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Electrochemical Preparation and Characterization of Aluminum Oxide Nanoparticles

[1]Aqeel Mahdi Alduhaidahawi, [2]Fouad A.A. Al-Saady and [1]Amer Musa Jouda
[1]Faculty of Science, University of Kufa, Kufa, Iraq
[2]Faculty of Pharmacy, Al-Mustansriyah University, Baghdad, Iraq

Abstract: Aluminum oxide Nanoparticles (Al_2O_3 NPs) were prepared by electrochemical method in solution contain $NaNO_3$ as electrolyte and Poly Vinyl Alcohol (PVA) as stabilizer. The synthesized Al_2O_3 NPs were characterized by FTIR, SEM, TEM and AFM studies. FTIR spectrum of Al_2O_3 NPs exhibited absorptions at (605 and 883 Cm^{-1}) are attributed to Al-O bond. SEM micrographs givesirregular agglomerated small (flower) like structure. From TEM study the size of Al_2O_3 NPs was found to be <40 nm and the shape of nanoparticles was uniform thread like structure . The average size of synthesized Al_2O_3 NPs from AFM study was found to be 41.16 nm.

Key words: Electrochemical preparation, aluminum oxide nanoparticles, AFM, SEM, TEM, thread

INTRODUCTION

Nanotechnology includes the study, control and manipulation of chemical materials at the nanometer scales, typically they having dimensions <100 nm (Mallikarjuna *et al*., 2012; Kannan and Subbalaxmi, 2011). The properties of such materials are novel and can be engineered by controlling the dimensions of these structures and their installation by physical, chemical or biological procedures. Singh *et al*. (2011) The characters of metal and metal oxide nanomaterials such as optical, magnetic, electronic and catalytic are depending on their shape, size and chemical surroundings (Theivasanthi and Alagar, 2010). Nanomaterials are of large heed because of various uses in chemical, biological and environmental fields (Awual *et al*., 2015; Kumar *et al*., 2014; Shahat *et al*., 2015). The size and shape of the nanoparticles are main operatorsto diversiformtheir properties such as optical, magnetic, electrical, catalytic and antimicrobial (Katwal *et al*., 2015). Metal oxide nanoparticles possesses widely developedin the past decenniums. They have been vastly used in many applications such as catalysts, sensors, semiconductors, capacitors, batteries, cosmetic, medical and engineering science (Ueda *et al*., 2008; Gessner *et al*., 2000; Kim *et al*., 2005; Pria, 2007; Farsi and Gobal, 2007; Dillon *et al*., 2008). Among them aluminium oxide is one the most widely used ceramic materials as catalysts, catalyst supports and absorbents and also wear resistant coating (Aghababazade *et al*., 2007).

Aluminum oxide Nanoparticles (Al_2O_3 NPs) have attracted significant attention due totheir lots uses in several fields. Ceramics based on Al_2O_3 possesses a largelyused innew industry due to their singular characteristics like, high hardness, high mechanical strength and good chemical stability. They have been using in create various catalytically active complexes for cleaning industrial emissions for the oil industry (Jiao *et al*., 2012). Al_2O_3 is inert atroom temperature and is insoluble in all common chemical reagents. It appears distingue wearresistance and can be polished to a surface finish. Various allotropes of Al_2O_3 have been reportedin the literature (Jiao *et al*., 2012), however, Al_2O_3 Nanoparticles (Al_2O_3 NPs) were prepared by various methods involving; ball milling, pyrolysis, solgel method, sputtering, hydrothermal, laser ablation and electrochemical methods (Reid *et al*., 2008; Mirjalili *et al*., 2010; Kavitha and Jayaram, 2006; Trinh *et al*., 2008; Qu *et al*., 2005; Yatsui *et al*., 2000; Shelke and Rajbhoj, 2017). Among them, the electrochemical method which have advantages such as easy installatio, relative shortened time, high purity product is obtained, facilitate isolation and no side products formation (Shelke and Rajbhoj, 2017).

