

Niobium compounds in transesterification reactions of soybean oil: a promising use of NbCl₅.

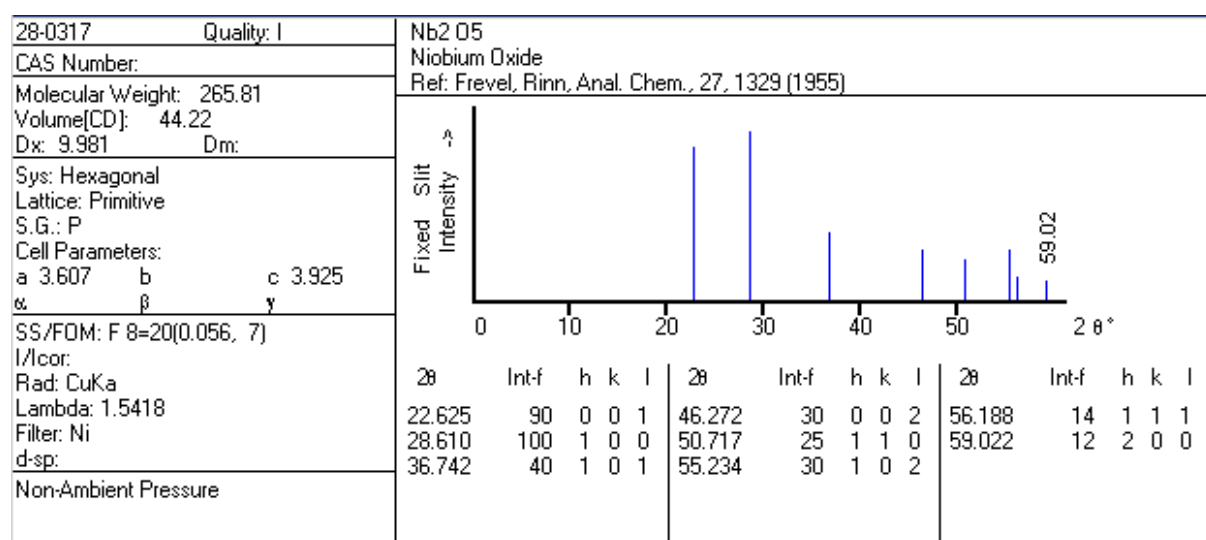
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Supplementary Information

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**Figure S1:** Crystallographic record (JCPDS 28-0317) of the hexagonal phase of Nb₂O₅.

