Highly crystalline zeolite — a from flyash of bituminous and lignite coal combustion

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Abstract

Flyash is being generated in voluminous amounts by large scale coal combustion process. It poses a serious threat to thermal power industries specifically, in India, wherein the percent of utilisation of flyash is very poor (3–5%). In view of this problem, newer methods of its disposal and utilisation are being explored. The synthesis of zeolite from flyash appears to be one of the most promising alternatives as it has emphasis on value addition to waste material.

Flyashes originating from different sources of coal differ in their characteristics and have implications in this work on Zeolite-A production. These factors have been thoroughly investigated and the conditions favourable for formation of Zeolite-A have been delineated. The reactivity of flyash towards zeolite formation is directly dependent on the SiO_2/Al_2O_3 ratio, Fe_2O_3 and CaO content. Amongst the flyashes investigated, so far the sub-bituminous coal based flyash with SiO_2/Al_2O_3 ratio of 3.47 appears to be a suitable substrate for Zeolite-A synthesis. These zeolites have been characterised with respect to XRD crystallinity, calcium binding capacity (CBC) and sorption capacity, wherein the crystallinity ranges from 50 to 100%, the CBC ranges from 290 to 560 meq/100 g and sorption capacity ranges from 16.6 to 23.8%. © 2001 Elsevier Science B.V. All rights reserved.

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1. Introduction

Increased thermal power generation has resulted in discharge of an uncontrolled amount of flyash into the environment. Due to its adverse effects, attributed mostly to its fine structure and toxic elements, regulations for its land disposal are stringent. Development of alternative methods of flyash disposal or proper utilisation with emphasis on resource recovery has thus become imperative [1].

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Flyash utilisation methods include extraction of alumina/magnetite, production of light aggregates, flyash bricks, portland pozzolan cement, and concrete which are all low value utilisation. Currently, synthesis of zeolites from flyash, a high value added product, is receiving increasing attention. Zeolites are crystalline, hydrated aluminosilicates with over 280 species of naturally occurring and synthetic compositions and are prepared from sodium silicate and aluminate solutions [2]. In this context, Indian flyashes with high SiO_2 and Al_2O_3 [2] content appear to be suitable substrates for synthesis of zeolites.

Amongst the possibility of synthesis of various types of zeolite from flyash, the synthesis of Zeolite-A is the most promising technology. This is attributed to the fact that Zeolite-A can be used as a substitute for conventional phosphatic builder, viz. sodium tripolyphosphate (STP). The STPs are known to cause eutrophiction (decaying of lakes) and this problem can be resolved by substituting with Zeolite-A. The market demand for STP is 800,000 tonnes per year and anticipating a 25% shift to zeolite based detergents, the demand of Zeolite-A is expected to be about 200,000 tonnes per year [3]. Based on the mass balance studies it is envisaged that production of 100,000 tonnes per year of Zeolite-A would require approximately 1,000,000 tonnes per year of flyash. Thus, the synthesis of Zeolite-A from flyash would account for flyash utilisation of 1–2% of the total flyash production, which is quite substantial taking into account the current low amount of flyash utilisation.

At present, much attention is being paid to these waste materials or natural silicate raw material as a potential source of inexpensive Zeolite-A [4]. Amongst the above materials, kaolin clay is the most promising one [5]. The method of synthesis, starting from kaolin involves its thermal treatment at 700°C for its transformation into metakaolin and then its recrystallisation into Zeolite-A [6].

Zeolite-A has been synthesised from allophane in Japan. The treatment of diatomite with 10–15% solution of sodium hydroxide has been used in Russia for Zeolite-A synthesis [7]. On similar lines, flyash has also been used as a raw material for zeolite synthesis as it contains ≈60% SiO_2 and ≈30% Al_2O_3 [8–13]. However, the work reported so far results in formation of mixture phases [14,15]. The process developed by Rayalu et al. details the synthesis of Zeolite-A from flyash. The process developed is environmentally benign as no solid waste residue is generated and the emissions are negligible. It is proposed to used the mother liquor in the process; however, there may be build-up of toxic elements which can be avoided by removal of a bleed stream and addition of fresh water. Alternately, the mother liquor may be contacted with an adsorbent (viz. zeolites) to remove trace elements (unpublished data).

This paper addresses the selective synthesis of Zeolite-A from flyashes originating from different coal sources in India, including optimisation of reaction conditions and detailed characterisation of zeolites synthesised.

2. Materials and methods

2.1. Materials

Flyash samples originating from two different sources, namely lignite based flyash (FAL) and sub-bituminous coal (FASBC), were collected from Neyveli Lignite Corp. (NLC) and

Koradi Thermal Power Station (KTPS) All flyash samples were obtained from the hopper of an electrostatic precipitator.