MATERIALS AND METHODS

Experimental section: The electrochemical system for preparation of aluminium oxide (Al_2O_3) nanopowders is shown schematically in Fig. 1. A 250 mL-glass beaker, pure aluminium foil (0.25 mm thin) as anode (positive) electrode, graphite plate as cathode (negative)electrode, D.C-regulated power supply (30 V maximum voltage and

Corresponding Author: Aqeel Mahdi Alduhaidahawi, Faculty of Science, University of Kufa, Kufa, Iraq

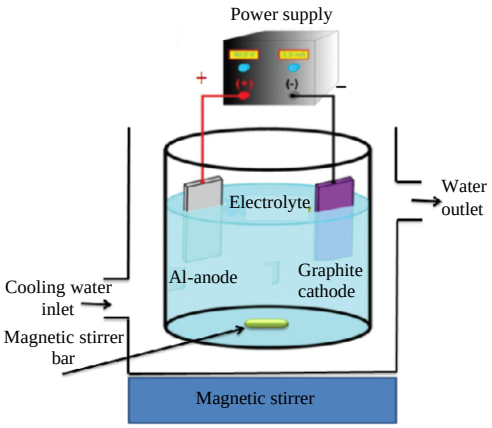

Fig. 1: The electrochemical system for preparation of Al_2O_3 nanoparticles in PVA and $NaNO_3$

5000 mA maximum current) was used for providing and measuring , stirrer plate, magnetic stirrer bar and cooling water bath.

Prior of electrolysis, both cathode and anode were washed by acetone and ethanol and then deionized water to cleaning the two electrodes surfaces from any organic materials.

In typical electrochemical procedure the electrochemical cell filled with 200 mL solution contain of (15 mL of 10 g/100 mL $NaNO_3$ as electrolyte, 10 mL of 1 g/100 mL Poly Vinyl Alcohol (PVA) as stabilizer and deionized water). An pure aluminium foil (1×4 cm) and inert graphite electrode (2×5 cm) were as anode and cathode, respectively being vertically and placed face to face in electrochemical cell solution with 2 cm apart. The electrolysis reaction was performed in an undivided electrolytic cell for 60 min with stirrer at temperature of 15-30°C. The temperature is increased during the electrolysis, then the cooling water bath was used for control and prevent the increasing in temperature. The voltage at range of 20-30 V.

After 10-15 min of starting of electrolysis, milky suspended solution is produced of Al_2O_3 nanoparticles. The pH of solution rises during of electrolysis reaches to a maximum value of 11.6 and stays constant even to end of electrolysis. The aluminium electrode is used once of each experiment.

The resulting white precipitates of Al_2O_3 nanoparticles were centrifuged washing with deionized water and ethanol for several times and then dried at 60°C for 1 h.

RESULTS AND DISCUSSION

FT-IR study: The functional groups of prepared Al_2O_3 nanoparticles during electrochemical process was studied

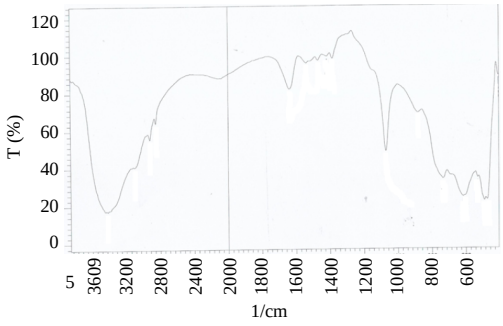

Fig. 2: FT-IR spectrum of electrochemical prepared Al_2O_3-NPs in presence of PVA (1 g/100 mL) as stabilizer and $NaNO_3$ (10 g/100 m) as electrolyte

by FT-IR spectroscopy (Fig. 2). The absorption band at 3419 cm^{-1} was due to the O-H vibrations. The two peaks at (605 and 883cm^{-1}) are back to Al-O bond.

Scanning electron spectroscopy: The morphology of electrochemical synthesized Al_2O_3 Nanoparticles in presence of 15 mL of 10 g/100 mL $NaNO_3$, 10 mL of 1 g/100 mL PVA was examined by Scanning Electron Spectroscope (SEM) as shown in Fig. 3, the SEM images reveals irregular agglomerated small (flower) like structure.

Transmission electron spectroscopy: The morphology and size of Al_2O_3 NPs which is electrochemically synthesized in 15 mL of 10 g/100 mL $NaNO_3$, 10 mL of 1 g/100 mL PVA was examined by Transmission Electron Spectroscope (TEM) micrograph of Al_2O_3 NPs as shown in Fig. 4 which illustrates the TEM micrograph of Al_2O_3 NPs .The nanoparticles exhibited a uniform thread like shape having a sizes in the range of 20-40 nm.

Atomic force microscopy analysis: Atomic Force Microscope (AFM) topography imaging is a useful tool to obtain information and study about morphology, topography and textile of various surfaces. In the AFM image of Al_2O_3 NPs (Fig. 5) the dark colors are the low structures while the bright colors are the high structures due to different directions of Al_2O_3 grains.

