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Optimization of Combined Catalytic Ethanolysis of *Ceiba pentandra* (L.) Seed Oil and Analysis of Metal Trace Elements in Produced Biodiesel

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Abstract Ethanolysis of seeds oil from *Ceiba pentandra* with ethanol in combined catalysis was investigated in this study. In addition, the metal trace elements (TMEs) of the formulated biodiesel were quantified. For this purpose, the formulation of ethyl esters was done in two steps (acidic and basic) followed by purification. The influence of the molar ratio of ethanol to oil (6:1-8:1), the ratio of the mass of catalyst to oil (1.0; 1.1; 1.25 and 1.5) and the reaction temperature (30-60)°C were studied in order to optimize the operating conditions of this reaction. Subsequently, part of oil was converted to ethyl esters with the optimal reaction parameters. The analysis of metal trace elements using inductively coupled plasma mass spectrometry (ICP-CRC-MS, Agilent 7500) lead to determine the metal trace elements in both samples (oil and biodiesel) after mineralization. Calibration was performed with aqueous standards to avoid the use of organic standards which are often unstable. Fatty acids in the oil were converted into ethyl esters under optimal conditions (ethanol/oil molar ratio of (40:1), the amount of catalyst (H_2SO_4) (10% w/w) and a temperature of 78°C) for a fatty acid content equal to 0.46% (w/w). Triglycerides in oil were transesterified under basic catalysis with the optimal operating conditions (ethanol/oil molar ratio = 6:1, amount of catalyst = 1.1% (w/w) and reaction temperature = 60°C) yielded of improved ethyl esters equal to 98.91% (w/w). The MTEs of the vegetable oil samples and the formulated biodiesel revealed relatively low concentrations. On the other hand, other TMEs (Mg, Na, K and Ca) were quantified with concentrations that are higher (sup 0.1 µg/Kg) but lower than the recommended value (<500 µg/Kg) (ASTM D6751/ EN 14214). Data recovery was in the range of 90 to 100% for the two samples studied.

Keywords Ethanolysis, Combined catalysis, Metal trace elements, Vegetable oil, Formulated biodiesel, *Ceiba pentandra*, ICP-MS

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1. Introduction

Concern about the disappearance and gradual depletion of the world's fossil fuel reserves has prompted scientifics to research and produce alternative renewable fuels [1]. Curiosity about green fuels as an additive or alternative to petroleum has grown considerably over the past two decades for political, financial and environmental reasons [2,3]. Thus, several developed fund have supported researches for more environmentally friendly and renewable resources in order to reduce climate change. One of options was the production of biodiesel. Indeed, biodiesel is a mixture of alkyl esters obtained by the transesterification reaction from vegetable oils. In order to respond effectively to food self-sufficiency, the use of non-edible vegetable oils for the formulation of biodiesel is nowadays promoted. It is shown that the use of biodiesel allows the reduction of carbon dioxide (CO_2) emissions [4].

Ceiba pentandra (L.) known as Kapokier is a tropical plant which belong to the family of Bombacaceae. Kapokier is known for its soft and white silk [5]. It is a large tree with conical thorns on the trunk and twigs having a rapid growth and becomes productive between 4 to 5 years.

