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Influence of the crystallization temperatureon the synthesis of Y zeolite

[1]Khalid Yamni, [2]NajibTijani,[3]Said Lamsiah
[1,2,3]*Département de chimie, Equipe Matériaux, Membranes et Procédés de SéparationFaculté des Sciences, Université Moulay Ismail, Meknès, Maroc*

Abstract:*In this paper, the study of the behavior temperature of the Y zeolite is discussed. In first we have synthetized the product from the two precursors of aluminate and silicate. The characterization was carried out by using X-ray diffraction powder. In the last step of the synthesis, we have chosen the crystallization temperature. The obtention of the Y zeolite is mostly affected by this parameter. In order to follow the effect of this, 3 values are chosen for this synthesis. The 3 powders were characterized by XRD. The obtained spectrawere decomposed and indexed to obtain the Laue group space. The research of the best space group is undertaken in CheckCell program.*
The analysis of the different solution with different CFOM allowed the choice of the true solution. To validate the result, profile matching with Fullprof program is made. This transition let us think that the evolution from cubic to tetragonal systems can be explained by the relation group-subgroup.
Keywords*: Y zeolite, Crystallization Temperature, XRD, Fullprof, CheckCell*

I. Introduction

Zeolites are porous crystalline aluminosilicates. The zeolite materials were introduced by Cronstedt who discovered in 1756 [1]; they play an important role in many areas (petrochemical, pharmaceutical, adsorption etc.) [2,3,4]. In the present work we opted to synthesize a Y zeolite with a variable crystallization temperature program (60 ° C, 80 ° C and 100 ° C). The temperature is a very important factor, which affects a manner different zeolite synthesis[5].It can control (increase or decrease) the rate of crystallization, but also the type of zeolite formed. Generally high synthesis temperatures lead to the formation of dense phases. The Characterization by XRD showed that the zeolite Y (pure) corresponds to samples which have undergone a crystallization temperature in 60 ° C. when the crystallization temperature is closed to 100 ° C; we have seen the formation of another phase matching zeolite P.

II. Experimental

The synthesis [6] procedure is divided into two stages:

- Preparation of germination and the growth gel.
- Mixture of the precursors previously prepared.

The germination gel is prepared by the following method:in first NaOHand aluminate (NaAlO2 0.0065 mol) of sodium are dissolved in distilled water. After the solution of sodium silicate (Na2Si3O7 0.023 mol) are added to the aluminate precursor . The obtained mixture is aging for 24 h at room temperature under stirring.

The growth gel is prepared by the following manner:NaOHand of aluminate (NaAlO2 0.04 mol) of sodium are dissolved in distilled water. Thesolution of sodium silicate (0.147mol Na2Si3O7)areaddedvery slowly on the aluminate precursorunder vigorous stirring. The gel is made to mature for two hours after the end of addition.Then germination gel is added on the growth gel under vigorous stirring and the whole is placed in an oven at (120 ° C, 100 ° C, 80 ° C, 60 ° C) for 24 hours in a closed polyethylene container. After the zeolite is filtered and washed with distilled water on a Buchner funnel until the pH 10 and finally dried in an oven at 100 °Cfor 12H.

III. Characterization of the Y zeolites sample by X-ray diffraction

X-ray powder diffraction data were collected with a 6100 Shimadzu diffractometer using CuKα radiation and a monochromator. The measured 2 theta range (5–90°) was scanned in steps of 0.02° with a counting time of 3s/step. This 2 theta range was used for the refinement of cell parameter. The aperture and the soller slits were set at 1.0°.
The geometry is the θ -2θ. The sample is ground.

In order to follow the effect of the crystallization temperatureon thesynthesis of Y zeolite, we have elaborated three zeolites at different temperatures. All others parameters of the synthesis are the same.The first elaborated product are the Y zeolite at 80°C figure 1.

