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Full Length Research Paper

How can "Instantaneous Pressure-Drop DIC" Texture Camelina Seeds, Increase Extraction Yields and Preserve Vegetal Oil Quality?

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Texturing by "Instant Controlled Pressure-Drop" (DIC) used as pretreatment technique in both solvent and pressing processes allowed getting higher yields and better kinetics with perfect preservation of quality of Camelina-sativa seed vegetal oil. Response Surface Methodology (RSM) was used and led to optimize DIC processing parameters of temperature (T) and time (t) at 158 °C for 41 s. For both pressing and solvent, yields were 0.3153 and 0.4490 g oil/g db (dry basis), which were 38% and 22%, respectively higher than the yields issued from the crude Camelina seeds. In addition, the composition of vegetal oil issued from DIC-textured Camelina seeds was similar to the crude raw material oil, thus relating a good preservation of the quality.

Keywords. *Camelina sativa*; "Instant controlled pressure drop" (DIC); Vegetal Oil pressing; Vegetal Oil solvent extraction; Process intensification; Response Surface Methodology (RSM).

INTRODUCTION

Camelina (*Camelina sativa* L. Crantz) seeds have a global interest due to their high content of oilseeds, generally between 300 and 460 g/kg (Eynck and Falk, 2013), with high functional properties that allow numerous uses in the food, cosmetic and biofuel sectors (Zhao et al., 2015). The large amounts of unsaturated fats (approximately 90%), which characterize the Camelina vegetal oil allow it to dry so quickly that it can be consumed for manufacturing polymers, varnishes, paints, cosmetics and dermatological products (Obour et al., 2017). Since Camelina seed vegetal oil is rich in α-linolenic acid and tocopherol, it is a real healthy alternative for food applications (Moser, 2010). Moreover, it has been shown that this oil is very suitable for the manufacture of biodiesel and renewable aviation fuel (Ciubota-Rosie et al., 2013), (Mupondwa et al., 2016), (Li and Mupondwa, 2014) . Camelina biofuels may imply a reduction of greenhouse emissions by more than 60% compared to petrol-based fuels (Gesch et al., 2016). By-product oil pressing meal was traditionally used as feed ingredient (Zubr, 2003). The growth features of Camelina make it suitable as a transitional crop in Mediterranean zones, where it can be proposed with other main crops, as alternated or double crop (Royo-Esnal et al., 2017), (Jung et al., 2016), (Berti et al., 2016). Camelina sativa has been shown to grow on marginal lands (Bansal and Durrett, 2016), with no notable need for fertilizers and pesticides (Avram et al., 2015).

Extraction of vegetal oil from Camelina seeds is performed by various processes such as cold pressing, solvent or supercritical CO_2 extraction, etc. (Avram et al., 2015). The efficiency of mechanical press is affected by the initial and issued characteristics of the samples, such as heating/cooking pretreatments, moisture content, pressing temperature, and structure shape of the mechanical press, as axis and sizes of the compression chamber (Martins et al., 2010). It is recommended that the vegetal oil obtained from Camelina sativa seeds by pressing requires filtration and deodorization stages; while the neutralization, degumming, bleaching, etc. are not requisite, and actually can have reverse effects on the quality of the vegetal oil (Zubr, 1997).

Cold pressing associates specific convenience with low cost and small scale. It also has as benefit the possibility to use residue (cake) as animal feed or fertilizer, since it is free of toxic solvents. In brief, it is less injurious and hazardous compared to solvent extraction because of chemical nature (Moses, 2014). Solvent extraction is technically more effective and efficient for vegetal oil extraction from seeds. Its yields can reach up to 98% (Moses, 2014), to be recognized as the most efficient method (George, 1984). Extraction yields and kinetics normally depend on the sizes and area of the solid phase, solvent ratio, temperature, and moisture content (Avram et al., 2014). However, the natural structure is so compact that it greatly reduces the diffusivity of solvent within seeds and the availability of vegetal oil.

