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(54) Title: CATALYTIC CRACKING OF ORGANIC COMPOUNDS USING ZEOLITE ITQ-33

(57) Abstract: The present invention relates to a process for the catalytic cracking of organic compounds characterized in that it comprises at least: a. introducing at least one first zeolitic material, ITQ-33, inside a reactor, b. feeding the reactor with at least one organic compound, c. allowing the zeolitic material ITQ-33 and the organic compound to remain in contact the time necessary for the reaction to occur.

CATALYTIC CRACKING OF ORGANIC COMPOUNDS USING ZEOLITE ITQ-33

TECHNICAL FIELD OF THE INVENTION

Catalytic cracking catalysts.

PRIOR ART

The general trend in catalytic processes, among them those belonging to the field of refining, is cost reduction due to an increase in activity of the catalysts used, and especially due to optimizing their selectivity ("*Marcilly, C., Journal of Catalysis 216, 47 (2003)*"). On the other hand, transformation processes are acquiring greater importance due to the reduction of heavy fuel consumption. It is expected that the fluid catalytic cracking (FCC) process will remain the main transformation process for vacuum distillates, due to its large processing capacity and its flexibility, which allows its production to be directed to gases (propylene and butenes), gasoline or diesel, with minimum modifications to the unit or operating conditions.

World consumption of liquid fuels continues to increase, but the demand has been changing in recent years, with a clear increase in diesel demand and a decrease in that of gasoline. This trend is much more marked in the European market ("*Hydrocarbon Processing vol 82, No. 9 p 47 (2003)*"). The demand for light olefins for the petrochemical industry is also in continuous growth ("*Marcilly C., Studies in Surface Science and Catalysis 135, 37 (2001); Hydrocarbon Processing vol. 80, No. 6 p 23 (2001)*"). Fluid catalytic cracking or its variant, deep catalytic cracking (DCC), together with steam cracking, are the units that contribute the most to the production of C_3-C_5 olefins. Catalytic cracking especially produces a large amount of propylene, one of the olefins in greatest demand. Moreover, FCC contributes approximately 30% of the gasoline stream in a refinery, and at the present time when there is, especially in Europe, an excess global production of gasoline, selectivity to propylene in the FCC unit may be increased, optimizing the economic yield of the unit.

Propylene production in the FCC may be increased by modifying the operating conditions in the unit, such as for example increasing the temperature of the reactor. Nevertheless, this solution produces a significant increase in gases, and especially of the undesirable dry gas. Better results are obtained using novel catalyst compositions that involve the use of zeolite mixtures. The use of zeolite ZSM-5 as an additive in FCC catalysts also leads to an increase in C_3 and C_4 olefins (see for example US-3758403, US-3769202; US-3894931; US-3894933;

US-3894934; US-3926782; US-4309280; US-4309279; US-437458 and *Buchanan, J.S. and Adewuyi, Y.G., Applied Catalysis: A General, 134, 247 (1996); Madon, R.J., Journal of Catalysis 129 (1), 275 (1991))*. However, it is known from "*Studies in Surface Science and Catalysis, vol. 76, 499 (1993)*" that the introduction of zeolite ZSM-5 has little or no influence upon total transformation.

Both from the point of view of increasing the barrel octane number of gasoline and that of increasing the yield of C_3-C_4 olefins, and especially propylene, it would be advantageous to find other zeolites that would also allow conversion of feed. Numerous medium- and large-pore zeolites have been studied to this end. The use of MCM-22, Omega, L, mordenite and BEA zeolites is of note (see for example: *J. Catal, 165, 102 (1997); Stud. Surf. Sci. and Catal. 46, 115 (1989)*; US-5314612; EP-489324; US-474292; US-4137152; EP-350331; FR-2661621). It would be especially advantageous if that greater background transformation translated into a greater yield of diesel, without losing the selectivity to C_3-C_4 olefins, and maintaining a high RON and MON octane index for the gasoline.

