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Greener Technology for Synthesis of Zeolite by Sonication and their Characterizations

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Abstract: Zeolite is major production aspect with their respective use in environmental science. Ultrasonic method is a green technology for synthesisof zeolite. The current study is focused on sustainable development of green method for the synthesis of zeolite in greener technology i.e. sonication. Coal fly ash was used to synthesis by alkali fusion and aging by mechanical stirrer.The curing step is supplanted by ultrasonic method. The synthesized zeolite was characterized by classical weight chemical analysis, scanning electron microscopy (SEM), X-ray diffraction (XRD).

Key Words: Zeolite, Sustainable development, Greener Technology, Sonication, Coal fly ash.

I.INTRODUCTION

Coal fly ash remains is produced by thermal power plants have been enormous measure of dump material expanding all through the world. The dumping of such an immense amount of coal fly ash has turned into a land possessed issue. It is more than 112 million tons of fly ash debris is being created yearly in India [1] with more than 80,000 sections of land of land being involved by ash pond [2]. Coal fly ash for the most part utilized as building materials and in other structural designing works. Mass and agglomerate use is traditionally separated as takes after [3].

• Agglomerate applications, for example cement and Solid assembling, creation of blocks and Light weight aggregates and refractory materials,and additives for the ceramic industry.

• Bulk applications, such as road and rail bases, Pavements, land filling in mining activities, and soil amendmentmaterial.

Other than above utilization of fly ash debris there is a standout among the most essential use of fly ash is to be utilized as a part of synthesis of zeolites it is a standout among the most vital gatherings of inorganic materials[4]. Zeolites are microspores crystalline material which is aluminosilicate tetrahedral units with standard structure comprising of sub-atomic measured pores and channels [5].

The creation of both naturally and synthetically zeolite are essential use in environmental benign i.e. heterogeneous catalysis and all the more as of late in a few developing fields, for example, wellbeing and medication concerning their employments. Zeolites have an amazingly high significance in mechanical applications adsorbents, ion exchangers and catalysis. Specifically, the quantity of uses of zeolites as profoundly dynamic, particular and stable impetuses in expansive scale advances relentlessly increments. They incorporate oil refinery, petrochemistry, generation of fine chemicals and environment catalysis [4].

In current study the ultrasonic method (Sonication technique) is use for synthesis of zeolites, to enhance the crystallite size of zeolites and reduce curing time [6]. In this study, how coal fly ash effortlessly converted to zeolite, in concerning with physicochemical characteristics. Coal fly ash is use

as effective silica source for synthesis of zeolite. Coal fly ash has pretreatment as like sieving for evacuate bigger particle, calcination for expelling if present any volatile material, Mineral acid treatment for enhance activity[2]. On considering green methodologies we need to utilize ultrasonic waves for synthesis of zeolite. Ultrasonic method recognizes new technique for synthesis of zeolite. Ultrasonic is acoustic energy in the form sound waves having frequency more than 20,000 Hz.

II. Experimental:

II. 1 Materials:

The crude raw material of coal fly ash samples were collected from thermal power plant Nagpur, India.Table 1 presents the physicochemical properties of the coal fly ash samples utilized as the part of present investigation. As can be seen from this table, the coal fly ash samples used were of 'Class F' type with SiO2, Al2O3 and Fe2O3 as the major constituents.

II.2 Synthesis of zeolite by ultrasonic methods:

Ultrasonic method is use for synthesis of zeolite, ultrasonic wave are extents from more than 20 KHz. The present study for synthesis of zeolite utilizes coal fly ash. It was mixing with sodium hydroxide in nickel crucible and fuse on Bunsen burner, the resultant fuse mass is obtained. The fused mass is cooled at ambient temperature, grinding and pour into conductivity water to make slurry. The slurryalong these obtained was agitated using mechanical stirrer (Remi, India) at 400 rpm for 1 hour. It sonicated (FRITSCH Analysette, Germany) by ultrasonic waves at 80 db for 10 min. with steady stirring.The flow chart of synthesis of zeolite process is shown in figure No.1

3.1.1a Spectrophotometry analysis of SiO_2:

Silicic acid react with a solution of molybdate in the acidic medium to give an intense yellow coloration due to the formation of complex silicomolybdic acid H4(SiMo12O40) which can be measured at 400 nm wavelength. It is better to reduce the complex acid to molybdenum blue. A solution of Ascorbic acid is generally used after reduction, the absorbance can bemeasured at 810 nm wavelength. Compute the percentage of SiO2 from the standard graph.

