

Modifications in the kinetics of transesterification of sunflower oil over strontium supported on silica by barium doping

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ABSTRACT

The effect of doping of strontium/silica (Sr40/ Si-A_{octa}) catalyst by barium on its kinetics of methanol transesterification of sunflower oil (MTSFO) to biodiesel was investigated. The experimental data of MTSFO over Sr40/Si-A_{octa} appear to be good fit into pseudo first order kinetic mechanism. However, the zero-order rate equation is the best fit model for MTSFO over BaSr40/Si-A_{octa}, compared with the pseudo-first order and second-order rate kinetic models. On the other hand, the intraparticle diffusion kinetics model may also play an important role in the MTSFO over BaSr40/Si-A_{octa}. In addition, a significant change was observed in Arrhenius activation energy of MTSFO over Sr40/Si-A_{octa} when it dopped by Ba. Where, Arrhenius activation energy values were found to be 16.6 and 49.6 kJ mol⁻¹ for Sr40/Si-A_{octa} and BaSr40/Si-A_{octa}, respectively. These results can be suggested that addition of Ba to Sr40/Si-A_{octa} can enhance its catalytic performance in selective MTSFO.

Keywords: Transesterification, biodiesel, kinetics, strontium, barium, catalysts.

INTRODUCTION

Heterogenous catalytic production of biodiesel from transesterification of vegetable oils using methanol received a great attention in last few years, from energy cost and environmental views^[1-5]. Not only, basic homogenous catalysts such as alcoholic NaOH and KOH perform the problems of forming soap, but also other problems may arise such as the formation of high quantities of by-products beside corrosion problems. Thus, the using of solid basic heterogenous catalysts are always essential to overcome these problems in esterification process^[6].

Different alkaline earth metals (viz., Ca, Mg, and Ba) doped SiO₂ catalysts were used for the biodiesel production^[7]. The demand in the use of alkaline earth metal oxides as heterogeneous basic catalysts has increased due to their availability, non-toxicity, the ability to be reused, low cost, and high amount of surface basic sites that perform the activity^[8].

Understanding the kinetics of the methanol transesterification to biodiesel over heterogenous catalyst is essential for improving catalyst performance at desired reaction conditions. In general, kinetics of heterogeneously catalyzed transesterification over monophasic basic catalyst follows pseudo first order mechanism^[9-12]. In the present study, the kinetics of MTSFO was investigated not only on monophasic SrO supported on silica but also on biphasic Ba-SrO-BaO/silica. Different kinetics models (viz., zero, first, second and interparticle diffusion) for esterification reaction of sunflower oil with methanol were tested.

EXPERIMENTAL

1. Preparation of mesoporous silicate by using n-octadecylamine as a surfactant

In the preparation process 64.8ml of deionized water, 0.16 ml of HCL and 3.25 g of n-octadecylamine were added in a flask with magnetic stirrer to form mixture A. 22.32 ml of TEOS and 37.9 ml of anhydrous ethanol were fully mixed in beaker to form mixture B. Then solution B was poured into the flask containing solution A drop by drop for 20-30 min. The resulting mixture was sequentially stirred at room temperature for 2h, and then left the mixture for 18 h. The obtained silica particles were filtrated and fully washed with adequate deionized water and were dried at 60°C for 12h and then calcined at 550 °C for 5 h^[13]. The obtained silica support denoted as Si-A_{octa}.

2. Preparation of strontium oxide supported on silica

Strontium oxide supported on Si-A_{octa} with loading 40w.t% was prepared using deposition method. 0.7 M Sr(NO₃)₂ and 0.3 M NaOH were prepared separately by dissolving appropriate quantities of salts in water. The high concentration of Sr(NO₃)₂ was necessary in order to reach a high supersaturation degree, an important requirement for the production of nanoparticles. The Sr(NO₃)₂ solution was heated to 60°C. Then silica support was added to strontium nitrate solution at 60°C with vigorous continuous stirring. The precipitation reaction of took place by dripping the NaOH solution into the strontium salt /silica solution, under vigorous stirring for 2-3h, keeping the temperature constant at 60°C within ±1°C^[14,15]. When the precipitation of Sr(OH)₂ on support was completed, the mixture was continuously and vigorously stirred for a further 2-3h in order to deagglomerate the white precipitate as soon as possible. The catalyst aqueous suspension was cooled down to room temperature and left to decant for 24 h. The supernatant solution was removed with a pipette and the remaining suspension was washed three times with deionized cold water (leaving the suspension to decant every time for 24 h) to take off the more soluble sodium nitrate byproduct. Finally, the suspension was filtrated and washed by deionized water^[16]. The slurry was dried for 24h at 100°C then calcination at 700°C for 5h. The obtained catalyst denoted as Sr40/Si-A_{octa}.

