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Dielectric Study Of NH4-Heulandite

Dr.V.P.Deshpande
Shivaji College Kannad Dist.Aurangabad
Corresponding Author: Dr.V.P.Deshpande

Abstract: Natural Zeolite Heulandite belongs to Group VII were collected near Ellora Ajanta belt. characterization was made using XRD,IR at NCL Pune. Dielectric study of NH4-Helundite was studied using LCR Bridge.

Keywords: NH4- Heulandite,Characterization,Dielectric study

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I. Introduction

Heulandite zeolites are among the most abundant useful zeolites found in the nature with Si/Al ratio ranging from 3 to 5. the general formula for Heulandite is [(Na,K) $Ca_4(Al_9\ Si_{27}\ O_{72})$ 24 H_2O]. Heulandite is determined as the mineral with a ratio Si/Al < 4.They are monoclinic zeolite minerals C2/m group with the following unit cell parameters a

a=17.73 A° , b=17.92 A° , c=7.43 A° , β=116.24 ° (1)

Their structure is characterized by the large intersecting open channels of 10 and 8 member tetrahedral rings. In natural zeolites these channels are predominantly occupied by Na , K, Ca and H_2O (2). The kind and the population of channel cations influence the stability of the cavities of a zeolite and play role, of the crucial importance, in it's thermal behavior (3). The cation positions are found in the channels of a hydrated natural, " Heulandite", group zeolites (4). The key difference between Heulandite and Clinoptilolite is thermally stable to temperature in excess of 450°C, while Heulandite undergoes structural collapse below 450°C.

It is well known that Heulandite and Clinoptilolite react differently up on heating, depending on their chemical composition (5)

2 Sample Preparations

Heulandite were collected from the quaries of Ajanta – Elora caves, Marathwada. Sample crushed and sieved to get 106 μm sized crystals. For ion exchange sample is treated with 1 M solution of Ammonium Nitrate with stirring at 95°C for six hours. NH_4 ion exchanged form of Heulandite is heated at 250°C for 48 hours for getting H- Heulandite .

3 Characterization

X-ray diffraction: For the characterization of Heulandite, X – Ray diffractograms were recorded between 2θ values from 5° to 50° on Phillips (PW 1710) having wavelength 1.54056 A°. Different three forms of Heulandite are recorded & result of d values are reported in table 1

