

Study of effects of some reaction conditions on ethanolysis of rapeseed oil with dispergation

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Abstract

The alkaline-catalyzed (KOH) ethanolysis of rapeseed oil with the help of a mechanical disperser was researched in this study. The effects of chosen reaction conditions (independent variables: the reaction temperature and time, the amount of catalyst, the molar ratio ethanol to oil and the rotation frequency of the disperser) on the ethanolysis process and ways for the improvement of the transesterification and separation process were studied. The transesterification of oil, the separation of the ester phase from the glycerol phase and qualitative properties of the ester phase were monitored by many dependent variables (e.g. the yield of the ester phase, the weight concentration of glycerides, the content of potassium ions etc). The measured data was analyzed by multi-linear regression with the help of many statistical tests (significance of parameters, exclusion of outliers etc.). The created mathematical models describing the relations between the independent and dependent variables were verified by several independent experiments.

1 Introduction

Recently, the world importance of biodiesel production has increased significantly. The decreasing of the carbon dioxide emissions, the dependence on imported crude oil and better sales possibilities for farmers are the main reasons for the usage of biodiesel. Nowadays, the mixing of fatty acid methyl esters (FAME) into fossil diesel fuel is requested by law in many countries.

Biodiesel production proceeds mainly by transesterification reaction. Alkaline, acid or enzymatic catalyses are possible. There is also a method without the using of a catalyst

where alcohol reacts in a supercritical state [Warabi et al., 2004]. But alkaline catalysis is the most effective and widely used method [Meneghetti et al., 2006].

The most used alcohol is methanol, but it has some disadvantages: the toxicity and it is difficult to produce it from renewable raw materials. Thereby there is a tendency to use ethanol in biodiesel production. On the other hand, ethanol has lower reactivity in comparison with methanol during transesterification [Warabi et al., 2004], [Issariyakul et al., 2007], which is caused by a longer carbon chain [Nimcevic et al., 2000]. The reaction time of transesterification takes about one hour and more.

Alkaline catalyzed transesterification is very complicated process: the reaction system is complex [Mittelbach and Remschmidt, 2004] (see also Fig. 1) and the presence of a two-phase system makes the process more difficult. The improvement of the alkaline-catalyzed transesterification process is desired and therefore some improved methods are researched: ultrasonic and hydrodynamic cavitation [Wang et al., 2006], microwave assistance [Hernando et al., 2007], [Azcan et al., 2008] and ultrasound assistance [Santos et al., 2009].

Some authors tried to create a kinetic model of transesterification [Komers et al., 2002], [Stamenkovic et al., 2008] but statistical models are frequently used. The statistical design of experiments is the most often used for the study of transesterification. The factorial design matrix for linear models or central composite designs for non-linear models and response surface methodology are frequent [Domingos et al., 2008], [Bouaid et al., 2007]. The effects of three or four independent variables (reaction conditions) on the yield or conversion of oil to esters are mostly investigated.

This work is focused on the ethanolysis of rapeseed oil with the dispergation. The aim and the exceptionality of this paper consists of the investigation of the effects of several independent variables on many dependent variables which monitor the self transesterification process, the separation of phases and the qualitative properties of the ester phase.

2 Materials and methods

2.1 Researching strategy

The creation of a large interfacial surface is necessary for the transesterification (two-phase system of immiscibility liquids ethanol–oil). A mechanical disperser, which enables the emulsification of the mixture, has a better effect than common stirrers. The reaction temperature, the reaction time, the weight ratio of the catalyst (KOH) to oil, the molar ratio ethanol to oil and revolutions of the disperser (see also 2.2) were selected as the independent variables because these variables influence the kinetic and thermodynamic aspects of the transesterification process. For example, the reaction temperature increases the reaction speed, changes the equilibrium of reversible reactions and influences the phase equilibrium. The investigated intervals of independent variables for all 17 experiments are shown in Tab. 1. The intervals were selected on the basis of preliminary experiments and results of other authors the regard to the slower speed of ethanolysis in comparison with methanolysis and the requirement of a suitable conversion of rapeseed oil. The experiments were designed on the basis of the Plackett-Burman designs.

Many dependent variables were monitored: relative amounts of ethyl esters, content of soaps, monoglycerides, diglycerides and triglycerides in the reaction mixture; yield,

concentrations of glycerides, potassium ions and free glycerol in the ester phase; the weight of phase and concentration of ethyl ester in the glycerol phase (see also 2.2). The monitoring of many dependent variables enables us to formulate the obtained findings more globally.