3. Preparation of Sr40/Si-A_{octa} doped by Barium

The Sr40/Si-A_{octa} promoted by Ba with 10wt.% loaded, was prepared by impregnation using metal nitrates. The preparation process was as follows: Appropriate quantities of metal nitrate (Ba(NO₃)₂) was initially dissolved in 25ml deionized water, stirring until the mixture become clear then added into the solution under vigorous stirring appropriate quantities of Sr40/Si-A_{octa}. The suspension was stirred during 1 h, then the suspension was kept in the beaker for 24 h and then heated at 80 °C under vigorous stirring, until dryness. The formed paste was dried overnight at 110°C and then crushed in a Pyrex mortar. The powders were calcined for 5 h in a muffle at 700°C for 4h with a heating rate of 5 °C/min. Catalyst prepared was denoted BaSr/Si-A_{octa}.

4. Kinetic study

To determine the kinetics of the transesterification reaction, the effect of reaction temperature and reaction time were studied for both Sr40/Si-A_{octa} and 10 wt. % Ba Sr40/Si-A_{octa} catalyst. All reactions were achieved in a 250 ml glass reactor containing a condenser. The catalyst was spread throughout the 30:1 methanol to oil molar ratio with magnetic stirring of 500 rpm. Then, the sunflower oil was added, and the mixture was heated at reflux at selected reaction

Modifications in the kinetics of transesterification of sunflower oil over strontium supported on silica by barium doping

temperatures and times. The reaction temperature varied from 50-60 °C and reaction time varied from 0 to 60 min. After the reaction has finished, two layers were created. The upper layer contained biodiesel and methanol, while the bottom layer contained glycerol. Methanol that was present in excess was evaporated, while the catalyst was separated using centrifugation. Finally, a decanter was used to separate the biodiesel and glycerol using gravity. The sample concentration determined by the mole fraction at the time specified by Iodometric titration^[17,18].

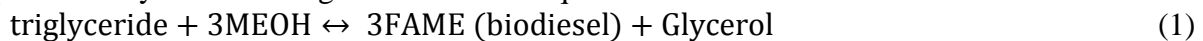
RESULTS AND DISCUSSION

1. Kinetics of MTSFO over Sr40/Si-A_{octa} and BaSr/Si-A_{octa} catalysts

Transesterification is generally assumed to follow pseudo 1st order kinetics as alcohol in such reactions is employed in excess on the required stoichiometric amount of 3:1 alcohol to oil molar ratio. The present work also optimized alcohol to SFO molar ratio at 30:1 and hence, the Sr40/Si-A_{octa} and BaSr/Si-A_{octa} catalysts showed high catalytic activity for biodiesel production 75% and 97%, respectively. Thus, kinetics of Sr40/Si-A_{octa} and BaSr/Si-A_{octa} catalyzed transesterification of SFO were studied.

2. Kinetic equation and Pseudo 1st order Model

It is well known that the general equation of transesterification of triglycerides (TG) can be presented by the following stoichiometric Eq. 1:



This reaction follows of three steps to convert triglyceride to fatty acid methyl ester (FAME; biodiesel) and glycerol.

In the first step TG reacts with one molecule of methanol to produced diglyceride (DG) and one molecule of methyl ester. Then, the molecule of DG reacts with another molecule of methanol to produce monoglyceride (MG) and one molecule of methyl ester. Finally, the MG reacts with methanol to produce ME and glycerol.

Based on previously reported mechanistic considerations, it was assumed that the reaction is a single step transesterification, and the intermediate reactions of DG and MG have been ignored^[19-22]. Thus, the kinetic equation of transesterification reaction can be expressed as:

$$-r = \frac{-d[\text{TG}]}{dt} = K^{\cdot} \cdot [\text{TG}] \cdot [\text{ROH}]^3 \quad (2)$$

Where, K^{\cdot} is the equilibrium rate constant.

Considering, Eq. 2, this overall conversion should follow a fourth order reaction rate law^[22]. However, due to large amount of the used alcohol in the reaction mixture the reaction could be safely considered follows the pseudo-first order kinetics^[20, 23-24].