$$Na_{1.54}\ K_{0..95}\ Ca_3\ [\ Si_{26.66}\ A_{l8.49}\ O_{72}\ ]\ 29\ H_2O$$

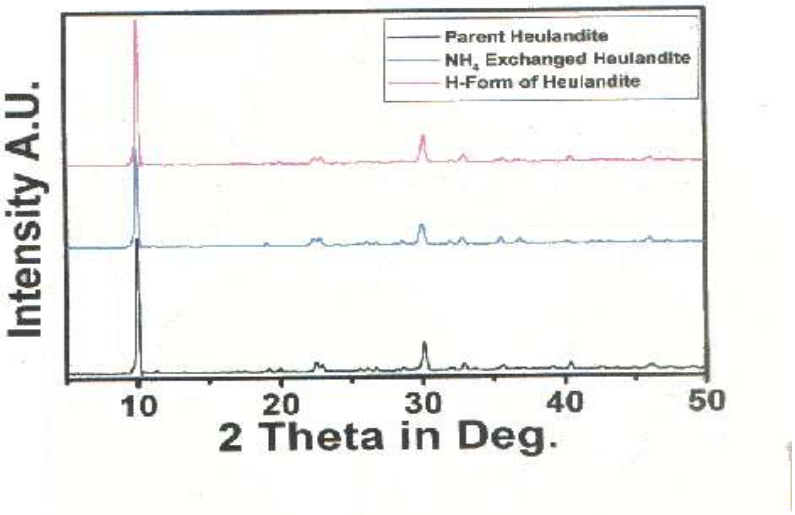

Fig. 1 *XRD of NH4-Heulandite*

2 Theta	d- Value	Peak Width	Intensity
9.895	8.9315	0.08	100
11.11	7.9573	0.24	2
13.12	6.7424	0.64	0.7
15.83	5.5938	0.2	1.6
16.895	5.2435	0.12	2.4
19.08	4.6476	0.16	4.5
22.2	4.001	0.28	12
22.715	3.9114	0.12	11.7
25.02	3.5561	0.12	1.3
25.92	3.4346	0.16	2.3
26.325	3.3827	0.12	3.6
26.82	3.3214	0.24	1
28.21	3.1608	0.12	3.8
30.02	2.9742	0.16	12
31.885	2.8044	0.32	2.6
32.685	2.7375	0.28	4.9
35.35	2.537	0.24	1.8
36.775	2.4419	0.64	1.8
39.775	2.2655	0.48	0.3
44.74	2.0239	0.32	0.3
45.715	1.983	0.24	1
47.11	1.9275	0.32	0.6
48.66	1.8697	0.24	0.8
49.21	1.85	0.06	0.3

Table 1*- XRD Data for NH4-Heulandite (After Background Subtraction)*

IR- From IR studies external linkage, Asymmetric stretch is observed at 1200 cm^{-1} and symmetric stretch is at 795 cm^{-1}. In water bands region hydroxyl stretch is observed at 3740 cm^{-1} & water bands assigned at 1655 cm^{-1} for Internal tetrahedral (Structure sensitive) mode Asymmetric stretch is at 1095 cm^{-1} & symmetric stretch is 750 cm^{-1} from the IR bands of parent form, NH_4 – form & H-form of Heulandite it is observed that there is no major change in bands expect water bands. This confirms the stability of Heulandite zeolite.

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
Heulandite	1200	795	599	1095	750	490	3740	1655

Table 2 IR assignments in Cm^{-1}

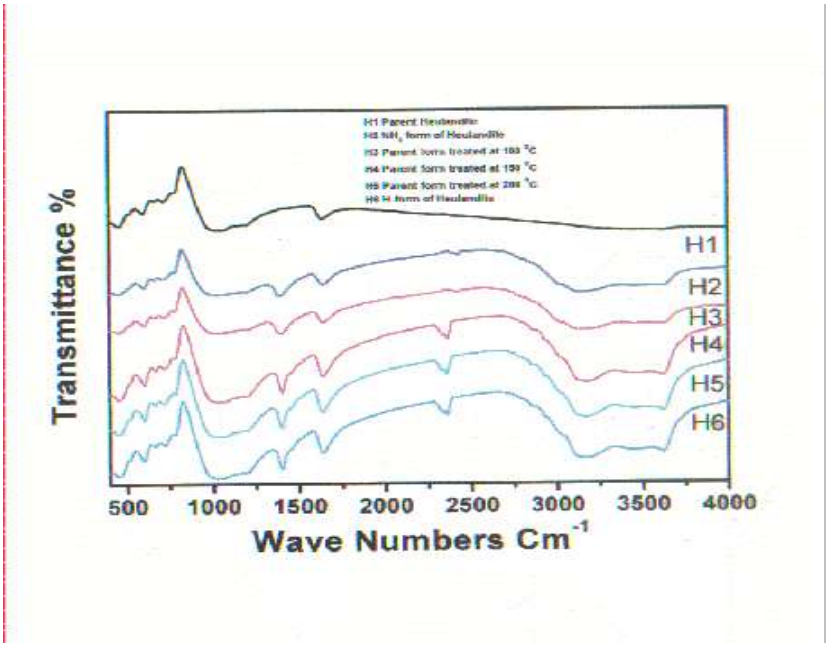

Fig. 2 IR OF NH4- Heulandite from 400-4000

II. Results And Discussion

XRD:- XRD pattern of the parent Heulandite NH_4 - exchanged Heulandite and H- form Heulandite is shown in fig 1 From diffractogram we conclude the crystalline nature of Heulandite d –values are compared with standard 'd' values. This confirms the Heulandite structure. From the three diffractograms we come to the point that there is no major change in these three forms of Heulandite samples. This confirms the stability of Heulandite zeolite.