In order to improve the technological/structure aptitude in terms of extraction, seeds can be ground, cut, heated, etc. This results in higher yields and better kinetics, but mostly coupled to significant thermal degradations. In our laboratory, numerous studies on the effect of soft structural expansion have been performed. In several cases of extraction, increased porosity results in greater diffusivity (Ben-Amor et al., 2008). Therefore, the well-known swelling operation of "Instant Controlled Pressure Drop DIC" can be used for large range of plant-based materials without implying significant thermal degradation (Bouallegue et al., 2016).

The aim of this study is to investigate the impact of DIC in the case of Camelina S. seeds in terms of extraction yields and chemical and physical properties of extracted oils.

Experimental protocol

Raw material and chemicals

Natural sun-dried Camelina sativa seeds were harvested in August, 2014 obtained from France fields. Ethanol 95% and n-Hexane (HPLC grade, 99.9%) were purchased from Merck.

Treatment and processes

Instant controlled pressure drop technology

Instant controlled pressure drop (DIC) technology has been initially defined, patented, and developed by ALLAF and collaborators, since 1988, at the University of Technology of Compiegne (France) and since 1994, at the University of La Rochelle. The main DIC parameters are the treatment temperature (usually between ca 80 and 170 °C), the high steam pressure (usually between 0.07 and 0.7 MPa) - treatment time (usually between 5 and 60 s) and a quick pressure drop (higher than 5 MPa s^{-1}) towards a vacuum (ca. 5 kPa) (Setyopratomo et al., 2009), (Jung et al., 2011), (Berka-Zougali et al., 2010).

DIC implies both effects of expansion and rapid cooling of the product (Jung et al., 2011). The abrupt decompression causes an autovaporization of volatile molecules, which implies instant cooling and further expansion of the sample structure (Allaf et al., 2014) and usually huge preservation of product color, taste and vitamins. It also results in great effective decontamination and gets rid of insects. Therefore, DIC treated products usually have a shelf life of more than two years (Jung et al., 2011). In addition, thanks to the well-controlled texturing and the possible destruction of cell walls, DIC can imply higher yields, better kinetics and lower energy consumption, which inexorably involves a decrease in the cost (Berka-Zougali et al., 2010), (VAN Cuong, 2010). Moreover, DIC usually induces higher evaporation of volatile compounds (VAN Cuong, 2010). The new functional behavior and technological aptitudes result in well-controlled intensification of solvent extraction through a more active washing step and greater diffusivity using less solvent (Allaf et al., 2014).

The high temperature of the process is produced by exposing the raw material to high pressure of dry saturated steam (Allaf et al., 2014). Figure 1 shows a schematic diagram of the DIC configuration.

The treatment vessel (1) is a double-jacket chamber. The vacuum tank (2) has a volume 130 times bigger than the processing vessel. (2) is connected to a water ring-pump (at temperature lower than 18-20 °C) in order to maintain a vacuum level of 3.5-5 kPa in all the experiments. The pneumatic valve (3) between (1) and (2) can be opened in less than 0.05 s; this ensures an abrupt pressure-drop within the treatment vessel. According to the experimental design, 13 samples from

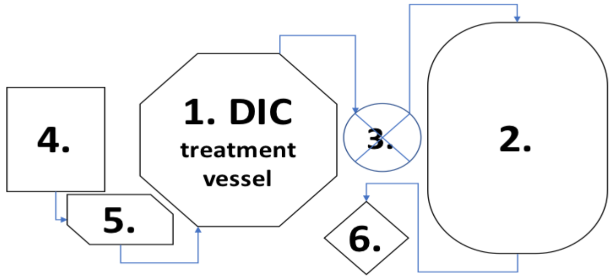

Figure 1. Schematic diagram of the industrial scale DIC unit: 1. DIC Treatment Vessel; 2. Vacuum System Tank; 3. Large diameter instantaneous opening valve; 4. Steam generator; 5. Steam trap; 6. Vacuum lock

Table 1. The factorial design matrix with the actual and coded levels of the independent variables.

