

A PRELIMINARY INVESTIGATION CONCERNING METAL OXIDES AS CATALYSTS FOR ESTERIFICATION OF LAURIC ACID WITH ISOPROPANOL

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Abstract. This paper reports the synthesis of isopropyl laurate while using a mixture of metal oxides as a catalyst. The best conversion value was obtained in the following conditions: 393 K, isopropanol:lauric acid molar ratio is 10:1, composition of the catalyst is 33 % CuO, 33 % Ag₂O and 33 % Al₂O₃, catalyst/lauric acid ratio is 8 % (w/w), and reaction time 2 h. Isolation of the catalysts after esterification showed that the oxides were not converted into other materials.

Keywords: metal oxide, isopropyl laurate, esterification, lauric acid, isopropanol.

1. Introduction

The development of more efficient methods to synthesize isopropyl laurate has been gaining relevance in the scientific community in the recent years, especially because this compound can be used in the composition of different products, such as pigments, tonners and viscosity adjustors [1, 2]. The high demand for the products cited above, together with the unfavorable thermodynamic factors regarding isopropyl laurate synthesis, justifies its high commercial value (US\$ 852.84 *per gram*) [3]. Despite the high price of this compound, it can be synthesized through a reaction between lauric acid and isopropanol, two low priced and easily obtained compounds.

There are several methods to synthesize this ester. The most usually described, according to the literature [4] is the esterification reaction catalyzed by an inorganic acid, generally, sulfuric acid. Although it presents a satisfactory conversion [4, 5] the separation between the products and the catalyst is difficult, since it is a homogeneous catalyst. Furthermore, it leads to a huge problem from the environmental point of view: the generated residues from the catalyst washing process are

extremely toxic for the environment. They also present high carcinogenic activity in humans [6, 7].

An alternative to the esterification with sulfuric acid is the use of heterogeneous catalysts such as inorganic oxides. Several cases of esterification with inorganic oxides have been reported in the literature [8-10]. In these cases, not only the catalyst separation is performed in an easy manner (because the catalyst is a solid and immiscible material in the reaction system), but the used oxides are Lewis acids of lower toxicity in relation to sulfuric acid. Therefore, the damage to the environment is greatly reduced [7]. However, with the use of heterogeneous catalysts, more extreme conditions regarding the temperature and reagents are necessary in order to obtain a satisfactory reaction yield. Despite the adversities cited above, the possibility of the catalyst recovery and the decrease of environmental impact make up those disadvantages.

In order to perform an organized study about the reaction conditions of an esterification system, it is necessary to use a factorial design – a very useful statistic methodology. The factorial design was successfully used, according to the literature, to study several esterification reactions [11].

While there are several studies that analyzed heterogeneous catalyst in esterification reactions with many acids and organic alcohols, there are no descriptions in the literature that include a ternary mixture of oxides as a catalyst for an esterification system. It is also important to mention that, although many different types of catalysts showed very high catalytic activity in esterification reactions, the synthesis of these highly active compounds uses very expensive reagents, which are very time-consuming and demand specific apparatus that many starting laboratories cannot afford. In this context, it is preferable to use catalysts that are relatively cheap, and without complex stages of synthesis/preparation (such as some metal oxides).

Therefore, the objective of this work was to evaluate, through experiments suggested by a factorial design, how a ternary oxide mixture acts in the catalysis of

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esterification of lauric acid with isopropanol. After determining the proportions between the chosen oxides that lead to the highest conversion gain compared to the reaction without catalysts. The other reaction conditions were studied with this oxide mixture and another factorial design.

2. Experimental

2.1. Preparation of Catalysts

The chosen oxides for the esterification reactions were based on: (1) the research and analysis of previous papers involving esterification reactions with lauric acid; and (2) the assumption that such oxides must not lose their structural characteristics after the esterification reaction. Therefore, the selected oxides for this study were CuO, Al₂O₃ and AgO. These products, acquired from Sigma-Aldrich, VETEC and Synth, were previously dried at approximately 423 K, in order to eliminate adsorbed water molecules on the respective oxide surfaces.

