

## RESEARCH ARTICLE

# Synthesis of Valuable Zeolitic Material from Coal Fly Ash as Industrial Waste, its post modification and Characterizations

Kapure GP

Department of Physics, S.G.B. College, Purna (Jn) Maharashtra, India- 431 511

Email: [gpk\\_2010@rediffmail.com](mailto:gpk_2010@rediffmail.com).

## Manuscript Details

Received : 17.06.2017

Revised :01.08.2017

Accepted: 05.08.2017

Published: 06.08.2017

ISSN: 2322-0015

Editor: Dr. Arvind Chavhan

## Cite this article as:

Kapure GP. Synthesis of Valuable Zeolitic Material from Coal Fly Ash as Industrial Waste, Its Post modification and Characterizations. *Int. Res. Journal of Science & Engineering*, 2017, 5 (4): 49-58.

© The Author(s). 2017 Open Access

This article is distributed under the terms of the Creative Commons Attribution 4.0 International License

(<http://creativecommons.org/licenses/by/4.0/>), which permits unrestricted use, distribution, and reproduction in any medium, provided you give appropriate credit to the original author(s) and the source, provide a link to the Creative Commons license, and indicate if changes were made.

## ABSTRACT

Coal fly ash (CFA) is byproduct which can be used to synthesize X type zeolite followed by hydrothermal treatment. The utilization of CFA for synthesis of zeolite not only solves environmental problems but also use to form a value added materials. The conversion of CFA into zeolite which is useful for protection of echo friendly environment. Depending on the calcinations temperature, time and chemical composition, the zeolite NaX have been successfully synthesized. The crystallinity of the prepared zeolite NaX was found to change with fusion temperature and a maximum value was obtained at 823K. The synthesized zeolite was characterized by XRD, SEM, FTIR, and BET techniques.

The results show that the ion exchange capacities of the treated fly ash in the forms of zeolite NaX, for Cs<sup>+</sup> ions relative to those of the commercial zeolite NaX. Our results suggest that the treated CFA may be potentially useful in ion-exchange waste-water treatment etc.

**Keywords:** Coal Fly ash (CFA); Zeolite NaX; Hydrothermal treatment; Post modification; XRD; SEM; FTIR; and BET techniques.

## INTRODUCTION

In recent years, various environmentally unsupportive processes in synthesis of bulk and fine chemical industries are being replaced with cleaner catalytic processes. In many industrial processes such as petro chemistry and the manufacture of organic chemicals Solid acid heterogeneous catalyst have been playing an important role as green catalysts and attracted special interest owing to their special features such as shape selectivity, controlled variability, their reusability and eco-friendly nature [1].

Zeolite is an important class of micro porous material that exhibits excellent molecular sieving capabilities for ion-exchange, catalytic reaction, adsorption, and it is utilized in various processes for environmental protection. Zeolites and zeolite-like materials are micro porous, crystalline materials composed, primarily, of  $[\text{SiO}_4]_2^-$  tetrahedra connected to form a framework with 1-, 2-, or 3-dimensional network of pores ranging in diameter from 2 to 10 Å

Zeolite crystallization represents one of the most complex structural and chemical problems in crystallization phenomena. Formation under conditions of high metastability leads to a dependence of the crystallization of a specific zeolite phase found under equilibrium conditions on a number of variables in addition to the reaction mixture, temperature and time. Fyfe reported the higher probability of the crystallization of the metastable phase from highly reactive gel mixtures.

In such a system, the initial maximum free energy excess must be high with respect to the final stable state. Therefore, the each component in the reacting mixture contributes specific characteristics of the gel and final material obtained. In many instances achieving the right gel often becomes the initial requirements for the crystalline products.

In this study we have repeated the synthesis of zeolite using waste material instead of general chemicals used. Therefore in the first few attempts of synthesis we have established the synthesis producer only using pure chemicals and then same system is adopted by the use of Coal Fly Ash (CFA) as one of the source of silica [2-3] and number of attempts have been used for crystallization of the NaX zeolite by using CFA. The CFA obtained from thermal power station located near to the Nanded district i.e. One at Parli TPD and Chandrapur TPD. Chandrapur TPS is largest thermal power generation plant out of five Thermal Power stations in India. It was observed that we obtained highly crystalline and phase purity of NaX even though we use the silica and alumina from CFA instead of chemicals. Hence the utilization of CFA for synthesis of zeolite not only solves environmental problems but also use to form a value added materials. Synthesis of microporous materials is one of the potential applications of waste fly ash to obtain high value industrial products with environmental technology utilization. The synthesis of such materials from fly ash is of great significance from fundamental as well as commercial point of view which is quite evident from the fact that several researchers are working on this research topic all over the world.



TPS at Parli (MS)



TPS at Chandrapur (MS)

Fig. 1: The Parli TPS is the major electricity generation plant in our Marathwada region as shown below  
Coal Fly Ash as Source of Silica:

CFA collected from Parali and Chandrapur TPS. Powder X-ray diffraction patterns were obtained using Cu-K $\alpha$  radiation with a Rigaku Desktop Model. The XRD peak information is important to quantity changes in the composition of Quartz and Mullite reactants that affecting reaction conditions of hydrothermal synthesis of zeolite and reaction products. After the complete analysis of Collected Fly Ash it was decided to prepare NaX zeolites due to their structural peculiarities and cation exchanges properties, they have large Industrial and Eco friendly applications.