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Under optimal conditions an adult tree can produce up to 400 fruits each year, yielding 15-18 kilograms of fiber and about 30 kilograms of seeds per fruiting [6]. Fruit is in a spindle-shaped membranous envelope containing black spherical seeds representing about 28% m/m of the fruit and covered with fine, silky hairs. Seeds are a source of oil and therefore an excellent raw material in the paint and biodiesel production industries [7,8]. Previous study reported that the main constituents of ethyl biodiesel derived from *Ceiba pentandra* vegetable oil are palmitate (C16:0) (24.53%), oleate (C18:1) (19.6%) and linoleate (C18:2) (36.92%) esters [8]. Oils in addition to the main elements such as carbon (82-87%) and hydrogen (12-15%) [9]; contained trace metals such as sulfur (0.05-5%), oxygen (< 2%) and nitrogen (< 0.1%) which came respectively in fourth and fifth position of the most abundant elements [10]. Other elements such as vanadium, nickel, calcium, potassium, iron, copper, zinc, boron, arsenic, selenium, silicon, phosphorus are generally present in oils in small proportions (0.01-0.1%) or in ultra trace levels (<0.01%). Indeed, trace metal elements (TMEs), are incisive pollutants that have a toxic and harmful impact on alkyl esters (biodiesels) and their use. Among these TMEs, Lead, Zinc, Copper, Potassium, Sulfur, Sodium have an important place. Their presence and accumulation in oil and biodiesel have several origins including, the supply and misuse of fertilizers based on these elements. In addition, TMEs play an important role in the clogging of vehicle fuel lines and can lead to the production of undesirable residues of metal oxides in engine. Moreover, some TMEs acted as poisons during the catalytic cracking process of oils in the refining process, particularly vanadium and nickel, which can occur at high concentrations [11]. Other metal trace elements can cause corrosion of refinery equipment [12]. Mercury can be deposited in engine equipment, requiring careful monitoring and maintenance [13]. Therefore, the determination of trace metal elements in seeds oils with could lead to, know the type of specific metals which should be removed upstream and downstream [14]. On the other hand, some heavy metals are toxic at high doses. Thus, monitoring of these elements (Ca, Cu, Fe, Mg, Mn, Na and P etc.) is necessary because of their ability to affect the behavior of biodiesel by forming undesirable compounds in engine. Then, the present study aims to optimize the combined catalytic transesterification reaction of non-edible seed oil from *Ceiba pentandra* and quantify metal trace elements in seeds oil and biodiesel produced.

2. Material and Methods

2.1. Sampling and Oil Extraction

Ceiba pentandra seeds were collected in northwestern Benin 9° 39′ 42″ North then 1° 23′05″ East. Oil was extracted from seeds soxhlet extraction method and stored in dark at 25℃ until use [5,8].

2.2. Transesterification Process

Biodiesel was produced by the transesterification reaction in combined catalysis (acid and basic) from non-conventional oil obtained. Ethanolysis reactions were carried out, following the two-step procedure (combined acid and base homogeneous catalysis), considering the acidity higher than 1% (m/m) oil [5].

2.3. Fischer's Esterification

The first step consisted of esterification of free fatty acids from oil by homogeneous acid catalysis. It was conducted with anhydrous ethanol: oil molar ratios of (9:1, 14:1, 20:1 and 30:1) in the presence of concentrated sulfuric acid (H_2SO_4) (1 to 5% (w/w oil) at 78℃ for one hour under moderate stirring.

At the end of this step, the reaction mixture was neutralized with 20% (w/w oil) at 2% (w/w water) sodium bicarbonate solution for 10 min under slow stirring, and then transferred to a separatory funnel. To accelerate the separation of the 100% (v/v) phases, hexane was added to the mixture. After phase separation, drying was performed by addition anhydrous sodium sulfate (Na_2SO_4). After filtration the ester phase, the solvent was evaporated under reduced pressure. The ester-rich phase was then weighed and free fatty acid content was determined by colorimetric titration.

Transesterification of triglycerides by homogeneous basic catalysis.

Several experiments of the synthesis of ethyl esters by the transesterification reaction were carried out to evaluate the stoichiometric amount of ethanol to be used. For this purpose, 5g of non-conventional seeds oil were transesterified by varying the amount of ethanol to analyze the effect of the molar ratio (ethanol: oil) on the reaction. Indeed, 6:1 and 8:1 ethanol excesses were used to transesterify oil samples [15].