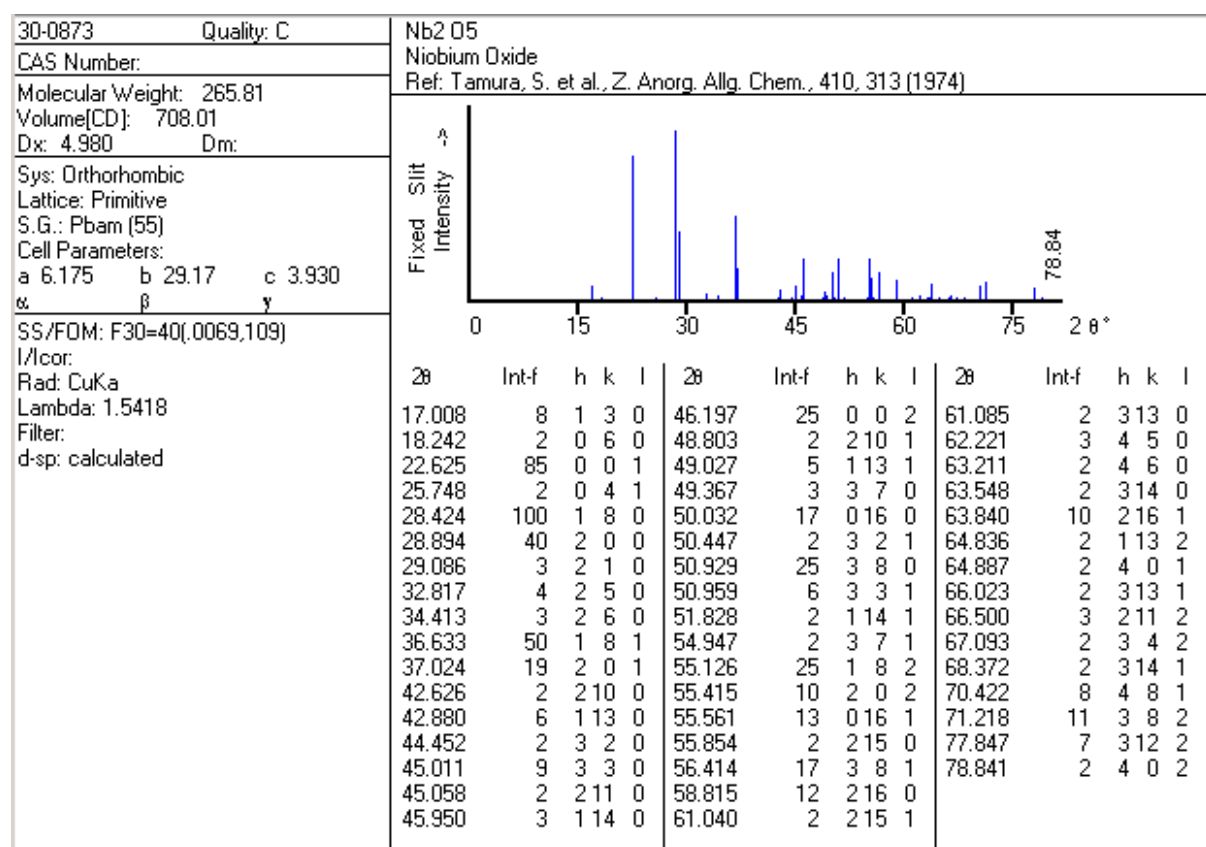


Figure S2: Crystallographic record (JCPDS 30-0873) of the orthorhombic phase of Nb₂O₅.

Figure S3 shows the optimized chromatogram with the retention times of the 1.0 μL injection of the methyl esters mixture (standard-primary) of the fatty acids at the concentration of 100 mg/L. The elution order and their respective detection times were identified by first injecting each methyl ester.

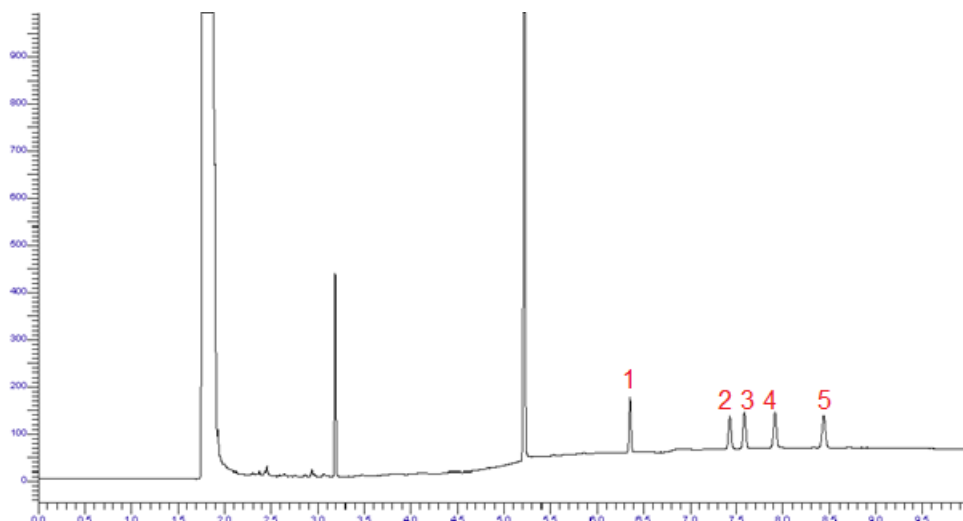


Figure S3: Chromatogram obtained after the injection of 1.0 μL of standard stock solution containing the palmitate esters of methyl (1), methyl stearate (2), methyl oleate (3), methyl linoleate (4) and methyl linolenate (5).