Linear models for the description of the relation between dependent and independent variables were used because the natural patterns of these dependences are unknown and linear models are the simplest. Before the statistical analysis, the independent variables were normalized into interval $(-1;+1)$. The building of the linear models was carried out with the help of statistical testing (significance of parameters, exclusion of outliers etc.) according to [Meloun and Militký, 2004].

2.2 Symbols and variables

EP, GP – ester phase, glycerol phase.

TG, DG, MG – triglycerides, diglycerides, monoglycerides. All these substances are referred to as glycerides.

EE, S, K, FG – ethyl esters, soaps, potassium (potassium ions), free glycerol.

T – reaction temperature. [$^{\circ}\text{C}$]

t – reaction time. [hours]

C – weight ratio of catalyst (KOH) to oil. [wt-%]

MR – molar ratio ethanol to oil. [-]

f – rotation frequency of disperser. [rpm]

Y_{EP} – yield of ester phase (biodiesel) [%]. It is calculated as the ratio of measured weight of EP and theoretical weight of EE multiplied by 100. Theoretical weight of EE formed from 1 g of rapeseed oil is 1.051 g (if rapeseed oil has $M_R=879.1 \text{ g}\cdot\text{mol}^{-1}$ and ethyl ester of rapeseed oil has $M_R=308.1 \text{ g}\cdot\text{mol}^{-1}$).

w_{EE} , w_S , w_{MG} , w_{DG} , w_{TG} – weight ratio of ethyl esters, soaps, monoglycerides, diglycerides and triglycerides in the whole reaction mixture to the initial weight of oil (key component) [wt-%]. These weight ratios were calculated as the sum of the substance in both phases together divided by the initial weight of oil (900 g) as the key component and multiply by 100.

$w_{TG(EP)}$, $w_{DG(EP)}$, $w_{MG(EP)}$, $w_{K(EP)}$, $w_{FG(EP)}$ – weight concentration of triglycerides, diglycerides, monoglycerides, potassium ions and free glycerol in EP. [wt-%]

$w_{EE(GP)}$ – weight concentrations of ethyl esters in GP. [wt-%]

2.3 Chemicals

Cold-pressed, filtrated rapeseed oil, free of erucic acid (acid number $0.6 \text{ mg KOH}\cdot\text{g}^{-1}$, water content $600 \text{ mg}\cdot\text{kg}^{-1}$ and density $920 \text{ kg}\cdot\text{m}^{-3}$, produced by firm RPN Slatiňany), Absolute alcohol (water content 0.12 %), Potassium hydroxide p. a. (purity 90 %), Carbon dioxide (for food processing industry).

2.4 Apparatus

The double-walled laboratory reactor IKA[®] LR 2000 (volume 2 l) was used (Fig. 2). A toothed disc stirrer served as the main stirrer. The high-performance disperser T-25 digital ULTRA-TURRAX[®] (maximal 24000 rpm) was installed into the reactor. The reactor was joined to a water pump and thermostat [Hájek et al., 2009].

2.5 Procedure

900 g of rapeseed oil was put into the reaction vessel; the catalyst was dissolved in ethanol and both liquids were thermostated separately to the reaction temperature. Then the disperser was switched on, the solution of KOH in ethanol was quickly added into the reaction vessel and this point was considered as the start of the reaction. The stirrer was set to 200 rpm during the transesterification process because of satisfactory heat

transfer. After the chosen reaction time, the reaction was stopped by neutralisation of the catalyst by gaseous CO₂ dosed into a reaction mixture until pH fell to a minimum value (approximately 5 minutes). Potassium carbonate and potassium bicarbonate were formed from KOH; they have no catalytic effect in this system [Skopal et al., 2001]. Then the reaction mixture was evaporated for 40 min at 80°C at a pressure of approximately 3 kPa (water pump) and thereby the excess of ethanol was removed (deethanolisation). After cooling down to 25°C the reaction mixture was separated for 24 hours to the EP and GP by gravitation in a separatory funnel. Then both formed phases were analyzed.

2.6 Analytical methods

2.6.1 Determination of glycerides and ethyl esters

The content of glycerides in both phases were determined by the GC method according to EN 14105 by Shimadzu GC-2010 with the help of linear calibration curves (monoolein, diolein and triolein were used as standards).

The content of ethyl esters in the EP [wt-%] was calculated as the difference 100 % minus the sum of concentrations of all glycerides in the ester phase, providing that the concentrations of other substances in the EP are insignificant.

