SYNTHESIS, CHARACTERIZATION AND APPLICATION OF Al-SBA-15 CATALYST

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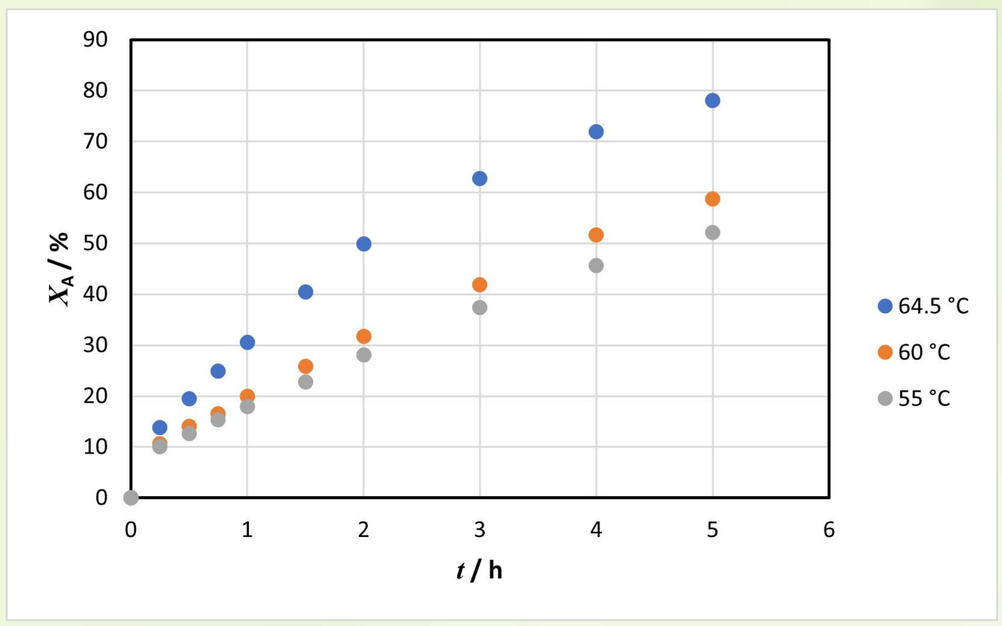
Abstract

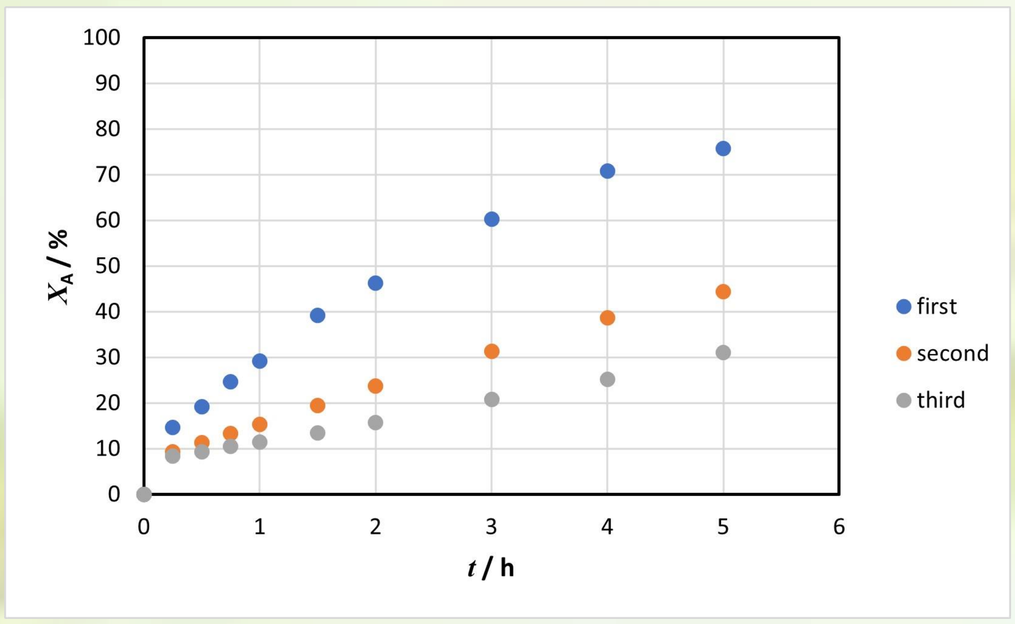
The focus of this study was to synthesize a solid acid catalyst based on mesoporous silica SBA-15 with incorporated metal Al. The synthesized catalyst was characterized by nitrogen adsorption-desorption (BET), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), thermogravimetric analysis (TGA). Furthermore, using this catalyst, a series of oleic acid esterification reactions in methanol were carried out with the production of oleic acid methyl ester. All experiments were performed in a laboratory batch reactor. The influence of reaction temperature (50, 55, 60, and 64.5) °C and catalyst mass (0.025, 0.05, 0.1, 0.2 and 0.3 g) on the reaction conversion were studied. The reaction efficiency was monitored by determining the concentration of oleic acid and methyl oleate using GC-FID. We also checked the loss of catalyst activity after repeated use. The results show that the conversion increases with increasing temperature and mass of the catalyst up to 0.2 g. Further mass increase didn't result in increased conversion. The study was upgraded by determining the kinetic parameters; thus, the Langmuir-Hinshelwood-Hougen-Watson model was employed.

Introduction

The growing demand for safe industrial processes has led to the development and use of environmentally friendly solid catalysts intended for use in acid-catalyzed value-added reactions. Acid catalysis is one of the most common processes in the chemical industry. Industrially important organic transformations include esterification and transesterification, etherification, dehydration, oxidation, acetylation, silylation and biodiesel synthesis [1,2]. Solid acid catalysts, known as "green catalysts", are used as a substitute for homogeneous acid catalysts. They contain more environmentally friendly components, while providing greater activity and selectivity compared to existing homogeneous catalysts [3]. An example of a heterogeneous catalyst is mesoporous silica - modified SBA-15, which is used in many organic reactions.



