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Dielectric Study of NH4-Stilbite

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ABSTRACT: *Natural Zeolite Stilbite belongs to Group VII were collected near Ellora Ajanta belt. Characterization was made using XRD, IR at NCL Pune. Dielectric study of NH4-Stilbite was studied using LCR Bridge.*

KEYWORDS: *NH4- Stilbite,Characterization,Dielectric study*

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I. INTRODISATION

Natural zeolites are characterized by micro porosity. The micro porosity results from the specific crystalline structure of the zeolite particles which in turn depends upon it's composition. Existence of apertures of fixed diameters causes the molecular sieving action of zeolites. Under ideal conditions some molecules can pass through the apertures to the internal structure filling the available adsorptive space. Others (of larger size) cannot enter and remain on the outer surface of the zeolites grain.

Stilbite is a natural zeolite belonging to the Heulandite group with unit cell formula $Ca_4[(AlO_2)_8(SiO_2)_{28}]28H_2O$(1). The zeolite framework can be described as a fundamental polyhedral configuration containing four and five member rings of tetrahedra. Two sets of interconnected channels exist in the Stilbite structure. A larger 10 – member ring channel with 4.91A°x6.2 A°. window size in the [100] direction and a smaller eight ring channel with the size of 2.7 A° x 5.6 A° in the [101] direction (2). The inner surface should therefore be readily accessible to the small molecules. Stilbite is a monoclinic zeolite .the unit cell constants is a = 13.61A° , b = 18.24A° , c = 11.27A°, β = 127°54' Stilbite zeolite could potentially be used as an adsorbent and catalyst. It is generally believed that collapse of the Stilbite structure during dehydration is due to the presence of exchangeable cations (3). When removing the water molecule legends, the cations must coordinate directly to the oxygen in the framework. Because of their high charge density, the cations can disort the frame work to achieve the best environment for possible coordination. The distortion in Stilbite is so serious that some of the bonds connected to tetrahedral atoms (Silicon or aluminum) breck, rendering the zeolite almost useless as an industrial catalyst or dehydrating agent (4)Since destruction of the Stilbite framework is closely resulted to the exchangeable cations it might be possible to stabilize the framework by reducing the number of exchangeable cations. Beyer et al (5) performed a thermogravimetric study of the de ammonation of NH_4 – Stilbite in oxidizing and insert atmosphere and reported the structure variation of the H-Stilbite thus formed Mortier and coworkers (6) investigates the crystal structure of NH_4 – Stilbite dehydrated at 300°C, and found only a minor distortion with respect to the hydrated form. The framework remains open and the residual cations are located in eight rings.In the case of Stilbite, the hydrogen form is more stable (7,8). The framework of NH_4 – Stilbite heated in a shallow bed reactor remains upto the temperature of 500°C. The thermal stability of the zeolite increases with increasing degree of ammonium exchange. The thermal behaviour of Stilbite is studied recently (9). The collapse of the frame work can be prevented by reduction of the cation / framework interaction.

II. SAMPLE PREPARATIONS

Stilbite were collected from the quaries of Ajanta caves. Sample crushed and sieved to get 106 µm sized crystals for NH_4 – ion exchanged form of Stilbite, sample is treated with 1 M solution of NH_4NO_3 with stirring at 95°C for six hours. To form H-form Stilbite, NH_4- Stilbite heated at 250°C.

III. CHARACTERIZATION

X-ray diffraction- For the characterization of Stilbite, X-ray diffractograms were recorded between 2θ values from 5° to 50° on Phillips (PW 1710) having the wavelength 1.94056 A°. Diffractograms of three forms of Stilbite are recorded.

