Coupling of Texturing/Cooling using Instant Controlled Pressure Drop and Transesterfication for Biodiesel Production from Camelina Sativa

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Abstract

Although Camelina Sativa as oleaginous seeds has obvious advantages as a feed of wonder health benefits, it has been recommended as a highly promising environmental sustainable energy crop and a perfect source of biodiesel. The current work deals with the industrial significance of intensifying the oil and biodiesel yield from Camelina seeds by incorporation of a pretreatment stage for raw material texturing using Instant Controlled Pressure-Drop (DIC) process. The texturing process proved to promote the yield of oils produced by pressing the seeds, and extraction the seeds using solvent, by an amount of 75.9 and 82.9 kg oil /1000 kg seeds, respectively compared to the raw untreated seeds. Consequently, the transformation of the oil using conventional transesterification and in-situ transesterification reactions showed similar trend for increasing the level of biodiesel yield by 86.58 and 155.8 kg/1000kg seeds from conventional transesterification and in-situ transesterification, respectively after texturing the seeds by DIC at 5 bars saturated steam for 40s. The promising findings of the current work could be considered as an innovative approach for cost effective biodiesel production for industrial purposes.

Index terms— biodiesel; in-situ transesterification; instant controlled pressure drop; optimization.

1 Introduction

uring the last century, the consumption of energy has greatly increased due to the change in the life style and the significant growth of population. This increase of energy demand has caused growing emissions of combustion generated pollutants and, simultaneously, the scarcity of conventional fossil fuels **??**1]. This induces increasing extraction costs and makes alternative energy sources more attractive [2]. Biomass is considered as one of the most promising alternative sources of energy that would be economically efficient, socially equitable, and environmentally sound. To meet the rising energy demand and replace reducing low-cost petroleum reserves, biodiesel is in the forefront of alternative technologies [2].

Despite the large fall in price of fossil fuels, there have been substantial increases in biodiesel production in recent years, and this trend is expected to continue [3]. For example, U.S. Biodiesel production during December 2016 was 1 million gallons higher than production in November 2016, it reached 143 million gallons in December 2016. To compare between the three years ago, biodiesel productions in 2014, 2015 and 2016 were 1271, 1268 and 1566 million gallons respectively [4].

Biodiesel can be produced from a great variety of feed stocks. These feed stocks include most common vegetable oils, animal fats and waste oils. The choice of feedstock depends largely on geography. The routes of biodiesel production is dependable on the origin and quality of the feedstock [5].

The archaeological excavations in Europe have revealed the existence of Camelina sativa as far back as 1500 B.C., however, it is a new crop for the western United States, where cultivation began in the 1980s [6,7].

Biodiesel production from Camelina seeds can be classified as a new-generation or a more relevant type of second generation of biodiesel. Camelina's feed potential and its competition with feed grains is limited because Camelina is high in euricic acid and glucosinolates, these two main anti-nutritional factors limits the amount of Camelina meal that can be fed.

Hence, Camelina has more potential for production with less competition with other feed and food crops. Also, land used to grow Camelina, even fallow land, may positively impact that land's productivity for later food production [7].

Camelina possesses important agronomic traits that recommend it as an ideal production platform for biofuels and industrial feed stocks [8]. Camelina is promising sustainable alternative energy crops because it possesses a short-season crop and can be grown as a crop twice during the year under different climatic and soil conditions with the exception of heavy clay and organic soils [9][10][11]. Interest in Camelina sativa has been renewed due to the fact that the crop does not require high inputs of nutrients and pesticides. It grows well in semiarid regions, and in the soil with low fertility researched as a fall-seeded cover crop within soybean and sunflower, the double crop yield returns were higher compared to the mono cropped counterparts [8].

The main product of Camelina sativa is the oil. The oil content of Camelina seeds ranged from 29.9 to 38.3% [12,14], however values as high as 48% have been reported [15]. Table 1 listed the oil yield from several oil bearing seeds. Camelina biodiesel system starts with planting, followed by harvesting and crushing the seeds. This results in two major products; Camelina meal and oil. The meal is fed to livestock, and the oil is processed into biodiesel [7].

