

Thermal studies of low silica zeolites and their magnesium exchanged forms

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Abstract

Zeolites are crystalline aluminosilicate polymers with uniform pores, channels and cavities. They can be synthesised using a variety of raw materials and the history of the zeolite influences some of its properties. Thermal treatment dehydroxylates the zeolites, breaks down the structure and converts them to X-ray amorphous material. Further heating results in the crystallisation of ceramic phases depending upon the chemical composition of the system. The ion exchange capacity of the zeolite helps to introduce various cations into the structure and thereby extending the possibility of the formation of different types of ceramics. A systematic study has been carried out on the sintering behaviour of low silica zeolites i.e., NaA and NaX of different origin (synthesised from kaolin/sodium aluminosilicate gel) and their magnesium exchanged forms. The final products were albite, nepheline and jadeite for clay based zeolites. Cristobalite also is likely to form from the kaolin-based zeolite NaA and carnegite from the corresponding NaX. Sodalite was the main product from the gel route zeolites. The magnesium exchange was partial and the remaining sodium present in the system was found to influence the sintering properties. The magnesium zeolites were found to be more heat resistant compared to their sodium forms. Mullite and magnesium spinel were the major phases formed along with small amount of albite. An important observation is that cordierite is formed at 1150 °C only from the clay based magnesium zeolites. The fired samples contain different crystalline and glassy phases and the physical properties represent an overall behaviour of this heterogeneous system. © 2002 Elsevier Science Ltd and Techna S.r.l. All rights reserved.

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1. Introduction

Zeolites are crystalline aluminosilicates with indefinitely extending three dimensional network of aluminium and silicon tetrahedra linked by sharing of oxygen atoms. They have channels and interconnected voids which are occupied by alkali and alkaline earth metal cations (usually Na^+ ions) and water molecules. These cations are mobile and can be easily exchanged with other metal ions to the extent that the charge neutrality is maintained. In studies related to the thermal stability of natural and synthetic zeolites and their ion exchanged forms, the formation of various mineral powders has been reported. The hydrothermal transformation of zeolites at different temperatures time and pressure is also studied earlier [1]. Recently zeolites and their ion exchanged forms have been reported as precursors for

various aluminosilicate ceramics [2–17]. There are some advantages for this novel route of ceramic preparation compared to the traditional methods of mixing of oxides and non-conventional use of sol gel precursors followed by sintering.

The unique properties of “low silica zeolites” i.e. NaA and NaX with Si/Al ratios ~ 1 and 1–1.5 respectively, such as ion exchange capacity, sorption and catalytic activity, make them ideal for various industrial applications. Generally, zeolites are synthesised from freshly prepared sodium aluminosilicate gel from various silica and alumina sources. Kaolinite having a Si/Al ratio = 1 is an ideal, combined source for silica and alumina for the synthesis of low silica zeolites which is reported as early as in 1964 [1]. The zeolites from kaolinites are always contaminated with trace amounts of Fe, Ti, Ca, Mg etc. which are originally present as ancillary minerals in the naturally occurring kaolin. Even though processing of the raw clay improves the quality of the clay to some extent, traces of these impurities still remain in the zeolite either in the structure or as associated ions.

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There is a possibility that these impurities may influence the ion exchange and sintering behaviour of these zeolites and the properties of the sintered products.

The synthesis of ceramics from zeolites was studied for the first time by Choudhary et al in 1989 where monolithic anorthite, anorthite-cordierite or cordierite based ceramics were prepared from Ca, Ca-Mg and Mg forms of zeolites A, X and Y [2]. Later, a series of studies on the synthesis of leucite pollucite and spodumene based ceramics from zeolites B, W and ZK 19 was reported by Bedard et al. [3–10]. Recently, syntheses of pollucite and celsian ceramics have been reported by Pareira and Hoghoogi et al. respectively [16–17]. All these work has been carried out using zeolites synthesised by the Gel Route i.e. separate sources of silica and alumina.

The present paper deals with a systematic study on the sintering of sodium zeolites A and X synthesised from two different raw materials, i.e. clay route (CR) and gel route (GR) and their magnesium exchanged forms. The zeolites were thermally treated at different temperatures from 750 to 1150 °C at an interval of 100 °C and the sintered products were analysed by X-ray diffraction. The physical properties such as water absorption, shrinkage and apparent density of the fired products were measured. The influence of synthesis route and the cation in the ion-exchanged zeolites on the sintering behaviour was investigated.

2. Experimental

2.1. Synthesis of zeolites

2.1.1. Clay route

A coating grade kaolin (English Indian Clays Ltd., Thiruvananthapuram, India) was used as the raw material for the synthesis of zeolites NaA and NaX. The clay was calcined in a muffle furnace to get metakaolin which was subjected to hydrothermal reaction with aqueous alkali at 90 ± 2 °C for $3\frac{1}{2}$ h under stirred conditions to give zeolite NaA [18]. The molar ratios $\text{SiO}_2/\text{Al}_2\text{O}_3 = 2$; $\text{Na}_2\text{O}/\text{SiO}_2 = 2.5$ and $\text{H}_2\text{O}/\text{Na}_2\text{O} = 40$ were maintained during the reaction. In order to prepare zeolite NaX, calculated amount of sodium silicate was added to the metakaolin-alkali mixture and the molar ratios $\text{SiO}_2/\text{Al}_2\text{O}_3 = 3$; $\text{Na}_2\text{O}/\text{SiO}_2 = 1.1$ and $\text{H}_2\text{O}/\text{Na}_2\text{O} = 40$ were maintained. The reaction mixture was aged for 24 h at ambient temperatures further subjected to hydrothermal reaction in an air oven at 87 ± 2 °C for 15 h under static conditions to get phase pure zeolite NaX [19]. The samples were filtered washed with alkaline water (pH 9–10) and dried. The zeolites were rehydrated to their equilibrium water content by exposing them to water vapour from saturated sodium chloride solution and characterised by various methods. The samples were designated as CR zeolites (clay route).