Crystallization of the zeolitic materials occurs through nucleation reaction and crystal growth. Nucleation again depends upon the alkalinity. Thus, crystallinity of the resultant products is a function of the alkalinity<sup>4</sup> of the reaction mixture. The crystallinity of synthesized zeolite increases with increase in NaOH/fly ash ratio up to a certain value after which it shows a decreasing trend. As mentioned earlier, alkali present in the fusion mixture reacts with the silica and alumina present in the fly ash and form silicate and aluminate salts.

The mineralogical study indicated the presence of mullite and quartz [5-6] as major reactive phases during zeolite synthesis from fly ash after alkali activation.

## EXPERIMENTAL

### *Synthesis of zeolite NaX without coal fly ash (CFA)*

Zeolites are crystalline hydrated aluminosilicates having rigid three-dimensional infinitely extended framework structure. It encloses the cavities and channels of molecular dimensions. The framework structures are built from corner sharing of all four oxygens of the [SiO<sub>4</sub>]<sup>4-</sup> and [AlO<sub>4</sub>]<sup>5-</sup> tetrahedra as the primary-building units [7]. The general empirical crystallographic unit cell formula of zeolite is expressed as (1) M<sub>x</sub>/n [(AlO<sub>2</sub>)<sub>x</sub> (SiO<sub>2</sub>)<sub>y</sub>]<sub>z</sub> H<sub>2</sub>O Where M is charge compensating cations of valence n, x and y represent the number of mol's of AlO<sub>2</sub> and SiO<sub>2</sub> respectively, where y x and z is the number of water molecules.

The Si-O bond in silicate tetrahedron has 50% covalent character and the bond is very much short. Therefore even at standard temperature and pressure and easily in hydrothermal conditions, these tetrahedra can share vertices to form large varieties of polymeric zeolite structures. The [AlO<sub>4</sub>]<sup>5-</sup> tetrahedron is also isostructural

[8] with [SiO<sub>4</sub>]<sup>4-</sup> units and Al-O bond length is 1.75 Å. The most of the natural zeolites are aluminum isomorphs. Therefore zeolite is one of the most interesting classes of tectosilicate having open framework in which [AlO<sub>4</sub>]<sup>5-</sup> is partially substituted for [SiO<sub>4</sub>]<sup>4-</sup> in silicate polymorph. In nature, Al substitution up to an Si/Al ratio of about 1:1 is observed. Al avoids Al-O-Al bonds as per Loewenstein's rule (2), therefore Al substitution generally. Crystallization of zeolite is usually performed from the system containing sources of silica, alumina, an aqueous base and in some cases an organic compound such as quaternary ammonium compound or an amine [9-11]. Zeolite X has been synthesized with/without organic template and in pure sodium form or mixed cation form.

The number of patents were filed with the various claims and compositional changes and ingredients.

Zeolite NaX is conventionally formed as an aluminosilicate from as



Where M is charge compensating cations of valence n, x and y represent the number of mol of AlO<sub>2</sub> and SiO<sub>2</sub> respectively, where y x and z is the number of water molecules. The Si-O bond in silicate tetrahedron has 50% covalent character and the bond is very much short (1.62 Å). Therefore, even at standard temperature and pressure and easily in hydrothermal conditions, these tetrahedra can share vertices to form large varieties of polymeric zeolite structures. The [AlO<sub>4</sub>]<sup>5-</sup> tetrahedron is also isostructural with [SiO<sub>4</sub>]<sup>4-</sup> units and Al-O bond length is 1.75 Å. Most of the natural zeolites are aluminum isomorphs. Therefore, zeolite is one of the most interesting classes of tectosilicate having open framework in which [AlO<sub>4</sub>]<sup>5-</sup> is partially substituted for [SiO<sub>4</sub>]<sup>4-</sup> in silicate polymorph. In nature, Al substitution up to an Si/Al ratio of about 1:1 is observed. Al avoids Al-O-Al bonds as per Lowenstein's rule (2), therefore Al substitution generally does not occur below the ratio of 1:1.

The compositions can be prepared by utilizing the materials [12], which supply the appropriate oxides. Zeolite NaX is a metastable phase meaning that other types of zeolite phases such as P, A or sodalite may form if this recipe not followed carefully.

**Synthesis of zeolite NaX with coal fly ash (CFA):**

The present section is concerned with the utilization of coal fly ash for synthesis of value added product like Zeolite NaX. The fly ash used zeolites have wide applications in ion exchange, as molecular sieves, catalysts, and as adsorbents. The collected CFA from two different TPS contained both amorphous SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and crystalline components (quartz and mullite). The compositional similarity of fly ash to the naturally occurring zeolites started the work on the synthesis of zeolites. After this initial work, many patents and technical reports have appeared on different methods of synthesis of zeolite from fly ash and also its several applications were proposed. The physico-chemical properties of the coal fly ash samples discussed here.

**Prior treatment to coal fly ash (Fuse method)**

There are three phases in fly ash from which the aluminum and silicon come from

- 1) Amorphous aluminosilicate glass,
- 2) Quartz, and
- 3) mullite.

The aluminosilicate glass phase is the largest and most unstable of these phases in the hydrothermal environment, and therefore has the highest rate of dissolution. The amounts of the main components of ash viz. both amorphous (mainly SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>) and crystalline components (mainly quartz and mullite) show few variations with the type of coal and its decided type. The fly ash used present investigation is Type "F". The treated Fly ash collected from

Chandrapur TPS and Parali TPS was screened by sieve of 80-mesh size to eradicate the larger particles. The unburnt carbon along with other volatile materials present in CFA was removed by calcination at 800°C for 5h in muffle furnace (shown in Fig 2.) CFA was then treated with dilute hydrochloric acid to remove iron to a certain extent (if any) or magnetically stirred thereby increasing the activity, thermal stability and acidity of the zeolite.