2.4. General Process in Basic Homogeneous Catalysis

Unreacted triglycerides and mono- and di-glycerides contained in the ester-rich phase were transesterified under basic catalysis in second step. Ethanol and catalyst were added in the amounts established for each experiment, the stirring system was connected (250 rpm) and this time represented the start of the reaction. The final mixture was neutralized with a 3% $NaHCO_3$ solution (w/w water) to pH=7 and then transferred to a separatory funnel. After separation, the ester phase was then dried with anhydrous sodium sulfate (Na_2SO_4) and filtered through filter paper. Residual ethanol and water were removed by evaporation at 90℃ under reduced pressure. The yields of ethyl biodiesel from each oil were evaluated and expressed as mass percentage [15].

2.5. Trace Metal Analysis by Inductively Coupled Plasma Mass Spectrometry (ICP-MS)

Inductively coupled plasma mass spectrometry

(ICP-CRC-MS, Agilent 7500) with a CRC-2 collision-reaction cell was used to determine the inorganic constituents in the seeds oils and biodiesels formulated after mineralization. The system (7500cs) equipped with a simultaneous dynode, discrete dual mode (pulse/analog) detector with nine orders of linear dynamic range uses a Scott double pass chamber and a low flow efficient microconcentric nebulizer and spray cooled Scott chambers. The ICP-MS is controlled by an Agilent Chemstation computer system. For sample preparation and mineralization, 200 µL of sample was added to 1.5 mL of oxygenated water (H_2O_2) and 2.5 mL of 65% Suprapur® nitric acid (HNO_3) (Ref. 1.00441) in a 50 mL tube. Seeds oils are complex mixtures of organic materials, with a high organic load and viscosity, which can cause polyatomic and spectrometric interferences during the determination, and instability or even plasma quenching. Therefore, a pre-treatment is necessary before their introduction in the analytical instruments. Thus, the mineralization of the sample was done in a Digiprep system for 8h at 80℃.

Table 1. Operating conditions for inductively coupled plasma mass spectrometry

Plasma gas source	: Ultra pure Argon (99.999%)
Plasma power	: 1500 W
Plasma gas flow rate	: 14 L/min
Nebulizer gas flow rate	: 1.05 L/min
Auxiliary gas flow rate	: 1.9 L/min
Pump speed	: 0.1 rpm
Delivery system	: Scott chamber
Nebulizer	: Microconcentric

The final analysis is performed by ICP-MS, after dilution to 50mL, without addition of internal standard. Standards and calibration solutions are prepared from the stock solution (Agilent 5183-4688 Multi-element Standard Solution) by dilution in 5mL vials. A method blank containing oxygenated water and nitric acid was prepared under the same conditions of mineralization, dilution for analysis. The analysis was performed in multimode (without cell, in He (Collision) mode, then in H_2 (Reaction) mode depending on the interfered elements [16].

3. Results and Discussion

3.1. Transformation of Free Fatty Acids into Ethyl Esters by Fischer Esterification in Acid Catalysis

Table 2 presented the results of transformation of free fatty acids into ethyl esters by Fischer esterification reaction. For the various tests carried out, a decrease in free fatty acid content is noted as a function of the anhydrous ethanol/oil molar ratio and the concentration of the catalyst. Thus, for a molar ratio (anhydrous ethanol / oil) and a concentration of catalyst (sulfuric acid) respectively (40:1) and 10% (m/m) of the mass of vegetable oil used, it was observed a better reduction of the free fatty acid content.

Table 2. Transformation of free fatty acids into ethyl esters by Fischer esterification reaction

Experiments	Molar ratio ethanol: oil	Catalyst (H_2SO_4), % (w/w oil)	T, (℃)	[FFA], %(w/w)
CP1	9:1	1.5	78	2.16
CP2	14:1	3.0	78	1.44
CP3	30:1	5.0	78	0.68
CP4	40:1	10.0	78	0.46

