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THE ROLE OF EPOXIDATION ON CAMELINA *Sativa* BIODIESEL PROPERTIES

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ABSTRACT

An unstandarised biodiesel made from Camelina *sativa* oil, having over a 90 percent by weight of unsaturated fatty acids, were transformed into an epoxidised biodiesel that satisfy the requirements for iodine value, linolenic acid methyl ester content, cetane number, polyunsaturated fatty acids content, and viscosity established by the EN 14214 and ASTM D 6751 standards. The epoxidation reaction was carried out at 60 °C using peroxyacetic acid generated *in situ* and sulphuric acid as catalyst. A conversion of 60 % of double bonds was reached after 3 hours of reaction. However, only one hour was needed to get standard requirements and to avoid the epoxide ring opening side reaction that leads to hydroxyl groups. Besides, it was also observed that the formation of hydroxyl groups increases the kinematic viscosity of the biodiesel, being deleterious for the biodiesel properties.

Keywords: EN 14214; ASTM D 6751; Unsaturation degree; Iodine value; Cetane number; Linolenic acid methyl ester.

1. Introduction

Biodiesel, an alternative diesel fuel, is made from renewable biological sources such as vegetable oils and animal fats. This fuel is biodegradable and non-toxic and has low emission profiles as compared to petroleum diesel (Lapuerta *et al.*, 2008). The properties of biodiesel depend on the fatty acid composition of raw materials because the fatty acid profile of biodiesel is identical to that of the parent oil or fat. In turn, the properties of fatty acid methyl esters (FAME) are determined by the chemical structure of the fatty acids. Molecular weight, degree of unsaturation, branching of the chain, and the presence of chemical functional groups such as hydroxyl and epoxide affect the physical and chemical properties of biodiesel (Wandumesthrige *et al.*, 2009; Ramos *et al.*, 2009; Canakci and Sanli, 2008).

Camelina *sativa* (L.) Crantz, also known as false flax or gold-of-pleasure, is a broadleaf oilseed flowering plant of the Brassicaceae family (mustard, rapes, canola and crambe) that grows optimally in warm climates. Camelina *sativa* yields anywhere from 336 to 2,240 kg of seeds per hectare at maturity, with the lipid content of individual seeds ranging between 35 and 45 wt.% with a protein content within 27 to 32 wt.%.

The oil from Camelina *sativa* contains approximately 90 wt.% of unsaturated fatty acids. This unusual fatty acid pattern is the result of the abundance of C18:1 (12.8 – 14.7 %), C18:2 (16.3 – 17.2 %), C18:3 (36.2 – 39.4 %) and C20:1 (14.0 – 15.5 %) fatty acids. The high C18:3 content is incompatible with EN 14214 specifications and negatively affects biodiesel properties such as the iodine value, the cetane

number (CN), the oxidation stability index (OSI) and the linolenic acid methyl ester content. Other critical parameters are distillation temperature and the polyunsaturated methyl ester content (PUFA). For these reasons Camelina *sativa* biodiesel (CSB) presents various problems for motor fuel applications, being necessary to reduce its high degree of unsaturation, and molecular weight.

To improve the properties of biodiesel, various strategies can be carried out: use of additives, winterization, and alteration of the fatty acid composition or genetic modification (Wandumesthrige *et al.*, 2009; Kongyai *et al.*, 2013; Pérez *et al.*, 2010). The addition of additives to biodiesel is probably the simplest method. However, the problem of additive compatibility and the unintended effects on the other fuel properties are of concern. Therefore, the alteration of the fatty ester composition is often preferred and might be better (Kongyai *et al.*, 2013).

