Characterization and Dielectric study of H- Clinoptilolite

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Abstract— **Clinoptilolite were collected from the quarries of Ajanta caves , Marathwada (Maharashtra). Sample was crushed and sieved to get 106 µm sized crystals for the ion exchanged. The sample is treated with 1 M solution of Ammonium Nitrate with stirring at 95°C. for the six hours to get NH4 ion exchanged form of Clinoptilolite ,again heated at 250°C for 48 hours for getting H – Clinoptilolite. Characterization was made using XRD,IR at NCL Pune Dielectric study was made using LCR Bridge. Pellets of H- Clinoptilolite were prepared..Variation of dielectric constant, dielectric loss, dielectric conductivity and relaxsession time were measured from 20Hz to 20KHz**

Index Terms— **H-Clinoptilolite,Characterization,Dielectric study**

I. Introduction

Natural Zeolite Clinoptilolite is a silica rich zeolite that belongs to 7th group (1)of platy zeolites. Heulandite, another platy zeolite of the same group and Clinoptilolite are isostructural but their thermal stability, Si /Al ratio and the cation contents are different. Boles(2)investigated the relationship between the chemical composition and thermal behavior of these zeolites and proposed the name of the zeolite as Clinoptilolite if Si/Al> 4 and if Si/Al <4, the zeolite is termed as Heulandite. There are two varieties of Clinoptilolite, one silica rich is called as simply Clinoptilolite, whereas the low silica Clinoptilolite is known as ca-clinoptilolite[3].

The unit cell parameter of Clinoptilolite is
a= 17.62A° , b=17.91A° , c=7.39A° , β=116° , 18'

The frame work structure of the Clinoptilolite consists of a common unit which contains 10 nodes, known as the 4-4-1 unit. These units are connected so as to share one or two nodes in zeolites.It is a monoclinic zeolite.

Clinoptilolite has been utilized intensively in environmental applications such as treatment of waste water from nuclear factories (5), remidition of radioactive soils (6), the treatment of sewage and agricultural effluents (7) etc. In such environmental applications, Clinoptilolite is valued for it's high cation exchange selectivity for Cs , Sr and NH_4^+ during ion exchange

Clinoptilolite were collected from the quarries of Ajanta caves , Marathwada (Maharashtra). Sample was crushed and sieved to get 106 µm sized crystals for the ion exchanged. The sample is treated with 1 M solution of Ammonium Nitrate with stirring at 95°C. for the six hours. NH4 ion exchanged form of Clinoptilolite is heated at 250°C for 48 hours for getting H – Clinoptilolite.

Manuscript received March 17, 2022

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II. Characterization

X-ray diffraction patterns were recorded between 2θ values from 5° to 50° on Phillips model (PW 1710)with Cu Kα wavelength= 1.54056 A°. Diffractogram are recorded for the parent Clinoptilolite , NH_4 – exchanged Clinoptilolite and H – form Clinoptilolite. D values & intensities are recorded in table 1

Infrared Studies:

The infrared spectra of Clinoptilolite was recorded on perkin – Elmer – 221 Spectrophotometer in the frequency range 400 – 4000 cm^{-1} of NH_4 – form, H – form & parent form at 100°C, 150°C and 200°C. The observed IR bands and assignments are given in table 2

III. Results and Discussion:

XRD – Pattern of the parent Clinoptilolite, NH_4 – exchanged Clinoptilolite and H – form Clinoptilolite is shown in fig.1. From diffractogram we determine the crystalline nature of Clinoptilolite d- values are compared with standard 'd' values. This confirms the Clinoptilolite structure. There is no major change in diffrcatograms of these three forms. The intensity in NH_4 – Clinoptilolite and H – form Clinoptilolite increases.