It is worthy to note that fuel obtained from renewable and eco-friendly sources has low contain of sulfur and aromatics, and is totally biodegradable [17]. The optimized biodiesel from Camelina met the related ASTM D6571 and EN 14214 biodiesel standards. They are comparable to those of the regular petroleum diesel fuels and can be used for diesel engines as qualified fuel [9,18].

Generally, biodiesel is made through transesterification of triglyceride in the natural oils with alcohol. The transesterification of vegetable oils or fats can be done in a simple process. There are different ways of biodiesel production, normally depending on the kinds of raw materials: refined, crude or frying oils. Transesterification is performed using different types of catalyst; basic, acids, ion exchange resins, lipases and supercritical fluids [19]. Thus, the process conditions must be carefully controlled to achieve optimal yield at the optimal temperature and reaction time [20].

Ideally, transesterification is potentially a less expensive way of transforming the large, branched molecular structure of bio-oils into smaller, straightchain molecules of the type required in regular diesel combustion engines [2]. Oil usually used as raw material for transesterification process is habitually produced by solvent extraction or by mechanical pressing usually followed by solvent extraction to extract any remaining oil [21][22][23]. The efficiency of a mechanical-expression process rarely exceeds 80% [24]. Solvent extraction is more efficient, especially for oilseeds with lower oil contents such as soybeans. Seeds with higher oil contents are pre-pressed before extraction by solvent [25]. The majority of the output of the biodiesel production process is meal (in terms of weight and volume). Meal should be consumed as close as possible to the point of production to avoid transportation costs [7].

For improving technological aptitude of raw material in terms of extraction, one may modify the premier structure of the seeds. In our laboratory, modifying the raw materials is carried out by applying texturing and structural expansion using the Instant Controlled Pressure Drop (DIC) technology [26]. In terms of vegetal oil production, it had been proved that DIC-textured oleaginous plants could get about 10% higher oil yields. Also, the higher the expansion ratio, the better the diffusivity constant predicted. Oil transesterification is by far the most common method to produce biodiesel [5,27] Although the reaction is not so energyconsuming and the conversion efficiencies are good [28], the operation is known as expensive because of the numerous steps between harvesting of oilseeds and final production of biodiesel after intermediate steps of oil extraction and refining [29]. The price of raw material can account for 65 to 75% of the cost of production of biodiesel. Increasing the yield has a great interest in improving the process and the economy and profitability of biodiesel production [28]. However, it has been reported that biodiesel yields were possibly reduced during conventional transesterification because of the existence of gums and extraneous material in the crude vegetable oil, hence, refining and purification of extracted oil become crucial stages before transesterification [30].

There is another way to produce biodiesel; that is 'in-situ transesterification'. transesterification produced lower yield of biodiesel than the conventional transesterification method in different percentages according to the raw material. However, ISTE is interesting because it greatly simplifies the process and makes it more suitable for distributed production [29], [31][32][33].

The current work aimed at defining various intensification routs of producing biodiesel based on Year 2017 G using (DIC). Fatty Acid Methyl Esters (FAMEs) were manufactured by transesterification of oil produced by pressing and solvent extraction of the raw seeds, transesterification of pressed oil and oil extracted by solvent from DIC textured seeds, and in-situ transesterification of raw and the DIC-textured seeds.

2 II.

3 Material And Methods

4 a) Raw materials

Dried Camelina Sativa seeds were provided by Sanctum Mediterranean harvested from France fields (Les Combes, 30250 Junas). Methanol 99.9%, toluene 98%, anhydrous Sodium phosphate, n-Hexane (HPLC grade, 99.9) were purchased from Merck. Sulfuric acid 99% from Sigma-Aldrich.