2.1.2. Gel route

The same molar ratios as in the clay route synthesis were maintained here also, but the zeolitisation was carried out at static conditions. A sodium aluminosilicate gel was prepared by mixing calculated amounts of sodium aluminate, sodium silicate and sodium hydroxide solutions and the reaction mixture was heated in an air oven maintained at 85 ± 2 °C for about $3\frac{1}{2}$ h for preparing zeolite NaA [20]. The molar ratios were changed for zeolite NaX synthesis and the reaction mixture was heated at 90 ± 2 °C for 15 h [21]. After the reaction, the samples were filtered, washed with alkaline water (pH 9–10) and dried. The products were rehydrated, characterised and designated as GRzeolites (gel route).

2.2. Ion exchange of the zeolites

The zeolites NaA and NaX synthesised by two different routes were first exchanged with NH_4^+ ions and then exchanged with magnesium ions by conventional method. Nearly 10 g of the zeolite was added to 100 ml of 10% ammonium nitrate solution and stirred for 8 h. Then the slurry was kept for about 16 h, centrifuged, the supernatant liquid was decanted and the solids were again treated with a fresh solution of corresponding nitrate in a similar manner. This experiment was repeated five times to ensure that maximum ion exchange has taken place. These samples were subjected to ion exchange using magnesium nitrate solution in a similar way. The magnesium-exchanged zeolites thus prepared, were filtered, washed thoroughly and dried.

2.3. Sintering

The Na zeolites and their magnesium exchanged forms were pressed into pellets of 10 mm diameter under a pressure of 125 MPa and heated in a programmable furnace at temperatures 750, 850, 950, 1050 and 1150 °C at a rate of 3 °C/min with a soaking time of 3 h.

2.4. Characterisation

The zeolites NaA and NaX, the magnesium exchanged forms and the sintered products were characterised by various methods including chemical, X-ray Diffraction (XRD) and thermal analysis. Particle size distribution analysis and brightness of the zeolites and kaolin have also been determined. The characterisation of the sintered products included measurement of water absorption, shrinkage and apparent density.

2.4.1. Chemical analysis

Standard wet chemical analysis along with instrumental methods were adopted to estimate SiO_2 gravimetrically, Al_2O_3 , CaO and MgO by complexometry,

Fe₂O₃ and TiO₂ by colorimetry and Na₂O by flame photometry [22].

2.4.2. X-ray diffraction analysis

Powder XRD patterns were recorded on a Philips PW 1710 X-ray Diffractometer with Ni filtered CuK_α radiation.

2.4.3. Thermal analysis

Differential thermal analysis (DTA) and thermogravimetric analysis (TGA) were carried out on a Shimadzu Thermal Analyser at a heating rate of 40 °C/min.

2.4.4. Water absorption

Standard methods were followed for the determination of water absorption [23].

2.4.5. Shrinkage

The dimensional changes during firing were also calculated by measuring the diameter of the pellets before and after firing using a Vernier caliper.

2.4.6. Density

The apparent density was measured using Archimedes' principle.

2.4.7. Particle size distribution

Sedigraph 5100 model particle size analyser (Micromeritics) was used for determining the particle size distribution of the kaolin and the zeolites.

2.4.8. Brightness (%ISO)

The brightness was measured using a Colour Touch™ Model ISO spectrophotometer from Technidyne Corporation, USA.

3. Results and discussion

3.1. Phase analysis

The XRD patterns of the zeolites and their corresponding Mg exchanged products are found identical indicating that the structures of the zeolites remain the same during ion exchange. The dÅ values of the zeolite NaA (CR and GR) and their Mg exchanged products along with the standard values [24] are given in Table 1. Similarly, the values for different forms of NaX are shown in Table 2.

3.2. Chemical analysis

The chemical analysis of the sodium forms of the CR and GR zeolites are given in Table 3 and their particle size distribution in Table 4. The sodium content is slightly less in the zeolite NaXGR and almost same in

Table 1

d Values of standard zeolite NaA, CR and GR NaA and Mg A, Ref. [24]

Std.	Na zeolites		Mg zeolites	
	NaACR	NaAGR	MgACR	MgAGR
12.31	12.46	12.28	12.46	12.28
8.70	8.77	8.68	8.77	8.68
7.10	7.15	7.10	7.15	7.10
5.50	5.54	5.51	5.54	5.51
4.16	4.13	4.10	4.13	4.10
3.71	3.74	3.71	3.74	3.71
3.41	3.43	3.41	3.43	3.41
3.29	3.31	3.30	3.31	3.30
2.98	3.00	2.99	3.00	2.99
2.90	2.92	2.97	2.92	2.97
2.75	2.77	2.76	2.77	2.76
2.62	2.64	2.62	2.64	2.62
2.51	2.52	2.51	2.52	2.51

Table 2

d Values of standard zeolite NaX, CR and GR NaX and Mg X, Ref. [24]