The content of glycerides in the GP was determined by the same GC method, but the sample had to be pre-treated before GC analysis: The sample of the GP was acidified by H₃PO₄. Soaps were transformed into fatty acids and the glycerol phase was distributed into a lighter non-polar phase (esters, glycerides, fatty acids and other non-polar substances) and a heavier polar phase (glycerol, water, potassium salts and other polar substances). The separation of phases was accelerated by water addition and heating up (80°C). Then the non-polar phase was analyzed as described in EN 14105. The

concentrations of monoglycerides (and other glycerides by analogy) in the glycerol phase were calculated according to (1).

$$W_{MG(GP)} = W_{MG(non-polar\ phase)} \cdot \frac{m_{non-polar\ phase}}{m_{GP}} \quad (1)$$

The concentration of ethyl esters in the GP was determined by GC method together with the determination of glycerides in one analysis: The peak of ethyl esters was identified in the chromatogram of the non-polar phase. The concentration of ethyl esters in the non-polar phase was calculated according to the calibration curve. Then the concentration of ethyl esters in the glycerol phase was calculated analogously to (1).

2.6.2 Determination of soaps, carbonates and bicarbonates in the glycerol phase

This determination is based on the combination of acidimetric titration of the GP and the measuring of volume of CO₂ formed during acidification of the GP [Kwiecien et. al., 2009]. The first step of the acidimetric titration curve of the GP corresponds with the forming of bicarbonates from carbonates. The second step corresponds with the forming of fatty acids from soaps and CO₂ from bicarbonates (sum of the original bicarbonates and bicarbonates formed in the first step). Measuring of the volume of the forming CO₂ is necessary to calculate the concentrations. This volume was determined separately because it is impossible to make it together with the titration.

2.6.3 Determination of potassium ions and free glycerol in the ester phase

The concentration of potassium ions in the EP was determined by flame photometry (Flame photometer 410, Sherwood) with the help of a calibration curve. Calibration solutions were prepared by the mixing of a small amount of very concentrated ethanolic KOH solution into the biodiesel matrix without potassium ions. The biodiesel matrix was prepared by the washing of biodiesel by citric acid aqueous solution.

The concentration of free glycerol was determined by HPLC method [Hájek et al., 2006].

3 Results and discussion

The most important experimental data are stated in Tab. 2. The reaction mixtures of experiments 8 and 14 could not be separated to the EP and GP at all at used conditions. The same problem was registered at ethanolsis of other vegetable oils in [Domingos et al., 2008], [Oliviera et al., 2008]. The mixtures from experiment 8 and 14 were separable only at a higher temperature (approximately 80°C).

3.1 Transesterification

Transesterification process was monitored by the determination of weight ratios of ethyl esters, soaps, MG, DG and TG in the total reaction mixture (EP and GP together) relative to the initial weight of the oil as the key component (see also 2.2). These variables do not depend on the separation but only on the transesterification process. The weight concentrations of TG, DG and MG only in the ester phase are presented in the chapter 3.3.

$$wr_{TG} = 0.19 - 0.13 \cdot T - 0.13 \cdot C - 0.18 \cdot MR \quad R^2 = 0.964 \quad (2)$$

$$wr_{EE} = 97.27 - 1.60 \cdot T - 0.38 \cdot C + 0.63 \cdot MR \quad R^2 = 0.941 \quad (3)$$

$$wr_S = 4.30 + 1.21 \cdot T + 0.89 \cdot C - 0.30 \cdot MR \quad R^2 = 0.962 \quad (4)$$

$$wr_{DG} = 1.00 - 0.33 \cdot T - 0.60 \cdot C - 0.62 \cdot MR \quad R^2 = 0.923 \quad (5)$$

$$wr_{MG} = 2.29 + 0.19 \cdot T - 0.51 \cdot C - 0.47 \cdot MR \quad R^2 = 0.903 \quad (6)$$

Weight ratio of TG (2) decreases with the increasing of T , C and MR , since the ethanol and catalyst are included in the kinetic equations of transesterification and

saponification of TG (see diagram of reaction system and kinetic equations from [Komers et al., 2002]).

Weight ratio of EE (3) decreases with the increasing of T and C and increase with the increasing of MR , while the weight ratio of soaps (4) is affected exactly vice versa. This is caused by the competition between the forming of EE and S (see Fig. 1). The soaps are mainly formed by the saponification of oil, but a minor part of the soaps is formed by the neutralization of free fatty acids (acid number of oil was $0.6 \text{ mg KOH}\cdot\text{g}^{-1}$) which corresponds with 5–14 % of total formed soaps.