The not referenced peaks on the Figure S3 chromatogram correspond from left to right to heptane (solvent), methyl octanoate and 4-methyl-phenol, respectively. These substances were not quantified in this work. The chromatograms and their quantitative data for the 1.0 μL injections of the transesterification reactions of soybean oil using NbCl_5 and the different synthesized niobium pentoxide catalysts (amorphous Nb_2O_5 , SG400, SG500, SG600, SG700, SG800) are shown in the Figure S4 to S10.

•*NbCl₅*

Figure S4 shows chromatogram of the 1.0 μL injection of the transesterification reaction of soybean oil using NbCl_5 as catalyst. It was observed that the peaks exceeded the limit of quantification of the optimized method, not allowing its analysis. Thus, a 1:1000 dilution of the reaction in heptane was performed repeating the analysis. The data is presented in the Figure S5 and in the Table 1.

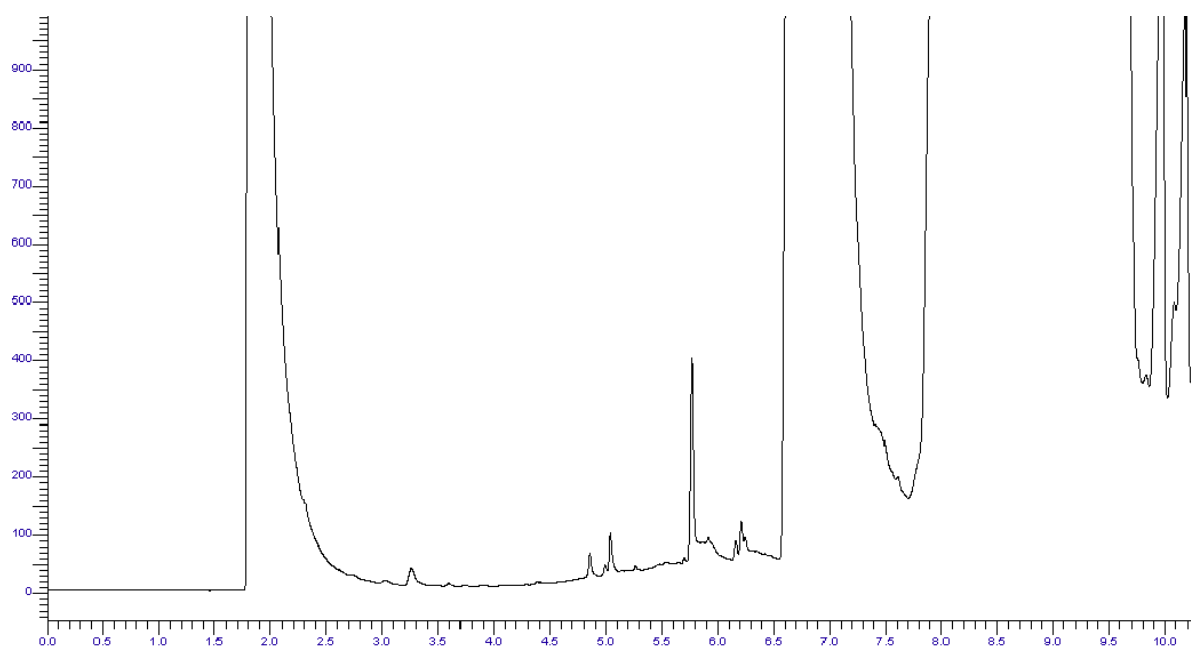


Figure S4: Chromatogram obtained after the injection of 1.0 μL of the transesterification reaction using NbCl_5 as a catalyst.

•Amorphous Nb₂O₅

Figure S5 and Table 1 show the chromatogram and quantitative data, respectively, of the 1.0 µL injection of the soybean oil transesterification reaction using commercial and amorphous Nb₂O₅ as catalyst.