IR- From IR studies external linkage, Asymmetric stretch is observed at 1200 cm^{-1} and symmetric stretch is at 795 cm^{-1}. In water bands region hydroxyl stretch is observed at 3740 cm^{-1} & water bands assigned at 1655 cm^{-1} for Internal tetrahedral (Structure sensitive) mode Asymmetric stretch is at 1095 cm^{-1} & symmetric stretch is 750 cm^{-1} from the IR bands of parent form, NH_4 – form & H-form of Heulandite it is observed that there is no major change in bands expect water bands. This confirms the stability of Heulandite zeolite.

6 Dielectric Study of NH4 – Heulandite

Dielectric Constant (Є') :- In the fig 3 shows that dielectric constant of NH4 – ion exchange Heulandite goes on decreasing up to 6000 KHz after that it goes on increasing slowly or remain constant.

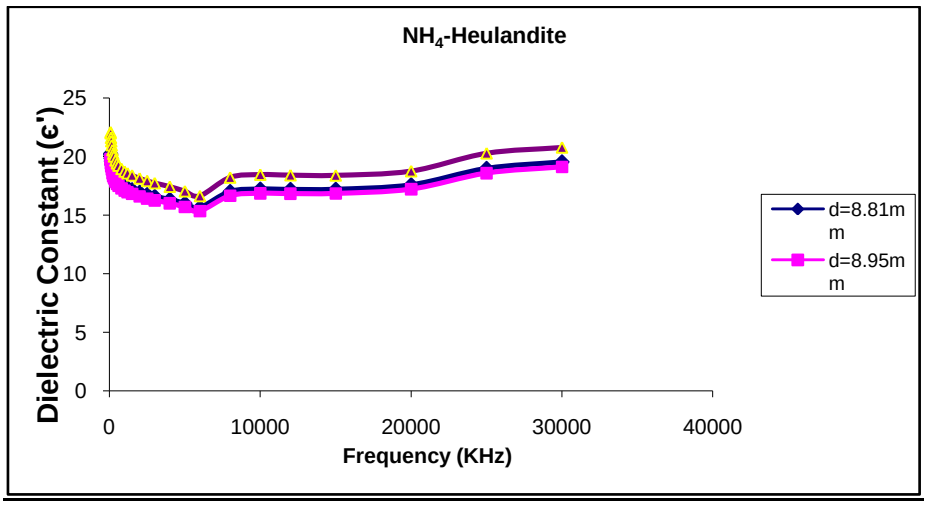

Fig. 3 variation of dielectric constant as frequency in NH_4 Heulandite

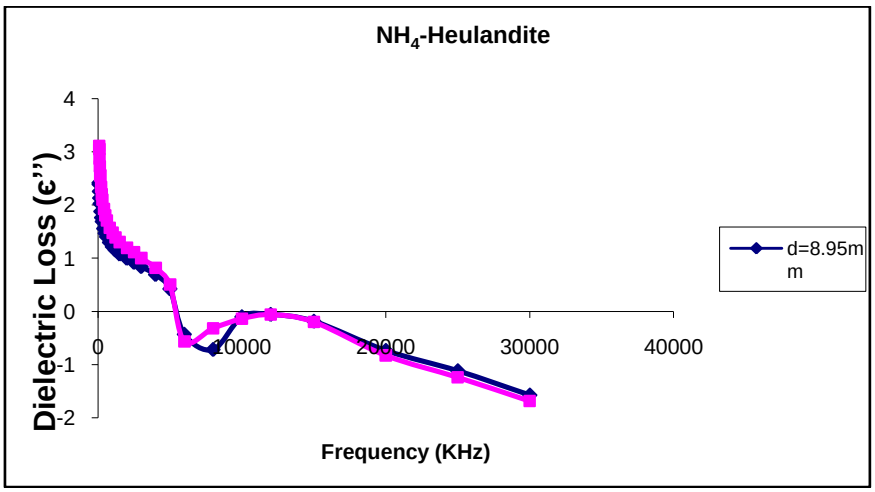