Conversion regarding time at different temperatures, f_m = 600 min^{-1}, m_{cat} = 0.025 g



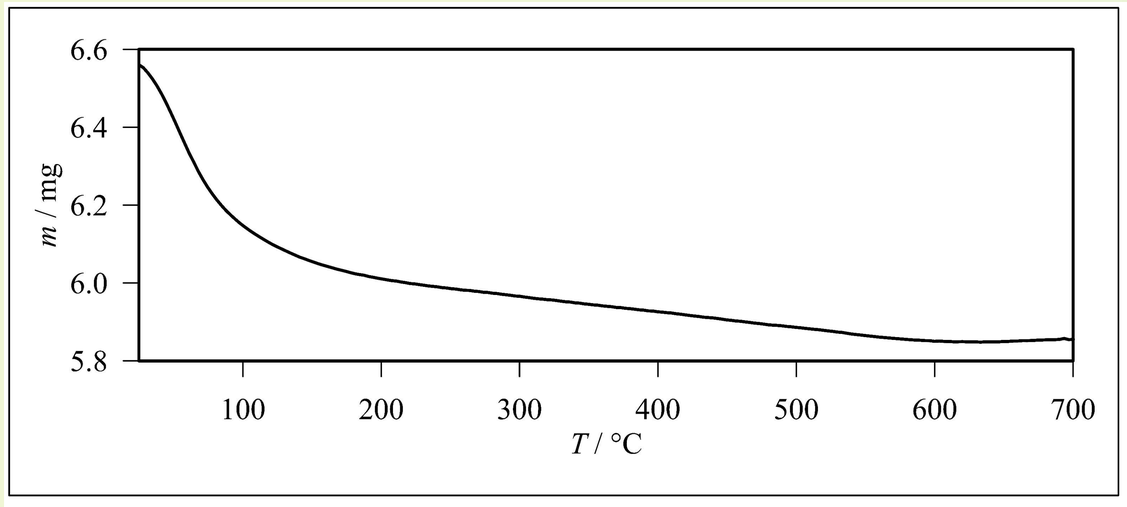
Conversion regarding time for three consecutive reactions, ϑ = 55 °C, f_m = 600 min^{-1}, m_{cat} = 0.1 g

Experimental conditions:

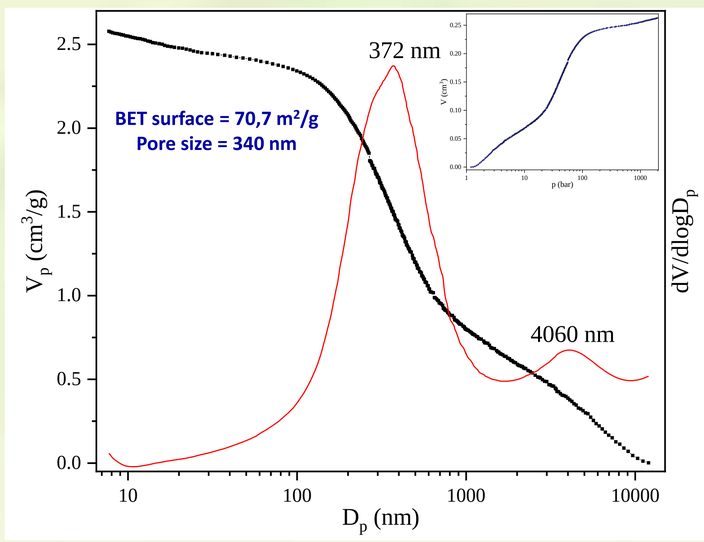
T = (55, 60, 64.5) °C

f_m = 600 min^{-1}

m_{cat} = (0.025, 0.05, 0.1, 0.2 and 0.3) g



TGA thermogram



Results of BET analyse

Langmuir-Hinshelwood-Hougen-Watson model:

$$\frac{dX_A}{dt} = k\, c_{A0}\,(1 - X_A)(M - X_A)$$

Aktivation energies at different mass of the catalyst

m_{cat} / g	E_a / kJ/mol
0.025	73.5
0.1	58.0
0.2	21.8
0.3	10.0

Conclusion

We synthesized a solid acid catalyst based on mesoporous silica SBA-15 with incorporated metal Al. The catalyst has a high catalytic activity in the esterification reaction of oleic acid with methanol. After the reaction is complete, it is easily separated from the reaction medium. When the catalyst is reused, its activity is slightly reduced. The kinetic parameters of the reaction were determined. The activation energy decreases with increasing mass of the catalyst.

Acknowledgement

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References

[1] Léon CIS, Song D, Su F, An S, Liu H, Gao J, et al., (2015), Propylsulfonic acid and methyl bifunctionalized TiSBA-15 silica as an efficient heterogeneous acid catalyst for esterification and transesterification, Microporous and Mesoporous Materials, 204, 218–25.

[2] Dhainaut J, Dacquin J-P, Lee AF, Wilson K., (2010), Hierarchical macroporous–mesoporous SBA-15 sulfonic acid catalysts for biodiesel synthesis, Green Chem., 12, 296–303.

[3] Zheng Y, Li J, Zhao N, Wei W, Sun Y., (2006), One-pot synthesis of mesostructured AlSBA-15-SO3H effective catalysts for the esterification of salicylic acid with dimethyl carbonate, Microporous and Mesoporous Materials, 92, 195–200.

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