Therefore, before any synthesis treatment, the sample of fly ash (calcined and sieved) was milled and fused in silica crucible with sodium hydroxide in different ratio at 550°C for 1 hour at controlled heating rate (1°C/ min) and cooled by natural convection at room temperature in fused material grind. The ratio of activation solution to coal fly ash (i.e. NaOH/SiO<sub>2</sub>) along with optimization of temperature, pressure and reaction time results in the formation of various types of zeolite material. An amorphous SiO<sub>2</sub> component in the CFA was used as Si-source for the further investigations.

**Chemical Composition of Coal Fly Ash:**

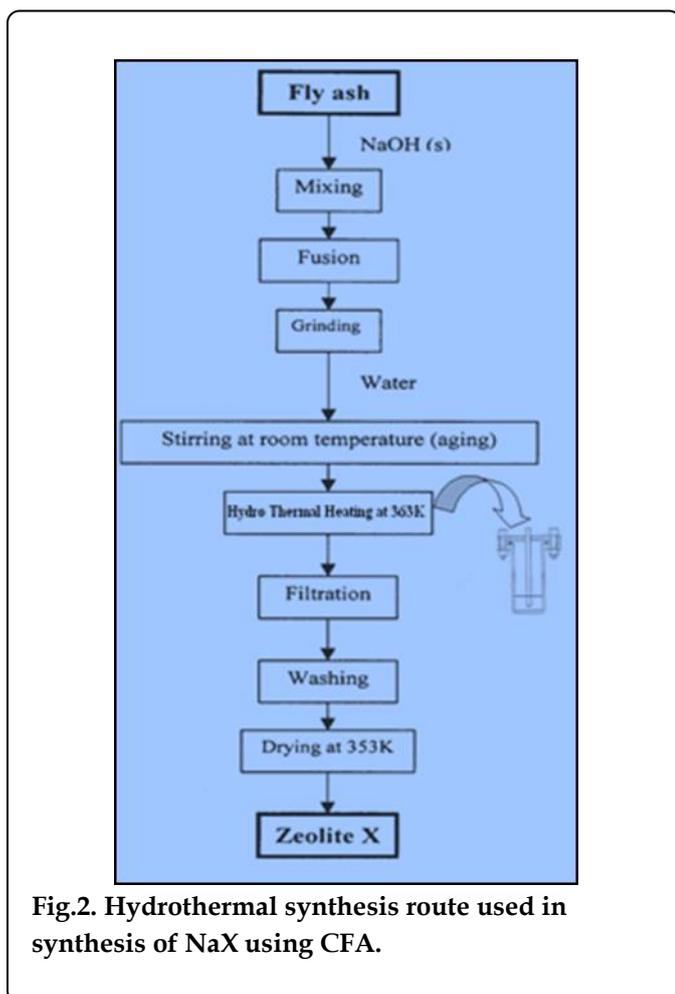
The Chemical compositions of fly ash used in the present study are given in Table 2. The chemical composition of samples was established by estimating EDX as well as silicon by gravimetric [13-15]. The qualitative and quantitative analysis results of fly ash, obtained using the FP method, are shown Table 1. A 3g power sample was directly inserted into a powder-sample container with 5 µm polypropylene adhered to the base.

**Table 1: Chemical Analysis of CFA obtained from Chandrapur and Parli TPS as:**

Chandrapur Thermal Power Station on % weight		Parali Thermal Power Station On % Weight	
Fly Ash Content	In Percent	Fly Ash Content	In Percent
Silica (SiO <sub>2</sub> )	65.20	SiO <sub>2</sub>	70.10
Alumina(Al <sub>2</sub> O <sub>3</sub> )	21.37	Al <sub>2</sub> O <sub>3</sub>	20.18
Iron Oxide (Fe <sub>2</sub> O <sub>3</sub> )	6.68	Fe <sub>2</sub> O <sub>3</sub>	7.0
(TiO <sub>2</sub> )	2.12	Na <sub>2</sub> O	0.23
Lime (CaO)	1.56	CaO	1.42
Magnesia (MgO)	0.69	MgO	0.32
Sodium Oxide (Na <sub>2</sub> O)	0.24	K <sub>2</sub> O	0.40
Potassium Oxide (K <sub>2</sub> O)	0.56	Mn <sub>3</sub> O <sub>4</sub>	0.20
<b>Total</b>	<b>99.47</b>	<b>Total</b>	<b>100.05</b>
SiO <sub>2</sub> /Al <sub>2</sub> O <sub>3</sub> =70.10/20.18 = 3.47 On Molebasis Si/Al=1.73Na <sub>2</sub> O/Al <sub>2</sub> O <sub>3</sub> =0.11		SiO <sub>2</sub> /Al <sub>2</sub> O <sub>3</sub> = 65.20/21.37 =3.05 On Molebasis Si/Al=1.52Na <sub>2</sub> O/Al <sub>2</sub> O <sub>3</sub> =0.12	

The estimation procedures followed are as under: The 0.5 to 1 g of fly ash was weighed accurately in a pre-weighed platinum crucible. The sample was then ignited on the burner till constant weight. From the difference in the weights, % loss on ignition was calculated. The ignited sample was moistened with the few drops of sulphuric acid to which 5-ml hydrofluoric acid (48 % electronic grade) was added. It was then slowly evaporated till the hydrofluoric acid completely removed [16]. The HF treatment was repeated thrice. After the last HF treatment and removal of  $H_2SO_4$ , the sample was ignited strongly on burner. Finally, %  $SiO_2$  was estimated from the difference in the weights (before and after HF treatment).