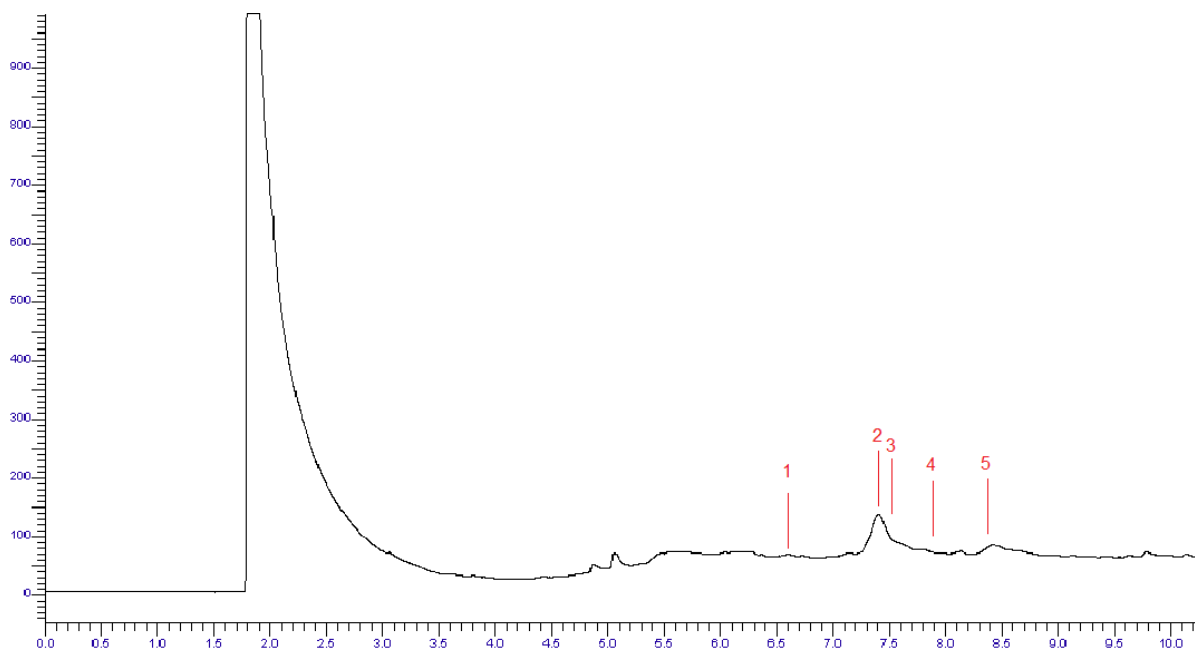


Figure S5: Chromatogram obtained after the injection of 1.0 µL of the transesterification reaction using commercial and amorphous Nb₂O₅ as catalyst containing methyl palmitate (1), methyl stearate (2), methyl oleate (3), methyl linoleate (4) and methyl linolenate (5).

Table 1: Data obtained by injecting 1.0 µL of the transesterification reaction using commercial Nb₂O₅ and amorphous as catalyst.

Index	Component	Time (min)	Area	Concentration (µg.L ⁻¹)
1	methyl palmitate	6.598	11423,22	7.5
2	methyl stearate	7.498	-	-
3	methyl oleate	7.699	5102,12	2.7
4	methyl linoleate	7.874	-	-
5	methyl linolenate	8.299	2507,92	1.4

•SG400

Figure S6 and Table 2 show the chromatogram and quantitative data, respectively, of 1.0 μL injection of the soybean oil transesterification reaction using SG400 (nano Nb_2O_5 synthered at 400 $^\circ\text{C}$) as catalyst.