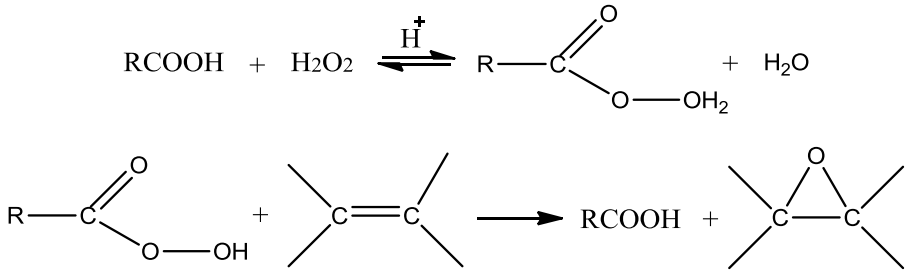

Figure 1. Mechanism of epoxidation reaction

Epoxidation is a reaction of a double bond with active oxygen, which results in the addition of an oxygen atom, converting the original double bond into a three membered epoxide currently named oxirane ring (Figure 1). In general, olefins can be epoxidised with peroxyacids, of which m-chloroperbenzoic acid has been the most often used. Other peroxyacids, especially peracetic and perbenzoic, have also been used (Gamage *et al.*, 2009). Epoxidation of vegetable oils has been extensively studied due to its impact on polymers and resins synthesis. For this reaction, an organic acid like acetic acid, or formic acid, is reacted with hydrogen peroxide to generate peroxyacetic acid as oxygen carrier. These peroxyacids can be used as a separate reagent or can be synthesised *in situ*, but the most common method is by using a peroxyacid generated *in situ* due to relative easy and safety manipulation (Gan *et al.*, 1992). *In situ* epoxidation of vegetable oil with H_2O_2 and acetic acid has been previously reported (Vijayagopalan and Gopalakrishnan, 1971; Gamage *et al.*, 2009). It was found that molar ratio of reactants, temperature and catalysts are critical in obtaining acceptable oxirane content. The epoxidation of a vegetable oil must be carried out with great care to prevent any possible side reactions. Figure 2 shows oxirane ring opening reaction that leads to hydroxyl groups.

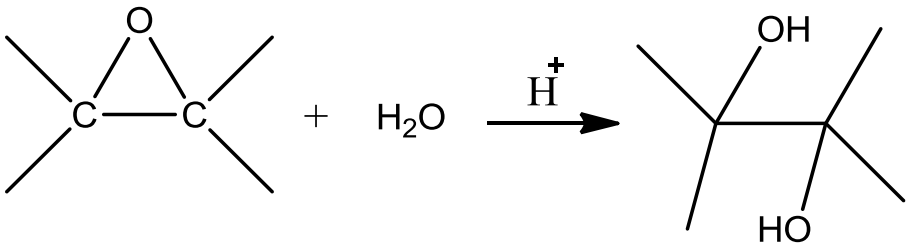

Figure 2. Oxirane ring opening reaction

Taking into account that Camelina *sativa* biodiesel does not meet the requirements of EN 14214 and ASTM D 6751 standards, the aim of this work is to enhance the biofuel quality modifying the chemical structure of fatty acid methyl esters by catalytic epoxidation. This treatment has not been described in literature before. To accomplish this goal, the catalytic epoxidation of the Camelina *sativa* biodiesel was carried out at 60 °C by using peroxyacetic acid generated *in situ* and the physical and chemical properties of the modified biodiesel (iodine value, cetane number, oxidation stability and linolenic acid

methyl ester content, etc.) were compared with those requirements established in the two biodiesel standards (ASTM D6751 and EN 14214).

2. Material and methods

2.1 Materials

Camelina *sativa* seeds were ground in Spain. All materials used in the extraction processes; oil characterisation, oil refining process, transesterification process, and characterisation of biodiesel were supplied by Fluka, Panreac, Sigma Aldrich, and Supelco. Reference materials used for biodiesel characterisation were supplied by Conostan, NIST Stanhope-Seta, IIS, IRMM and LGC Standards. Glacial acetic acid (>99.7%), aqueous hydrogen peroxide (50%), sulfuric acid (95-97%), 1-butanol (>99.5%), hydrogen bromide solution (33% in acetic acid), Tetrahydrofuran (THF) (>99.9%) were supplied by Sigma-Aldrich. Diethyl ether (99.7%), sodium hydroxide (98-100%), ethanol (96%), cyclohexane, Wij's reagent, sodium thiosulphate ethanolic solution (0.1 N) and potassium iodide were supplied by Panreac.

