2321-8045 Science Park Research Journal Vol-2, Issue-34, 5th March 2015
Impact Factor : 1.6200[UIF-2013]

Original Article

Characterization And Thermal Behavior Of Natural Stilbite From Marathwada, India

S. D. Ghan

ABSTRACT

Zeolite crystals were collected from the nearby areas of Aurangabad city,(M.S.) India. The collected crystals were characterized as stilbite by using x-ray diffraction, infrared spectroscopy, thermal analysis(TGA/DTA) and wet chemical analysis method. The collected stilbite crystals possess high crystalinity with Si/Al equal to 3.25 and thermally stable up to 500^{0}C.

Keywords:
zeolite, stilbite, thermal stability .

S. D. Ghan

From
Department of Electronics,Yogeshwari Mahavidyalaya, Ambajogai,Dist. Beed (M.S.)

The Article Is Published On March 2015 Issue & Available At
www.scienceparks.in

DOI:10.9780/23218045/1202013/49

Introduction

Stilbite is a naturally occurring platy zeolite belonging to VII group of zeolites[1]. It is the first natural zeolite, discovered by the Cronstedt in 1756.

The structure of stilbite was determined by Galli and Gottardi[2] and refined and studied by many researchers[3-5]. According to these researchers, the structure of stilbite is characterized by large intersecting channels of 10-membered ring of (Si,Al)O4 tetrahedra (diameter 4.9 x6.1?m) and 8-membered ring (diameter 2.7 x4.0?m) . All the channels are occupied by cations and water molecules. The cations have two sites in the channels. One Ca^{+2} fully occupied a site in the center of main channel parallel to a- direction and is completely surrounded by eight water molecules without any coordination with framework oxygen atoms. Second Na^{+} is in a site, in the main channel nearer to the framework so as to coordinate not only water molecules but also framework oxygen atoms. The uses and applications of stilbite have been reported by many researchers[6,7].

In the present investigation, the collected crystals from the nearby areas of Aurangabad city were fully characterized for its zeolitic nature, Si/Al ratio, dehydration behavior and thermal stability.

2. Results And Discussion

2.1 Materials

The as grown samples of stilbite were separated from geodes, then cleaned, crushed and sieved to get 106µm sized crystals. The powdered sample was 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 Characterization

X-ray diffraction studies

Fig.1 shows the x-ray diffractogram of stilbite sample and table .1

indicates the relative intensities and `d` values.The x-ray powder diffractogram of the collected sample was recorded on a Philips diffractometer using Nickel filter ,CuK_{α} radiation (?= 1.5406 A^0) scanning with the chart speed of 40 / min. in the 2? values ranging from 5^0-50^0 at room temperature. The relative intensities and `d` values were compared with the standard `d `values . All the characteristics peaks of stilbite were found to be present in the x-ray diffractogram, hence confirming its identity. The x-ray diffractogram reveals that, the stilbite sample possesses high crystalinity.

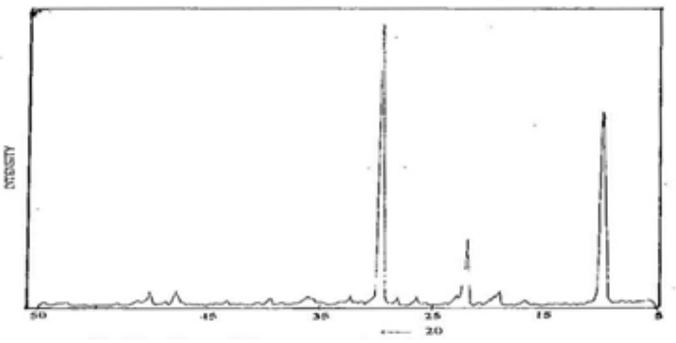

Fig.1: x-ray diffractogram of stilbite sample

Table.1: x-ray powder data for stilbite sample

Intensity	Calculated d(A^0)	Reported d(A^0)
VS	9.073	9.120
VW	5.336	5.374
W	4.667	4.674
W	4.560	4.560
S	4.039	4.039
W	3.169	3.169
S	3.025	3.024
W	2.784	2.784
MS	1.822	1.824

VS-very strong,VW-very weak, S-strong,W-weak

IR Studies

Infrared spectra of the stilbite sample were recorded on `Shimadzu C ` infrared spectrophotometer in range 400cm^{-1}—4000cm^{-1} , using Nujol mull technique as depicted in Fig.2 . The observed frequencies have been assigned with the various structural groups present in the zeolite structure are given in table.2. The IR spectra confirm the zeolitic nature of the sample, the observed frequencies are in good agreement with the reported data[8,9].

