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Research Article

Hydrothermal Synthesis of Mordenite Type Zeolite

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Abstract:

Zeolite is a crystalline alumino-silicate microporous material, which has been widely used as ion-exchangers, adsorbent and catalyst. Recently, several researches highlights the progress of zeolite-based catalysts in the CO_2 conversion to valuable products. In the present work, hydrothermal synthesis of mordenite zeolite crystals, with composition of the chemical products Al_2O_3, 12.5 SiO_2,2.4 NaOH, 110 H_2O at 160°C for 96 hours (4 days) at Ph 11 is described. Results of characterization of the mordenite zeolite by XRD, FTIR, EDAX, SEM, TGA, are presented and discussed. In the present work, hydrothermal synthesis and characterization of mordenite zeolite crystals is described.

1. Introduction

Zeolites are used as absorbents and desiccants in gas purification processes, and separation processes such as para xylene from its isomers [1,2]. They are also used as potential catalysts in petroleum production, petrochemicals and refining. Catalytic cracking on Y and ZSM-5 zeolites is by far the largest application followed by catalytic hydrocracking on mordenite zeolites [3-5]. Tons of zeolites are currently on the market as part of detergent formulations. Zeolite A is used exclusively as a phosphate substitute. Other natural zeolites are used in a number of applications such as wastewater treatment, nuclear effluents and CO_2 conversion [6-8]. The main user countries of zeolites worldwide are the USA, Japan and Western European countries
Nowadays, zeolites have an economic impact in some areas, notably in the field of ion exchange reactions, which make zeolites economically attractive as small quantities of zeolites are sufficient to treat large volumes of water. Zeolites have great potential for ecosystem protection, ranging from wastewater and gas treatment to the replacement of undesirable and environmentally damaging polyphosphates [9-14]. The industrial exploitation of zeolites often requires their preparation. They are obtained by hydrothermal crystallisation of

aluminisilicates and active alkali metals at moderate temperatures and pressures.
In the present work, hydrothermal synthesis and characterization of mordenite zeolite crystals is described.

2. Material and Methods

Mordenite zeolite was prepared by hydrothermal crystallisation at 160°C for 96 hours (4 days) at Ph 11, from starting gels of molar composition Al_2O_3, 12.5 SiO_2,2.4 NaOH, 110 H_2O. After the crystallization process the obtained powder was washed with distillate water and dried at 120°C.
Chemical components of products are SiO_2 :41.6%. Al_2O_3 :77%. The obtained powder was analyzed by powder X ray diffractometer (Philips PW 1800, using Cu Ke radiation), infrared spectroscopy (Philips PU 9800), scanning electron microscope (Philips XL 30) and differential thermal analyzer (M2 BDL SETARAM).

3. Results and Discussions

3.1 XRD analysis

The XRD patterns of as-elaborated samples are given in figure 1. They are in good agreement with jcpds card 0110155. We observe in table 1 the appearance of all peaks related to the presence of mordenite zeolite, confirming the high crystallinity.
According to these results, we attest the absence of other phases than mordenite. When we compare between the results found by the XRD and those presented in the jcpds 0110155 card, also shown in Table 1. We certify that there is a very good similarity between the measured parameters and those present in this card.

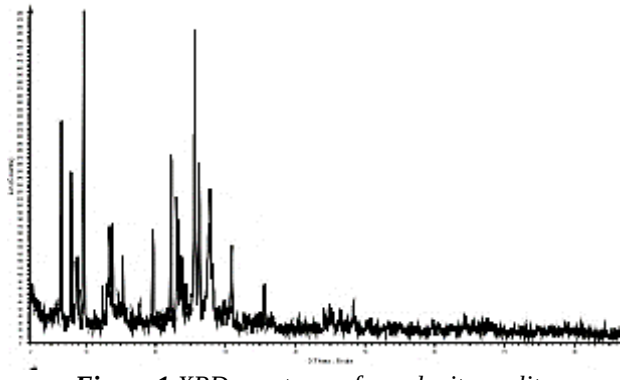


Figure.1 *XRD spectrum of mordenite zeolite*

The table 1 represents the parameters of XRD peaks of mordenite zeolite and its reference parameters.

