



Θαλής - ΤΕΙ Καβάλας - Nanocapillary

SBA & MCM

MIS 375233

	μ	
1.		3
2. MCM		4
2.1	MCM	4
2.2		5
2.3	μ	9
3. SBA		10
3.1	SBA	10
3.2		11
3.3	μ	12
4.		15
5.	μ μ	16



MCM SBA

Nanocapillary

MCM SBA.

μ μ μ 2

50nm IUPAC. μ μ μ

(). ,

· μ μ

μ μ μ MCM

SBA μ μ SBA – 15 MCM – 41.

μ Alicante μ μ Antwerp,

μ μ , μ

μ μ μ

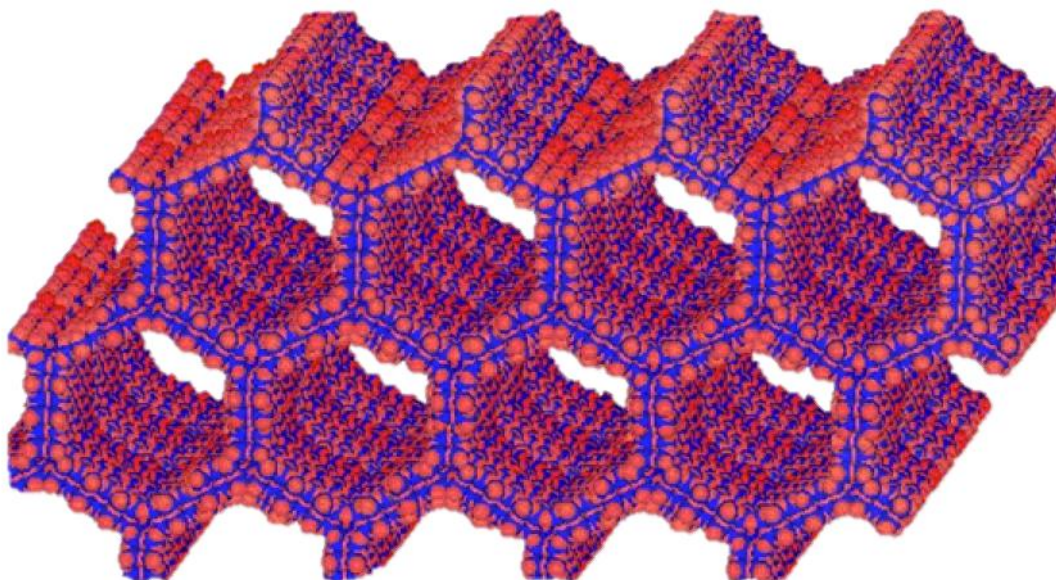
μ μ μ



1. MCM

1.1 MCM

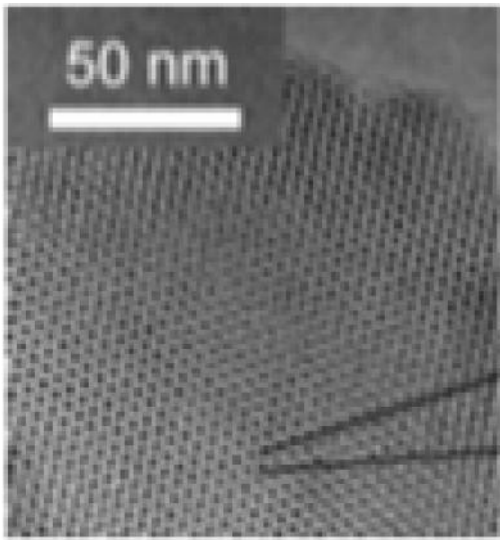
MCM (Mobil Composition of Matter) μ
1992 Mobil (μ)
μ) . μ , μ
μ μ 30-100 μ
μ μ μ μ μ



. 1. MCM - 41



MCM – 41
 μ μ μ
 μ μ μ
 μ (. 1) μ (surface area)
 1200m²/gr μ μ
 μ μ μ μ μ μ
 μ μ μm (2).



. 2.