For considering MTSFO to be pseudo-first order mechanism, the following hypothesis were proposed: i) the transformation of TG follows as the pseudo-first order transesterification reaction kinetics, ii) the reverse reaction could be ignored, iii) consuming catalyst concentration during the reaction can be proposed to be negligible due to using of sufficient amount of catalyst with respect to sunflower oil to shift the reaction equilibrium to the forward reaction, and iv) the concentration of methanol (MeOH) was almost constant due to the excess amount of methanol (Methanol/ oil ratio 30:1). Thus, Eq. 2 can be modified to the pseudo-first order rate law and can be expressed as Eq. 3:

$$-r = \frac{-d[\text{TG}]}{dt} = K \cdot [\text{TG}] \quad (3)$$

Where K is the modified rate constant,

$$K = K' \cdot [\text{MeOH}]^3 \quad (4)$$

By the integration of Eq. 3:

$$\ln. [\text{TG}]^\circ - \ln. [\text{TG}] = K \cdot t \quad (5)$$

Based on the mass balance, conversion (X) of MeOH is given by Eq. 6.

$$X = 1 - \frac{[\text{TG}]}{[\text{TG}]^\circ} \quad (6)$$

$$[\text{TG}] = [\text{TG}]^\circ (1 - X) \quad (7)$$

Also,

$$\frac{dX}{dt} = K(1 - X) \quad (8)$$

Finally the 1st order kinetic model is given by equation (9) ,

$$-\ln(1 - X) = Kt \quad (9)$$

3.2. Kinetics plots and estimated kinetics parameters

Figures (1-6) show the plots of $\ln(1 - X)$ versus reaction time by applying pseudo-first order kinetics model. In addition, the estimated rate constants at different temperatures and regression coefficient values are listed in Table (1). As shown in Table 1 and Figures (1-3), the pseudo-first-order rate equation fitted well MTSFO data over Sr40/Si-A_{octa}, where a good regression coefficient (R^2) was obtained with a good linear nature of the plot between $\ln(1 - X)$ vs. t. However, as shown in Table 1a relatively low regression coefficient (R^2) was obtained for pseudo-first-order rate equation of MTSFO over BaSr40/Si-A_{octa} compared with that of Sr40/Si-A_{octa}. Which reveals a deviation from pseudo first order kinetic model (Figs. 4 -6). Thus, another three different kinetic models have been used to investigate MTSFO over BaSr40/Si-A_{octa} namely, pseudo-second order, zero order and intraparticle diffusion kinetics models. The linear forms of the three different kinetic models; pseudo-second order, zero order and intraparticle diffusion can be written as equations 10,11 and 12, respectively:

$$Kt = a/(1 - X) - 1/a \quad (10)$$

Where, a is initial concentration of assuming equal unity for 100% conversion.

$$X = Kt \quad (11)$$

$$X = Kt^{0.5} + \text{constant} \quad (12)$$

It is worth noting that from Figure (7) and Table (2) (R^2 , values) the pseudo-second order model did not describe well MTSFO over BaSr40/Si-A_{octa}. Interestingly, as shown in Table (2) and Figure (8), the zero-order rate equation is the best fit model for MTSFO over BaSr40/Si-A_{octa}, where a good regression coefficient (R^2) was obtained for the zero-order model compared with the pseudo-first-order and pseudo-second-order rate kinetic models (Table 2). On the other hand, the intraparticle diffusion kinetics model (Table 2, Fig. 9) may be also played an important role in the MTSFO over BaSr40/Si-A_{octa} (R^2 for intraparticle diffusion is ca. 0.94).

To calculate the activation energy, the Arrhenius equation was employed to estimate the activation energy (Ea) Eq. 13:

$$\ln k = \ln A - E_a/RT \quad (13)$$

Modifications in the kinetics of transesterification of sunflower oil over strontium supported on silica by barium doping

Where, E_a is the activation energy (KJ mol^{-1}), A is the preexponential factor (min^{-1}), R is the gas constant ($8.314 \text{ J K}^{-1} \text{ mol}^{-1}$) and T is the reaction temperature (K). A plot between $\ln k$ vs. $1/T$ is shown in Figures (10 and 11), the values of E_a from the plot was listed in Table (2). The observed E_a value in the present study (16.6 and 49.6 KJ mol^{-1}) was found within the range of the reported values ($26\text{--}82 \text{ KJ mol}^{-1}$) for the transesterification reaction catalyzed by heterogeneous catalysts.

Table 1. The estimated rate constants values from first order model at different temperatures and regression coefficient of MTSFO over Sr40/Si- A_{octa} and BaSr/Si- A_{octa} catalysts.

T (°C)	Sr40/Si- A_{octa}			BaSr/Si- A_{octa}		
	K (min^{-1})	R^2	E_a (KJ mol^{-1})	K (min^{-1})	R^2	E_a (KJ mol^{-1})
50 °C	0.019	0.9709	16.62	0.0402	0.829	49.61
55 °C	0.0196	0.9952		0.0434	0.8696	
60 °C	0.0229	0.9898		0.07	0.809	

Table 2. The estimated rate constants values from pseudo-second, zero order and interparticle-diffusion models at different temperatures and regression coefficient of MTSFO over BaSr/Si- A_{octa} catalysts.