In the zeolite synthesis method the fly ash precipitates were fused with sodium hydroxide (NaOH) or (KOH) in a different (say 1:1.2, 1:1.5, 1:2 etc.) ratio at 500 to 600°C for about 1-2 h.



The fused product was then mixed thoroughly with distilled water and the slurry was subjected to aging for different hours. After aging the slurry was subjected to crystallisation at 70 to 90°C for 0 h to 12 h. The solid product was recovered by filtration and washed thoroughly with deionised water until the filtrate had a pH of 10-11. The product was then dried at a temperature of 80°C. The mechanism for zeolite synthesis from fly ash in a batch hydrothermal synthesis process has three stages;

- 1) The dissolution of aluminum and silicon from fly ash,
- 2) The deposition of aluminosilicate gel on ash surface,
- 3) The crystallization of zeolite from aluminosilicate gel

In addition to these we also used ultrasonic treatment for precipitated reaction gel to enhance crystallization rate.

## RESULTS AND DISCUSSION

### XRD Pattern of Collected Fly Ash :

The different minerals have different unit cell composition therefore XRD technique allows for qualitative identification of the phases present in the collected mineral. From the fig. 3, these XRD patterns it can be seen that the major crystalline phases found in the coal fly ash are common mineralogical phases such as quartz, mullite [17] and aluminosilicate glass amorphous material forming during the combustion process.

Fly ash is mainly composed of some oxides derived from inorganic compounds, which remain after combustion of the coal. The amounts of the main components of ash viz. both amorphous (mainly  $SiO_2$ ,  $Al_2O_3$ ) and crystalline components (mainly quartz and mullite) show few variations with the type of coal. The Chemical compositions of fly ash used in the present study are given in Table 1. The compositional similarity of fly ash to the naturally occurring zeolites started the work on the synthesis of zeolites from this waste material. After this initial work, many patents and technical reports have appeared on different methods of synthesis of zeolite from fly ash and also its several applications were proposed. The ratio of activation solution to coal fly ash (i.e.  $NaOH/SiO_2$ ) along with optimization of temperature, pressure and reaction time results in the formation of various types of zeolite material.