Std.	Na zeolites		Mg zeolites	
	NaXCR	NaXGR	MgXCR	MgXGR
14.28	14.31	14.31	14.31	14.31
8.75	8.82	8.81	8.82	8.81
7.46	7.57	7.47	7.57	7.47
5.68	5.76	5.76	5.76	5.76
4.76	4.83	4.79	4.83	4.79
4.37	4.44	4.44	4.44	4.44
3.91	3.98	3.98	3.98	3.98
3.46	3.52	3.51	3.52	2.51
3.31	3.36	3.36	3.36	3.36
3.02	3.07	3.08	3.07	3.08
2.92	2.96	2.96	2.96	2.96
2.86	2.90	2.89	2.90	2.89
2.81	2.81	2.81	2.81	2.81
2.63	2.67	2.67	2.67	2.67
2.43	2.41	2.41	2.41	2.41

Table 3

Chemical analysis of Na zeolites

	NaACR	NaXCR	NaAGR	NaXGR
SiO ₂	28.68	34.01	28.97	33.43
Al ₂ O ₃	24.92	23.46	21.11	22.78
Fe ₂ O ₃	0.48	0.35	0.20	0.15
TiO ₂	0.73	0.78	0.05	0.05
Na ₂ O	12.61	12.03	12.81	10.78
Si/Al	0.98	1.23	1.17	1.24
Brightness ISO	78.19	74.91	90.09	90.62

other zeolites. The iron and titanium percentages are higher in the clay-based samples, which originally come from the parent clay. The brightness is affected by these coloured impurities and this is evident from the values

Table 4
Particle size distribution of the zeolites

	CR zeolites		GR zeolites		Kaolin
	NaA	NaX	NaA	NaX	
Microns +45	0.98	32.14	47.00	29.00	0.8
Microns –45 +10	1.24	14.43	11.26	10.65	2.5
Microns –10 +2	96.54	50.90	39.75	57.69	16.7
Microns –2	1.24	2.55	1.99	2.66	80.0

given in Table 3. The particle size distribution shows tremendous change when the ultrafine kaolin is converted to the coarser narrow size ranged particles of zeolite.

The chemical analysis of the magnesium exchanged zeolite show that the exchange is only partial especially in MgXGR (Table 5). Hence, all the samples contain some amount of Na₂O the presence of which plays an important role in determining the sintering behaviour of the zeolites.

3.3. Thermal analysis

The thermal analysis data of the sodium and magnesium zeolites are given in Table 6. The sodium forms of the CR zeolites have endotherms at ~188 °C whereas the GR products show endotherms at lower temperatures especially the NaAGR. This endotherm corresponds to the dehydroxylation and the zeolite NaAGR has the maximum weight loss on heating. All the zeolites show exotherms at higher temperature (750–950 °C) which can be attributed to the decomposition of the

Table 5
Chemical analysis of Mg exchanged zeolites

	MgACR	MgXCR	MgAGR	MgXGR
SiO ₂	32.9	36.22	31.24	38.89
Al ₂ O ₃	27.95	23.56	24.47	24.75
Fe ₂ O ₃	0.42	0.61	0.25	0.25
TiO ₂	0.67	0.66	0.06	0.05
Na ₂ O	2.26	2.92	2.17	4.15
Si/Al	1.00	1.30	1.00	1.33
MgO	7.54	7.68	10.09	4.95

Table 6
Thermal analysis data of the zeolites

Samples	Endotherm °C	Exotherm °C	Loss % TGA
NaACR	188	924	21.8
NaXCR	188	896	23.5
NaAGR	124	759	23.8
NaXGR	170	942	21.2
MgACR	160, 232	349, 837, 1014	24.9
MgXCR	203	337, 906, 1031	29.4
MgAGR	196	371, 752, 880, 975	25.2
MgXGR	196	373, 722, 927, 997	24.0

zeolite structure and crystallisation of new phases. This reaction also takes place at a lower temperature in zeolite NaAGR.

The endotherms of the magnesium zeolites corresponding to dehydroxylation are found at a higher temperature compared to their sodium forms. The loss in weight on heating is almost in the same range for the entire ion exchanged forms. The higher values compared to the Na zeolites are due to some NH₄⁺ ions remaining in the sample. Three to four exotherms are clearly observed within the temperature range 330–1030 °C, which are indicative of more than one reaction or crystallisation of more phases. The exotherm at ~350 °C corresponds to the liberation of NH₃ (left out during ion exchange). Other exotherms can be assigned to structural collapse of zeolite giving amorphous product and crystallisation of different phases at higher temperature.

3.4. Sintering studies

The sodium zeolites NaA and NaX prepared by clay and gel route and their magnesium exchanged forms heated at different temperatures were analysed by X-ray diffraction for the phase formation. The physical properties like water absorption, shrinkage and apparent density of the sample pellets were also determined. This study gives information on the sintering behaviour of the zeolites. X-ray diffraction analysis indicate reactions like amorphisation of zeolites, crystallisation of various phases in the system etc. taking place on thermal treatment of the zeolites and Figs. 1–4 give the XRD patterns. It is reported that the temperature at which the zeolites become amorphous (T_{am}) derived by X-ray analysis is an unambiguous measure of the thermal stability of zeolites. This is the temperature at which the lattice collapse begins. However the T_{am} generally does not coincide with the temperature of the first exothermic peak T_1 of the DTA curve [25]. The physical properties are represented in the Figs. 5 and 6. However, it is to be noted that the combinational effect of different ceramic and glassy phases in various quantities is playing a significant role.