With regard to diesel production, the characteristics of the catalytic cracking catalyst will also be decisive. It has been observed that it is possible to increase diesel production in FCC by means of the use of novel materials as matrix components (sepiolites, in *Applied Catalysis A: General, 84 (1), 31 (1992)*), of novel materials as active components (the mesoporous material MCM-41, in *Journal of Catalysis 159 (2), 375 (1996)*), or by modifying the zeolite Y conventionally used as an active component in order to increase the accessibility of active centers and the product diffusion (reduction in crystal size, such as in *Studies in Surface Science and Catalysis 154C, 2296 (2004)*).

The present invention relates to a cracking process for organic compounds and preferably for oil-derived or synthetic hydrocarbon fractions, using the zeolitic material called ITQ-33 as the active zeolitic component, the structure of which is defined by a characteristic X-ray diffractogram. The relative positions, widths, and intensities of the diffraction peaks may be modified according to the chemical composition of the material, as well as by the degree of hydration and the crystal size of the zeolite.

The specific pore topology of this zeolitic material will be shown to provide it with activity for cracking the feeds of catalytic cracking units, as well as for producing, surprisingly, on its own and/or in combination with other zeolites, and at the same time, a high yield of diesel and a high yield of light olefins, especially propylene.

DESCRIPTION OF THE INVENTION

The present invention relates to a catalytic cracking process for organic compounds that uses at least one zeolitic material called ITQ-33.

This zeolitic material ITQ-33 used as a catalyst in the process of the present invention exhibits a three-directional channel system, with straight ultra-large pore channels parallel to the *c* axis, defined by circular openings formed by 18 atoms in tetrahedral coordination. These channels are interconnected by a system of channels or windows formed by 10 atoms in tetrahedral coordination located in the *ab* plane, and with openings of 6.1 x 4.3 Å.

This zeolitic material exhibits a new structure or topology that is characterized by a specific X-ray diffraction pattern. The main peaks of the zeolite ITQ-33 diffractogram as synthesized, that differentiate it from other known zeolites, are shown in Table 1.

Table 1: Uncalcined zeolite ITQ-33

d ± 0.2 (Å)	I_{rel}[(a)]
16.59	VS
11.63	VW
9.57	VW
8.31	VW
6.76	VW
4.81	VW
4.62	VW
4.27	W
3.64	W
3.15	W

[(a)] Relative intensity, obtained by assigning a value of 100 to the most intense peak; VW (very weak) corresponds to a value of less than 20, W (weak) from 20 to 40, M (medium) between 40 and 60, S (strong) between 60 and 80 and VS (very strong) between 80 and 100.

Once calcined, the crystalline structure of zeolite ITQ-33 is characterized by an X-ray diffraction pattern the most characteristic lines of which are shown in Table 2.

Table 2: Calcined zeolite ITQ-33

d ± 0.2 (Å)	I_{rel}[(a)]

16.81	VS
11.50	VW
9.71	VW
8.41	VW
6.79	VW
4.84	VW
4.65	VW
4.24	VW
3.66	VW
3.17	VW

[(a)] Relative intensity, obtained by assigning a value of 100 to the most intense peak; VW (very weak) corresponds to a value of less than 20, W (weak) from 20 to 40, M (medium) between 40 and 60, S (strong) between 60 and 80 and VS (very strong) between 80 and 100.

Zeolite ITQ-33 may be synthesized in fluoride medium or in OH^- medium, using an organic structure-directing agent, such as for example the hexamethonium cation, and forming a gel containing in its composition the source of at least one T^{IV} element, amongst which Si and Ge are preferred, and of one or more T^{III} elements, amongst which Al, B, Fe and Ga are preferred, preferably Al, B or a mixture of both, the T^{IV}/T^{III} ratio being at least 5. The synthesized zeolite is subjected to an elimination process to eliminate any organic matter occluded inside the material, which is performed by means of extraction, thermal treatment at temperatures greater than 250°C for a period of time between 2 minutes and 25 hours, or a combination of both, such that, directly or via conventional ion exchanges, including exchanges with rare earths, the acid form that is the preferred form in the catalytic composition is obtained.