	Processing temperature °C	Processing time (s)
Minimum level (-α)	115	15
Point (-1):	122	19
Central point	140	30
Point (+1):	158	41
Maximum level (+α)	165	45

Table 2. The DoE trials using various combinations of the independent variables with water content and yields of oil produced from Camelina seeds and cake, as responses.

	Processing		Water content	Yields (g oil/g db)		
No.	**Temperature °C**	**Time (s)**	**(g H_2O/g db)**	**$Y_{Pressing}$ (g oil/g db)**	**$Y_{Seed-solvent}$**	**$Y_{meal-solvent}$**
control			0.0443	0.2277	0.3681	0.0929
DIC1	140.0	30.0	0.0446	0.2936	0.4296	0.0687
DIC2	165.0	30.0	0.0489	0.2421	0.4379	0.0845
DIC3	140.0	45.0	0.0512	0.2952	0.4430	0.0650
DIC4	140.0	30.0	0.0506	0.2927	0.4277	0.0709
DIC5	158.0	41	0.0480	0.3153	0.4490	0.0585
DIC6	158.0	19.0	0.0473	0.2449	0.4169	0.0784
DIC7	140.0	30.0	0.0534	0.2930	0.4253	0.0697
DIC8	122.0	19.0	0.0467	0.2859	0.3918	0.0778
DIC9	122.0	41	0.0498	0.2679	0.4267	0.0629
DIC10	140.0	30.0	0.0518	0.2818	0.4205	0.0715
DIC11	115.0	30.0	0.0518	0.2911	0.4208	0.0733
DIC12	140.0	15.0	0.0497	0.2787	0.4001	0.0702
DIC13	140.0	30.0	0.0502	0.2635	0.4168	0.0687

Camelina seeds were treated by DIC at different temperature and time.

Design of Experiments (DoE) and Response Surface Methodology (RSM)

In order to reduce the number of experimental trials required to study the effects and optimize the values of the main operating parameters (independent variables of steam temperature T and processing time t), a 2-component rotary experimental design was defined using 13 experiments including 2^2=4 factorial points, 2*2=4 star-points and 5 replicates for the central point. The experiments were performed randomly. This should minimize the effects of unexpected variability on the observed responses due to unusual responses. Table 1 lists the independent variables and actual and coded levels, while Table 2 inclines the trials performed in the DIC treatments.

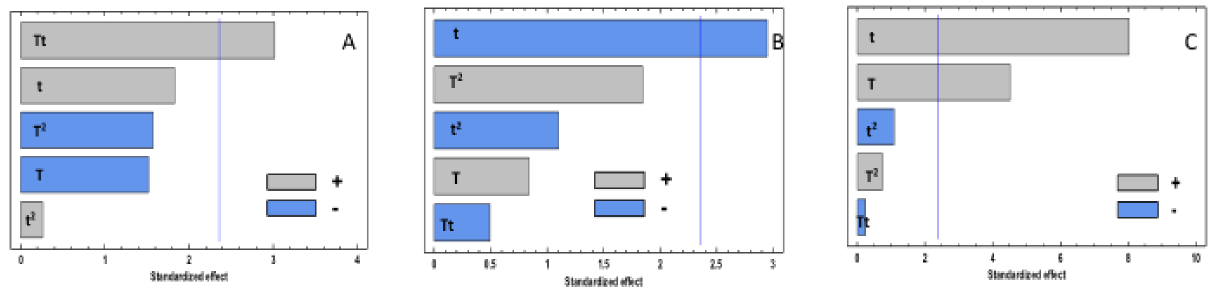

Figure 2. Standardized Pareto Chart for Yield defined by RSM analysis from design of experiments DoE (A) pressing extraction for DIC treatment seeds, (B) solvent extraction for cake from pressing, and (C) solvent extraction for DIC treatment seeds. With temperature (T) and total thermal treatment time (t), as DIC operating parameters.

