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Research Article

Preparation and Characterization of ZSM-5 Zeolite

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Abstract:

Recently, zeolites attracted considerable research attention because they exhibit adsorption properties, ion exchange capabilities and a good catalytic property, a specific pore size distribution; a unique chemical and thermal properties and several applications. Actually zeolites play a crucial role as a catalyst to capture and convert Carbone dioxide into valuable products. In the present work, hydrothermal synthesis of ZSM-5 zeolite crystals, with composition of the chemical products 0.01 Al_2O_3,1SiO_2,2 NaOH, 0.15 TPABr, 18.34 H_2O at 170°C for 48 hours (2 days) at Ph 11 is described. Results of characterization of the ZSM-5 zeolite by XRD, FTIR, EDAX, SEM, TGA, are presented and discussed.

1. Introduction

Nowadays, microporous solid materials occupy a privileged place in the industrial field. Zeolites are microporous crystalline aluminosilicates that have a similar composition to the clay minerals but with their well-defined three-dimensional microporous structures. Zeolites were among the first solids to be widely used in materials science [1-3]. Their applications, which are too varied, particularly in the field of catalysis and the environment, are increasing in recent times [4-7]. Compared to other materials, zeolites have very interesting physical and chemical properties. They have a microporous character with uniform pore size, a shape selectivity which allows certain hydrocarbon molecules with a crack diameter less than or equal to that of the crystal to diffuse into the pores of the crystal, ion exchange properties. They have the possibility of developing an internal acidity which allows zeolites to be interesting materials for catalysis [8] and a high thermal stability [9]. Since November ,1972, when the synthesis of a new type of zeolite, denoted by ZSM-5 (Zeolite Socony Mobil Number 5), was filed, several research papers as well as patents related to its synthesis have been issued [10-15]. This zeolite can be distinguished by its unique properties. It has

significant applications such as a catalysis, gas separation and purification industry.
In the present work, hydrothermal synthesis and characterization of ZSM-5 zeolite crystals is described.

2. Material and Methods

The production of ZSM-5 generally follows three steps that are divided into the formation of a primary gel, the subsequent hydrothermal conversion to a secondary gel with the final crystallization step.
ZSM-5 zeolite was prepared by hydrothermal crystallization at 170°C for 48 hours (2 days) at Ph 11, from starting gels of molar composition 0.01 Al_2O_3,1SiO_2,2 NaOH, 0.15 TPABr, 18.34 H_2O, as it is shown below:

H_2O

↓

1 ← NaO

↓

2 ← Al_2O_3 (with stirring)

↓

3 ← SiO_2 et TPABr (Without stirring)

↓

4 ← Autoclave (T=170°C, 48h)

↓

5 ← H_2O Washing

After the crystallization process the obtained powder was washed with distillate water and dried at 120°C.
Chemical products used: 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 card048-0134. In Table I, the appearance of all peaks related to the presence of ZSM-5 was observed, confirming the high crystallinity.
According to these results, the absence of other phases than ZSM-5 were confirmed.
The comparison between the results found by the XRD analysis and those presented in the jcpds 048-0134 card, as shown in Table I, certify that there is a very good similarity between the measured parameters and those present in this card. The table .1 represents the parameters of XRD peaks of ZSM-5 zeolite and its reference parameters.

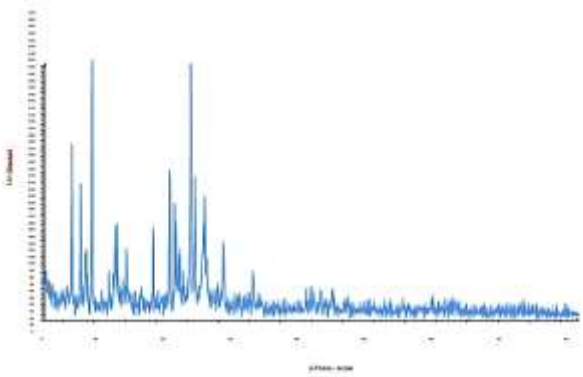

Figure.1 *XRD spectrum of ZSM-5 zeolite*

Table.1 *Parameters of XRD peaks of ZSM-5 zeolite and reference parameters.*

According to jcpds 048-0134			**ZSM-5 powder**	
hkl	d(A°)	I(%)	d(A°)	I(%)
101	11.163	96	11.158	100
200	9.997	62	10.067	31
002	6.682	13	6.707	4
102	6.348	18	6.065	1
301	5.981	20	6.003	9
131	5.701	13	5.713	7
202	5.574	16	5.578	8
122	5.372	7	5.368	2
400	4.996	13	5.034	3
103	4.353	18	4.364	4
421	4.250	22	4.261	5
203	4.095	14	4.087	1
501	3.858	100	3.857	43
133	3.655	29	3.649	14
432	3.437	14	3.444	5
104	3.312	16	3.308	4
080	2.488	6	2.491	1
1000	2.014	11	2.013	6
0100	1.991	11	1.994	4

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 ZSM-5 zeolite shown in figure.2. The main bands of absorption obtained from Figure 2, are shown in Table 2.