2.2 Experimental procedure

The oil was extracted from the seeds by using an oil press KOMET CA 59 G, with a feeding capacity of 5-8 kg h^{-1}, and an extruder of 0.5 mm. The extraction of the wet cake oil was performed using a Soxhlet apparatus. The oil refining procedure was performed as described by Fernández *et al.*, (2010). The biodiesel was synthesised on a laboratory scale via the conventional homogeneous alkaline catalysed transesterification using a methanol/oil molar ratio of 6:1 and sodium metoxide as catalyst. A molar ratio catalyst/oil of 0.1:1 was used (Casas *et al.*, 2010). The epoxidation of Camelina *sativa* biodiesel was performed in a jacket borosilicate glass reactor of 1 litre equipped with digital control of stirring from 40 to 2,000 revolutions per minute and a thermostatic bath for temperature regulation withing -10 to 100°C. This reactor is provided with a lid of three necks in which a reflux condenser, a thermocouple with temperature indicator and the head of stirring are located.

In this work, 0.3 kg of Camelina *sativa* biodiesel and 0.5 mol of acetic acid per mol of instauration were added to the reactor, being the temperature fixed at 40 °C and the agitation at 400 revolutions per minute. Once the desired temperature was reached, the catalyst H_2SO_4 (2% by weight of the aqueous phase) was added. Further, the required amount of aqueous H_2O_2 at 50% by weight (two moles per mol of insaturation) was added drop-wise during half an hour. The reaction was performed at a fixed temperature of 60 °C under an agitation of 900 revolutions per minute, and a nitrogen atmosphere during 4 hours, considering the completion of H_2O_2 addition as the initial time. For characterization purposes, samples were taken out at 30, 60,120 and 180 minutes. In the purification of the samples, diethyl ether was used as solvent to separate the organic phase from the aqueous phase in a separating funnel. The residual acetic acid and sulfuric acid in the organic sample were removed by ion exchange in a fix bed column filled with the strongly basic anionic exchange resin Amberlite IRA 402.

2.3 Analytical methods

The characterisation methods, analysis techniques and quality controls for the feedstock and the biodiesel were adopted from Carrero and Pérez, (2012). To assess the quality of the oil used in the production of biodiesel, the methods listed in the AOCS Analytical Guidelines Ck 1-07 were used. The biodiesel parameters were tested according to procedures described in the EN 14214 and ASTM D6751 standards. All measurements were carried out in triplicate, and no statistically significant differences were observed among them.

The percentage of oxirane rings was determined by direct method using hydrobromic acid solution in glacial acetic acid according to AOCS Official Method Cd 9-57. The determination of hydroxyl index, in mg KOH per gram of product, was carried out according to AOCS Official Method Tx 1a-66. The presence of functional groups in the biodiesel and the epoxidized biodiesel were confirmed qualitatively by FTIR spectroscopy.

The CN has been calculated based on the biodiesel methyl esters composition by using their individual cetane numbers, according to the equation 1 (Knothe, 2005)

$$CN = \sum CN_i \times w_i \quad (1)$$

where CN_i is the cetane number for an individual methyl ester, w_i is the mass fraction of each methyl ester, and i is a counter.

3. Results and discussion

3.1 Composition of Camelina sativa oil

The total oil yield from Camelina *sativa* seeds was 43.9 wt.%, obtaining a 38.29 wt.% with the mechanical press and 5.61 wt.% with the soxhlet extraction. This oil is constituted by saturated (8.63 wt.%), monounsaturated (32.96 wt.%), unsaturated of 2 or 3 double bonds (54.10 wt.%) and polyunsaturated with 4 or more double bonds (2.48 wt.%) fatty acids, being the linolenic acid (C18:3) the most predominant.

3.2 Characterisation of Camelina sativa biodiesel

The transesterification reaction was satisfactorily performed with a yield of 97.5 wt.% methyl ester content. The separation and purification post-treatments stages were performed properly since the product accomplished with the quality requirements of the EN 14214 standard (glycerol, methanol and metal contents). The properties of *Camelina sativa* biodiesel (CSB), requirements of the biodiesel standards ASTM D6751 and EN 14214, and the test methods are summarised in Table 1.

