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## KINETICS OF ACETIC ACID – METHANOL ESTERIFICATION REACTION OVER SULFONIC ACID-FUNCTIONALIZED MCF CATALYST

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### ABSTRACT

The esterification of acetic acid with methanol was studied in the presence of heterogeneous acidic catalyst - sulfonic acid functionalized silica mesoporous cellular foam (MCF) in order to determine its potentials. The effects of catalyst loading, molar ratio of substrates and temperature on the reaction rate were discussed. The kinetic model for this reaction was proposed.

Przedstawiono wyniki badań kinetycznych reakcji estryfikacji kwasu octowego metanolem w obecności stałego kwasowego katalizatora – mezoporowatego materiału krzemionkowego o strukturze piankowej, funkcjonalizowanego grupami sulfonowymi. Określono wpływ ilości katalizatora, stosunku molowego substratów oraz temperatury na szybkość reakcji. Zaproponowano model kinetyczny reakcji przeprowadzonej w obecności otrzymanego katalizatora.

### 1. INTRODUCTION

Although traditionally in the industrial processes acids are esterified with alcohols in the presence of strong homogeneous catalyst such as *p*-toluenesulfonic or sulphuric acid, the usage of heterogeneous catalyst is more advantageous from environmental and economic point of view. Minimalization of the amount of wastes, easier separation and recovery of catalyst from the reaction medium, reducing equipment corrosion are the main benefits of replacing the homogeneous catalysts by the solid ones. A numerous of active solid acidic catalysts are proposed in the literature. Among them: ion-exchange resins [1,2,3], zeolites [3,4,5],

supported heteropolyacids [6] or advanced porous materials with incorporated acidic groups [7,8,9]. Considering a huge number of different catalysts features it seems to be very likely that some of them are more favorable for selected applications than the others. It is clear, that all of them have their own drawbacks and recommendations: the applicability of zeolites is reduced due to their small surface area and pores diameters and interrelated diffusional effects; the usage of the acidic ion-exchange resins (Amberlyst, Nafion), is limited by their low thermal stability; supported heteropolyacids are very sensitive to water and they can be easily deactivated. Therefore acidic acid functionalized mesoporous materials seem to be good alternative for above mentioned catalysts. Recently, influence of the carrier structure on the reaction rate have been proved. [7,10,11]. In our previous study the activity of catalysts of various mesoporous structures (SBA-15, MCF, MN Kieselgel 60), in the esterification reaction of acetic acid with butanol, have been compared. Among various materials studied, those of mesostructured cellular foams appeared to be particularly attractive for liquid phase applications. They are characterized by open structure, large specific surface area – up to  $1000 \text{ m}^2 \cdot \text{g}^{-1}$  and their 3D cage-like spherical pores in the range of 24-42 nm in diameter which are interconnected by windows with diameters up to 22 nm [12, 13]. These values are notably larger than in other materials. Due to these features MCFs proved to be excellent candidates for the use as catalyst/biocatalyst supports [14-20]. Following our previous promising results [11, 21], herein we present a kinetic portrayal of the esterification of acetic acid with methanol using a sulfonic acid-functionalized silica MCF catalyst.

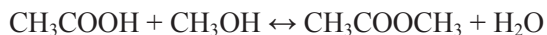
## 2. EXPERIMENTAL

### 2.1. CATALYST PREPARATION

Mesostructured cellular silica foams were synthesized according to the procedure described elsewhere [12, 13]. Detailed description of MCFs functionalization was given in [21]. Shortly, MCF was prepared from tetraethoxysilane (TEOS) using microemulsion templating method. Pure silica was grafted with organosulfonic acid precursor - 2-(4-chlorosulfonylphenyl)ethyltrimethoxysilane (CSPTMS), in the amount of 1 mmol/g. Additionally, methyl groups ( $1 \text{ mmol} \cdot \text{g}^{-1}$ ) were introduced to increase the hydrophobicity of the catalysts surface. The precursor of the methyl groups was chlorotrimethylsilane.

