International Journal Of Engineering Research And Development
e-ISSN: 2278-067X, p-ISSN: 2278-800X, www.ijerd.com
Volume 13, Issue 9 (September 2017), PP.31-44

Swell-Texturing assisted in-Situ Transesterification of Camelina Seeds Biodiesel

Fanar Bamerni[1,2], Ibtisam Kamal[1,3], Karim Allaf[1,]
[1]*University of La Rochelle, Laboratory of Sciences and Engineering for the Environment,*
LaSIE UMR-CNRS , France.
[2]*Faculty of Science, Chemistry Department, Zakho University, Iraq*
[3]*Faculty of Science, Soran University, Iraq*
*Corresponding Author: *Fanar Bamerni*

ABSTRACT:- Camelina seeds are a captivating source of vegetal oil, used for both food and industrial applications. The current work aims at intensifying In-Situ Transesterification (ISTE) using swell-texturing by Instant Controlled Pressure-Drop (DIC). This DIC swell-texturing produced twice more biodiesel (0.7731 against 0.39 35 g FAME/g ddb).

Keywords: Biodiesel; In-situ transesterification; Instant Controlled Pressure-Drop DIC swell-texturing; Modeling; Response Surface Methodology.

I. INTRODUCTION

Camelina (*Camelina sativa* L.) is a cruciferous oilseed plant belonging to the (mustard) family with different common names including false flax, Dutch flax, German sesame, Siberian oilseed and Gold of Pleasure ([1], [2], [3]). It is an ancient crop grown as far back as 1000 BC [4]. Camelina is indigenous native to northern Europe and Central Asia. It held promise as a source of edible oil and animal feed products in Eastern Europe in the middle ages. Conventionally, Camelina seeds were crushed and boiled to release oil for food, medicinal and lamp oil uses. Today, various processes such as cold pressing, solvent extraction or supercritical CO_2 extraction are used to produce this oil, whose fatty acid profile is so unique that it is a good candidate for large nutritional and bio-based industrial applications. The high amounts of unsaturated fatty acids allow this oil to have growing commercial prominence ([5], [6]).

Increasingly, Camelina is becoming a good alternative crop for industrial feedstocks [6],[7]), or biofuel ([2],[5],[8],[9],[10],[11]) because of its high oil content of 35%-45% ([6],[12],[13]) and more consistent yields estimated to be 106 to 907 L ha^{-1}. This is significantly greater than soybean (247-562 L ha^{-1}) and sunflower (500-750 L ha^{-1}). Furthermore, Camelina is much less weather dependent, and cheaper to produce than other new crops such as rapeseed, corn, and soybean. It also has been reported that Camelina exhibits some herbicidal properties, which could potentially reduce the requirements for tillage and weed control ([8], [14]). Also, Camelina oil has high content of two anti-nutritional factors (euricic acid and glucosinolates); these factors limit Camelina's feed competition with other feed and food crops [15].

In the current work, the possibility of producing biodiesel from Camelina seeds have been investigated. The route applied can be classified as a new generation or a more relevant second generation biodiesel type ([16], [17], [18], [19], [20]. [21]. [22]. [23]). In fact, Camelina can be grown in short marginal periods without being a competitor of food land since it can be used as a natural fertilizer when it is combined with edible crops on good arable land. On the other hand, issues with this 2nd generation biofuels were the cost-effective additional processes such as purification, needed to develop conversion of biomass to fuel. However, life-cycle evaluations of biodiesel produced from Camelina grown under different conditions showed a reduction in energy inputs and overall greenhouse gas emissions compared to diesel, as well as other biodiesel oleaginous crops ([8],[6],[24]). Furthermore, it was reported that the optimized Camelina biodiesel complied with the ASTM D6571 and EN 14214 standards related to biodiesel and are comparable to those of regular diesel fuels [5],[7]). Moreover, compared to microalgae source, Camelina has much lower water content. This presents a highly technical advantage of consuming much lower additional energy for cultivation, harvesting, dewatering and drying ([18], [20]). So, this novel fuel molecules made from Camelina, can greatly contribute to play a key role in the fuel market until the transesterification technology is economically realistic ([20], [23]).

Theoretically, numerous lipids such as Camelina oil and waste oils, can be used as a fuel directly [25]. Nevertheless, long-term engine durability studies show that fueling diesel engines with 100% vegetable oil causes engine failure [26] and significantly much lower thermal efficiency, more particularly because of the sticky nature (high viscosity) of the vegetable oils, mainly due to its high molecular weight. Thus, good use of oil may be obtained through various physical techniques, like dilution and composition, heating, etc. However, research works have been focused on chemically changing the vegetable oils into biodiesel, to get comparable properties to conventional petroleum diesel ([27], [25]) and be much more appropriate for current engines. The chemical reaction called transesterification (TE) converts lipids into biodiesel ([28], [29],[30],[31]). It is based on the transformation of fatty acids into fatty acid methyl esters (FAMEs). It requires numerous initial phases of oil extraction, filtration and refining, and, after achieving adequate transesterification, new separation and filtration processes required ([32], [33], [34]). Alkalis, acids, or enzymes catalysts are usually used to improve both rate and yields of this conversion reaction of lipids into fatty acid methyl ester ([35],[36],[37]). Acid catalysts act on both esterification and transesterification by avoiding saponification and FFA directly converted to ester through esterification, as well as glycerides converted to ester through transesterification. Whereas, bases only catalyze transesterification ([22], [29], [38]). The conditions of transesterification of unrefined Camelina oil were optimized to be, temperature of 33.6°C, molar ratio of methanol/oil 6.9:1, and acid catalyst concentration 1.66% leading to an optimized biodiesel yield of 95.6% wt., while solvent extraction of oil usually yields up to 81% [39].

To increase convenience and possibly reduce industrial difficulties of the transesterification, researchers and engineers proposed to directly execute this reaction inside the oleaginous matrix itself. This *In-Situ* Transesterification ISTE process effectively results in more appropriate industrial operation ([40], [41], [42], [28]). In this study of the appealing oleaginous plant of Camelina seeds, the combination of swell-texturing of oleaginous seeds and ISTE aims at relevantly intensifying biofuel production. The current work objectives have been to study the impact of instantaneous pressure drop (DIC) as a reliable swelling technique prior to ISTE transforming plant lipids in biodiesel in a pot reaction vessel, and to optimize DIC operating parameters it terms of FAME yields using Response Surface Methodology (RSM).

II. MATERIALS AND METHODS

A. Materials

The biodiesel feedstock was dried Camelina Sativa seeds harvested from France fields (Sanctum Mediterranean). The properties and characteristics of Camelina oil are assembled in

Table **1**. Chemicals; methanol 99.9%, toluene 98%, and anhydrous Sodium phosphate, as well as Isopropanol, potassium hydroxide, phenolphthalein indicator, iodine, glacial acetic acid, bromine water, potassium iodide, starch indicator, sodium thiosulfate and sulfuric acid 99% were obtained from Sigma-Aldrich.

Table 1. Camelina Oil Properties And Characteristics

Water content (g H2O/g db)	Free Fatty Acids Content wt.%	Kinematic Viscosity at 40 °C (mm^2/s)	Iodine Number (g I_2/100 g Oil)	Saponification Value (mg KOH/g Oil)
0.04	2.2±0.1	27.9±0.8	112.1±0.5	188.2±0.3

Acid number (mg KOH/g)	Density at 25°C (g/cm3)	Ester value (mg KOH/g)	% glycerin	Peroxide Value (m eq O2/kg)	Refractive Index at 25 °C
4.42±0.1	0.92±0.07	183.82	10.05	2.380±0.003	1.478±0.003

A. Treatments and transformations

In-situ transesterification operation

In-situ transesterification (ISTE) process for producing Camelina biodiesel, was performed under the following conditions: solvent type (Methanol: Toluene (90:10)), reaction temperature of 60°C, 600 rpm, catalyst type H_2SO_4-methanol (4%, w/v), and reaction time of 2 h [44]. With the objective to identify conditions of the highest yields (Y) of Fatty Acid Methyl Esters FAMEs, the impact of solvent/seeds ratio (S) and catalyst/solvent ratio (C) were investigated as experimental parameters. Preliminary experiments allowed us to identify the ranges of S from 10:1 to 50:1 (v/w) and C from 1% to 10% (v/v of solvent). Each trial started by separately preparing a reactive mixture with adequate amounts of solutions of methanol/toluene, and acid catalyst in a 500-mL round bottom flask with reflux condenser, the mixture was heated using a magnetic stirrer hot plate (LabT Ech, korea). The mixture was shaken at 600 rpm until the catalyst was completely dissolved, and at the same time, pre-heated to the desired reaction temperature (60°C). A predetermined amount of Camelina seeds according to the experimental design in Table 2, was soaked in 10 ml of the reactive mixture for 10 min., and then charged to the round bottom flask when the methanolic/catalytic solution had reached the desired temperature. The reaction was kept at 60°C with mechanical mixing at constant speed (600 rpm) for 2 hr. After the reaction completion, the round bottom flask was cooled to room temperature. The cooled mixture was filtered, and sodium hydroxide was added to the filtrate to neutralize the acid catalyst, thus ensuring that the transesterification reaction had completely stopped. On the other hand, fundamental studies of solvent extractions and/or solvent reactions performed on vegetal materials, have demonstrated that the kinetics of such operations are well controlled by the mass diffusivity which normally proceeds with weak kinetics and poor yields [45]. However, the intensification of such processes can normally be done through a texturing stage of the raw vegetal materials to get higher porosity and, then, greater diffusivity. Hence, the instant controlled pressure drop process is a specific texturing way for enhancing the extraction and/or solvent reaction processes without involving thermal or chemical degradation.