T (°C)	Pseudo-Second order		Zero order		Interparticle-diffusion	
	K (min^{-1})	R^2	K (min^{-1})	R^2	K (min^{-1})	R^2
50 °C	0.153	0.697	0.0140	0.967	0.168	0.900
55 °C	0.179	0.715	0.0144	0.988	0.161	0.938
60 °C	0.850	0.638	0.0156	0.988	0.166	0.947

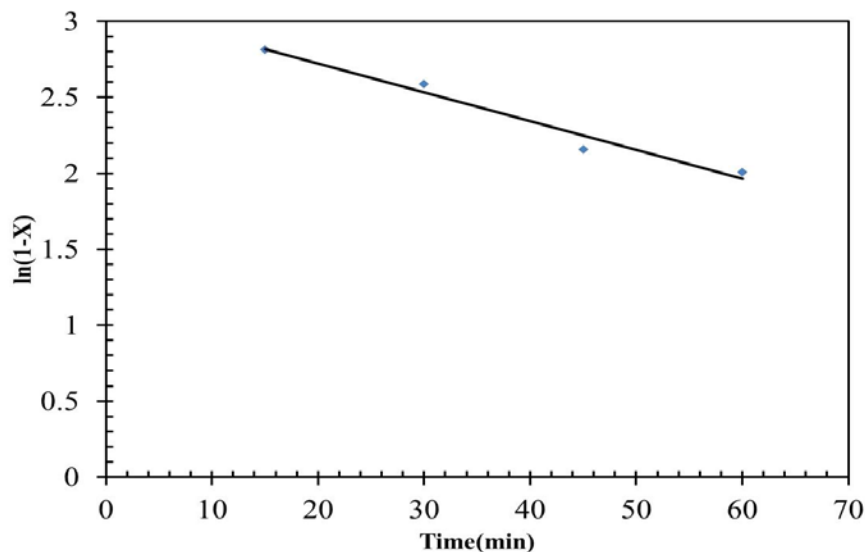


Fig. 1. First order kinetics plot of MTSFO over Sr40/Si- A_{octa} . Reaction conditions: methanol/oil ratio 30:1, the reaction time 1.0 h and catalyst /oil ratio 1.0 wt.% at 50 °C .

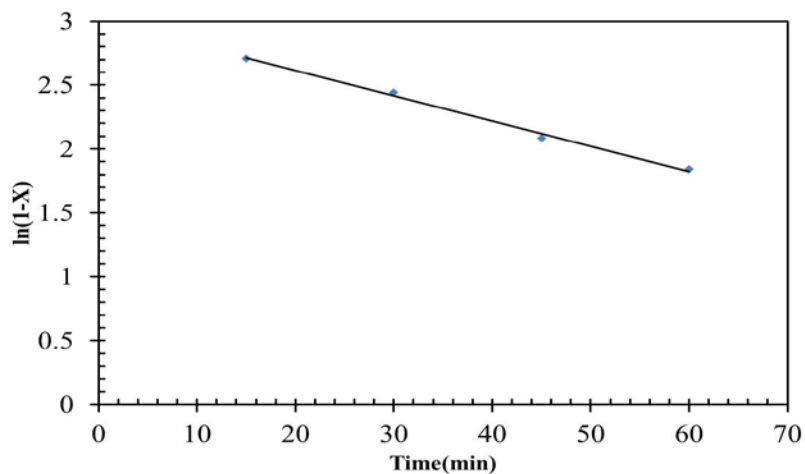


Fig. 2. First order kinetics plot of MTSFO over Sr40/Si-A_{octa}. Reaction conditions: methanol/oil ratio 30:1, the reaction time 1.0 h and catalyst /oil ratio 1.0 wt.% at 55 °C.

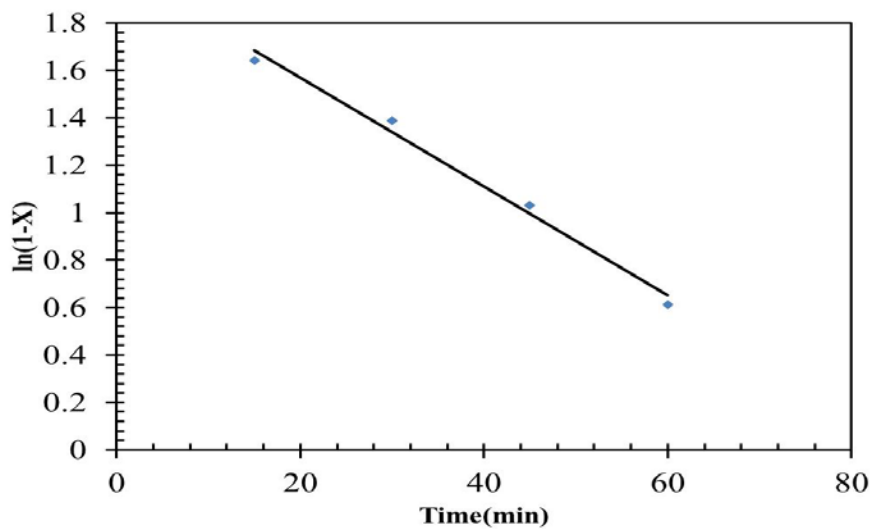


Fig. 3. First order kinetics plot of MTSFO over Sr40/Si-A_{octa}. Reaction conditions: methanol/oil ratio 30:1, the reaction time 1.0 h and catalyst /oil ratio 1.0 wt.% 60 °C.