TEM

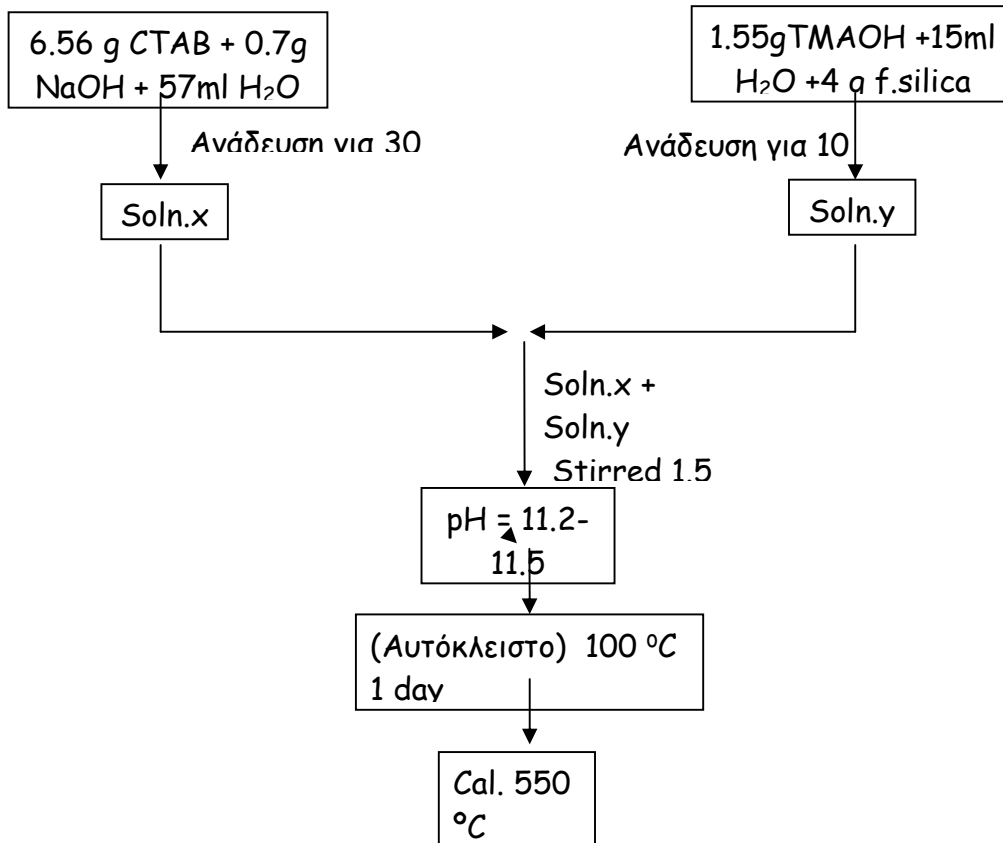
MCM

1.2

MCM – 41 μ μ 5
 , μ μ μ 1.5
 20nm. μ μ μ 1 1.5
 nm, μ μ μ



μ μ Antwerp, μ μ MCM – 41
 μ μ μ MCM-41:



μ μ MCM-41 :
 μ , μ μ (. . Cab-o-sil) μ ()
 μ μ (TMOS: (CH₃O)₄Si)



μ) μ SiO_2 (μ μ
 μ 100 °C μ 48 μ 1
 μ .
 μ μ μ Si μ μ
 μ μ μ , μ μ Si (SiO_2)
 μ μ μ μ SiO_2
 μ .

MCM – 41. μ

MCM – 41 ((fumed silica))

μ μ μ μ μ .

:

- μ 6.2gr CTMABr μ 40.4 gr μ 20 μ μ
 μ μ
- μ 20% μ TEAOH
- μ 4.1gr μ 2 μ 70 C
- μ 24 μ μ .
- μ μ μ μ μ 130 – 150 oC
- 48
- μ μ , μ
- μ μ 150 mL H₂O μ μ 130 –
 150 oC 72

- μ μ 10C μ μ 550oC 6

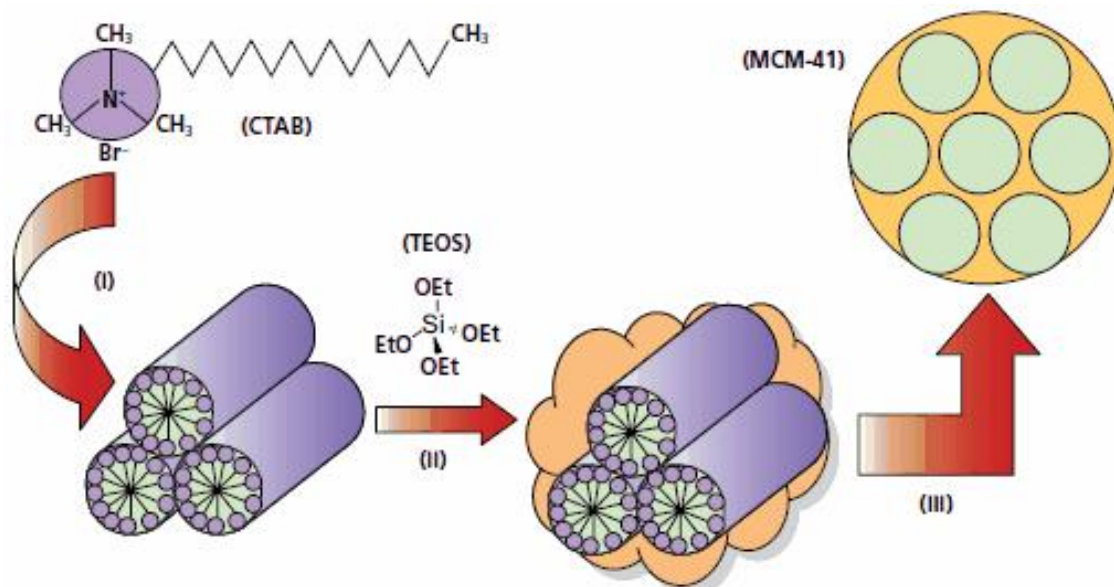
MCM-41 (μ)

μ μ

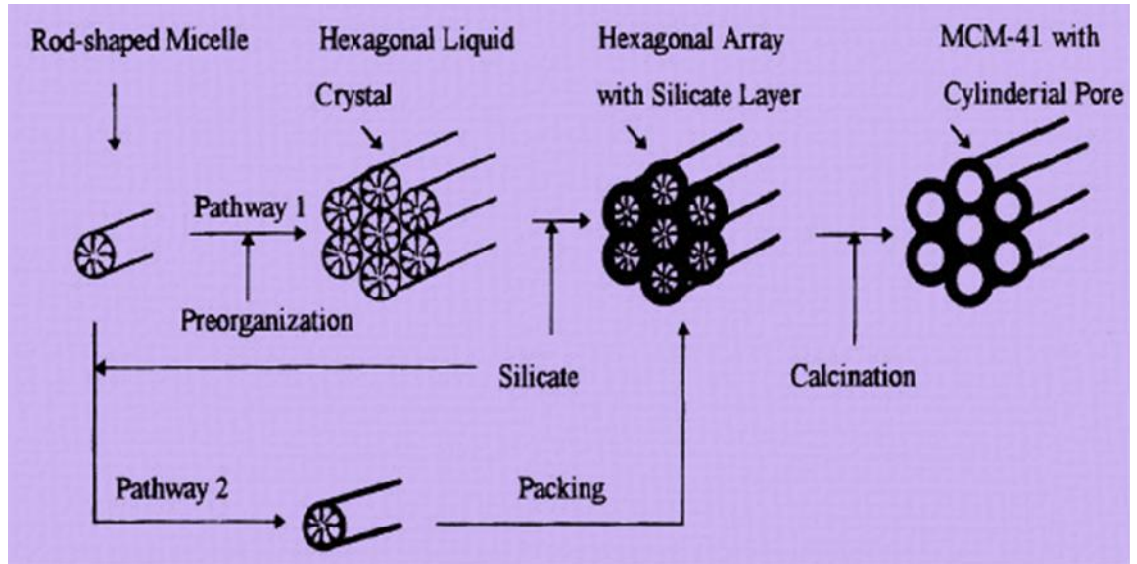
μ μ μ

:

- 400mL μ 8.13gr μ Na
 - 120gr μ μ 30
 - μ 20% μ TEAOH
 - μ 4.84gr μ $\mu\mu$ μ 30gr
 - 10gr EtOH μ 30
 - μ 2 μ μ 30
- μ



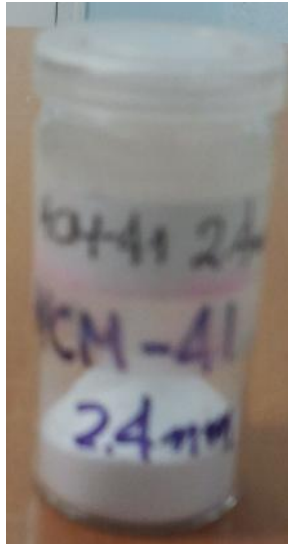
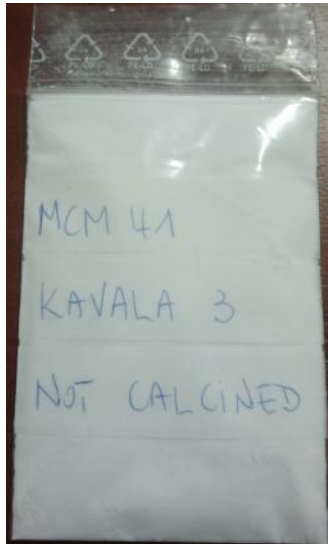
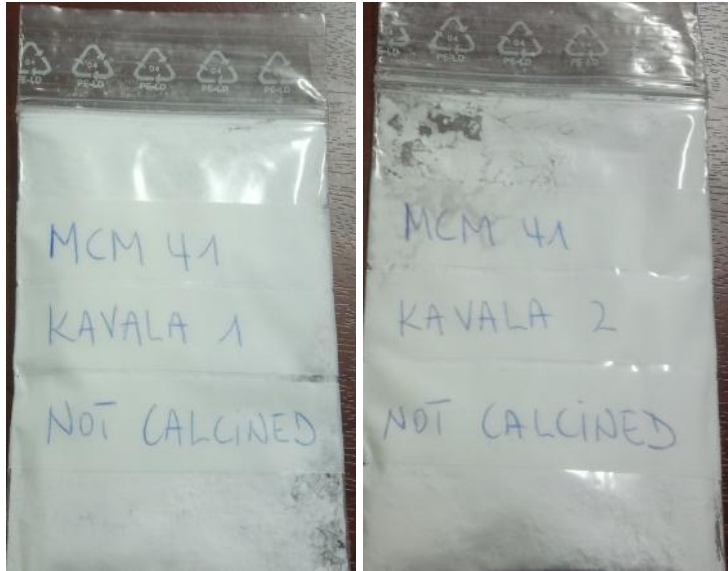
. 3. μμ MCM CTAB.



. 4. μμ MCM Micelle.

3 4 μ
MCM. 3 CTAB
TEOS μ μ calcination MCM – 41.
4 MCM – 41 μ μ
μ .

1.3 μ



5. μ MCM – 41

TEI AM

3 μ

MCM – 41 μ

calcination.

μ μ μ μ μ

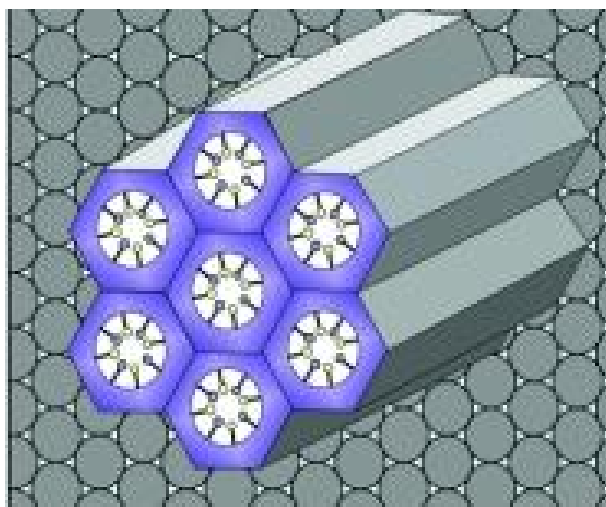
μ SAXS, XRD, SEM, TEM, Nitrogen

Porosimetry .

2. SBA

2.1 SBA

To SBA 6 MCM μ
 μ Santa Barbara (Santa Barbara Amorphous). SBA
 μ μ , μ
 . μ SBA ,
 , μ
 SBA SBA – 15 μ μ μ μ
 μ 4 – 14nm. μ μ
 μ SBA – 15 μ μ
 μ μ SBA – 15.