Table 2. *The main bands of IR absorption and associated bond vibration of ZSM-5 zeolite.*

	Link type	Frequency cm^{1} D.W.Breck	ZSM-5
Internal vibrations (tétraédres TO_4)	Asymmetric elongation Si-O-Al. Si-O-Si.	1250-950	1223
	Symmetric elongation Al-O. stretching	720-650	680
	Bending T-O link.	500-420	438
External vibrations	Double cycle $2C_4$ et $2C_6$ Si, Al-O.	650-500	547
	Pores openning	420-300	446
	Symmetric elongation Si-O-Al. Si-O-Si.	820-750	792
	Asymmetric elongation Si-O-Al.	1150-1050	1084

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.

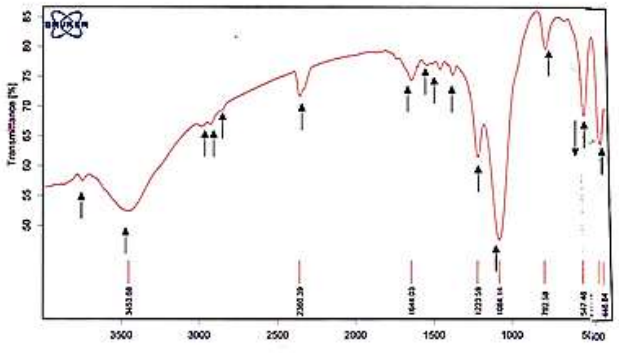

Figure.2 *FTIR absorption spectrum of ZSM-5 zeolite*

3.3 SEM characterization

The morphology of the crystalline phases was demonstrated by observation with a scanning electron microscope. The figure 3 illustrates the micrographs of these samples.

The morphology of the crystals is generally regular and that the dimensions of the ZSM-5 samples are of the order of 5-10 µm.

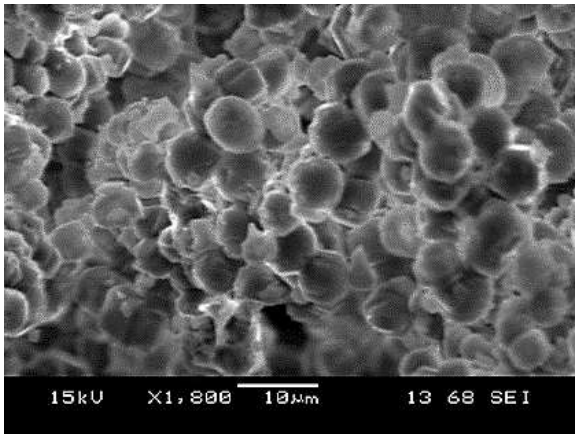

Figure .3 *Scanning electron micrographs of ZSM-5 zeolite*

According to the EDS characterization, the chemical composition of the as prepared powder is presented in figure. 4 and table.3

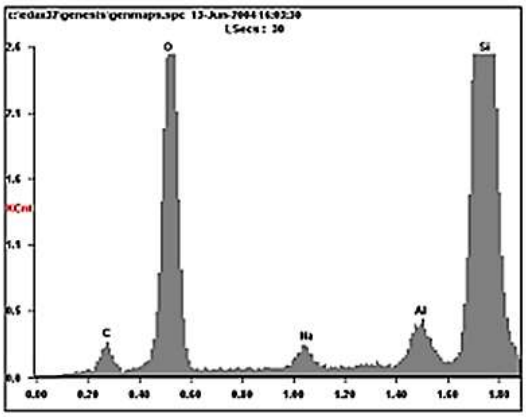

Figure.4 *EDS analysis of ZSM-5 zeolite*

Table.3 *Chemical composition of ZSM-5 zeolite*

Element	Weight %	Atomic%
O	49.57	53.88
Na	1.63	1.24
Al	1.77	1.14
Si	29.37	18.18
C	17.66	25.56

3.4 DTA analysis

Differential thermal analysis (DTA) of the sample revealed the organic structurant agent occluded in its basic structure. a succession of endotherms ranging were observed from 50 to 200 °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 200 where the dehydration is complete.

The two most important transformations observed in the ATD curve of ZSM-5 zeolite presented in figure .5 can be interpreted as follows:

An endothermic peak at a temperature of 300 reflects the elimination of the organic structuring agent ((C3H7)4NBr) associated with the OH- ions and its subsequent removal by thermal decomposition.

A second exothermic peak at a temperature of 790 which probably translates into the beginning of the decomposition of the zeolite structure and finally a final peak at 1200 which necessarily translates into the total decomposition of the latter

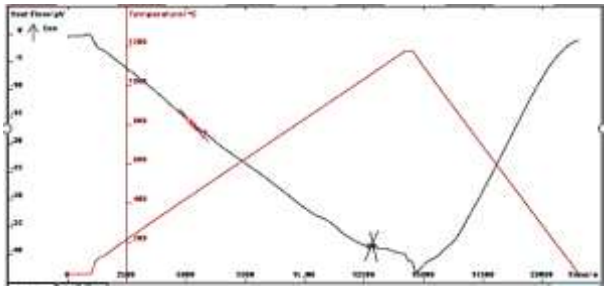

***Figure**.5 DTA curve of ZSM-5 zeolite*

4. Conclusions

In the present work, pure ZSM-5 zeolite type is obtained for contact times of 48 hours, at temperatures of 160°C and under autogenous pressure.

The skeletal structure of the ZSM-5 material is thermally stable. The initial structural change starts to take place from 1026°C and above 1200°C the structure decomposes. The differential thermal diagram proves the stability of this structure.

The infrared spectroscopy technique allowed the characterization 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 ZSM-5 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

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- **Ethical approval:** The conducted research is not related to either human or animal use.
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