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Preparation and Characterization of Alumina Nanoparticles from Coal Fly Ash

Myat Myat Thaw[1] and Khine Thin Thin Aye[2]

Abstract

In the present research, coal fly ash samples were collected in Tigypt Power Plant from Southwest Shan State in Myanmar. Alumina nanoparticles was synthesized from coal fly ash by using alkali lime sintering method. Alumina nanoparticles were obtained by using six steps (leaching step, pre-desilication step, leaching step, desilication step, carbonation step, and calcination step at 1000° C for 3 h. The alumina nanoparticles obtained from coal fly ash is in agreement with aluminium nitrate by using lactic acid as a standard reference. It was found that average crystallite size of alumina nanoparticles from chemical source and coal fly ash were to be 47.83 nm and 63.55 nm by using Debye Scherer equation. According to Lattice parameter values, the crystal structure of alumina nanoparticles from coal fly ash were observed to be hexagonal. The morphology of alumina nanoparticles from chemical source and coal fly ash were found to be spherical in shape by using field emission scanning electron microscope.

Keywords : **coal fly ash, alumina, FE SEM, XRD, Debye Scherrer**

Introduction

Application and occurrence of fly ash

Fly ash can be applied in construction industry, ceramic industry environmental protection, and catalysis, zeolite synthesis and valuable metal recovery. Fly ash can be found in China, India, U.S and EU countries (Goswami et al., 2013).

Applications of alumina nanoparticles

Manufactured nanoparticles of aluminum oxide (nano-alumina) have been widely used in the environment; however, their potential toxicity provides a growing concern for human health. Aluminum is relatively stable in the form of alumina (aluminum oxide) and can enter the body through drinking water, food intake, inhalation, and skin contact. In addition, specific medical interventions,

[1] Lecturer, Dr, Department of Chemistry, University of Yangon

[2] Mres, Department of Chemistry, University of Yangon

such as dialysis or certain aluminum-containing drugs, may lead to aluminum accumulation in the tissues (Hongbin 2010).

Aim and Objectives

The preparation, characterization of alumina nanoparticles from coal fly ash was studied. Coal fly ash sample from raw coal fly ash by magnetic separator was prepared. Coal fly ash sample was characterized by using EDXRF, XRD, FT IR, and SEM techniques. Alumina nanoparticles from coal fly ash by using alkaline leaching sintering method. Alumina nanoparticles was alumina nanoparticles was characterized by using XRD, FT IR, SEM, and FE SEM techniques.

Materials and Methods

Sample collection

Coal fly ash samples was collected in Tigyit Power Plant from South-west Shan State

in Myanmar.

Chemicals

Aluminium nitrate, lactic acid and polyvinylpyrrolidone (PVP) Sodium hydroxide Calcium hydroxide and Distilled water were used.

Apparatus

Grinder, 200 μm mesh sieve, and magnetic separation machine,a magnetic stirrer, a centrifuge, a pH meter and an oven, test tubes, a hot plate, a constant temperature bath, an autoclave, a hot air sterilizer, an incubator, an electronic balance, spirit burner, pipettes (0.1, 1 and 10 mL), 100 mL and 50 mL measuring cylinders, a glass rod, 250 mL conical flasks, 250 mL beakers, 250 mL volumetric flasks, and X-ray diffractometer (Rigaku-D-max 2200, Japan), Scanning Electron Microscope (JOEL-JSM-5610 Japan), Iron Sputter (JFC-1600),and field Emission Scanning Electron Microscope (EVO 60, Brand: Card ZEISS, Germany) were used.

Procedure

A 100 g of coal fly ash with 150 mL of 4 M sodium hydroxide solution were leached in a beaker (500 mL, Pyrex) as a leaching step. The mixed solution were heated at 100 °C in a covered 500 mL beaker for 3 hours to dissolve silica. Then the solution was filtered with Whatman No. 1 filter paper into a beaker. It was carefully wiped and rinsed with distilled water to avoid the loss of residues. The residue on the filter paper were washed with 100 mL hot distilled water several times and dried in an oven at 105 °C for 3 hours. The dried leached ash was placed into a muffle furnace at 1000 °C for 3 hours. A 70 g leached ashes were mixed with 94 g lime stone and 42 g soda ash. The mixture sample was placed into a muffle furnace at 1000 °C for 3 hours to produce clinker. Sodium hydroxide solution (5 %) was added into the clinker (1:3). The mixed solution was heated at 90 °C in a covered 500 mL beaker for 30 minutes with constant stirring to dissolve the silica and to produce sodium aluminate solution. The solution was filtered with Whatman No. 1 filter paper and washed with hot distilled water. The sodium aluminate solution were deeply desilicated by adding saturated lime milk at

1000 °C for 1 hour. The sodium aluminate were obtained by desilication. Then, the aluminium hydroxide were prepared by passing carbon dioxide into the sodium aluminate solution for carbonation decomposition. Alumina nanoparticles were obtained by sintering aluminum hydroxide at 1000 °C for 3 h (Cheng .,2012).