. 6

SBA - 15

μ

μ

μ

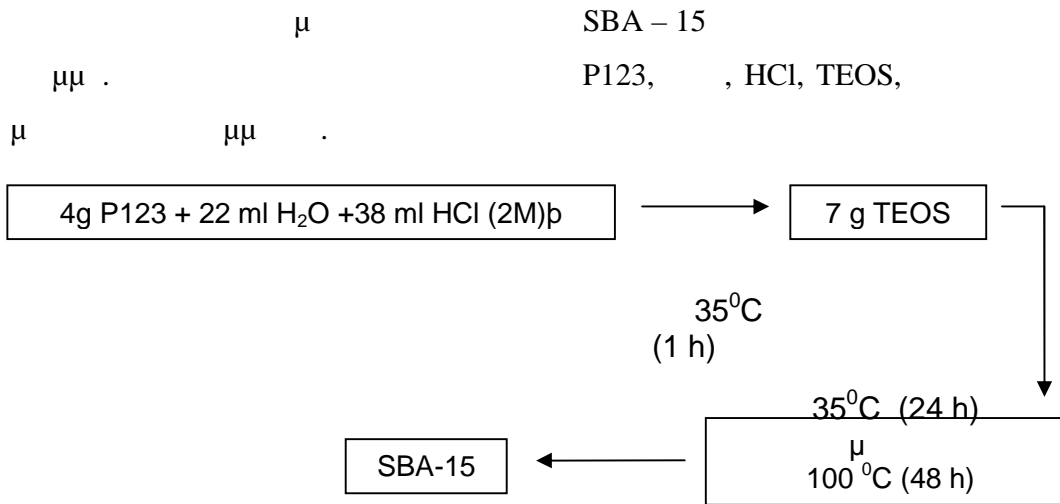
μ

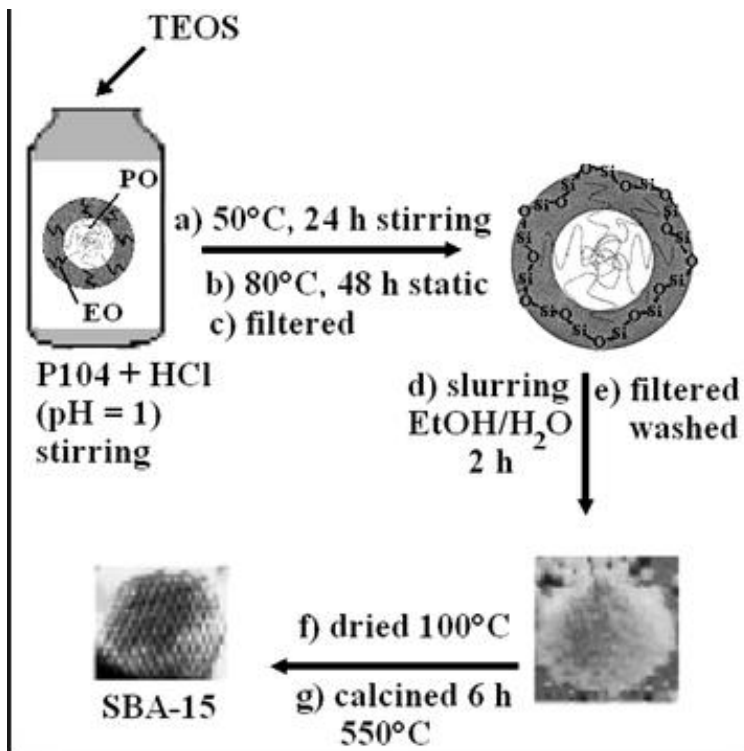
SBA – 15

(PEO)

μ .

2.2



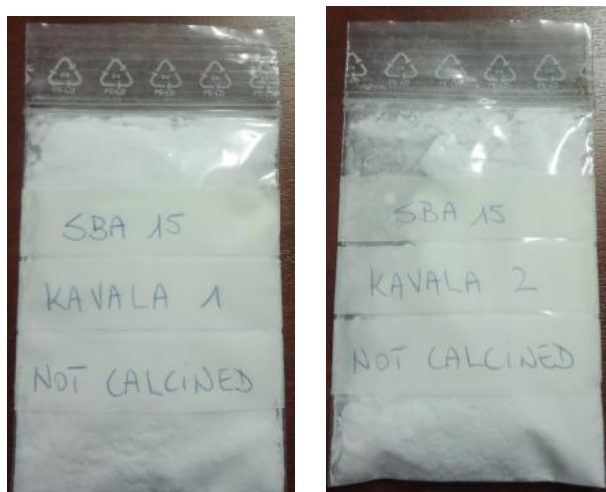


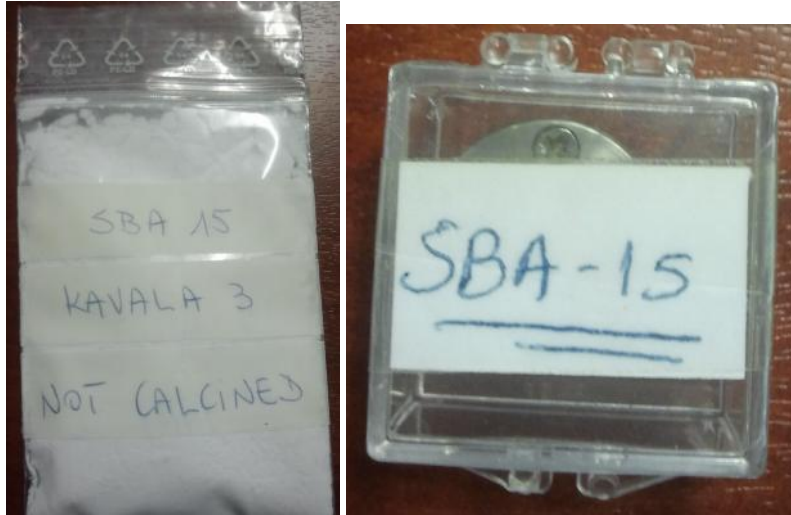
.7

SBA – 15

2.3

μ





8. μ SBA – 15

ΤΕΙ ΑΜ

-15 μ , 3 μ SBA
 μ calcination. μ
 μ μ μ μ ,
 μ SAXS, XRD, SEM, TEM, Nitrogen Porosimetry .