### 2.2. CATALYTIC EXPERIMENTS

The catalyst was tested in esterification reaction of acetic acid with methanol:



Methanol and acetic acid, (all pure p.a.) for catalytic experiments were obtained from POCh, Gliwice, Poland. The experiments were performed in a three-necked glass

flask with a volume of 75 ml equipped with Teflon-coated magnetic stirrer, thermometer, tube for sample withdrawal and water cooled condenser to avoid loss of chemicals. The temperature of the reacting mixture was measured with accuracy of  $\pm 0.1$  K. The reaction was carried out for 6 hours. After reaction the catalyst was separated from the reaction mixture by filtration. A gas chromatograph HP 5890 Series II with a TCD detector was used to analyze reaction mixture composition.

At the beginning, acetic acid and methanol were heated to desired temperature and after that the warm substrates were loaded into the reactor. Finally, 1 wt % of the catalyst (pre-dried at 473 K for 2 hours) was added. The moment of catalyst adding was taken as the beginning of the reaction. The conversion was calculated on the basis of acetic acid content in the reaction mixture.

### 3. RESULTS AND DISCUSSION

#### 3.1. SAMPLE CHARACTERIZATION

Detailed sample characterization was already presented in [21], but the most important textural properties are given below. The nitrogen adsorption isotherm was of type IV according to IUPAC classification and it showed step hysteresis of type H1 located in the relative pressure range of 0.7–1.0 which is typical for large-pore mesoporous solids. The surface area of the synthesized catalyst was  $260 \text{ m}^2 \cdot \text{g}^{-1}$ , while the surface area of silica support was  $640 \text{ m}^2 \cdot \text{g}^{-1}$ . The decrease in its value was due to the surface coverage by functional groups. However, the shape of the isotherm did not change. Pore size distribution was determined using the BJH model. The pore diameters of spherical pores were taken off from the adsorption curve while the diameters of connecting windows from the desorption curve. The average diameter of spherical cages was ca. 29 nm and the average diameter of windows was ca. 14 nm.

The thermal stability of the functionalized material was determined by thermogravimetric analysis (TGA). The decomposition curve revealed good thermal stability of the catalyst, up to 623 K, which is very attractive from the catalytic point of view. It is noteworthy that a typical polymeric catalyst – Amberlyst15 remains stable only up to 393 K.

Previous experiments also showed good catalytic stability of the catalyst in repeated esterification cycles [21]. After 6 cycles activity decreased about 10%, while the texture was still well preserved.

#### 3.2. CATALYTIC EXPERIMENTS

##### 3.2.1 EFFECTS OF MASS TRANSFER RESISTANCES

In order to determine and eliminate the effect of the external mass transfer constraints, the initial experiments were carried out with a stirring of 200, 500 and

1000 rpm. It was found that a stirrer speed of about 500 rpm was enough to keep all the catalyst particles in suspension and that the stirrer speed did not affect the rate of reaction above this speed. The lack of external mass transfer limitations above 500 rpm was observed. It was due to the extremely low grain size (about 10  $\mu\text{m}$ ) of the catalyst particles and hence very large external surface area. Thus, all further experiments were carried out with the speed of 1000 rpm to ensure that measured reaction rate was free from external diffusion.

To examine the influence of internal diffusion on the rate of the esterification calculations of the Thiele modulus  $\Phi$  were performed.

The Thiele modulus defined as [22]:

$$\phi = R_0 \sqrt{\frac{r_K}{D_{ef} \cdot c_K}} \quad (1)$$

calculated for external conditions of the temperature 343 K and the reaction rate  $1.74 \text{ mol} \cdot \text{m}^{-3} \cdot \text{s}^{-1}$  was  $\Phi = 5.49 \cdot 10^{-5}$ . This value indicates that diffusional constraints of the reacting species inside the pores of the catalyst can be neglected.

### 3.2.2 EFFECT OF THE CATALYST LOADING

The influence of the catalyst concentration on the acetic acid conversion was investigated for 0.5, 1 and 2 wt % of the catalyst (relative to substrates) and molar ratio of acid : alcohol equal to 1:1. The reaction was carried out at 323 K for 6 hours. The increase in acetic acid conversion is illustrated in Figure 1. The higher the catalyst loading the faster the reaction rate because of the increase in total number of available active sites for reactants.