Fig. 4 variation of dielectric loss as frequency in NH_4 Heulandite

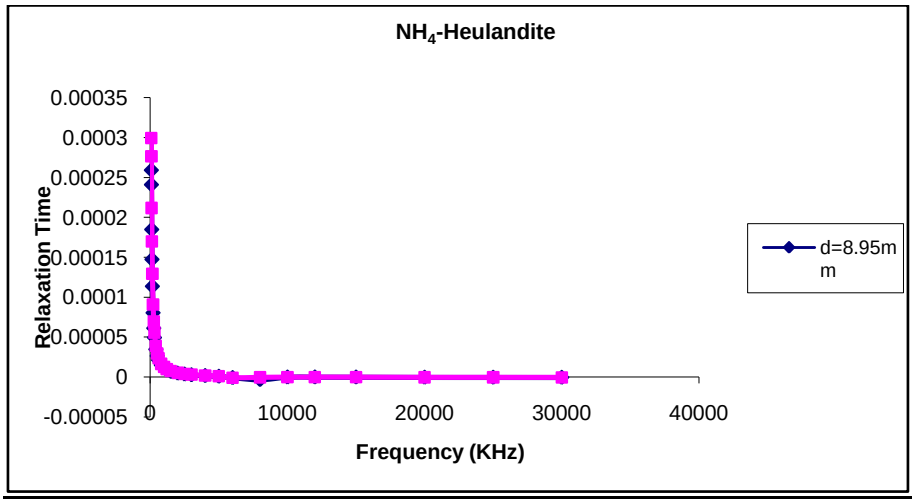

Fig. 5 variation of relaxation time as frequency in NH_4 Heulandite

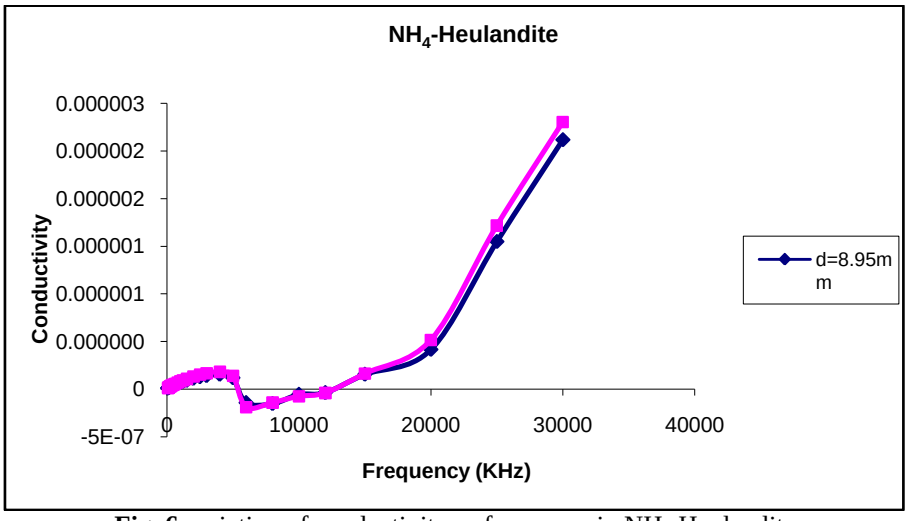

Fig. 6 variation of conductivity as frequency in NH_4 Heulandite

Dielectric Loss (Є''):- Fig 4 Indicates the variation Є'' against the frequency. There is decrease in Є'' by increasing frequency. Decrease up to 5000 KHz is slow then Є' increases again. From 12000 KHz Є' decreases.

Relaxation Time (τ): – Fig 5 shows the frequency verses Relaxation Time. As frequency increases the value of τ decreases.

A.C. conductivity (б) :- A.C. conductivity goes on increasing as frequency increases. In fig 6 б- increases up to 5000 KHz. Then decreases and again increase from 6000 KHz.

III. Conclusions

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

Acknowledgements

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