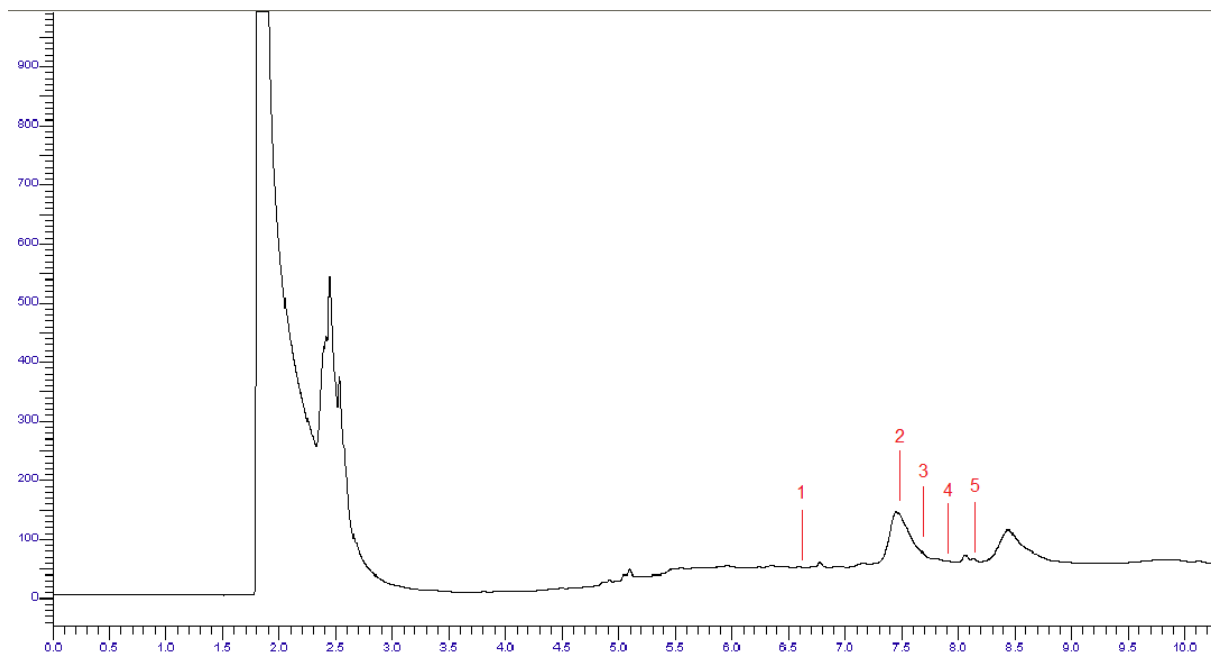


Figure S6: Chromatogram obtained after the injection of 1.0 μL of the transesterification reaction using SG400 as catalyst containing methyl palmitate (1), methyl stearate (2), methyl oleate (3), methyl linoleate (4) and methyl linolenate (5).

Table 2: Data obtained by injecting 1.0 μL of the transesterification reaction using SG400 as catalyst.

Index	Component	Time (min)	Area	Concentration ($\mu\text{g}.\text{L}^{-1}$)
1	methyl palmitate	6.576	92431.35	58.6
2	methyl stearate	7.469	23474.83	11.5
3	methyl oleate	7.685	156053.6	70.1
4	methyl linoleate	7.871	22211.3	9.9
5	methyl linolenate	8.296	45049.19	18.9

Figure S7 and Table 3 show the chromatogram and quantitative data, respectively, of 1.0 μL injection of the soybean oil transesterification reaction using SG500 (nano Nb_2O_5 synthered at 500 $^\circ\text{C}$) as catalyst.

