

STUDIES ON CHARACTERIZATION AND THERMAL BEHAVIOUR OF NATROLITE

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ABSTRACT

Characterization of natrolite crystals (secured from Ajanta, Aurangabad District, Maharashtra, India) by X-ray diffraction, IR-Spectroscopy and Chemical analysis was carried out. Thermal behaviour of transparent and translucent crystals was studied using thermogravimetric/differential thermal analysis (TG/DTG/DTA).

Key words: Zeolite, Natrolite, Thermal behaviour.

INTRODUCTION

The structural characteristics of the group V zeolites were established by Pauling¹ and Taylor² followed by several workers³⁻⁷. The zeolites of this group are called fibrous zeolites. All the structures in this group are based upon cross linked chains of tetrahedra. The three possible ways of cross linking the chains produce the three types of framework structures of group V zeolites. One mode is the characteristics of the framework of natrolite, scolecite and mesolite. The characteristics fibrous habit of zeolite crystals is therefore parallel to c-axis. The crystal has Fdd 2 symmetry.

EXPERIMENTAL

Materials

In the present case of investigation the crystals collected from Ajanta caves, Aurangabad M.S. India. The collected crystals of natrolite were separated from geodes. The transparent and translucent part of the crystal were cleaned and crushed and sieved to get 106 µm sized crystals. The samples were characterized in order to investigate the physico-chemical properties using X-ray diffraction, IR, TG/DTG/DTA, Chemical analysis etc.

The samples so prepared were designated as follows :

The transparent natrolites as A and translucent natrolites as B.

Characterization

X-ray Diffraction : The X-ray diffractograms were recorded between 2θ values ranging from 5° to 50° with a scan speed of 1° / min, on a Phillips X-ray diffractometer and Ni filtered CuK_α radiation (λ = 1.5406 Å). The relative intensities and 'd' values are found to be in close agreement with Galli & Alberti⁸ for natrolite samples.

Chemical Analysis

The chemical analysis were carried out by wet chemical analysis methods. Atomic absorption spectroscopy (Hitachi 8000) was used to obtain content of cation of the sample. The chemical formulae obtained by chemical analysis for sample A & B are as follows :

- i) $H_{1.96} N_{96.64} K_{2.4} [Si_{18.12} Al_{11} O_{72}] 14H_2O$
 ii) $H_{1.89} N_{96.61} K_{2.5} [Si_{19.97} Al_{102} O_{72}] 16H_2O$

IR Studies

The infrared spectra were recorded on Perkin - Elmer -221 Spectrometer in freq. range 500-1500 cm using Nujolmull technique. The observed bands and assignment are given in Table 1. The structure sensitive, structure insensitive bands are compared with the reported data and found in close agreement with the standard values. The IR spectra of sample A and sample B are very similar. In the