IR

The IR spectra of Stilbite were recorded on Perkin – Elmer . 221 spectrophotometer in the frequency range 400 to 4000 cm^{-1} , of parent form, NH_4 – form, Parent at 100°C, Parent form at 150°C, Parent form 200 °C & H – Form of Stilbite.
The observed IR bands and assignment are given in fig 2

Chemical Formula of Stilbite

The chemical formula of Stilbite is as follows

1) Parent Stilbite

$Na_{0.5}\ K_{0.07}\ Ca_{3.37}\ [Si_{24.36}Al_{7.30}O_{72}]\ 31\ H_2O$

2) H – form Stilbite

$H_{2.94}\ Ca_{1.0}\ [\ Si_{24.36}Al_{7.3}O_{72}\ ]\ 31\ H_2O$

IV. RESULTS AND DISCUSSION

XRD Pattern of parent Stilbite, NH_4 – form of Stilbite & H – form of Stilbite is shown in fig.1. from diffractogram. There are no major changes in these three forms which confirm the stability of Stilbite. ‘d’ values are compared with standard ‘d’ values.

2 Theta	d-Value	Peak Width	Intensity
09.75	9.08	0.10	100
16.78	5.29	0.24	2.7
19.09	4.65	0.24	11
20.76	4.28	0.12	6.3
21.91	4.06	0.18	56
22.24	4.00	0.12	5.4
23.87	3.73	0.24	4.6
26.25	3.40	0.20	6
28.00	3.19	0.08	11
28.76	3.10	0.24	2.7
29.43	3.04	0.12	25.
29.51	3.03	0.10	32.6
29.81	3.00	0.16	10.6
31.09	2.88	0.24	2
32.25	2.78	0.08	13.5
32.91	2.72	0.12	2.5
35.04	2.56	0.20	4.6
38.36	2.35	0.24	3.6
40.55	2.22	0.16	2
42.57	2.12	0.24	1.7
43.83	2.06	0.32	2.5
44.66	2.03	0.24	3.9
47.90	1.90	0.32	1.4

Table 1- XRD Data For Stilbite (After Background Subtraction)

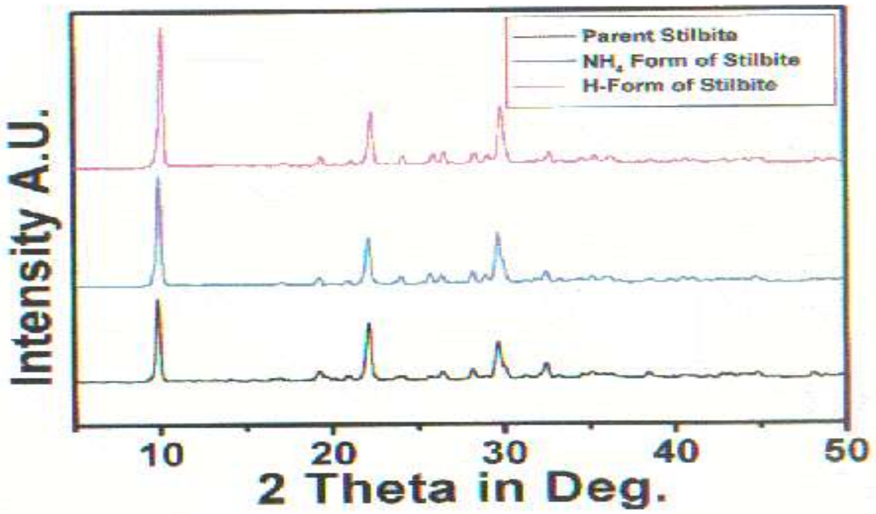

Fig. 1 XRD pattarn of stilbite

IR – From IR studies structure sensitive Asymmetric stretch is at 1417 cm^{-1} and symmetric stretch is at 833 cm^{-1}.Water bands of hydroxy stretch is at 3605 cm^{-1} & water bands at 1659 cm^{-1} . Internal tetrahedral symmetric stretch is at 443 cm^{-1}. Asymmetric stretch is at 1045 cm^{-1} . Assignment for T-O bending is at 443 cm^{-1} . The nature of IR bands is same for parent form, NH$_4$ – form & H – form which confirms the stability of Stilbite.

