The Synthesis of MSU-X Mesoporous Materials from Sodium Silicate

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Abstract

MSU-x (MSU represents Michigan State University) materials are a family of ordered mesoporous silica obtained by using nonionic poly(ethylene oxide)-based surfactants [1]. MSU materials have many application areas due to their mesoporous structure, high mass oil cracking activity and high thermal stability [2]. In recent years MSU mesoporous materials were used in adsorption, catalytic applications, separation process, and drug delivery system [2-6].

In this study, MSU mesoporous silica materials were synthesized from sodium metasilicate pentahydrate (SMP) which is more cost-effective than the tetraethyl orthosilicate (TEOS) as a silica source and Pluronic 123 as a nonionic structure-directing triblock copolymer surfactant in an acidic medium. The synthesized materials were characterized by X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR) analyses. The obtained results showed that the MSU-x was successfully synthesized.

Keywords-component; Mesoporous, MSU-x, silicate, XRD, FT-IR.

1 Introduction

Porous materials are the composite of the solid phases and the pores formed in the solid phases. Main difference between porous materials and solid materials are the existence of pores. The features and structure of the pores determine the ways in which porous materials can be used in which process. The basic parameters for the porous materials are pore size, porosity, pore shape, and specific surface area [7]. Compared to dense solid materials, porous materials have large specific surface area, good energy adsorption, and low density [8].

Porous materials can be classified by different criteria such as pore shape, pore size, and production method. The international Union of Pure and Applied Chemistry (UIPAC) has recommended specific definition of porous materials due to their pore size: micropores (pore diameter < 2 nm), mesoporous (2nm < pore diameter < 50 nm], or macro porous (50 nm < pore diameter) [9]. Cheremskoj also has another classification pore types such as macropores (Pore diameter > 1000nm), micropores (100 < porediameter < 1000nm), submicropores (pore diameter < 100nm), and ultramicropores (pore diameter < 0.5 nm) [10]. Due to the special properties of porous materials it has many applications ranging from catalysis, adsorption, sensing, separation, biotechnology, and fluid distributors [8, 11, 12].

Mesoporous molecular sieves have attracted much attention in scientific research projects and for practical applications. Recently, one of the popular topics is mesoporous materials which include Santa Barbara Amorphous (SBA-15), Mobil Composition of Matter No. 41 (MCM-41), MSU-x and hexagonal mesoporous silica (HMS). Among them, MSU type materials which is a kind of the ordered silica based mesoporous material synthesized from nonionic poly(ethylene oxide)-based surfactants. Due to the mesoporous structure, high mass oil cracking activity and high thermal stability of MSU materials, they have many application areas such as adsorption, separation, drug delivery system, and catalysis.

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In the present study MSU-x mesoporous materials was synthesized from sodium metasilicate solution as a pure silica source. In addition, the synthesized material was characterized using XRD and FTIR analysis.

2 Materials and Methods

In the present study, sodium metasilicate ($Na_2SiO_3 \cdot 9H_2O$, Sigma Aldrich >97%) was used as a pure silica source. Pluronic Triblock copolymer, $EO_{20}PO_{70}EO_{20}$, (P123) was used as a template that supplied from Sigma Aldrich. The other chemicals used in the synthesis of MSU-x materials were hydrochloric acid (Merck, 37%) and sodium floride (Merck, >99.5%).

An infrared spectrum of the synthesized sample was obtained using a Perkin Elmer Spectrum One Fourier-transform infrared (FTIR) spectrometer using dry KBr pellet technique in the range of 4000– 450 cm^{-1} (Fig. 1). The synthesized sample was mixed with KBr in the weight ratio of 1:100 in order to prepare the pellet [13].

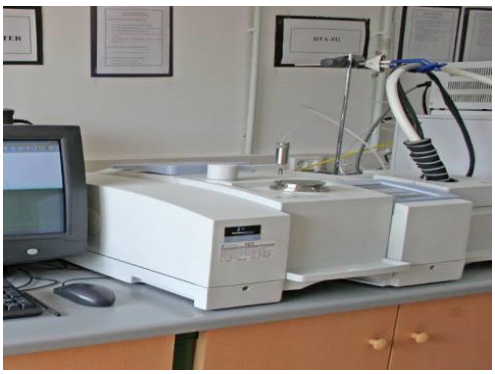

Figure 1. Image of the FT-IR equipment

The small-angle XRD patterns of the sample was recorded on a Philips Panalytical X'Pert-Pro X-ray diffractometer, using Cu K◎ radiation (◎ = 1.540 A°) in 45 kV and 40 mA (Fig. 2).

Figure 2. Image of the XRD device

2.1 Synthesis of MSU-X

The synthesis procedure of the MSU-x material consists of three steps. In the first step of study, a given amount of P123 was dissolved in distillated water. Then, pH of the obtained mixture was adjusted to 2 by addition of 37% of aqueous hydrochloric acid (HCl) at the room temperature. In the second step, a given amount of sodium metasilicate was dissolved in the distillated water and the pH of the solution was adjusted to 2 with 37% of aqueous HCl. After the both solutions became clear, sodium metasilicate solution was added to the P123 solution drop by drop and then the mixture was stirred for 1 hour at 50°C. In the final step, a given amount of sodium fluoride was added the obtained solution for the condensation. After aging for 72 hours at 50°C, the resulted product was filtered, dried in an oven, and calcined in air under static conditions at 550°C for 6 hours. The image of the resulted product was given in Fig. 3 and synthesis scheme of MSU-X was shown in Fig. 4.