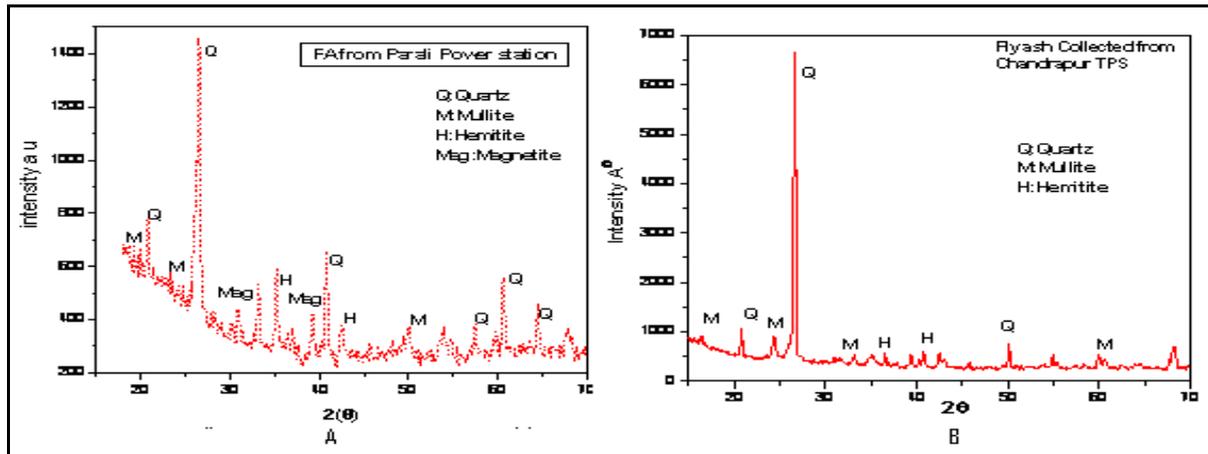


Fig. 3 (a,b): XRD of CFA from Parli and Chaddrapur TPS

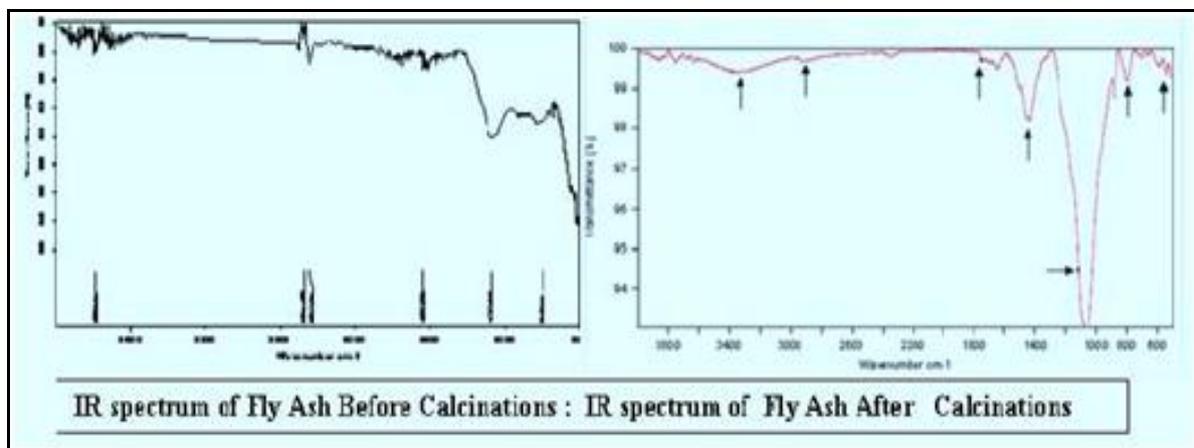


Fig. 4: IR Of CFA Before and After Calcination

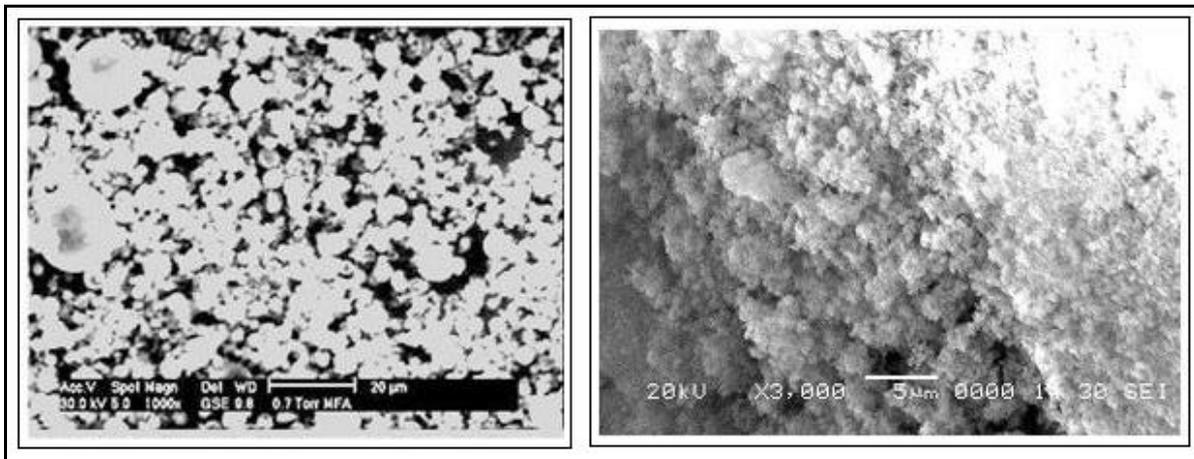


Fig. 5 : (SEM) of the original and treated CFA

#### Infrared spectroscopy:

FTIR spectra provide valuable information about the basic characteristics of the molecule, namely, the nature of atoms, their spatial arrangement and their chemical linkage forces. Infrared spectroscopy [18-20] has been

extensively used for identifying the various functional groups of the support, as well as identifying the various functional groups of the active component. The mid infrared region of the spectrum contains the fundamental frame work vibration of  $\text{Si(Al)O}_4$  grouping.

The absorption band in between the wave numbers 980-1320 cm<sup>-1</sup> in IR spectrum of fly ash and treated fly ash represent the presence of substituted Al atoms in the tetrahedral forms of silica frame work.

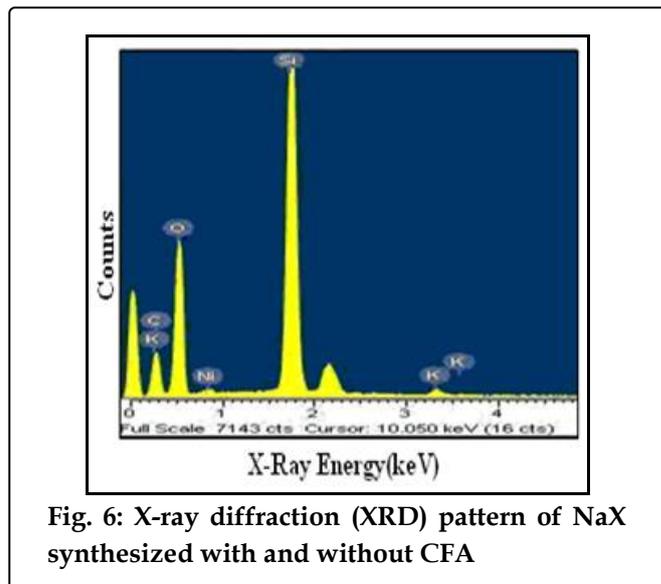
**SEM:**

For SEM analysis, solid fly ash samples (from Parali and Chandrapur TPS) were set into an epoxy resin and polished to give a smooth cross section of particles for the morphological structure of the raw fly ash, the bulk composition was also estimated from SEM by indirect method. The elemental composition of samples was first determined from SEM and from these data the percentage of oxides was calculated. It can be seen that there is significant chemical and mineralogical variation between different ash particles and even within a single particle [21].

EDS of CFA particles show strong concentrations of Oxygen, Aluminium and Silicon, with lower levels of sodium, magnesium, potassium, calcium, titanium, nickel and iron as depicted in EDS spectrum Fig.

**The effect of Fusion Temperature :**

The properties of synthesized zeolite NaX are also affected by fusion temperature (FT). The effect of fusion temperature on SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> ratio was studied, the ratio first increases with fusion temperature and then attains the maximum value again it decreases with increase in the temperature. During fusion, the silica and alumina present in the fly ash react with the NaOH and form Na-salts which is soluble in water. Therefore, crystallinity was also found to change with fusion temperature.



