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Article

Microwave Assisted Biodiesel Production from Trap Grease

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Ésteres metílicos e etílicos foram preparados a partir de ácido láurico, sob aquecimento convencional e irradiação de microondas para determinar as melhores condições de esterificação de ácidos graxos. Estas condições foram então aplicadas na conversão de resíduo de caixa de gordura a biodiesel. Este resíduo é uma matéria-prima potencial para a produção de biodiesel, devido ao seu baixo custo e fácil esterificação. A esterificação catalítica do resíduo com metanol foi realizada sob aquecimento convencional e irradiação de microondas. Foi possível obter conversões elevadas com o aquecimento convencional, mas sob aquecimento por microondas, o equilíbrio da reação de esterificação foi atingido muito mais rapidamente. O biodiesel obtido a partir do resíduo em reações com irradiação de microondas mostrou uma alta conversão (96%), mesmo em condições brandas de reação (razão molar gordura:metanol 1:6, 1,0% H_2SO_4, 393 K, 10 minutos). Embora estas experiências iniciais tenham sido realizados com um aparelho de laboratório de pequena escala, o procedimento poderia ser facilmente adaptado para um processo comercial contínuo.

Methyl and ethyl esters were prepared from lauric acid under conventional heating and microwave irradiation to determine the best fatty acid esterification conditions. These conditions were then applied in the conversion of trap grease (brown grease) to biodiesel. The trap grease is a potential feedstock for biodiesel production, due to its low cost and easy esterification. In this paper, trap grease catalytic esterification with methanol was performed under conventional heating and microwave irradiation. It was possible to obtain high conversions with conventional heating, but under microwave heating, the esterification reaction equilibrium was attained much faster. The trap grease biodiesel obtained from microwave assisted reactions showed a high conversion (96%) even at mild reaction conditions (trap grease:methanol molar ratio 1:6, 1.0% H_2SO_4, 393 K, 10 minutes). While these initial experiments were performed in a small-scale laboratory unit, the procedure could be easily scaled-up to a commercial continuous process.

Keywords: biodiesel, esterification, trap grease, microwave

Introduction

Since the 1973 and 1978 oil crises, various alternative energy sources have been studied to replace products derived from oil. Nowadays, the great instability related to oil prices is fuelling that search again. Besides the economic aspect, there is the fact that fossil fuels are the main contributor to the greenhouse effect.

In this context, there is an intense interest in the development of renewable fuels, both clean and economically viable.[1-4] Among several alternatives to diesel oil, biodiesel is a substitute produced from fatty materials and short-chain alcohols. A barrier to the production of biodiesel on a commercial scale is the fact that its final cost is higher than that of diesel oil. The main reason for this high cost is the high price of virgin vegetable oils[5] or high-quality animal fats,[6] which constitute 75% of the total biodiesel production cost.

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Some strategies have been developed to make biodiesel production economically attractive, such as tax incentives. Moreover, it is necessary to seek out new raw material sources at a lower cost, such as high acid residual oils and fats of inferior quality.

The classic process of biodiesel production is based on alkaline transesterification of oils or fats with short chain alcohols. In this process, it is essential that the free fatty acids (FFA) are previously neutralized and removed, because they react with the catalyst to form soap, reducing the efficiency of the process. This method is used for oils or fats with a FFA content between 0.5 and 5.0% (by weight). For raw materials with more than 5.0% FFA, the strategy is to convert the FFA fraction into biodiesel separately. In this case, the conversion to biodiesel is conducted in two consecutive steps: first, the esterification of FFA is catalyzed by a mineral acid, and then a conventional step of alkaline transesterification to produce esters from triglycerides.[7] When the fatty acid content is above 20%, it is more economical to use only one acid catalyst step under conditions where both the esterification and transesterification reactions occur simultaneously.