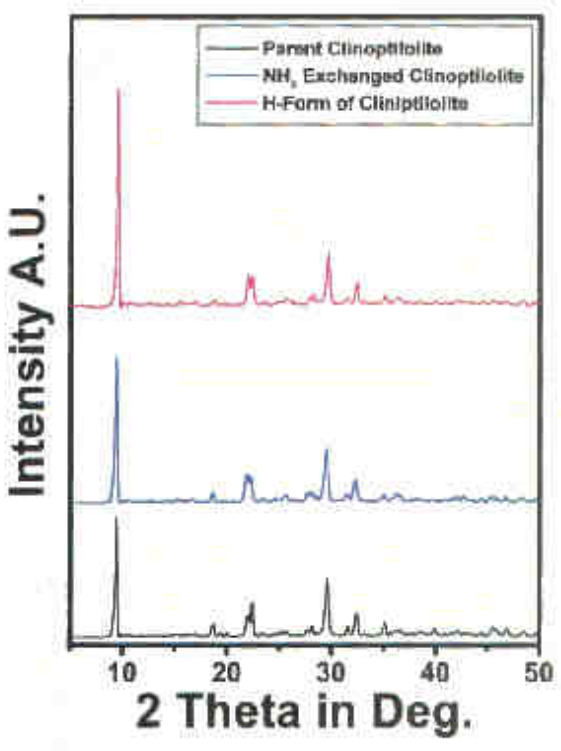

Fig 1 XRD pattarn of clinoptililote

2 Theta	d-Value	Peak Width	Intensity
09.91	08.93	0.12	100
16.64	05.33	0.06	1.3
19.11	04.65	0.06	6.4
19.83	04.48	0.12	1.9
22.28	03.99	0.16	3.7
22.75	03.91	0.16	3.8
23.84	03.73	0.24	0.9
25.46	03.50	0.16	1.2
28.54	03.13	0.16	1.5
29.95	02.98	0.12	15
30.23	02.96	0.16	7.3
31.95	02.80	0.16	1.8
32.89	02.72	0.28	2.7
35.52	02.53	0.20	1.6
40.28	02.24	0.16	2.6
42.47	02.13	0.24	1.1
44.67	02.03	0.64	0.4
45.97	01.97	0.24	1.5
47.27	01.92	0.32	0.6
48.96	01.86	0.40	0.4

Table.1- XRD Data For Clinoptilolite (After Background Subtraction)

IR

IR bands for Clinoptilolite are shown in table 2. in external linkage, asymmetric stretch is observed at 1391 cm^{-1}& symmetric stretch is at 795 cm^{-1}, OH- stretch is at 3625 cm^{-1} and the water bands are 1630 cm^{-1}. Double ring is observed at 598 cm^{-1} . Zeolite structure is insensitive to the asymmetric stretch at 1250 and symmetric stretch at 750 cm^{-1} bands at 490 is observed due to the vibration of Si – O or Al – O bond. As we heat the parent sample at 100°C, 150°C , 200°C & H – form Clinoptilolite there is no major change in IR spectrum this confirms the stability of the Clinoptilolite. Only water bonds become more intense and OH – stretching is more intense than the parent form of Clinoptilolite.

Sample Name	External linkage cm^{-1} Str. sensitive		Double ring	Internal Tetrahedral Str Insensitive cm^{-1}		T – 0 Bend	Water Bands	
	Asymmetric Stretch	Symmetric stretch		Asymmetric Stretch	Symmetric stretch		OH-stretch	H_20 Bands
Clinoptilolite	1391	772	598	1250	750	490	3625	1630

Table2 . IR assignments in Cm^{-1}

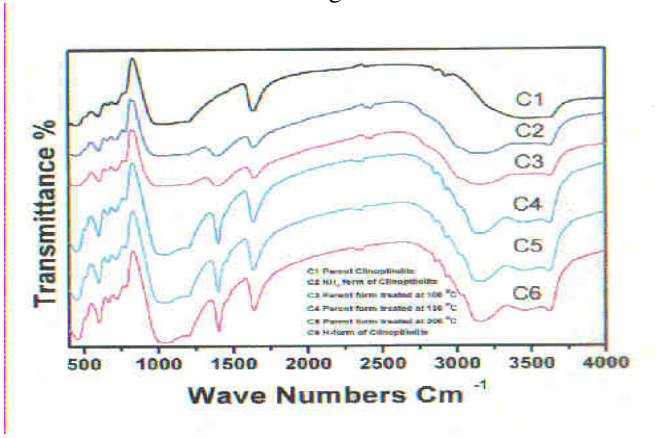

Fig. 2 IR of clinoptilolite from 400-4000

Dielectric studies

Dielectric Constant:-Fig 3 indicates the variation of dielectric constant against frequency in H – form Clinoptilolite Decrease in dielectric constant up to

5000 KHz is observed. Then dielectric constant slowly increases or nearly remains constant.

Dielectric Loss (Є'') :- Fig 4 shows the variation of dielectric loss to the frequency of

H – form Clinoptilolite. This shows that decrease in Є'' is observed as increase in the frequency up to 5000 KHz. Decrease is fast. Then Є'' is remain constant.

Relaxation Time (τ) :- Fig 5 shows the variation of relaxation time with frequency of

H – form Clinoptilolite. This shows that decrease in τ is observed in H- Clinoptilolite as increase in the frequency.

A.C. Conductivity (б) :- Fig 6 shows the variation in AC Conductivity with frequency

inH – form Clinoptilolite . From fig. б conductivity goes on increasing as frequency increases linearly.