**Fig. 6: X-ray diffraction (XRD) pattern of NaX synthesized with and without CFA**

The X-ray powder diffraction (XRD) patterns of sample were recorded to ascertain the phase purity and also to detect the change in crystallinity during the different synthesis runs and modification treatments by ion exchange. The XRD patterns were collected over 2θ range of 5° to 40° using Ni filtered CuK (λ = 1.54041 Å) radiation by step scan with a 0.05° step and a 5.0 s. counting time, using a Rigaku D Max-III VC X-ray diffractometer. The most crystalline with no impurity sample was treated as parent or reference sample in the system. The degree of crystallization of the solid product was estimated from the formula

$$\% \text{ Crystallization} = \frac{\text{Peak Area}^* \text{ of the product}}{\text{Peak area}^* \text{ of the reference sample}}$$

(\*Peak Area of prominent peak between 2θ = 5-40°)

**Table 2 : XRD data of Na-X with chemically and without Chemically prepared**

NaX Chemically Prepared			NaX without Chemically prepared (with CFA)	
[hkl]	d(Å)	(I/I <sub>0</sub> )	d(Å)	(I/I <sub>0</sub> )
111	14.46	100	14.51	100
220	8.84	18	8.92	25
311	7.53	12	7.60	24
331	5.73	18	5.77	54
333	4.81	5	4.74	17
440	4.41	9	4.43	30
531	4.22	1	4.24	4
620	3.94	4	3.96	15
533	3.80	21	3.82	89
622	3.76	3	3.67	20
444	3.60	1	3.61	11
711	3.50	1	3.38	76

X-ray diffraction pattern for NaX (100 % crystalline) sample produced with chemically and they are compared with XRD data obtained by using coal fly ash in the Table. 2. The  $d$  ( $\text{\AA}$ ) values were found to be closely matches with the chemically produced.

#### Effect Synthesis Temperature on NaX crystalline:

The XRD pattern of three synthesis temperature 70, 80, 90°C is reported in the Figure 6. The percent crystallinity of the samples drawn at different three temperature 70, 80, 90°C was calculated based on the ratio of the sum of the areas of prominent peaks (with  $2\theta \cong 6.0, 9.9, 11.6, 15.3, 18.3, 20.0, 23.3, 26.6, 29.2, 30.3, 30.9, 31.9, 32.6, 33.6, 34.2$  and  $37.3$  values). These patterns show the typical progressive development of the zeolite NaX phase obtained from CFA after 8 h of crystallization period. The characteristic peaks of zeolite NaX starts appearing after the two hour and the fully crystalline phase is obtained at 8 hr. This unusual shorter crystallization period may be due to ultrasonic treatment given to reaction gel. Perhaps higher reactivity of the  $\text{SiO}_2/\text{NaOH}$  ratio at the time of fusion of Fly ash may also responsible for this.

The fig. 7 also illustrated that absence of impurity peaks and amorphous halo region in powder XRD profiles of NaX sample of 8 h. indicated highly pure and crystalline nature of the samples. This most crystalline sample in the system was arbitrarily assumed to be 100 % crystalline. It also observed from the powder XRD profiles that obtained characteristics peaks were closely matches with the chemically produced NaX data in above Table 3.

The obtained values crystallinity for 70, 80 and 90 °C were calculated and they are 30 %, 80 % and 100 % crystalline .

The Scanning Electron Microscope (SEM) picture of the samples obtained after 8 h at 90° C shown in Fig. 8. and it was found to be fully crystalline (free from amorphous impurities) as revealed by the XRD. The crystal habit is nearly spherical in shape with 2-3  $\mu\text{m}$  in size. The crystal morphology is often influenced by different synthesis parameters.

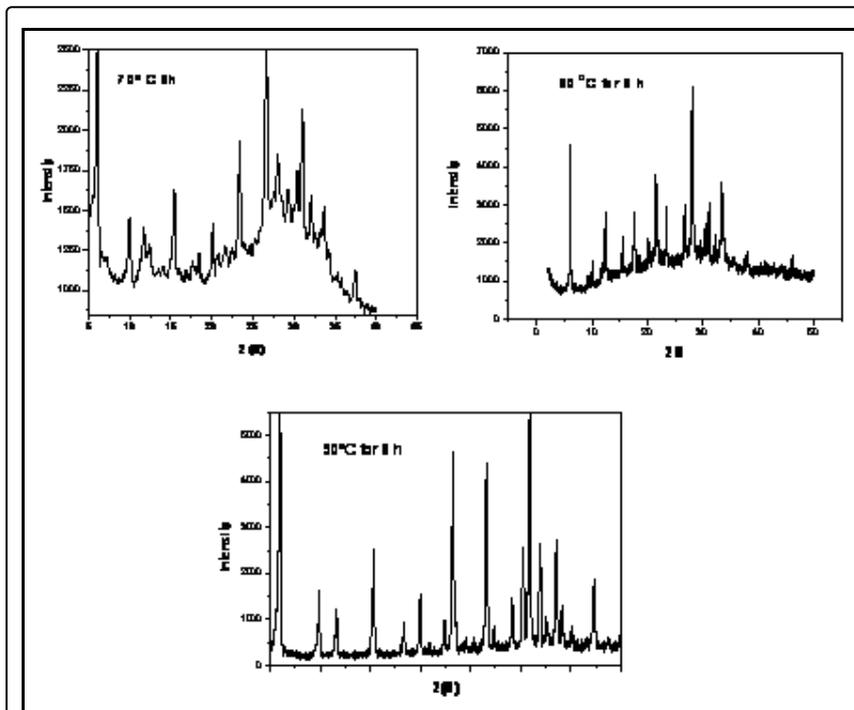


Fig.7: powder XRD of NaX at Temp.70, 80 and 90°C.