Texturing by DIC as pretreatment for In-Situ Transesterification ISTE

The definition and first studies of the Instant Controlled Pressure Drop (DIC) process were carried out in 1988, mainly based on the thermodynamics of instantaneous transformations. Since then, DIC technology has been used and successfully applied at both laboratory and industry scales to improve structure expansion, processing kinetics (drying, extraction of volatile compounds and non-volatile compounds, bleaching and sterilization). Usually, DIC has resulted in higher process performance, better quality of final products in terms of functional and organoleptic properties of natural products, and equipment reliability ([46], [47], [48]).

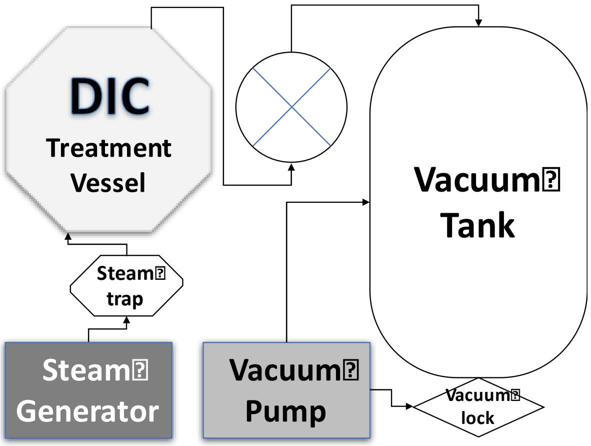


Fig. 1: Schematic diagram of the industrial-scale DIC unit.

DIC is a High-Temperature/Short-Time process. It involves a first stage of introducing in the treatment vessel fresh and/or partially dried materials, heating them usually up to 160 °C using high-pressure (up to 0.8 MPa) of saturated steam, for a short period of time (some seconds or dozens of seconds). Once the equilibrium of both temperature and water content within the product is attained, the second main stage is performed inferring an abrupt pressure-drop whose rate $\Delta P/\Delta t$ is higher than 0.5 MPa s^{-1}, towards a vacuum (usually at 5 kPa). Fig. 1 shows schematic diagram of industrial scale DIC set-up.

Since porosity and texturing ratio normally depends on both amount of auto-vaporized water, and rheological behavior involving glass transition of the product, the effects of DIC on *in-situ* transesterification is dependable on DIC temperature, vacuum level, and pressure-drop rate. Hence, the authors decided to carry out a comparative study of ISTE performances of un-textured and differently DIC-textured Camelina seeds. Both DIC vacuum level (3.5 kPa) and pressure drop rate (65 MPa s^{-1}) were kept constant based on preliminary experiments. Consequently, the effects of other DIC operating parameters including treatment temperature T, (115-165 °C), and processing time t, (15-45 s) on biodiesel (Fatty Acid Methyl Esters FAMEs) yield were investigated. It is worthy to note that the DIC treatment temperature is strictly correlated with DIC saturated steam pressure.

Assessment methods

Measurements of characteristics of Fatty Acid Methyl Esters FAMEs were conducted after FAME purification. Hot distilled water was added to the crude FAME filtrate produced from each experiment (2/1) v/v, then the solution was transferred to a separation funnel. The upper FAME layer was separated, washed with distilled water several times until removing the traces of the acidic catalyst. The solvent was evaporated using rotary evaporator (55-60°C) under vacuum conditions (Buchi R-215, Switzerland). The obtained FAMEs (i.e. biodiesel) were dried over adding 0.5 gm of anhydrous sodium phosphate then filtrated to remove the sodium phosphate. Finally, the FAMEs were collected with molecular sieves to make sure getting rid of the remnants of moisture in a dark glass container and kept in the refrigerator. The biodiesel yield was calculated using Eq. 1:

$$Y(biodiesel\ yield)\% = \frac{Weight\ of\ biodiesel\ (g)}{mass\ of\ seeds(g)} * Lipid\ content\ (\%) \qquad Eq.\ 1$$

To determine FAME density, two methods can be used, the pycnometer method (EN ISO 12185) and hydrometer method. Pycnometer is the most precise and accurate method. Indeed, hydrometer is affected by many factors such as temperature, alcohol present in biodiesel, etc. making measurements not accurate. The detailed method for measuring FAME density has been reported in literature [49].

Specific gravity or Relative density (RD) is an interesting FAME parameter. It is the ratio of the material to water densities at a selected temperature, usually 15.6℃ (60℉) [49]. API gravity is defined by the American Petroleum Institute to characterize heavy and light weight fuel fractions. Although density, specific gravity, and API gravity are not included in the specifications outlined in the ASTM standards for petrodiesel (D 975), biodiesel (D 6751), or biodiesel/petrodiesel blends (D 7467), a minimum API gravity at 30° is recommended to maintain good power production and fuel economy for engine manufacturers [51], API gravity can be calculated using equation 2:

$$API = \frac{141.5}{SG\ at\ (15.6^{\circ}C)} \text{-}131.5 \qquad Eq.\ 2$$

On another hand, there are various instruments used within industry practice to accurately estimate, determine, and predict kinematic viscosity ASTM D -445. They include capillary, orifice, and rotational viscometers. Capillary viscometers are frequently used to measure the viscosity of a transparent Newtonian liquid. They are simple and demand only a small volume portion of the liquid fuel. In the recent study, capillary viscometer (Viscometer, Cannon Fenske Routine, Eurostar Scientific LTD,PSL ASTM-IP 100, UK) was used following the method reported in literature [52].

The acid number EN 14104 of biodiesel samples was measured using titration methodology to determine the amount of existent FFA [49].The iodine value IV reveals the number of double bonds in a FAME sample. It is expressed in g of I/100g of biodiesel and specifies the quantity of iodine in gram that are consumed by 100 g of the biodiesel sample under specific conditions. The determination of IV is conducted by titration with a solution of sodium thiosulfate following the European standard EN 14111 [49].Along with, the chemical and physical properties of fuel play important role in delay period of fuel combustion. The Cetane Number (CN) of the fuel is the most significant parameter which is responsible for the delay period [53]. The higher the carbon chain length, the higher the cetane number. Thus, cetane number usually accepted by diesel engines should be ranged between 40 and 55 [54]. In this study, Cetane number was determined by Zeltex 101C, Portable Near-Infrared Octane/Cetane Analyzer (Hagerstown, MD 21740, USA). The detailed method to estimate cetane number is highlighted in literature [55]. Near Infrared (NIR) analyzer is used to obtain the absorption spectrum of diesel which then is fed to a chemometric model. Since its reproducibility depends on the engine, the same engine is used as reference [56].Furthermore, numerous Equivalent Test Methods (IP 13, ISO 6615, DIN 51551, JIS K 2270, and AFNOR T60-116) can be used to assess the value of Conradson carbon residue produced from burning fuel. This is an approximation of the fuel tendency to compose deposits in vaporizing pot-type and sleeve-type burners. The detailed D 189 method is reported in literature [57].

Also, the Cold Filter Plugging Point (CFPP) of a fuel is suitable to estimate the lowest temperature at which a fuel will give trouble-free flow in specific fuel systems. The detailed method ASTM D 6371 has been reported [57]. This test method is technically equivalent to test methods IP 309 and EN 116 [58].Besides, equivalent Test Methods of IP 15, ISO 3016, DIN 51597, JIS K 2269, and AFNOR T60-105 can be used to determine the “Pour Point". Pour point is an index of the lowest temperature of its utility for certain applications. The detailed method has been reported in literature [57]. On another hand, Cloud point is the temperature of a liquid fuel when the smallest observable cluster of wax crystals first appears upon cooling at fixed conditions. The detailed method ASTM D 2500-02 was reported in literature [59].