3.4.1. Zeolites

The zeolite NaACR is found to be quite stable even at 750 °C and formation of nepheline (NaAlSiO₄), carnegite (NaAlSiO₄) and/or cristobalite (SiO₂) in very small quantities is indicated. The zeolite NaXCR on heating to 750 °C totally decomposes and gives peaks of nepheline, albite (NaAlSi₃O₈), jadeite (NaAlSi₂O₆), carnegite and/or cristobalite. At higher temperatures, the phases formed were the same for both the zeolites but varied in their percentage crystallinity. Zeolite NaACR when fired at 950, 1050 and 1150 °C gives products with almost the same water absorption and shrinkage. The apparent

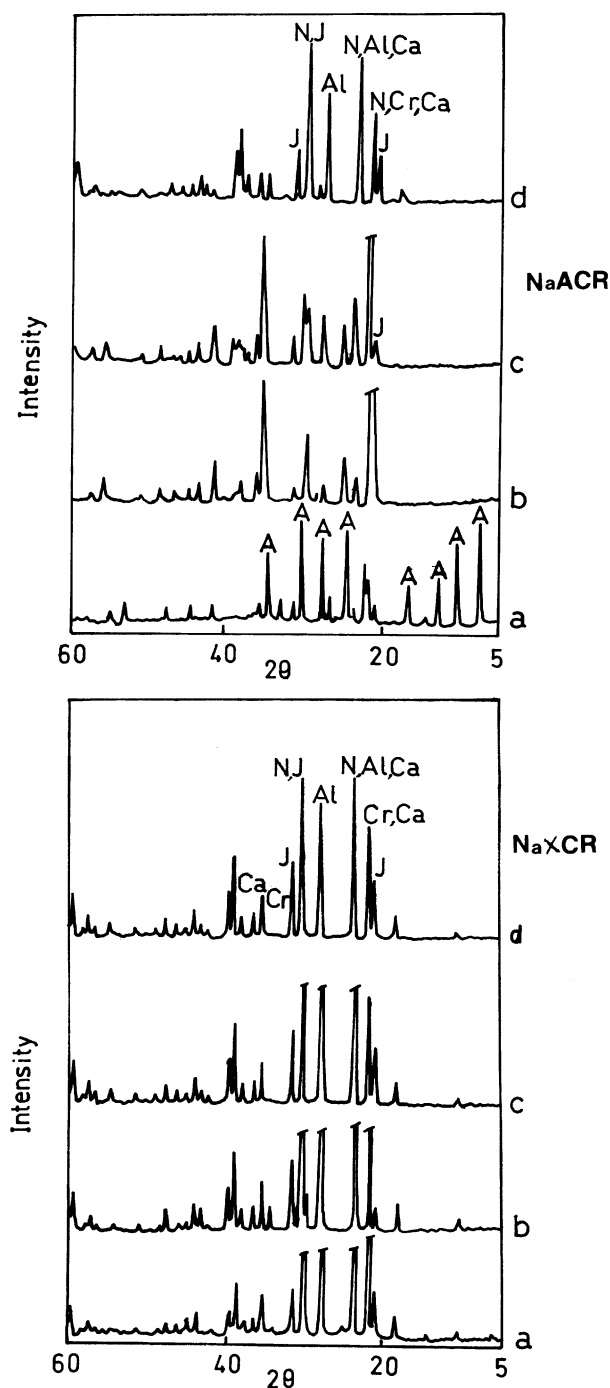


Fig. 1. XRD patterns of CR zeolite NaA and NaX fired at different temperatures; (a) 750 °C; (b) 850 °C; (c) 950 °C; and (d) 1150 °C. A—zeolite NaA; Al—albite; Ca—carnegieite; Cr—cristobalite; J—jadeite; N—nepheline.

density values also do not vary much and all the samples were found to be highly porous. The phases which crystallise in the samples are the same and are found to be nepheline, albite, carnegieite/cristobalite and jadeite. Carnegieite and cristobalite have XRD peaks with almost the same $d\text{\AA}$ values since the crystal structure is the same. Cristobalite is known to have lower density