Viscosity, cetane number, cloud point, distillation curve and iodine value are directly related to the chemical composition of the CSB. Flash point, methanol content, metal content, sulphur level, acid number, and cold soak filterability are related to the purity of the CSB, the production process, transport and storage (Ciubota-Rosie *et al.*, 2013).

These specifications clearly demonstrate the important relationships between certain properties and highlight the significance of the computed average unsaturation, which is highly correlated with several other properties. Although the ASTM D6751 standard does not limit the iodine value (IV), the EN 14214 standard limits the IV to a maximum of 1.2 g I_2 g^{-1}. Thus, the iodine value for CSB (1.52-1.53 g I_2 g^{-1}) is very high and does not satisfy the EN 14214 standards. Engine manufacturers have argued that biodiesel with a high iodine value polymerizes at high temperature, forming deposits on injector nozzles, piston rings and piston ring grooves when these surfaces are heated (Mittelbach and Remschmidt, 2004).

The cetane number (CN) increases with the length chain of the fatty acid and decreases with an increase in the number of double bonds (Ramírez Verduzco *et al.*, 2012). This parameter is very important in the characterization of a biodiesel because it gives a measurement of the combustion quality during ignition. It provides information about the ignition delay of a diesel fuel upon injection into the combustion chamber. Biodiesel with low cetane number tends to cause poor combustion, smoking, and excessive carbon deposits on compression-ignition engine (Srivastava and Prasad, 2000).

CSB has an unsatisfactory oxidation stability of 1.3 h, which is lower than the specified values ASTM D6751 (OSI > 3 h) and EN 14214 (OSI > 8 h). Low oxidation stability is commonly found in fuels with a high content of polyunsaturated esters. Methylene groups adjacent to double bonds are particularly susceptible to be attacked by free radicals as the first step of fuel oxidation (Monyem and Van Gerpen, 2001).

According to the above the fatty acids profile (over 90 wt.% unsaturated fatty acids content) of biodiesel from Camelina *sativa* does not meet the EN 14214 and ASTM D 6751 requirements and thus, it is necessary its chemical modification, being the epoxidation of CSB proposed for this purpose.

Table 1. Standard requirements, Camelina *sativa* biodiesel properties and test methods.

Property	Units	ASTM D6751-12	UNE-EN 14214:2012	CSB	Test Method
Density at 15 °C	kg m^{-3}	-	860 – 900	888	EN ISO 12185
Kinematic Viscosity at 40 °C	$mm^2 s^{-1}$	1.9 – 6	3.5 – 5	4.3	EN ISO 3104
Cold Filter Plugging Point	°C	-	According to climate zone	- 4	EN 116
Cloud Point	°C	According to climate zone	-	0	ASTM D 2500
Cetane number	-	≥ 47	≥ 51	42.76	EN 15195
Methyl ester content	wt.%	-	≥ 96.5	97.5	EN 14103
Distillation temperature AET, 90 % recovered	°C	≤ 360	-	369	ASTM D 1160
Flash Point	°C	≥ 93	≥ 101	152	EN ISO 3679
Sulphur content	mg kg^{-1}	≤ 15	≤ 10	0.57	EN ISO 20846
Carbon residue	wt.%	≤ 0.05[a]	-	0.019[a]	EN ISO 10370
Sulphated ash content	wt.%	≤ 0.02	≤ 0.02	0.0013	ISO 3987
Water content	mg kg^{-1}	-	≤ 500	120	EN ISO 12937
Total contamination	mg kg^{-1}	-	≤ 24	7.3	EN 12662
Copper strip corrosion (3 h,50 °C)	classification	3	1	1A	EN ISO 2160
Oxidation stability, 110 °C	hours	≥ 3	≥ 8	1.3	EN 14112
Acid value	mg KOH g^{-1}	≤ 0.5	≤ 0.5	0.15	EN 14104
Iodine value	g I_2 g^{-1}	-	≤ 1.2	1.52	EN 14111
Cold Soak Filterability Test	seconds	≤ 360	-	246	ASTM D 7501
Linolenic acid methyl ester	wt.%	-	≤ 12.0	34.2	EN 14103
Polyunsaturated (≥ 4 double bonds) methyl esters	wt.%	-	≤ 1	2.08	EN 15779
Methanol content	wt.%	≤ 0.2 or flash point ≥ 130 °C	≤ 0.2	0.0121	EN 14110
Monoglyceride content	wt.%	0.4	≤ 0.7	0.579	EN 14105
Diglyceride content	wt.%	-	≤ 0.2	0.171	EN 14105
Triglyceride content	wt.%	-	≤ 0.2	0.107	EN 14105
Free glycerol	wt.%	≤ 0.02	≤ 0.02	0.006	EN 14105
Total glycerol	wt.%	≤ 0.240	≤ 0.25	0.189	EN 14105
Group I metals (Na+K) Group II metals (Ca+Mg)	mg kg^{-1}	≤ 5.0 ≤ 5.0	≤ 5.0 ≤ 5.0	0.11 0.16	EN 14538
Phosphorus content	mg kg^{-1}	≤ 10	≤ 4.0	< 0.1	EN 14107
Water & Sediment	% volume	< 0.2	-	0	ASTM D 2709