Moreover, freezing point increases with molecular weight, and is strongly influenced by molecular shape. Molecules that fit more readily into a crystal structure have higher freezing points [50]. Freezing point determined by Oil Product Low-temperature Characteristics Meter (OPLCM, SHATOX Co. LLC, Tomsk Russian Federation) meet all ISO 9001 requirements [60]. Also, Flash point is the tendency of the test specimen to form a flammable mixture with air under controlled conditions. The Flash point was determined by (*GD-261-1* Pensky-Martens *Closed Cup Flash Point Tester* Chongqing Gold Mechanical & Electrical Equipment Co., Ltd, China). It is equivalent to other test methods IP 34, ISO 2719, DIN 51758, JIS K 2265, and AFNOR M07-019 [59].

Response Surface Methodology (RSM)

Response surface methodology RSM is a collection of mathematical and statistical techniques based on the fit of a polynomial equation to describe the experimental data of dependent variables versus number of factors (independent variables). The objective of using RSM is to develop procedures that would bring a better understanding of the combined effects of the key processing variables on the desired responses to attain the best mathematical system performance of statistical previsions. RSM is normally used after performing an adequate Design of Experiments DoE selecting operating parameters and their own ranges. This is normally based on collecting data from literature, initial experience, and preliminary experiments. Statgraphics for Windows software (5.1 version), SIGMA PLUS Neuilly/Seine (France) for designing experiments and statistically treating the responses was used, leading to a generalized 2nd polynomial empirical model. This is to express the response Y (dependent variable) versus the independent variables X_i and X_j:

$$Y = \beta_0 + \sum_{i=1}^{n} \beta_i \chi_i + \sum_{i=1}^{n} \beta_{ii} \chi_i^2 + \sum_{i=1}^{n-1} \sum_{j=2}^{n} \beta_{ij} \chi_i \chi_j \qquad Eq.\ 3$$

Where β_0, β_i, β_{ii}, and β_{ij} are the regression coefficients for intercept, linear, quadratic and interaction terms, respectively estimated by the model.

In this study, two distinct 2-operating parameter Central Composite Designs CCD with, each, 2^2=4 factorial points, 2*2 star-points and 4 repetitions of central point, were used to optimize: 1/ The operations of *In-Situ* Transesterification ISTE transformation parameters (S: seeds/solvent ratio and C: catalyst/solvent ratio) for Camelina raw material, and 2/ The DIC texturing parameters ; the processing temperature, T and the treatment time, t. Response was principally the yields of AFMEs produced by the ISTE *reactions*.

Experimental design of ISTE for the fresh (un-textured) Camelina seeds

The coded and natural levels of *in-situ* transesterification (ISTE) processing parameters, which follow a central composite design CDD for different experiment conditions carried out on raw Camelina seeds are listed in

Table **2**. These processing parameters used as independent variables were the solvent/seeds ratio S, and the catalytic /solvent ratio C.

Table **3** shows the FAME yields Y as response value (dependent variable) expressed in g FAME/ g ddb (dry dry basis) for different CDD trials.

Table 2. Real and coded values of independent parameters (solvent/seeds ratio S, and catalytic /solvent ratio C)

Coded level	-α	-1	0	+1	+α
S: Solvent / Seeds (ml/g)	10	15.9	30	44.1	50
C: Catalyst/ Solvent (v/v %)	1%	2.3%	5.5%	8.7%	10%

Table 3. Trials of central composite design CDD for ISTE of raw Camelina seeds. The independent parameters: solvent/seeds ratio S, and catalytic /solvent ratio C, FAME yields Y as response (dependent variable)

Exp. No.	S: Solvent solution: seed weight (v/w) (ml/g)	C: Catalyst/Solvent (v:v)% of solvent solution	Y: ISTE-FAME (g FAME/g ddb)
1, 4, 7, 10, 13	30	5.5%	0.3066±0.0005
2	50	5.5%	0.3815
3	30	10%	0.3399
5	44.1	8.7%	0.3626
6	44.1	2.3%	0.3494
8	15.9	2.3%	0.2573
9	15.9	8.7%	0.2678
11	10	5.5%	0.2414
12	30	1%	0.2793

Experimental design of ISTE for (DIC-textured) Camelina seeds

After optimizing the *in-situ* transesterification (ISTE) conditions for the raw untreated Camelina seeds, another 2-parameter 5-level central composite design CDD was adopted to study the effect of DIC operating parameters on FAME yields. 13 DIC-textured samples were *in-situ* transesterified using the optimized processing conditions (catalyst:solvent, 10% v/v) and (solvent/seed ratio, 50:1 v/wt) estimated from the response surface analysis (RSA) of the data listed in

Table **3**.

The investigated DIC operating variables (independent variables) were the treatment temperature T, and the processing time t. The coded and natural levels of the independent DIC parameters are listed in Table 4.

Table **5** shows the FAME yields Y as the response values for different ISTE trials. The saturated steam pressure was not analyzed by RSM. Its levels were listed in Table 4 and

Table **5** just to express the normal close dependence of T and the saturated steam pressure P.

Table 4. Values of DIC independent parameters (processing temperature, T and processing time, t)

Coded level	-α	-1	0	+1	+α
DIC processing temperature T (°C)	115.0	122.3	140	157.7	165.0
DIC saturated steam pressure (MPa)	0.17	0.21	0.36	0.58	0.7
DIC Treatment Time t (s)	15.0	19.4	30.0	40.6	45.0

Table 5. Trials of central composite design for DIC swell-texturing treatment of raw Camelina seeds. DIC processing parameters are temperature, T and treatment time, t as independent variables.

No.	Treatment Temperature T (°C)	Processing time t (s)	Saturated steam pressure (MPa)	ISTE FAME Yields (g FAME/g ddb)	
				Observed values	**Fitted values**
Control	/	/	/	0.3935	
DIC1, 4, 7, 10, 13	140	30	0.36	0.66 ± 0.01	0.6592
DIC 2	165	30	0.7	0.6056	0.6662
DIC3	140	45	0.36	0.5859	0.6369
DIC5	157.7	41	0.58	0.7731	0.7081
DIC6	157.7	19	0.58	0.6083	0.5680
DIC8	122.3	19	0.21	0.5742	0.5719
DIC9	122.3	41	0.21	0.5926	0.5657
DIC11	115	30	0.17	0.5616	0.5684
DIC12	140	15	0.36	0.5260	0.5422

III. RESULTS AND DISCUSSION

B. *RSM modeling and optimization of* In-Situ *Transesterification for fresh (un-textured) Camelina seeds*

Response Surface Analysis was applied to model and optimize the effect of the ISTE parameters on FAME yield. The experimental ISTE data were fitted to a mathematical model and the model was tested for significance and suitability. The significances of all terms in a second-degree polynomial equation were statistically analyzed through Pareto charts based on ANOVA value with p-value of 0.05. The model is analyzed using the lack-of fit test, coefficient of determination (R^2) and adjusted-R^2 analysis. The larger the value of R^2 (up to 1), the more adequate the model is versus the experimental results.

The RSA results of the experimental data listed in (

Table **3.**) were reported and plotted in (Fig.2: Pareto-charts, general trends, response surface, etc.). The polynomial multiple linear regression model (Eq. 4) is estimated. The vertical line on Pareto chart determines the effects that are statistically significant with a p-value of 0.05. The standardized effect is the estimated effect divided by its standard error. Hence a low standardized effect can mean either a low effect of the parameter or a too large experimental range. The estimated second order polynomial model (p-value <0.05) showing the effect of the in-situ transesterification conditions on biodiesel yield is shown in Eq. 4:

$$Biodiesel\ yield = 19.633 + 0.2692S + 0.2544C + 0.001057S^2 + 0.001496S + 0.01169C^2 \quad Eq.\ 4$$

where C is the catalyst concentration (v/v) %, S is solvent/seeds (v/w), R^2 = 97.55%, Adjusted R^2 for d.f. = 95.80%.