2.2. Tests with Catalysts

Primarily, seventeen esterification experiments (generated from a mixture factorial design) were executed in order to determine which used oxide proportion has the best catalyst activity in the isopropyl laurate synthesis. All reactions were executed at 393 K, with 10:1 isopropanol:lauric acid molar ratio and 8 % catalyst (in comparison to the total lauric acid mass) and 2 h of reaction time. This amount of the catalyst had its composition changed according to the factorial design presented in Table 1.

The esterification tests were performed in a Büchiglassuster miniclave drive pressurized steel reactor with an external rotation control at 500 rpm. Temperature control was achieved with a circulatory heating system (model Julabo HE-4) coupled to the reactor. Based on previous tests, 500 rpm was chosen as the ideal stirring value in order to ensure a potential impact of mass transfer, and because higher stirring values (750 rpm, 1000 rpm, *etc.*) did not lead to a significant increase in the acid-ester conversion.

The reactions were conducted through the following steps: the lauric acid, isopropanol and catalyst were introduced into the reactor, which was tightly sealed and heated, reaching a reaction temperature of 393 K in approximately 15 min. At the end of the scheduled period for reaction (2 h), the system temperature was decreased with the aid of a ventilator for about 20 min until mild condition and the reaction mixture was transferred to 100 ml volumetric flask. The excess alcohol was removed by a rotary evaporation under reduced pressure, at 353 K for isopropanol. The system pressure was governed by the vapor pressure of isopropanol, the most volatile component in the reaction medium, for reaction temperatures of 393 K, the pressure was of 200 kPa.

The conversion of lauric acid into isopropyl laurate was measured by the remaining acid quantification methodology, Ca-40-method of the American Oil Chemist's Society (AOCS) that uses NaOH solution of 0.1 mol/l, standardized with potassium biftalate. The results were expressed as a percentage conversion to ester, due to the use of a well established commercial sample, in this case the tested lauric acid.

Table 1

Factorial design experiments

Exp	CuO (%)	Ag ₂ O (%)	Al ₂ O ₃ (%)
1	100.0	0.000	0.000
2	66.66	33.33	0.000
3	66.66	0.000	33.33
4	33.33	66.66	0.000
5	33.33	33.33	33.33
6	33.33	0.000	66.66
7	0.000	100.0	0.000
8	0.000	66.66	33.33
9	0.000	33.33	66.66
10	0.000	0.000	100.0
11	66.66	16.66	16.66
12	16.66	66.66	16.66
13	16.66	16.66	66.66
14	0.000	33.33	66.66
15	50.00	50.00	0.000
16	50.00	0.000	50.00
17	0.000	50.00	50.00

Table 2

Factorial design experiments (second stage)

Exp.	Temperature, K	Molar ratio	Time, h
1	393	6	2
2	413	6	2
3	393	10	2
4	413	10	2
5	393	6	3
6	413	6	3
7	393	10	3
8	413	10	3
9	386.18	8	2.5
10	419.82	8	2.5
11	403	4.64	2.5
12	403	11.36	2.5
13	403	8	1.66
14	403	8	3.34
15	403	8	2.5
16	403	8	2.5
17	403	8	2.5

2.3. Chemical Characterization

In order to characterize the used oxides, the technique of x-ray powder diffraction (XPRD) was used. Experiments were conducted in a Shimadzu XDR-6000 equipment, using $\text{CuK}\alpha$ radiation of $\lambda = 1.5418 \text{ \AA}$, current of 30 mA, tension of 40 kV and a dwell time of 2 K/min. The samples were placed in glass sample holders. The vibrational modes present in the compounds were analyzed by a Fourier transform infrared spectroscopy (FTIR). Measurements were made in KBr (spectroscopic grade, Vetec) discs and collected in a Bio-Rad FTS 3500GX spectrophotometer, in the range of $400\text{--}4000 \text{ cm}^{-1}$, with a resolution of 4 cm^{-1} and accumulation of 32 scans.

To verify that the catalysts structures (both before and after reactions) were consistent with that of metal oxides, all obtained compounds were analyzed once again by XPRD and FTIR.

2.4. Determination of Reaction Conditions

After determining the best quantities of each oxide to be used as a catalyst mixture, a new central composite factorial design (with seventeen experiments) was used in order to study the conversion of lauric acid to isopropyl laurate while considering temperature, isopropanol:lauric acid molar ratio and reaction time. The arranged experiments sequence is shown in Table 2. In this stage, a catalyst percentage of 8 % (in comparison to the lauric acid mass) was applied in each experiment.