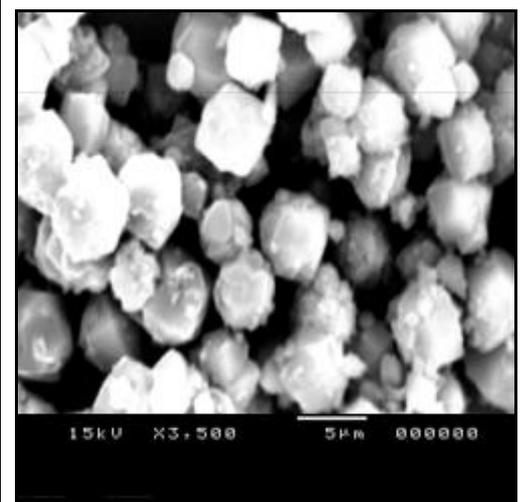


Fig. 8: SEM pictures of NaX zeolites with 100 % crystallinity

**Table 3: Surface Area Measurements**

% crystallinity	BET Specific Surface Area (m <sup>2</sup> ,g <sup>-1</sup> )	Micropore Volume (cc, g <sup>-1</sup> )	% crystallinity
Amorphous (0%)	60.5	0.023	Amorphous (0%)
30 %	290	0.10	30 %
80 %	645	0.240	80 %

**Surface Area Measurements:**

NOVA-1200 (supplied by Quatachrome Corporation U.S.A) high speed surface area and micropore size analyzer was used for the measurement of low-pressure nitrogen adsorption to determine the surface area [22-24]. The samples were degassed (activated) at 373 K for 8 h to make it free from contaminants such as water under the high vacuum. NOVA Enhanced Data Reduction Software Ver.3.70 was used to analyze adsorption data. It can be seen that as percent crystallinity increases specific surface area and micropore volume increases. This is in agreement that as the crystallinity increases more open structure is form.

**Infrared spectroscopy of NaX (IR):**

The most reliable alternative is infrared spectroscopy which can be use to identify the zeolitic phase, the structural details and crystallinity. Generally, the mid-IR region is examined because the absorbed water in the zeolite masks the OH region. The framework IR spectra were recorded using noel mull technique, with KCN as

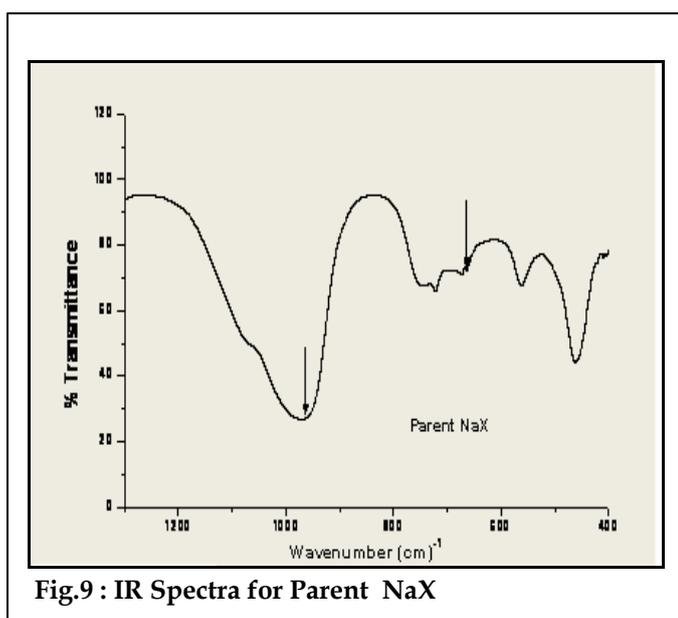
internal standard, for different crystallinity sample obtained in kinetics study of NaX zeolite are shown in Fig.9.

A broad and strong band around 1080 cm<sup>-1</sup>, 740 cm<sup>-1</sup> and 460 cm<sup>-1</sup> are characteristics of amorphous aluminosilicate [25]. As the type X zeolite lattice start to crystallize a band around 974 cm<sup>-1</sup> appear with increasing intensity and shifted to higher frequency. The main asymmetric stretch is at 974 cm<sup>-1</sup> with a shoulder at 1050 cm<sup>-1</sup> (in 100 % crystalline sample). The symmetric stretches are at 740 cm<sup>-1</sup> and 673 cm<sup>-1</sup>. The band near 560 cm<sup>-1</sup> is associated with the double six rings that connect the sodalite cages. A TO<sub>4</sub> bending vibration occurs at 465 cm<sup>-1</sup>. The impurities phases can also easily identified by IR.

**CONCLUSION**

All the characterization techniques performed in this study reveals that well ordered porous material of uniform hexagonal array can be synthesized very conveniently and in a very short span of time from an industrial waste coal fly ash. The degree of exchange was monitored by number of repeated ion exchange treatments at 368 K in each series.

