium iodide, Ionics 23: 1711–1719

[4] Elfadly A. M., Badawi A. M., Yehia F. Z., Mohamed Y. A., Betiha M. A., Rabie A. M. 2013. Selective nano alumina supported vanadium oxide catalysts for

oxidative dehydrogenation of ethylbenzene to styrene using CO_2 as soft oxidant, Egyptian Journal of Petroleum. 22: 373-380.

[5] Khanbolouk F., Akia M., Arandiyan H., Yazdani F., Dortaj Y. 2017.Utilization of spray-dried nanoporous gamma alumina support in biodiesel production from waste cooking oil, Journal of Nanostructure in Chemistry. pp. 7191-7200.

[6] Chinnakoti P., Chunduri A. L.A., Vankayala R. K., S. Patnaik, Kamisetti V. 2017. Enhanced fluoride adsorption by nano crystalline c-alumina: adsorption kinetics, isotherm modeling and thermodynamic studies, Appl. Water Sci. 7:2413-2423.

[7] Afkhami A., Tehrani M. S., Bagheri H. 2010. Simultaneous removal of heavy-metal ions in wastewater samples using nano-alumina modified with 2,4-dinitrophenylhydrazine, J. Hazard. Mater. 181: 836-844.

[8] Akmil N., Luqman C. A., Ahmad M., Zaman K. 2012. Improved mechanical properties of HDPE/nano-alumina composite through silane coupling agent, AIP Conf. Proc. 1502: 500-510.

[9] Brostow W., Lobland H. E. H., Hnatchuk N., Perez J. M.2017. Improvement of Scratch and Wear Resistance of Polymers by Fillers Including Nanofillers, Nanomaterials. 7, 66: 12.

[10] Ghabezi P., Farahani M. R.2017. Experimental investigation of nanoparticles effects on cohesive model and bridging laws of mode I fracture in the adhesive joints, J. Adhes. Sci. Technol. 31: 1807-1823.

[11] Esther C. M., Sridhara N., Sebastian S. V., Bera P., Anandan C., Aruna S. T., Rangappa D., Sharma A. K., Dey A. 2015. Optical and RF transparent protective alumina thin films, J. Mater. Sci. Mater. Electron. 26 :9707-9716.

[12] Parida K. M., Pradhan A. C., Das J., Sahu N. 2009. Synthesis and characterization of nano-sized porous gamma-alumina by control precipitation method, Mater. Chem. Phys. 113:244-248.

[13] IsmailR.A., Zaidan S. A., Kadhim R. M. 2017. Preparation and characterization of aluminium oxide nanoparticles by laser ablation in liquid as passivating and anti-reflection coating for silicon photodiodes. Appl. Nanosci.7: 477-487.

[14] Dubey S., Singh A., Nim B., Singh I. B. 2017. Optimization of molar concentration of AlCl3 salt in the sol–gel synthesis of nanoparticles of gamma alumina and their application in the removal of fluoride of water, J Sol-Gel Sci Technol. 82: 468-477.

[15] Rogojan R., Andronescu E., Ghiţulică C., Vasile B. Ş.2011. Synthesis and Characterization of Alumina Nano-Powder Obtained by Sol-Gel Method, U.P.B. Sci. Bull., Series B. 73:67-76.

[16] Mohammed A.-H. A.K., Hussein H. Q., Mohammed M. S. 2017. The Effect of Temperature on the Synthesis of Nano-Gamma Alumina Using Hydrothermal Method, Iq. J. Chem. Petro. Eng. 18(March):16.

[17] Lu C. L., Lv J. G., Xu L., Guo X. F., Hou W. H., Hu Y., Huang H. 2009. Crystalline nanotubes of γ-AlOOH and γ-Al2O3: hydrothermal synthesis, formation mechanism and catalytic performance, Nanotechnology. 20(21).

[18] Sivadasan A. K., Selvam I. P. and Potty S. N. 2010. Microwave assisted hydrolysis of Aluminium metal and preparation of high surface area g Al2O3 powder, Bull. Mater. Sci. 33: 737-740.

[19] Piao L. Y., Liu X. Z., Mao L. J., Ju S. T.2009. Preparation of Nano-Alumina by Reverse Microemulsion Method, Acta Physico-Chimica Sinica. 25: 2232-2236.

[20] Sherikar N. B., Umarji A. M. 2013. Synthesis of g-Alumina by Solution Combustion Method Using Mixed Fuel Approach (Urea+Glycine Fuel), IC-RICE Conference, Gulbarga, India

[21] Pervaiz M., Butt K. M., Raza M. A., Rasheed A., Ahmad S., Adnan A. 2015. Extraction and applications of aluminium hydroxide from bauxite for commercial consumption, Chemistry International. 1: 99-105.

[22] Plunkert P. A. 2000. Bauxite and Alumina, U.S. Geological Survey Minerals Yearbook, USA.

[23] Fahad M., Iqbal Y. 2009. Bauxite deposit in Pakistan: An Introduction, Journal of Material Society. 3:41-44.

[24] Rodríguez J. A., García M. F. 2007. Synthesis, Properties, and Applications of Oxide Nanomaterials. John Wiley & Sons, New Jersey, USA.

ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

[25] Afkhami A., Tehrani M. S., Bagheri H. 2010. Simultaneous removal of heavy-metal ions in wastewater samples using nano-alumina modified with 2, 4-dinitrophenylhydrazine. Journal of Hazardous Materials. 181: 836-844.

[26] Hosseini S. A., Niaei A., Salari D., Production of γ-Al_2O_3. 2011. Chem. Mater. Sci. J. 1: 23-27.

[27] Karim M. R., Rahman M. A., Miah M. A. J., Ahmad H., Yanagisawa M., Ito M. 2011. Synthesis of γ -Alumina Particles and Surface Characterization, The Open Colloid Science Journal. 4: 32-36.