A Study on the Synthesis of MCM-41

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Abstract

The discovery of mesoporous material has great interest to many scientists. MCM-41 is a well-known mesoporous molecular sieve which includes alumina and silica. Material containing pores with diameters between 2 and 50 nm. MCM-41 widely used for catalysis, ion exchange, drug delivery, optics, gas sensing, and sorption. MCM-41 was prepared by combining with ultrasound treatment and high pressure autoclave hydrothermal reaction. Samples which are taken before and after calcination have been analyzed with X-ray powder diffraction, Fourier transform infrared spectroscopy and Scanning electron microscope.

Key words: MCM-41, FT-IR, TEOS, XRD, SEM.

1. Introduction

MCM-41 (Mobil Crystalline Materials No 41) was first discovered by researchers of Mobil Research and Development Corporation in 1992. MCM-41 is an ordered mesoporous hexagonal structure, which has diameters ranging from 2 to 10 nm. MCM-41 has a large internal surface area and favorable uniformity. Nevertheless it's easy to control the porous size of the material depends on that it becomes an advantage for material science and industrial applications [1]. Ordered mesoporous silica materials have great interest by many researchers. They are mostly attracting by the scientific community. Also, the chemical industry offers many promising approaches for new high-tech materials [2]. MCM-41 widely used for catalysis, ion exchange, drug delivery, optics, gas sensing , molecular sieving, filter membranes, laser media, and sorption[3-12].

There are kind of parameters which effect the properties of synthesized materials such as aging period, type and concentration of raw materials, synthesis method, ph and calcination [1].In an ordinary synthesis of MCM-41 an aqueous silica solution such as fumed silica, sodium silicate or tetraethyl orthosilicate is added to a fresh aqueous alkaline solution of a micelle-forming surfactant under steady stirring [13].

In this study, MCM-41 was synthesized by combining with ultrasound treatment and high pressure autoclave hydrothermal reaction. The obtained material was characterized before and after calcination by X-ray powder diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and Scanning electron microscope (SEM). The XRD pattern of calcined sample shows that highly ordered MCM-41 material was synthesized.

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2. Materials and Method

Tetraethyl orthosilicate (TEOS; C8H20O4Si, 99 wt%, Merck) as the silica source and hexadecyltrimethylammonium bromide, (HDTMA-Br; $CH_3(CH_2)15N(CH_3)_3Br$, 99 wt %, Merck) as the structure-directing agent were used as the reagents for the synthesis of MCM-41.
XRD patterns of samples were performed with a diffractometer (Philips PANalyticalX'Pert Pro) using Cu Kα radiation at 45 kV and 40 mA. FTIR analysis of samples was performed in the 4000-450 cm^{-1} region with a spectrometer (Perkin Elmer Spectrum One) at a resolution of 4 $cm-^{1}$. A fixed weight of each sample was mixed with KBr and then the pellet prepared before the FTIR analysis. The morphological properties of samples were investigated by SEM (CamScan APOLLO D-300).

2.1. Synthesis of MCM-41

In this study, MCM-41 was synthesized by two consecutive steps production processes. In the first step of synthesis, ultrasonic irradiation was used for the preparation of reaction solution. Firstly, HDTMA-Br was dissolved in deionized water under ultrasonic irradiation. After preparation of the solution sodium hydroxide and TEOS were added to final mixture quietly and allowed to stand ultrasonic irradiation. The pH of the mixture was adjusted to approximately 11 by addition of sulphuric acid. In the second step, the mixture was transferred into a Teflon-lined stainless steel autoclave to carry out hydrothermal treatment at 90°C for 24 h. After the cooling of product to room temperature, the obtained precipitate was washed with water and dried at 100°C overnight. This sample was denoted as as-synthesized MCM-41. The as-synthesized sample was calcined at 550°C for 6 hours to removing of structure-directing agent and characterized by XRD, FTIR, and SEM analyses. This sample was denoted as calcined MCM-41.

3. Results

The XRD pattern of as-synthesized and calcined MCM-41 was given in Figure 1. It was shown that MCM-41 sample obtained exhibited a very intense (100) diffraction peak and three weak (110), (200), and (210) peaks which are characteristic of long range ordered hexagonal MCM-41 mesoporous phase. After the calcination, the intensities of peaks increased significantly as a result of removal of organic phase. In addition, these peaks shifted to the higher angles.