All the chemicals used were procured from Merck. The chemicals used were analytical grade sodium hydroxide, aluminium hydroxide, sodium silicate, sodium aluminate and hydrochloric acid. The indigenous commercial Zeolite-A was procured from Zinco Lab Pvt. Ltd., Mumbai, and the international grade Zeolite-A were procured from Wako, Japan and Degussa, Germany.

3. Experimental

3.1. Characterisation of flyash

The flyash samples were characterised with respect to SiO_2, Al_2O_3, Fe_2O_3, TiO_2, MnO, CaO, MgO, Na_2O and K_2O using the LIMBO method [16].

3.2. Synthesis

The flyash based Zeolite-A (FAZ-A) samples were synthesised by fusing flyash and sodium hydroxide. A homogeneous fusion mixture was obtained by grinding flyash and sodium hydroxide together. The mixture was heated at a temperature ranging between 550 and 650°C for a period of time. The resultant fused mass was cooled, milled and mixed thoroughly in distilled water with simultaneous addition of sodium aluminate. Commercial Zeolite-A was added as seeding to above mixture. The slurry so obtained was subjected to ageing for a period of time. The amorphous aluminosilicate gel was then subjected to crystallisation at a temperature ranging between 90 and 105°C. Solid crystalline product was separated by filtration and washed thoroughly until the filtrate pH was 10–11 and dried at a temperature of 50–60°C. The details of this process are provided elsewhere [17].

Four variables were studied in synthesising Zeolite-A of high crystallinity, CBC, and optical brightness (OB). The possibility of using sodium aluminate as a source alumina enrichment to maintain SiO_2/Al_2O_3 ratio favourable for synthesis of Zeolite-A from flyash was studied in detail. To start with the composition (SAI) and volume (20 ml) was arrived at to maintain the SiO_2/Al_2O_3 ratio of (1.0–1.1) in FAZ-A reaction systems using crystallinity, CBC and OB as diagonistic parameters. However, as the sample resulting from addition of sodium aluminate of SAI, composition (20 ml vol.) has lower OB various other compositions and volumes of sodium aluminate addition were studied in detail. The sodium aluminate of various compositions employed are designated as SA-I (Na_2O: 50.3%, Al_2O_3: 29.4%), SA-II (Na_2O: 50.3%, Al_2O_3: 39.0%), SA-III (Na_2O: 7.3%, Al_2O_3: 14%), SA-IV (Na_2O: 15%, Al_2O_3: 28%), SA-V (Na_2O: 20.2%, Al_2O_3: 14%), and SA-VI (Na_2O: 17%, Al_2O_3: 16%).

Depending upon the chemical composition of flyash samples and to improve the OB, the flyash samples were subjected to certain pre-treatment processes such as sieving, magnetic separation and acid treatment for removal of iron. Impurities like Fe_2O_3, which decreases calcium binding capacity (CBC) of FAZ-A and imparts colour must be removed. The flyash was acid treated by refluxing it with 8 N hydrochloric acid for 2 h. The acid treated flyash

was then washed thoroughly prior to subjecting it to a fusion step. The removal of magnetic iron was effected by subjecting the flyash slurry to a magnetic field. The amount of iron removed in five to six cycles was 70–75%.

3.3. Characterisation of FAZ-A

The CBC of FAZ-A samples was determined by following standard method. For determining CBC 0.05% $CaCl_2$ stock solution was prepared with 1 N NaOH and its pH was adjusted to 10–11. A pre-weighed (0.5 g) FAZ-A sample and Zeolite-A standard were added to 500 ml of stock solution and stirred for 15 min. The solutions were then filtered. These Ca^{2+} solutions along with the stock solution were titrated complexometrically using standardised 0.01 M EDTA solution. The amount of Ca^{2+} ions exchanged by zeolite was expressed in terms of meq of Ca^{2+}/100 g of zeolite on anhydrous basis. The sorption capacity of FAZ-A was determined by heating it at 800°C for 1 h. Powder X-ray diffraction (XRD) analysis was performed using Cu Kα as a source of X-rays in the range of 5–60° using an X-ray diffractometer, Model Philips PN-1830. The *d*-spacing values reported in the literature for Zeolite-A (12.2 ± 0.2, 8.60 ± 0.20, 7.05 ± 0.15, 4.07 ± 0.08, 3.68 ± 0.07, 3.26 ± 0.05, 2.96 ± 0.05, 2.73 ± 0.05, 2.60 ± 0.05) were used as a basis for identification and quantification of crystalline phase [18]. For the elemental analysis, FAZ-A samples were dissolved in a mixture of acetic and nitric acid after subjecting the sample to the LIMBO method of elemental digestion and analysed by ICP–AES (Model: YJ24) for Al_2O_3, SiO_2 was determined using instrumental/conventional methods. Na_2O was estimated by using flame photometer (Modiflame 127 FPM compressor unit 122). The particle size analyses were measured using Fritsch particle size analysette 22. The colour of the samples was examined by measuring brightness with a Carl Zeiss photoelectric reflection photometer (ELREPHO) with $BaSO_4$ as standard. The surface morphology of the zeolite was examined by Jeol-840 A scanning electron microscope (SEM). The sorption capacity was determined as loss on ignition (LOI) at 800°C for 2 h. The TCLP test was conducted as per the standard method of USEPA [19].

