



Synthesis of Zeolite Derived from Fly Ash Collected from Chandrapur Super Thermal Power Station by Alkali Fusion followed by hydrothermal treatment

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Abstract:

The quartz main crystalline phase of fly ash can be converted to pure Zeolite X-type at employed treatment conditions. The properties of zeolitic material formed strongly depended upon the treatment conditions and composition of the raw materials. FTIR spectra of zeolite composed of the peaks of vibration of framework resulted from the asymmetric stretching and bending vibrations of Si-O or Al-O bonds. XRD patterns of different synthesized zeolitic were scanned from 10.330 (2θ , where θ is angle of diffraction).

Keywords:

Zeolite, Zeolite -X, Coal fly ash, FTIR, XRD.

Introduction:

Fly ash is one of the residues generated in combustion, which comprises of fine particles exit from flue gases (that does not rise is termed bottom ash). Industrially, fly ash produced during combustion of coal, like in thermal plants that gets captured by electrostatic precipitators/particle filtration equipment before the flue gases reach chimneys of coal-fired power plants, and together with bottom ash removed from the bottom of the furnace is in this case jointly known as coal ash. The coal source/makeup/quality being burned, the components of fly ash vary considerably, but all fly ash includes substantial amounts of silicon dioxide (SiO_2) (both amorphous and crystalline) and calcium oxide (CaO), both being endemic ingredients in many coal-bearing rock strata.

The fly ash produced from burning pulverized coal in a coal-fired boiler is a fine-grained, powdery particulate material that is carried off in the flue gas and usually collected by means of electrostatic precipitators, baghouses, or mechanical collection devices such as cyclones. In general, there are three types of coal-fired boiler furnaces used in the electric utility industry. They are referred to as dry-bottom boilers, wet-bottom boilers, and cyclone furnaces. The most common type of coal burning furnace is the dry-bottom furnace. When pulverized coal is combusted in a dry-ash, dry-bottom boiler, about 80 percent of all the ash leaves the furnace as fly ash entrained in the flue gas. When pulverized coal is combusted in a wet-bottom (or slag-tap) furnace, as much as 50 percent of the ash is retained in the furnace, with the other 50 percent being entrained in the flue gas. In a cyclone furnace, where crushed coal is used as a fuel, 70 to 80 percent of the ash is retained as boiler slag and only 20 to 30 percent leaves the furnace as dry ash in the flue gas.





Coal is composed primarily of carbon and hydrogen, but all coal also contains some mineral matter (for example, clays, shales, quartz, and calcite); the percentage varies by coal type and source. Coal ash is the mineral matter that is collected after the coal is combusted, along with some unburned carbon. The amount of coal ash produced at a power plant depends on the volume of coal burned, the amount of mineral matter in the coal, and the combustion conditions.

Various methods of synthesis of zeolite from fly ash have so far been invented & patented some of the important techniques are alkali fusion followed by hydrothermal treatment slurry method. Fusion method is found to be the most efficient & a general method for synthesis of X-type Y-type & A-type from a large variety of fly ash. A modified fusion process to synthesize zeolite A & X from the fly ash. It was found that the addition of aluminium hydroxide to the fused fly ash solution followed by hydrothermal treatment at 60°C produced single phase zeolite A & X depending on the source of the ash received fly ash. The result confirms that the quantity of dissolved aluminium species is critical for the type of zeolite formed from fused fly ashes.

Material and Methods:

I-Material:

Zeolite synthesized from fly ash (from CSTPS, Chandrapur Super Thermal Power Station) by hydrothermal treatment. NaOH procured Ranbaxy. All reagents were analytically pure (99%) & used without purification.

II Zeolite synthesis method:

Mixture of NaOH (activator, adjusts sodium contents in starting material) & fly ash (pre-determined ratio) milled & fused in mortar and pestle at varied temp for 1 hr and cooled room temperature, grind further & added to water (10g fly ash / 100mL water). Slurry agitated mechanically in glass beaker for several hours and kept at around 90°C for 6 hrs. The resultant precipitate was then repeatedly washed with distilled water to remove NaOH filtered & dried. Mullite & α -quartz present in fly ash sources of aluminium & silicon respectively, for zeolite formation. Steps in zeolite synthesis shown below:

Result and Discussion:

