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Thermal Behavior of Group V Zeolites from Marathwada, India

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Abstract **- The zeolites natrolite, scolecite and mesolite are group V zeolites .These zeolites were collected from different parts of Marathwada region (M.S.), India. The collected zeolites were characterized by x-ray diffraction, infrared spectroscopy, thermal analysis (TGA/DTA) and wet chemical analysis method. It has found that the collected zeolites are of low silica type zeolites and possess thermal stability up to 500^0C.**

Keywords: Natrolite, Scolecite, Mesolite ,Thermal Stability

1. INTRODUCTION

The natrolite ,scolecite and mesolite are naturally occurring fibrous zeolites belonging to group V of zeolites[1]. The common structure of these zeolites have been studied by many researchers [2-4]. The framework structure of these zeolites consists of cross linked chains of $(Si,Al)O_4$ tetrahedra. An individual chain is comprised of linked units of five tetrahedra and cross linking the chains, produce the framework of these zeolites. The characteristics fibrous habit of the zeolites is parallel to c-axis. Two types of channels run through the structure ,produce small cavities contain the water molecules and exchangeable cations. The main channels which run perpendicular to c-axis between adjacent chains have a minimum diameter of $2.60A^0$ as compare to $2.80A^0$ for the c-parallel channels. The cations Na^+ and Ca^{+2} along with the water molecules are arranged in the channels in clusters i.e. the water molecules are clustered around the cations. In case of natrolite ,each Na^+ ion has as nearest neighbours four framework oxygen atoms and two water molecules. In scolecite, the Ca^{+2} ions are in 7-fold coordination with four oxygen atoms of framework and three water molecules. In mesolite, water molecules are shared by two adjacent sodium atoms along the channel. The ca^{+2} cations are coordinated to four oxygen atoms[5]. In the present investigation an attempt has been made to study the thermal behavior of these fibrous zeolites which are available in the Marathwada region,India.

2. EXPERIMENTAL

2.1 Materials

The crystals of natrolite and scolecite were collected from the nearby area of Ajantha caves, Aurangabad, India and mesolite was secured from the nearby area of parbhani city ,India. The collected crystals were separated from geodes, then cleaned, crushed and sieved to get 106µm sized crystals. The powdered samples were washed repeatedly with distilled water to remove soluble impurities and then dried.

The characterization of the samples were done by using x-ray diffraction, infrared spectroscopy ,thermal analysis (TGA/DTA) and wet chemical analysis method.

2.2 Chemical Analysis

The chemical analyses of the zeolites were carried out by wet chemical analysis methods. Atomic absorption spectroscopy (Hitachi Z-8000) was used to obtain content of cations of the zeolites. The chemical formulae obtained by chemical analysis for the zeolites are as follows

Natrolite sample

$H_1Na_7K_3\ [Si_{20}\ Al_{11}\ O_{72}]\ 15\ H_2O$

giving Si/Al –1.81

Scolecite sample

$H_2\ Ca_4\ Na\ \ [Si_{22}\ Al_{12}\ O_{72}]\ .\ 19\ H_2O$

giving Si/Al – 1.83

Mesolite sample

$H\ Ca_4\ Na_2\ [Si_{19}\ Al_{11}\ O_{72}]\ 17\ H_2O$

giving Si/Al – 1.72

2.3Thermal Analysis

The TGA/DTA/DTG curves for the zeolites were recorded on Setaram-92 thermal analyzer from ambient temperature to 1000^0C in air atmosphere at a heating rate 10^0C/min. using calcined alumina as a reference material.