Canakci and Van Gerpen[8] studied this technique to convert the FFA in feedstock materials. The process was developed for pre-treating feedstocks with high FFA content using an acid catalyst until FFA content fell below 1%. The second step was a transesterification reaction to convert the remaining triglycerides to biodiesel. The authors studied the effect of methanol concentration, catalyst concentration and reaction time. They used a test material for this experiment that consisted of a mixture of 20% palmitic acid in soybean oil. The mixture presented an acid number of 41.3 mg KOH *per* g. In a typical test, the authors reacted methanol with the FFA-rich mixture in a 1:9 molar ratio of oil to methanol with 5% sulfuric acid as catalyst, a reaction time of one hour and a temperature of 338 K. Under these conditions, the acid number was reduced to 1.77 mg KOH *per* g, which indicated an FFA conversion of 95.7%. After the acidity of the oil was reduced to less than 2.0 mg KOH *per* g at the end of the first stage, the alkaline transesterification was performed. In this stage, several alkaline catalysts were tested, including KOH, NaOH, sodium methoxide and metallic sodium. The results obtained in this series of tests showed that sodium methoxide was more tolerant to residual acidity than NaOH or KOH were, with both requiring an oil with an acid number less than 1 mg KOH *per* g to produce good quality esters (high triglyceride conversion).

Hasuntree *et al.*[9] investigated the possibility of using restaurant trap grease as feedstock in the production of biodiesel via acid catalyzed esterification. In their study, sulfuric acid was used as a catalyst for the esterification reaction of FFA and methanol. The FFA levels of restaurant trap greases were reduced from 60.38 ± 2.22 mg KOH *per* g to 11.60 ± 1.60 mg KOH *per* g when conditions for biodiesel production were as follows: methanol to FFA ratio of 5:1, 5.0 wt.% H_2SO_4, and a reaction temperature at 333 K with a reaction time of 60 minutes. During the acid-catalyzed esterification, the percentage of methyl esters resulting from conversion of FFA in the obtained product was 83.59 ± 1.51% based on the result of ^{1}H nuclear magnetic resonance (NMR) analysis.

Park *et al.*[10] investigated the feasibility of the production of biodiesel from trap grease containing 51.5% FFA. The authors proposed a two-step process: the esterification of FFA by an acid catalyst (H_2SO_4 or Amberlyst 15) followed by the transesterification of the remaining triglycerides by an alkali catalyst (KOH). After the esterification of the trap grease using 0.5 wt.% sulfuric acid and a molar ratio of methanol to oil of 9:1, the acid value decreased from 102.9 to 2.75 mg KOH *per* g (97.3% FFA conversion). The product with 2.75 mg KOH *per* g acid value was then transesterified using potassium hydroxide. With a molar ratio of methanol to oil of 6:1, 0.8 wt.% of potassium hydroxide, 353 K and 0.5 h, fatty acid methyl ester (FAME) content reached 92.4%. In the esterification test using 21% of Amberlyst 15, a molar ratio of methanol to oil of 8.37:1, 368 K and 3 h, the acid value decreased to 3.23 mg KOH *per* g. With the transesterification using 1.2% potassium hydroxide, a molar ratio of methanol to oil of 6:1, 353 K and 0.5 h, FAME content increased to 94.1%.

Charoenchaitrakool *et al.*[11] developed a similar study to investigate the optimum conditions in biodiesel production from waste frying oil using two-step catalyzed process. In the first step, sulfuric acid was used as a catalyst for the esterification reaction of free fatty acid and methanol in order to reduce the free fatty acid content to be approximate 0.5%. In the second step, the product from the first step was further reacted with methanol using potassium hydroxide as a catalyst. The best results for biodiesel production were obtained using a methanol to oil molar ratio of 6.1:1, 0.68 wt.% of sulfuric acid, 324 K and a reaction time of 60 minutes in the first step, followed by using molar ratio of methanol to product from the first step of 9.1:1, 1 wt.% KOH, 328 K and a reaction time of 60 minutes in the second step. The percentage of methyl ester in the final product was 90.56 ± 0.28%.

Lu *et al.*[12] conducted a study using a 100 t *per* year biodiesel pilot scale production system, mainly consisting of a fixed-bed and a down-stream plug-flow reactors. The unit was setup to test different feedstock oils, especially trap grease, for their feasibility as biodiesel feedstock. The following raw materials were used in the tests: rapeseed oil, chinese wood oil, and trap grease. A type of ion-exchange resin was used to fill in the fixed-bed reactor and used as the esterification catalyst for pretreating of the highly acidified oil. With a fresh catalyst, the acid value of trap grease could be reduced from 114.0 to about 2.0 mg KOH *per* g (this corresponding to approximately 98% conversion, after 13 h at 348 K, catalyst load of 15 wt.% of oil, methanol addition of 20 wt.% of oil).