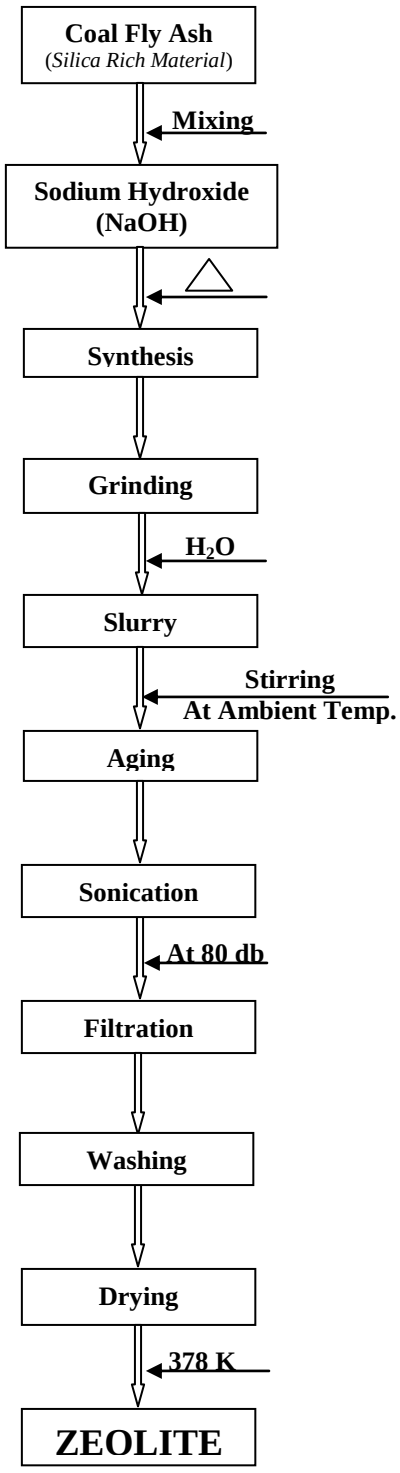


Fig. 1: Flow Chart for Synthesis of Zeolite.

3.1.1b Spectrophotometry analysis of TiO_2:

Take an aliquot equivalent to 0.1 gm, include 5 ml phosphoric acid and 5 ml 1:1 sulphuric acid (final acidity may vary from 105 to 3.5 N), add 5 ml dilute hydrogen peroxide. A yellow color will develop. Make up the volume to 100 ml in a volumetric flask and measure the absorbance in a spectrophotometer (SHIMADZU, Japan) at 410 nm. Compute the percentage of SiO2 from the standard graph.

3.1.2: Titrimetric analysis

3.1.2a Determination of Al_2O_3 (EDTA Complexometry):

The pH of the principle aliquot is acclimated to a suitable range (5.5). An excess of EDTA solution added and boiled to form a stable complex. After cooling, the excess of EDTA is determined by titrating with a solution of zinc acetate and the percentage of Al_2O_3 is calculated from the amount of actual EDTA used up.

3.1.2b Determination of Fe_2O_3 (Volumetry):

The main aliquot is disintegrated with concentrated hydrochloric acid, and iron is reduced to its divalent state with stannous chloride. The excess of reductant is removed by oxidation with mercuric chloride. Ferrous iron is determined by sulphuric-phophoric acid medium, employing diphenylamine barium sulphonate as indicator.

3.1.3: Determination of Na_2O, MgO and CaO:

Take 0.1 gm of sample in a platinum crucible and add 3 gm of Boric acid mix well and fuse in muffle furnace, the temperature increasing gradually to the full temperature of the furnace. Keep in the molten condition for 15-20 minutes. Remove the crucible from the furnace and allow to cool. Dissolve the melt in 100 ml water in borosil beaker, add HCl (1:1) and boil for 15 minutes. Allow to cool at room temperature and make up in 250 ml volumetric flask. Mix thoroughly and allow to settle for about 2 hours. Decant the supernatant liquid through a dry funnel in a dry beaker and determine the Na_2O by flame photometry (flame photometer 128, Systronic).MgO and CaOis determine by Inductively Coupled Plasma Atomic Emission Spectrometer (ICP Thermo elemnetal, USA).

3.1.4 Determination of loss on ignition (Gravimetry): Weigh accurately one gram of sample into a weighed platinum crucible. Heat gently first and then at a gradually increasing temperature. Finally, ignite at 1000OC for 30 minutes, cool in a desiccators and weigh.

$$L.O.I = \{[(A-B) \times 100] / C\}$$

Where

A = initial weight in gm of the crucible with the sample.

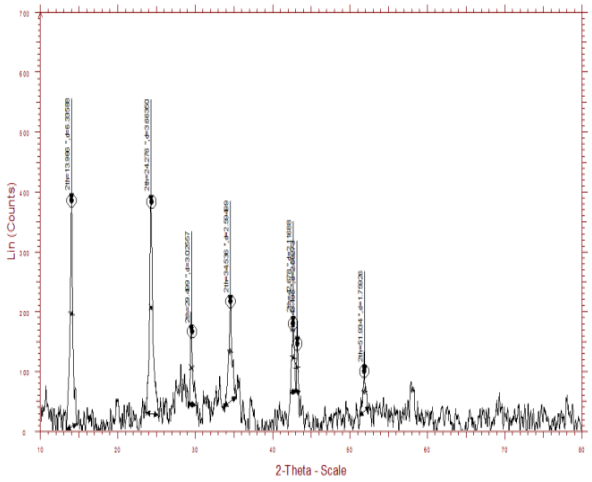
B = final weight in gm of the crucible with the residue after ignition.

and C = weight in gm of the sample taken.