Results and Discussion

Technologies of alumina extraction from coal fly ash can be classified into acidic, alkali and acid alkali methods. Among these methods, alkaline leaching sintering method was used to obtain alumina nanoparticles. Non-magnetic portion of coal fly ash samples were obtained by using magnetic separation machine. The coal fly ash samples were sieved to get the 200 mesh size after separation (Nawawi 2013).

Analysis of alumina nanoparticles from coal fly ash and aluminium nitrate by XRD

The X-ray diffraction is used to determine crystallinity of polymerase materials. Crystalline nature of alumina nanoparticles from coal fly ash was investigated by XRD technique. Extraction of alumina nanoparticles from coal fly ash samples by alkaline leaching sintering method was done and calcined at different temperatures (800°, 900°, and 1000° C) . Crystalline nature of alumina nanoparticles was not found clearly at 800° and 900° C for 3 hours. In this work, crystalline nature of alumina nanoparticles was found at 1000° C for 3 hours. Figure 1. shows XRD diffractogram of alumina nanoparticles synthesized from coal fly ash samples by alkaline leaching sintering method at calcination temperature at 1000° C for 3 hours. XRD diffraction peaks located at the 2 θ values of 25.71°, 35.26°, 43.44°, 52.50°, and 57.58° corresponding to Miller indices (012), (104), (113), (024), and (116) planes, respectively indicated the formation of alumina nanoparticles. The diffraction peaks of alumina are matched with standard PDF data 80-0826 and 73-112 corundum Al_2O_3 standard. Average crystalline size of alumina nanoparticles was calculated and found to be 65 nm by Debye Scherer equation. The crystal structure of alumina nanoparticles found to be hexagonal according to lattice parameters (a = b = 4.75 Å, and c = 12.8632 Å). It was found the average crystallite size of alumina nanoparticles from aluminium nitrate was observed to be 47 nm according to XRD data. XRD diffractogram of alumina nanoparticles from aluminium nitrate is shown in Figure 2. The XRD diffractogram reveals diffraction peaks of (012), (104), (110), (113), (024), (116), (214), and (300) correspond to 2 θ values 25.7°, 35.2°, 43.44°, 52.5°, 57.5° respectively (Table 3.3). Alumina nanoparicles from aluminium nitrate are hexagonal ccording to lacttice parameters a= 4.771Å and c= 12.7454Å (Table 1and 2). It can be seen that eight diffractogram of alumina nanoparticles obtained from coal fly ash at 1000° C for 3 hours are similar and exhibit diffraction pattern of alumina nanoparticles from aluminium nitrate.