Fig.2 shows the estimated RSA plots reflecting the effect of the ISTE operating parameters in terms of biodiesel yield (g FAMEs/g of ddb) Camelina seeds. It is well noticed that both independent variables were significant. However, S (solvent/seeds ratio) had the most significant impact. This finding can be easily explained considering that increasing the solvent/seeds ratio would enhance the interaction between the reactants whatever the porosity, diffusivity and lipid availability in the raw material are, thus increasing the reaction kinetics and the biodiesel conversion yields. As shown in Eq. 4, only the linear terms were significant and their regression coefficients had positive coefficient values. This indicates that the higher the amount of solvent/seed ratio and the acid catalyst concentration, the higher the FAME yield. Similar findings were reported in the literature [9], [5], [10], [11], [6]. The amount of alcohol in the reaction plays a vital role. Indeed, since the transesterification is an equilibrium reaction, the molar ratio of methanol to total fatty acids used for ISTE is normally higher than the stoichiometric value to favor product formation. The highest experimental value of biodiesel yield for the un-textured raw Camelina was 0.3815 g FAMEs/g ddb. The empirical model estimated from RSA allowed identifying the optimal value of biodiesel yield to be 0.4020 g FAMEs/g of ddb Camelina seeds achieved at S = 50 ml solvent/g of Camelina seeds, and C = 10 %.

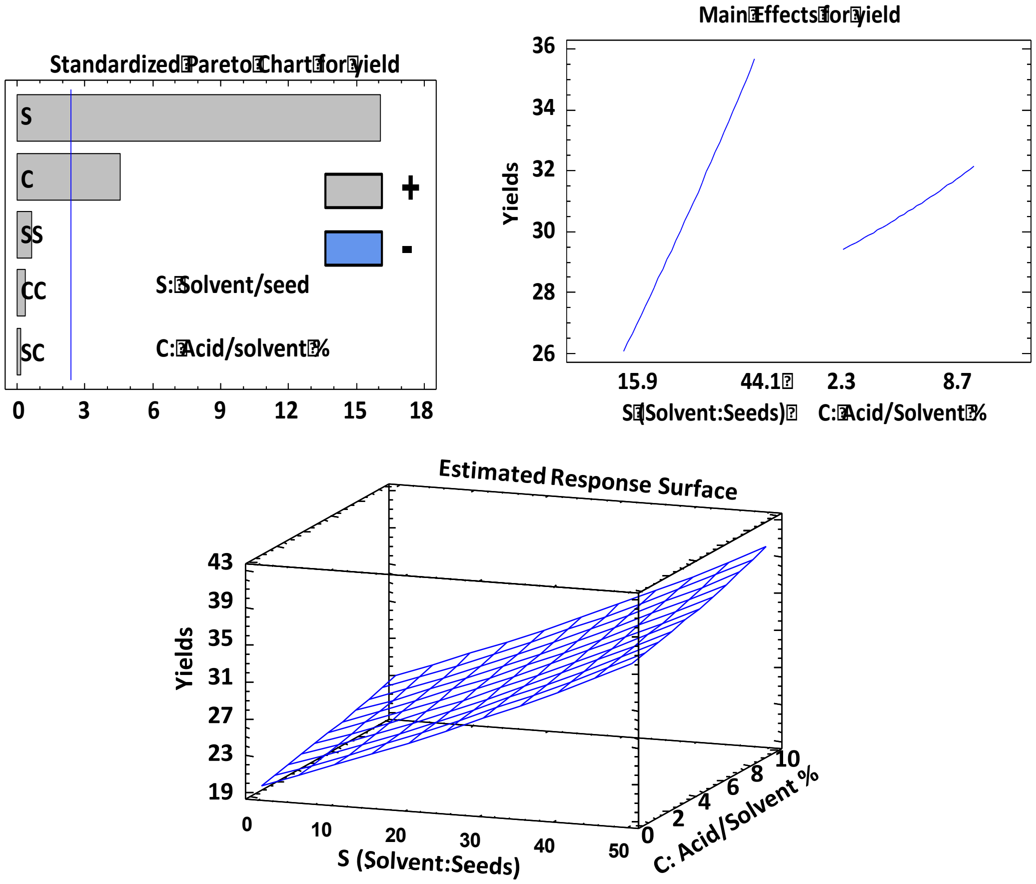


Fig.2: Pareto chart, general trends, and response surface of the effect of solvent/seeds ratio and catalyst/solvent ratio on biodiesel FAME yield (g FAMEs/100 g ddb)

C. RSM modeling and optimization of the impact of DIC-texturing of Camelina seeds on FAME yield obtained by *In-Situ* Transesterification

The optimized in-situ transesterification reaction conditions estimated from the RSM analysis of the raw seeds were applied to study the impact of DIC process on FAME yield throughout ISTE of 13 DIC-textured samples (Table 4 and 5). The ISTE experimental results are plotted in Fig. 3. It is obvious to note that all the DIC treated samples had higher FAME yield compared to the un-textured raw material. A significant increase in FAME yield was observed for Sample no. 5 (DIC-textured at 157.7 $^{\circ}$C for 41 s) which had approximately twice more FAME yield (0.7731 g FAME/g ddb seeds) compared to (0.3935 g FAME/g ddb seeds) from the un-textured raw material.

To analyze the experimental data, Statgraphic methods were exploited. The estimated effects and interactions can be reflected by p-values, which allowed DIC operating variables; treatment time, t and temperature, T to get significant impacts on FAME yields, for p-value < 7%. F-Ratio and P-Value estimated from ANOVA corresponded to DIC operating variables are listed in Table 6.

Table 6. ANOVA data of ISTE FAME yields Y (dependent variable) versus various terms of independent parameters of DIC temperature, T and time, t

Source	DIC Treatment Time t	DIC Treatment Temperature T	t^2	tT	T^2
F-Ratio	4.64	4.95	4.36	2.77	1.58
P-Value	0.0683	0.0614	0.0751	0.1401	0.2491

The standard error of the fitted values shows 0.0440 as a standard deviation of the residuals, while the average value of the residuals is revealed by 0.02386 as the mean absolute error (MAE).

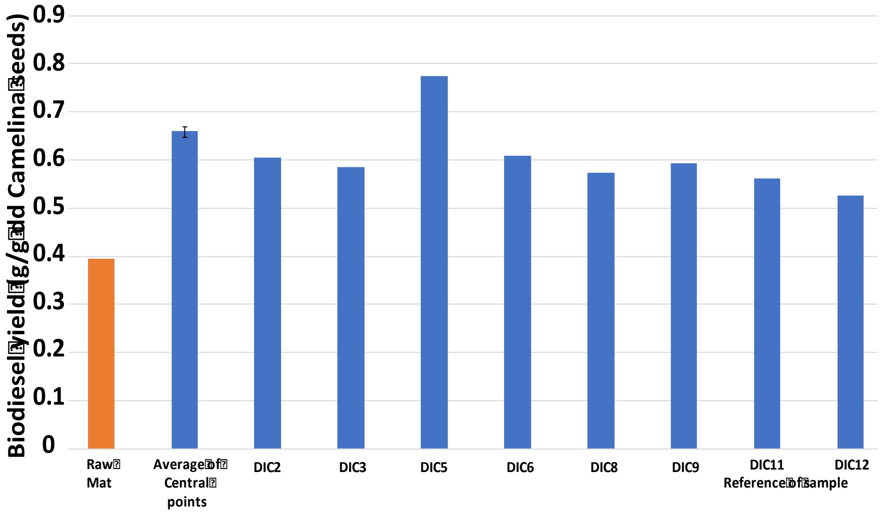


Fig. 3: ISTE biodiesel yields for the raw un-textured (Conventional ISTE process: in red) and (innovative DIC/ISTE process: in blue) from Camelina seeds

To plot the estimation of importance, Pareto Charts (Fig. 4) reveals the statistical significance of the effects; however, the estimated P-value was greater than 5.0 %, revealing that there is no indication of serial autocorrelation in the residuals at the 5.0% significance level. Accordingly, the DIC processing variables within the range applied in the current work seemed not significant, however the estimated regression factor R^2 equaled 71.7% and adjusted R^2 was limited to 51.4%. The polynomial second order equation showing the effect of DIC operating variables on FAME yield is shown in equation 5.

$$Y = \text{-}0.4831\text{-}0{,}0056t + 0{,}0148T + \text{-}0{,}0003t^2 + 0{,}0002tT\text{-}0{,}00007T^2 \qquad \textit{Eq. 5}$$

Where T is the DIC processing Temperature, t is the DIC treatment time, and Y is FAME yield.