The 3-D AFM image Fig. 5a indicates the formation of homogeneous distribution of Al_2O_3 NPs and no agglomerated was observed . The obtained data from Granularity Cumulation Distribution (GCD) about the particles size distribution in Fig. 6 which gives the range of particles diameters of Al_2O_3 NPs between 32-84 nm and the average diameter is found to be 41.16 nm. The other topographic information data were summarized in Table 1.

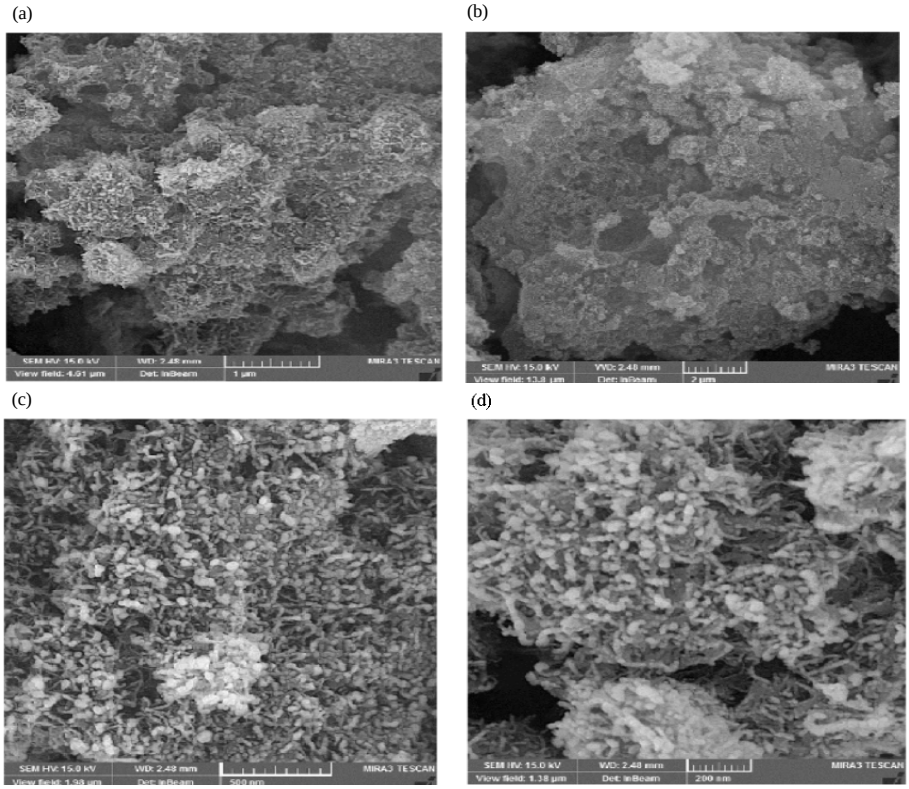

Fig. 3: SEM micrograph of electrochemical prepared Al_2O_3-NPs in presence of PVA (1 g/100 mm) as stabilizer and $NaNO_3$ (10 g/100 mL) as electrolyte

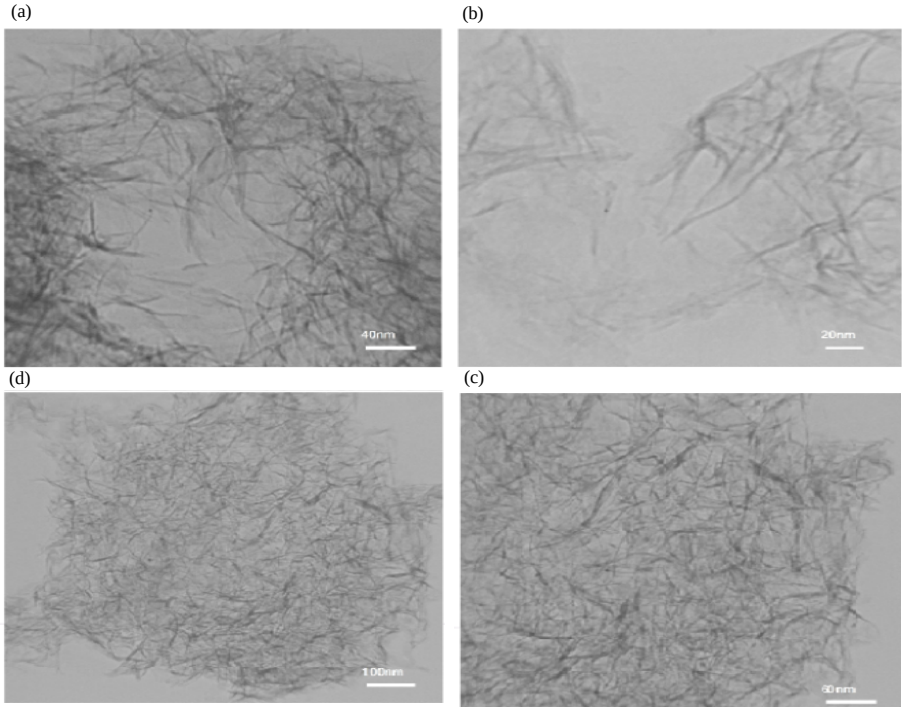

Fig. 4: TEM micrograph of electrochemical prepared Al_2O_3-NPs in presence of PVA (1 g/100 mL) as stabilizer and $NaNO_3$ (10 g/100 mL) as electrolyte