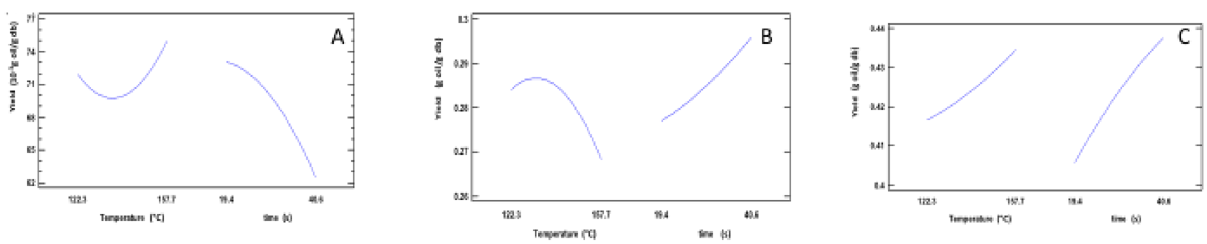

Figure 3. Estimated 1-D Main Effects Plot for Yield defined by RSM analysis from design of experiments DoE (A) pressing extraction for DIC treatment seeds, (B) solvent extraction for cake from pressing, and (C) solvent extraction for DIC treatment seeds. With temperature (T) and total thermal treatment time (t), as DIC operating parameters.

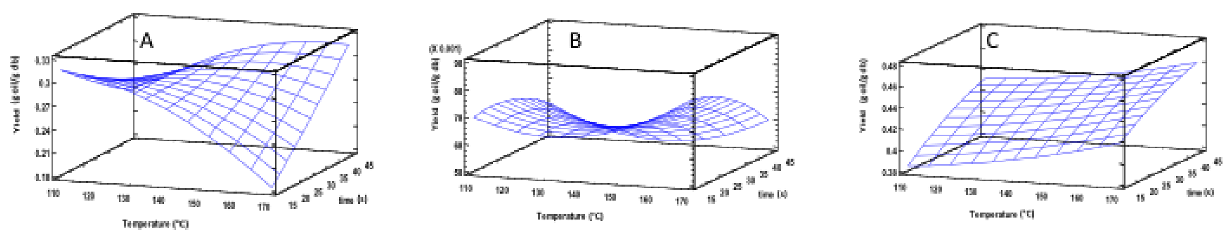

Figure 4. Estimated 3-D Response Surface for Yield defined by RSM analysis from design of experiments DoE (A) pressing extraction for DIC treatment seeds, (B) solvent extraction for cake from pressing, and (C) solvent extraction for DIC treatment seeds. With temperature (T) and total thermal treatment time (t), as DIC operating parameters.

The very relevant, convenient and powerful tool of surface response methodology RSM was used through the analysis program of Statgraphics for Windows software (5.1 version, SIGMA PLUS Neuilly/Seine, France) for designing experiments and statistically treating the responses.

Extraction of vegetal oil from Camelina sativa.

Common methods of vegetable oil extraction include solvent extraction (maceration, etc.) and cold press. Each method possesses its advantages and disadvantages. Camelina vegetal oil is extracted from the seeds by pressing and solvent extraction (Avram et al., 2015).

Mechanical pressing

To obtain the vegetal oil of Camelina sativa by pressing, a screw press type Skeppsta Maskin AB (Oil Tàby Press Type 40 A, Sweden) was used. The restricted size of the press cake outlet can vary by placing different sized nozzles. In the case of Camelina S. seeds, a nozzle of 7 mm allowed pressing to get the best results. The yield of vegetal oil collected by pressing was evaluated through the measurements of the difference in vegetal oil contents between the initial seeds and the press cake. Table 2 shows the percentage of oil produced from seeds by mechanical pressing.