3.

Antwerp μ μ μ μ
 Alicante μ .



.9. μ

ΤΕΙ ΑΜ

- GP-83B
- 21B
- GP-61B
- GP-77
- Gt-64#4
- C2
- C1
- C2 powder
- C1 powder
- C3
- C3 powder
- S2(b)
- 55B
- 55C
- GT - 64A
- GT - 64B
- GT - 64C
- Zn3Al - TiO2

- $(Cu+Zn)(Al - TiO_2)$
- TiO_2 Seeds
- $(Cu+Zn)/(Fe+Ti)(TiO_2)$
- Zn_3Al
- Zn_3Fe
- $(Cu+Zn)/Fe-TiO_2$
- $(Cu+Zn)/(Fe+Ti)$
- $Zn(Fe+Ti)$
- $Zn/(Fe+Ti)- TiO_2$
- $(Cu+Zn)/Fe$
- $(Cu+Zn)/Al$
- $Zn_3Fe- TiO_2$
- NH_4F (3.7ml)
- HF (0.6ml)
- HF (1.0 ml)
- NH_4F (2.2ml)
- NaF (33ml)
- NaF (20ml)

μ

μ

μ

μ

,

μ

μ

,

μ

.

4. Κινητικότητα

Nanocapillary –

μ

μ

μ

μ

μ

μ

.

μ

14 – 18/1/2013 μ

Dr. J. W. Nolan

μ

Antwerp,

μ

Prof. E. F. Vansant

Prof.



P. Cool. μ μμ
 μ MCM SBA. 5 8. μμ Dr. No lan
 μμ
 μ μ Prof. E. F. Vansant Prof. P. Cool
 μ μ
 μ 22 – 27/10/2013 μ Antwerp μ
 Dr. J. W. Nolan μ Antwerp μ
 μ μ μ Prof. Vansant,
 μ μ Antwerp μ
 μ μ
 μ μ μ
 μ μ μ
 μ μ μ
 μ μ μ

5. Πειραματικά Δεδομένα

MCM – 41

**MCM - Mobil Composition of Matter (1992)
 Mesoporous Material**

Description

Hexagonally ordered mesoporous material with small mesopores.
 Type of material: Silica