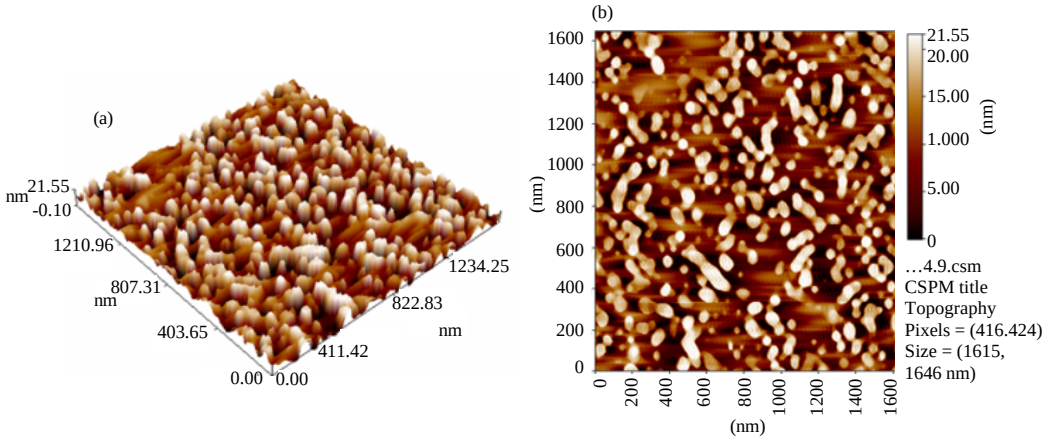

Fig. 5: AFM images of electrochemical prepared Al_2O_3-NPs in presence of PVA (1g/100 mL) as stabilizer and $NaNO_3$ (10 g/100 mL) as electrolyte

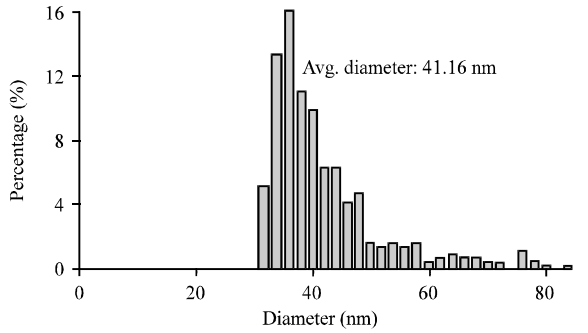

Fig. 6: GCD-chart of electrochemical prepared Al_2O_3-NPs in presence of PVA (1 g/100 mL) as stabilizer and $NaNO_3$ (10 g/100 mL) as electrolyte; Granularity cumulation distribution chart

Table. 1: The topographic information data which were obtained from AFM

CSPM imager surface roughness analysis	Values
Roughness average (Sa) nm	5.39
Root mean square (Sq) nm	6.24
Surface skewness (Ssk) nm	0.0604
Surface kurtosis (Sku)	1.82
Peak-peak (Sy) nm	21.6
Ten point height (Sz) nm	21.6
Mean summit curvature (Ssc) nm^{-1}	-0.0933
Root mean square slope(Sdq) nm^{-1}	0.674
Surface area ratio (Sdr)	18.3
Surface bearing index (Sbi)	4.92
Core fluid retention index (Sci)	1.53
Valley fluid retention index (Svi)	0.0674
Reduced submmit height (Spk) nm	3.18
Core roughness depth (Sk) nm	18
Reduced valley depth (Svk) nm	0.619

CONCLUSION

It has been proposed successfully electrochemical preparation of Al_2O_3-NPs including dissolution of aluminium electrode in $NaNO_3$ as electrolyte and PVA as stabilizer of nanoparticles. This is an easy, economic and time budge procedure. The average size of Al_2O_3-NPs is found to be 41.16 nm which is obtained from GCD and <40 nm which is obtained from TEM micrographs. According to TEM micrographs the nanoparticles having uniform thread like structure.

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