Table.1 *Parameters of XRD peaks of mordenite zeolite and reference parameters.*

According to jcpd card0110155			**Mordenite powder**	
hkl	d(A°)	I(%)	d(A°)	I(%)
110	13.581	100	13.569	88
020	10.265	13.1	10.197	34
200	9.055	56.4	9.089	100
220	6.791	0.3	6.764	1
111	6.584	40.4	6.556	49
130	6.402	29.1	6.367	9
021	6.071	13..3	6.035	5
310	5.791	9.5	5.808	9
040	5.133	0.6	5.098	2
221	5.042	2.3	5.027	2
131	4.877	4.4	4.850	1
311	4.59	3.2	4.589	5
330	4.527	22.7	4.523	16
240	4.465	0.6	4.446	1
041	4.241	1.4	4.214	1
420	4.142	5.7	4.150	1
150	4.004	46.1	3.979	21
331	3.880	1.0	3.871	1
241	3.840	16.9	3.823	9
002	3.764	6.9	3.744	4
421	3.629	4.5	3.630	1
112	3.627	0.3	3.609	1
510	3.567	2.3	3.579	1
151	3.535	0.3	3.514	2
022	3.534	4.3	3.514	2

3.2 FTIR analysis

The investigation by infrared spectroscopy technique in the mid-infrared region of the zeolite spectrum is useful in this regard since it contains the fundamental vibrations of the framework Al, Si-4 or (T 4) tetrahedral. A different vibration bands are observed in the FTIR absorption spectrum of mordenite zeolite shown in figure 2. The first class of vibrations found at 1223 -1230, 680 -700 and 438-454 cm-' are assigned to the internal tetrahedral. The second group of frequencies observed at 547-546, 446,792-789 and 1084-1102 cm-' are assigned to the linkages between tetrahedral and the topology of the units of structure of samples. The main bands of absorption obtained from Figure 2, are shown in Table 2.

3.3 SEM characterization

The morphology of the crystalline phases was demonstrated by observation with a scanning electron microscope.

Table .2 *The main bands of IR absorption and associated bond vibration of mordenite zeolite*

	Link type	Frequency cm^{-1} D.W.Breck	Mordenite
Internal vibrations (tétraèdres TO_4)	Asymmetric elongation Si-O-Al. Si-O-Si.	1250-950	1230
	Symmetric elongation Al-O. stretching	720-650	700
	Bending T-O link.	500-420	450
External vibrations	Double cycle $2C_4$ et $2C_6$ Si, Al-O.	650-500	550
	Pores openning	420-300	400
	Symmetric elongation Si-O-Al, Si-O-Si.	820-750	800
	Asymmetric elongation Si-O-Al.	1150-1050	1010

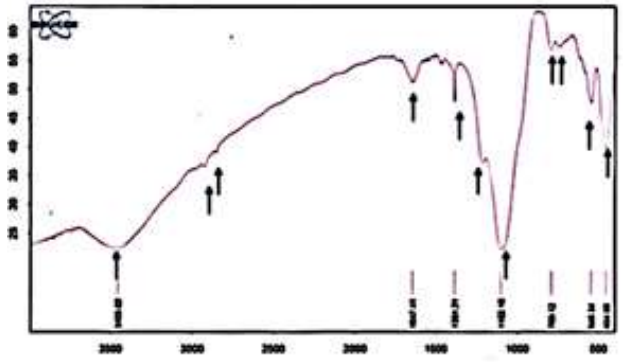


Figure.2 *FTIR absorption spectrum of mordenite zeolite*

Figures 3 illustrates the micrographs of these samples. We note that the morphology of the crystals is generally hexagonal prismatic, regular and that the dimensions of mordenite samples are of 10-15 µm respectively. According to the EDS characterization, the chemical composition of the as prepared powder is presented in figure 4. and table 3.

3.4 DTA analysis

According to the DTA analysis shown in figure 5, we observe a succession of endotherms ranging from