Table 3. Yields of ethyl esters from ethanolysis under basic catalysis

Experiments	Molar ratio ethanol: oil	Catalyst (KOH), % (w/w oil)	T, (℃)	Time (min)	Crude ethyl ester content, % (w/w)
CP1	6:1	1.0	35	120	56.8
CP2	6:1	1.1	35	120	82.6
CP3	6:1	1.25	35	120	80.8
CP4	6:1	1.5	35	120	75.8
CP5	6:1	1.0	60	120	60.71
CP6	6:1	1.1	60	120	98.91
CP7	6:1	1.25	60	120	89.35
CP8	6:1	1.5	60	120	81.62
CP9	8:1	1.0	35	120	42.8
CP10	8:1	1.1	35	120	61.15
CP11	8:1	1.25	35	120	70.4
CP12	8:1	1.5	35	120	65.01
CP13	8:1	1.0	60	120	68.2
CP14	8:1	1.1	60	120	84.08
CP15	8:1	1.25	60	120	98.15
CP16	8:1	1.5	60	120	86.74

3.2. Transesterification of Vegetable Oil by Basic Catalysis

Table 3 showed the yields of ethyl esters from ethanolysis under basic catalysis. The results obtained indicate that parameters such as molar ratio, amount of catalyst and temperature greatly influence the yield of oil conversion to esters. Thus, the variation of these factors allowed to optimize, the ethanolysis in basic catalysis. Subsequently, the different factors that influence this conversion were analyzed and a mode of ethanolysis by basic homogeneous catalysis was improved.

3.3. Influence of Ethanol: Oil Molar Ratios (6:1 and 8:1) on Yield at Ethyl Esters

The experimental results of ethanolysis of oil (Figure 1) indicated that when the amount of ethanol is increased, an improvement in yield is observed. At 35℃ and maintaining cKOH=1.1% of the oil mass, a conversion rate of 82.6% (w/w) was obtained in 120 minutes for an ethanol:oil molar ratio of 6:1, while for the same reaction time, we found an ester yield of 61.15% (w/w) by increasing the ethanol:oil molar ratio to 8:1. The same findings were made by performing the reaction under the same operating conditions with an increase in temperature to 60℃ but with an improvement in the conversion rate (ω) in both cases (6:1; ω=98.91% (w/w) and 8:1; ω = 84.08% (w/w)). This result, shows the positive impact of temperature on the basic catalysis ethanolysis of *C. pentandra* seeds oil.

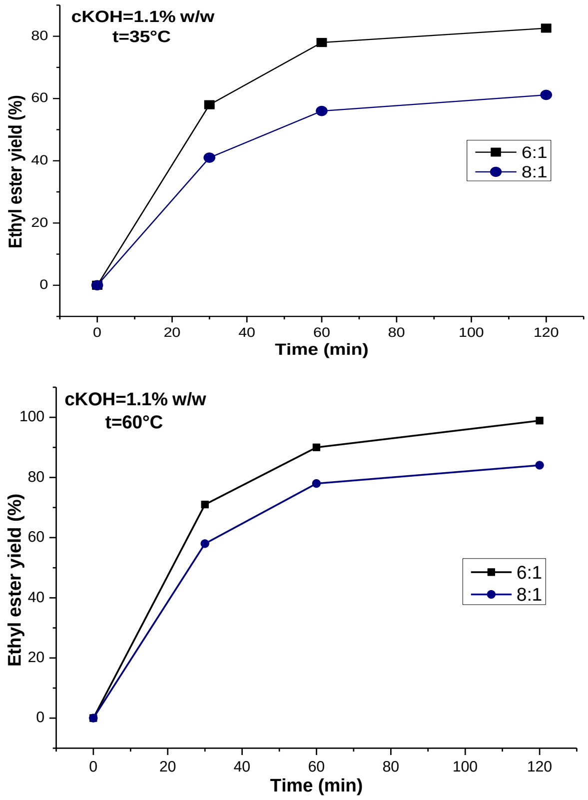

Figure 1. Effect of molar ratio (6:1 and 8:1) on the yield of at ethyl esters from *C. pentandra* seeds oil with cKOH=1.10% at 35 and 60℃

In conclusion, the study of effect of the ethanol: oil molar ratio and of temperature on ester conversion rate allowed us to better understand the importance of these two factors during our different tests. The variation in temperature also allowed us to achieve a conversion rate in line with the European specification in terms of total ester content. However, a further increase in molar ratio from 6:1 to 8:1 is also influenced by temperature variation.