The present invention relates to a process for the catalytic cracking of organic compounds characterized in that it comprises at least:

a. introducing at least one first zeolitic material, ITQ-33, inside a reactor,

b. feeding the reactor with at least one organic compound,

c. allowing the zeolitic material ITQ-33 and the organic compound to remain in contact for the time necessary for the reaction to occur.

According to a particular embodiment, said catalytic cracking process comprises, as well as the zeolitic material ITQ-33, a second zeolitic material.

According to that mentioned above, the zeolitic material ITQ-33 used in the

process of the present invention may form part, as a single zeolitic component, of catalytic cracking catalysts for organic compounds, or it may also combine with one or more zeolitic components.

According to a particular embodiment, the second zeolitic material that combines with ITQ-33 is chosen from zeolites with structures containing pores delimited by rings chosen from 14-membered rings, 12-membered rings, 11-membered rings, 10-membered rings and combinations thereof.

According to the previous embodiment, the second zeolitic material is preferably chosen from CIT-5, UTD-1, zeolite Beta, ITQ-7, zeolite Y, SSZ-33; NU-86; ZSM-5, SAPO-11, MCM-22 and combinations thereof.

More preferably, the second zeolitic material is chosen from zeolite Y, zeolite Beta, ZSM-5 and combinations thereof.

According to this particular embodiment, the first zeolitic material ITQ-33 and the second zeolitic material may be present in the same catalyst particle or in separate particles. In both cases the composition of the cracking products is determined by the combined effect of the different zeolitic components. The zeolitic material ITQ-33 may be incorporated in a matrix via a mixture made up of this zeolite as the single zeolitic component, or this zeolite together with another or other zeolite(s) according to that disclosed in the previous paragraph, as well as the binder and any other conventional additives in catalytic cracking catalysts, such as for example kaolin, silica, alumina.

According to the particular embodiment in which said catalytic cracking process comprises a second zeolitic material as well as the zeolitic material ITQ-33, this second zeolitic material may be found in a proportion by weight with regard to the first zeolitic material ITQ-33 of between 2 and 80%.

According to another particular embodiment of the present invention, the first zeolitic material ITQ-33 may be modified by ion exchange preferably chosen from total or partial ion exchange.

Preferably, the ions involved in the ion exchange are chosen from divalent ions, trivalent ions, rare earth cations and combinations thereof.

The catalytic cracking process of the present invention may be performed at a

temperature between 400 and 800°C, and preferably between 450 and 650°C.

The catalytic cracking process described above is a catalytic cracking process for organic compounds.

According to a preferred embodiment, said organic compound is at least a hydrocarbon fraction. This hydrocarbon fraction may be derived from oil or be synthetic.

The catalytic cracking process of the present invention may be used preferably between deep catalytic cracking (DCC) processes and fluid catalytic cracking (FCC) processes.

The amount of the first zeolitic material ITQ-33 in the catalytic cracking catalyst or catalytic cracking additive is between 2 and 60% by weight.

BRIEF DESCRIPTION OF THE DRAWING

Figure 1: X-ray diffraction pattern for uncalcined ITQ-33.

Examples are shown below that illustrate the essence of this invention.

EXAMPLES

Example 1: Synthesis of a sample of the zeolitic component ITQ-33.