The study provides an efficient porous catalyst from Flash ash which possesses higher catalytic activity and yield of the product to a greater extent. The phase purity and crystalline of Cs<sup>+</sup> the cation-exchanged sample was examined by powder XRD. Since the crystalline and characteristic peak position remained essentially same, the influence of extra framework cationic size on the change in the relative intensities of characteristic peaks was examined, as a function of nature and concentration. The remarkable advantages offered by this method are: catalyst is inexpensive, non-toxic, easy handling and reusable, simple work-up procedure, short reaction time, high yields of product with better purity and green aspect by avoiding toxic catalyst and hazardous solvent.

**Fig.9 : IR Spectra for Parent NaX**

## REFERENCES

1. Ferraiolo G, Zilli M, Converti A. Fly ash disposal and utilization, *J. Chem. Technol. Biotechnol.* 47, 281-305, (1990).
2. Carlson CL, Adriano DC. Environmental impact of coal combustion residue, *J. Environ. Qual.* 22, 227-247, (1993).
3. Gitari WM. Evaluation of the Leachate Chemistry and Contaminants Attention in Acid Mine Drainage by Fly Ash and its derivatives PhD Thesis, University of the Western Cape, South Africa, (2006).
4. Ciccù R, Ghiyani M, Muntoni S, Perreti A, Zucca A, Orsenigo R, Quattroni G. *International Ash Utilisation symposium, Center for applied energy research, University of Kentucky, (1999);* paper # 84
5. Tanaka H, Matsumura S, Furusawa S, Hino R, Conversion of coal fly ash to Na-X zeolites, *J. Mater. Sci. Lett.* 2002; 22, 323-325.
6. Keka Oza, Narayan C Pradhan; Amarnath Samanta, *Bull Matter science.*, 2004; 27:555-564.
7. Coal Resources of India S. Farooq Department of Geology AMU.
8. Adler KB, Mossman BT, Butler GB, Jean LM, Craighead JE, Interaction of Mount St. Helens' volcanic ash with cells of the respiratory epithelium. *Environ Res.*, 1984; 35:346-361.
9. Akematsu T, Dodson RF, Williams MG, Hurst GA. The short-term effects of volcanic ash on the small airways of the respiratory system. *Environ Res.*, 1982; 29:358-370.
10. Anderson HR. Health effects of air pollution episodes. In : Holgate ST, Samet J.M., Koren H.S., Maynard R.L. (eds) *Air pollution and health. Academic, San Diego*, 1999; 461-482.
11. Babolini G, Inferrera C, Girbino G, Grosso M. The clinical and morpho-anatomic aspects of the liparitis. In: Condorelli L., Teodori U., Anguissola A.B., Sangiorgi M. (eds) *XIV International Congress of Internal Medicine, Excerpta Medica, Rome*, 1978; 2: 829-838.
12. Baris YI, Artvinli M, Sahin AA *Environmental mesothelioma in Turkey. Ann NY Acad Sci.*, 1979; 330:423-432.
13. Baxter PJ. The eruption of El Reventador volcano 2002: health hazards and the implications for volcano risk management in Ecuador. Report to the *Pan-American Health Organization, Pan American Health Organization, Washington DC* (2003).
14. Baxter PJ, Ing R, Falk F, French J, Stein GF, Bernstein RS, Merchant JA, Allard J Mount St Helens eruptions, May 18 to June 12 1980. *J Am Med Assoc.*, 1981; 246: 2585-2589.
15. Baxter PJ, Ing R, Falk H, Plikaytis B Mount St. Helens eruptions : the acute respiratory effects of volcanic ash in a North American community. *Arch Environ Health*, 1983; 38:138-14316.
16. Baxter PJ, Woo G, Pomonis A Preliminary assessment of volcanic risk on Montserrat. *Montserrat Volcano Observatory, Montserrat*, p 32, (1998).
17. Beck JS, Vartulli JC, Kennedy GJ, Kresge CT, Roth WJ, Schramm SE. Molecular or Supramolecular Templating: Defining the Role of Surfactant Chemistry in the Formation of Microporous and Mesoporous Molecular Sieves, *Chem. Mater.* 1994; 6, 1816.
18. Beckett W (2000) Occupational respiratory disease. *N Engl J Med*, 1982; 342:406-413.
19. Fao Food Outlook, *Food and Agriculture Organization of the United Nations*, November, (2011).
20. Erikson S and Prior, M., "The Briquetting of Agricultural Waste for Fuel", *FAO Environment and Energy paper* 11, FAO of the UN, Rome. (1990).
21. Ekwall P. In *Advances in Liquid Crystals*; Brown G. H., Ed.; *Academic Press Inc.: New York*, p 1; (1971).
22. Francis W and Peters MC. *Fuel and Fuel Technology*", Pergamon Press Publishing, United Kingdom pages 101-106, (1965).
23. Forestry Residues, *Proceedings of the sixth Canadian Bio-energy R & D Seminar held out Richmond, British Columbia*, 16-18.
24. Grover PD. And Mishra, S.K. "Biomass Briquetting; Technology and Practices", *FAO of the UN, Bangkok*, (1996).
25. Mukhyonov, JP. "Fundamentals of Chemical Technology", *Mir Publishers, Moscow*, pages 200-201, (1986).

© 2017 | Published by IRJSE

### Submit your manuscript to a IRJSE journal and benefit from:

- ✓ Convenient online submission
- ✓ Rigorous peer review
- ✓ Immediate publication on acceptance
- ✓ Open access: articles freely available online
- ✓ High visibility within the field

---

Email your next manuscript to IRJSE

: [editorirjse@gmail.com](mailto:editorirjse@gmail.com)

---