Modifications in the kinetics of transesterification of sunflower oil over strontium supported on silica by barium doping

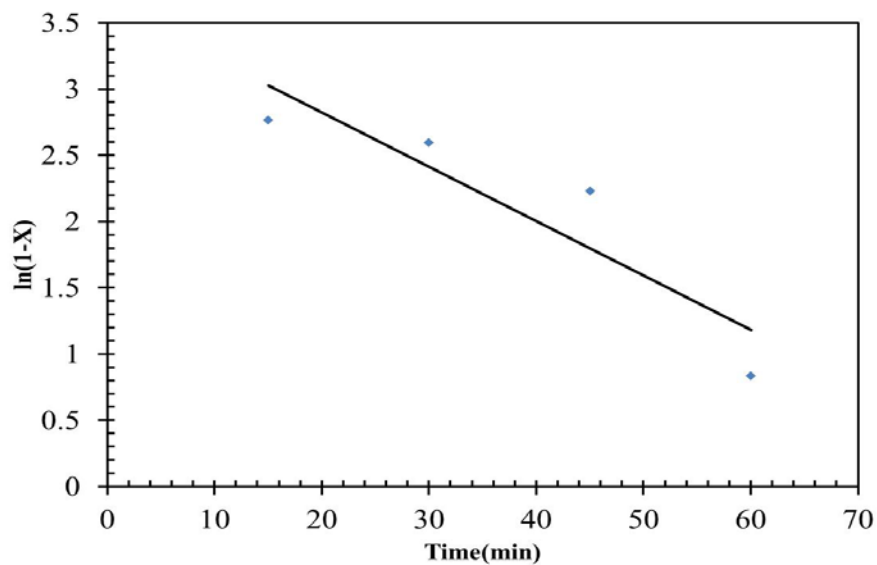


Fig. 4. First order kinetics plot of MTSFO over BaSr/Si-A_{octa}. Reaction conditions: methanol/oil ratio 30:1, the reaction time 1.0 h and catalyst /oil ratio 1.0 wt. % 50 °C.

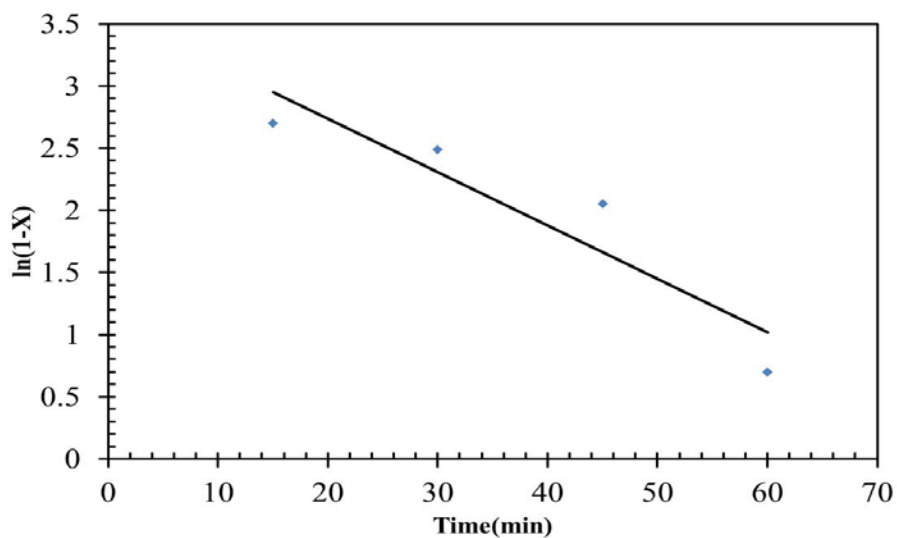


Fig. 5. First order kinetics plot of MTSFO over BaSr/Si-A_{octa}. Reaction conditions: methanol/oil ratio 30:1, the reaction time 1.0 h and catalyst /oil ratio 1.0 wt. % 55 °C.