4. Results and discussion

4.1. Flyash characterisation

The chemical analysis of flyash from different sources is given in Table 1. Of the two flyash samples the FASBC has a higher alumina content than FAL, whereas these flyash samples have comparable silica contents. The FASBC with higher Al_2O_3/SiO_2 ratio is more favourable for use as a raw material for Zeolite-A synthesis it requires less sodium aluminate addition thereby reducing the cost of production of Zeolite-A.

The FAL has a higher CaO content compared to FASBC. The higher content of CaO in FAL results in lesser formation of mullite phases. The low level of mullite phases encourages zeolite synthesis, this result is attributed to the fact that mullite is resistant to direct caustic treatment. The FAL flyash also contains a large amount of unburnt carbon which interferes

Table 1
Comparative chemical composition of different flyash samples

Type of flyash	Sub-bituminous coal based flyash (KTPS) (%)	Lignite based flyash (NLC) (%)
SiO_2	62.27	61.83
Al_2O_3	30.96	19.04
Fe_2O_3	1.25	6.05
TiO_2	1.67	1.29
MnO	Trace	Trace
CaO	3.02	7.22
MgO	Nil	1.75
Na_2O	0.12	1.26
K_2O	0.41	0.36
LOI	0.29	1.19

in the fusion step thereby affecting the quality of fused product. This unburnt carbon is estimated on the basis of LOI content. It is apparent from Table 1 that LOI level in FASBC is less than FAL.

The iron oxide in flyash is decomposed during the fusion process and has high probability of being incorporated into the zeolite matrix. This process yield a brownish tinge for the Zeolite-A product. Thus, lower percentage of iron oxide content in flyash is favourable for synthesis of zeolite with better OB. FAL has highest percentage of iron oxide content; FASBC has a lower content. The iron oxide is present mostly as magnetite; and hence the problem of colour can be resolved by magnetic separation.

The other elements present in trace amount include TiO_2, MnO, MgO, Na_2O and K_2O. These elements along with other anions like carbonate, sulphate, etc. appear to promote nucleation and crystallisation of FAZ-A.

The reaction conditions including fusion temperature, fusion time and ageing time were studied thoroughly in exploring the possibility of synthesising A type zeolite [17]. The results clearly indicate that flyash yields x or y type of zeolite [18] and therefore it can be concluded that variation in those parameters does not influence Zeolite-A synthesis significantly in comparison to particle size of flyash, crystallisation time and sodium aluminate composition [17,18]. Hence amongst the variables explored for synthesis of Zeolite-A, studies were restricted to wide ranging variations in particle size of flyash, crystallisation time, sodium aluminate compositions and seeding effect to illustrate their effect on Zeolite-A synthesis. The formation of zeolite and its standardisation was monitored with respect to crystallinity, colour and CBC.

4.1.1. Zeolite-A from FASBC

FAZ-A was synthesised from FASBC (ZA-I–ZA-13) under conditions delineated in Table 2. The quantity of flyash and NaOH taken for each experimental run was 20 and 24 g, respectively. For experimental runs ZA-1–ZA-4 unsieved flyash with approximate particle sizes ranging between 44 and 250 μ was used. FAZ-A synthesised under conditions delineated for ZA-I using 20 ml of sodium aluminate of composition SA-I (Na_2O: 50.3%, Al_2O_3: 39%) shows low crystallinity of 50% and CBC of 300 meq/100 g. This result may

Table 2
Variation of crystallinity sorption capacity and CBC with respect to experimental conditions for FASBC