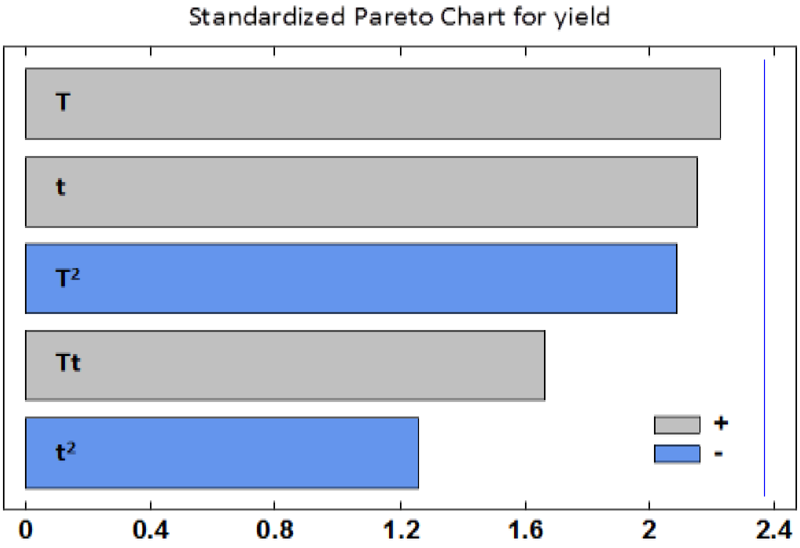


Fig. 4: Standardized Pareto Chart (p-value=5%) of the processing temperature T and the processing time t for ISTE yield of DIC textured Camelina seeds.

The highest FAME yields was estimated to be $Y_{optimized}$ = 0.72 g FAME/g ddb correlated with DIC-textured Camelina seeds at a Treatment Time $t_{optimized}$ = 43 s and a processing temperature $T_{optimized}$ =165 °C. The contour plots (Fig. 5) clarify the situation. These optimized DIC conditions allowed the extensive yield of biodiesel to be so moderate that it resulted in a convenient, high performance and cost-effective *in-situ* transesterification manufacturing biodiesel.

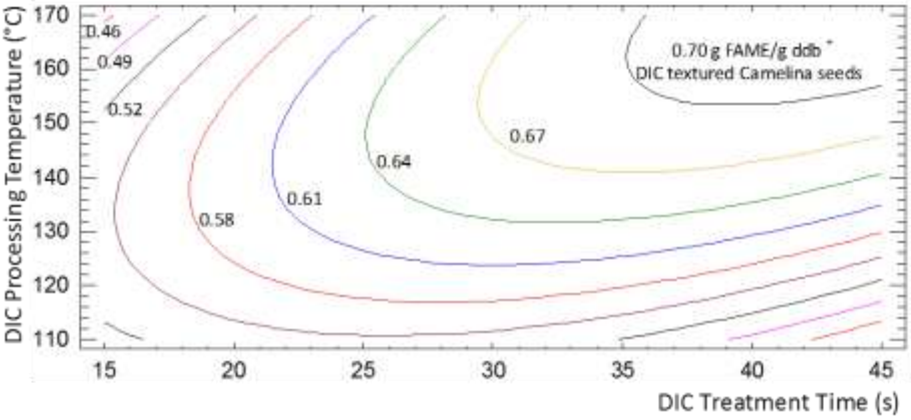


Fig. 5: Contours of estimated response surfaces of the effects of DIC processing temperature, T and processing time, t on FAME yield of DIC textured Camelina seeds

D. *Evaluation of Fuel Properties of Camelina Biodiesel*

The properties of ISTE biodiesel prepared from Camelina seeds DIC-expanded at 158 °C (Saturated steam pressure of 0.58 MPa) for 41 s, are summarized in Acid value or neutralization number is a measure of free fatty acids contained in a fresh fuel sample or issued from degradation in aged samples. In the current operation, the acid value was 0.33 mg KOH/g which is within the ASTM D 6751-09 and UNE-EN 14214 standards [70]. This means lower corrosion and minor engine deposits, particularly in the fuel injectors [71].

The iodine value is the measure of unsaturation degree in oil. Iodine value is a useful parameter in studying oxidative rancidity and chemical stability properties of different oils and biodiesel fuels. Higher quantity of double bonds in the sample has greater potential to polymerize and hence lesser stability [72]. The iodine value in the current study was lower than all other researches' (145 against 151 – 166.2 g I2 /g) while particularly high and exceeds the allowable limit specified in PN-EN 14214 (<120 g I2/g). This should be correlated with the presence of great amount of unsaturated fatty acids in Camelina oil.

Table 7 together with the standards specified in the ASTM D6751 and EN 14214 as well as the published values in literature. The characteristics investigated were Cloud point, Pour point, Cold filter plugging point, Kinematic viscosity, Acid value, Iodine value, Cetane Number, Density, Flash point, Carbon residue, Water content, Specific gravity, API, Calorific value, and Freezing point.

The density, kinematic viscosity, and acid number were tested. All of them adhere to the specifications in ASTM D6751 and EN 14214 standards. The relative density of DIC/ISTE FAME was slightly lower than conventional fuel (0.882 vs 0.91 to 0.887). This has a direct influence on the volumetric fuel consumption and fuel performance. Moreover, the lower the fuel density, the higher the quality of atomization (spraying) and combustion. Although biodiesel density can progress depending on the conditions of manufacturing, storage, transportation and distribution process, this impact keeps relatively interesting because lowering the fuel density causes the reduction of the particulates emission. In some cases, lower fuel density also causes the decrease of the nitrogen oxides emission. Besides, decreasing the fuel density is linked with the drop of its calorific value, which affects the engine performance. The density of esters depends on the molar mass, the free fatty acid content, and the water content ([61] and [62]).

Additionally, DIC/ISTE fuel presented cetane number, heating value and viscosity strongly correlated normal requirements of engine. Kinematic viscosity of DIC/ISTE biodiesel was well-located in the normal range of conventional fuel (3.58±0.03 against 3.5-5 cSt for ASTM D 445). Thus, this low level of kinematic viscosity of our DIC/ISTE biodiesel positively affects the start of injection, injection pressure and the fuel spray characteristics, which are the main parameters that define engine performance and exhaust emission. It is worth noting that kinematic viscosity increases with chain length of either the fatty acid or alcohol moiety in a fatty ester or in an aliphatic hydrocarbon, the increase in kinematic viscosity over a certain number of carbons is smaller in aliphatic hydrocarbons than in fatty compounds. The kinematic viscosity of unsaturated fatty compounds strongly depends on the nature and number of double bonds with double bond position affecting viscosity less. Terminal double bonds in aliphatic hydrocarbons have smaller viscosity-reducing effect. Branching in the alcohol moiety doesn't significantly affect viscosity compared to straight-chain analogues. At the opposite, free fatty acids or complexed with hydroxyl-groups possess significantly higher viscosity ([63],[64]).

Acid value or neutralization number is a measure of free fatty acids contained in a fresh fuel sample or issued from degradation in aged samples. In the current operation, the acid value was 0.33 mg KOH/g which is within the ASTM D 6751-09 and UNE-EN 14214 standards [70]. This means lower corrosion and minor engine deposits, particularly in the fuel injectors [71].

The iodine value is the measure of unsaturation degree in oil. Iodine value is a useful parameter in studying oxidative rancidity and chemical stability properties of different oils and biodiesel fuels. Higher quantity of double bonds in the sample has greater potential to polymerize and hence lesser stability [72]. The iodine value in the current study was lower than all other researches' (145 against 151 – 166.2 g I_2 /g) while particularly high and exceeds the allowable limit specified in PN-EN 14214 (<120 g I_2/g). This should be correlated with the presence of great amount of unsaturated fatty acids in Camelina oil.