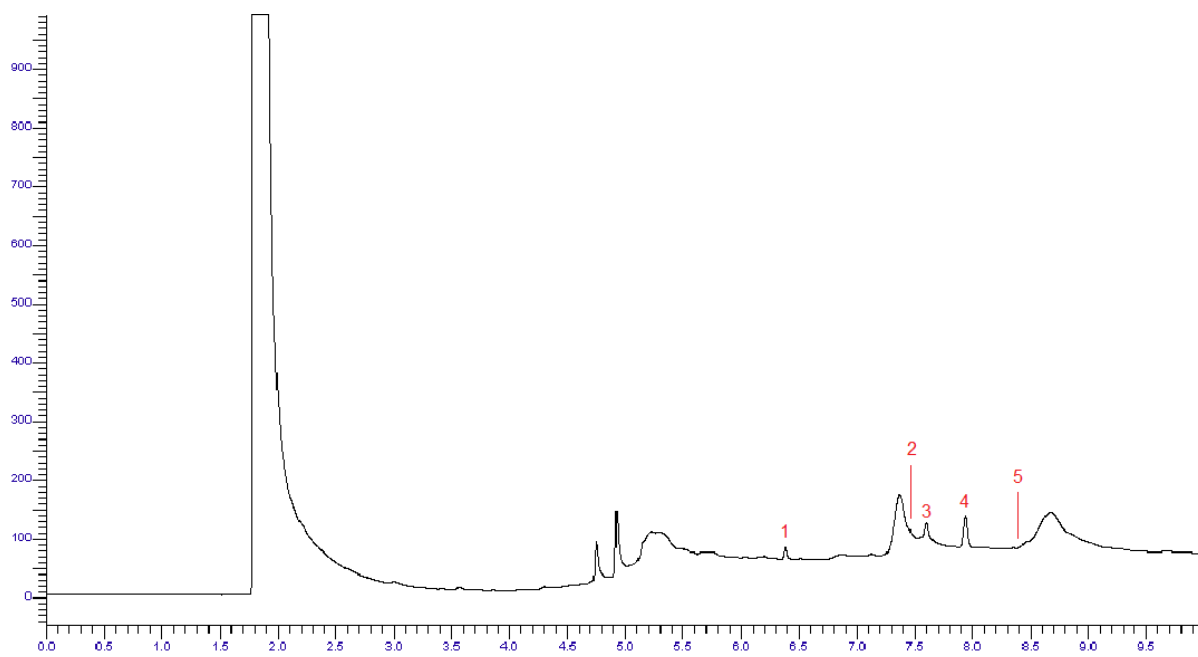


Figure S7: Chromatogram obtained after the injection of 1.0 μL of the transesterification reaction using SG500 as catalyst containing methyl palmitate (1), methyl stearate (2), methyl oleate (3), methyl linoleate (4) and methyl linolenate (5).

Table 3: Data obtained by injecting 1.0 μL of the transesterification reaction using SG500 as catalyst.

Index	Component	Time (min)	Area	Concentration ($\mu\text{g.L}^{-1}$)
1	methyl palmitate	6.423	114112.81	72.4
2	methyl stearate	7.448	28981.27	14.2
3	methyl oleate	7.559	192658.82	86.5
4	methyl linoleate	7.824	274213.65	12.3
5	methyl linolenate	8.413	55616.28	23.4

•SG600

Figure S8 and Table 4 show the chromatogram and quantitative data, respectively, of 1.0 μL injection of the soybean oil transesterification reaction using SG600 (nano Nb_2O_5 synthered at 600 $^\circ\text{C}$) as catalyst.