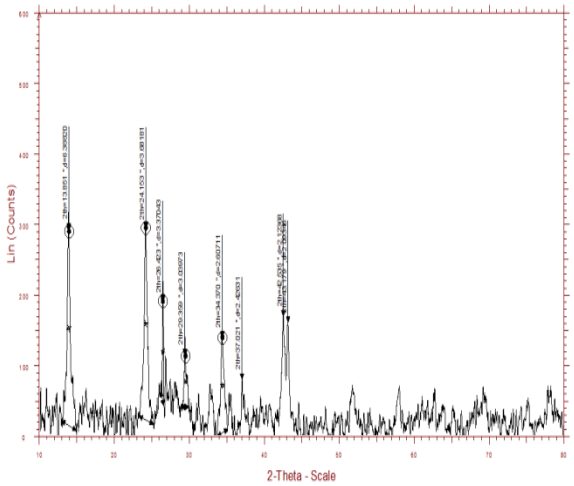
Table No. 1: Physicochemical characterization of coal fly ash and zeolite.

Samples	Conc.	Al_2O_3	Fe_2O_3	TiO_2	SiO_2	Na_2O	CaO	MgO	LOI
Coal Fly Ash	%	20.31	7.67	1.12	63.39	0.08	1.01	0.35	0.20
Zeolite	%	21.22	7.50	1.33	39.64	18.50	1.19	0.40	9.83

3.2 X-ray diffraction patterns (XRD) were recorded utilizing the X-ray diffraction (XRD) spectra of synthetic zeolites were obtained using Bruker AXS D8 advance analysis from 180^0 C to 430^0C measuring circle diameter 435, 500 and 600mm pre define angle range 360^0 max. usable angular range 3^0 to 135^0 x- ray source Cu , wavelength 1.5406 A^0 detector Si(Li) PSD**.** The samples were scanned from 10–50° (2θ, where θistheangle of diffraction). Various crystalline phases exists in the samples were identified with the help of JCPDS(Joint Committee on Powder Diffraction Standards) files for Inorganic compounds. Quantitative measure of thecrystallinity of the synthesized zeolite was made by using the summed heights of major peaks in the X-ray diffract were ascertained.



(a)

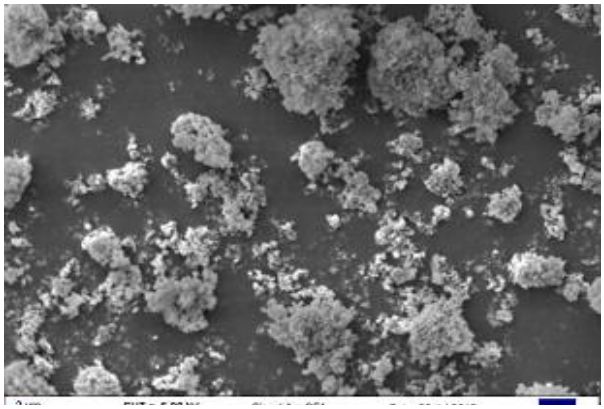


(b)

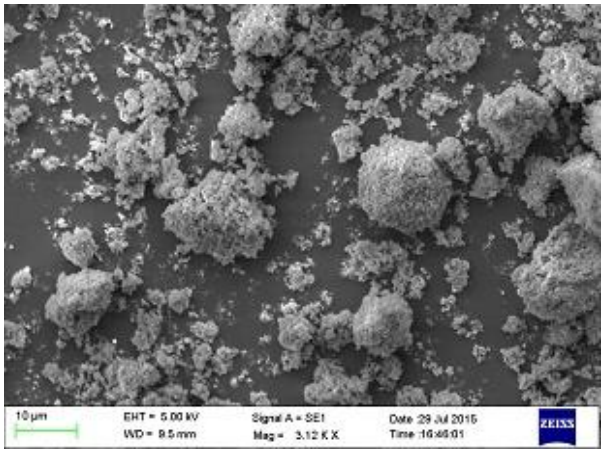
FigureNo. 2: XRD patterns of a) coal fly ash and b) Zeolite

3.3 Scanning electron microscopy (SEM)

Scanning electron microscope was performed on ZEISS. SEM experiments were directed to explore of the morphology of coal fly ash and zeolite structure Fig. 2 a and 2b. showsSEM micrographs of the coal fly ash at 6.32KX and Zeolite at 3.12 KX.



(a)



(b)

Figure No. 3: Morphological study by SEM of a) Coal fly ash b) zeolite

3. Conclusion:

From physicochemical characterization studied that, the ratio of elementsAl:Si:Na contain in zeolite is 1:2:1.The above information demonstrated that unadulterated and crystalline zeolite was effectively synthesized by a method utilizing ultrasonic assisted at curing step. The zeolite is characterized by classical wet chemical analysis, SEM and XRD.

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