Figure 3. Synthesized MSU-X

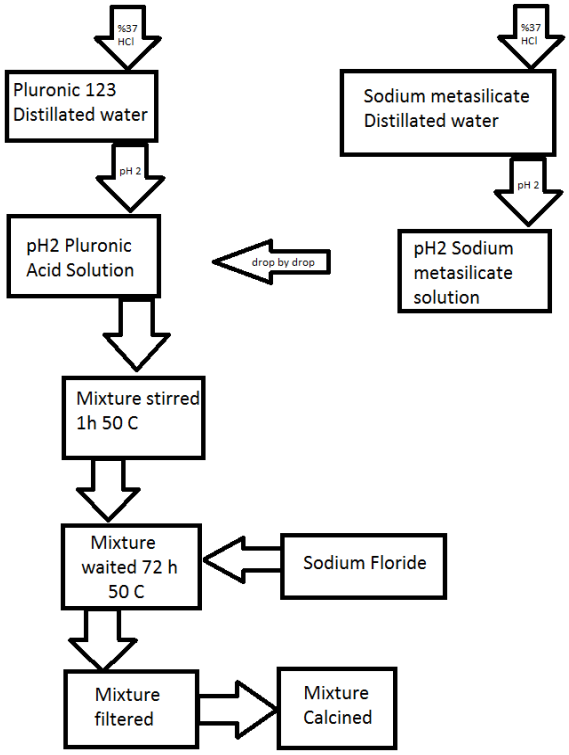

Figure 4. Synthesis scheme of MSU-X

I. Results And Discussion

The obtained FTIR absorption bands were given in Fig. 5. The main features of the MSU-x spectra included a broad band around 3445 cm^{-1} due to stretching vibrations of physically adsorbed water or structural –OH groups. Another peak at 1653 cm^{-1} can be assigned to OH bending vibrations of the adsorbed water molecules. The broad strong peak at 1090 cm^{-1} can be attributed to the asymmetric stretching of Si–O–Si groups. The band at 799 cm^{-1} can be attributed to the typical symmetric stretching modes of Si–O–Si. The bands around at 799 and 465 cm^{-1} indicate Si-OH vibrations generated by the presence of defect sites and Si-O-Si bending mode [14-15].

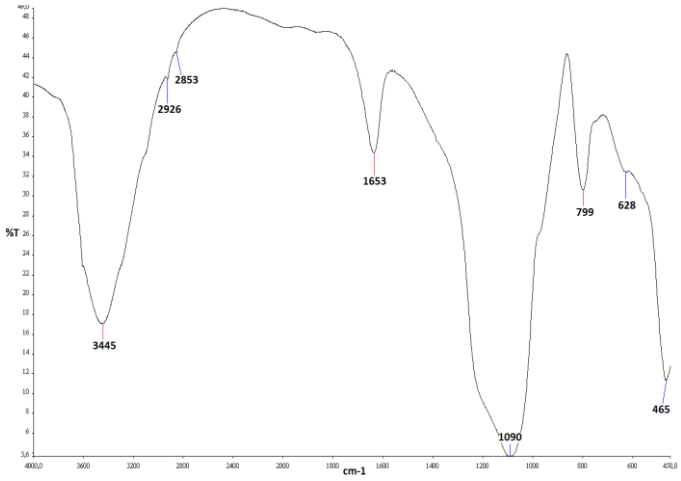

Figure 5. FT-IR spectrum of MSU-X

The small angle XRD pattern of the synthesized sample was given in Fig. 6. It was seen that sample demonstrated single d_{100} diffraction peak at the range of 0-1°, which was similar to the MSU-type materials [16].

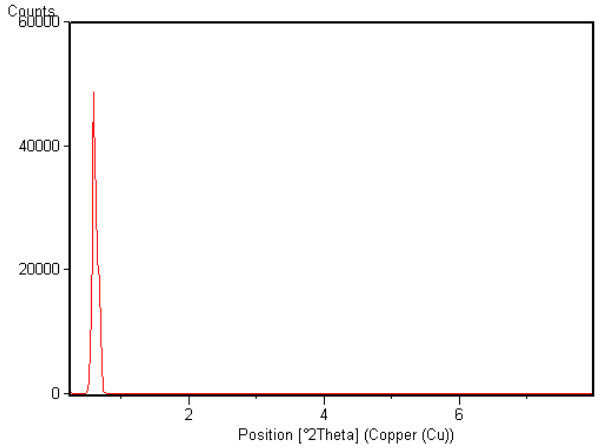

Figure 6. XRD pattern of synthesized MSU-x

3 Conclusion

In this study, MSU-x mesoporous silica materials was successfully synthesized from sodium metasilicate solution. The crystal structure of the synthesized sample was determined by XRD. It was observed that XRD pattern of the sample similar to the MSU-type materials. In addition, the chemical structure of the sample was investigated by FT-IR spectroscopy.

4 References

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