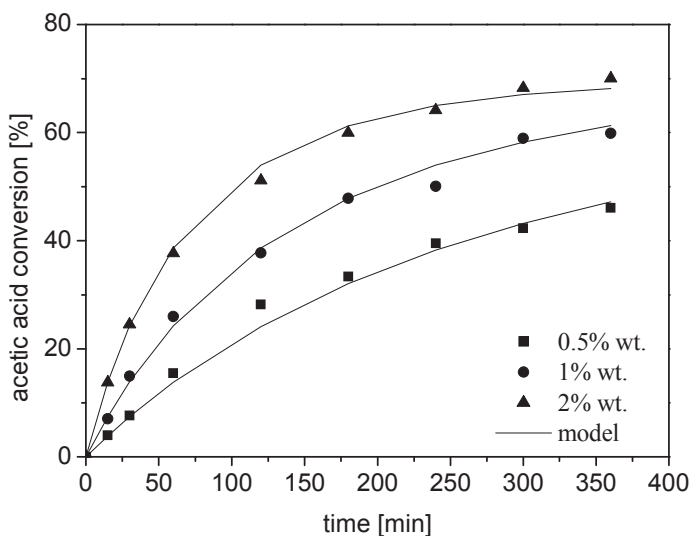


Fig. 1. Effect of catalyst loading on acetic acid conversion. Temperature 323 K, acetic acid : methanol 1:1, stirrer speed 1000 rpm

Rys. 1. Wpływ ilości katalizatora na konwersję kwasu octowego. Warunki reakcji: temperatura 323 K, kwas octowy : metanol 1:1, szybkość mieszania 1000 obr/min

### 3.2.3 EFFECT OF THE SUBSTRATES MOLAR RATIO

The influence of the molar ratio of the reactants on the conversion of acetic acid was studied for molar ratios of acetic acid to methanol equal to 1:1, 1:3, 1:5 at 323 K and catalyst loading of 1 wt %. The results are shown in Figure 2. The conversion of acetic acid (after 360 min.) increased from 60% to 85% with the rise in methanol excess (molar ratio) from 1:1 to 1:5.

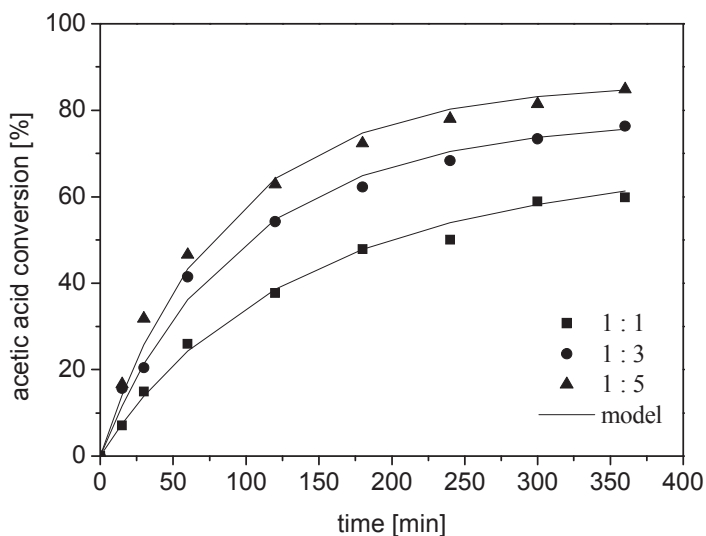


Fig. 2. Effect of acetic acid to methanol molar ratio on acetic acid conversion. Temperature 323 K, catalyst loading 1 wt %, stirrer speed 1000 rpm

Rys. 2. Wpływ stosunku molowego substratów na konwersję kwasu octowego. Warunki reakcji: temperatura 323 K, ilość katalizatora: 1%, szybkość mieszania 1000 obr/min

### 3.2.4 EFFECT OF REACTION TEMPERATURE

Figure 3 illustrates the effect of reaction temperature on the course of esterification. Experiments were carried out in the temperature range of 313–343 K while keeping the acid: alcohol molar ratio at 1:1 and 1 wt % catalyst loading. With an increase in reaction temperature the conversion of the acetic acid was found to increase substantially. It shows that the higher temperature yields the greater conversion of acetic acid at a fixed contact time.