[a] (on 100 % sample)

3.3 Epoxidation of Camelina sativa biodiesel

Figure 3 shows the trends for different FAMEs as function of the reaction time. As it was expected, the epoxidation reaction decreases the unsaturation degree. Table 2 shows the properties of epoxidised Camelina sativa biodiesel (ECSB) with the reaction time.

The total unsaturated FAME decreased more than 40 % after one hour of epoxidation reaction. Obviously, the main contribution to this decrease was due to the abatement of the C18:2 and C18:3

compounds, attending to the highest original contents in the CSB. In the same way, a conversion of PUFA closed to 50 wt.% was obtained.

These results confirm that after one hour of the epoxidation reaction, ECSB satisfy the standard limits: Iodine value ≤ 120 g I_2/100 g, C18:3 ≤ 12 wt.%, PUFA ≤ 1 wt.%, kinematic viscosity values within 1.9-6 ASTM / 3.5-5 EN mm^2/s and cetane number ≥ 47 ASTM / 51 EN.

Table 2. Properties of epoxidised Camelina *sativa* biodiesel with reaction time

Property	CSB	ECSB 30 min	ECSB 60 min	ECSB 120 min	ECSB 180 min
C14:0 (wt.%)	0.05	0.1	0.1	1.4	4.6
C15:0 (wt.%)	0	0	0.3	1.2	4.2
C16:0 (wt.%)	5.2	5.6	6.2	8.3	15.8
C18:0 (wt.%)	2.7	2.9	3.2	4.3	8.1
C18:1 (wt.%)	15.2	13.2	10.6	9.1	12.4
C18:2 (wt.%)	17.9	15.9	11.2	7.9	0
C18:3 (wt.%)	34.2	24.7	12.0	8.1	7.8
C20:0 (wt.%)	1.4	1.5	1.7	2.3	4.4
C20:1 (wt.%)	15.1	13.3	10.8	9.4	13.0
C20:2 (wt.%)	2.2	1.8	1.3	0.9	1.3
C20:4 (wt.%)	1.5	1.0	0.5	0.4	0
C22:0 (wt.%)	0.3	0.3	0.3	0.5	0.9
C22:1 (wt.%)	2.6	2.3	1.9	1.7	2.6
C22:6 (wt.%)	0.62	0.6	0.5	0.5	0
Oxidation Stability, 110 °C (h)	1.30	1.66	2.25	2.71	2.93
Iodine Value (g I_2 g^{-1})	1.52	1.30	1.027	0.721	0.614
PUFA (wt.%)	2.08	1.6	1.0	0.9	0.0
Kinematic Viscosity, 40 °C (mm^2 s^{-1})	4.3	4.5	6.0	12.9	42.2
Cetane Number	45.8	48.8	52.7	55.1	60.9
Cloud Point (°C)	0	+ 1	+ 2	+ 7.3	+ 11