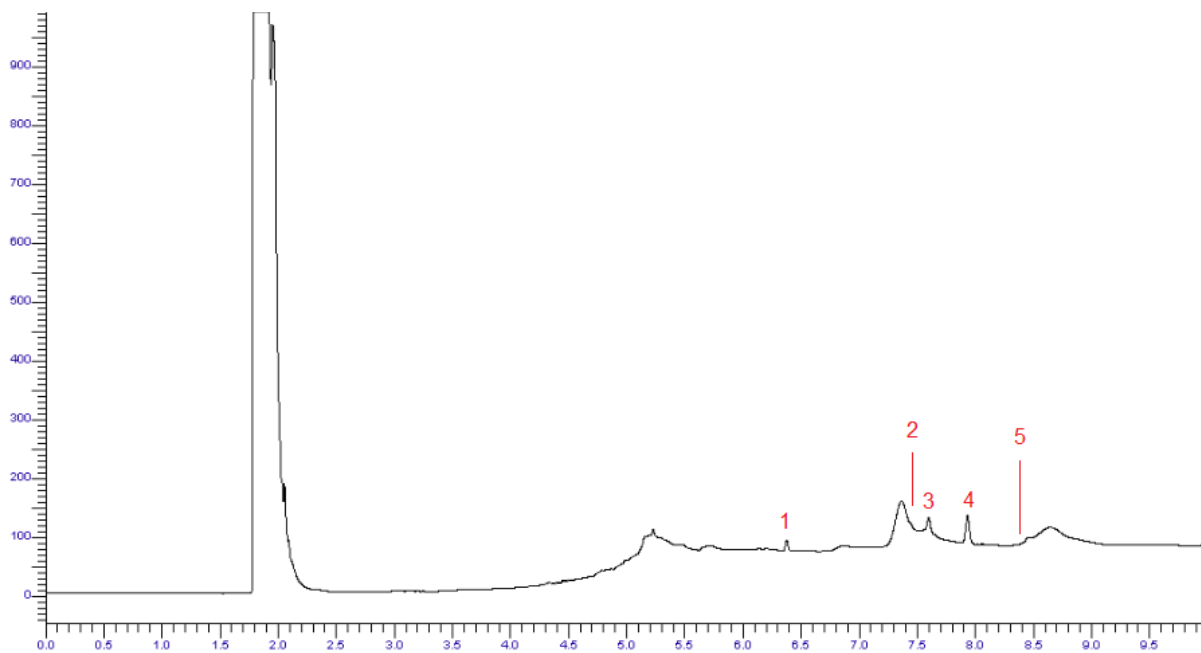


Figure S8: Chromatogram obtained after the injection of 1.0 μL of the transesterification reaction using SG600 as catalyst containing methyl palmitate (1), methyl stearate (2), methyl oleate (3), methyl linoleate (4) and methyl linolenate (5).

Table 4: Data obtained by injecting 1.0 μL of the transesterification reaction using SG600 as catalyst.

Index	Component	Time (min)	Area	Concentration ($\mu\text{g.L}^{-1}$)
1	methyl palmitate	6.421	102701.5	65.1
2	methyl stearate	7.433	26083.14	12.8
3	methyl oleate	7.578	173392.9	77.8
4	methyl linoleate	7.855	246792.2	11.1
5	methyl linolenate	8.399	50054.65	21.0

•SG700

Figure S9 and Table 5 show the chromatogram and quantitative data, respectively, of 1.0 μL injection of the soybean oil transesterification reaction using SG700 (nano Nb_2O_5 synthered at 700 $^\circ\text{C}$) as catalyst.

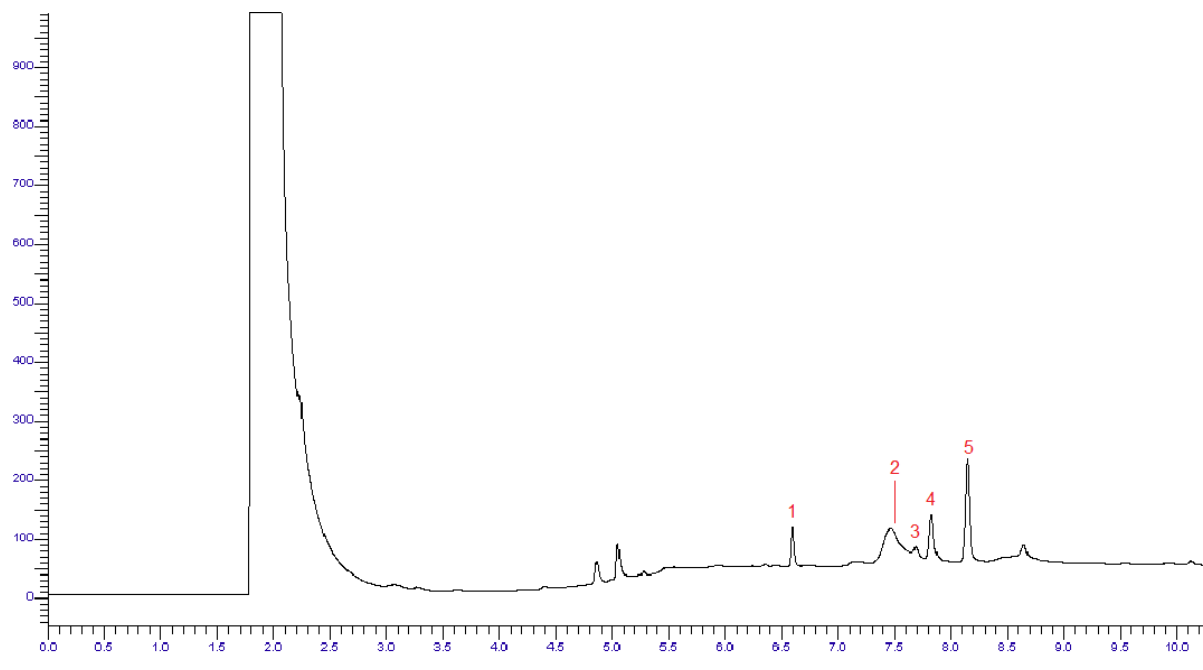


Figure S9: Chromatogram obtained after the injection of 1.0 μL of the transesterification reaction using SG700 as catalyst containing methyl palmitate (1), methyl stearate (2), methyl oleate (3), methyl linoleate (4) and methyl linolenate.