3.484 g of GeO_2 are dissolved in 14.543 g of hexamethonium hydroxide solution (24.5% by weight) and 8.035 g of hexamethonium bromide solution (50% by weight). 1.043 g of aluminum isopropoxide and 14.205 g of tetraethyl orthosilicate (TEOS) are added. Finally, having hydrolyzed the alkoxides, 1.254 g of hydrofluoric acid solution (48% by weight) are added and the mixture is kept stirring allowing it to evaporate until the reaction mixture reaches a final composition of:

0.67 SiO_2 : 0.33 GeO_2 : 0.025 Al_2O_3 : 0.15 $R(OH)_2$: 0.10 $R(Br)_2$: 0.30 HF : 1.5 H_2O

wherein R is hexamethonium.

The gel is heated to 175°C without stirring for 20 hours in steel autoclaves with an internal Teflon sheath. The solid obtained after filtering, washing with distilled water and drying at 100°C is ITQ-33.

The X-ray powder diffraction pattern from the solid obtained after filtering, washing and drying at 100°C is shown in Figure 1 and in Table 3.

Table 3: Uncalcined zeolite ITQ-33.

(d ± 0.2) (Å)	Relative Intensity
16.59	vs
11.63	vw
9.57	vw
8.31	vw
6.76	vw
4.81	vw
4.62	vw
4.27	w
4.02	vw
3.86	vw
3.82	vw
3.64	w
3.33	vw
3.15	w
2.78	vw
2.75	vw
2.54	vw
2.38	vw
2.27	vw

The relative intensity of the lines is calculated as the percentage with respect to the most intense peak, and is considered very strong (vs)= 80-100, strong (s)=60-80, medium (m)= 40-20, weak (w)=20-40, and very weak (vw)= 0-20.

Example 2: Activation by means of calcination of the zeolitic component ITQ-33.

The zeolite obtained from Example 1 is calcined in air flow at 540°C for 3 hours. The X-ray diffraction pattern of the calcined material is shown in Table 4.

Table 4: Calcined zeolite ITQ-33.

(d ± 0.2) (Å)	Relative Intensity
16.81	VS
11.50	VW
9.71	VW

8.41	VW
6.79	VW
4.84	VW
4.65	VW
4.24	VW
4.01	VW
3.84	W
3.81	VW
3.66	VW
3.36	W
3.17	VW
2.81	VW
2.56	VW
2.37	VW
2.27	VW

The relative intensity of the lines is calculated as the percentage with respect to the most intense peak, and is considered very strong (VS)= 80-100, strong (S)=60-80, medium (M)= 40-60, weak (W)=20-40, and very weak (VW)= 0-20.

Example 3: Catalytic cracking of a vacuum gas oil with a catalyst containing ITQ-33.

A first catalyst, Catalyst A, was prepared in this example, with the zeolite from Example 1. The uncalcined zeolite was first pelleted, ground in a mortar and sieved, taking the fraction between 0.59 and 0.84 mm in diameter. 0.67 g of this sample were mixed with SiO_2 (2.5 g) sieved to a particle size of 0.25-0.42 mm, and this mixture was calcined "in-situ" for 3 hours at 540°C in air flow (100 ml/min).

The catalytic cracking reaction of the vacuum gas oil (Table 5) with this catalyst was carried out in a fixed bed reactor of the "Microactivity Test" (MAT) type, at 500°C, with a 60-second feed time, and at different catalyst/feed ratios expressed by weight of zeolite/feed weight.

Table 5: Vacuum gas oil properties.

Density (15°C) g cc^{-1}	0.9172
Aniline point (°C)	79.2
Sulfur (% by weight)	1.65
Nitrogen (ppm)	1261

Na (ppm)	0.18
Cu (ppm)	< 0.1
Fe (ppm)	0.30
Ni (ppm)	0.2
V (ppm)	0.40
ASTM D-1160 (°C)	
5%	319
10%	352
30%	414
50%	436
70%	459
90%	512
VABP (°C)	435
K (UOP)	11.82
Mean molecular weight	407
Aromatic carbon (weight %)	22.96
Naphthenic carbon (weight %)	15.16
Paraffinic carbon (weight %)	61.88

Table 6 shows the interpolated yields at 86% total transformation, the transformation being defined as the sum of gasoline, diesel (LCO), gases and coke yields.