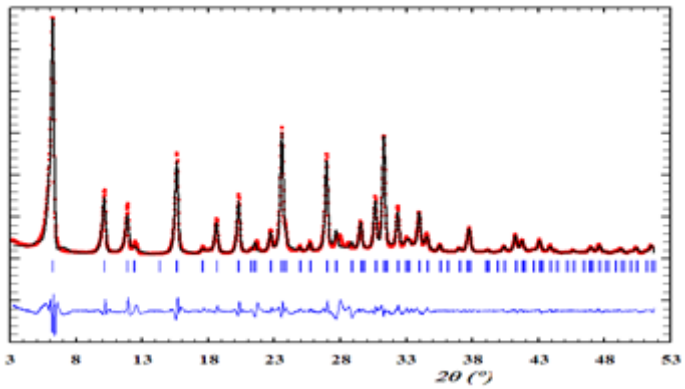

Figure 1: x ray spectrum of the Y zeolite at 80°C

The decomposition of the spectra and the indexation were made by DicVolprogram [7]. The table 1summarizes the result given by this software.

Table 1: parameters of cellby DicVol program

	a(Å)	b(Å)	c(Å)	Alpha	beta	gamma	Space group
Y zeolite 80	24.7974	24.7974	24.7974	90.000	90.000	90.000	P m-3m

The space group is the holoeder P m3m.Thesearchof the best space groupwas done by two programs ChekCell and CheckGroup [8]. The result is Fd-3m. To fit the spectrum, we use Fullprof program [9] with Pseudo Voigt as function of profile. We can note on the spectra in figure 2 the existence of some satellitespeaksnot fitted by this space group. That let usthink that process of crystallization is probably in evolution.

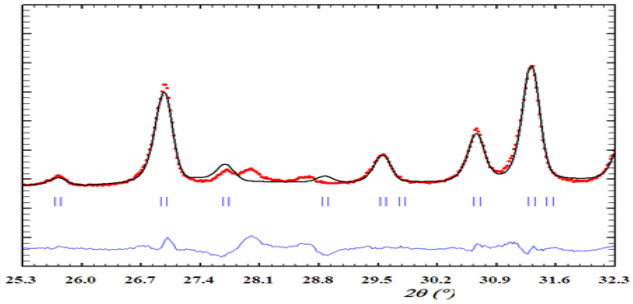

Figure 2: some satellites peaks not fitted by profile function

So we have decided to synthesizetwo Y zeolitesat lower and higher temperaturesthan 80°C.When we recorded anX ray spectrum of the Y zeolite at 60°C, we note the absence of the satellitespeaks. And the spectrum of this Y zeolite is pure. The figure 3 shows the obtained spectrum.

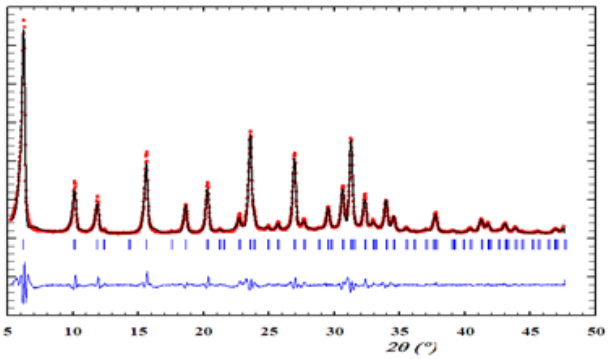

Figure 3: x ray pattern of the Y zeolite at 60°C

Also we present in the figure 4 the region that clearly shows the absence of the satellite peaks.

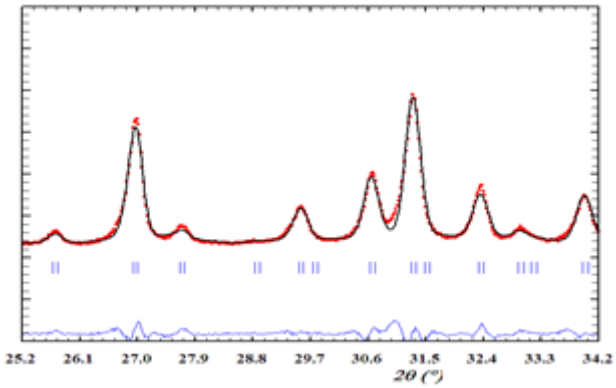

Figure 4: absence of satellites peaks in the Y zeolite elaborated at 60°C

The Y zeolite elaborated at 100°C shows a change of crystal system. It becomes a P zeolite and crystallizes in the tetragonal crystal system. The table 2 below gives the metric of the lattice.The profile matching applied on the data gives the refinement parameters below.

Table 2: parameters of lattice of the P zeolite elaborated at 100°C

	a(Å)	b(Å)	c(Å)	Alpha	beta	gamma
P zeolite	9.9958	9.9958	10.0184	90	90	120

With the CheckCell program, we find the space group I-4. The figure 5 shows the refinement obtained by profile matching.