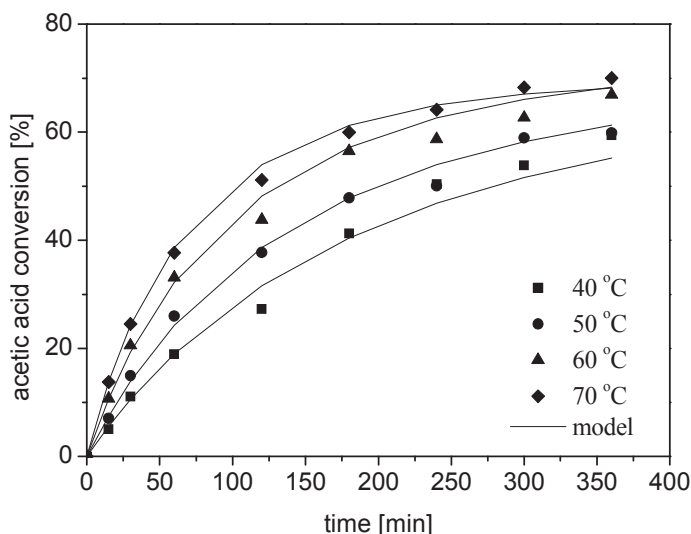


Fig. 3. Effect of reaction temperature on acetic acid conversion. Acetic acid : methanol 1:1, catalyst loading 1 wt. %, stirrer speed 1000 rpm

Rys. 3. Wpływ temperatury reakcji na konwersję kwasu octowego. Warunki reakcji: kwas octowy : metanol 1:1, ilość katalizatora: 1%, szybkość mieszania 1000 obr/min

### 3.3. KINETIC EQUATION

Esterification reactions are known to be reversible reactions of second-order. For heterogeneously catalyzed reaction, the Langmuir-Hinshelwood, Eley-Rideal or pseudo-homogeneous models were proposed [23-28]. The pseudo-homogenous model was chosen with respect to the form of the catalyst (particles of diameters ca. 10  $\mu\text{m}$ ). According to general kinetic expression for the second-order reversible reaction, the reaction rate of esterification may be written as [23, 24]:

$$r = -\frac{dc_A}{dt} = k_1 \cdot (c_K \cdot c_A - \frac{c_E \cdot c_W}{K}) \quad (2)$$

The equilibrium constant  $K$  was determined as:

$$K = \frac{k_1}{k_{-1}} = \frac{c_E \cdot c_W}{c_K \cdot c_A} \quad (3)$$

and its value was calculated for each reaction temperature based on the experimental molar ratio of the reactants at equilibrium. The equilibrium constant was found to be weakly dependent of the temperature (Figure 4). It corresponds to low value of the heat of reaction ( $\Delta H = -2.27 \text{ kJ}\cdot\text{mol}^{-1}$ ) during esterification of acetic acid with methanol.

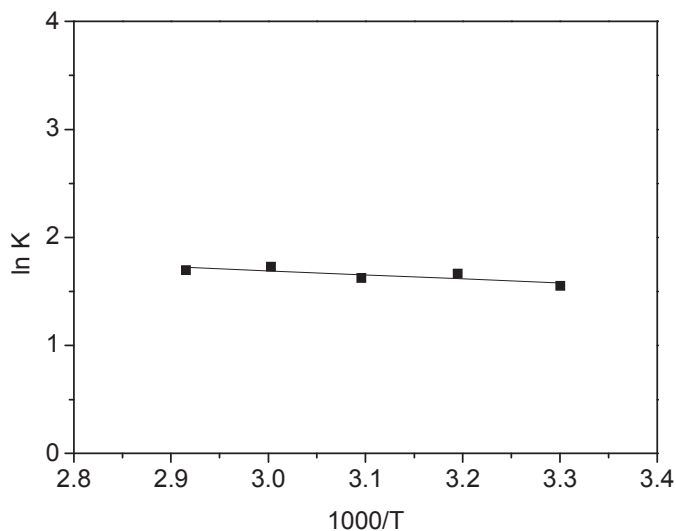


Fig. 4. The equilibrium constants dependence of temperature  
Rys. 4. Zależność stałej równowagi reakcji od temperatury