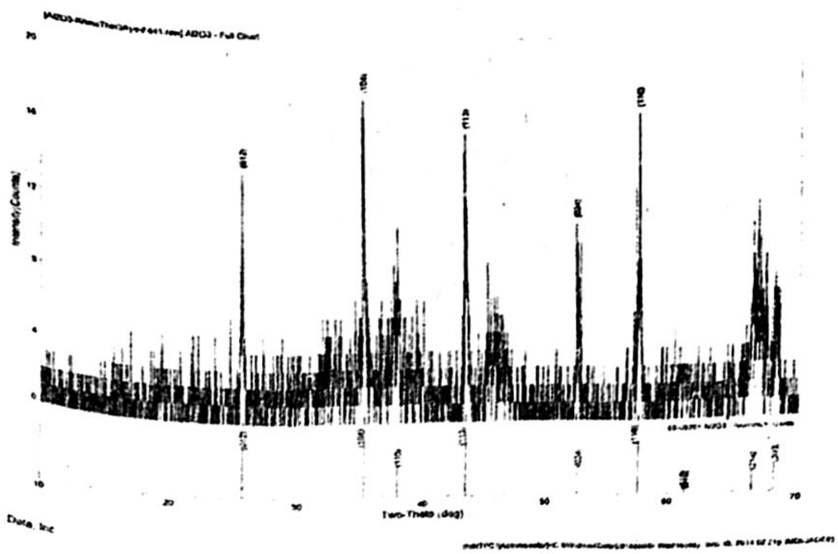


Figure 1. XRD diffractogram of alumina nanoparticles from coal fly ash

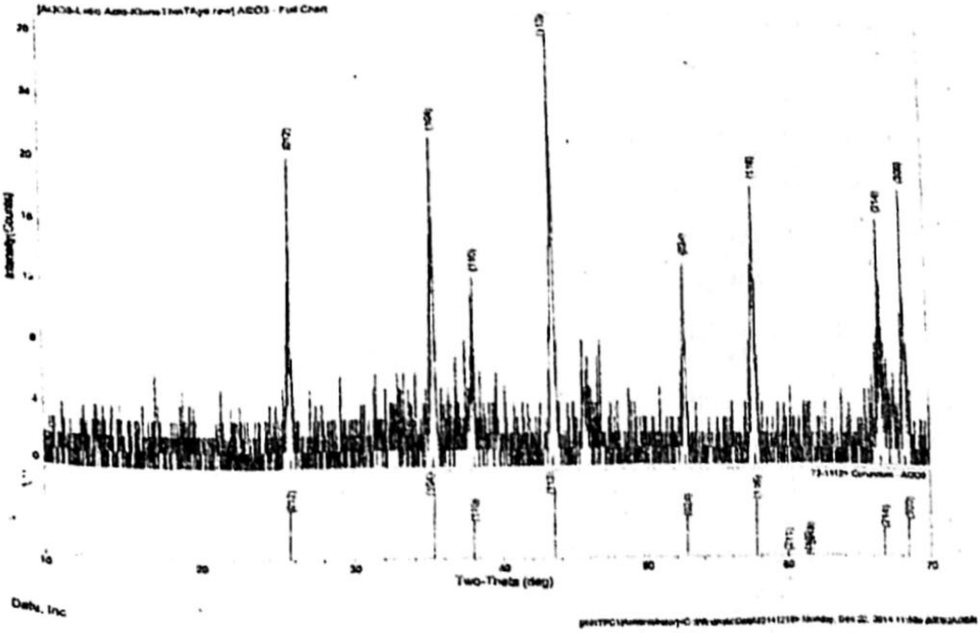


Figure 2. XRD diffractogram of alumina nanoparticles from aluminium nitrate

Field emission scanning electron microscopic measurements of alumina nanoparticles from coal fly ash and aluminium nitrate

The field emission scanning electron microscopic (FE-SEM) measurement was carried out EVO 60, Brand: Card ZESIS instrument in order to analyze the structure and morphology of synthesized samples. FE-SEM was used to indicate the morphology. From the FE SEM image, the synthesized alumina nanoparticles sample is spherical in shapes for 3 hours calcinations in muffle furnance. The morphology and particles size of alumina nanoparticles after the calcination temperature at 1000° C were investigated by FESEM (Figures 3. and 4). It was found that the morphology of alumina nanoparicles look like spherical in shape. The particles size of alumina nanoparticles from coal fly ash is larger than that of alumina nanoparticles from aluminium nitrate.

Table 1. XRD Data of Alumina Nanoparticles from Aluminium Nitrate with Lactic Acid