Table 7. Properties of Camelina biodiesel vs standards of ASTM D6751 and EN 14214, and published values in literature

Properties	Literature	Test Method	Present FAME	Present Test Method	ASTM D 6751-09	UNE-EN 14214
Density gm /ml at 15 °C	0.91 [11], 0.888 [65], 0.887 [66],	EN ISO 12185	0.886±0.2	EN ISO 12185	-	0.860-0.900
Specific gravity	0.87-0.88 [67]		0.881			
API gravity			29.11			
Kinematic viscosity cSt, at 40 °C	4.3 [65], 4.15 [68], 2.9 to 3.15 [67], 3.9 [66]	ASTM D 445	3.58±0.03	ASTM D 445	1.9-6	3.5-5
Acid value mg KOH /g	0.76 [11], 0.31 [68], 0.43 [69], 0.25 [66], 0.15 [65]	EN 14104	0.33±0.1	EN 14104	≤ 0.50	≤ 0.50
Iodine value g I2 /g	152 [65], 151 [68], 156 [69], 166.2 [66]	EN 14111	145±0.03	EN 14111	-	≤ 120
Cetane number	49.26 to 51.17 [67], 49.7 [66]	EN 15195	53.6±0.2	ASTM E1655	≥ 47	≥ 51
Carbon residue Wt.%	0.019 (100% sample) [65], 0.003 [66]	EN ISO 3679	0.052±0.01	D 189	≤ 0.05 (100% sample)	≤ 0.3 (on 10% distillation residue)
Cold filter plugging point °C	-5 [69],	EN 116	-3.2±0.05	EN 116	-	According to climate zone
Pour point °C	-4 [68], -11 to -8 [67]	ASTM D97	-4.6±0.1	ASTM D97	-15 to 10	
Cloud point °C	0 [65], 3 [68], 7 [69],-1.6 [66]	ASTM D 2500	0±0.02	ASTM D 2500	According to climate zone	
Freezing point °C		ASTM D2386	-8.3			
Flash point °C	152 [65], 132 to 140 [67], 152 [66]	ASTM D 7501	146.3	ASTM D 98	≥ 93	≥ 101

Cetane number (CN) of DIC/ISTE biodiesel issued from Camelina seeds was 53.6±0.2 which is higher than literature's (49.26 to 51.17). This parameter is important for evaluating the quality of biodiesel because, it is conceptually similar to the octane number for gasoline, and consequently, it is the prime indicator of biodiesel ignition performance inside combustion cylinder [73]. Indeed, it reveals the fuel's ignition delay, defined as a time period between injection of the fuel into the cylinder and the first identifiable pressure increase during the fuel combustion. Thus, the cetane number basically represents how quickly the fuel starts ignition during combustion in a diesel engine cylinder. The higher the biodiesel cetane number, the quicker the ignition [50].

The flash point (FP) of DIC/ISTE Camelina biodiesel (146.3 ˚C) was much higher than of 93˚C as defined in the standard (ASTM D 6751-09), and 101 °C (UNE-EN 14214) implying that Camelina biodiesel is safer to be handled during the process of transport and storage.

Compared to standards and conventional transesterified Camelina oil, DIC/ISTE Camelina seed biodiesel presented interesting values of Pour point, Cloud point, and Freezing point at -4.6±0.1, 0±0.02, and -8.3°C, respectively. Indeed, the cold flow properties of biodiesel are extremely critical in assessing the quality of biodiesel. The poor cold flow ability is a common concern in the biodiesel industry as all diesel fuels should be susceptible to start-up and be operational at low-temperature under cold weather conditions. This is mainly caused by the formation of crystals and solid precipitates at low temperatures, which could clog the vehicle fuel lines and filters [74]. It has also been documented that contaminants may originate from parent vegetable oils or be introduced during the transesterification, purification and even storage processes of biodiesel may affect cold flow properties [75]. Cloud point (CP) is defined as the temperature at which biodiesel starts forming cloudy suspensions and visible crystals. Generally, a relatively high CP value indicates a poor cold flowability. CP has been related to biodiesel composition in some published works [76] including degree of unsaturation such as CP decreases with increasing unsaturation degree. Although Camelina biodiesel inherently contains a high percentage of unsaturated fatty acid esters, CP value of our DIC/ISTE Camelina biodiesel was determined to be relatively low (-1, 0°C) which is lower than some reported CPs ([77], [78], [79]). This indicates that the DIC/ISTE biodiesel of Camelina should have good cold flowability. Nevertheless, although the value of CP well represents biodiesel cold flow properties, other "in-vivo" measurements within conventional engines thus adequately assessing biodiesel's low temperature operability or performance in both vehicle tank and fuel system [75].

IV. CONCLUSION

The general conclusions may be withdrawn from the current study are: first, *In-situ* transesterification (ISTE) process has been imagined as an intensification way of producing biodiesel. This is a much more convenient operation performed in only one transesterification stage excluding steps of the extraction and purification of oil bearing material. Second, response Surface methodology is a valuable tool to reduce the number of experiments, thus minimizing cost and efforts needed to accomplished chemical and engineering processes as well as optimization and modeling the processes for laboratory and industrial scale operations. An optimum biodiesel yield (0.4019 g FAME/g ddb) from Camelina seeds can be obtained by ISTE of the seeds at 60^oC for 2 hours using Methanol: Toluene (90:10) as solvent, and H_2SO_4 as catalyst, with 10% (v/wt.) solvent /catalyst ratio and 50 ml solvent/g Camelina seeds respectively. Third, the combination of ISTE with the innovative technology of Instant controlled Pressure Drop Process (DIC) resulted in intensifying the ISTE process leading to significant increase biodiesel production yield. The DIC-texturing assisted ISTE (DIC/ISTE) allowed reaching up to 191% higher biodiesel yields than ISTE without using DIC. Fourth, the DIC/ISTE biodiesel meet most of the quality specifications in the EN 14214 and ASTM D6751 standards.

ACKNOWLEDGMENT

Authors wish to acknowledge ABCAR-DIC Process (La Rochelle France) and Sanctum Méditerranée for their technical contribution for developing and maintaining this specific work through providing DIC equipment and Camelina, respectively.

REFERENCES

[1]. M. Avram, M. Stroescu, A. Stoica-guzun, and O. Floarea, "Optimization of the oil extraction from Camelina (Camelina sativa) seeds using response surface methodology," Rev. Chim., vol. 66, pp. 417–421, 2015.

[2]. A. Sampath, "Chemical characterization of camelina seed oil," Master Thesis, The State University of New Jersey, Graduate School-New Brunswick, 2009.

[3]. H. Abramovič, B. Butinar, and V. Nikolič, "Changes occurring in phenolic content, tocopherol composition and oxidative stability of Camelina sativa oil during storage," Food Chem., vol. 104, no. 3, pp. 903–909, 2007.

[4]. D. H. Putnam, J. T. Budin, L. A. Field, and W. M. Breene, "Camelina: A promising low-input oilseed," New Crops, John Wiley & Sons, New York, NY. pp. 314–322, 1993.

[5]. E. A. Waraich et al., "Camelina sativa, a climate proof crop, has high nutritive value and multiple-uses: A review," Aust. J. Crop Sci., vol. 7, no. 10, pp. 1551–1559, 2013.

[6]. K. A. Obour, Y. H. Sintim, E. Obeng, and D. V. Jeliazkov, "Oilseed Camelina (Camelina sativa L Crantz): Production Systems, Prospects and Challenges in the USA Great Plains," Adv. Plants Agric. Res., vol. 2, no. 2, pp. 1–10, 2015.

[7]. P. D. Patil, V. G. Gude, and S. Deng, "Biodiesel production from jatropha curcas, waste cooking, and camelina sativa oils," Ind. Eng. Chem. Res., vol. 48, no. 24, pp. 10850–10856, 2009.

[8]. S. Bansal and T. P. Durrett, "Camelina sativa: An ideal platform for the metabolic engineering and field production of industrial lipids," Biochimie, vol. 120, pp. 9–16, 2016.

[9]. O. U. Dairo, T. M. A. Olayanju, E. S. A. Ajisegiri, O. J. Alamu, and A. A.E, "Optimization of in-situ Biodiesel Production from Raw Castor Oil-Bean Seed," Energy Technol. Policy, vol. 3, no. 13, pp. 14–20, 2013.

[10]. B. A. Abdulkadir, W. Danbature, F. Y. Yirankinyuki, B. Magaji, and M. M. Muzakkir, "In Situ Transesterification of Rubber Seeds (Hevea brasiliensis)," Greener J. Phys. Sci., vol. Vol. 4 (3), pp. 38–44, 2014.

[11]. D. Y. C. Leung, X. Wu, and M. K. H. Leung, "A review on biodiesel production using catalyzed transesterification," Appl. Energy, vol. 87, no. 4, pp. 1083–1095, 2010.

[12]. J. G. Crowley and A. Fröhlich, Factors Affecting the Composition and Use of Camelina. Dublin: Citeseer, 1998.

[13]. H. Abramovič and V. Abram, "Physico-chemical properties, composition and oxidative stability of camelina sativa oil," Food Technol. Biotechnol., vol. 43, no. 1, pp. 63–70, 2005.

[14]. M. A. Berhow et al., "Optimized analysis and quantification of glucosinolates from Camelina sativa seeds by reverse-phase liquid chromatography," Ind. Crops Prod., vol. 43, no. 1, pp. 119–125, 2013.