Table 6: Comparison between activity and selectivity at constant transformation.

Zeolite	Catalyst A ITQ-33	Catalyst B USY-1	Catalyst C Beta
Transformation (%)	86	86	86
CAT/OIL	0.69	0.34	0.74
Yield (%):			
Gasoline	33.7	38.5	31.7
Diesel	23.1	20.6	13.6
Gases	20.0	23.1	36.5
Coke	9.2	3.9	4.3
Hydrogen	0.6	0.1	0.2
Methane	1.0	0.6	0.7
Ethane	1.3	0.8	1.0
Ethylene	1.3	1.5	1.8
Propane	2.0	3.0	4.4

Propylene	4.0	4.2	8.0
Isobutane	4.1	6.8	8.2
n-Butane	1.1	1.9	2.7
T2-butene	1.1	1.2	2.0
1-butene	1.1	1.2	2.0
Isobutene	1.5	1.0	3.8
C2-butene	0.9	1.0	1.8

These tables compare the catalytic cracking results for vacuum gas oil obtained using a commercial USY zeolite (Zeolyst CBV720), that we will call zeolite USY-1 with a unit cell of 24.32 Å (Catalyst B), and with a commercial zeolite Beta (Zeolyst CP811) with a Si/Al ratio=13 (Catalyst C). Catalysts B and C were prepared from commercial zeolites (0.5 g of zeolite), by mixing them with silica (2.5 g of silica) and forming them in the same manner as Catalyst A. The results show that Catalyst A, with the zeolite the use of which is claimed in this patent, is more active than Catalyst C and produces a greater yield of diesel fraction than Catalysts B and C (Table 6). The amount of total butenes and propylene obtained at constant transformation is in the order of that obtained with the catalyst based on zeolite USY-1 (Table 6).

Example 4: Catalytic cracking of a vacuum gas oil with a catalyst containing ITQ-33 combined with zeolite ZSM-5.

This example compares the activity and selectivity in the cracking of a vacuum gas oil (Table 7) obtained by combining the zeolite from Example 1 with a zeolite ZSM-5 with a Si/Al ratio = 40 (Zeolyst CBV8020), in a zeolite ITQ-33/zeolite ZSM-5 ratio of 1:0.2 by weight (Catalyst D), with those obtained by a combination of zeolite USY-1, referenced in Example 3, with a zeolite ZSM-5 with a Si/Al ratio = 40 (Zeolyst CBV8020), in a zeolite USY-1/zeolite ZSM-5 ratio of 1:0.2 by weight (Catalyst E). Both for Catalyst D and for Catalyst E the zeolites have been arranged in two consecutive beds such as disclosed in Corma, A. and Martínez-Triguero, J., Journal of Catalysis 165 (1997) 102-120. The reaction has been carried out in the experimental conditions provided in Example 3.

Table 7: Comparison between activity and selectivity at constant transformation.

	Catalyst D	Catalyst E
Zeolite	ITQ-33 + ZSM-5	USY-1 + ZSM-5
Transformation (%)	86	86
CAT/OIL	0.72	0.37

Yield (%):		
Gasoline	25.2	33.7
Diesel	23.3	18.1
Gases	28.9	32.0
Coke	8.6	2.1
Hydrogen	0.6	0.1
Methane	0.8	0.6
Ethane	1.2	0.9
Ethylene	3.1	2.8
Propane	2.5	4.2
Propylene	9.0	7.5
Isobutane	2.8	7.0
n-Butane	1.2	2.5
T2-butene	1.6	1.4
1-butene	1.6	1.4
Isobutene	3.2	2.7
C2-butene	2.1	1.1

The results obtained (Tables 7 and 8) show that Catalyst D, based on the combination of the zeolite forming the subject of this patent with a zeolite ZSM-5, produces a greater amount of propylene and butenes than Catalyst E, formed by a combination of zeolite USY-1 (24.32 Å) and a zeolite ZSM-5. Moreover, Catalyst D produces ~5 points more diesel than Catalyst E, showing the advantage of the catalyst that combines zeolite ITQ-33 and ZSM-5 with regard to that combining USY and ZSM-5 in order to simultaneously produce high yields of diesel and LPG olefins, and especially propylene.