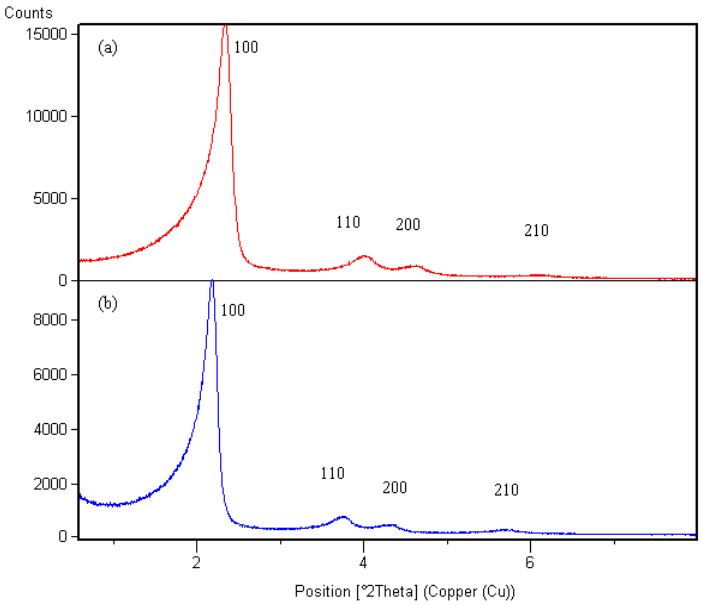


Figure 1.XRD pattern of obtained samples a) Calcined MCM-41 and b) As-synthesized MCM-41.

The FTIR spectra of MCM-41 before and after calcination are illustrated in Figure 2. It can be seen that the spectrum of MCM-41 a broad band around 2921 and 2851 cm^{-1} due to stretching vibrations of physically adsorbed water or structural -OH groups. Another peak at 1648 cm^{-1} can be assigned to OH bending vibrations of the adsorbed water molecules. The broad strong peak at 1064 cm^{-1} can be attributed to the asymmetric stretching of Si–O–Si groups. The band at 803 cm^{-1} can be attributed to the typical symmetric stretching modes of Si–O–Si [15]. The differences between of two spectrum were exhibited in the bands at 1488, 2851, and 2921 cm^{-1} can be attributed to the structure-directing agent. It was seen that these bands were disappeared after the calcination of the sample.