The reaction order and the rate constants were determined by fitting the experimental data to kinetic equations of various orders. The equation selected was the one which gave the least scatter of the reaction rate constant  $k$ . The evaluated reaction rate expression for the targeted esterification reaction is given below:

$$k_1 = 2.759 \cdot \exp\left(-\frac{33910}{R \cdot T}\right) \quad (4)$$

The Arrhenius plot of Eq. 4 is presented in Figure 5. The values of the frequency factor and activation energy are found to be:  $k_{1\infty}=2.76 \text{ dm}^3 \cdot \text{mol}^{-1} \cdot \text{s}^{-1}$ ,  $E=33.91 \text{ kJ} \cdot \text{mol}^{-1}$  respectively. For comparison, the activation energy for the reaction in the presence of cation exchange resin Indion 130 was found to be  $38.13 \text{ kJ} \cdot \text{mol}^{-1}$  [24] and in the presence of hydrogen iodide it was  $55.8 \text{ kJ} \cdot \text{mol}^{-1}$  [28].



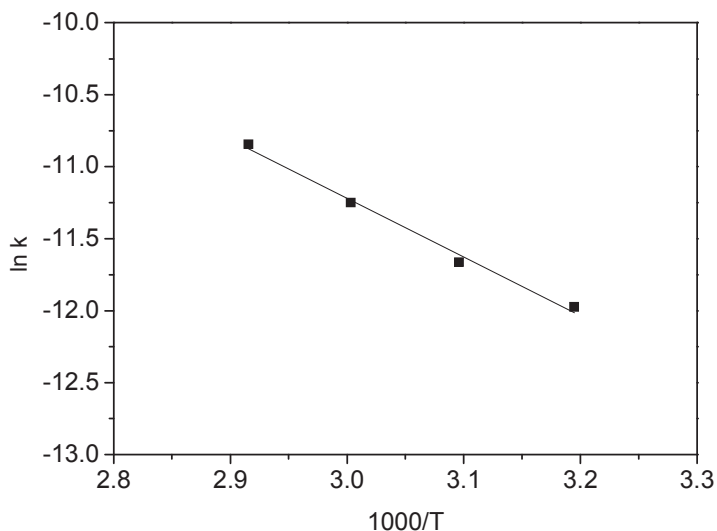


Fig. 5. Arrhenius plot  
Rys. 5. Wykres Arrheniusa

In Figures 1–3, the points represent the experimental results, while the curves portray predictions for a batch reactor with the reaction rate equation given by Eq. 2. The comparison of experimental and calculated data showed quite good accuracy of the derived expression.

#### CONCLUSIONS

- The esterification of acetic acid with methanol can be effectively performed using the proposed heterogeneous catalyst.
- The value of activation energy and linear relationship in the Arrhenius plot tend to indicate the lack of mass transfer limitations and that the process is kinetically controlled. That can be ascribed to very open structure of the catalyst particles and their very small size in the range of 10  $\mu\text{m}$ .
- Comparison of experimental with calculated data indicate that the reaction can be successfully described with the help of the homogeneous model for the reversible second-order reactions.