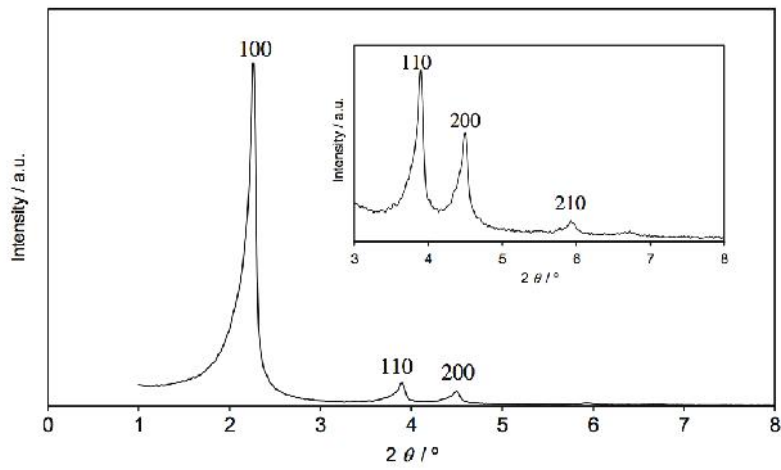
Pore Size Distribution

BJH 1.0cm³/gr
 2.5-3 nm pore size
 =0.34gr.mL



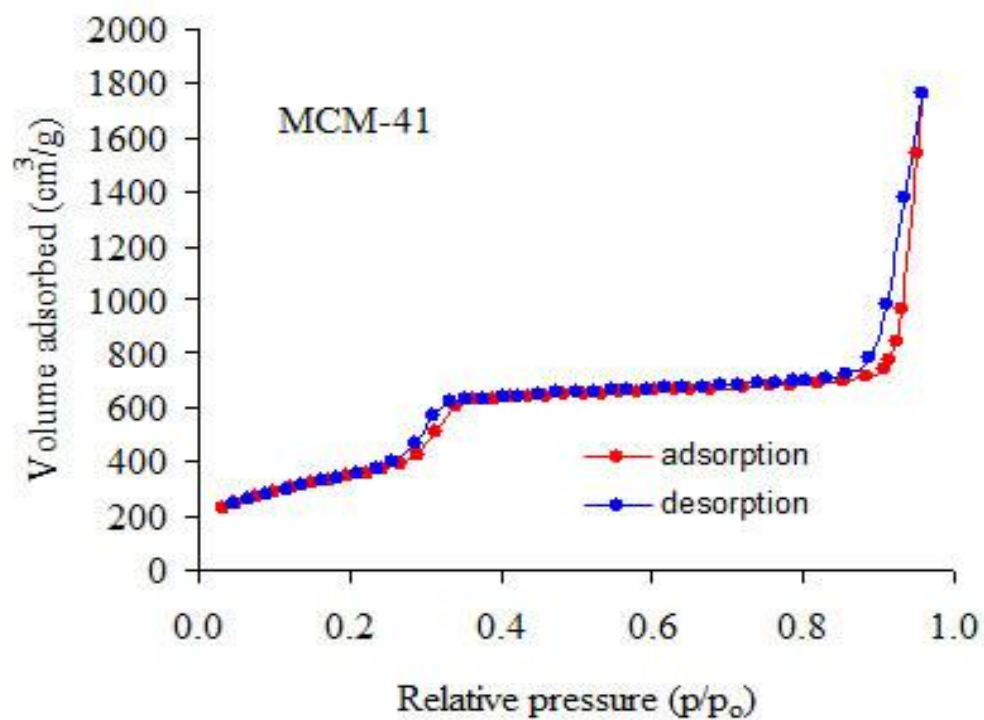


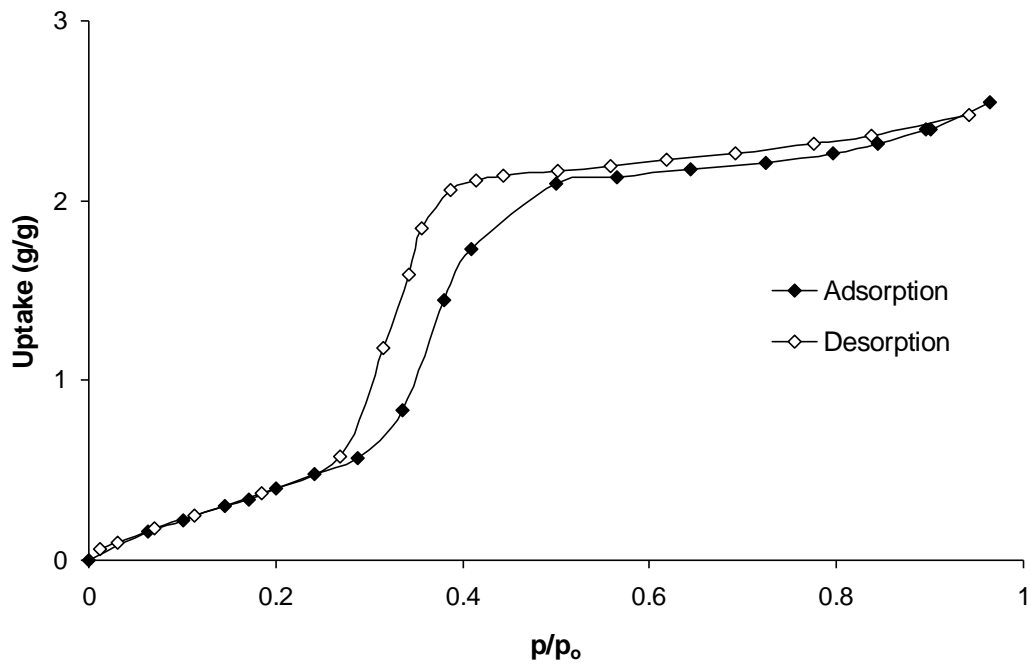
XRD Spectra



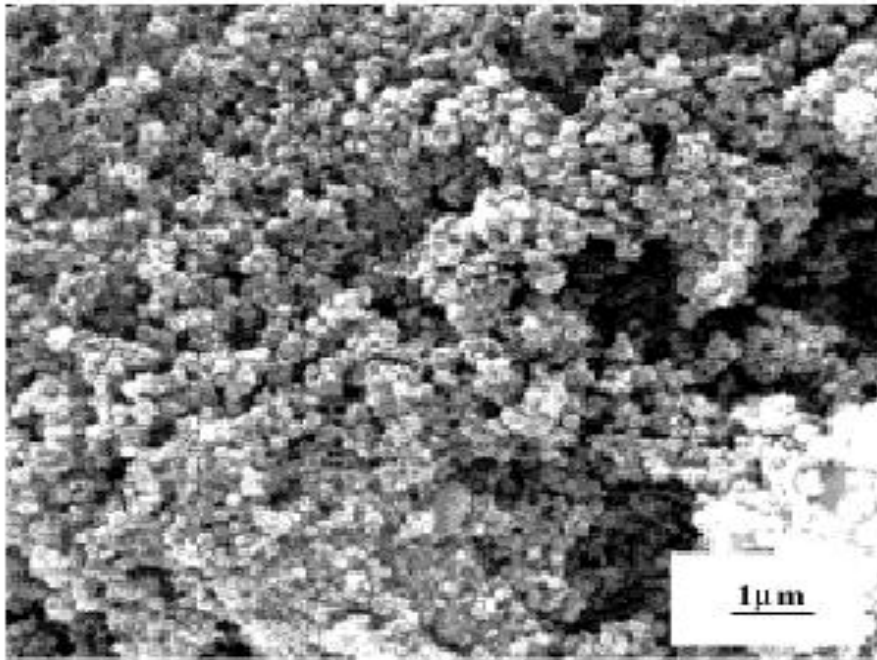
Nitrogen Porosimetry - Isotherm



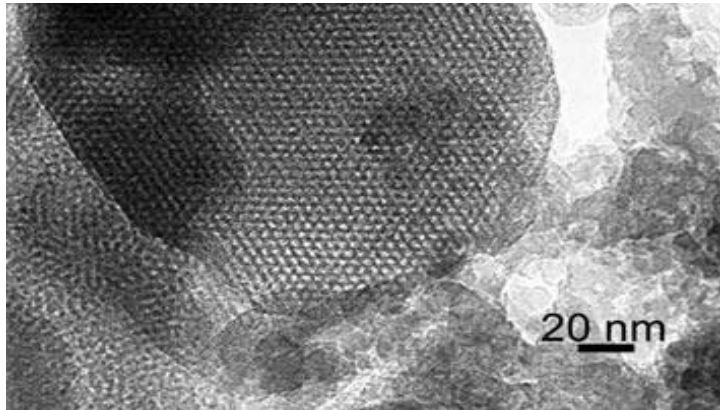




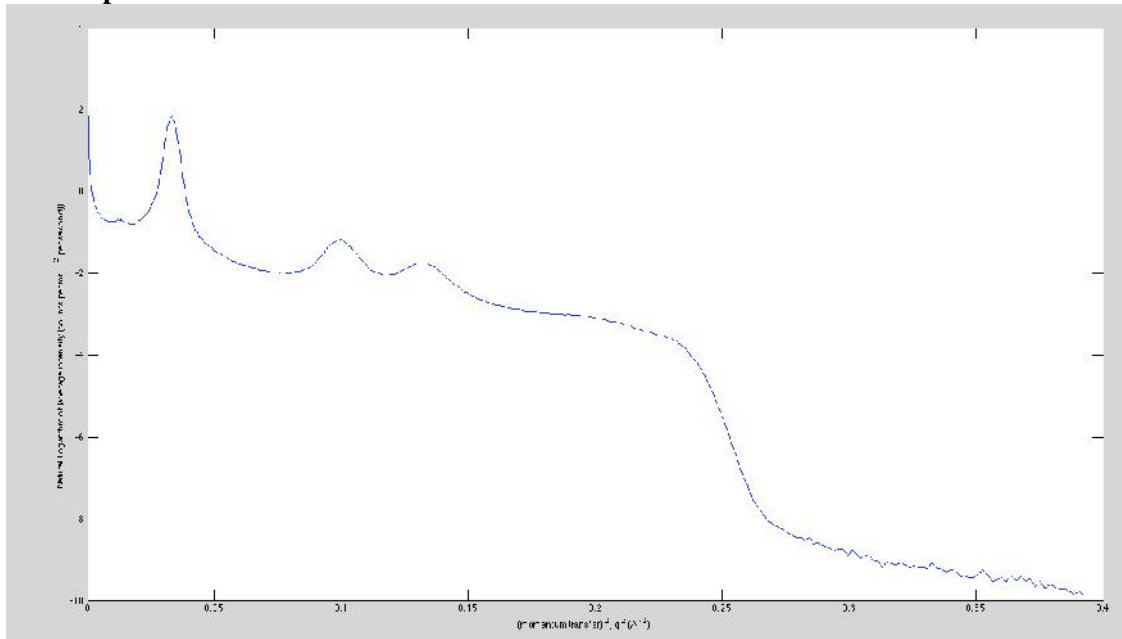
SEM – Picture



TEM – Picture



SAXS Spectra



Recipe

MCM-41 (from fumed silica)*

Batch composition: 1 SiO₂:0.25 CTMABr:39.36 H₂O:0.20 TEAOH. Source of materials:

Cetyltrimethylammonium bromide 99+% (CTMABr) (Acros Organics).

Tetraethylammoniumhydroxide 20% (TEAOH 20%) (Sigma– Aldrich).

Fumed silica (aerosil 380, Degussa).

Batch preparation:

1. Add 6.2 g CTMABr to 40.4 g H₂O, stir at room temperature until dissolved.
2. Add 10 g of TEAOH 20% solution.
3. Add 4.1 g fumed silica and stir at 70 °C for 2 h (stirring is crucial. If stirring is stopped due to a high viscosity, it is possible to add between 1 and 5 ml of water to keep it stirring).
4. Stir for a duration of 24 h at room temperature.
5. Transfer the viscous solution into an autoclave and heat to 130–150 °C for 48 h.
6. After the heat treatment, quench the autoclave and filter the solution.
7. Wash the solid with 150 mL H₂O.
8. Transfer the solid into an autoclave and add some fresh water (until the solid is just covered) and heat it a second time to 130–150 °C for 72 h.

REFERENCES

Beck, J.S., Vartuli, J.C., Roth, W.J., Leonowicz, M.E., Kresge, C.T., Schmitt, K.D., Chu, C.T.-W., Olson, D.H., Sheppard, E.W., McCullen, S.B., Higgins, J.B., Schlenker, J.L.
A new family of mesoporous molecular sieves prepared with liquid crystal templates
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DOCUMENT TYPE: Article

SOURCE: Scopus

Huo, Q., Margolese, D.I., Stucky, G.D.
Surfactant Control of Phases in the Synthesis of Mesoporous Silica-Based Materials