3.4. Influence of Catalyst Concentration (cKOH) on Yield at Ethyl Esters

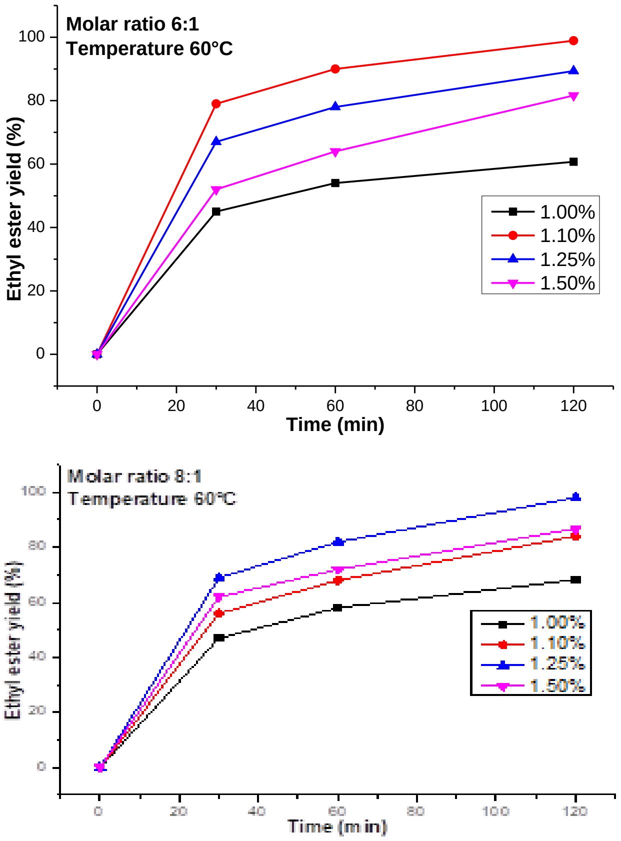

Figure 2. Effect of catalyst concentration (cKOH) on *C. pentandra* seeds oil conversion rate as a function of reaction time: ethanol:oil molar ratios (6:1 and 8:1) and temperature

Figure 2 presented the results of the effect of catalyst con-centration (cKOH) on *C. pentandra* seeds oil conversion rate as a function of reaction time: ethanol:oil molar ratios (6:1 and 8:1) and temperature. The results of these tests have made it possible to specify the effect of amount of KOH and temperature on the rate of conversion of this oil into ethyl esters Indeed, the tests gave a better conversion rate with cKOH equal to 1.10%. But with higher cKOH (1.25 and 1.5%) or lower (1%), an incomplete reaction and or the appearance of secondary reaction (saponification) leading to a low yield of ethyl esters were observed. The highest yield of esters or conversion rate is 98.91% (w/w) after 120 minutes of reac-tion at 60℃ with an ethanol: oil molar ratio of 6: 1 and 98.15% (w/w) under the same conditions but with a molar ratio of 8: 1. Indeed, the graphs show a high yield of esters from the first 60 minutes of reaction and then asymptotic curves until the end of reaction (120 min).

However, assay lead to record ethyl ester yields above the EN 14214 limit set at 96.5% for ethyl esters made at the same catalyst concentration (1.10%) at 60℃ and after 30 min of reaction. In sum, the concentration of catalyst in transesterification reaction varies with the chemical composition and acidity of oil. These results are similar to those of Anastopoulo et *al.* [16] on the ethanolysis of rape seed and sunflower oil, and in the ethanolysis of peanut oil [17].