The current research on biodiesel is not only focused on the search for alternative raw materials,[6] but it also includes the search for more efficient catalysts with reduced waste, the development of simpler methods of biodiesel purification and the commercial application of glycerin.

The use of microwaves to provide heat for reactions is one of the alternative methods for developing the synthesis of biodiesel. Microwave-assisted reactions[13-16] have shorter reaction times and good yields, sometimes with a drastic reduction in the formation of co-products.

In this context, Mazzocchia *et al.*[17] conducted a series of transesterification reactions under microwave irradiation. The tests were performed in a Milestone Ethos 1600 unit with an output of up to 1000 W. The transesterification reactions were carried out on rapeseed oil with barium hydroxide as the catalyst and methanol as the alcohol. The tests were conducted with a rapeseed oil to methanol molar ratio of 1:9, 1:18 and 1:30 and with different catalyst concentrations. The best result obtained by the author was 99% conversion, with an oil:alcohol molar ratio of 1:9, 1.5% catalyst loading, 3.5 bar and 376 K for 10 minutes.

In another study by Lertsathapornsuk *et al.*,[18] a domestic microwave oven (Ecolux, 800 W, 2450 Hz) was modified and used as a continuous reactor in biodiesel synthesis from waste palm oil containing 4.5% FFA. The oil was treated with an alkaline alcoholic solution containing 3.0% NaOH in ethanol. Part of the added NaOH was used in the neutralization of the FFA, and the rest served as a catalyst for the transesterification reaction. Initially, the authors used an oil:alcohol molar ratio of 1:6 and 3.0% NaOH for 30 seconds, which led to a conversion of only 30% of the residual oil. When the oil:alcohol molar ratio was increased to 1:9 and all other conditions were kept constant, the conversion of residual oil increased to 82%. In the same way, a third test was performed with an oil:alcohol molar ratio of 1:12 and the conversion reached over 97%.

In this work, a laboratory microwave (MW) unit was used to obtain biodiesel from lauric acid. The reaction parameters of temperature, lauric acid:alcohol molar ratio, reaction time, catalyst concentration and type of alcohol (methanol or ethanol) were studied. For comparison, conventional tests were also carried out in a conventional autoclave reactor in addition to the microwave tests.

Experimental

Reagents

The following reagents were used in the experiments: lauric acid PA (Vetec), trap grease collected directly in IME facilities, methanol PA (Vetec) and ethanol PA (Vetec). Sulfuric acid 98% (Vetec) was used as the catalyst and *N*-methyl-*N*-trimethylsilyl trifluoroacetamide (MSTFA) was used as a derivatizing agent.

Apparatus

Tests were conducted in a Discover microwave lab unit (CEM) operating with programmable power from 1 to 300 W. A 50 mL glass reactor was used with magnetic stirring set at 400 rpm. The pressure in these tests was essentially the vapor pressure of the alcohol (ethanol or methanol) at the test temperature, with the system being able to operate at a maximum pressure of 10 bar. The conventional heating tests were conducted in a steel autoclave (Parr Instruments) with an internal Teflon reaction recipient (45 mL) equipped with magnetic stirring, also set at 400 rpm. The autoclave was heated by a silicone oil bath. The pressure in these tests was essentially the vapor pressure of the alcohol (ethanol or methanol) at the test temperature. This second system could operate with a maximum pressure of 200 bar.

In this study a standard 10 mL borosilicate vessel was used in the reaction between grease/lauric acid and alcohol under H_2SO_4 catalysis and submitted to MW irradiation (Monowave 300 or CEM Discover) reactor and the temperature was monitored by an infrared sensor and/or a fiber optics probe. The microwave power applied was the fast possible mode offered by the Monowave 300 software.

Analytical procedures

The acid number of the product of the reaction was determined using the American Oil Chemists' Society (AOCS) Te 1a-64 method. As the acid number of lauric acid is 279 mg KOH *per* g and the biodiesel (ester) is not an acid, the decrease in this value was directly proportional to the progress of the reaction, and the conversion could be calculated using the following equation:

$$\%\ \text{Conversion} = 100 \times (279 - \text{acid number of the product}) / 279 \quad (1)$$

Hydrogen nuclear magnetic resonance (^{1}H NMR) was used to characterize the biodiesel products. The spectra were recorded on a Varian spectrometer, model Unity 300 (300 MHz). Deuterated chloroform and tetramethylsilane

(TMS) were used as solvent and internal reference, respectively. The conditions used were 303 K, 5-mm diameter tubes and 80 seconds of acquisition time.