5 b) Measurement of moisture content

Camelina Sativa seeds were sun dried. The moisture content of the samples was measured by IR moisture analyzer (MB 45, Infrared halogen Moisture Analyzer. OHAUS -Switzerland), and by oven method (105 °C for 24 hours). The initial water content of the dried Camelina seeds has been determined to be 0.0443 g H 2 O/g db. The safe moisture content for storage of oilseeds decreases with increase in oil content of the oilseed [34]. Camelina seeds moisture should be not more than 8% for best storage. Maximum oil contents also based on moisture content [34][35][36][37][38]. c) Instant controlled pressure drop technology Instant controlled pressure drop (DIC) technology was initially developed by ALLAF and collaborators, (Since 1988) at the University of La Rochelle. It applies an instant pressure drop to modify the texture of the material and intensify functional behavior [39]. Instant controlled pressure drop DIC is a High-Temperature, High Steam Pressure (ranged between 0.1 and 0.7 MPa)/Short-Time (usually between 5 and 60 s) treatment followed by an abrupt pressure drop towards a vacuum (about 5 kPa) [40,41]. A schematic diagram of DIC set-up is shown in Figure 1.1.1.1.1. Figure 1: Schematic diagram of DIC unit:

The treatment vessel where we place and treat the samples. The vacuum system, which consists mainly of a vacuum tank with a volume 130 times greater than the processing reactor, and a suitable vacuum pump. The initial vacuum level was preserved at 50 kPa in all the experiments. A pneumatic valve assures the connection/separation between the vacuum tank and the processing vessel. It can be opened in less than 0.2 seconds, this ensures the abrupt instant pressure drop (?P/?t>0.5 MPa/s) within the reactor.

DIC is distinguished by a pressure-drop rate higher than 0.5 MPa/s implying an expansion and a rapid cooling of the product [40]. The pressure drop induces a whole swelling of the product and a possible controlled destruction of cell walls; it also may release volatile compounds [42].

The high temperature of the process is generated by subjecting the raw material for a short time-frame to saturated steam high-pressure [43], This generates an auto-vaporization of volatile molecules, implying instant cooling and expansion of the sample. DIC allows the structure to be more expanded [43] and usually preserves the product color, flavor and vitamins; it also decontaminates and gets rid of insects. Therefore DIC treated products have normally a minimum shelf life of two years [40]. In addition, texturing by DIC results in increasing operation performances through lower energy consumption and processing time [40]. It allows undertaking much more effective solvent extraction Year 2017 G through higher effective starting accessibility and diffusivity, which greatly reduces processing time [43]. i. Mechanical pressing Pressing is the most common method in the world to separate oil from oils oilseeds on small to medium level [44].

To obtain the Camelina Sativa oil by pressing, a mechanical screw press (Täby Örebro, Sweden) was used. The restricted size of the press cake outlet can vary by placing different sized nozzles (7 mm) to get the best results in terms of extracted oil. After pressing the pressed cake was analyzed for oil content.

6 ii. Solvent Extraction for Camelina cake and Seeds

Oil extraction from Camelina seeds as well as cakes produced by pressing was achieved by reflux apparatus using n-hexane. Processing conditions were estimated from the literature [11] with some modifications: seeds/solvent ratio 1:26 extraction temperature 60 ± 2 ?C. Extraction time and agitation speed were selected to be 2 hours, and 600 rpm, Percentages of oil extracted from the seeds and the cake are listed in table 2.

7 e) Experimental design

Statgraphics for Windows software (5.1 version), SIGMA PLUS Neuilly/Seine (France) for designing experiments and statistically treating the responses was employed. 2-parameter 5-level central composite designs was adopted to study the effect of DIC operating parameters on biodiesel (FAMs) yield. 13 DIC-textured samples were transesterified using the optimized conditions estimated from response surface analysis (RSA) for data based on ISTE of the raw Camelina seeds.

8 f) Conventional and in Situ Transesterification

Transesterification (TE) of Camelina oil, and In-Situ Transesterification (ISTE) of Camelina seeds were carried out based on experimental design using Response Surface Methodology. The main response (dependent variable) was the yield of biodiesel. Sulfuric acid was used as the catalyst in the two processes. The optimum conditions used in the conventional transesterification (TE) process were: reaction time (36.24) min., solvent/oil volume ratio (17:1), and catalyst to solvent volume ratio (2:100). The transesterification processes were carried out at constant temperature (60 ?C) and agitation (600 rpm).