Sample	Fusion		Mixing ageing time (h)	Sodium aluminate (NaAlO$_2$) (ml)	Seeding (g)	Crystallisation time (h)	Pre-treatment	Sorption capacity (%)	Calcium binding capacity (meq/100 g)	Crystallinity (%)	Sodium aluminate composition
	Temperature	Time									
ZA-1	600	1.5	8	20	No	3		18.7	300	50	SA-I (Na$_2$O: 50.3%, Al$_2$O$_3$: 29.4%)
ZA-2	600	1.5	8	20	Yes	3		22.7	470	85	SA-II (Na$_2$O: 50.3%, Al$_2$O$_3$: 39%)
ZA-3	600	1.5	8	30	Yes	3		18.6	360	70	SA-II (Na$_2$O: 50.3%, Al$_2$O$_3$: 39%)
ZA-4	600	1.5	8	20	No	3.5		19.1	430	100	SA-II (Na$_2$O: 50.3%, Al$_2$O$_3$: 39%)
ZA-5	600	1.5	8	20	Yes	3	Sieving by 170 μ	21.5	500	100	SA-II (Na$_2$O: 50.3%, Al$_2$O$_3$: 39%)
ZA-6	600	1.5	8	20	Yes	3	Sieving by 53 μ	21.0	470	100	SA-II (Na$_2$O: 50.3%, Al$_2$O$_3$: 39%)
ZA-7	600	1.5	8	20	Yes	3.5	Sieving by 53 μ	21.0	540	100	SA-II (Na$_2$O: 50.3%, Al$_2$O$_3$: 39%)
ZA-8	600	1.5	8	100	No	3		21.0	530	97	SA-III (Na$_2$O: 7.3%, Al$_2$O$_3$: 14%)
ZA-9	600	1.5	8	100	No	3		20.8	460	88	SA-IV (Na$_2$O: 15%, Al$_2$O$_3$: 28%)
ZA-10	600	1.5	8	112	No	3	Sieving by 53 μ	21.2	527	100	SA-V (Na$_2$O: 20.2%, Al$_2$O$_3$: 14%)
ZA-11	600	1.5	8	100	Yes	2	Sieved by 53 μ magnetic separation ferric hydroxide removed from SA-VI		505	100	SA-VI (Na$_2$O: 20.2%, Al$_2$O$_3$: 18.4%)
ZA-12	600	1.5	8	42	Yes	3	Sieved by 53 μ magnetic separation ferric hydroxide removed from SA-VI	21.0	540	100	SA-VI (Na$_2$O: 20.2%, Al$_2$O$_3$: 18.4%)
ZA-13	600	1.5	8	20	No	3	8 N HCl for 2 h refluxing	22.4	520	85	SA-VII, 50 ml of neutralised acid extract + 12 g Al(OH)$_3$ + 13 g NaOH

be due to insufficient addition of alumina; this problem was overcome by modifying the composition of sodium aluminate in specific by increasing the alumina content.

The usage of 20 ml of sodium aluminate of composition SA-II (Na_2O: 50.3%, Al_2O_3: 39%), i.e. ZA-2 shows a definite increase in crystallinity to approximately 85% with CBC of 470 meq/100 g. To further improve the crystallinity of FAZ-A, 30 ml of SA-II were added in the ageing step, but this step resulted in a decrease of CBC as well as crystallinity (ZA-3). This result may be due to excess addition of alumina and soda which results in the formation of other phases like sodalite. An increase in the crystallisation time from 3 to 3.5 h with same sodium aluminate; composition (SA-II) resulted in the formation of highly crystalline sample (ZA-4) with no significant increase in its CBC.

The larger particle size fraction of flyash having iron oxide impurities was removed mechanically by sieving. The flyash with particle size of 170 μ was used for the synthesis of FAZ-A using 20 ml sodium aluminate of SA-II composition. The FAZ-A so formed (ZA-5) shows significant improvement in crystallinity (100%) and CBC (500 meq/100 g). The usage of flyash with particle size of 53 μ using 20 ml of SA-II and crystallisation time of 3.5 h resulted in (ZA-7) significant improvement in CBC (540 meq/100 g) with 100% crystallinity. This result illustrates marked effect of particle size of flyash on Zeolite-A synthesis.

Several modifications in aluminate compositions were made to improve crystallinity, CBC and OB of FAZ-A. FAZ-A (ZA-8) was synthesised using sodium aluminate of composition SA-III (Na_2O: 7.3%, Al_2O_3: 14%) with 97% crystallinity and CBC of 530 meq/100 g. However, the use of sodium aluminate of composition SA-IV (Na_2O: 15%, Al_2O_3: 28%) resulted in decrease in crystallinity and CBC of FAZ-A (ZA-9). Also no improvement in colour was observed. This shows that the sodium aluminate does not affect the OB of the FAZ-A.

The FAZ-A samples synthesised so far had an OB of approximately 60%. To improve OB, certain syntheses were performed using sodium aluminates of different compositions. FAZ-A (ZA-1) synthesised using SA-I is brighter with comparatively lower CBC and crystallinity than those synthesised using SA-II. However, by using sodium aluminate of composition SA-V (Na_2O: 14%, Al_2O_3: 20.2%), CBC and crystallinity of FAZ-A synthesised are not altered but there is slight improvement in colour.

To improve the colour of FAZ-A, iron oxide impurities were removed by sieving and by selective magnetic separation of flyash. The FAZ-A synthesised (ZA-11) from above flyash using 100 ml SA-VI (Na_2O: 20.2%, Al_2O_3: 18.4%) is 100% crystalline with CBC 505 meq/100 g and FAZ-A synthesised (ZA-12) using 42 ml SA-VI (Na_2O: 20.2%, Al_2O_3: 18.4%) shows marked increase in CBC (540 meq/100 g) with 100% crystallinity and significant improvement in colour (80%).