Table 5: Data obtained by injecting 1.0 μL of the transesterification reaction using SG700 as catalyst.

Index	Component	Time (min)	Area	Concentration ($\mu\text{g.L}^{-1}$)
1	methyl palmitate	6.599	126792.03	80.4
2	methyl stearate	7.497	32201.41	15.8
3	methyl oleate	7.701	214065.32	96.1
4	methyl linoleate	7.828	304681.80	136.8
5	methyl linolenate	8.296	61795.87	25.9

•SG800

Figure S10 and Table 6 show the chromatogram and quantitative data, respectively, of 1.0 μL injection of the soybean oil transesterification reaction using SG800 (nano Nb_2O_5 synthered at 800 $^\circ\text{C}$) as catalyst.

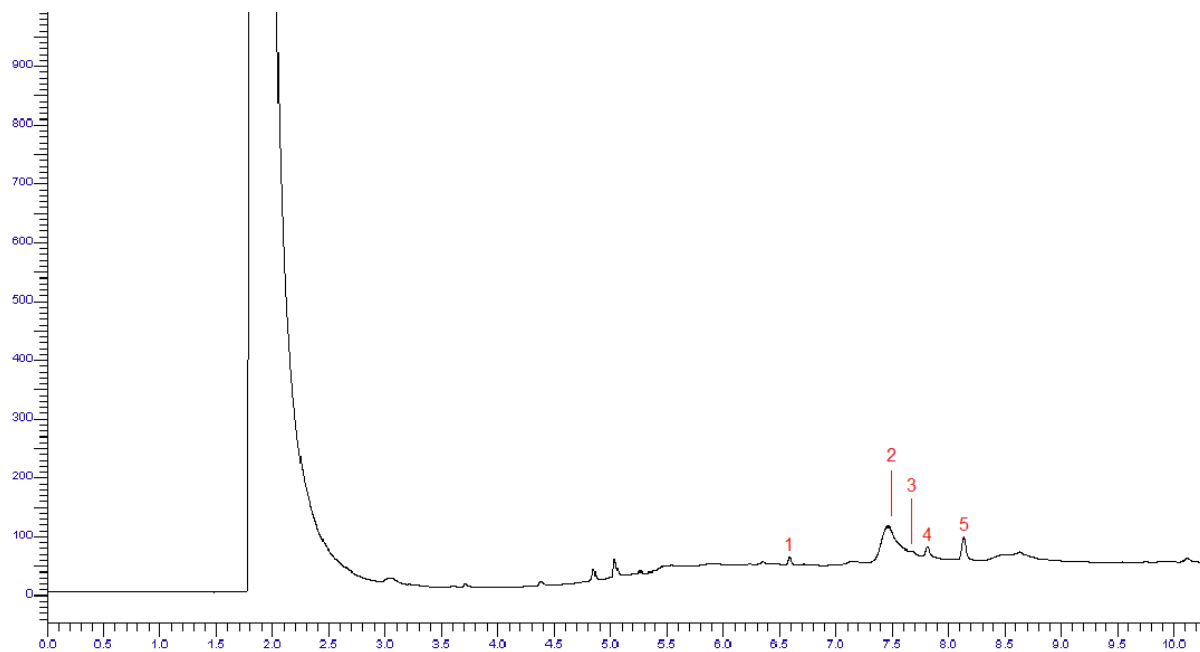


Figure S10: Chromatogram obtained after the injection of 1.0 μL of the transesterification reaction using SG700 as catalyst containing methyl palmitate (1), methyl stearate (2), methyl oleate (3), methyl linoleate (4) and methyl linolenate.

Table 6: Data obtained by injecting 1.0 μL of the transesterification reaction using SG800 as catalyst.

Index	Component	Time (min)	Area	Concentration ($\mu\text{g.L}^{-1}$)
1	methyl palmitate	6.591	26850.65	17.3
2	methyl stearate	7.501	4320.66	2.3
3	methyl oleate	7.709	45678.57	20.8
4	methyl linoleate	7.825	108326.68	46.9
5	methyl linolenate	8.293	25318.59	10.8