[15]. T. Foulke, M. Geiger, and B. Hess, "Is Biodiesel from Camelina right for you ?" University of Wyoming, College of Agriculture and Natural Resources, Laramie, Wyoming., USA, pp. 1–19, 2013.

[16]. A. C. Ahmia, F. Danane, R. Bessah, and I. Boumesbah, “Raw material for biodiesel production. Valorization of used edible oil,” Rev. des Energies Renouvelables, vol. 17, no. 2, pp. 335–343, 2014.
[17]. M. Etemadzadeh, “Biodiesel Production From Microalgae,” Master Thesis. Politecnico di Milano University, Industrial Engineering Faculty, 2014.
[18]. K. Dutta, A. Daverey, and J. G. Lin, “Evolution retrospective for alternative fuels: First to fourth generation,” Renew. Energy, vol. 69, pp. 114–122, 2014.
[19]. A. L. Ahmad, N. H. M. Yasin, C. J. C. Derek, and J. K. Lim, “Microalgae as a sustainable energy source for biodiesel production: A review,” Renew. Sustain. Energy Rev., vol. 15, no. 1, pp. 584–593, 2011.
[20]. R. A. Lee and J.-M. Lavoie, “From first- to third-generation biofuels: challenges of producing a commodity from a biomass of increasing complexity,” Anim. Front., vol. 3, no. 2, pp. 6–11, 2013.
[21]. Y. Wang, “Microalgae as the Third Generation Biofuel: Production , Usage , Challenges and Prospects.,” Master’s Thesis, Department of Earth Sciences, Uppsala University, 2013.
[22]. Y. Dhaman and P. Roy, “Challenges and Generations of Biofuels: Will Algae Fuel the World?,” Ferment. Technol., vol. 2, no. 1, pp. 1–6, 2013.
[23]. J. Lu, C. Sheahan, and P. Fu, “Metabolic engineering of algae for fourth generation biofuels production,” Energy Environ. Sci., vol. 4, no. 7, pp. 2451–2466, 2011.
[24]. D. R. Shonnard, L. Williams, and T. N. Kalnes, “Camelina-Derived Jet Fuel and Diesel: Sustainable Advanced Biofuels,” Environ. Prog. Sustain. Energy, vol. 29, no. 3, p. 382, 2010.
[25]. M. Sam Mannan, Y. Wang, C. Zhang, and H. H. West, “Application of inherently safer design principles in biodiesel production process,” Inst. Chem. Eng. Symp. Ser., no. 151, pp. 982–989, 2006.
[26]. J. Jones and C. L. Peterson, “Using unmodified vegetable oil as a diesel fuel extender,” Univ. Idaho, USA, 2002.
[27]. I. Thomas, N. A. Porter, and P. and Lappas, “Assessment of the Viability of Vegetable Oil Based Fuels,” in Sustainable Automotive Technologies 2013, Springer, 2014, pp. 15–25.
[28]. S. Kumar, A. K. Gupta, and S. N. Naik, “Conversion of non-edible oil into biodiesel,” J. Sci. Ind. Res. (India)., vol. 62, no. 1, pp. 124–132, 2003.
[29]. H. Fukuda, A. Kondo, and H. Noda, “Biodiesel fuel production by transesterification of oils,” J. Biosci. Bioeng., vol. 92, no. 5, pp. 405–416, 2001.
[30]. U. Schuchardt, R. Sercheli, and R. M. Vargas, “Transesterification of vegetable oils: A review,” J. Braz. Chem. Soc., vol. 9, pp. 199–210, 1998.
[31]. G. Knothe and L. F. Razon, “Biodiesel fuels,” Prog. Energy Combust. Sci., vol. 58, pp. 36–59, 2017.
[32]. N. C. Om Tapanes, D. A. Gomes Aranda, J. W. de Mesquita Carneiro, and O. A. Ceva Antunes, “Transesterification of Jatropha curcas oil glycerides: Theoretical and experimental studies of biodiesel reaction,” Fuel, vol. 87, no. 10–11, pp. 2286–2295, 2008.
[33]. H. M. Mahmudul, F. Y. Hagos, R. Mamat, A. A. Adam, W. F. W. Ishak, and R. Alenezi, “Production, characterization and performance of biodiesel as an alternative fuel in diesel engines – A review,” Renew. Sustain. Energy Rev., vol. 72, no. November 2016, pp. 497–509, 2017.
[34]. O. O. Babajide, “Optimisation of Biodiesel Production via Different Catalytic and Process Systems,” Phd thesis - Department of Chemistry University of the Western Cape, 2011.
[35]. A. Rajalingam, S. P. Jani, A. S. Kumar, and M. A. Khan, “Review Article Production methods of biodiesel,” vol. 8, no. 3, pp. 170–173, 2016.
[36]. L. Wu, T. Wei, Z. Lin, Y. Zou, Z. Tong, and J. Sun, “Bentonite-enhanced biodiesel production by NaOH-catalyzed transesterification: Process optimization and kinetics and thermodynamic analysis,” Fuel, vol. 182, pp. 920–927, 2016.
[37]. G. D. Yadav and S. B. Dhoot, “Immobilized lipase-catalysed synthesis of cinnamyl laurate in non-aqueous media,” J. Mol. Catal. B Enzym., vol. 57, no. 1–4, pp. 34–39, 2009.
[38]. A. Kundu, A. Mukherjee, G. Halder, and D. Datta, “A Kinetic Study on Acid Catalyzed Esterification of Free Fatty Acids in Ricinus Communis Oil for the Production of Biodiesel,” Int. J. Res. Eng. Technol., vol. 5, no. Special Issue: 01, pp. 31–44, 2016.
[39]. J. Yang, T. Astatkie, and Q. S. He, “A comparative study on the effect of unsaturation degree of camelina and canola oils on the optimization of bio-diesel production,” Energy Reports, vol. 2, pp. 211–217, 2016.
[40]. J. Park, B. Kim, and J. W. Lee, “In-situ transesterification of wet spent coffee grounds for sustainable biodiesel production,” Bioresour. Technol., vol. 221, pp. 55–60, 2016.
[41]. M. Barekati-Goudarzi, D. Boldor, and D. B. Nde, “In-situ transesterification of seeds of invasive Chinese tallow trees (Triadica sebifera L.) in a microwave batch system (GREEN3) using hexane as co-solvent: Biodiesel production and process optimization,” Bioresour. Technol., vol. 201, pp. 97–104, 2016.
[42]. G. Kildiran, S. Ö. Yücel, and S. Türkay, “In-situ alcoholysis of soybean oil,” J. Am. Oil Chem. Soc., vol. 73, no. 2, pp. 225–228, 1996.
[43]. K. G. Georgogianni, M. G. Kontominas, P. J. Pomonis, D. Avlonitis, and V. Gergis, “Conventional and in situ transesterification of sunflower seed oil for the production of biodiesel,” Fuel Process. Technol., vol. 89, no. 5, pp. 503–509, 2008.
[44]. B. Lebreton et al., “Trophic importance of diatoms in a inertidal Zostera noltii seagrassbed: Evidence from stable isotope and fatty acid analyses,” Estuarine, Coast. Self Sci., vol. 92, no. 1, pp. 140–153, 2011.
[45]. L. Wang and C. L. Weller, “Recent advances in extraction of nutraceuticals from plants,” Trends Food Sci. Technol., vol. 17, no. 6, pp. 300–312, 2006.

[46]. T. Allaf and K. Allaf, Instant Controlled Pressure Drop (D . I . C .) in Food Processing, From Fundamental to Industrial Applications. Springer New York Heidelberg Dordrecht London, 2014.

[47]. B. Berka-Zougali, A. Hassani, C. Besombes, and K. Allaf, “Extraction of essential oils from Algerian myrtle leaves using instant controlled pressure drop technology,” J. Chromatogr. A, vol. 1217, no. 40, pp. 6134–6142, 2010.

[48]. K. Ibtisam and K. Allaf, “Optimization of Instant Controlled Pressure Drop Dic-Assisted-Solvent Extraction of Total Phenols of Green Coffee Beans,” J. Food Stud. Publ. by Macrothink Inst., vol. 2, no. 1, pp. 42–61, 2013.

[49]. Z. Taghizade, “Determination of biodiesel quality parameters for optimization of production process conditions,” Master thesis, Chemical Engineering, Polytechnic Institute of Bragança, Portugal, 2016.

[50]. J. Bacha et al., “Diesel Fuels Technical Review,” Chevron Glob. Mark., pp. 1–116, 2007.