Table 8: Comparison of activity and selectivity at constant gases yield.

	Catalyst D	Catalyst E
Zeolite	ITQ-33 + ZSM-5	USY-1 + ZSM-5
Yield at C1-C4 gases (%)	28	28
CAT/OIL	0.72	0.72
Transformation (%)	86.1	80.8
Yield (%):		
Gasoline	25.2	30.5
Diesel	23.3	19.0
Coke	8.6	3.3
Hydrogen	0.6	0.1

Methane	0.8	0.5
Ethane	1.2	0.8
Ethylene	3.1	2.3
Propane	2.5	3.6
Propylene	9.0	6.6
Isobutane	2.8	6.0
n-Butane	1.2	2.1
T2-butene	1.6	1.2
1-butene	1.6	1.3
Isobutene	3.2	2.5
C2-butene	1.3	1.1

Example 5. Quality of the gasoline obtained during catalytic cracking of a vacuum gas oil using ITQ-33 as the zeolitic component.

This example shows (Table 9) the PIONA analysis of the gasoline obtained during the cracking of the vacuum gas oil in Examples 3 and 4.

Table 9: PIONA analysis of the gasoline obtained.

Zeolite	Catalyst A ITQ-33	Catalyst B USY-1	Catalyst D ITQ-33 + ZSM-5	Catalyst E USY-1 + ZSM-5
Transformation, weight %	88.1	88.3	86.1	87.0
n-paraffins, weight %	7.3	2.7	3.4	2.2
i-paraffins, weight %	16.4	21.7	12.3	13.9
Olefins, weight %	7.3	5.6	3.9	2.3
Naphthenes, weight %	9.2	9.3	5.9	7.5
Aromatics, weight %	52.2	55.1	64.0	69.6

Others, weight %	11.6	5.6	10.5	4.5

CLAIMS

1. Catalytic cracking process for organic compounds characterized in that it comprises at least:
   a. introducing at least one first zeolitic material, ITQ-33, inside a reactor,
   b. feeding the reactor with at least one organic compound,
   c. allowing the zeolitic material ITQ-33 and the organic compound to remain in contact for the time necessary for the reaction to occur.

2. Catalytic cracking process according to Claim 1, characterized in that it also comprises a second zeolitic material.

3. Catalytic cracking process according to Claim 2, characterized in that said second zeolitic material is chosen from zeolites with structures containing pores delimited by rings chosen from 14-membered rings, 12-membered rings, 11-membered rings, 10-membered rings, and combinations thereof.

4. Catalytic cracking process according to one of Claims 2 and 3, characterized in that the second zeolitic material is chosen from zeolites CIT-5, UTD-1, Beta, ITQ-7, zeolite Y, SSZ-33, NU-86, ZSM-5, SAPO-11, MCM-22 and combinations thereof.

5. Catalytic cracking process according to one of Claims 2 to 4, characterized in that the second zeolitic material is chosen from zeolite Y, zeolite Beta, ZSM-5 and combinations thereof.

6. Catalytic cracking process according to one of Claims 2 to 5, characterized in that the first zeolitic material ITQ-33 and the second zeolitic material are present in the catalyst in separate particles.

7. Catalytic cracking process according to one of Claims 2 to 5, characterized in that the first zeolitic material ITQ-33 and the second zeolitic material are present in the same catalyst particle.