3.5. Influence of Temperature on Ester Yield

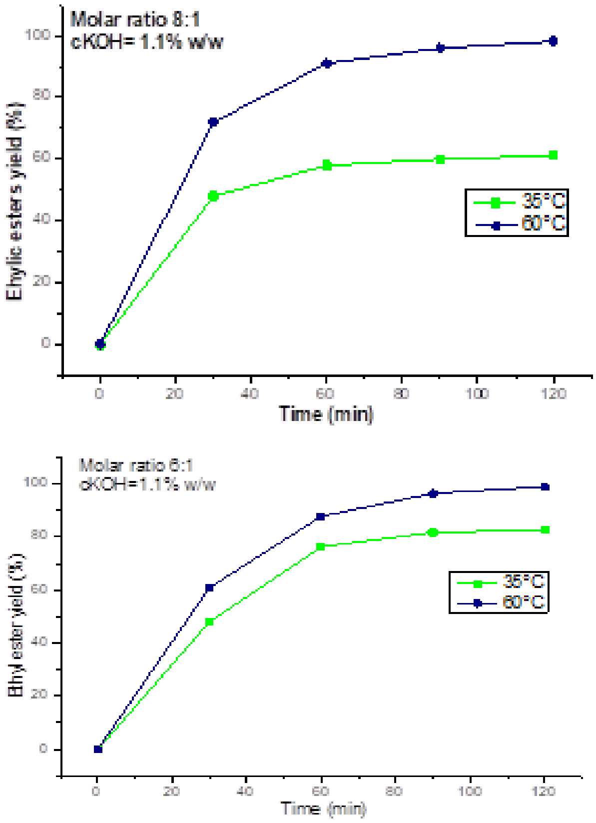

Figure 3. Influence of temperature on yield of esters from *C. pentandra* seeds oil: Ethanol: oil molar ratios (6:1; 8:1), cKOH=1.1% (m/m)

Figure 3 presented results of the influence of temperature on yield of esters from C. pentandra seeds oil: Ethanol: oil molar ratios (6:1; 8:1), cKOH=1.1% (m/m). Results showed the variation of the conversion rate to esters of C. pentandra oil as a function of time. In these experimentations, it was observed an improvement in the rate of conversion to esters, when reaction is conducted at 60℃. These are 98.91 and 98.15% (w/w) of conversion for molar ratios of 6:1 and 8:1 respectively. This finding, allows to say that the ethanolysis of C. pentandra oil is very efficient at a reaction temperature of 60℃ than at 35℃. These results lead to define the optimal operating conditions for ethanolysis under basic catalysis after the transformation of free fatty acids into ethyl esters by the Fischer esterification reaction (Table 1). It appears that the increase in temperature allowed chemical equilib-rium to be reached faster by reducing reaction time. This behavior can be explained by exothermicity of transesteri-fication reaction by basic homogeneous catalysis as demonstrated by Nitièma-Yefanova and al. [18] during ethanolysis of seeds oils from *Balanites aegyptiacas*, *Azadirachta india*, and *Jatropha curcas*.