To further improve OB, the flyash was acid treated by refluxing with 8 N HCl for 2 h. The acid-treated flyash was filtered and washed thoroughly with double distilled water until the pH of the filtrate decreased to approximately 7. The acid filtrate was reused for synthesis of sodium aluminate SA-VII after precipitating ferric hydroxide by adding excess of sodium hydroxide. This step was carried out to avoid any alumina loss occurring during acid treatment. FAZ-A synthesised (ZA-13) from acid-treated flyash shows significant improvement in colour. However, a slight decrease in crystallinity and CBC value was observed.

Table 3
Variation of crystallinity sorption capacity and CBC with respect to experimental conditions for FAL

Sample	Fusion		Mixing ageing time (h)	Sodium aluminate (NaAlO$_2$) (ml)	Seeding (g)	Crystallisation time (h)	Pre-treatment	Sorption capacity (%)	Calcium binding capacity (meq/100 g)	Crystallinity (%)	Sodium aluminate composition
	Temperature	Time									
ZA-14	600	1.5	8	20	No	2.5		16.62	290	91.7	SA-I (Na$_2$O: 50.3%, Al$_2$O$_3$: 29.4%)
ZA-15	600	1.5	8	20	No	2.5		16.6	410	93	SA-II (Na$_2$O: 50.3%, Al$_2$O$_3$: 29.4%)
ZA-16	600	1.5	8	22	No	2.5		15.7	353	66.5	SA-II (Na$_2$O: 50.3%, Al$_2$O$_3$: 29.4%)
ZA-17	600	1.5	8	30	Yes	2.5		22.31	415	100	SA-II (Na$_2$O: 50.3%, Al$_2$O$_3$: 29.4%)
ZA-18	600	1.5	8	20	Yes	3		18.19	330	82	SA-II (Na$_2$O: 50.3%, Al$_2$O$_3$: 39%)
ZA-19	600	1.5	8	SA-VI	Yes	2.5	Acid treatment was done	17	390	60	SA-VI 50 neutralised and filtered acid extract + 12 g Al(OH) + 13 g NaOH

Seeding is an important step in zeolite synthesis and it is known to promote nucleation and crystallisation process. The effect of seeding is apparent from samples ZA-2, 3, 5–7, 11, 12. It does not show marked improvement in CBC and crystallinity in comparison with the other modifications (sodium aluminate concentration, crystallisation time, etc.) introduced in the process. The effect of different seeding methods was studied with respect to degree of crystallinity and CBC. It is clear from the synthesis data that synthetic Zeolite-A having a particle size of 1 μm is better for seeding than commercial Zeolite-A of particle size 4–5 μm (unpublished data). Thus, it is clear that the seeding effect is more pronounced for decreasing particle seed size. This result is consistent with data reported elsewhere. The increase in the rate of nucleation and crystallisation with decrease in particle size of seeding may be due to increase in cumulative surface area.

4.1.2. Zeolite-A from FAL

FAZ-A was synthesised from FAL (ZA-14–ZA-19) under conditions delineated in Table 3. ZA-14 was synthesised using sodium aluminate (20 ml) of composition SA-I (Na$_2$O: 50.3%, Al$_2$O$_3$: 29.4%). The product has 91.7% crystallinity and a lower value of CBC (290 meq/100 g). This result is attributed to lower alumina content in the lignite flyash. FAZ-A (ZA-15) synthesised using 20 ml of sodium aluminate SA-II composition (Na$_2$O: 50.3%, Al$_2$O$_3$: 39.0%) has a higher crystallinity (93%) and CBC of 410 meq/100 g. An increase in sodium aluminate addition from 20 to 22 ml resulted in ZA-16 with a significant increase in crystallinity (100%) as well as CBC (415 meq/100 g). Further increase in sodium aluminate addition resulted in (ZA-17) decrease in crystallinity to about 66.5% with a consequent decrease in CBC (353 meq/100 g). In another experiment the crystallisation time was increased from 2.5 to 3 h to observe effect of time on crystallisation reaction. The increase in crystallisation time resulted in FAZ-A with lower crystallinity and CBC.