Table.3 *Chemical composition of mordenite zeolite*

Element	Weight %	Atomic%
O	50.29	59.79
Na	4.18	3.46
Al	4.58	3.23
Si	34.57	23.42

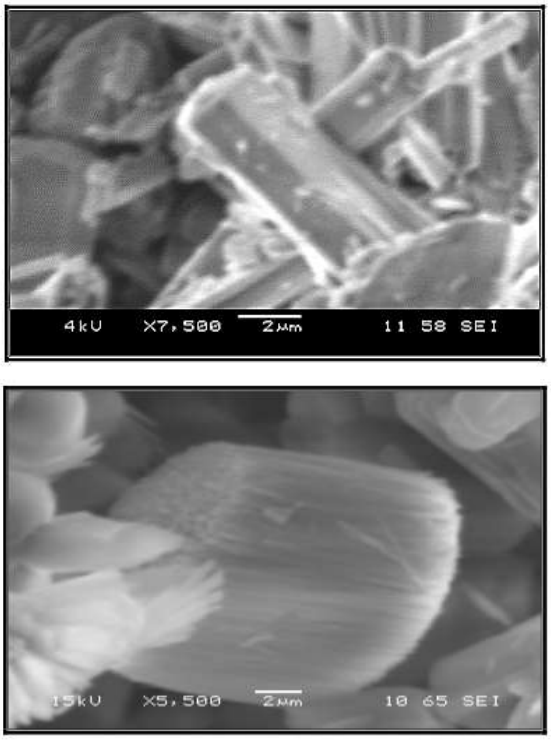
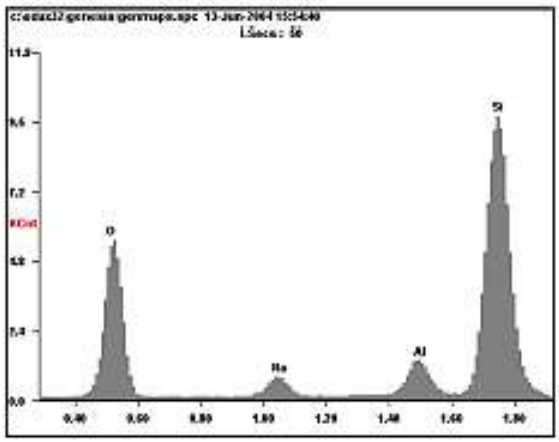


Figure .3 *Scanning electron micrographs of mordenite zeolite*



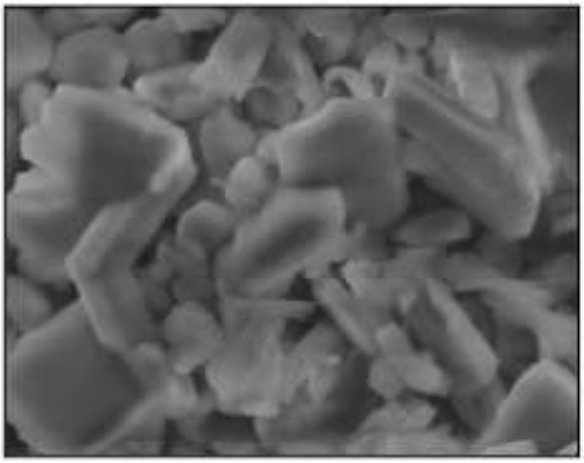


Figure.4 *EDS analysis of mordenite zeolite*

50 to 250 °C. The differential thermal analysis of the sample allowed us to determine that the departure of the physisorbed water takes place from 100 to about 250 where the dehydration is complete. We noticed the vitreous state of the mordenite sample after characterization which explains the vitreous transformation of this material at a temperature exceeding transformation of this material at a temperature exceeding 1200°C.

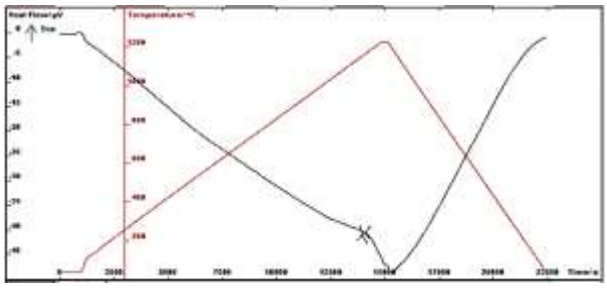


***Figure**.5 DTA curve of mordenite zeolite*

4. Conclusions

In the present work, the pure Mordenite zeolite types are obtained for contact times of 96 hours, at temperatures of 170 C and under autogenous pressure. The skeletal structure of the Mordenite material is very thermally stable. The initial structural change starts to take place from 1100°C onwards and above 1250°C the Mordenite structure vitrifies. The differential thermal diagram proves the stability of this structure.

The infrared spectroscopy technique allowed the characterisation of the absorption bands attributed to the different Si-O-Si, Si-O-Al, Si-O and Al-O bonds linked to the tetrahedra forming the structures of the Mordenite phases. Sites belonging to hydroxyl groups -OH were also revealed by this technique.

With their dual capacity for ion exchange and adsorption, these materials can be used in the very long term in the decontamination of any very dilute waste water.

Author Statements:

- **Ethical approval:** The conducted research is not related to either human or animal use.
- **Conflict of interest:** The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper
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