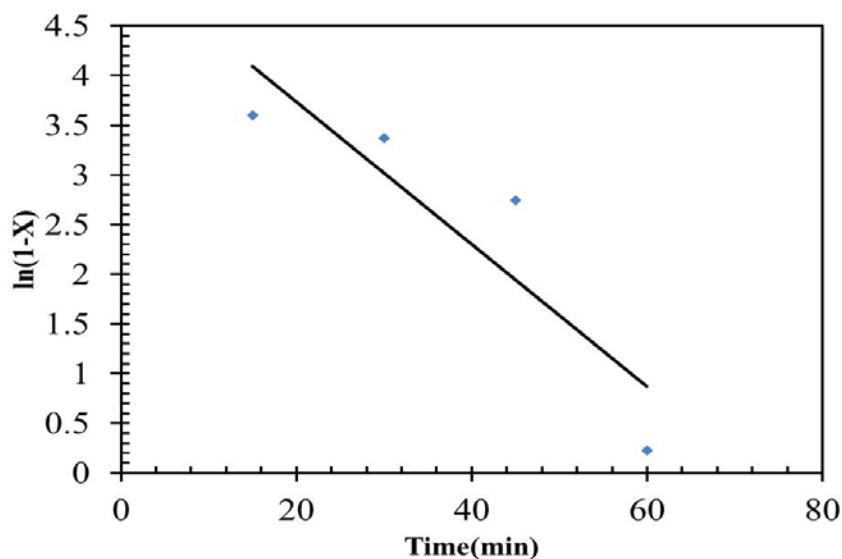


Fig. 6. First order kinetics plot of MTSFO over BaSr/Si-A_{octa}. Reaction conditions: methanol/oil ratio 30:1, the reaction time 1.0 h and catalyst /oil ratio 1.0 wt.% at 60 °C.

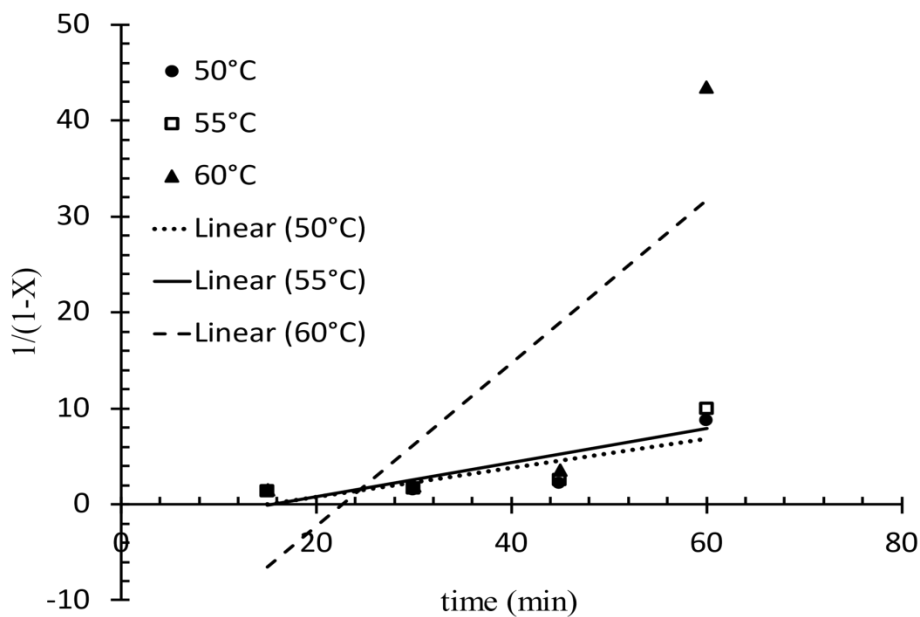


Fig. 7. Pseudo-second order kinetics plots of MTSFO over BaSr40/Si-A_{octa}. Reaction conditions: methanol/oil ratio 30:1, reaction time 1.0 h and catalyst /oil ratio 1.0 wt.% at different temperatures.

Modifications in the kinetics of transesterification of sunflower oil over strontium supported on silica by barium doping