Thus, it is apparent that increasing Al$_2$O$_3$ content of reaction system through sodium aluminate addition resulted in a definite increase in crystallinity and CBC value up to ZA-17 synthesis. With a further increase in sodium aluminate addition, there is a decrease
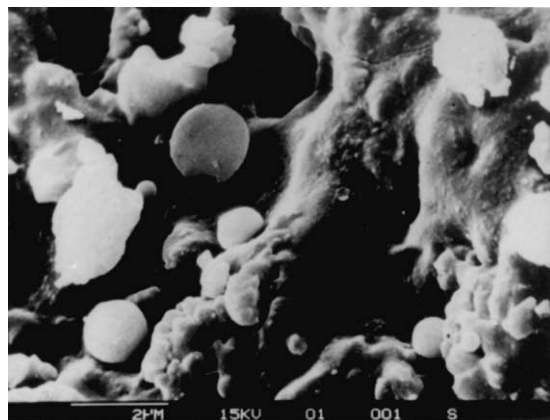

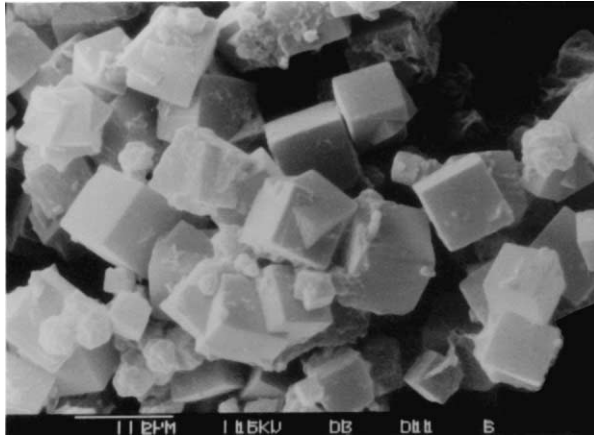
Fig. 1. XRD pattern of FAZ-A (FASBC).

Fig. 2. XRD pattern of FAZ-A (FAL).

in CBC and crystallinity as observed for ZA-18. Thus, it may be concluded that excess addition of sodium aluminate results in formation of amorphous phases and/or sodalite formation, which reduces the crystallinity and CBC. A similar trend has been observed for increase in crystallisation time beyond 2.5 h.