In the ^{1}H NMR spectrum, ethyl biodiesel was characterized by the presence of a quartet around 4.1 ppm, corresponding to the –CH2–O– hydrogens. The methyl biodiesel showed a characteristic singlet at 3.6 ppm, corresponding to the methoxy hydrogens.

Gas chromatography coupled to mass spectrometry (GC-MS) analysis was performed by using a modified method based on EN 14105. Free fatty acids and ethanol were transformed into more volatile silylated derivatives in the presence of pyridine and MSTFA. All GC-MS measurements were carried out in duplicate using a DB 5-HT (Agilent, J & W Scientific®, USA) capillary column (10 m × 0.32 mm × 0.1 µm). The GC-MS samples were prepared by dissolving 0.1 g of the final product in 1 mL of *n*-heptane. One hundred µL of this solution and pyridine solutions of butanetriol (1 mg mL^{-1}) and tricaprine (8 mg mL^{-1}), used as internal standards, were added to a flask followed by addition of 100 µL of MSTFA. After 15 minutes, these reactants were dissolved in 8 mL *n*-heptane. One µL of this sample was then injected into a Shimadzu CG2010 equipment. The quantification was done based on calibration curves of the internal standards.

Results and Discussion

For all tests, the biodiesel (ester) production was confirmed by ^{1}H NMR analysis and gas chromatography coupled to mass spectrometry of the isolated products.

Lauric acid esterification

Both microwave assisted and conventional heating tests were performed with methanol and ethanol, lauric acid:alcohol molar ratios of 1:3, 1:6, 1:8 and 1:12 and catalyst concentrations of 2.5, 5.0 and 7.5%. The temperature was varied from 393 to 403 K and the reaction time varied from 5 to 60 minutes. The results obtained with microwave heating and conventional heating are listed in Table 1 and 2, respectively.

Influence of the lauric acid:methanol molar ratio

In the microwave assisted tests 1 to 4 (Table 1), the lauric acid:methanol molar ratio was varied from 1:3 to 1:12, with all other reaction conditions kept constant. Clearly, the conversion of lauric acid to biodiesel increased with increasing amounts of methanol, with the highest conversion observed for the highest molar ratio of 1:12. This result was consistent with the fact that the esterification reaction is a reversible reaction, with the product formation being promoted by an increase in the concentration of one of the reagents.[9]

In the conventional heating tests 26, 16 and 17 (Table 2), the lauric acid:methanol molar ratio was varied from 1:6 to 1:12, respectively, with all other reaction conditions kept constant. Comparing these results with those obtained with microwave heating, the same trend of increasing ester conversion with increasing molar ratio was observed. Additionally, the conversions obtained with microwave irradiation were slightly higher than those obtained by conventional heating.

Table 1. Microwave assisted biodiesel production tests

entry	Type of alcohol	Molar ratio[a]	Reaction time / min	Temperature / K	Catalyst (H_2SO_4) /%	Conversion /%[b]
1	Methanol	1:3	30	393	5	89
2	Methanol	1:6	30	393	5	92
3	Methanol	1:8	30	393	5	94
4	Methanol	1:12	30	393	5	96
5	Methanol	1:6	30	393	2.5	88
6	Methanol	1:6	30	393	7.5	93
7	Methanol	1:8	30	393	7.5	94
8	Methanol	1:12	30	393	7.5	96
9	Methanol	1:6	5	393	5	95
10	Methanol	1:6	15	393	5	92
11	Methanol	1:6	60	393	5	92
12	Methanol	1:8	30	403	5	94
13	Methanol	1:12	30	403	5	95
14	Ethanol	1:8	30	393	5	94
15	Ethanol	1:12	30	393	5	95

[a]Molar ratio of lauric acid:alcohol; [b]based on acid number of the product.