3. RESULTS AND DISCUSSION

3.1Thermal study of natrolite

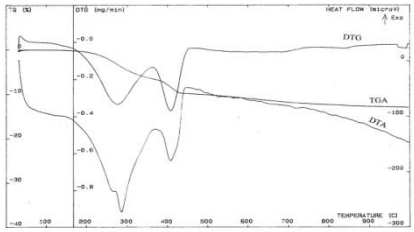

Fig.1: TGA/DTA curves of natrolite sample

Fig.1 depicts the TGA/DTA curves of natrolite sample. From the TGA curves, it has been observed that the dehydration of the zeolite is in two steps, corresponding to two water sites exist in the structure. The first step is observed at about 285^{0}C due to the escape of water molecules from the cations. At this stage weight loss due to dehydration has been estimated about 4%. The second step has been observed at 400^{0}C , may be due to the detachment of water molecules which are linked with the oxygen atoms of the framework . The weight loss at this stage is 9%. A total 10% weight loss has been estimated on heating the sample up to 1000^{0}C.

From the DTA curve, an endotherm has been observed at 280^{0}C due to desorption of water molecules. Because of dehydration , there exists a change in local charge balance. The Na^+ ions tend to move toward the framework oxygen atoms . This results in shrinkage of the framework structure and the new phase α-metanatrolite begins to form at this stage [6]. An exotherm at 380^{0}C corresponds to the completion of this phase . The high temperature exotherm at 460^{0}C attributed to structural collapse [7].

3.2 THERMAL STUDY OF SCOLECITE

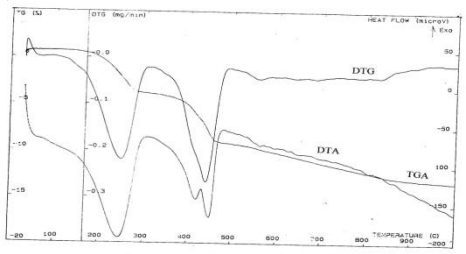

Fig.2: TGA/DTA curves of scolecite sample

Fig.2 shows the TGA/DTA curves of scolecite sample. The TGA curve suggests that the dehydration of scolecite is in two steps, corresponding to two water sites exist in the structure. The first one has been observed at 250^{0}C , showing 4% weight loss and second at 450^{0}C , shows 9% weight loss. A total of 11% weight loss has been estimated on heating the sample up to 1000^{0}C.

From the DTA curve , an endotherm has been observed at 250^{0}C due to loss of water molecules which occupy one of the two Na-sites in the channel. This partially dehydrated scolecite has contraction in the structure. The contraction in the structure is along a and b axis and elongation along c-axis[8]. An exotherm at 300^{0}C suggests the completion of this phase.

The two endotherms of the DTA curve one at 420^{0}C and another at 450^{0}C reveal the loss of water molecules clustered around cations. These two endotherms may be due to two types of cations associated with water molecules.

The high temperature exotherm observed at 490^{0}C attributed to structural collapse.

3.3Thermal study of mesolite

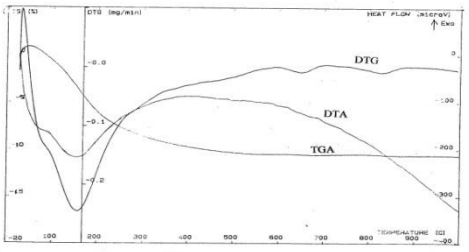

Fig.3: TGA/TDA curves of mesolite sample

Fig.3 indicates TGA/DTA curves of mesolite sample. The TGA curve reveals that the dehydration of mesolite sample is in single step. 9% weight loss due desorption of water has been recorded when sample is heated up to200^{0}C . A total 10% weight loss has been estimated when sample is heated up to 1000^{0}C.

From the DTA curve ,an endotherm is observed at 180^{0}C .this may indicates the detachment of water molecules from the cations. A broad exotherm observed at about 410^{0}C, corresponds to structural collapse.

4. CONCLUSION

The collected group V fibrous zeolites natrolite, scolecite and mesolite are low silica zeolites. TGA study confirms the dehydration of natrolite and scolecite is in two steps and of mesolite is in one step. All the zeolites possess thermal stability up to 500^{0}C.

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