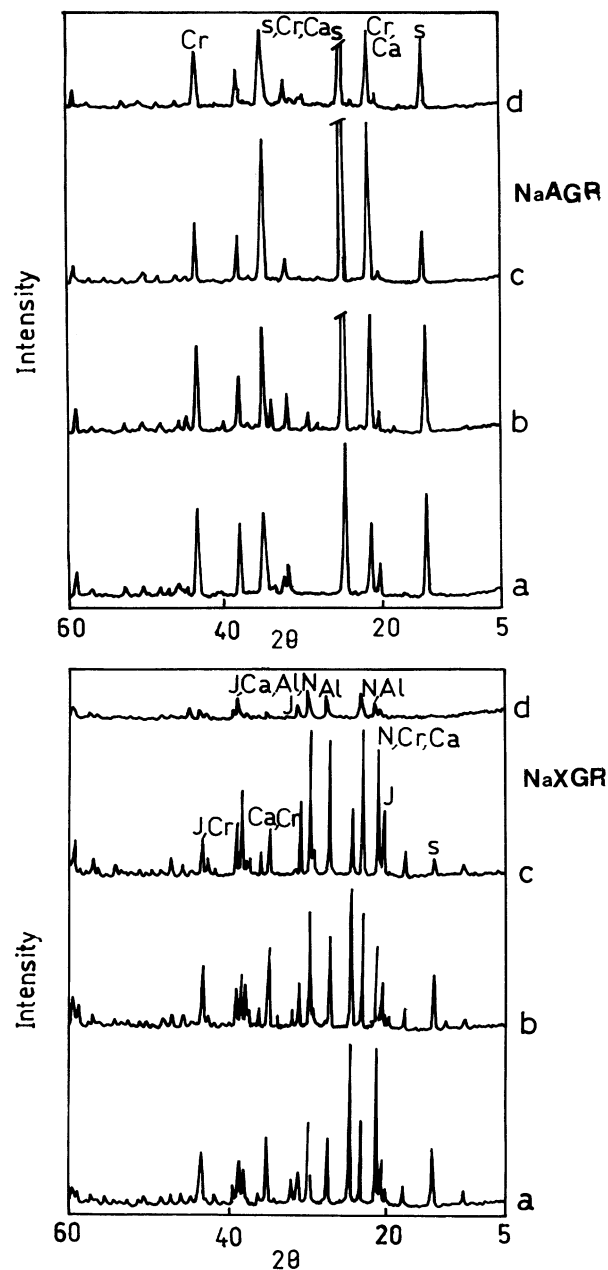


Fig. 2. XRD patterns of CR zeolite NaA and NaX fired at different temperatures; (a) 750 °C; (b) 850 °C; (c) 950 °C; and (d) 1150 °C. Al—albite; Ca—carnegieite; Cr—cristobalite; J—jadeite; N—nepheline; S—hydroxy sodalite.

compared to carnegieite. The low density and high porosity of the NaACR products indicate that cristobalite and not carnegieite might have formed in these products. The zeolite NaXCR on firing at this temperature range shows sintering as indicated by the decrease in water absorption with corresponding increase in shrinkage. At 1150 °C, the products become nonporous with high shrinkage. The apparent density increases with the firing temperature. The low porosity and higher apparent densities of the products indicate that carnegieite might have preferably formed from this zeolite.

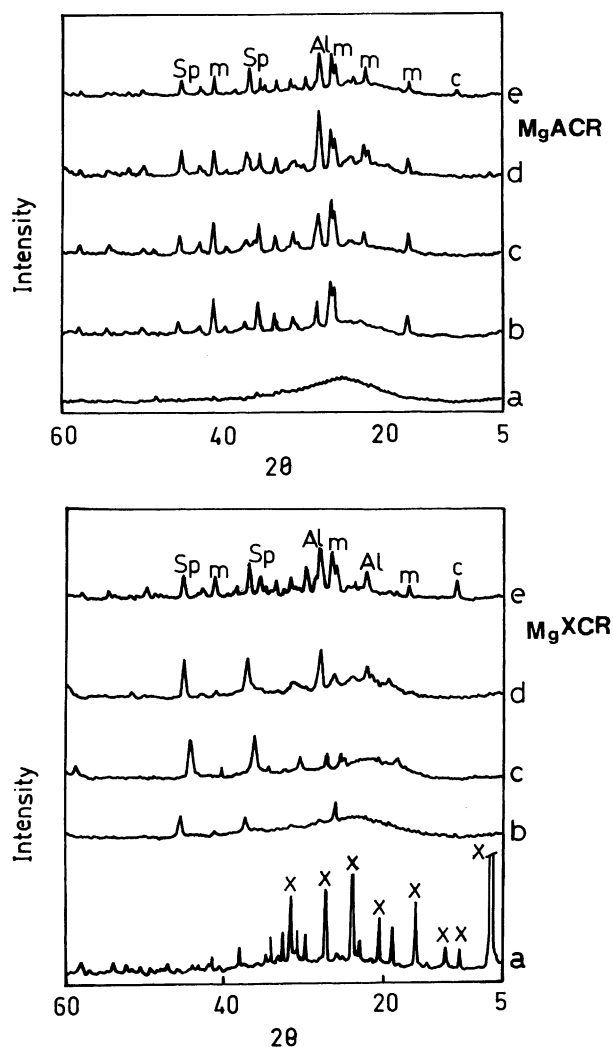


Fig. 3. XRD patterns of CR zeolite MgA and MgX fired at different temperatures; (a) 750 °C; (b) 850 °C; (c) 950 °C; (d) 1050 °C and (e) 1150 °C. Al—albite; C—cordierite; m—mullite; Sp—spinal; X—zeolite NaX.

The GR zeolites formed highly crystalline sodalite ($\text{Na}_6\text{Al}_6\text{Si}_6\text{O}_{24} \cdot 8\text{H}_2\text{O}$) at 750 °C. NaAGR gave cristobalite/carnegeite along with sodalite and the latter decreases with increase in the firing temperature. NaXGR gave nepheline, albite, and jadeite in addition to these two phases. Sodalite becomes almost insignificant in the products fired at 1150 °C. Some alkali might have remained in these zeolites due to improper washing, which favoured the formation of sodalite on heating.