1. FTIR: Fourier Transform Infra-Red spectra:

FTIR spectra of various synthesized zeolite-X types shown in Fig. 1a, 1b and 1c. FTIR spectra of zeolite composed of the peaks of vibration of framework and Si-O or Al-O e.g., two strong peaks occurring at wavelength number of 1007(ZX-a), 1004(ZX-b), 1001(ZX-c) and 460 cm⁻¹ (ZX-a), 448 cm⁻¹ (ZX-b), 461 cm⁻¹ (ZX-c) resulted from the asymmetric stretching and bending vibrations of Si-O or Al-O bonds. The small peak at 629 cm⁻¹ (ZX-a), 626 cm⁻¹ (ZX-b), 630 cm⁻¹ (ZX-c) is due to the vibration in the external linkage of tetrahedra. No significant broadening/peaks observed beyond 1140 cm⁻¹ (attribute to asymmetric stretching vibration of (Si/Al) O₄)





2. X-ray diffraction (XRD): The determination of structure of three synthesized zeolite-X was done by XRD (Bruker AXS, Diffractometer D8, Germany) using Cu-K_α as a source and Ni as a filter as shown in Figure 2 a

The X-ray (powder) diffraction (XRD) pattern of any crystalline material is the fingerprint of its structures. XRD patterns of different synthesized zeolitic were scanned from 10.330 (2θ, where θ is angle of diffraction). Various crystalline phase present in samples were identified by six prominent peaks namely at theta (θ) value 14.85, 17.60, 24.15, 31.19, 36.62, 54.32 and 70.73. The major peaks were selected specifically because they are least affected by the degree of hydration of samples & also by others.

Table 1: Normal range of chemical composition for fly ash produced from different coal types (expressed as percent by weight).

Sr. No.	Component	Bituminous (%)	Sub-bituminous (%)	Lignite (%)
1	SiO ₂	20-60	40-60	15-45
2	Al ₂ O ₃	5-35	20-30	10-25
3	Fe ₂ O ₃	10-40	4-10	4-15
4	CaO	1-12	5-30	15-40
5	MgO	0-5	1-6	3-10
6	SO ₃	0-4	0-2	0-10
7	Na ₂ O	0-4	0-2	0-6
8	K ₂ O	0-3	0-4	0-4
9	LOI	0-15	0-3	0-5

Table.2: Synthesis of zeolite at different temperatures.

Zeolite designation	Source of fly ash	NaOH/fly ash	Fusion temperature (K)			Curing time(hrs)
			600	800	1000	
Z-X	CSTPS	1:1.5	600	800	1000	6

Table.3: Peak least (Bookmark 1)

Pos. [°2 Thetaθ]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]
14.8572	20.40	0.4896	5.95789	31.64
17.6083	54.36	0.4080	5.03274	84.29
24.1523	51.76	0.4080	3.68192	80.25
31.1974	64.50	0.4896	2.86465	100.00
35.6278	28.72	0.4896	2.51793	44.54
54.3214	3.74	1.3056	1.68745	5.80
70.7301	5.43	0.4080	1.33089	8.41