[51]. R. O. Dunn, “Specific Gravity and API Gravity of Biodiesel and Ultra-Low-Sulfur Diesel (ULSD) Blends,” Trans. ASABE, vol. 54, no. 2, pp. 571–579, 2011.

[52]. D. S. Viswanath, T. K. Ghosh, D. H. L. Prasad, N. V. K. Dutt, and K. Y. Rani, “Theories of Viscosity,” in Viscosity of Liquids: Theory, Estimation, Experiment, and Data, Netherlands: Springer, 2007, pp. 109–133.

[53]. K. Sivaramakrishnan and P. Ravikumar, “Determination of cetane number of biodiesel and it’s influence on physical properties,” ARPN J. Eng. Appl. Sci., vol. 7, no. 2, pp. 205–211, 2012.

[54]. M. Angelovi, Z. Tkác, and J. Jablonický, “Determination Model for Cetane Number of Biodiesel at Different Fatty Acid Composition : a Review,” Anim. Sci. Biotechnol., vol. 47, no. 1, pp. 365–371, 2014.

[55]. “Zeltex ZX-101 XL, Portable Near-Infrared Octane / Cetane Analyzer, ISO 9001 Certified,” Hagerstown, MD 21740 USA, 1950. .

[56]. A. Barsamian, “Methods to Determine Diesel Cetane Number,” Engineering Consulting & Training Services, USA, pp. 4–5, 2009.

[57]. R. A. K. Nadkarni, Guide to ASTM Test Methods for the Analysis of Petroleum Products and Lubricants, Second Edi. USA: ASTM International, ASTM Manual Series; No. MNL44, 2000.

[58]. ASTM International United States, ASTM D6371: Standard Test Method for Cold Filter Plugging Point of Diesel and Heating Fuels. Designation: D 6371 – 99 An American National Standard, 2010, pp. 1–7.

[59]. S. Tayal, “Detection of Cold Flow Properties of Diesel and Biodiesel Fuel Using Optical Sensor,” Master Thesis, Faculty of the Graduate School University of Missouri- Columbia, 2006.

[60]. “Shatox, Devices for Express Analysis of Oil Products Quality,” Institute of Petroleum Chemistry, Shatox company. Russian Federation, Tomsk, 2010.

[61]. I. Barabás and I.-A. Todorut, Biodiesel Quality, Standards and Properties. Chaina: InTech Published, 2011.

[62]. W. Gis, A. Ĩóátowski, and A. Bocheȅska, “Properties of the Rapeseed Oil Methyl Esters and Comparing Them With the Diesel Oil Properties,” J. KONES Powertrain Transp., vol. 18, no. 4, 2011.

[63]. B. Tesfa, R. Mishra, F. Gu, and N. Powles, “Prediction models for density and viscosity of biodiesel and their effects on fuel supply system in CI engines,” Renew. Energy, vol. 35, no. 12, pp. 2752–2760, 2010.

[64]. G. Knothe and K. R. Steidley, “Kinematic viscosity of biodiesel fuel components and related compounds. Influence of compound structure and comparison to petrodiesel fuel components,” Fuel, vol. 84, no. 9, pp. 1059–1065, 2005.

[65]. C. Ciubota-Rosie, J. R. Ruiz, M. J. Ramos, and A. Perez, “Biodiesel from Camelina sativa: A comprehensive characterisation,” Fuel, vol. 105, pp. 572–577, 2013.

[66]. J. Yang, C. Caldwell, K. Corscadden, Q. S. He, and J. Li, “An evaluation of biodiesel production from Camelina sativa grown in Nova Scotia,” Ind. Crops Prod., vol. 81, pp. 162–168, 2016.

[67]. P. D. Patil and S. Deng, “Transesterification of camelina sativa oil using heterogeneous metal oxide catalysts,” Energy and Fuels, vol. 23, no. 9, pp. 4619–4624, 2009.

[68]. B. R. Moser and S. F. Vaughn, “Evaluation of alkyl esters from Camelina sativa oil as biodiesel and as blend components in ultra low-sulfur diesel fuel,” Bioresour. Technol., vol. 101, no. 2, pp. 646–653, 2010.

[69]. K. Kołodziejczyk and M. Owczuk, “Camelina sativa as biofuel as an alternative feedstock for the production of biofuels used in diesel engines,” Chemic, vol. 65, no. 6, pp. 531–536, 2011.

[70]. B. Adinew, “Biodiesel production from Trichilia emetica seeds using in-situ transesterification,” Bulg. Chem. Commun., vol. 46, no. 2, pp. 334–338, 2014.

[71]. N. Saifuddin, A. Z. Raziah, and H. N. Farah, “Production of Biodiesel from High Acid Value Waste Cooking Oil Using an Optimized Lipase Enzyme/Acid-Catalyzed Hybrid Process,” E-Journal Chem., vol. 6, pp. S485–S495, 2009.

[72]. M. Jesikha, “Fatty Acid Methyl Esters Characteristic and Esterification of Some Vegetable Oils for Production of Biodiesel,” Int. J. Eng. Sci., vol. 1, no. 12, pp. 50–53, 2012.

[73]. European Commission et al., “The impact of Biofuels on transport and the enviroment, and their connection with agricultural development in Europe,” Dir. Intern. Policies Policy Dep. Struct. Cohes. Policies, no. 2, pp. 48–49, 2015.

[74]. G. Dwivedi and M. P. Sharma, “Cold flow behaviour of biodiesel-A review,” Int. J. Renew. Energy Res., vol. 3, no. 4, pp. 827–836, 2013.

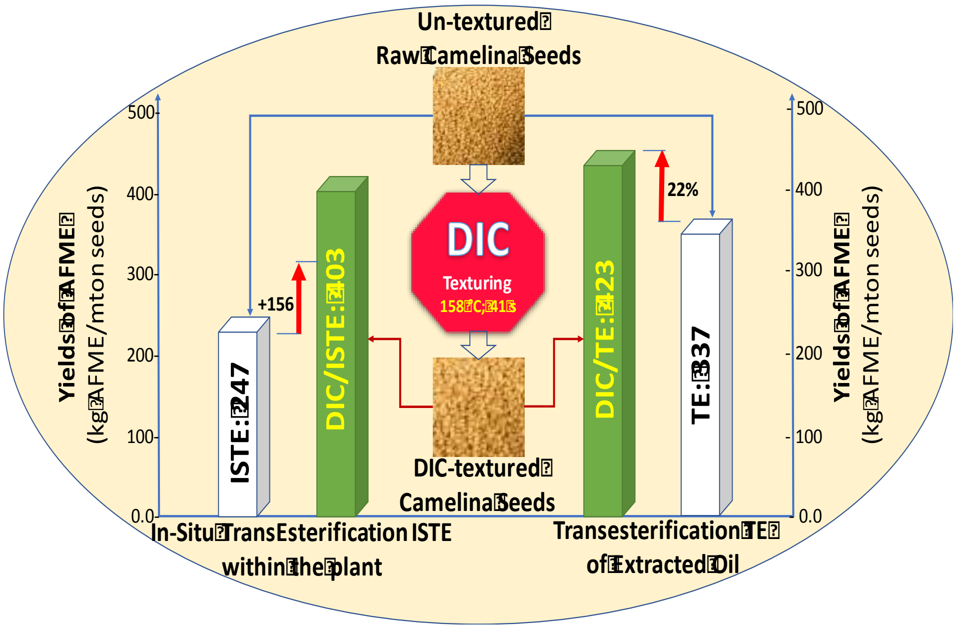
[75]. J. Yang, “Evaluating The Feasibility of Biodiesel Production from Camelina Sativa,” Master Thesis, Dalhousie University Halifax, Nova Scotia, 2016.

[76]. G. Knothe, “‘Designer’ biodiesel: Optimizing fatty ester composition to improve fuel properties,” Energy and Fuels, vol. 22, no. 2, pp. 1358–1364, 2008.

[77]. F. D. Gunstone, Vegetable Oils in Food Technology Composition, Properties and Uses, Second Edi. UK: Wiley - Blackwell, 2011.

[78]. the Japanese Pharmacopoeia, General Tests, Processes, 14th Editi. Japan: The Minister of Health, Labour and Welfare, 2011.

[79]. M. Ishizuka, Ed., Japan's Specifications and Standards for Food Additives, Eighth Edi. Japan: The Ministry of Health, Labour and Welfare, 2009.



Added Graphic

*Fanar Bamerni. "Swell-Texturing assisted in-Situ Transesterification of Camelina Seeds Biodiesel." International Journal Of Engineering Research And Development, vol. 13, no. 09, 2017, pp. 31–44.