The negative effect of the catalyst on the formation of esters is unexpected but a similar result was observed in the ethanolysis of cottonseed oil when the weight ratio of the KOH was over approximately 1.07 wt-% [Joshi et al., 2008]. The negative effect of the increasing temperature on the ester formation was also found in [Bouauid et al., 2007] but some authors observed negligible effects of the temperature [Domingos et al., 2008] or a positive effect [Encinar et al., 2007] on the ethanolysis of rapeseed oil. Probably, the effect of the catalyst and temperature on the formation of esters significantly depends on investigated intervals of these variables and other reaction conditions, so the results cannot be generalized.

The weight ratio of MG (6) increases with increasing T and decreases with increasing C and MR . The weight ratio of DG (5) is affected similarly except T . Decreasing of MG and DG concentrations with increasing C is caused by saponification because only soaps increase with amount of the KOH catalyst (4). On the contrary decreasing of MG and DG with increasing MR is caused by transesterification because only esters increase with that MR (3). These findings are valid only at the investigated interval of the

amount of the catalyst; too a low weight ratio of alkaline catalyst causes the deterioration of transesterification [Rashid et al., 2008]

Only weight ratio of MG is increasing with increasing T whereas TG, DG and EE are decreasing (soaps increase too). It may be caused by the positive reaction enthalpy of the equilibrium reaction of MG to EE (the last step of transesterification). The kinetic or thermodynamic data are not known for ethanolysis of vegetable oil; but in the case of methanolysis the reaction enthalpy of the equilibrium reaction of MG to esters (calculated from activation energies) is positive [Bambase, 2007].

The extension of MR has an unambiguously positive effect. Increasing MR decreases the weight ratios of MG, DG and TG in a desired direction – forming of EE. Increasing of the values of MR over the upper limit of the investigated interval (7.5:1) could be taken into consideration, especially in technologies which can simply recuperate the excess of ethanol.

The increasing of the catalyst (C) has debatable effects. More catalyst means that all glycerides are converted into products faster; but on the other hand more soaps are formed.

Higher reaction temperature (65 °C) is not suitable because of forming soaps in place of esters. The whole catalyst is saponified at 65°C after 1.5 or 2 hours, because carbonates and bicarbonates were not present in the glycerol phase. On the contrary, higher amounts of DG and TG remain in the reaction mixture at a lower temperature (25°C). Reaction time (t) has no significant effect on the reaction system. This may be caused by the narrow investigated interval of the reaction time (from 1.5 up to 2 hours). The second explanation is that the system of transesterification reactions is already after 1.5

hours in equilibrium and saponification is proceeding insignificantly after this reaction time.

Rotation frequency of disperser (f) has an insignificant effect on the reaction system at the investigated interval of rpm, more in the chapter 3.5.

The variables observed in this section (3.1) do not depend on separation and purification steps and could be compared with technological process with different separation.

3.2 Separation of phases

Separation of phases was monitored by the yield of the ester phase, weight of the glycerol phase and weight concentration of EE in the GP. The following equations were derived from the results:

$$Y_{EP} = 79.97 - 9.54 \cdot T - 4.67 \cdot C + 3.67 \cdot MR - 2.59 \cdot f \quad R^2 = 0.842 \quad (7)$$

$$m_{GP} = 274.8 + 90.1 \cdot T + 47.1 \cdot C - 34.3 \cdot MR + 23.1 \cdot f \quad R^2 = 0.841 \quad (8)$$

$$w_{EE(GP)} = 42.61 + 10.91 \cdot T + 3.71 \cdot C - 4.77 \cdot MR + 3.64 \cdot f \quad R^2 = 0.750 \quad (9)$$

The yield of biodiesel (7) decreases with increasing T , C and f and increase with increasing MR . The opposite effects is valid for the weight of the glycerol phase (8) because the weight sum of both phases must stay constant. Similar results are described in [Bouaid et al., 2009], where the yield of esters decreased when the temperature crossed over approximately 34°C, in [Rashid et al., 2008] and [Dorado et al., 2004], where esters yields decreased when the weight ratio of the catalyst got over approximately 0.8 - 1.0 wt-%. These findings confirm our model.