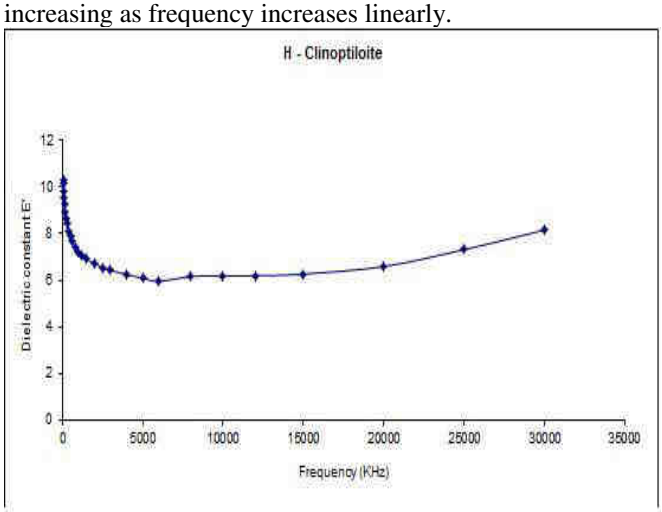

Fig. 3 Variation of dielectric constant as a frequency in H clinoptilolite

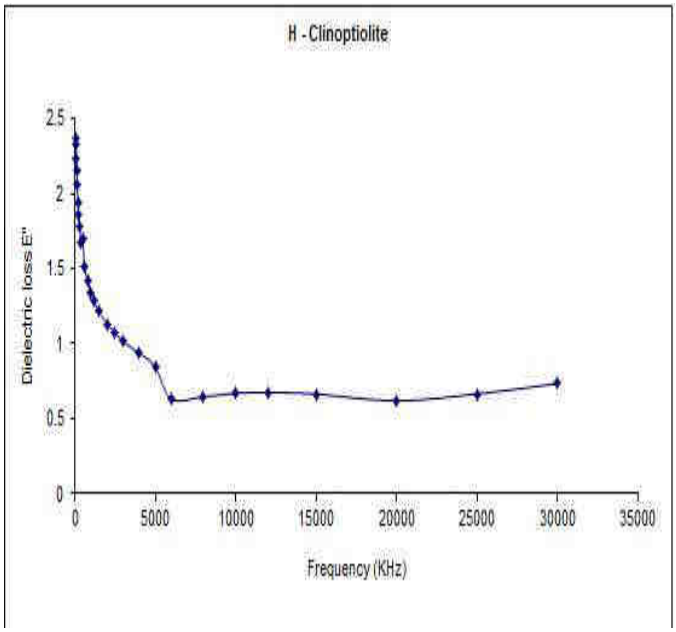

Fig. 4 Variation of dielectric loss as a frequency in H clinoptilolite

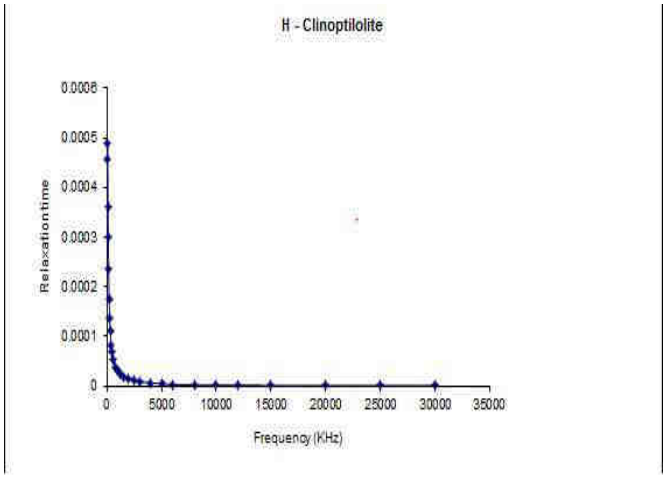

Fig.5 Variation of relaxation time as a frequency in H clinoptilolite

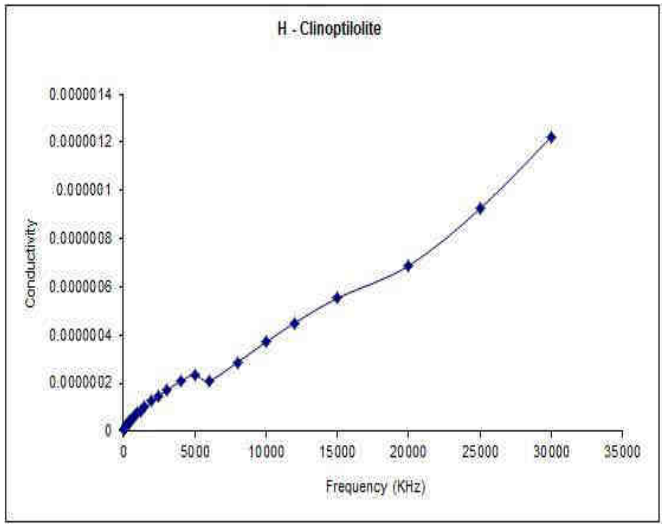

Fig.6 Variation of conductivity as a frequency in H clinoptililolite

CONCLUSION

FromXRD Pattern and IR bands confirm the thermal stability of H-Clinoptilolite there is no structural change in zeolite by ion exchange & H – form of zeolite except cation exchange.Dielectric study of H-Clinoptilolite plays an important role in stating the nature of zeolite.

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