The optimum conditions adopted for In-Situ Transesterification (ISTE) process were: solvent/seeds ratio (volume to weight 50:1), and catalyst to solvent volume ratio 10:100. The in situ transesterification reactions were carried out at constant temperature (60?C) and agitation (600 rpm). The In-situ transesterification process was performed under the following conditions: solvent type is Methanol: Toluene (90:10) v/v, catalyst type H 2 SO 4 mixed with methanol (4%, w/v), and reaction time 2 h. Each experiment started by preparing separately 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. The mixture was shaken 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 was soaked in 10 ml of the reactive mixture for 10 min., and then charged to the round bottom flask when methanol/catalytic solution had reached the desired temperature. After the reaction completion, the round bottom flask was cooled to room temperature, and then the cooled mixture was filtered.

The solution was transferred to a separation funnel to allow separation of glycerol from the ester phase. After separation, the crude biodiesel was washed 4-5 times with warm distilled water followed by 0.1 % sodium hydroxide to remove trace amounts of catalyst in the methyl ester. The washing was repeated until a clear water layer of neutral pH was obtained. The solvent was evaporated using rotary evaporator (55-60°C). The obtained FAMEs (i.e. biodiesel) were dried over anhydrous sodium phosphate then filtrated to remove the sodium phosphate. Finally, the FAMEs was 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:

(1)

9 G

The DIC operating variables for the in situ transesterification reaction were treatment temperature, T and processing time, t. The coded and natural levels of DIC independent parameters are listed in Table ??.

10 Table 2:

Real and coded values of DIC independent parameters (processing temperature, T and processing time, t)

According to the experimental design (13 samples) of Camelina seeds were treated by DIC at different processing temperature and time. In general, DIC treatment involves initial heating of the fresh biomass and/or partially dried feedstock usually up to 160 °C using high-pressure (up to 0.8 MPa) saturated steam, in a treatment vessel for a short period of time (some seconds or dozens of seconds). Once the equilibrium at both temperature and water content within product is attained, the second main stage is performed inferring an abrupt pressure-drop with a rate Î?”P/Î?”t higher than 0.5 MPa s -1 , towards a vacuum (usually 5 kPa).

Since the porosity and texturing ratio are usually function of the amount of auto-vaporized water from the textured material, the rheological behavior and the glass transition temperature of the material depend on DIC temperature, and pressure drop rate, hence, the DIC vacuum level and pressure drop were kept constant at (3.5 kPa) and (65 MPa s -1) respectively based on literature data [45] and after some preliminary experiments. A comparative study of ISTE for 13 samples of DIC-textured Camelina seeds was conducted. The DIC independent variables T and t were defined as 115.0-165.0 °C and 15-45 s, respectively as listed in Table ??. It is worthy to mention that the DIC treatment temperature is strictly correlated with the pressure of saturated steam.

11 III.

12 Results And Discussion

Studies at laboratory scale were established to identify and quantify the impact of DIC parameters on yield of oil extracted from Camelina seeds. DIC operating conditions were optimized relevant to maximum oil yield. DIC processing parameters were heating treatment time t (between 15 and 45 s) and the saturated steam temperature T (between 115.0 and 165.0 ?C) which corresponds to pressure P (between 0.17 and 0.7 MPa). The optimized processing parameters (157 °C or 0.58 MPa, and 41 s) were applied to identify the industrial significance of DIC process. Treatment capacity of the industrial scale DIC reactor was established to be about 8 tons/hour. The amount of extracted oil from raw seeds and DIC treated seeds by pressing and solvent extraction as well as the increase in oil yield is shown in Table 3. The results estimated revealed that by applying the optimized DIC texturing pre-treatment (0.58 MPa for 41 s), the oil yields from both solvent extraction and pressing increased by an amount of 75.9 and 82.9 kg oil /1000 kg seeds respectively. However, the pressing followed by solvent extraction (SE) of meal for DICtreated seeds allowed a total increasing of the oil yield of 50.9 kg/1000 kg seeds as shown in Table 3. The experimental results confirmed that the DIC samples produced higher biodiesel yield from both conventional and in-situ transesterification processes compared to raw untreated Camelina samples, however a significant increase was identified for biodiesel produced by in-situ transesterification compared to conventional transesterfication as shown in Table ??.