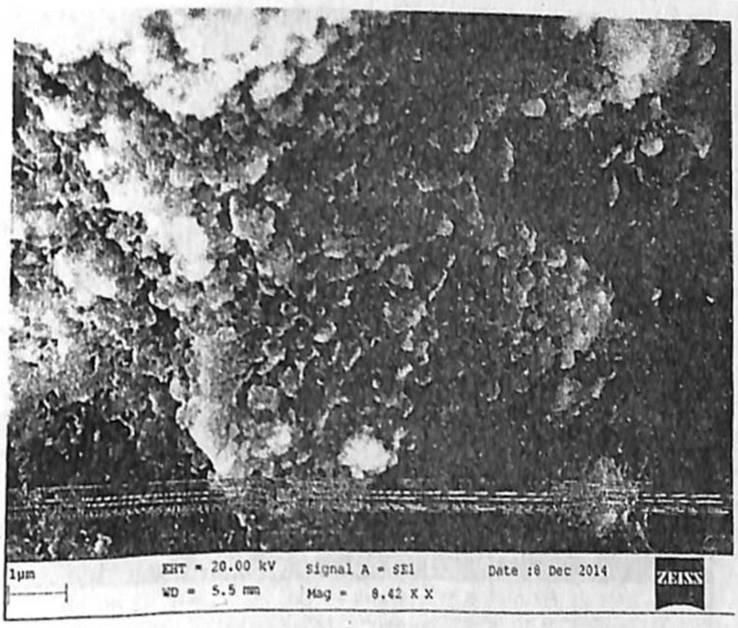
[Al2O3-Latic Acid-KhineThinTAye.raw] Al2O3 - Full Chart											Peak Search Report
SCAN: 10.0/70.0/0.02/0.06(sec), Cu(40kV,40mA), I(max)=28, 12/19/14 15:07											
PEAK: 17-pts/Quartic Filter, Threshold=1.0, Cutoff=0.0%, BG=1/0.5, Peak-Top=Summit											
NOTE: Intensity = Counts, 2T(0)=0.0(deg), Wavelength to Compute d-Spacing = 1.54056Å (Cu/K-alpha1)											
#	2-Theta	d(Å)	(h k l)	BG	Height	Height%	Area	Area%	FWHM	XS(Å)	P/N
1	25.768	3.4546	(0 1 2)	2	20	71.4	2.7	40.8	0.126	>1000	2.0
2	35.224	2.5458	(1 0 4)	2	21	75.0	5.0	76.7	0.224	417	2.1
3	38.006	2.3656	(1 1 0)	2	12	42.9	1.8	27.6	0.153	726	1.4
4	43.424	2.0822	(1 1 3)	1	28	100.0	6.5	100.0	0.205	477	2.6
5	52.678	1.7361	(0 2 4)	2	13	46.4	1.8	28.2	0.142	877	1.5
6	57.642	1.5978	(1 1 6)	2	18	64.3	4.0	61.0	0.211	487	1.9
7	66.585	1.4033	(2 1 4)	3	16	57.1	2.8	43.3	0.184	614	1.6
8	68.286	1.3724	(3 0 0)	2	18	64.3	2.6	39.6	0.137	>1000	1.9

Table 2. Average Lattice Constants Value of Alumina Nanoparticles from Aluminium Nitrate from XRD Data

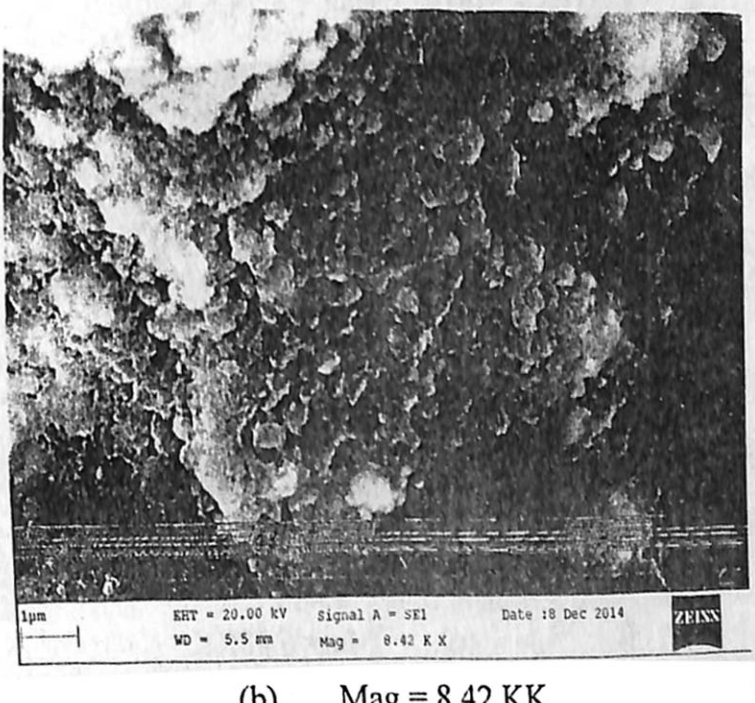
Lattice Constants from Peak Locations and Miller Indices

FILE: [Al2O3-Latic Acid-KhineThinTAye.raw] Al2O3 - Full Chart
SCAN: 10.0/70.0/0.02/0.06(sec), Cu(40kV,40mA), I(max)=28, 12/19/14 15:07
NOTE: [New Lattice Calculation] [Hexagonal]

2-Theta	d(Å)	h	k	l	a-Axis	b-Axis	c-Axis
25.768	3.4546	0	1	2	4.7047		13.0315
35.243	2.5445	1	0	4	4.7047		13.0315
43.424	2.0822	1	1	3	4.8725		12.0313
52.678	1.7361	0	2	4	4.7496		12.9538
57.642	1.5978	1	1	6	4.6980		13.0790
66.585	1.4033	2	1	4	4.8134		12.3453
				Average Lattice Constants =	4.7571		12.7454

