SYNTHESIS OF MCM-41 FROM FLY ASH

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INTRODUCTION

MCM-41 is a mesoporous material with a hierarchical structure from a family of silicate and alumosilicate solids [1,2]. It consists of a regular arrangement of cylindrical mesopores that form a one-dimensional pore system. It is characterized by an independently adjustable pore diameter, a sharp pore distribution, a large surface and a large pore volume. MCM-41,is widely used as catalytic cracking and also separations [3].

GOAL

The aim of these studies was to obtain mesoporous materials from fly ash, which is waste from the energy sector.

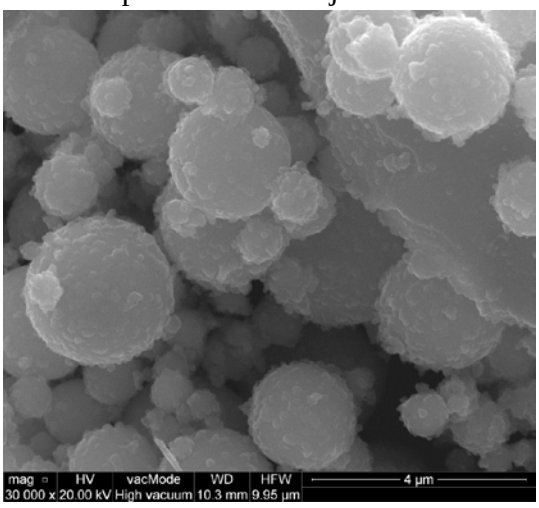
EXPERIMENTS

The starting material for the synthesis of MCM-41 wasfly ash from the Polish heat and power plant. The other ingredients were: CTAB - surfactant, NaOH, methyl acetate and distilled water. The chemical composition of used fly ash was determined using the XRF method. SEM analysis was performed to determine the porous structure and morphology. A point analysis of the chemical composition of grains - EDS was added to the study. Preparation of MCM-41 sieve from fly ash included two stages: extraction of Si and Al from fly ash, and proper synthesis based on the obtained filtrate. Two methods have been used, in order to extract silicon and aluminum from fly ash:

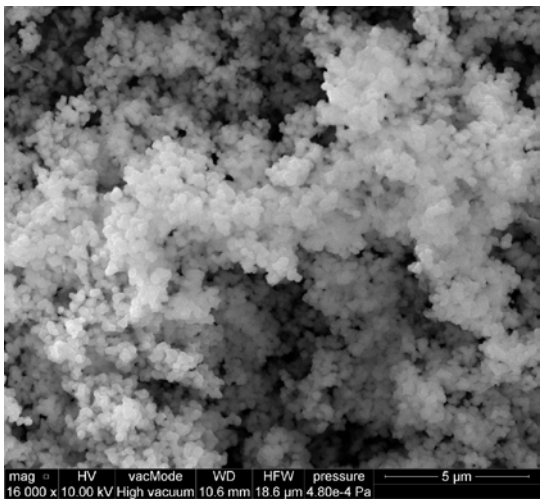
- the solution that remained from the synthesis of zeolites in the hydrothermal reaction

- fusion of fly ash with sodium hydroxide following with dissolution of the solid formed, in water

The obtained filtrate was placed in an oven, surfactant and methyl acetate were added. The resulting material was transferred to a mechanical stirrer and mixed, and then cooled to room temperature. Next the pHmeter was adjusted and material was mixed.



SEM photomicrograph of the fly ash used



SEM photomicrograph of the obtained MCM-41

RESULTS

As a result of the synthesis, four samples were obtained. The resulting products were subjected to XRD analysis. First samples (S1, S2) which were synthesized from the filtrate remained after fusion of fly ash with NaOH did not show mesoporous properties. However, it was achieved with remaining two samples (S3, S4) obtained on the basis of synthesis of zeolites in a hydrothermal reaction. For further description, SEM analysis and low-temperature nitrogen adsorption were performed. The characteristics of the pores were determined based on the DFT and BJH methods. After obtaining the results it was found that both the pore diameter and their volume are located near the border of the range of microporous materials with mesoporous ones. Therefore, it was decided to carry out the test, using the Dubinin-Raduszkiewicz method (Table 1).

Table 1. Selected properties of samples

Parameter	S3	S4
Dominant pore diameter (DFT) [nm]	2,703	2,583
Dominant pore diameter (BJH) [nm]	2,001	1,999
Dominant pore volume (DFT) [cm^3/g]	0,405	0,356
Dominant pore volume (BJH) [cm^3/g]	0,311	0,279
S_{BET} [m^2/g]	864,85	756,384
S_{DR} [m^2/g]	614,42	569,088
S_{DFT} [m^2/g]	670,236	610,514
S_{BJH} [m^2/g]	251,74	139,365

CONCLUSION

The difference in the way the materials were obtained undoubtedly influenced the result. The concentration of aluminum and silicon was not analyzed in the solutions used for synthesis, therefore it is not possible to state whether the reason for not receiving the expected products from filtrates after fusion is the result of low Si and Al concentrations or their inappropriate ratio.

SUMMARY

It might have seemed that the ash fusion with NaOH will allow higher concentrations of silicon and aluminum to be obtained in the filtrate compared to the hydrothermal solution solution. However upon the conducted analyzes, the presence of MCM-41 type material was found for the synthesis of which the filtrate was based on the hydrothermal reaction of obtaining zeolites. No MCM-41 structures were found for samples obtained from fused solutions.

REFERENCES

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