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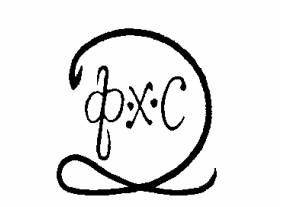

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THE INFLUENCE OF HYDROCHLORIC ACID ON THE FEATURES OF SBA-15 PARTICLES

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ABSTRACT
The template method synthesis of both SBA-15 materials with elongated and spherical particles was performed using a surfactant Pluronic P_{123}. The HCl (p.a.) was used to synthesize material with elongated particles connected in chain structures grouped into shapes resembling sheaves of wheat. In the synthesis of spherical SBA-15 with diameters ranging from 0.5 to 2 μm, a spent HCl solution which was obtained after chemical treatment of clay was used where the dominant presence of the spheres was confirmed by the SEM method. In addition to the methods mentioned above, XRD, EDS and FTIR methods characterize SBA-15 materials.

INTRODUCTION
The silicates have attracted a great deal of interest in the past decades because of their use in catalysis, separations, sensors, drug delivery, and optical devices. Many efforts have been devoted to the synthesis of silica spheres of defined size and pore diameter because the control of the particle morphology and pore size of mesoporous silica could open up new possibilities for its application as packing material in chromatography or as an easy-to-handle form for catalytic purposes [1-3]. Different morphologies of the SBA-15, such as fibres, platelets, spheres, monoliths, films, etc., can be synthesized by varying the reaction conditions during synthesis [4]. Silica spheres were obtained via a two-step synthesis process by using a triblock copolymer Pluronic P_{123} as a template in combination with an HCl solution used after use in the chemical treatment of clay.

METHODS
Both samples of SBA-15 were synthesized according to the standard procedure [5] by using Pluronic P_{123} (non-ionic triblock copolymer, $EO_{20}PO_{70}O_{20}$, BASF) as a surfactant and tetraethoxysilane (TEOS, 98%) as a source of silica. A 4.0 g sample of Pluronic P_{123} was dissolved in 30 ml of distilled water and 120 g of 2M HCl solution and stirred at 35 °C for 1.5 h. 8.5 g of TEOS were added dropwise into the solution and vigorously stirred at the same temperature for 1.5 h. According to the proposed method [5], the mixture was aged at 35 ºC for 20 h and then at 80 °C for 48 h. The final products were filtered, washed with 600 ml of distilled water, and dried at room temperature. Calcination was carried out in flowing air by slowly increasing the temperature from room temperature to 500 °C for 8 h and keeping it at 500 °C for 6 h to decompose triblock copolymer. In the synthesis of SBA-15 with elongated particles (SBA-15/E), HCl (p.a.) was used. In the synthesis of SBA-15 with spherical particles (SBA-15/S), a spent HCl solution after chemical treatment of clay was used (spent HCl solution) [6]. Methods SEM, XRD, EDS and FTIR were employed to characterize the phases, functional groups and microstructure of the obtained samples are described elsewhere [4,7,8].

RESULTS AND DISCUSSION

The SEM micrographs of the SBA-15 materials are shown in Figure 1.

The material SBA-15/E consists of many elongated particles of relatively uniformed sizes (up to 1μm). These elongated particles are aggregated into wheat like structures. Similar chain agglomerate structures were reported in the literature [5]. The spherical particles of (SBA-15/S), with diameters ranging approximately from 0.5 to 2 μm, are presented in Figure 1. The form and the size of the grains depend on the form and dimensions of the micelle, which was formed from the surface-active substance as a template [9]. Earlier researche presented that Pluronic P_{123} has never formed spheres only due to powerful hydrophobic forces that lead to the formation of elongated cylindrical silicate-surfactant micelles that are aggregated into wheat-like structures [10]. According to the literature data, various ionic species in the spent HCl solution could promote the formation of spherical particles [11]. Spheric SBA-15 is synthesized using the ionic surfactant cetyltrimethylammonium bromide (CTAB) under acidic condition [2]. Instead of using surfactant CTAB, we have demonstrated that sphere SBA-15/S can be obtained using a spent HCl solution.