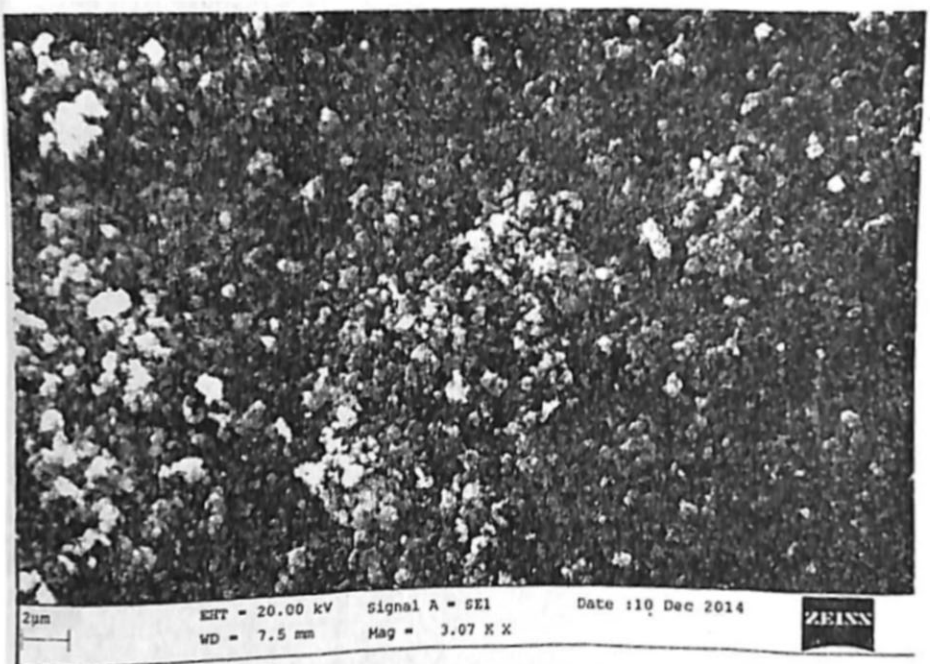


(a) Mag = 2.74 KK

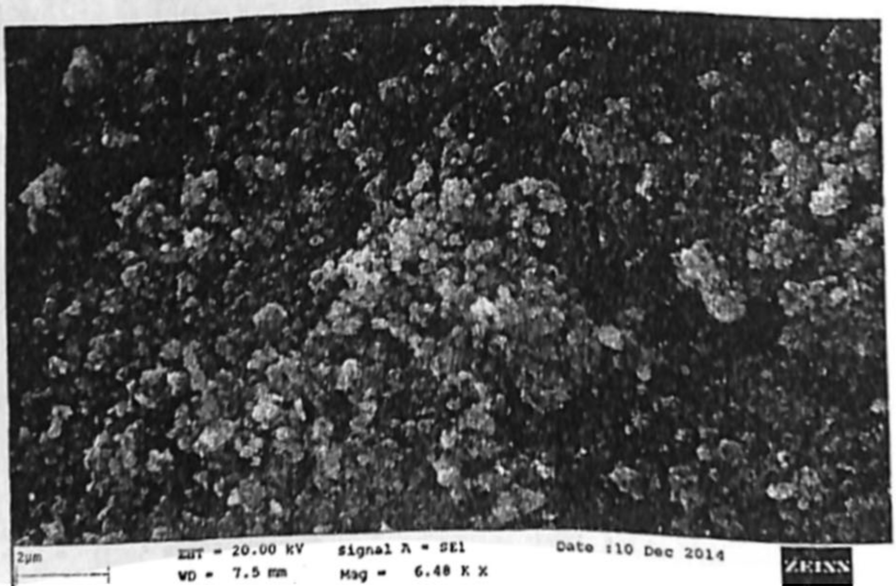


(b) Mag = 8.42 KK

Figure 3. Field emission scanning microscope images of alumina from coal fly ash (a) Mag= 2.74 KK (b) Mag= 8.42 KK



(a) Mag = 3.74 KK



(c) Mag=6.48KK

Figure 4. Field emission scanning microscope images of alumina nanoparticles from
aluminium nitrate with lactic acid
(a) Mag= 3.74 KK (b) Mag= 6.48 KK

Conclusion

In this study, alumina nanoparticles synthesized from coal fly ash are shown crystallite nature at 1000° C for 3 h. Alumina nanoparticles was synthesized from aluminum nitrate as the reference. According to lattice parameters (a=b=4.75 Å, c= 12.74 Å) the crystal structure of alumina nanoparticles was observed to be hexagonal. Alumina nanoparticles were found uniform in size and spherical in shape at different magnifications (2.74 KK, and 8.42 KK) by using field emission scanning electron micrograph.

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