These esterification tests were also performed in the Büchiglassuster miniclave drive pressurized steel

reactor, with the external rotation control at 500 rpm, which was used in the previous tests for determination of the best catalytic mixture. The conversion of lauric acid into isopropyl laurate was measured by the Ca-40-method of AOCS.

3. Results and Discussion

3.1. Chemical Characterization of Oxides

The XRD analysis (Fig. 1A) proves that the silver oxide (Ag_2O) particles are in accordance with the literature [12, 13]. The FTIR spectrum (Fig. 1B) shows the presence of some water molecules in the sample (O–H stretchings at 3626 cm^{-1} and adsorbed hydroxide groups at 3321 cm^{-1}) [14, 15], as well as carbon dioxide from the atmosphere (bands between 2390 and 2284 cm^{-1}) [16]. However, the main evidence that the starting compound is silver oxide can be obtained upon looking the Ag–O interaction bands from 673 to 438 cm^{-1} [13, 17].

From the XRD analysis (Fig. 2A) it is also confirmed that the starting copper oxide (CuO) particles can be indexed in accordance with the literature [18]. This FTIR spectrum (Fig. 2B) also shows the presence of some water molecules in the sample (O–H stretching vibrations at 3615 cm^{-1} , O–H symmetric stretching vibrations at 2897 cm^{-1} and adsorbed hydroxide groups at 3277 cm^{-1}) [14, 15, 19]. The band at approximately 1567 cm^{-1} can be attributed to O–H bending vibrations combined with copper atoms [15]. The Cu–O stretching modes can be confirmed from the bands at 575 and 512 cm^{-1} [15, 20].

Finally, from the XRD analysis (Fig. 3A) it is also confirmed that the starting aluminum oxide (Al_2O_3) is α -aluminum oxide (JCPDS card no. 46-1212). The FTIR spectrum (Fig. 3B) shows the characteristic spectroscopic bands of α -aluminum oxide (attributed to Al–O stretching vibrations) at 649, 586 and 446 cm^{-1} [16].

After three preliminary esterification reactions at 393 K, with 10:1 isopropanol:lauric acid molar ratio and

8 % catalyst (in comparison to the total lauric acid mass), no significant changes in the structure of oxides were observed, as can be seen in the X-ray diffraction patterns and FTIR spectra of compounds which were recovered after reactions (Fig. 4). All recovered compounds were washed with methanol and dried at 333 K in a laboratory oven. Therefore, it was concluded that all chosen oxides are suitable for further tests as heterogeneous catalysts.