Table 2. Conventional heating biodiesel production tests

entry	Type of alcohol	Molar ratio[a]	Reaction time / min	Temperature / K	Catalyst (H_2SO_4) /%	Conversion /%[b]
16	Methanol	1:8	30	393	5	94
17	Methanol	1:12	30	393	5	95
18	Methanol	1:8	30	393	7.5	94
19	Methanol	1:12	30	393	7.5	95
20	Ethanol	1:8	30	393	5	93
21	Ethanol	1:12	30	393	5	95
22	Ethanol	1:8	30	393	7.5	95
23	Ethanol	1:12	30	393	7.5	96
24	Methanol	1:6	5	393	5	74
25	Methanol	1:6	15	393	5	87
26	Methanol	1:6	30	393	5	91
27	Methanol	1:12	5	393	5	86
28	Methanol	1:12	15	393	5	90

[a]Molar ratio of lauric acid:alcohol; [b]based on acid number of the product.

Influence of the catalyst concentration

The previous protocol was repeated in tests 5 through 8, but with the sulfuric acid concentration varying from 2.5 to 7.5%. The result obtained with 2.5% of catalyst (test 5) was inferior to the results for the reactions performed under the same conditions with 5.0 and 7.5% (tests 2 and 6, respectively). However, it was observed that the conversions obtained with 5.0 and 7.5% of sulfuric acid were practically the same.

Repeating the previous protocol but using conventional heating, the sulfuric acid concentration was then varied from 5.0 to 7.5%. For methanol, as observed in the microwave tests, increasing the amount of catalyst did not significantly alter the conversion of biodiesel (tests 16 and 18, and tests 17 and 19). For ethanol, the results from tests 20 and 22, and 21 and 23, indicated a small increase in the conversion values when the sulfuric acid concentration increased from 5.0 to 7.5%.

Influence of the reaction time

In tests 9 through 11, the same standard reaction conditions were kept constant (300 W, lauric acid:methanol molar ratio of 1:6, 5.0% sulfuric acid and 393 K), but the reaction time was varied from 5 to 60 minutes. As can be observed for the conversion results, increasing the reaction time from 5 to 60 minutes led to no significant increase in biodiesel production. Most likely, the chemical equilibrium for these conditions was reached between 5 and 15 minutes of reaction time.

With conventional heating (tests 24 to 28), increasing the reaction time from 5 to 15 minutes (tests 24 and 25, respectively) led to an increase in biodiesel production. The conversions obtained at reaction times of 5 and 15 minutes were much lower than the results obtained for the microwave-assisted reactions under the same conditions, indicating the better efficiency of microwave heating.

Influence of temperature

The influence of the reaction temperature was studied only for microwave-assisted reactions. The study was conducted at lauric acid:methanol molar ratios of 1:8 and 1:12, varying the temperature from 393 to 403 K. The other reaction conditions were 300 W, 5.0% sulfuric acid and a reaction time of 30 minutes.

As the methyl esterification reaction is endothermic,[19] it was expected that a temperature increase would displace the reaction chemical equilibrium towards ester formation. However, comparing tests 3 and 4 (393 K) and 12 and 13 (403 K), it was observed that, for this temperature range and other conditions employed, the conversions did not change significantly.

Influence of the type of alcohol

Tests 14 and 15 were doubles of entries 3 and 4 using ethanol as the esterification agent in microwave assisted reactions. The conversion results obtained with ethanol were similar to values obtained for the equivalent methyl esterifications.

In tests 20 through 23, ethanol was used in the esterification under conventional heating. In this set of reactions, the reaction conditions used were: molar ratios of lauric acid:alcohol of 1:8 and 1:12, catalyst concentration of 5 and 7.5%, reaction time of 30 minutes and temperature of 393 K. The observed ethyl esterification conversion values were essentially the same values obtained for the methyl esterification.

Compared with the microwave test results for both methanol and ethanol, conventional heating had essentially the same levels of conversion with these reaction conditions.

Microwave-assisted esterification was compared with conventionally heated esterification for different reaction conditions. The main difference between the two sets of experiments is that the conventional heating tests usually require a longer reaction time to achieve the same level of conversion of the microwave-assisted tests. This large difference in reaction time can be attributed to the limitations of conventional heating in which the energy is first utilized to increase the temperature of the reaction vessel. Despite these observations, this comparison requires a study on the economic aspects, such as energy consumption, equipment, manpower, so we can say that microwave-assisted reactions are better than conventional heating.

Trap grease esterification

Using the information acquired with the lauric acid esterification tests, a new set of tests was performed using trap grease with both microwave and conventional heating.