The NaAGR zeolite at 950 °C gives product with water absorption ~5% which becomes almost zero at 1050 °C and remains the same at 1150 °C. The shrinkage and apparent density values do not vary much in the temperature range 950–1150 °C. NaXGR products at 950 °C are highly porous and the porosity decreases with firing temperature and reaches almost zero at 1150 °C when sintering occurs. The apparent density

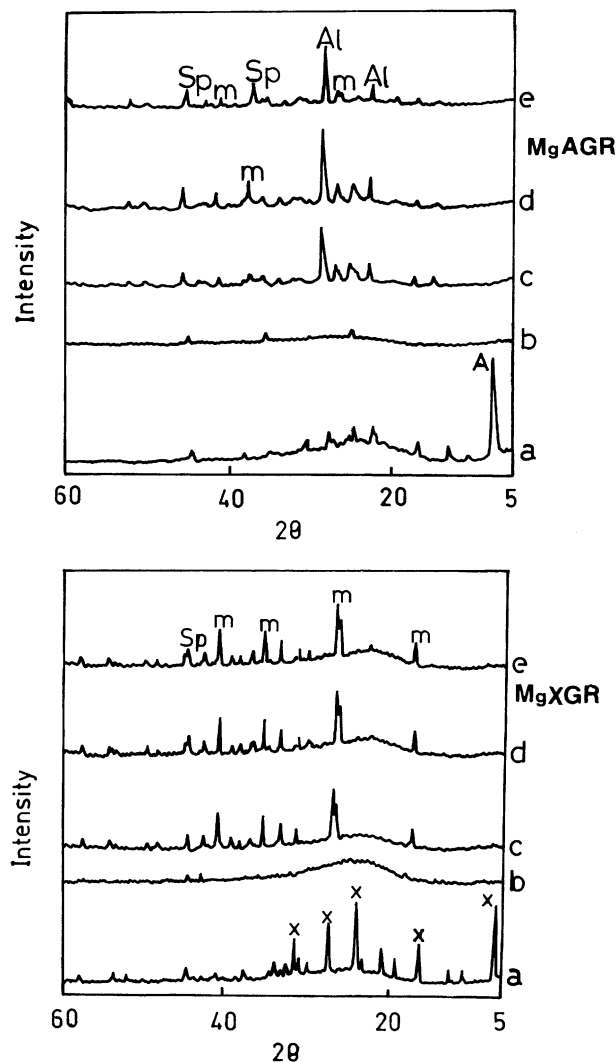


Fig. 4. XRD patterns of CR zeolite MgA and MgX fired at different temperatures; (a) 750 °C; (b) 850 °C; (c) 950 °C; (d) 1050 °C and (e) 1150 °C. A—zeolite; Al—albite; m—mullite; Sp—spinal; X—zeolite NaX.

and shrinkage increase with firing temperature, to some extent.

During sintering, crystalline forms of silica often appear as a secondary phase in the silica-rich compositions and this may be quartz, cristobalite or a mixture of the two depending upon the heat treatment and the presence of certain minor constituents, notably the oxides of alkali metals. Sometimes aluminosilicate crystals having structures close to those of crystalline forms of silica are also formed. The basic principles governing this formation were first formulated by Buerger [26]. The essential feature is that AlO_4 tetrahedra replace SiO_4 groups in the crystal lattice and in order to maintain overall electroneutrality, monovalent and divalent cations are accommodated in the interstitial sites. Quartz, cristobalite, tridymite and keatite, which are crystalline modifications of silica, have aluminosilicate