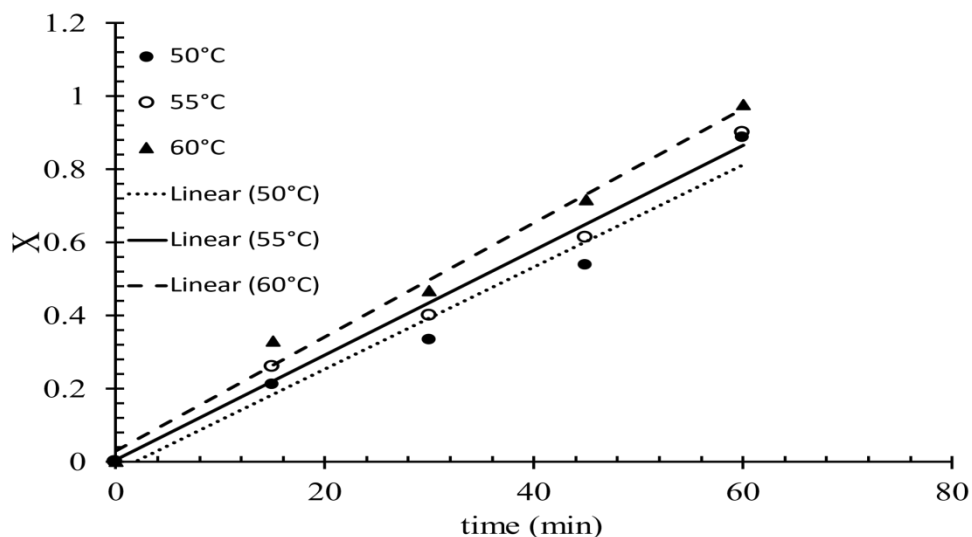


Fig. 8. Pseudo-zero order kinetics plots of MTSFO over BaSr40/Si-A_{octa}. Reaction conditions: methanol/oil ratio 30:1, reaction time 1.0 h and catalyst /oil ratio 1.0 wt.% at different temperatures.

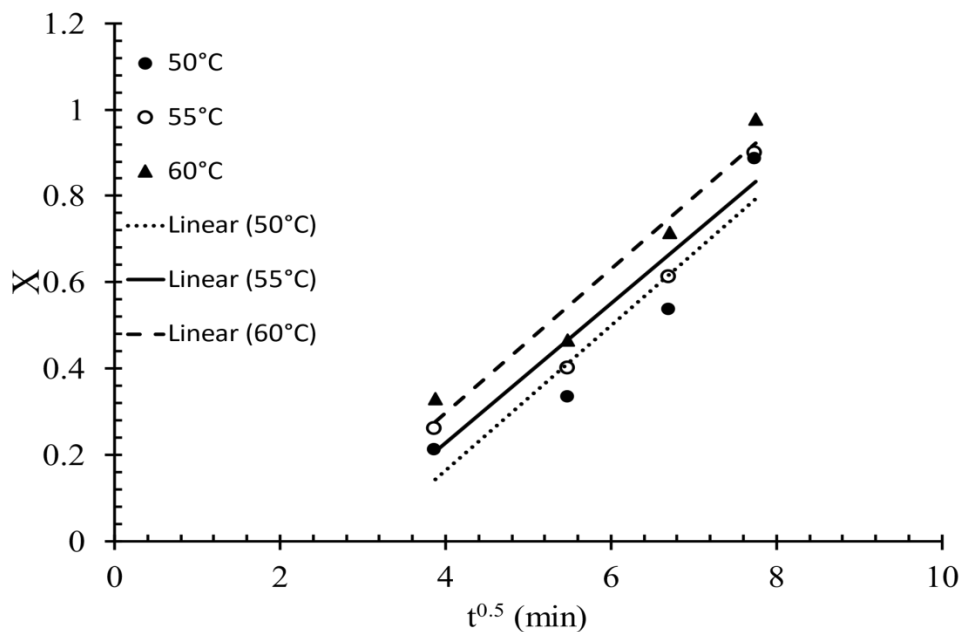


Fig. 9. Interparticle-diffusion kinetics plots of MTSFO over BaSr40/Si-A_{octa}. Reaction conditions: methanol/oil ratio 30:1, the reaction time 1.0 h and catalyst /oil ratio 1.0 wt.% at different temperatures.