3.6. Trace Metals Elements (TMEs) in Vegetable Oil

Table 4. Trace elements (metals) in unconventional vegetable oil

Elements	Mass ISTD	CPS or Ratio	Concentration (µg/kg)	RSD (%)	Limit values (µg/kg)
Be	9	126.6772	<0.000	0.49	<500
Na	23	117,514.8	14.83	2.93	<500
Mg	24	262,885.6	111.1	1.19	<500
Al	27	3,982,431	28.25	1.41	<500
K	39	303,194.1	170.1	1.05	<500
Ca	43	712.0506	72.45	9.89	<500
Ti	47	194.6764	114.5	4.60	<500
V	51	7,405.419	<0.000	22.12	<200
Cr	52	392,151.3	2.884	1.42	<500
Cr	53	15,881.90	1.046	1.31	<500
Fe	54	503,831.3	40.73	2.17	<500
Mn	55	139,446.7	0.6853	1.61	<500
Fe	56	205,306.9	15.25	1.64	<500
Fe	57	5,361.346	21.22	0.81	<500
Co	59	849.3978	0.09560	3.49	<500
Ni	60	7,014.502	0.2450	1.90	<500
Cu	63	45,009.84	0.5404	2.11	<500
Zn	66	62,184.22	2.866	0.58	<500
As	75	1.333385	0.3079	15.17	<500
Se	76	5.333542	0.1325	41.59	<500
Se	78	7.333620	0.1475	21.20	<500
Se	80	19.33409	0.1477	10.51	<500
Se	82	11.33388	0.3815	19.75	<500
Sr	88	9952.990	<0.000	3.21	<500
Mo	95	3618.163	0.2290	4.38	<500
Ag	107	537.3674	0.04422	4.07	<500
Ag	109	228.6780	0.04610	4.80	<500
Cd	111	153.3384	0.06648	7.03	<500
Cd	114	438.7082	0.08781	0.51	<500
Sb	121	325.3639	0.09453	0.56	<200
Sb	123	304.0300	0.08971	1.63	<200
Ba	137	3210.547	0.6857	5.85	<500
Ba	138	21137.01	0.6880	2.02	<500
Ti	205	49.33793	0.03973	0.19	<200
Pb	206	7093.388	0.1734	2.26	<100
Pb	207	4642.677	0.1459	3.43	<100
Pb	208	11371.81	0.07561	3.91	<100
Th	232	1249.513	0.04359	1.08	<100
U	238	1697.609	<0.04000	0.76	<100

The results of analysis of TMEs in seed oil performed by inductively coupled plasma mass spectrometry (ICP-MS) are presented in Table 4. Low values of concentration of analyzed minerals, such as Na, Mg, k, Ca, Ti, Mn, Zn and Fe, were obtained, when compared to those reported by Hossain et al. [19] This variation could be due to the edaphic conditions and the chemical composition of the investigated seed oil. On the other hand, some metal trace elements such as sodium, potassium, magnesium could be naturally present in this oil or introduced by pollution during chemical reactions with seed oil. However, it should be noted that all the contaminants quantified give lower values (<500 µg/Kg) as recommended by the ISO Standard (22241-2). However, the presence of TMEs such as Strontium, Selenium, Molybdenum, Silver, Barium and Lead in low concentrations (< 0.0001 µg/kg) in the seed oil is detected. It should be noted that the presence of TMEs (lead, cadmium) in seed oil at very low concentrations can be eliminated by the development of operating conditions of refining. Thus, the establishment of a quantification plan of these trace elements is necessary to have a reliable database to comply with current standards. Overall, the quantification of trace elements has allowed the evaluation of contaminants in seed oil. Moreover, the knowledge of these trace elements in the seed oil is necessary to avoid the formation of undesirable products during the transesterification reaction in basic catalysis.

3.7. Contaminants in Biodiesel from *Ceiba pentandra* Seeds Oil

The results of the analysis of contaminants in biodiesel derived from seed oil of *Ceiba pentandra* are presented in Table 5 produced contains 14.83 µg/kg of sodium (Na) and 170.1 µg/kg of potassium (K). It should be noted that these trace metals (sodium and potassium) are residues of the catalysts (NaOH or KOH) and must be removed during the washing of the final product. Other elements such as Iron, Nickel, Copper, Zinc, Barium are also quantified but in smaller quantities. The values of the analyzed minerals are all lower than the ISO standard (22241-2) fixed at <500 µg/kg for biofuels. Thus, the analysis of biodiesel formulated by ICP/MS shows a decreasing concentration in this order: Na > K > Ti > Ca > Zn > Fe = Cu > Cr = Se > Ni = As = Ba > Ag > Sr > Co > Cd (Table 4). However, it is important to note that these inorganic elements may be present in the feedstock or introduced during the biodiesel production processes. Previous study has reported that they can occur during base-catalyzed ethanolysis and storage of formulated biodiesel or added to the fuel as an additive to improve diesel engine performance [20,21]. However, some study indicate that the main source of metals in biodiesel is the feedstock used [22]. In addition, the content of TMEs can be related to air and soil pollution. The main conclusion of this work is that a large number of elements were found, but with very low concentrations and in accordance with the specifications of the fuels.