Attempts were made to remove CaO and Fe_2O_3 by acid treatment of flyash. The acid-treated flyash was washed and used for FAZ-A synthesis. The acid filtrate was neutralised
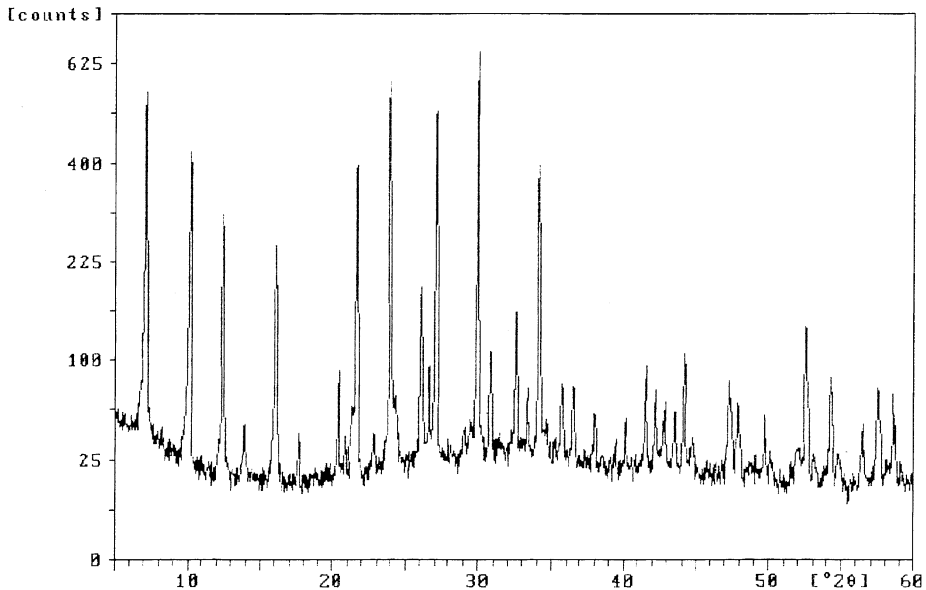
Fig. 3. XRD pattern of commercial Zeolite-A.
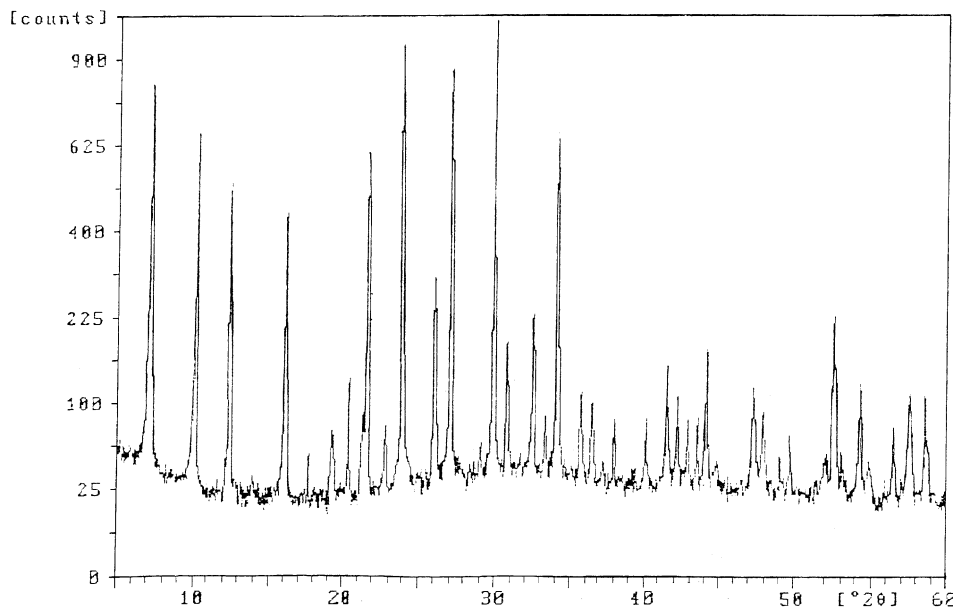
Fig. 4. Variation in crystallinity and CBC with chemical composition of flyash.
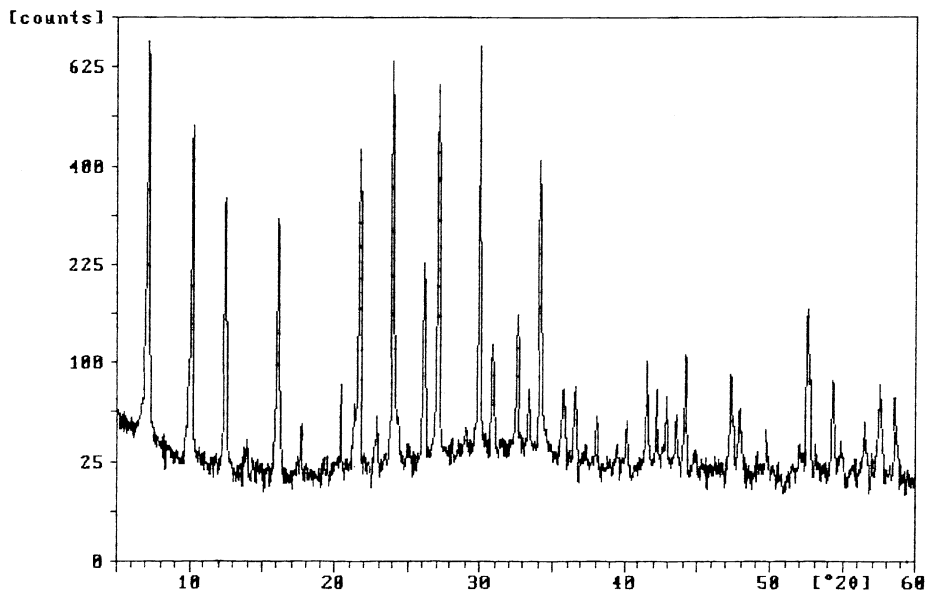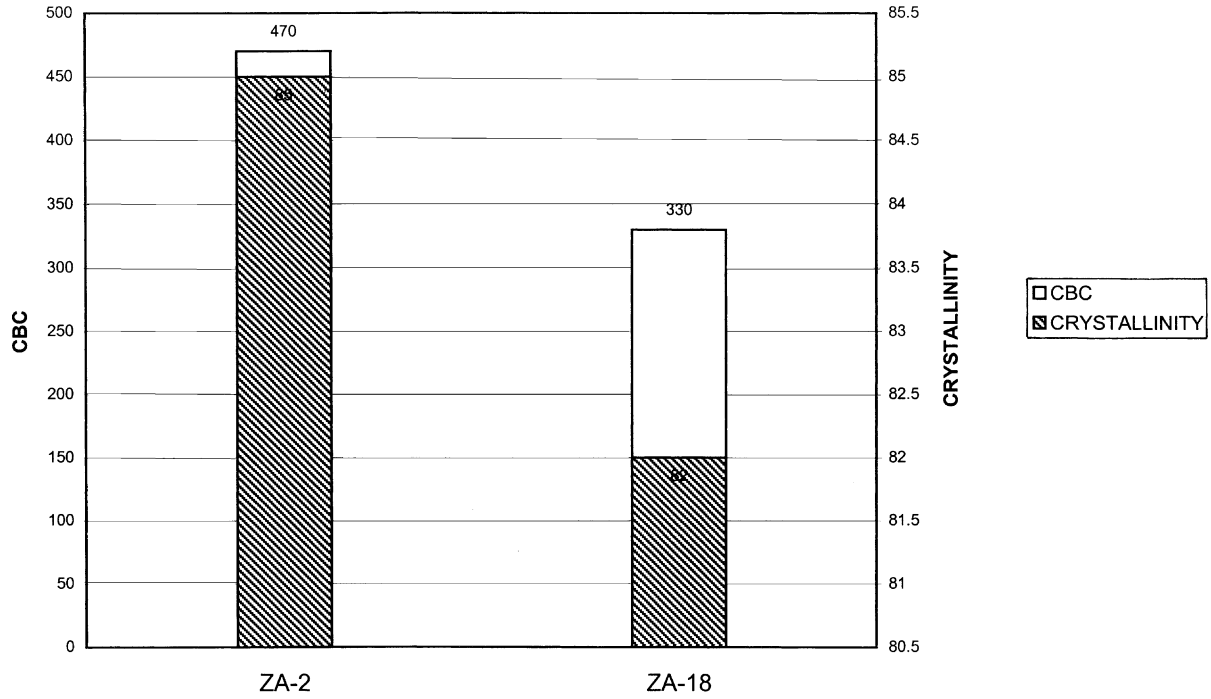