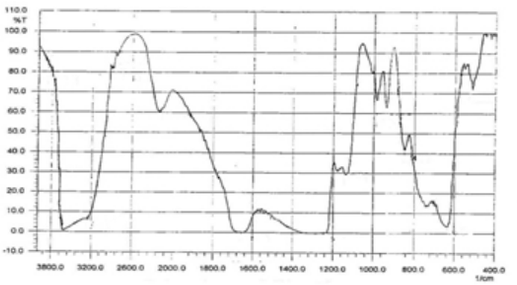

Fig.2: IR Spectra of stilbite sample

Table.2: IR data for stilbite sample

Internal Tetrahedra	Wave number(cm^{-1})
Asymmetric Stretch	985
Symmetric Stretch	632
T-O bend	430
External linkages	Wave number(cm^{-1})
Asymmetric Stretch	1134
Symmetric Stretch	760
Double ring	517
Water bands	Wave number(cm^{-1})
OH Stretch	2906,3248
H_2O Bend	1655

Chemical Analysis

The chemical analysis of the sample was carried out by wet chemical analysis method. Atomic absorption spectroscopy (Hitachi Z-8000) was used to obtain content of cations of the zeolite. The chemical formula obtained by chemical analysis for the stilbite sample is as follow

$$H_1Ca_2Na_3[Si_{26}Al_8O_{72}]23H_2O, \quad \text{giving } Si/Al - 3.25$$

Thermal analysis

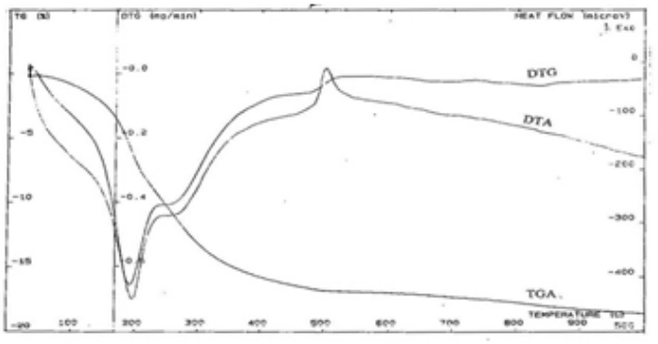

Fig.3: TGA/DTA curves of stilbite sample

TGA Study

The TG curve (Fig.3) reveals that the dehydration of stilbite is in two steps. The first step is observed between 50^0C and 200^0C, showing 8% of weight loss. This may be due to the escape of water molecules which are coordinated to cations. In the second step another 5% of weight loss has been estimated , when the sample is heated up to 290^0C. This weight loss may be due to the detachment of water molecules which are coordinated to cations and framework oxygen atoms. A total of 17% of weight loss has been estimated when the sample is heated up to 1000^0C.

DTA Study

The DTA curve as shown in Fig.3 , a sharp endotherm occurs at 200^0C which is attributed to dehydration of the sample. Due to dehydration there is shrinkage in unit cell, and the new phase-B begins to form at this temperature[10]. A small exotherm at 220^0C corresponds to the completion of this phase. A sharp exotherm is observed at 500^0C, attributed to structural collapse .

3. Conclusion

The sample of stilbite possesses good crystallinity with Si/Al ~ 3.25, and having thermal stability up to 500^{0}C

Acknowlegment

Author expresses sincere thanks to University Grants Commission (WRO),Pune for financial assist to the minor research project.

References

1.D.W. Breck., zeolite molecular sieve, John Wiley(New York),(1972)237
2.E. Galli and G.Gottardi , Natural zeolites Acta Crystalogr., Sect.B,27,(1971)833
3.M.Slaughter , crystal structure of Stilbite, Am. Miner.,55,(1970)387
4.M.Akizuki, Y.Kudov and Y.Satos, Crystal structure of orthorhombic (001)growth sector of stilbite .Eur.J.Miner.,05, (1993)839
5.D Howell, G Johnson, I.Taskar and P O`Hare, thermodynamic properties of zeolite stilbite, Zeolite,10(05),(1990)525-531.
6.K.Blazev,T Ivanova,Z.Panov,V.Paneva. Preliminary investigations into the mineralogy and potential uses of stilbite from Kratovo volcanic area. Comp. rend. Acad.Bulg.Sci.,65(02),(2012)187
7.S.D.Ghan, H.V.Bakshi and B.TBhoskar, Removal of ammonium ions from waste water using natural zeolites, Asian J. Chem.,17(01),(2005) 634
8.F.Pechar and D.Rykl.,Infrared spectra of natural zeolites of the stilbite group, Chem.zvesti,35(2),(1981)189.
9.Flanigen E M ,Khatami H C and Szymanski H A, Molecular sieve zeolites-1 Adv. Chem. Ser.,101,(1971)201
10.Cruciani G, Artioli G, A.Gaultiari, K.Stahl and J.Hanson. Dehydration dynamics of stilbite using synchrotron X-ray powder diffraction ,Am. Miner.,82,(1997)729

S. D. Ghan
Department of Electronics,Yogeshwari Mahavidyalaya, Ambajogai,Dist. Beed (M.S.)