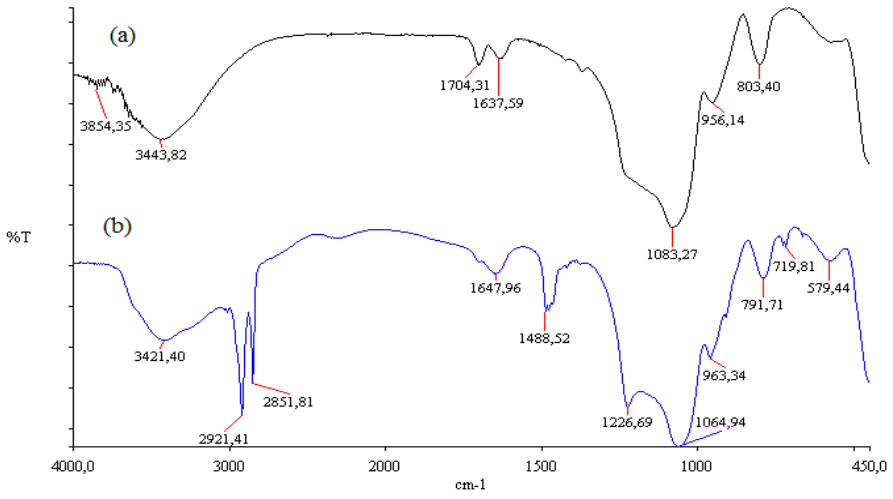
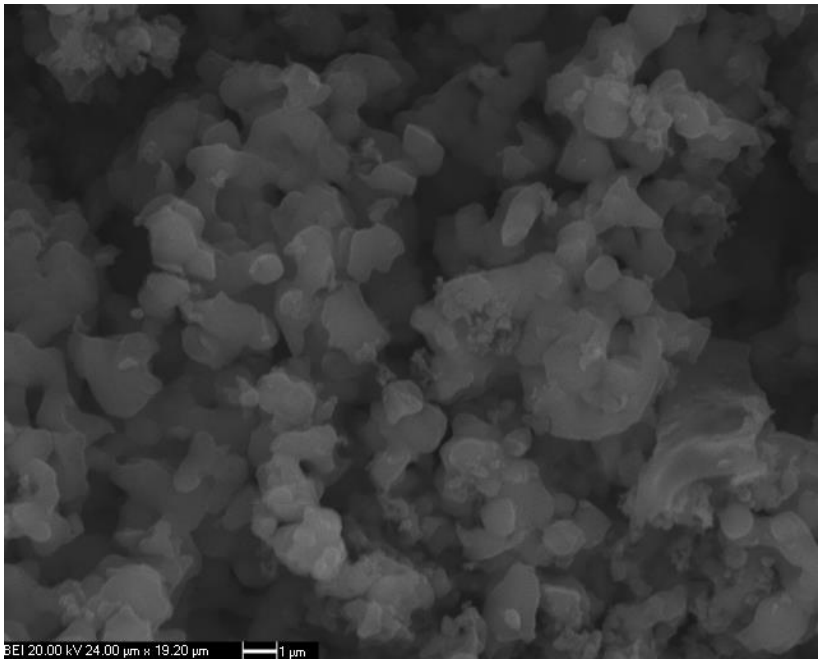
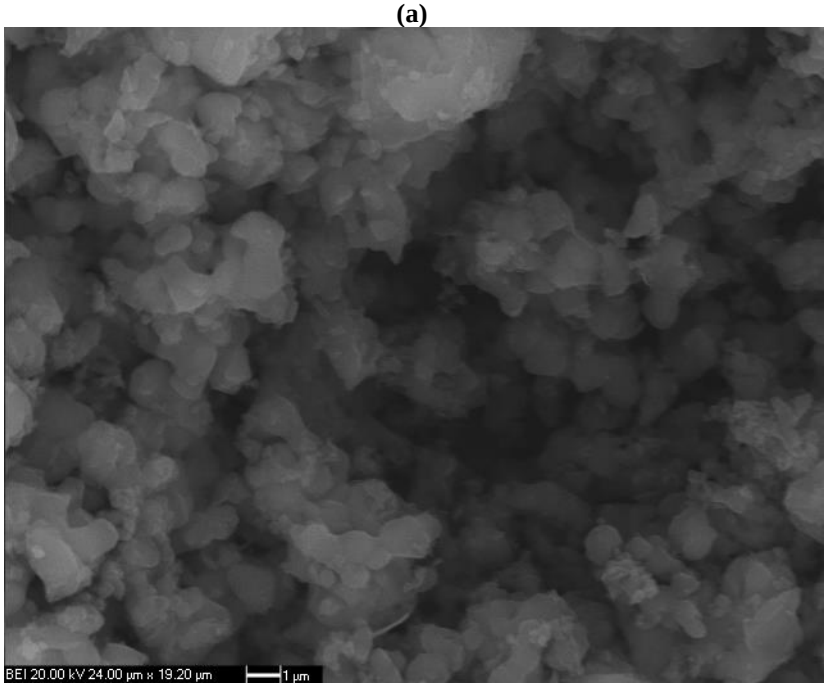


Figure 2. FT-IR pattern of obtained samples a) Calcined MCM-41 and b)As-synthesized MCM-41.

SEM micrographs of as-synthesized and calcined MCM-41 samples are demonstrated in Figs. 3 a and b, showing the morphology of the crystals. According to the SEM analysis, samples depict irregularly-shaped rods with an average length of 1.5 μm. Moreover, the particle size of sample was become smaller by calcination.



(a)



(b)

Figure 3. SEM image of samples a) As-synthesized MCM-41
b) Calcined MCM-41

4. Discussion

In this study, MCM-41 was synthesized by combining with ultrasound treatment and high pressure autoclave hydrothermal reaction. To our knowledge, limited research study has been published on synthesis of MCM-41 by combining production process.

Conclusions

In this study, the combination of ultrasound treatment and high pressure autoclave hydrothermal method were employed for synthesis of MCM-41. The chemical and morphological properties of synthesized materials were also investigated by XRD, FT-IR, and SEM analysis. According to analyses results MCM-41 was successfully synthesized. This study showed that using combine production method can be used as an alternative technique in the synthesis of MCM-41.

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