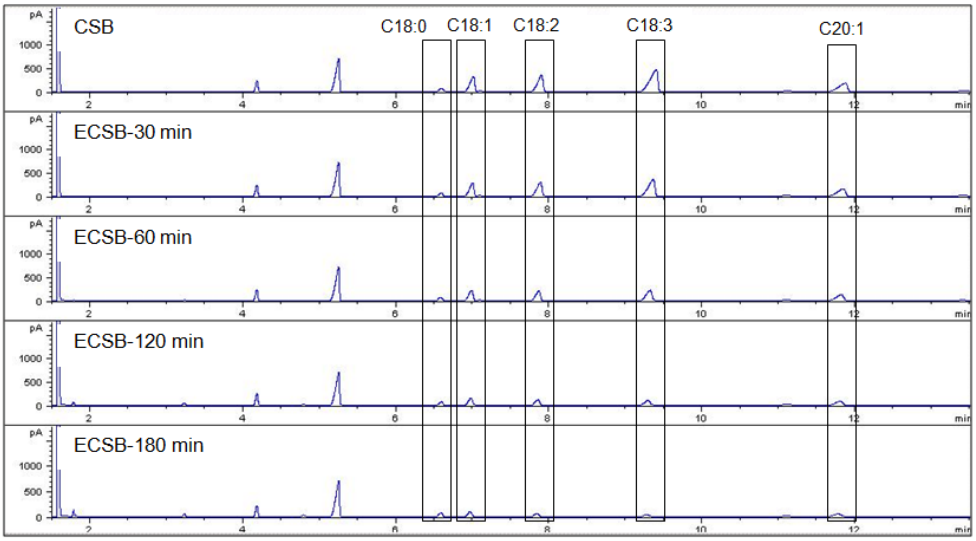

Figure 3. Chromatograms of CSB and ECSB

The epoxidation reaction causes the substitution of the unsaturations by the epoxy group, resulting in the decrease of unsaturated fatty acid content. Besides, the presence of different functional groups - unsaturated, epoxidized and hydroxyl-methyl esters may facilitate a closer packing arrangement, promoting the side-by-side parallel packing of the molecules, which would also result in an increase in

both cloud and flash points and also on the oxidation stability of epoxided biodiesel (Kongyai *et al.*, 2013).

As can be seen in Figure 4, a 60% of the unsaturated methyl esthers C18:1, C18:2, C18:3 and C20:1 was converted to oxirane rings and hydroxyl groups after 180 min under the epoxidation reaction. This figure shows that the secondary reaction -epoxide ring opening with water to form hydroxyl groups- takes place in this process. Besides, it is also observed that this reaction is turned into the main one for long time. These hydroxyl groups undergo a deleterious effect on epoxidized biodiesel (ECSB) which is critical because enlarge the viscosity (more than a 45 %).