13 Table 5: Summary of Response Surface Analysis results estimated from Analysis of variance (ANOVA)

The high regression coefficient(R^2) for both TE and ISTE processes reflected that the adopted models have high capability to explain the experimental results accurately.

On the other hand, the significance of the operating parameters for conventional transesterification and in-s itu transesterification reactions could be explained by the Pareto charts shown in Figures 2 and 3 respectively. Based on Pareto charts, is obvious to note that solvent/oil ratio is the most significant parameters affected biodiesel yield followed by reaction time, while catalyst/solvent ratio shows no significant for conventional transesterification as shown in Figure **??**. However, solvent/seeds ratio was also the top most significant for in situ transesterifications followed by catalyst/solvent ratio which show less significant effect (Figure 3).

14 Global

A comparative study for industrial production of biodiesel using 1000 kg of Camelina seeds was performed in the current work. The industrial production of biodiesel from Conventional Transesterification TE of Camelina oil, and In-Situ Transesterification ISTE of untextured raw seeds and DIC-textured seeds resulted in great difference in biodiesel yields as shown in (Table **??**).

The optimized DIC parameters correspond to the optimum experimental results when applied to industrial scale resulted in a clear view for the industrial significance of the DIC process.

Table 6 shows the estimated industrial yields of biodiesel produced by TE and ISTE operations of 1000 kg of un-textured and DIC-textured Camelina seeds. By incorporation of the optimized DIC texturing pre-treatment conditions, yields of biodiesel from both TE and ISTE increased as shown in Table 6. An increase in biodiesel yield of about 86.56 and 155.80 kg/ 1000g is produced from TE and ISTE respectively. Our findings are not in line with that recorded by other researches who reported that less amount of biodiesel is produced by ISTE compared to that produced by TE [46]. The result of the current work confirmed that when ISTE process is coupled with DIC treatment (DIC/ISTE) more biodiesel will be produced. The reason is attributed to structure expansion and texturing of the raw material by DIC which enhance the solvent diffusivity and extractability as well as the kinetics of the transformation process [43,47,48]..

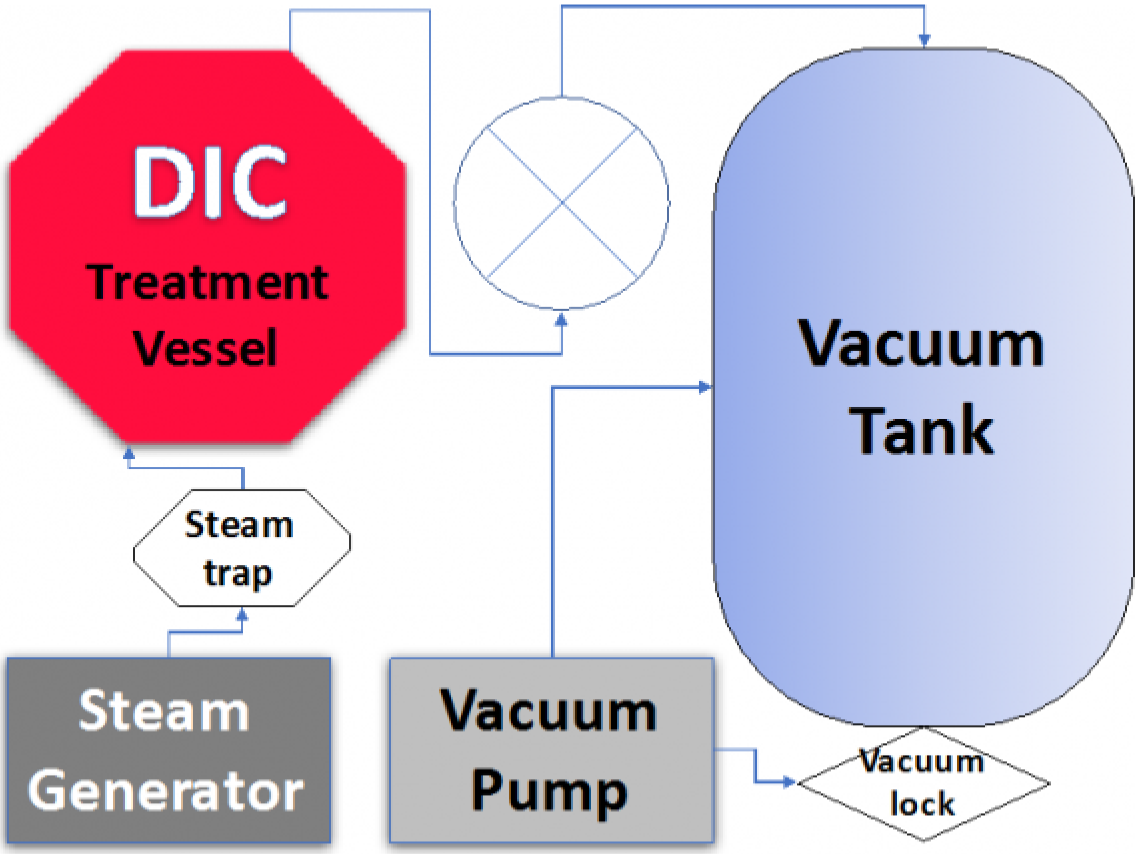
15 IV.