Fig. 5. SEM photograph of flyash.

Fig. 6. SEM photograph of FAZ-A (FASBC).

by NaOH to precipitate ferric hydroxide. The filtrate so obtained was reused for synthesis of sodium aluminate (SA-VI). This procedure was used to avoid any alumina loss occurring during acid treatment of flyash. There was no significant improvement in CBC and crystallinity (ZA-19) of the zeolite so formed.

Fig. 1 shows that morphologically flyash is amorphous with some cenospheres and pleurosphere. The morphological characteristics of FAZ-A (ZA-14) indicates its highly crystalline nature. It was observed that amorphous microparticles of aluminosilicate transforms into crystals of FAZ-A (Fig. 2) without appreciable change in size. The SEM photograph of FASBC is presented in the Fig. 2 and similar surface morphology has been observed for FAL.

The XRD pattern of Zeolite-A synthesised from FASBC, FAL and commercial Zeolite-A are presented in Figs. 3–5. The similarity in the XRD reflections of FASBC (Fig. 3) and FAL (Fig. 4) and the international zeolite standard (Fig. 5) shows the similarity in the product and formation of highly crystalline structure.

TCLP tests conducted for FAZ-A samples reveals that the leachate do not contain any of the toxic elements, viz. As, Ag, Ba, Cd, Cr, Pb, Se, Hg. Thus classifying FAZ-A as a non-toxic material.

5. Conclusion

In conclusion, it may be said that FAZ-A production from FASBC, and FAL is dependent on the chemical composition of the flyash raw material and the reaction conditions. Hence, by comparing FAZ-A synthesised from different flyashes under exactly identical conditions (without any pre-treatment) gives an insight into the most flyash-suitable substrate

Table 4
Comparative characteristics of flyash based and commercial zeolites

Sl. No.	Typical specifications	FAZ-A from FASBC	FAZ-A from FAL	Commercial Zeolite-A
1	Appearance	Creamish	White	White
2	Average particle size (μ)	2–3		2–3
3	Calcium binding capacity	150–160	120–130	160–180
4	Density (g/cm^3)	2.07	–	2
5	Tapped density	0.49	–	0.5
6	Moisture content (5) (ignition loss at 800°C)	20–21	19–21	19–20
7	pH of 1% slurry	9.5–10.5	8.0–8.5	10–11
8	Crystalline form	A type zeolite	A type zeolite	A type zeolite
9	Crystallinity (%)	96–100	93–100	80–85
10	Silica content (%)	32 ± 0.5	32 ± 0.5	33 ± 0.5
11	Alumina content %	27 ± 0.5	25.5 ± 0.5	27.5 ± 0.5
12	Na$_2$O content (%)	16 ± 0.5	9.5 ± 0.5	16.5 ± 0.5
13	Si/Al molar ratio	1.1–1.2	1.1–1.2	0.9–1
14	TCLP test	No toxic elements leached	No toxic elements leached	No toxic elements leached

for FAZ-A synthesis. Fig. 6 illustrates the influence of chemical composition of flyash on crystallinity and CBC of FAZ-A. It is evident from Fig. 6 that, under exactly similar conditions, FASBC is the most suitable substrate for FAZ-A synthesis. This type of database will prove to be useful in assessing the possibility of FAZ-A synthesis and the modification required for wide ranged flyash composition. Also the comparative characteristics of Zeolite-A synthesised from FASBC and FAL is presented in Table 4.

The process formulated for synthesis of FAZ-A from different flyash provide a good database for the synthesis of highly pure, crystalline, small and uniform cubic crystals. Synthesis of zeolitic from flyash a toxic waste material is high value added product. The characteristics like CBC, crystallinity, elemental analyses, particle size, and sorption capacity of FAZ-A are comparable to that of commercial Zeolite-A. These characteristics suggest the possibility of its usage as a substitute for detergent builder.

The process for synthesis of FAZ-A offers distinct advantages, viz. cost effective alternative to commercially available Zeolite-A, resolution of flyash disposal problem at least partially, commercially feasible substitute for STPs, conservation of conventional raw materials, viz. sodium silicate and aluminate and is environmentally benign.

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