The increasing weight of the GP is caused by the remainder of ethyl esters in the GP, which follows from the similar form of equations (8) and (9). The remainder of EE in the GP is caused by the influence of soaps which can emulsify non-polar esters in the polar GP (the correlation between weight ratio of soaps and concentration of ethyl esters

in the GP has $R^2=0.75$). Therefore the elimination of saponification reaction is very important. Identical findings were found in [di Felice et al., 2008].

Higher rotation frequency of the disperser causes the deterioration of the separation of the EP and GP but the explanation of that effect is not known.

The maximal value of biodiesel yield reaches up to 92 %. But in some cases it reached only approximately 60 % and most often it was about 88 %. This was caused by an unexpectedly high remainder of ethyl esters in the GP, because the total yield of EE depends also on the separation of esters between the EP and GP (weight concentrations of esters in the GP exceeded 50 % and the weight of the GP was high in experiments with a low yield of biodiesel). However, the GP with a higher amount of esters was unstable and part of the esters was separated from the GP after several days (ca 10–30 % of weigh of GP).

The transport of EE from the polar GP to the non-polar EP is the great scope for increasing of the biodiesel yield. In the work [Vicente et al., 2004], the material balance of methanolysis of sunflower oil supported by various alkaline catalysts showed, that the significant part of the ester losses is caused by the remainder of esters in the GP.

Equations (7), (8) and (9) have significantly lower values of the square of the correlation coefficient (R^2) compared with the equations of transesterification (2) - (5).

This may be caused by the lower suitability of the used linear model.

3.3 Properties of the ester phase

The following important variables were monitored: weight concentrations of TG, DG and MG, potassium ions and free glycerol because they are the important qualitative properties of biodiesel. Acid number, density and kinematic viscosity were monitored,

too. Norm of fatty acids ethyl esters do not exist and data was compared with the norm of fatty acids methyl esters (EN 14214).

The following equations were obtained for TG, DG and MG concentrations:

$$w_{TG(EP)} = 0.170 - 0.067 \cdot T - 0.069 \cdot C - 0.121 \cdot MR + 0.037 \cdot f \quad R^2 = 0.899 \quad (10)$$

$$w_{DG(EP)} = 0.549 - 0.125 \cdot C - 0.143 \cdot MR + 0.035 \cdot f \quad R^2 = 0.940 \quad (11)$$

$$w_{MG(EP)} = 0.739 + 0.038 \cdot T - 0.158 \cdot C \quad R^2 = 0.947 \quad (12)$$

These equations are valid only for the zone of lower concentrations of glycerides because the data of higher concentrations were outliers.

The equations of the concentration of TG, DG and MG in the EP have a different form in the comparison with the weight ratios of TG, DG and MG in the chapter 3.1. This may be caused by different distribution coefficients between the ester and glycerol phase. For example, TG are typically non-polar substances but MG have emulsive properties (emulsive properties of MG are shown and utilized in [Striūgas et al., 2008]). Increasing of C and MR and decreasing of f is necessary for decreasing of glyceride concentration. But increasing T has the reverse effect on TG and MG and thereby it is impossible to recommend uniquely some suitable reaction temperature to decrease the glycerides concentration.

The concentrations of MG and TG fulfilled the EN 14214:2003 in the major part of experiments but the concentration of DG reached the norm value in no experiment (normed values are maximum 0.8 % for MG, 0.2 % for DG and 0.2 % for TG). To fulfil the norm of DG concentration, it is necessary to go over the upper limit of investigated intervals of some independent variables (C and MR). The second alternative is the decreasing of DG concentration by the purification of the ester phase.

$$w_{K(EP)} = 31.15 - 10.16 \cdot T + 4.00 \cdot MR \quad R^2 = 0.825 \quad (13)$$

$$w_{FG(EP)} = 0.207 - 0.107 \cdot T - 0.069 \cdot C \quad R^2 = 0.823 \quad (14)$$

The concentration of potassium ions and free glycerol depend primarily on the purification conditions and it was not the focus of this research. But the measured values in all experiments and equations (13) and (14) show unambiguously, that the purification of the ester phase is necessary because both values exceed the EU norm manifold (norm values are $5 \text{ mg} \cdot \text{kg}^{-1}$ for K^+ and 0.2 % for FG). The extremely high concentrations of potassium ions in the EP (after 24 hours) in the experiments 5, 8, 9 and 14 may be caused by the slow rate of the phase separation in these experiments. But this problem was not studied further.

The gravitational separation of phases was not finished after 24 hours. All prepared ester phases were turbid after this time (turbidity of ester phase at 452 nm was from 45 up to 95 % on refined biodiesel as standard). The decreasing of potassium ions and free glycerol concentrations is possible by an extension of the separation time or the using a centrifuge.