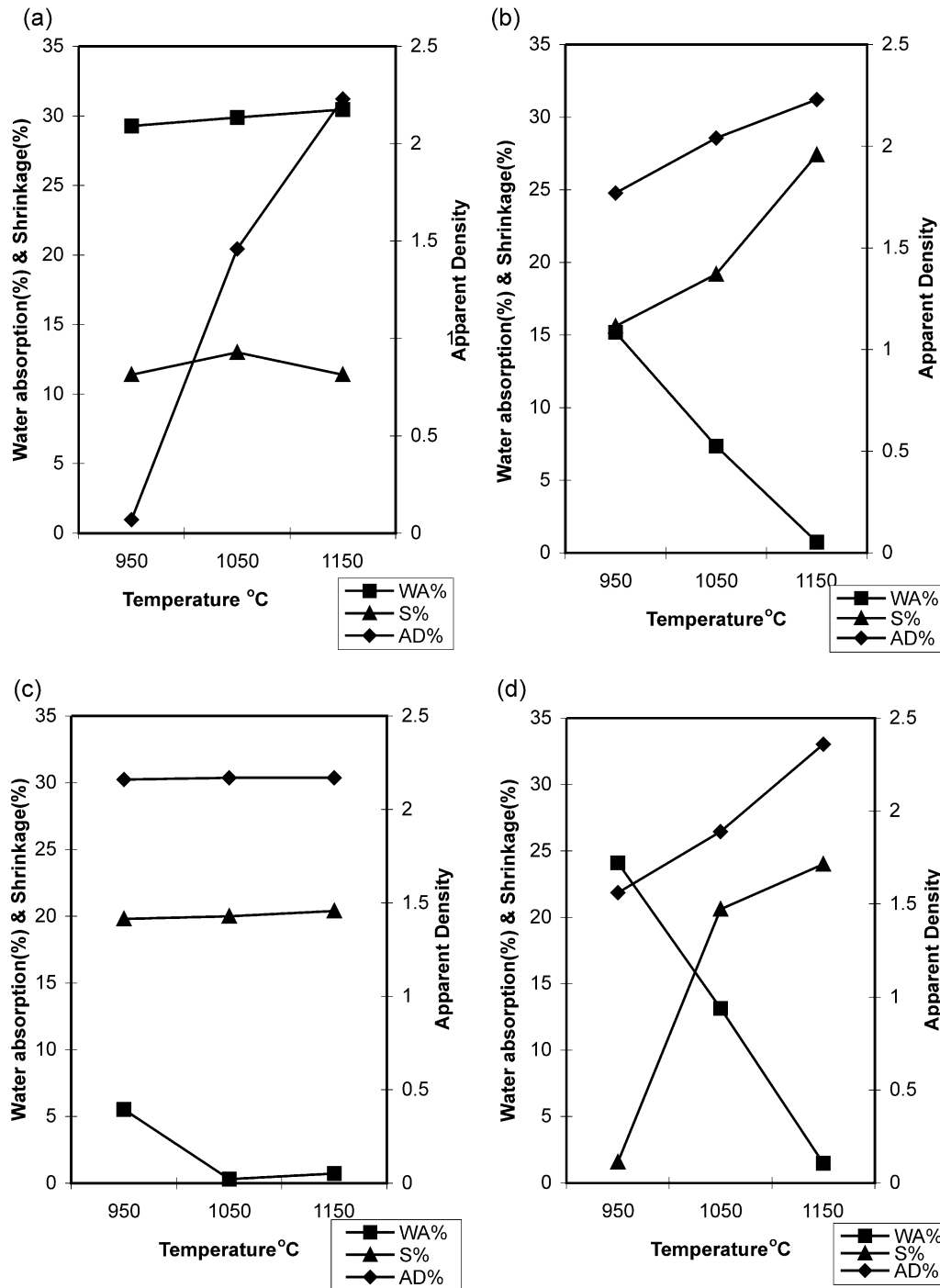


Fig. 5. Changes in properties of Na zeolites fired at different temperatures. (a) CRNaA; (b) CRNaX; (c) GRNaA and (d) GRNaX.

equivalents. These include carnegite and nepheline having cristobalite and tridymite structure respectively. Hence, it may be difficult to distinguish these silica forms from their aluminosilicate analogues based on XRD pattern.

3.4.2. Magnesium zeolites

The zeolite MgACR becomes amorphous on heating to 750 °C and, crystallisation of mullite ($\text{Al}_6\text{Si}_2\text{O}_{13}$),

albite and magnesium spinel (MgAl_2O_4) is observed at higher temperatures. Depending upon the firing temperature, there is some variation in the peak heights indicating the amounts crystallised. At 1150 °C, α -cordierite is found to crystallise. Almost similar behaviour is observed in the case of zeolite MgXCR except for the fact that the zeolite structure is more stable at 750 °C. At 850 °C, the product is almost amorphous with small amounts of mullite and spinel. At 950 °C and above,