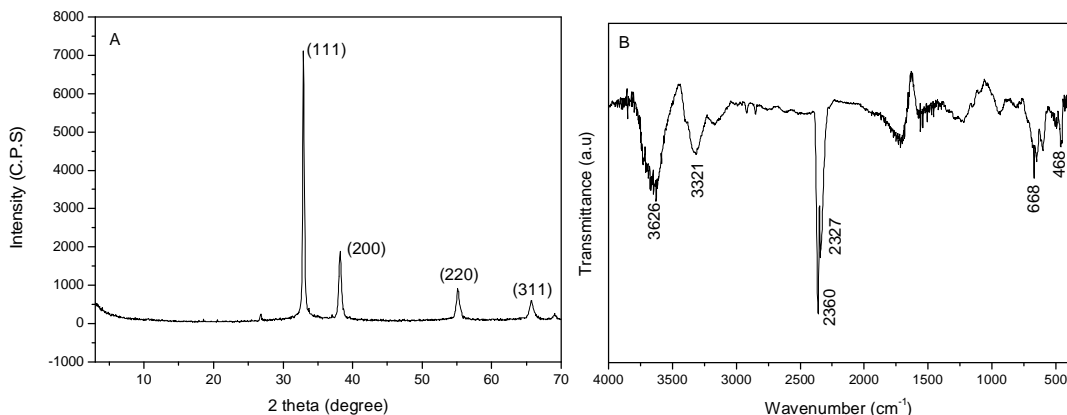


Fig. 1. X-ray diffraction pattern (A) and FTIR spectrum (B) of silver oxide

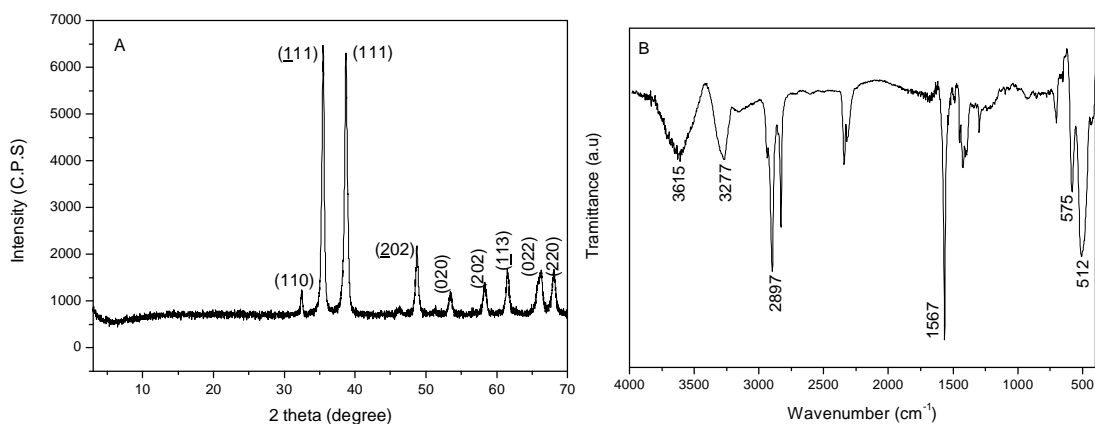


Fig. 2. X-ray diffraction pattern (A) and FTIR spectrum (B) of copper oxide

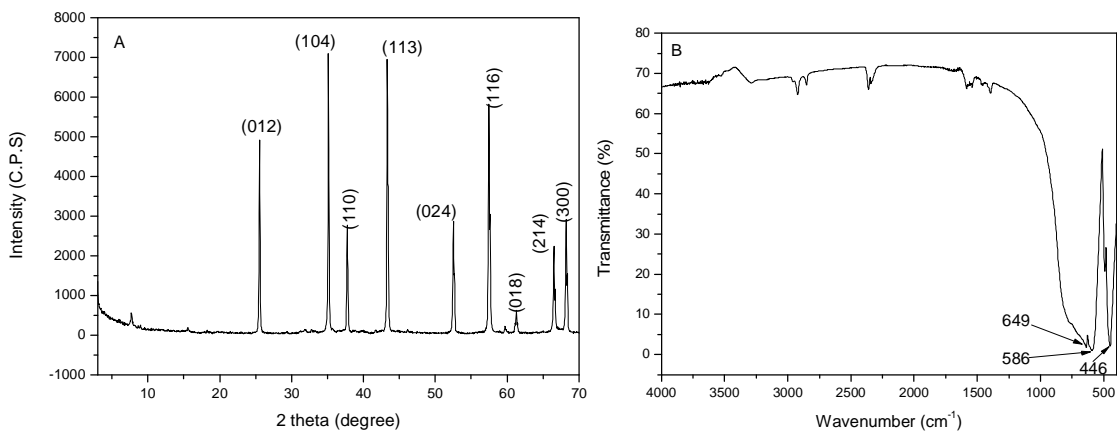


Fig. 3. X-ray diffraction pattern (A) and FTIR spectrum (B) of aluminum oxide

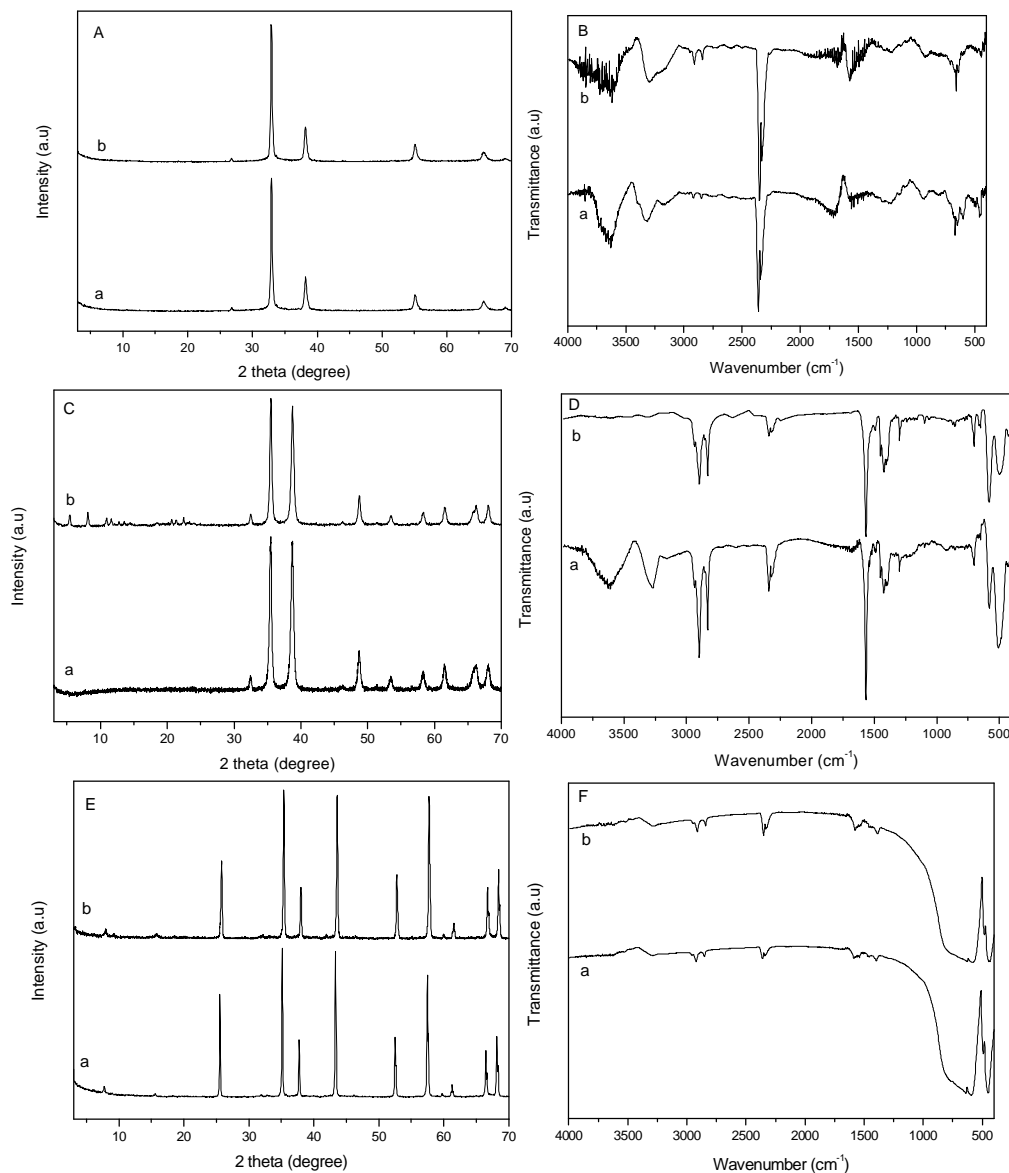


Fig. 4. X-ray diffraction patterns (A, C, E) and FTIR spectra (B, D, F) of silver oxide (A, B); copper oxide (C, D); aluminum oxide (E, F): the compounds before the reaction (a) and the compounds recovered after the reaction (b)

3.2. Esterification Reactions – Determination of the Most Adequate Oxide Mixture

For the first part of the study of the esterification reactions, the results obtained (conversion and conversion gain) with the different percentages of the oxides used as a catalytic mixture are presented in Table 3.

The conversion gain was calculated by taking as a reference the conversion of the non-catalyzed reaction (10.2 %) in the chosen reaction conditions.

Most of results (discounting their respective thermal conversions) were positive, which indicates the

effective contribution of metal oxides to convert lauric acid to isopropyl laurate, confirming the catalytic behavior of metal oxides.

The highest conversion value was obtained when only silver oxide was used as a catalyst (exp. 7), while the lowest conversion value (5.4 %) was obtained when the catalyst mixture was composed of 17 % CuO, 67 % Al₂O₃ and 17 % Ag₂O.

Upon analyzing reactions which used only one type of oxide, it can be noted that the metal oxides showed the following crescent order of catalytic activity and conversion gain: Al₂O₃<CuO<Ag₂O.