(1996) *Chemistry of Materials*, 8 (5), pp. 1147-1160. Cited 1193 times.

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Taguchi, A., Schüth, F.

Ordered mesoporous materials in catalysis

(2005) *Microporous and Mesoporous Materials*, 77 (1), pp. 1-45. Cited 983 times.

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DOCUMENT TYPE: Review

SOURCE: Scopus

Kruk, M., Jaroniec, M., Sayari, A.

Application of large pore MCM-41 molecular sieves to improve pore size analysis using nitrogen adsorption measurements

(1997) *Langmuir*, 13 (23), pp. 6267-6273. Cited 905 times.



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Conducting polyaniline filaments in a mesoporous channel host
(1994) *Science*, 264 (5166), pp. 1757-1759. Cited 903 times.
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SOURCE: Scopus

Maschmeyer, T., Rey, F., Sankar, G., Thomas, J.M.
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SOURCE: Scopus

Cassiers, K., Linssen, T., Mathieu, M., Benjelloun, M., Schrijnemakers, K., Van Der Voort, P., Cool, P., Vansant, E.F.
A detailed study of thermal, hydrothermal, and mechanical stabilities of a wide range of surfactant assembled mesoporous silicas
(2002) *Chemistry of Materials*, 14 (5), pp. 2317-2324. Cited 194 times.
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DOCUMENT TYPE: Article
SOURCE: Scopus

Van Der Voort, P., Mathieu, M., Mees, F., Vansant, E.F.
Synthesis of high-quality MCM-48 and MCM-41 by means of the GEMINI surfactant method
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SOURCE: Scopus

Patent

Beck, J. S. *U.S. Patent 5, 057, 296*. 1991

Recent patents

BI-MICROPOROUS-MESOPOROUS COMPOSITE MOLECULAR SIEVE Y-BETA/ MCM- 41 AND PREPARING METHOD THEREOF □□*PATENT COOPERATION TREATY APPLICATION*, April 2011 □ WANG, Dongqing ; LI, Quanzhi ; ZHANG, Zhihua ;