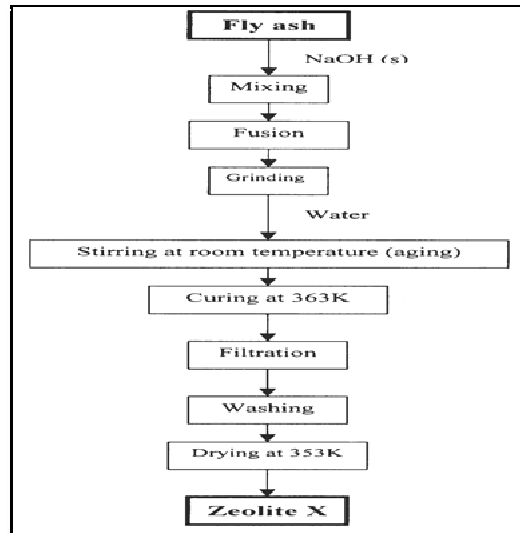


Figure.1: Process flow diagram for synthesis of zeolite X-type from fly ash

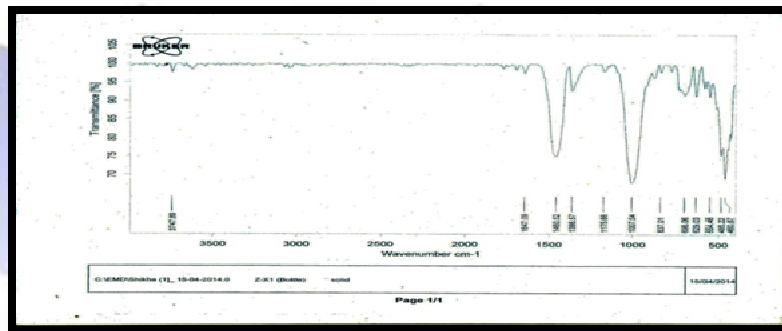


Figure. 1a: FTIR of Zeolite X-1

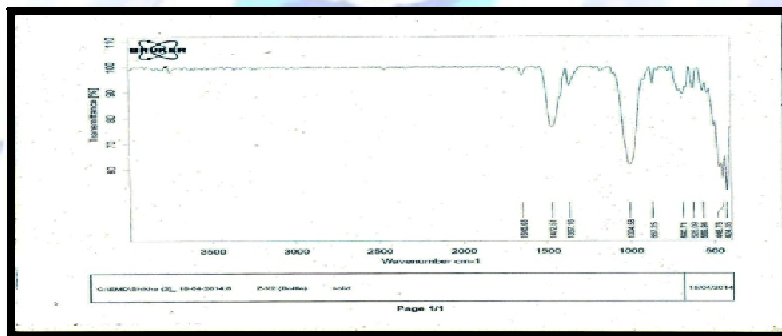


Figure. 1b: FTIR of Zeolite X 2

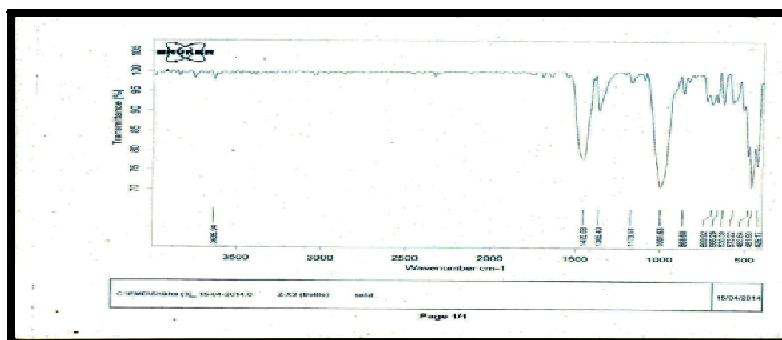


Figure 1c: FTIR of Zeolite X-3

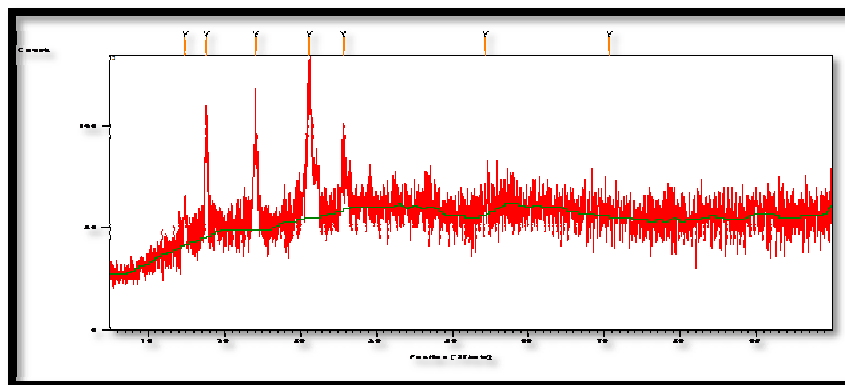


Figure. 2a: X-ray diffraction of zeolite

Conclusion:

Zeolite of X-type was synthesized from coal fly ash (Chandrapur Super Thermal Power Station, Chandrapur, (CSTPS) Maharashtra, India) by alkali fusion followed by hydrothermal treatment. The quartz main crystalline phase of fly ash can be converted to pure Zeolite X-type at employed treatment conditions. The zeolite-X was also successfully synthesized from coal fly ash under certain conditions. The properties of zeolitic material formed strongly depended upon the treatment conditions and composition of the raw materials. The crystallinity of synthesized zeolite changes with fusion temperature and a maximum value was obtained at 1000K. The best conversion of coal fly ash to Na-X zeolite was obtained at the following conditions: NaOH/Fly ash ratio, 1.1.5; fusion temperature, 1000K; aging at 90° C for 6 hrs.

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