It is interesting to note the poisoning effect of CuO in the catalytic activity of Ag₂O, since changing 33 % of

Ag₂O for CuO (exp. 7 and 4) reduces the catalyzed conversion from 44.6 to 17.2 %. However, this poisoning effect is greatly reduced when 50 % of CuO is used with 50 % of Ag₂O, since in this case the catalyzed conversion was reduced from 44.6 to 33.7 % (exp. 7 and 16).

The catalytic activity of Ag₂O is also negatively affected by Al₂O₃, since changing 33 % of Ag₂O for Al₂O₃ (exp. 7 and 8) reduces the catalyzed conversion from 44.6 to 18.9 %. Higher amounts of Al₂O₃ in the Ag₂O system also lead to reductions in the obtained catalyzed conversion value (although these reductions are less intense in comparison with the experiment pair 7/8).

Taking CuO and Al₂O₃ into consideration, they have a synergic effect on the reaction when 50 % of each are used (exp. 16). However, changing the CuO/Al₂O₃ ratio from 50/50 to 67/33 leads to a significant decrease in the obtained catalyzed conversion (from 31.6 to 13.7 %, exp. 16 and 3).

When the three oxides are analyzed at the same time, it can be noted that strong antagonistic effects (towards the catalyzed conversion) were observed while using 67 % of CuO or Al₂O₃, while maintaining the percentages of the other oxides fixed at 17 % (exp. 11 and 13). However, if 67 % of Ag₂O are used while maintaining the percentages of the other oxides fixed at 17 % (exp. 12), better conversion values can be obtained.

Through the analysis of the obtained data, it was possible to observe that the experiment catalyzed only by silver oxide (exp. 7) presented the highest conversion gain. The second highest conversion gain was observed in exp. 5, which used equal mass amounts of the three oxides

in question. In general, the same trends which were observed for the conversion values were also observed for the conversion gain values.

As the objective of this work is to analyze the esterification reaction catalyzed by a ternary mixture of oxides and, moreover, taking into consideration the high cost of silver oxide, it was concluded that it would be more beneficial to continue the study using approximately 33 % of each oxide in the reactions.

3.3. Esterification Reactions – Study of Additional Parameters

In this part of the study, seventeen experiments were performed with a fixed catalyst percentage of 8 % in relation to the lauric acid mass. This catalyst mass was composed of 33 % CuO, 33 % Ag₂O and 33 % Al₂O₃. In this step, the following reaction conditions were changed according to each experiment: reaction time, temperature, and isopropanol: lauric acid molar ratio.

For each point of the design, the respective experiment was also conducted without the presence of the catalyst, in order to obtain the conversion gain. The results are expressed in Table 4.

It is possible to conclude that two experiments that showed relatively high conversion values were exp. 2 and 6, both performed at a temperature of 413 K. However, the conversion gain in these cases was very low. This is strong evidence that the temperature has a more expressive influence in converting this reaction than the catalyst itself.

Table 3

Ester conversions upon variation of the oxide mixture

Exp	CuO, %	Ag ₂ O, %	Al ₂ O ₃ , %	Conversion, %	Conversion gain, %
1	100	0.00	0.00	26.9	16.7
2	66.7	33.3	0.00	27.8	17.6
3	66.7	0.00	33.3	13.7	3.50
4	33.3	66.7	0.00	17.2	7.00
5	33.3	33.3	33.3	34.3	24.1
6	33.3	0.00	66.7	31.9	21.7
7	0.00	100	0.00	44.6	34.4
8	0.00	66.7	33.3	18.9	8.70
9	0.00	33.3	66.7	23.5	13.3
10	0.00	0.00	100	20.3	10.1
11	66.7	16.7	16.7	12.3	2.10
12	16.7	66.7	16.7	30.7	20.5
13	16.7	16.7	66.7	5.40	0.00
14	0.00	33.3	66.7	15.7	5.50
15	50.0	50.0	0.00	33.7	23.5
16	50.0	0.00	50.0	31.6	21.4
17	0.00	50.0	50.0	22.5	12.3

