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# **Dielectric Study of H-Heulandite**

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**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 H-Helundite was studied using LCR Bridge.

**KEYWORDS**: H- 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

 $a=17.73 \text{ A}^{\circ}$ ,  $b=17.92 \text{ A}^{\circ}$ ,  $c=7.43 \text{ A}^{\circ}$ ,  $\beta=116.24 ^{\circ}$  (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)

### II. SAMPLE PREPARATIONS

Heulandite were collected from the quaries of Ajanta – Elora caves, Marathwada. Sample crushed and sieved to get 106  $\mu$ m sized crystals. For ion exchange sample is treated with 1 M solution of Ammonium Nitrate with stirring at 95°C for six hours. NH<sub>4</sub> ion exchanged form of Heulandite is heated at 250°C for 48 hours for getting H- Heulandite .

# III. CHARACTERIZATION

**X-ray diffraction**: For the characterization of Heulandite, X – Ray diffractograms were recorded between  $2\theta$  values from  $5^{\circ}$  to  $50^{\circ}$  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

 $Na_{1.54}~K_{0..95}~Ca_3$  [  $Si_{26.66}~A_{18.49}~O_{72}$  ] 29  $H_2O$ 

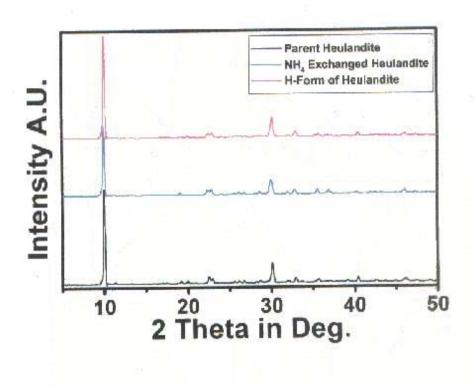


Fig. 1 XRD of NH4-Heulandite

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  $1200 \text{ cm}^{-1}$  and symmetric stretch is at  $795 \text{ cm}^{-1}$ . In water bands region hydroxyl stretch is observed at  $3740 \text{ cm}^{-1}$  & water bands assigned at  $1655 \text{ cm}^{-1}$  for Internal tetrahedral (Structure sensitive) mode Asymmetric stretch is at  $1095 \text{ cm}^{-1}$  & symmetric stretch is  $750 \text{ cm}^{-1}$  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 <sup>-1</sup> Str. sensitive		Double	Internal Tetrahedral Str Insensitive cm <sup>-1</sup>		T - 0	Water Ban	ds
	Asymmetric	Symmetric	ring	Asymmetric	Symmetric	Bend	OH-	$H_{2}O$
	Stretch	stretch		Stretch	stretch		stretch	Bands
Heulandite	1200	795	599	1095	750	490	3740	1655

**Table 2** IR assignments in Cm<sup>-1</sup>

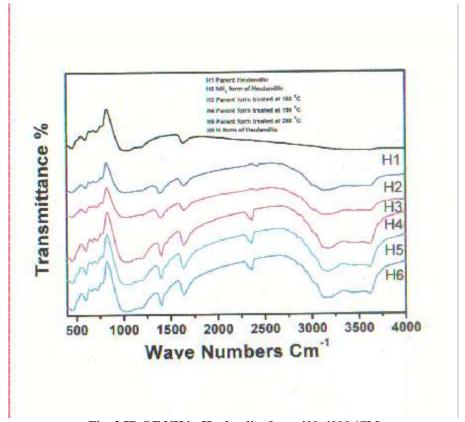


Fig. 2 IR OF NH4- Heulandite from 400-4000 /CM

# IV. 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<sup>-1</sup> and symmetric stretch is at 795 cm<sup>-1</sup>. In water bands region hydroxyl stretch is observed at 3740 cm<sup>-1</sup> & water bands assigned at 1655 cm<sup>-1</sup> for Internal tetrahedral (Structure sensitive) mode Asymmetric stretch is at 1095 cm<sup>-1</sup> & symmetric stretch is 750 cm<sup>-1</sup> from the IR bands of parent form, NH<sub>4</sub> – 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.

### Dielectric Study of H - Heulandite

**Dielectric Constant (C'):**-Fig.3 shows the variation of C' against frequency in H- form of Heulandite. Dielectric constant decreases initially upto 6000 KHz. Then remain nearly constant.

**Dielectric Loss (\mathcal{C}''):-** Fig 4 shows the frequency verses dielectric loss . As frequency increases  $\mathcal{C}$ '' decreases up to 6000 KHz. Then  $\mathcal{C}$ '' remains constant.

**Relaxation Time (T)**:- Fig 5 shows the variation of Relaxation time against frequency. This shows that T-decreases as frequency increases.

**A.C. conductivity (6)**: Fig 6 shows the increase in A.C. conductivity with increase in frequency in H – Heulandite.

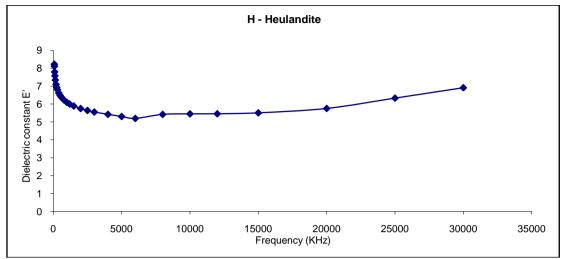


Fig. 3 variation of dielectric constant as frequency in H Heulandite

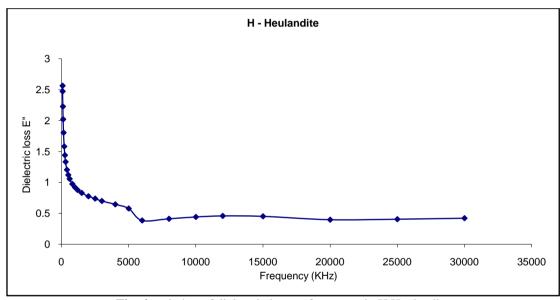


Fig. 4 variation of dielectric loss as frequency in H Heulandite

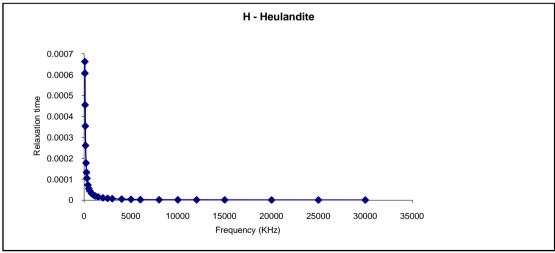


Fig. 5 variation of relaxation time as frequency in H Heulandite

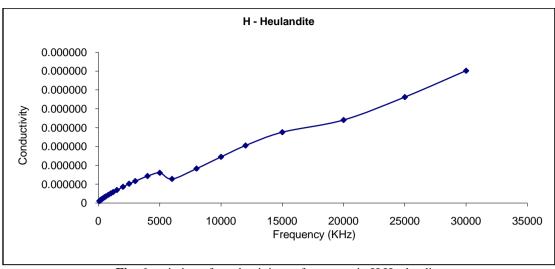


Fig. 6 variation of conductivity as frequency in H Heulandite

### V. CONCLUSIONS

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

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