## SYMBOLS - OZNACZENIA

$c$	– concentration of component, $\text{mol}\cdot\text{dm}^{-3}$ stężenie składnika, $\text{mol}\cdot\text{dm}^{-3}$
$k_I$	– reaction rate constant, $\text{m}^3\cdot\text{mol}^{-1}\cdot\text{s}^{-1}$ stała szybkości reakcji, $\text{m}^3\cdot\text{mol}^{-1}\cdot\text{s}^{-1}$
$K$	– equilibrium constant stała równowagi reakcji
$r$	– reaction rate, $\text{mol}\cdot\text{dm}^{-3}\cdot\text{s}^{-1}$ szybkość reakcji, $\text{mol}\cdot\text{dm}^{-3}\cdot\text{s}^{-1}$
$T$	– temperature, K Temperature, K
$\Phi$	– Thiele modulus moduł Thielego
$D_{ef}$	– effective diffusion coefficient, $\text{m}^2\cdot\text{s}^{-1}$ efektywny współczynnik dyfuzji, $\text{m}^2\cdot\text{s}^{-1}$
$R$	– the molar gas constant, $\text{J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$ stała gazowa, $\text{J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$
$R_o$	– the radius of the grain, cm średnica ziarna, cm

## SUBSCRIPTS AND SUPERSRIPTS - NDEKSY DOLNE I GÓRNE

A	– alcohol alkohol
K	– acid kwas
E	– ester ester
W	– water woda

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#### KINETYKA REAKCJI ESTRYFIKACJI KWASU OCTOWEGO METANOLEM W OBECNOŚCI KATALIZATORA MCF FUNKCJONALIZOWANEGO GRUPAMI KWASOWYMI

Zbadano kinetykę reakcji estryfikacji kwasu octowego metanolem w obecności opracowanego katalizatora. Katalizatorem był krzemionkowy materiał o strukturze mezoporowatych pianek komórkowych funkcjonalizowanych grupą etylofenylosulfonową. Otrzymany katalizator cechowała otwarta, mezoporowata struktura złożona z dużych kulistych porów o średnicach  $d_p=29$  nm połączonych oknami  $d_w=14$  nm. Powierzchnia właściwa otrzymanego katalizatora wynosiła  $S_{BET}=260$  m<sup>2</sup>·g<sup>-1</sup>. Przeprowadzone wcześniej testy wskazywały na dobrą stabilność właściwości strukturalnych i katalitycznych otrzymanego materiału. Przed przystąpieniem do badań określono warunki w których transport masy do powierzchni cząstek katalizatora nie ograniczał globalnej kinetyki reakcji. W tym celu określono wpływ intensywności mieszania na szybkość reakcji. Badania prowadzono w zakresie 200–1000 obrotów/minutę. Eksperymenty przeprowadzone dla ilości obrotów mieszadła powyżej 500 obr/min wykazały, że szybkość mieszania nie wywierała znaczącego wpływu na szybkość reakcji, co oznacza, że w tym zakresie nie występowały zewnętrzne opory wymiany masy. Wszystkie dalsze badania przeprowadzono mieszając zawiesinę z szybkością 1000 obr/min. Celem oszacowania wpływu dyfuzji wewnętrznej na szybkość procesu dla zewnętrznych warunków reakcji ( $T = 343$  K i  $r_K = 1,74$  mol·m<sup>-3</sup>·s<sup>-1</sup>) wyznaczono moduł Thielego. Wyznaczona wielkość  $\Phi=5,49 \cdot 10^{-5}$  pozwoliła na stwierdzenie, że opory dyfuzji wewnętrznej dla rozpatrywanego układu mogą być pominięte. Zbadano wpływ ilości katalizatora (0,5; 1; 2%), temperatury (313, 323, 333, 343 K) oraz stosunku molowego substratów (1:1, 1:3, 1:5) na konwersję kwasu octowego. Stwierdzono słabą zależność stałej równowagi reakcji od temperatury. Rząd i stałą szybkości reakcji wyznaczono metodą podstawienia. Wyznaczona dla reakcji energia aktywacji wynosiła 33,91 kJ·mol<sup>-1</sup>, natomiast współczynnik przedwykładniczy: 2,76 dm<sup>3</sup>·mol<sup>-1</sup>·s<sup>-1</sup>. Analiza kinetyczna procesu estryfikacji kwasu octowego metanolem przeprowadzonego w obecności otrzymanego katalizatora pozwoliła na stwierdzenie, że reakcja jest rzędu drugiego, pierwszego względem każdego ze składników, oraz że dane doświadczalne z dobrą dokładnością opisuje model pseudohomogeniczny.