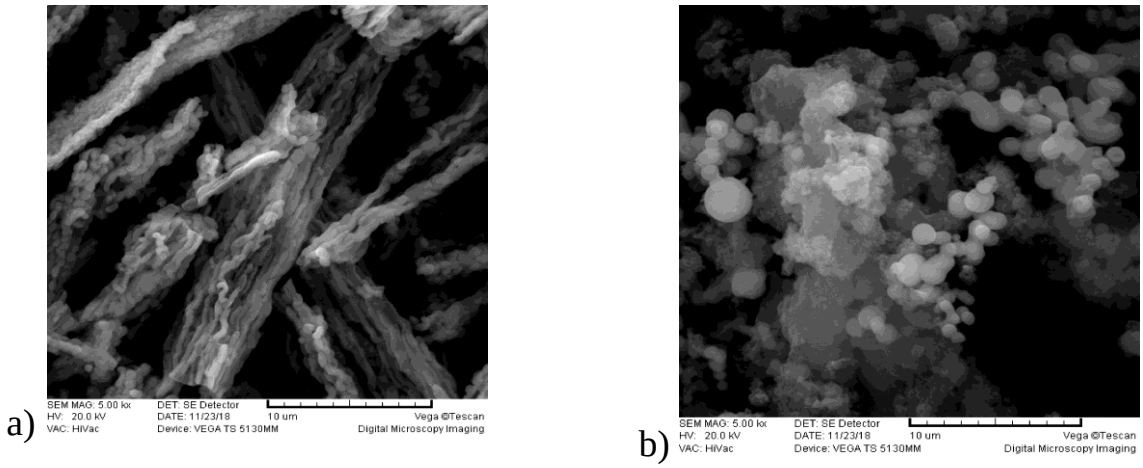

Figure 1. SEM micrographs of: a) SBA-15/E and b) SBA-15/S.

The EDS analysis showed that the particles of both test materials consist of SiO_2. The XRD analysis confirmed the presence of amorphous SiO_2 in the investigated samples. For SBA-15/E, the EDS analysis and diffractogram are presented elsewhere [4]. Figure 2. presents the FT-IR spectrums of SBA-15 with elongated and spherical particles. The FT-IR spectrums of both investigated SBA-15 materials are very similar. Bands at 1054 and 797 cm^{-1} belong to asymmetric and symmetric stretching vibrations of the Si-O-Si framework, respectively. The weak absorption, which peaks at 557 and 441 cm^{-1} could possibly be attributed to Si-O deformation. A weak band at 956 cm^{-1} represent Si-OH vibration [12–15].

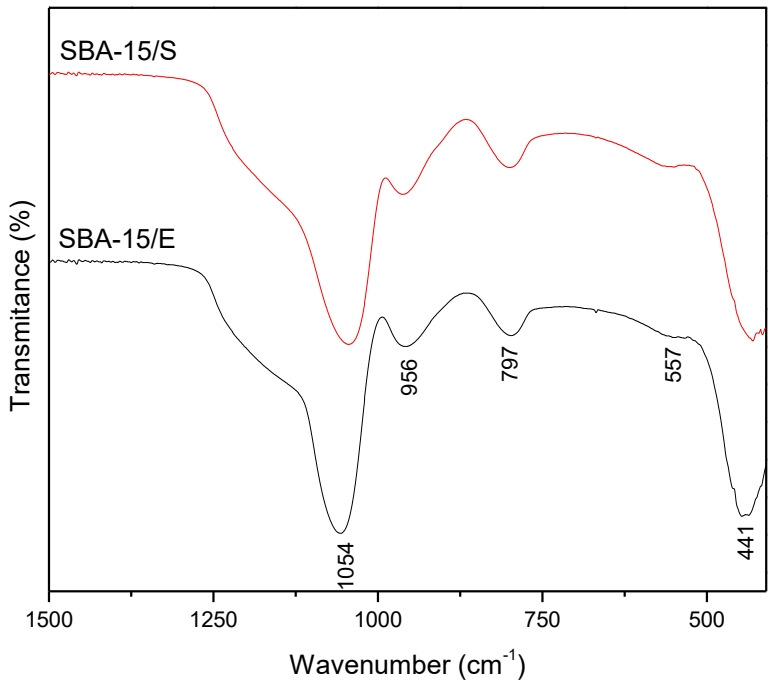

Figure 2. FT-IR spectra of: SBA-15/E and SBA-15/S.

CONCLUSION

The SBA-15 spheres were successfully synthesized. Investigations in this paper show that the differences in the structures of synthesized SBA-15 materials closely depend on the origin of reactants used in the synthesis of SBA-15. The spent HCl solution after use in chemical treatment of clay was used to synthesize SBA-15 spheres. The synthesis of spherical particles with diameters ranging approximately from 0.5 to 2 μm was promoted by various ionic species from spent HCl solution instead of using a commercial ionic surfactant.

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