16 Conclusion

The current study is a comparative study for production of biodiesel; Fatty Acid Methyl Esters Year 2017 G routes; conventional transesterification of oil extracted by pressing (PO-TE), conventional transesterification of oil extracted by solvent (SE-TE), transesterification of pressed oil from DIC textured seeds (DIC/PO-TE), transesterification of solvent extracted oil from (DIC/SE-TE), in-situ transesterification (ISTE) of un-textured raw material and in-situ transesterification (ISTE) of DICtextured seeds. The conclusions could be drawn from the current study revealed that coupling DIC texturing with transesterification, of the raw material will resulted in intensification of the transformation processes to promote the FAMEs yield by 86.58 and 155.8 kg/1000kg seeds by conventional transesterification and in-situ transesterification respectively. The findings are of potential importance from industrial point of view in term of cost effective biodiesel production.

1. Nada E.M. Elsolh, "The Manufacture of Biodiesel from the used vegetable oil," Master thesis, Faculty [1] [2]

[2] Coupling of Texturing/Cooling Using Instant Controlled Pressure Drop and Transesterfication for Biodiesel Production from Camelina Sativa



1

Figure 1: Figure 1 d)

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

Figure 2:

Figure 3: G

3

Figure 4: Figure 3 :

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	Camelina	Rapeseed	Soybean	Sunflower
Seed Yield (tons/ha)	0.90-2.24	2.68-3.39	2.14-2.84	1.44-1.70
Oil Content (wt. %)	35-45	40-44	18-22	39-49
Oil Yield (l/ha)	106-907	965-1342	347-562	505-750

Figure 5: Table 1 :

3

	Amount of extracted oil RM (kg)	Amount of extracted oil from DIC-textured seeds (kg)	Increased in yield after DIC textured (kg)
Solvent extraction from seeds	352.5	428.4	+75.9
Seed pressing	218	300.9	+82.90
Solvent extraction (SE) from meals	88	56	-32.00
Total of pressing and SE of meals	306	356.9	+50.90

b) Comparative Industrial Yields of Biodiesel

The actual values of DIC independent parameters and responses (biodiesel; FAMs yield from TE and ISTE) experiments carried out at laboratory are shown in

Figure 6: Table 3 :

44

Figure 7: Table 4 .Table 4 :

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	FAMEs from RM	FAMEs from DIC textured seeds	Increased in yield after DIC texturing
	(kg)	(kg)	(kg)
ISTE	247.3	403.1	155.80
TE	336.64	423.22	86.58

Figure 8: Table 6 :

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