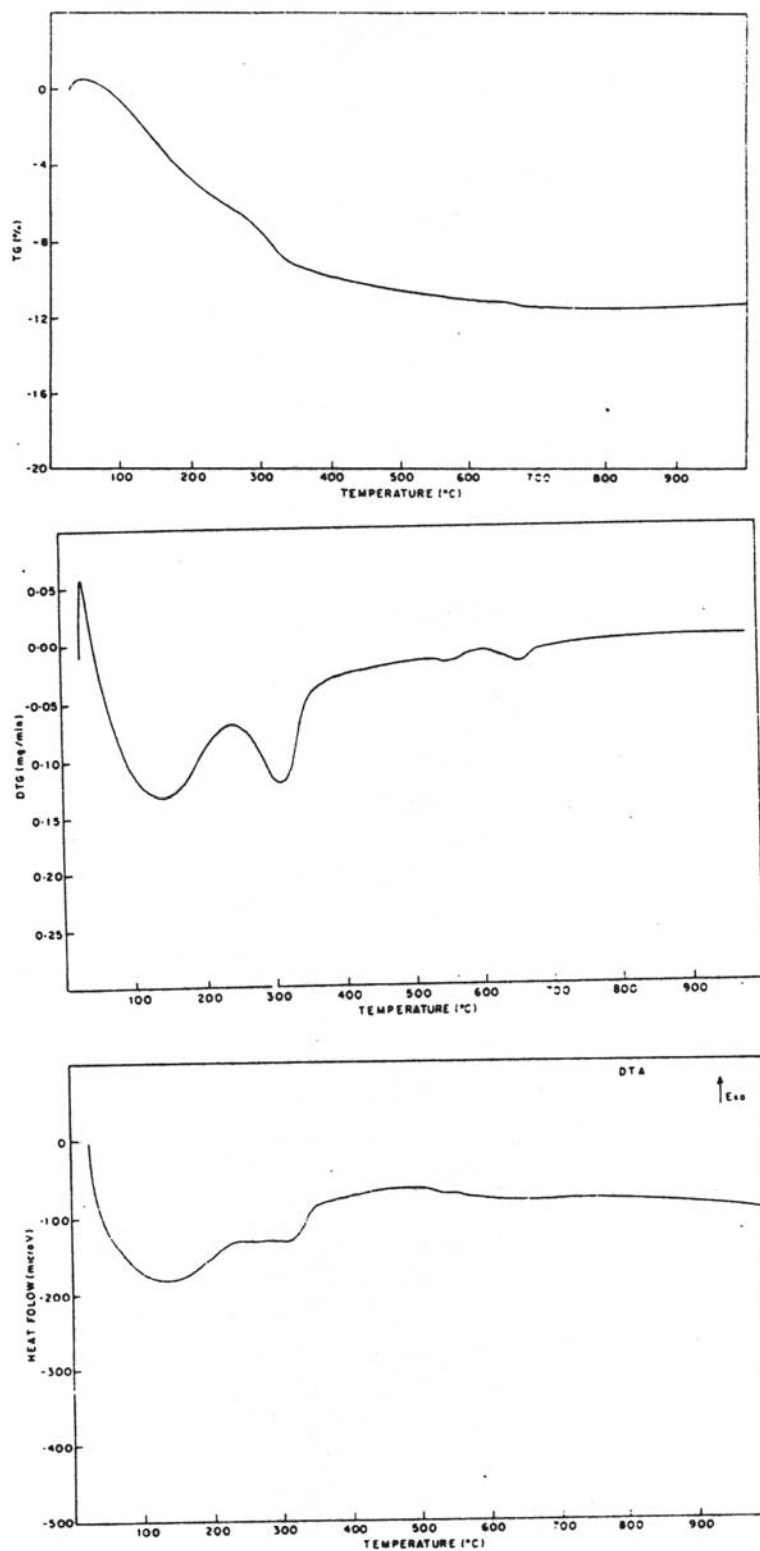


Fig. - 1(a): (TG/DTG/DTA) curves of sample A

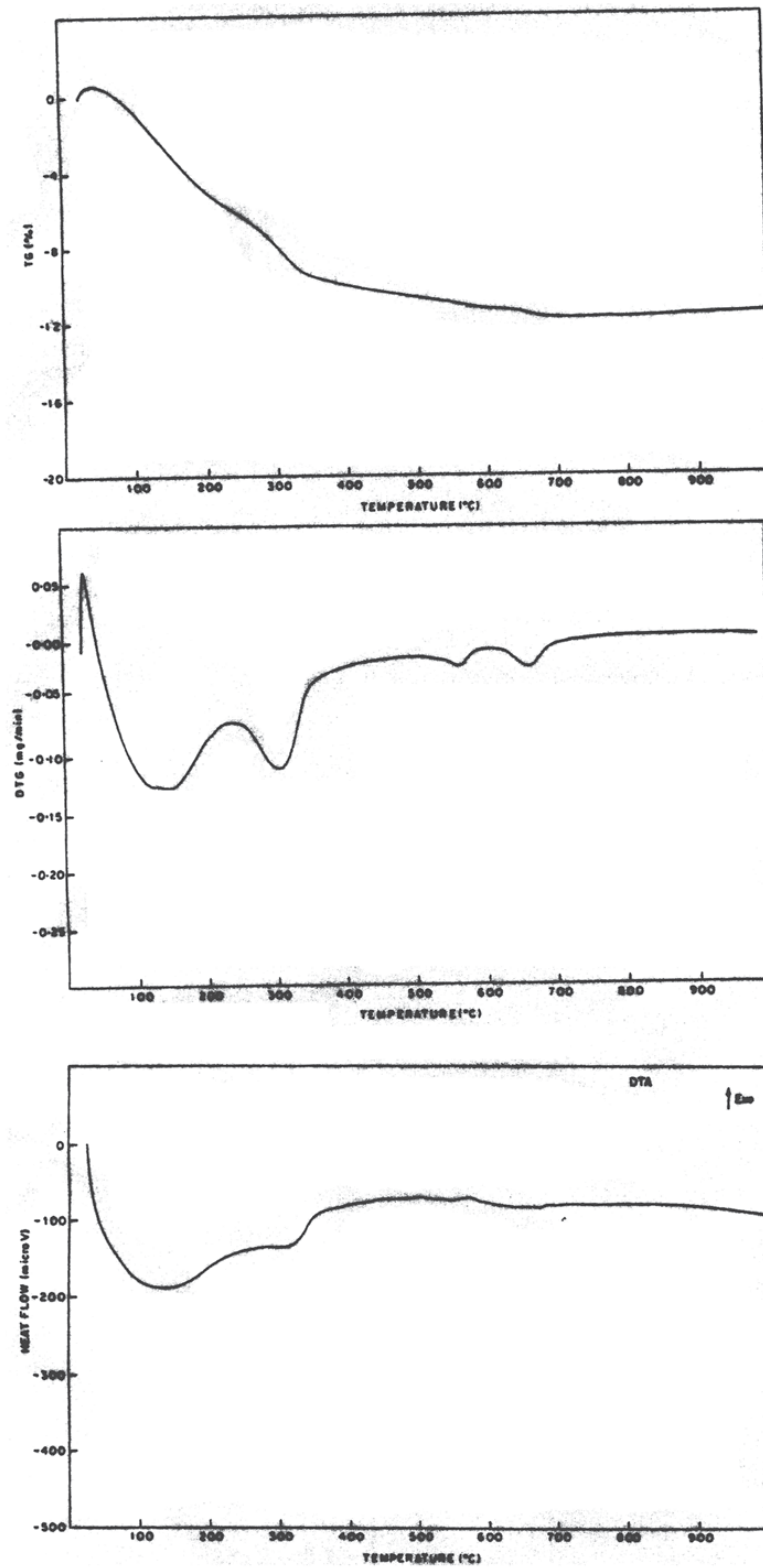


Fig. - 1(b): (TG/DTG/DTA) curves of sample B

present case of study the vibration at 526 cm^{-1} for the double ring in the spectrum of sample A shifts to 522 cm^{-1} in sample B.

Thermal analysis

The TG/DTG/DTA curves were recorded on a Setaram 92-12 thermal analyzer in air using precalcinated α alumina as a reference material. the curves obtained are as shown in Fig. 1(a) & (b).

The thermal properties of natrolite have been studied by many workers⁹⁻¹⁴. The TG/DTG/DTA curves for sample A and B are very similar. It is observed that most of the water is lost upto 550 K. Natural natrolite samples A and B have a sharp endotherm at 588 K due to one step dehydration and a very small endotherm at 820 K. There is an endotherm at 403 K. The endotherm at 588 K is caused by loss of water molecules bonded to Na ions while peak at 403 K is due to removal of water

molecules coordinated around K ions¹⁵.

In sample A, in the temperature range 355-542 K, 6.4%, in the range 577-638 K, 1.6% and in the range 899-986 K, 0.48% of weight loss is observed. Increase of sample B, in the temperature range 364-507 K, 5.76% in the range 559-655 K, 2.44% and finally in the temperature range 873-973 K, 0.48% weight loss is noted. The total weight loss is 11.86% and 11.04% incase of samples A and B respectively. The structure becomes amorphous at 973K in sample A and 986 K in sample B¹⁶⁻¹⁸.

Conclusion

Samples of fibrous zeolites collected maintain high degree of crystallinity. Transparency and translucency incase of natrolite samples in our case of investigation has not affected its thermal behaviour.

Table - 1: IR Data of samples A, B

Sample Number	External Linkage (cm^{-1}) (Structure Sensitive)		Double Ring	Internal Tetrahedra (Structure Insensitive cm^{-1})		
	Asymmetric Stretch	Symmetric Stretch		Asymmetric Stretch	Symmetric Stretch	T - O Bend
A	1370	722	526	1015	669	461
B	1376	722	522	1015	662	464

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