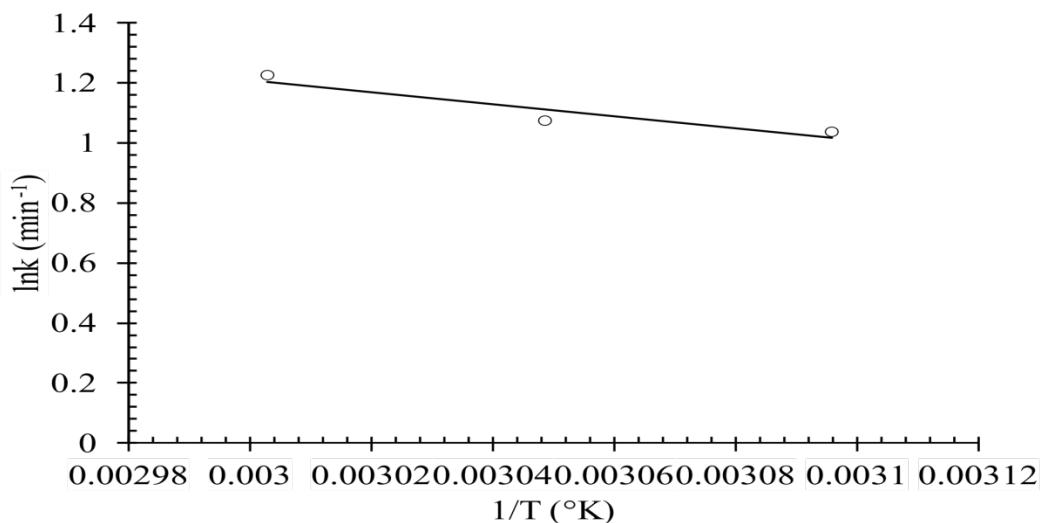


Fig. 10. Arrhenius plot of MSTFO over Sr40/Si-A_{octa}. Reaction conditions: methanol/oil ratio 30:1, the reaction time 1.0 h and catalyst /oil ratio 1.0 wt.%.

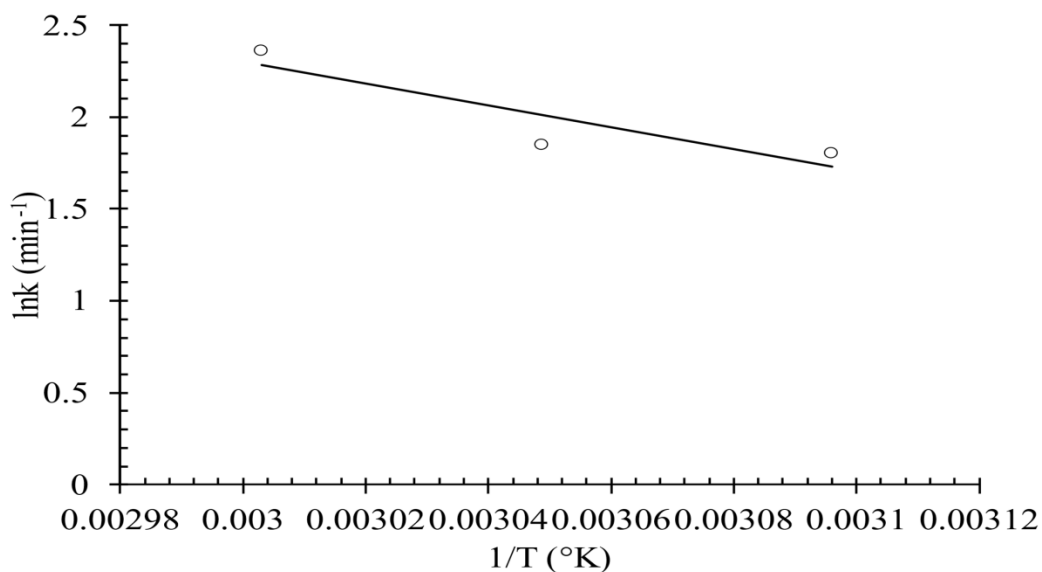


Fig. 11. Arrhenius plot of MTSFO over BaSr40/Si-A_{octa}. Reaction conditions: methanol/oil ratio 30:1, the reaction time 1.0 h and catalyst /oil ratio 1.0.

CONCLUSION

The kinetic data of methanol transesterification of sunflower oil (MTSFO) to biodiesel over single component -strontium supported on silica appear to good fit into pseudo first order kinetic model. Interestingly that, the doping of strontium/silica by barium decrease reaction order from pseudo first order to zero order. This indicates that the reaction mechanism is independent on concentration of reactants where, diffusion mechanism of reactants into catalyst active phases plays a significant role.

Modifications in the kinetics of transesterification of sunflower oil over strontium supported on silica by barium doping

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تعديلات في حركية الأسترة التبادلية لزيت عباد الشمس فوق السترونشيوم المدعوم بالسيليكا بواسطة منشطات الباريوم

منى عبد الفتاح ، صلاح أ.حسن ، عبد النبي محمد سالم ، هاني محمد عبد الدايم
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المستخلص

تم دراسة تأثير تعاطي المنشطات لمحفز السترونشيوم / السيليكا (Sr40 / Si-Aocta) بواسطة الباريوم على حركية أسترة الميثانول لزيت عباد الشمس (MTSFO) إلى وقود الديزل الحيوي. وجد أن البيانات التجريبية لـ MTSFO عبر Sr40 / Si-Aocta تتلاءم جيداً مع النموذج الحركي من الرتبة الأولى الزائفه عند استخدام السترونشيوم أحادي المكون المدعوم على السيليكا. ومن المثير للاهتمام، أن تدعيم السترونشيوم / السيليكا بالباريوم يقلل من رتبه التفاعل من الرتبة الأولى الزائفه إلى رتبه الصفر. وهذا يشير إلى أن آلية التفاعل لا تعتمد على تركيز المواد المتفاعلة، بينما تلعب آلية انتشار المواد المتفاعلة في المواضع النشطة للمحفز دوراً مهماً.