Table 4

Ester conversions upon variation of other parameters

Exp.	Temperature, K	Molar ratio	Time, h	Conversion, %	Conversion without catalyst, %	Conversion gain, %
1	393	6	2	22.9	6	16.9
2	413	6	2	27.9	26.8	1.1
3	393	10	2	34.3	10.2	24.1
4	413	10	2	30.5	21	9.5
5	393	6	3	20	8.1	11.9
6	413	6	3	30.9	29	1.9
7	393	10	3	20.4	5.3	15.1
8	413	10	3	24.7	17.7	7
9	386.18	8	2.5	20.7	18.7	2
10	419.82	8	2.5	43.8	30.2	13.6
11	403	4.64	2.5	20.4	18.2	2.2
12	403	11.36	2.5	14.9	6.8	8.1
13	403	8	1.66	20.6	8.6	12
14	403	8	3.34	34.3	27.8	6.5
15	403	8	2.5	22.2	10.4	11.8
16	403	8	2.5	19.2	10.4	8.8
17	403	8	2.5	18.3	10.4	7.9

The highest conversion gain values were observed in exp. 1, 3 and 7, which show that the catalyst oxides have greater effects on the reaction at lower temperatures. This justifies the purpose of our study, because from these results it can be concluded that, by using the catalyst, it is possible to obtain higher conversion gain values (in relation to the temperature of 413 K) while using lower amounts of thermal energy for reaction. It is important to note that the highest catalyzed conversion value was obtained with a temperature of 419.82 K (the highest temperature among all experiments) regardless of the reaction time and MR value. It could be stated that, if the conversion value is prioritized above all, it would not be necessary to analyze other reaction parameters. Just using a temperature value above 413 K is the key for obtained high conversion (gain) values. However, (thermal) energy costs are a major problem in many industries, and in these cases, experiments at high temperatures are not so attractive if better conversion gain values can be obtained at lower temperatures, even if we demand a greater amount of reagents.

After analyzing the reaction pairs 1/5, 3/7 and 4/8, it was primarily observed that changing the reaction time from 2 to 3 h leads to reductions in the conversion value. These reductions upon changing reaction time are more likely to occur when a molar ratio of 10 is used. However, when the temperature and MR are kept at 403 K and 8:1, respectively, and the reaction time is changed from 1.66 to 3.34 h, the conversion value increases from 20.6 to 34.3 %. Using a reaction time lower than 2 h is not beneficial to the system, even if higher temperature and MR values are used, as can be seen in exp. 13.

In most of the cases, increasing the MR values implies positive effects regarding conversion values, as can be seen in reaction pairs 1/3, 2/4 and 5/7. One exception was observed in the reaction pair 6/8, in which increasing MR was detrimental to the conversion value. Using a MR value higher than 11 decreases the conversion rate, as can be observed in exp. 12 and 15.

From these results it was possible to establish the positive or negative influence factors, in percentage points, for each variable or interaction between them. The analysis of isolated effects, as well as of combined interactions between each variable (Table 5), provides important data about the magnitude of each term.

Of all the terms, the temperature was the most important factor (4.05 %). This fact is confirmed by the results of exp. 1 and 2, for example: under constant conditions of time and MR, but increasing the temperature from 393 to 413 K, the 22.9 % conversion rate rises to 27.9 %. The same trend is observed in exp. 5 and 6, where the same type of change in temperature leads to the change in the conversion from 20 to 30.9 %, and in reaction pairs 7/8 and 9/10.

Table 5

Effects of isolated variables and their interactions

Variable/Interaction	Effect, %
Temperature	4.05
Molar ratio	-0.77
Time	0.25
Temperature:Molar ratio	-1.93
Temperature:Time	1.75
Molar ratio:Time	-2.48

Among all isolated effects, the molar ratio presented the most negative relevance (-0.77 %). This fact can be verified by observing that, although the increase of MR led to increases in the values of the conversion in reactions at 393 K with 2 h, and 413 K with 2 h, in the cases of reaction pairs 6/8 and 11/12, higher MR values led to reduction in the catalyzed conversion.

For the reaction time the positive effect (0.25 %) has the low magnitude justified by the fact that increasing the catalyst percentage caused relatively low increases (pairs, 2/6, 13/14 and 13/15) or even decreases (pairs 1/5, 3/7 and 4/8) for observed conversion values.