Table 5. Contaminants analyzed in ethyl biodiesel

Elements	Mass ISTD	CPS or Ratio	Concentration (µg/kg)	RSD (%)	Limit values (µg/kg)
Be	9	46.67078	<0.000	0.65	<500
Na	23	2,075,743	484.9	0.67	<500
Mg	24	6,156.351	<0.000	0.34	<500
Al	27	779,155.0	4.809	1.57	<500
K	39	658,353.3	388.6	1.50	<500
Ca	43	212.0101	9.698	35.59	<500
Ti	47	5,961.846	86.30	5.74	<500
V	51	49.33539	<0.000	1.71	<200
Cr	52	4,939.811	0.5063	1.17	<500
Cr	53	12,681.05	0.8324	5.83	<500
Fe	54	3,604.646	<0.000	0.45	<500
Mn	55	21,501.72	0.02672	9.32	<500
Fe	56	59,818.48	3.533	1.09	<500
Fe	57	1,560.159	10.18	1.82	<500
Co	59	357.3525	0.05588	2.16	<500
Ni	60	5,060.143	0.1823	3.08	<500
Cu	63	61,630.70	0.7863	3.25	<500
Zn	66	27,751.16	1.095	1.17	<500
As	75	196.6762	0.2842	12.91	<500
Se	76	49.33541	0.4964	19.97	<500
Se	78	14.66724	0.05349	12.02	<500
Se	80	27.33440	0.1685	15.63	<500
Se	82	13.33396	0.4064	21.65	<500
Sr	88	1,522.822	<0.000	0.78	<500
Mo	95	3,596.820	0.2283	3.11	<500
Mo	95	312.0171	0.1454	5.96	<500
Mo	98	4,381.136	0.2160	1.78	<500
Ag	107	65.33604	0.01541	4.99	<500
Ag	109	66.00290	0.03615	2.05	<500
Cd	111	36.88996	0.04324	2.36	<200
Cd	114	325.3647	0.08549	0.81	<200
Sb	121	905.4363	0.1048	1.55	<500
Sb	123	662.7376	0.09804	1.76	<500
Ba	137	947.4085	0.2376	6.44	<200
Ba	138	6,392.596	0.2512	2.40	<100
Tl	205	108.0093	0.04010	0.23	<100
Pb	206	1,754.952	0.06482	3.00	<100
Pb	207	1,453.544	0.07559	2.60	<100
Pb	208	3,754.265	<0.000	2.00	<100
Th	232	3,870.318	0.05866	2.29	<100
U	238	142.6800	<0.04000	0.25	<100

3.8. Comparison of TMEs in Seed Oil and Biodiesel Produced

Contaminants found in the seed oil such as Na, Mg, Al, K and Ca are at lower concentrations than those found in the formulated biodiesel. This increase of the contaminants in the formulated biodiesel should be due to the catalysts

(KOH and NaOH) used during ethanolysis. The concentrations and responses obtained in number of courses per second show linear relationships. The regression coefficients are all greater than 0.99 for each trace metal element.

4. Conclusions

This work is part of optimization of operating conditions such as molar ratio ethanol: oil, amount of catalyst and temperature in order to improve yield of biodiesel by com-bined catalysis on one hand and on other hand, quantifi-cation of trace metal elements (TMEs) present or introduced in the seed oil and formulated biodiesel. The experiments of ethanolysis of oil from *Ceiba pentandra* seeds allowed to identify the optimal operating conditions to reach the maximum conversion rate to biodiesel. Thus, a yield of 98.91% (m/m) of ethyl esters was achieved at optimal op-erating conditions (ethanol:oil molar ratio of 6:1, tempera-ture 60℃ and a quantity of catalyst depending on acidity of oil used).. Moreover, identification of TMEs in oil of *C. pentandra* is important to understand their formation and their impacts in proper functioning of diesel engines and on environment. In addition, it should be noted that most of the trace metals are naturally present in this oil, but others are introduced into biodiesel formulated during the basic cata-lytic ethanolysis processes.

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