8. Catalytic cracking process according to Claims 6 and 7, characterized in that the second zeolitic material is found in a proportion by weight with regard to the first zeolitic material ITQ-33 of between 2 and 80%.

9. Catalytic cracking process according to one of the preceding claims,

characterized in that the first zeolitic material ITQ-33 is modified by ion exchange.

10. Catalytic cracking process according to Claim 9, characterized in that said ion exchange is total.

11. Catalytic cracking process according to Claim 9, characterized in that said ion exchange is partial.

12. Catalytic cracking process according to one of Claims 9 to 11, characterized in that said ions are chosen from divalent ions, trivalent ions, rare earth cations and combinations thereof.

13. Catalytic cracking process according to Claim 1, characterized in that it is performed at a temperature between 400 and 800°C.

14. Catalytic cracking process according to Claim 13, characterized in that it is performed at a temperature between 450 and 650°C.

15. Catalytic cracking process according to one of the preceding claims, characterized in that the organic compound is at least a hydrocarbon fraction.

16. Catalytic cracking process according to Claim 15, characterized in that said hydrocarbon fraction is derived from oil.

17. Catalytic cracking process according to Claim 15, characterized in that said hydrocarbon fraction is synthetic.

18. Catalytic cracking process according to one of the preceding claims, characterized in that said process is chosen from the deep catalytic cracking DCC process and the fluid catalytic cracking FCC process.

19. Catalytic cracking process according to Claim 18, characterized in that the amount of the first zeolitic material ITQ-33 in the catalytic cracking catalyst or catalytic cracking additive is between 2 and 60% by weight.

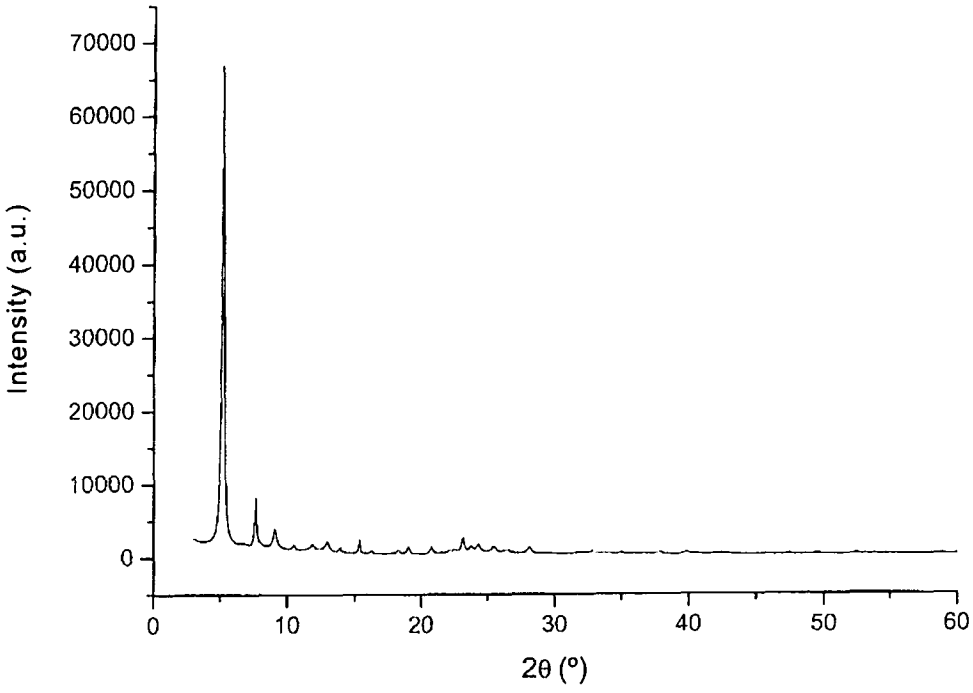
70000
60000
50000
40000
30000
20000
10000
0
Intensity (a.u.)
0
10
20
30
40
50
60
2θ (°)

Figure 1