The interactions temperature:MR and MR:time presented slightly negative effects for esterification reactions (-1.93 and -2.48 %, respectively), while temperature:time presented a slightly positive effect (1.75 %).

Therefore, the analysis of isolated effects, as well as of combined interactions between each variable, corroborates what was mentioned above: if the conversion value is prioritized above all, the recommended procedure is to use a very high temperature value while giving very low importance to the fact that MR and time values are high or not. However, upon remembering the relevance of conversion gains, another conclusion can be drawn.

By combining all factors, and taking into consideration that is very important to determine a reaction condition that demands lower amounts of time, reagents, and lower temperature values, while prioritizing high conversion gain values, it was concluded that point 3 (temperature 393 K, isopropanol:lauric acid molar ratio 10:1, and reaction time 2 h) provided the best result for a conversion gain.

The esterification reaction is nearly thermoneutral, as calculated from the enthalpies of formation of different esters, acids and alcohols. The average value of $\Delta H^0 = -3 \pm 2$ kJ/mol. A heat flux of only 0.8 kW would be generated, if these processes were designed to take place with a mean conversion rate of 1 kmol/h. From the calculation of the effect of temperature on the degree of esterification according to the Van't Hoff equation, a 0.4 % decrease is expected, around ambient temperatures, in the equilibrium constant per one Kelvin increase [21].

However, results [22] showed that under the equilibrium condition, the water produced in the reaction is mainly distributed in an alcohol-rich phase, with a small amount remaining in an ester-rich phase. The presence of water in a monophasic system would shift reaction equilibrium to the reactants because of the hydrolysis reaction. However, when two liquid phases are considered, the water formed in the reaction medium is not available in the ester-rich phase, allowing the reaction to proceed to products. Therefore, this fact justifies results which are superior in relation to calculated ones.

The reaction mechanism proposed for the synthesis of isopropyl laurate with metal oxides is similar to what was established in the literature for esterification of fatty acids using heterogeneous zinc catalysts: initially there is an acid-base interaction between the copper/silver/aluminum atom from the catalyst and the carbonyl group from the lauric acid, developing a high positive charge density on the carbonyl carbon. Isopropanol attacks the polarized carbonyl group in order to form a tetrahedral intermediate. Then, a proton is transferred between the oxygen atoms of this intermediate, generating a water molecule. This molecule is lost, while the isopropyl laurate is generated. Finally, the acid-base interaction between the copper/silver/aluminum atom from the catalyst and the carbonyl group from isopropyl laurate is finished, and this copper/silver/aluminum atom is free to begin another catalytic cycle [23-26].

4. Conclusions

All metal oxides showed a promising catalytic activity in the esterification of lauric acid with isopropanol. The most adequate conversion value was obtained from the reaction conducted in the following conditions: temperature 393 K, isopropanol:lauric acid molar ratio 10:1, composition of catalyst is 33 % CuO, 33 % Ag₂O and 33 % Al₂O₃, catalytic mixture/lauric acid ratio (w/w) 8 % and reaction time 2 h. The oxides were not converted into other materials during esterification. It was concluded that the materials tested as catalysts herein possess characteristics of heterogeneous catalysts.

The effectiveness of the investigated process could not be compared with other catalysts from the literature, since this is the first work which explores the synthesis of isopropyl laurate directly from lauric acid and isopropanol while employing metal oxides as catalysts.

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ДОСЛІДЖЕННЯ ОКСИДІВ МЕТАЛУ, ЯК КАТАЛІЗАТОРІВ ЕСТЕРИФІКАЦІЇ ЛАУРИНОВОЇ КИСЛОТИ З ІЗОПРОПАНОЛОМ

Анотація. Синтезовано ізопропілаурат з використанням як каталізатора суміші оксидів металів. Найвищу конверсію отримано за таких умов: 393 K, молярне співвідношення ізопропанол:лауринова кислота 10:1, склад каталізатора 33 % CuO , 33 % Ag_2O і 33 % Al_2O_3 , співвідношення каталізатор/лауринова кислота становить 8 мас %, час реакції 2 год. Показано, що оксиди каталізатора після естерифікації не перетворюються в інші речовини.

Ключові слова: оксид металу, ізопропілаурат, естерифікація, лауринова кислота, ізопропіловий спирт.