Another characteristic of the ester phase was determined. The acid number ranged between 0.01 and to $0.05 \text{ mgKOH} \cdot \text{g}^{-1}$ (norm value $0.5 \text{ mgKOH} \cdot \text{g}^{-1}$), density between 872 and $888 \text{ kg} \cdot \text{m}^{-3}$ (norm value from 860 up to $900 \text{ kg} \cdot \text{m}^{-3}$), kinematics viscosity at $40 \text{ }^\circ\text{C}$ about $4.7 \text{ mm}^2 \cdot \text{s}^{-1}$ (norm value from 3.5 up to $5 \text{ mm}^2 \cdot \text{s}^{-1}$).

3.4 Verification and reviewing of linear models

The found linear models were verified by several independent experiments (Tab. 3).

The correspondence between the predicted and measured data was characterized by error (15) which was calculated as the absolute difference between the measured and

predicted values divided by the dispersion of data used in regression (maximal measured value minus minimal measured value).

$$error = \frac{|x_{experimental} - x_{predicted}|}{x_{max} - x_{min}} \cdot 100 \quad (15)$$

The error values show the sufficient consistency of predictions and experiments and thereby the found linear models are applicable. The worst correspondence between the predicted and experimental data was found in the case of free glycerol in the EP at the verification experiment 1 (error 39.2 %). Then it follows that found model of free glycerol – equation (14) – has not a good prediction ability (prediction quality of models with R^2 less than 0.9 is not good).

The found linear models easily enable us to review the positive or negative effects of individual independent variables on the dependent variables. But do not enable us to specify the optimum reaction conditions. For example, the increasing weight ratio of the catalyst decreases the weight ratios of MG, DG and TG in the reaction mixture, but on the other hand decreases the yield of the ester phase and increases costs. The optimization of the ethanolsis process is not possible without the determination and the calculation of the main optimization criterion of each process – costs. But this was not the aim of this work. This work may assist in the mentioned optimization.

3.5 Comparison between disperser and classical stirrer

According to the chapter 3.1, the transesterification is not influenced by the rotation frequency of the disperser (f) at the investigated interval (from 10000 up to 20000 rpm). Therefore comparative experiments with different stirrers were made: one with the disperser at 10000 rpm and the other with the classical toothed disc at 800 rpm. The others condition (temperature, time etc) were the same. The results show (Tab. 4) that

the using of the disperser decreases weight ratios of MG, DG and TG and increases the weight ratio of ethyl esters, so the disperser has undoubtedly a positive effect in comparison with the classical stirrer. But increasing of its rotation frequency over 10000 rpm has a negative effect on the yield of biodiesel (see 3.1 and 3.2).

3.6 Deethanolisation

The residual ethanol affects the flash point of biodiesel, which is an important safety factor of fuels. EN 14214 permits flash point above 120 °C. The flash point of biodiesel is influenced primarily by residual alcohol [Mittelbach and Remschmidt, 2004].

Thereby the suitable degree of deethanolisation (evaporation at low pressure) is necessary. The deethanolisation of reaction mixture was carried out at constant time (40 min), constant pressure (3 kPa) and various temperatures. The deethanolisation temperature of 80 °C caused the flash point of subsequently formed ester phases varied in the interval 80–90 °C, 85 °C caused variation 100–110 °C and 90 °C caused variation 120–130 °C. The relatively wide intervals of the flash point values reached at the identical temperature of deethanolisation are caused by the impossibility of keeping an exactly identical pressure in the apparatus. The suitable temperature of deethanolisation is 90 °C to fulfill the EU norm for the flash point (120 °C).

4 Conclusions

The increasing of the molar ratio ethanol:oil has a positive effect unambiguously: it decreases the concentrations of glycerides and soaps in the reaction mixture and increases the ethyl ester concentration in the mixture and the yield of biodiesel.

The increasing of the weight ratio of the catalyst has ambiguous effects. The glycerides are converted faster and with a higher extent, unfortunately not only to biodiesel; a

relatively greater amount of undesirable soaps is formed. The increasing of temperature causes also a higher forming of soaps. The disperser has positive a effect on the transesterification process if the speed of its rotations is lower than 10000 rpm.

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Conflict of interest statement

The authors have no conflict of interest.

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Figure captions:

Fig. 1: The diagram of the reaction system of the alkaline-catalyzed transesterification

Fig. 2: Apparatus