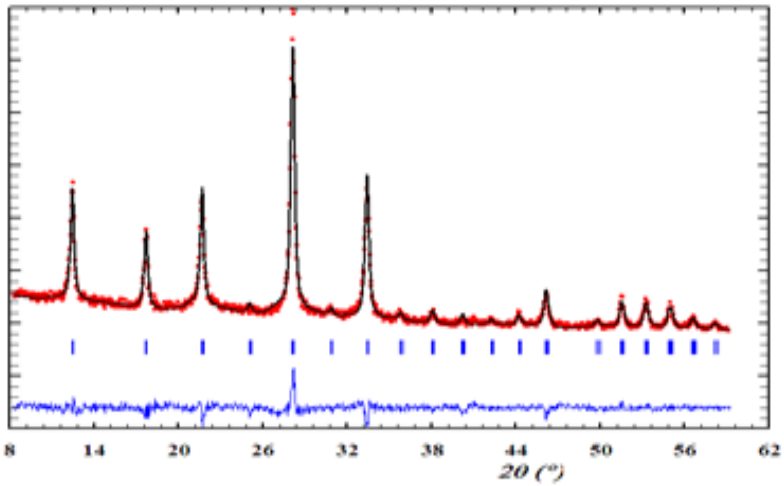

Figure 5: X ray spectrum of the Y zeolite elaborated at 100°C

IV. Conclusion

The synthesis of the Y zeolite is mostly affected by the crystallization temperature. In this work, the transformation is followed by X ray diffraction on the elaborated powders. The apparition of some peaks in the 2Θregion 25°-30° is a great sign for modification of the structure of the Y zeolite. The synthesis at lower and higher temperature than 80°Cgives the Yand the P zeolites respectively. The behavior of the Y zeolite versus crystallization temperature can be explained by the relation between group subgroup[10]. The figure 6 below resumes this relation:

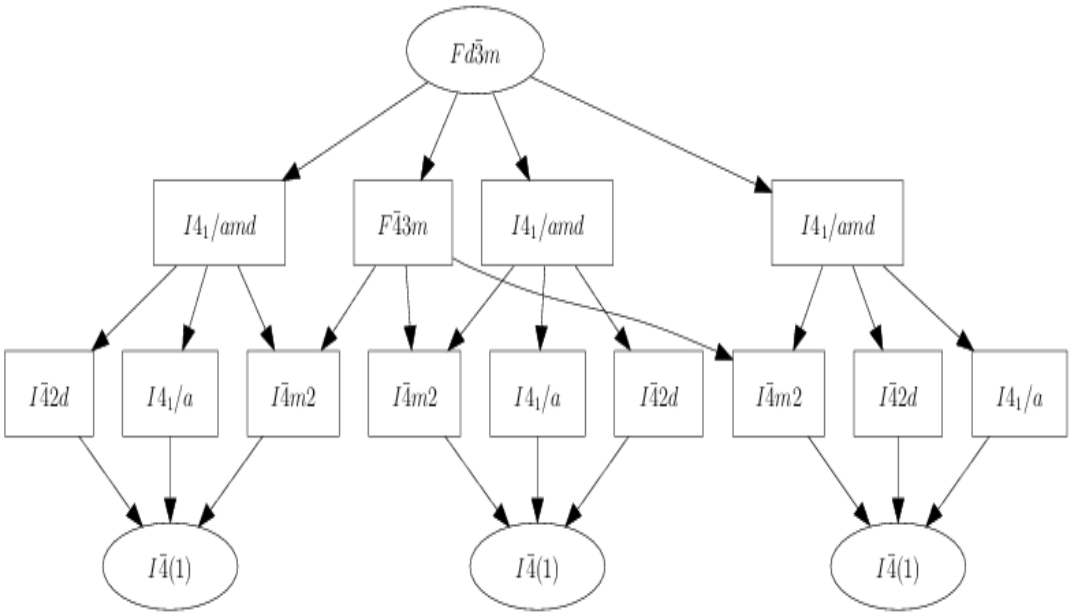

Figure 6: relation group subgroup between Fd-3m and I-4

The transformation between group subgroup and theexplanation ofthe splitting Wyckoffpositionsof this transition are understudy.

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