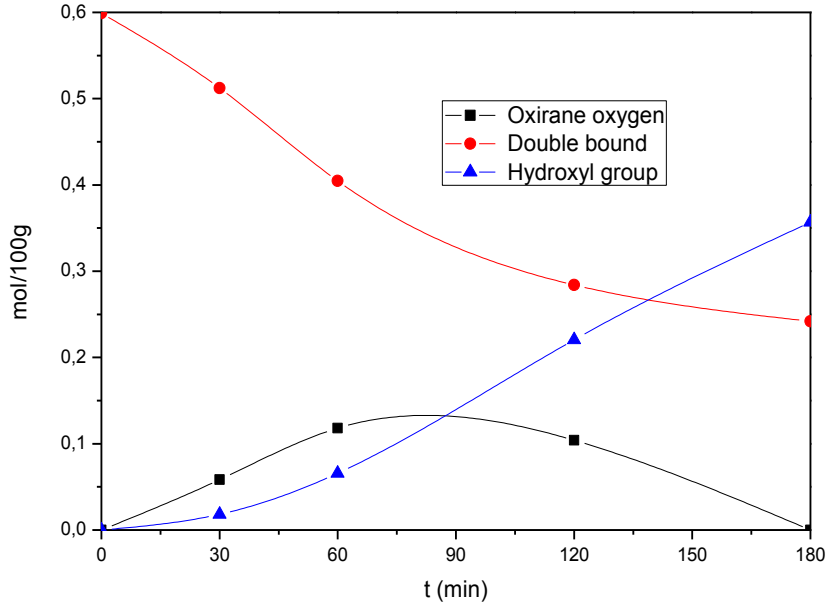

Figure 4. Variation of oxirane oxygen, double bonds and hydroxyl groups

The presence of functional groups in the biodiesel and the epoxidized biodiesel were confirmed qualitatively by FTIR spectroscopy as shown in Figure 5. Double bonds can be observed at the stretching bands 1656 and 3010 cm^{-1}, -OH groups present a strong stretching band at 3465 cm^{-1} and the characteristic doublet at 824–842 cm^{-1} represents the stretch of the oxirane ring.

This figure confirms that the higher the reaction time the lower the concentration of double bonds, the increase hydroxyl groups with time and how oxirane rings disappear for 180 min of reaction.

Finally, in order to achieve a higher conversion of double bonds to oxirane rings the secondary reaction must be avoided and suitable conditions must be researched.

4. Conclusions

The high content of unsaturated fatty acids of Camelina *sativa* biodiesel did not satisfy the requirements of EN 14214 and ASTM D 6751 standards. The epoxidation reaction allowed reaching a conversion of 60 % of double bonds after 3 hours of epoxidation reaction, modifying its properties. One hour of epoxidation was enough to transform the unstandarised biodiesel in an epoxidised biodiesel that satisfy the established limits for iodine value, C18:3, cetane number, PUFA and viscosity. Long reaction times cannot useful for this purpose because biodiesel viscosity increased sharply with the hydroxyl group content.

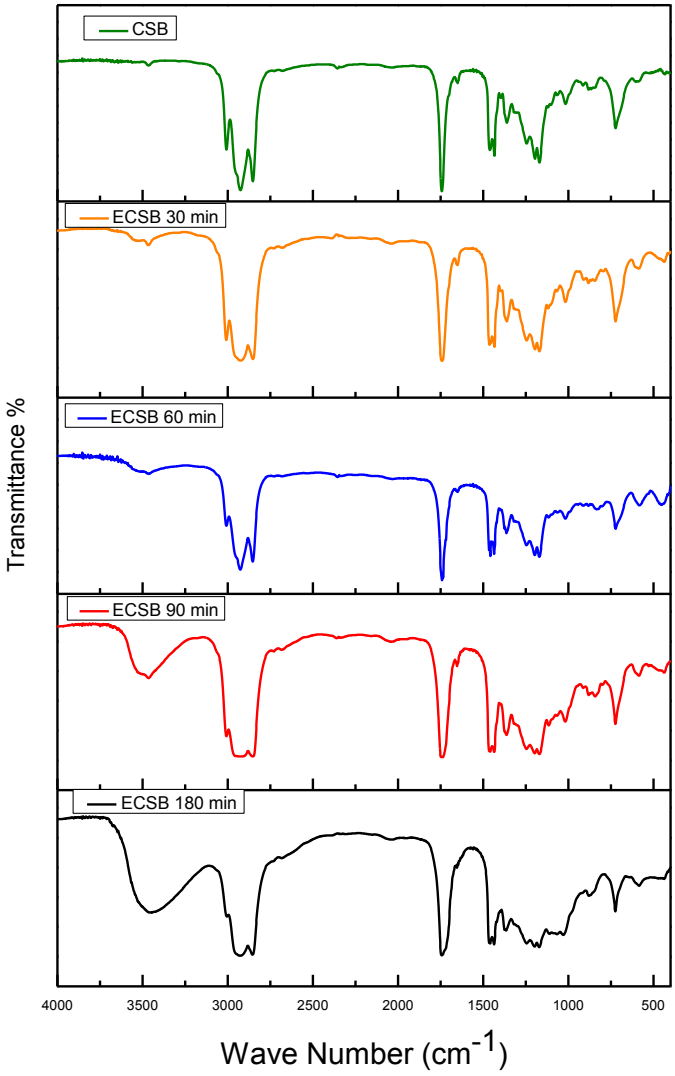

Figure 5. Comparation of FT-IR spectra of CSB and ECSB

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