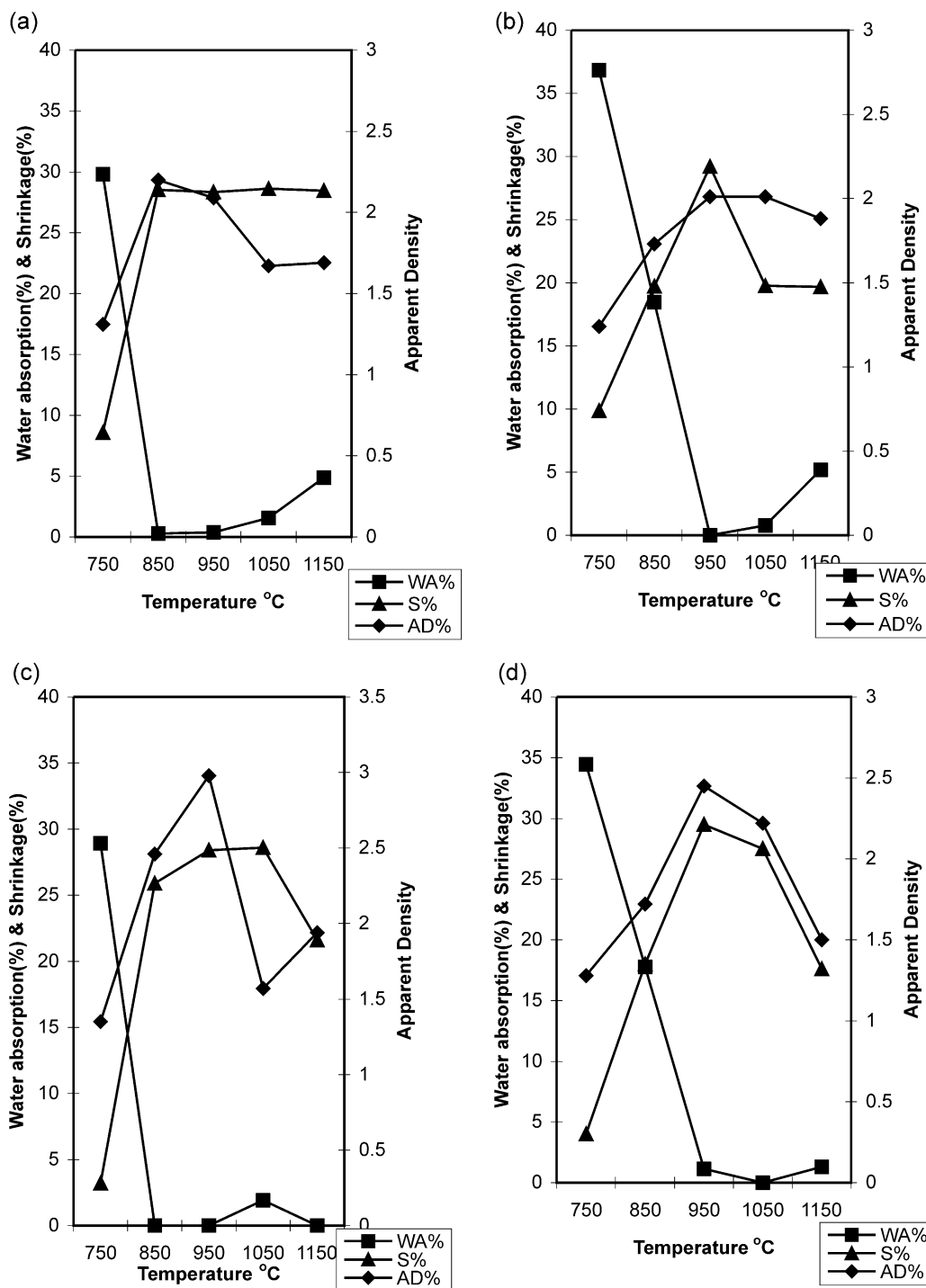


Fig. 6. Changes in properties of Na zeolites fired at different temperatures. (a) CRMgA; (b) CRMgX; (c) GRMgA and (d) GRMgX.

spinel, albite and mullite crystallise and albite becomes the predominant phase at 1050 and 1150 °C. The most important observation is that α -cordierite starts crystallising for the clay based zeolites.

MgACR zeolite is almost sintered at 850 °C with very low water absorption and high shrinkage. The water absorption increases with the firing temperature indicating that open porosity is introduced on heating. The

apparent density decrease with the temperature and the shrinkage does not change. The MgXCR is highly porous at 750 °C and becomes compact as the temperature is increased to 950 °C where water absorption is almost zero. The highest shrinkage and apparent density are also found for this sample. On further heating, a tendency to volume expansion is observed with increase in porosity, decrease in shrinkage and apparent density.

The tendency to volume expansion at higher temperature may be due to the formation of β -quartz or μ -cordierite which facilitates the crystallisation of α -cordierite.

Both the zeolites MgAGR and MgXGR retain some crystallinity at 750 °C and the crystallinity is lower for the former. Firing at 850 °C results in amorphisation of both the zeolites. At higher temperatures, albite, spinel and mullite are formed for MgAGR and the percent of albite increases with temperature. On the contrary, only mullite and spinel phases are formed from MgXGR. Crystallinity of the products remains almost the same from 950 to 1150 °C for MgXGR. However, cordierite formation was not observed within the 750–1150 °C range for the GR zeolites unlike in their CR analogues.

MgAGR is highly porous at 750 °C, mostly amorphous in nature containing small amounts of the zeolite and its shrinkage and apparent density are low. There is a sudden decrease in porosity at 850 °C to almost zero indicating that sintering has taken place at this temperature with the formation of X-ray amorphous glass phase. At 950 °C, the water absorption remains the same, which then increases with the firing temperature. The apparent density and shrinkage increase initially and then decrease with the temperature. MgXGR also behaves in a similar way with respect to these properties. The maximum sintering is observed for the products obtained by heating at 950 °C for both the zeolites.

The volume changes occurring during the formation of glass ceramics is attributed to the overall changes in the specific gravities of the crystalline phases formed as compared to those of the parent glass. The crystalline phases formed may have higher or lower densities than the glass so that the net effect of crystallisation can cause an expansion or contraction of the material. The rate of temperature rise is the most important factor, which controls the volume changes occurring during firing. This has been kept the same for all the firing schedules in the present study. Increase in water absorption and decrease in shrinkage and apparent density can be directly related to the volume expansion. The NaA zeolites do not show any contraction or expansion in the temperature range 950–1150 °C whereas contraction is found in the sodium X zeolites. On the contrary, volume expansion is detected in all the magnesium zeolites and is higher for the clay-based forms. The origin of the zeolites may be attributed to this behaviour. The influence of ancillary minerals of iron and titanium present in china clay on the sintering behaviour of clay based calcium and ammonium zeolites has been described in our earlier studies [27,28]. Stuffed β -quartz or μ -cordierite which facilitates the crystallisation of α -cordierite may be forming accompanied by some volume expansion. At 1150 °C, the clay-based zeolites give α -cordierite and the gel route zeolites may give the same at a higher temperature.

4. Conclusions

(i) The sodium forms of the low silica zeolites A and X synthesised from china clay give albite, nepheline and jadeite on thermal treatment. Cristobalite appears in the case of zeolite A whereas its aluminosilicate analogue, carnegite is formed from zeolite X.

(ii) The sodium forms of gel route zeolites A and X form sodalite as the major crystalline phase on heating and this may be due to the presence of some alkali in the zeolites. At higher temperatures, carnegite is also formed from zeolite A whereas nepheline, albite and jadeite are the additional phases crystallised from zeolite X.

(iii) The magnesium exchange was not complete in any of the zeolites and the exchanged forms were found to be more resistant to temperature compared to their sodium forms.

(iv) Mullite and magnesium spinel were the major crystalline phases formed when the magnesium exchanged zeolites were heated. In the case of clay based zeolites, α -cordierite also crystallised at 1150 °C. The origin of the zeolite i.e., clay based or gel route form, appears to have some influence on the sintering behaviour.

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