The trap grease reactions were performed only with methanol, using molar ratios of 1:3, 1:6 and 1:8 and catalyst concentrations from 0.5 to 5.0%. The temperature was kept at 393 K and the reaction time varied from 5 to 15 minutes. The results obtained with microwave heating are listed in Table 3.

Influence of the trap grease:methanol molar ratio

In tests 29 to 37, the trap grease:methanol molar ratio was varied from 1:3 to 1:8, with all other reaction conditions kept constant. As can be seen, the trap grease conversion to biodiesel increased with increasing amounts of methanol, with the highest conversion observed for the highest molar ratios of 1:6 and 1:8, with no significant increase in conversion when the molar ratio increases from 1:6 to 1:8.

Influence of the catalyst concentration

The previous protocol was repeated for entries 33, 39, 41 and 42, but with the sulfuric acid concentration varying from 0.5 to 5.0%. The conversion increases with the catalyst concentration, but there is little difference between the results obtained with 2.5 and 5.0% of sulfuric acid. Due to these results, all other tests were conducted using 5.0% sulfuric acid.

Influence of the reaction time

In tests 30, 33 and 36, the reaction conditions were kept constant (300 W, trap grease:methanol molar ratio of 1:6, 5.0% sulfuric acid and 393 K), varying only the reaction time from 5 to 15 minutes. With these reaction conditions, the conversion of trap grease to biodiesel is already very high with 5 minutes (97%), with no significant variation for reaction times over 10 minutes.

Additionally, three conventional heating tests were done using trap grease and the reaction conditions of tests 30, 40 and 41 (393 K, reaction times from 5 to 15 minutes and catalyst concentration from 1.0 to 5.0%). These conditions

Table 3. Microwave assisted biodiesel production tests (from trap grease)

Input	Molar ratio[a]	Reaction time / min	Temperature / K	Catalyst (H_2SO_4) /%	Conversion /%[b]
29	1:3	5	393	5.0	37
30	1:6	5	393	5.0	97
31	1:8	5	393	5.0	99
32	1:3	10	393	5.0	40
33	1:6	10	393	5.0	99
34	1:8	10	393	5.0	97
35	1:3	15	393	5.0	97
36	1:6	15	393	5.0	99
37	1:8	15	393	5.0	96
38	1:6	5	393	2.5	75
39	1:6	10	393	2.5	98
40	1:6	15	393	2.5	99
41	1:6	10	393	1.0	96
42	1:6	10	393	0.5	92

[a]Molar ratio of trap grease:alcohol; [b]based on GC-MS analysis.

Table 4. Biodiesel from trap grease: microwave-assisted and conventional heating

Molar ratio[a]	Reaction time / min	Catalyst (H_2SO_4) /%	Microwave-assisted conversion /%[b]	Conventional heating conversion /%[b]
1:6	5	5.0	97	76
1:6	15	2.5	99	68
1:6	10	1.0	96	50

[a]Trap grease:methanol molar ratio; [b]based on GC-MS analysis. All tests at 393 K.

were chosen to show how superior microwave heating is under short time and/or low catalyst concentration conditions. The results are indicated in Table 4.

Comparing the results obtained for both heating methods under the same reaction conditions, it is clear that the microwave assisted tests presented much higher conversions to biodiesel for short reaction times and low catalyst concentrations. Particularly, it was observed that the reaction is more responsive to catalyst concentration under microwave heating, probably due to the high microwave absorption capacity of the sulfuric acid. With 5.0% sulfuric acid and trap grease:methanol molar ratio of 1:6, only 5 minutes are necessary to achieve 97% conversion.

Conclusions

Microwave heating has proved to be very efficient for the conversion of fatty acids to esters in the biodiesel synthesis. High conversions were easily obtained, even with moderate reaction conditions such as a FFA:methanol molar ratio of 1:6, 2.5% of sulfuric acid catalyst, a reaction time of 15 minutes and temperature of 393 K. Moreover, no significant difference was observed in using methanol or ethanol in the esterification reactions.

Under microwave heating, the esterification reaction equilibrium was attained much faster. Using the standard test conditions, equilibrium was reached within 15 minutes under microwave heating, but only after 30 minutes with conventional heating.

Increasing the amount of alcohol shifts the reaction chemical equilibrium towards ester formation, increasing the conversion. The system was less sensible to increases in the catalyst amount, with no significant influence observed for values over 5.0%. No significant change was observed for varying the reaction temperature from 393 to 403 K in the microwave tests.

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