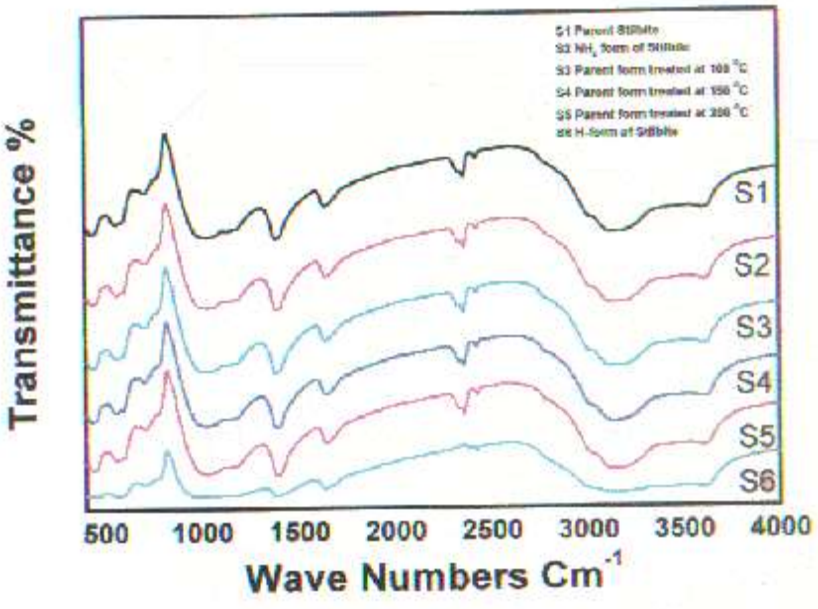

Fig. 2 IR OF Stilbite from 400- 4000

6 Dielectric Study of **NH4 – Stilbite**

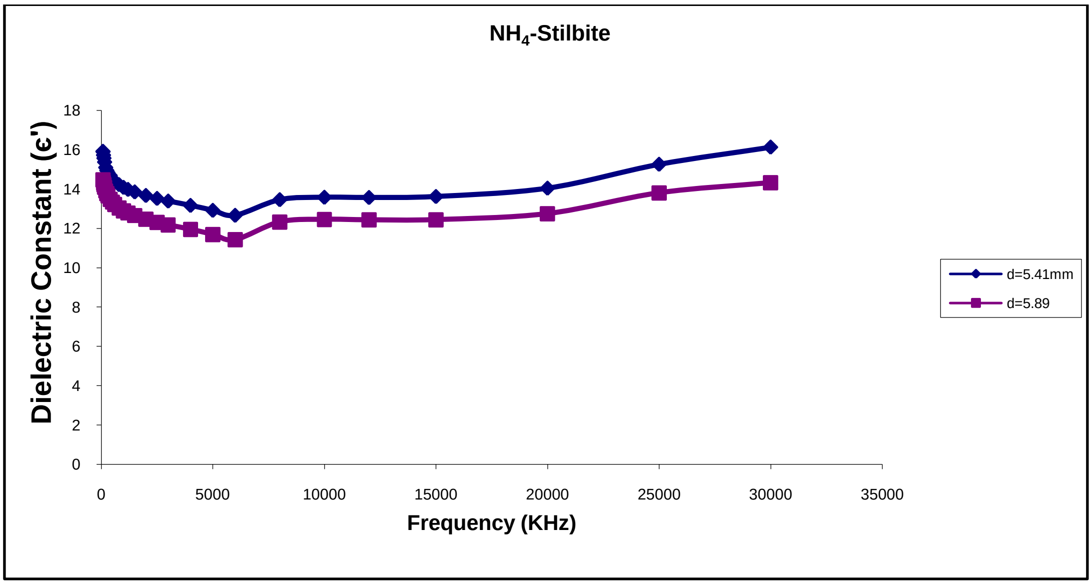

Fig.3 variation of dielectric constant as a frequency in NH_4 stilbite

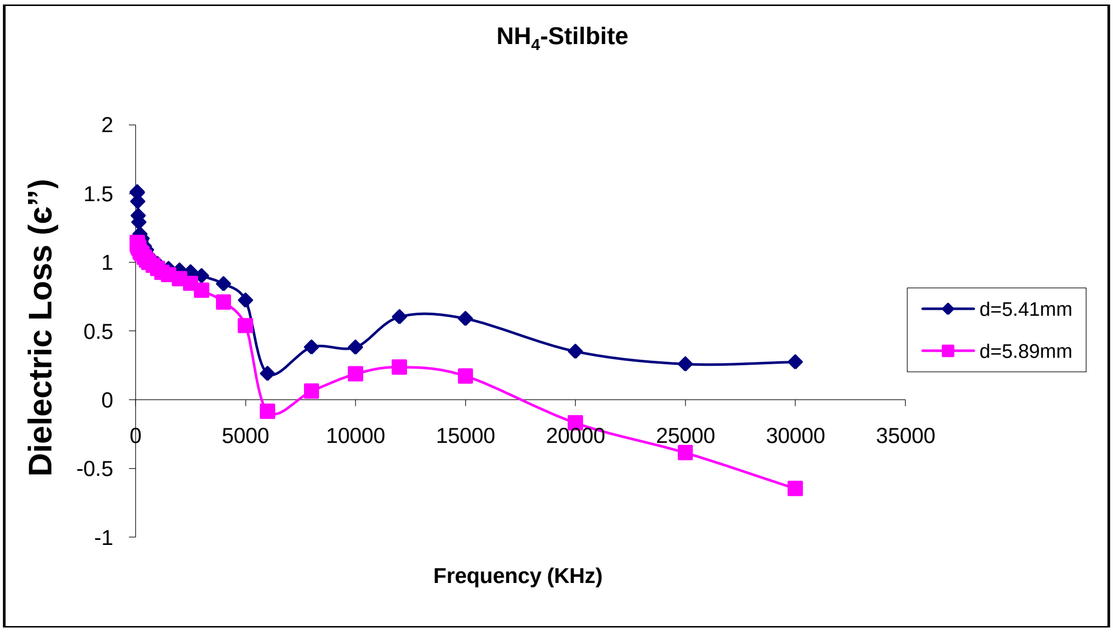

Fig 4 variation of dielectric loss as a frequency in NH_4 stilbite

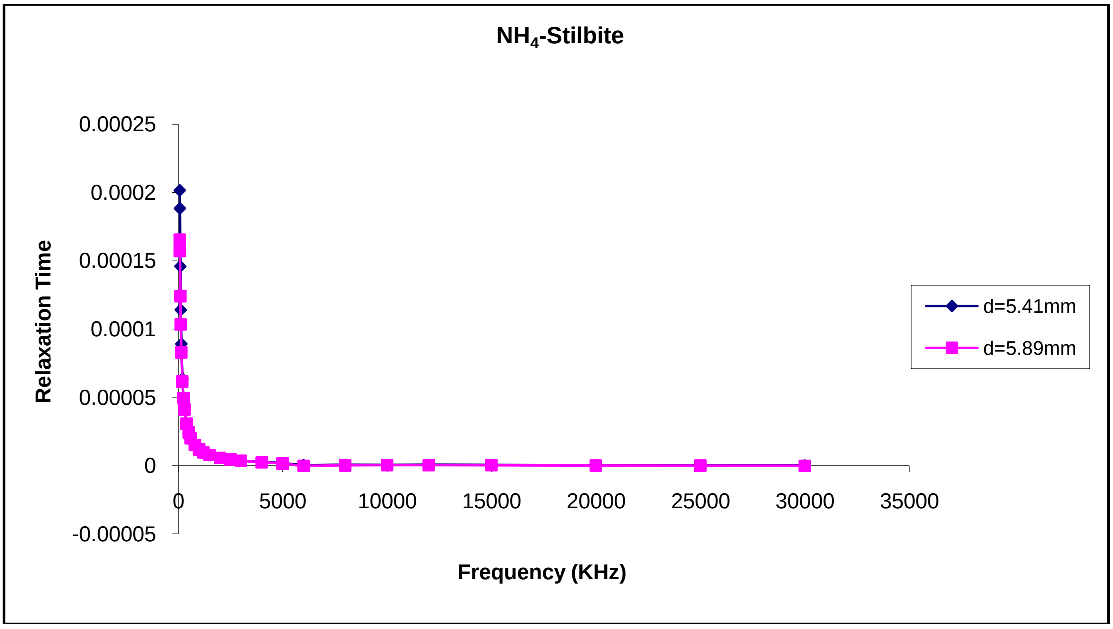

Fig.5 variation of relaxation time as a frequency in NH_4 stilbite

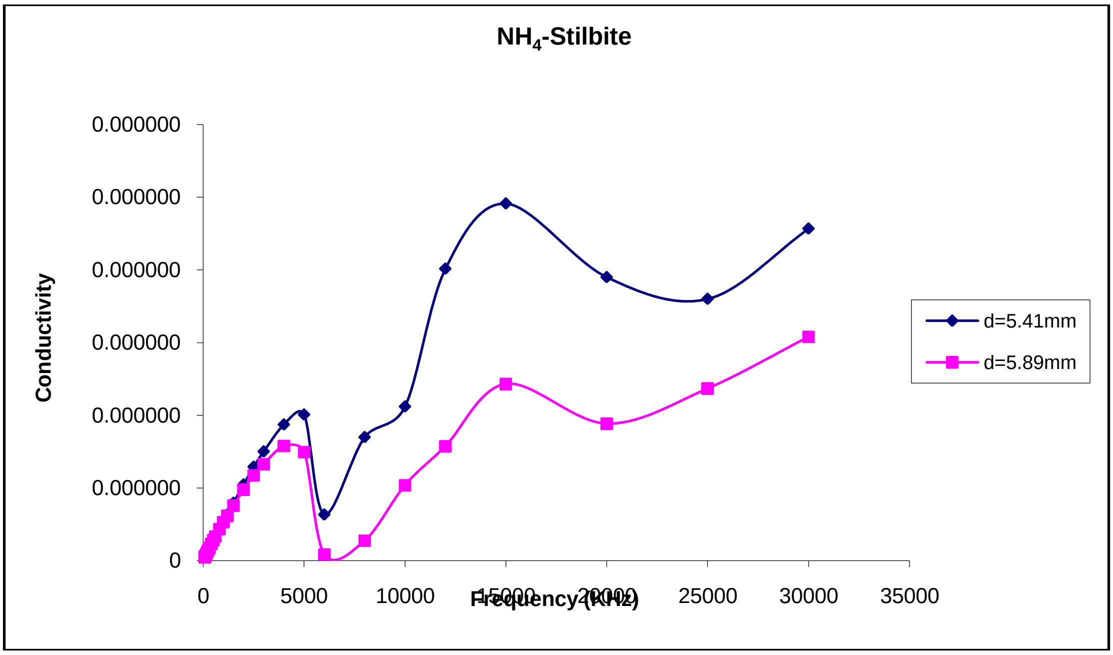

Fig.6 variation of conductivity as a frequency in NH_4 stilbite

Dielectric Constant (Є'):-In fig 3 there is decrease in Є' up to 6000 KHz Then increases slowly or nearly remains constant.

Dielectric Loss (Є''):-In fig 4, Є'' decreases as frequency increases, Є'' decreases up to 5000 KHz Then increases slowly. Then decreases.

Relaxation Time (τ) – Fig 5 Shows Relaxation Time against frequency. As frequency increases Relaxation time decreases.

A.C. Conductivity – Fig 6 Shows A.C. Conductivity verses frequency As frequency increases A.C. Conductivity goes on increasing also conductivity increases as thickness of the sample increases.

V. CONCLUSIONS

1) There is no major change in XRD Pattern of three forms of NH4- Stilbite
2) IR bands confirm the stability of NH4-Stilbite .
3) Dielectric study of NH4-Stilbite plays an important role in stating the nature of zeolite.

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