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Ευρωπαϊκή Ένωση
Ευρωπαϊκό Κοινωνικό Ταμείο



ΥΠΟΥΡΓΕΙΟ ΠΑΙΔΕΙΑΣ ΚΑΙ ΘΡΗΣΚΕΥΜΑΤΩΝ
ΕΙΔΙΚΗ ΥΠΗΡΕΣΙΑ ΔΙΑΧΕΙΡΙΣΗΣ

Με τη συγχρηματοδότηση της Ελλάδας και της Ευρωπαϊκής Ένωσης



ΕΥΡΩΠΑΪΚΟ ΚΟΙΝΩΝΙΚΟ ΤΑΜΕΙΟ

TIAN, Ran ; SUN, Famin ; LI, Xuguang ; YU, Chunmei ; DAI, Baoqin ; ZHAO, Ye ; WANG, Fucun ; HUANG, Yao ; LV, Qian ; GUO, Shuzhi ; LI, Haiyan ; ZHANG, Qinqwu ; MA, Shoutao ; CONG, Liru ; ZHANG, Quanguo ; BING, Shuqiu (PETROCHINA COMPANY LIMITED ; WANG, Dongqing ; LI, Quanzhi ; ZHANG, Zihua ; TIAN, Ran ; SUN, Famin ; LI, Xuguang ; YU, Chunmei ; DAI, Baoqin ; ZHAO, Ye ; WANG, Fucun ; HUANG, Yao ; LV, Qian ; GUO, Shuzhi ; LI, Haiyan ; ZHANG, Qinqwu ; MA, Shoutao ; CONG, Liru ; ZHANG, Quanguo ; BING, Shuqiu)

MCM- 41 TYPE MICROPOROUS MATERIALS CONTAINING TITANIUM AND THEIR UTILIZATION AS CATALYSTS IN ALPHA-PINENE OXIDATION □□EUROPEAN PATENT APPLICATION, January 2002 □CORMA CANOS, Avelino, Instituto de Tec. Quimica ; DOMINE, Marcelo E., Instituto de Tec. Quimica ; SUSARTE ROGEL, Manuel, Instituto de Tec. Quimica ; REY GARCIA, Fernando, Instituto de Tec. Quimica (CONSEJO SUPERIOR DE INVESTIGACIONES CIENTIFICAS ; UNIVERSIDAD POLITECNICA DE VALENCIA) (+)□□

MCM- 41 TYPE MICROPOROUS MATERIALS CONTAINING TITANIUM AND THEIR UTILIZATION AS CATALYSTS IN α -PINENE OXIDATION □□PATENT COOPERATION TREATY APPLICATION, September 2000 □CORMA CANOS, Avelino ; DOMINE, Marcelo, Eduardo ; SUSARTE ROGEL, Manuel ; REY GARCIA, Fernando (CONSEJO SUPERIOR DE INVESTIGACIONES CIENTIFICAS ; UNIVERSIDAD POLITECNICA DE VALENCIA

HYDROTHERMALLY STABLE METAL-CONTAINING MCM- 41 TYPE INTERMEDIATE FINE PORE MOLECULAR SIEVE □□PATENT ABSTRACTS OF JAPAN, September 2001 □TEI, SHUKUFUN ; TEKIHASAISHI, TATSUSHI (CHINESE PETROLEUM CORP) □Full text available at patent office. For more in-depth searching go to

Adsorption by Powders and Porous Solids, Second Edition: Principles, Methodology and Applications [Hardcover]

Jean Rouquerol (Author), Françoise Rouquerol (Author), Philip Llewellyn (Author), Guillaume Maurin (Author), Kenneth S.W. Sing (Author)

Characterization of Porous Solids and Powders: Surface Area, Pore Size and Density (Particle Technology Series) [Paperback]

Seymour Lowell (Author), Joan E. Shields (Author), Martin A. Thomas (Author), Matthias Thommes (Author)

Nanoporous Materials: Synthesis and Applications [Hardcover]

SBA – 15

Large pore hexagonal mesoporous material with micropores in the walls.

μ

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Ευρωπαϊκή Ένωση
Ευρωπαϊκό Κοινωνικό Ταμείο



ΥΠΟΥΡΓΕΙΟ ΠΑΙΔΕΙΑΣ ΚΑΙ ΘΡΗΣΚΕΥΜΑΤΩΝ
ΕΙΔΙΚΗ ΥΠΗΡΕΣΙΑ ΔΙΑΧΕΙΡΙΣΗΣ

Με τη συγχρηματοδότηση της Ελλάδας και της Ευρωπαϊκής Ένωσης



ΕΥΡΩΠΑΪΚΟ ΚΟΙΝΩΝΙΚΟ ΤΑΜΕΙΟ

Poly(ethylene glycol)-block-poly(propylene glycol)-block-poly (ethylene glycol) 4gr
<http://www.sigmaaldrich.com/catalog/product/aldrich/412325?lang=en®ion=GR>
H2O 130mL
HCL 20mL (<http://www.sigmaaldrich.com/catalog/product/sial/320331?lang=en®ion=GR>)
TEOS 9.14mL (<http://www.sigmaaldrich.com/catalog/product/aldrich/333859?lang=en®ion=GR>)

Poly(ethylene glycol)-block-poly(propylene glycol)-block-poly (ethylene glycol)

HCL

To HCl density
=1.2gr/mL 500 L μ Sigma
m= $V=1.2*500=600$ gr

m= $V=20*1.2=24$ gr

TEOS

TEOS density
=0.933gr/mL 25 L μ Sigma
m= $V=0.933*25=23.325$ gr

m= $V=0.933*9.14=8.52762$ gr