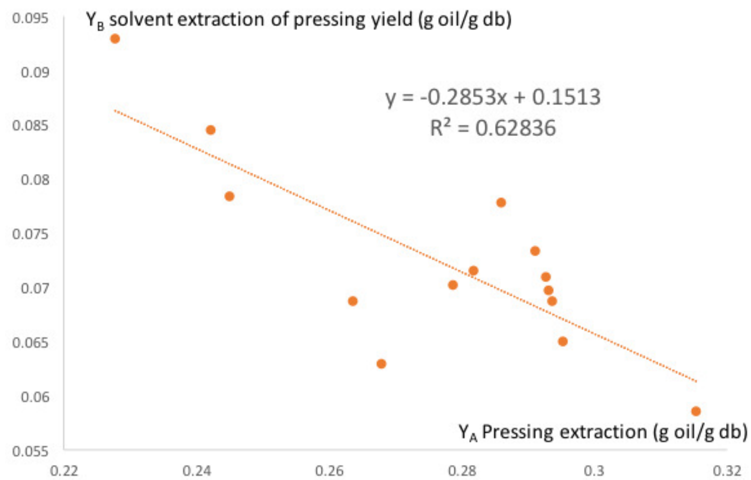

Figure 5. Evolutions of pressing yields and residual oil in the meal.

Solvent Extraction of Camelina Seeds and pressing cake

n-Hexane solvent extraction of vegetal oil from Camelina seeds (14 samples) and pressing cakes (14 samples), was made using reflux apparatus. Reflux reaction conditions were defined based on the literature (Avram et al., 2015) with slight modifications; ratio of seeds/solvent 1:26 (w/v), heating temperature 60±2 °C. We selected extraction time for 2 hours, with 600 rpm agitation speed. Table 2 shows the percentage of vegetal oil extracted from the seeds and the cakes.

Methods of Assessments

Measurement of moisture or water content

Camelina sativa seeds were sun-dried (natural drying). The moisture or water content of the samples was measured by two methods; oven (105°C for 24 h) and IR using moisture analyzer the Infrared halogen technology (OHAUS MB45 Moisture Analyzer, Switzerland). Water content was expressed in g H_2O/g db (dry basis) (Boone and Wengert, 1998):

$$W\ (water\ content: g(H_2O)/g\ db\ (dry-basis)) = \frac{Initial\ weight - dry\ weight}{dry\ weight} \quad (1)$$

The initial water content of the dried Camelina S. seeds has been determined to be 0.0443 g H_2O/g db. The safe water content for storage of oilseeds decreases with increase in vegetal oil content (Bargale, 1997). Camelina seeds water content should be less than 8% wb for best storage. Correctly, maximum oil contents also depend on water content. Thus, the maximum vegetal oil contents in soybean, sunflower and rapeseed are obtained for water at 13%, 12%, and 10.5% wb, respectively (Bargale, 1997), (Grady and Nleya, 2010), (Fleenor, 2012), (Yang et al., 2016).

Assessments and determination of the Physical and chemical Properties

The yield of vegetal oil was determined after pressing and solvent extraction process using expressed in g Oil/g db (Zhao et al., 2014).

$$Y\ (g\ Oil\ /g\ db\ ) = \frac{Oil\ extracted\ weight}{dry\ weight} \quad (2)$$

The density of vegetal oil was determined by pycnometer according to a procedure mention in (Chan et al., 2010).

$$Oil\ density(kg\ m^{-3}) = \frac{Oil\ extracted\ weight}{Oil\ volume} \quad (3)$$

Kinematic viscosity was determined using a viscometer (Viscometer, Cannon Fenske Routine, Eurostar Scientific LTD, PSL ASTM-IP 100, UK). The detailed method has been reported in (Beerens, 2017). refractive index has been measured according to ASTM D1218 (Oluwoye, 2013). The refractometer using is (Bellingham, Stanley Ltd. Abbe 60/95 refractometer). The peroxide value was determined according to a reported procedure (ASTM international, 2005):

$$Peroxide\ Value = \frac{(Sample\ titration - Blank\ titration)V_{thiosulfate*1000}}{Sample\ weight} \quad (4)$$

The acid value AV was determined according to a reported procedure (Hou J.–F. Shaw, 2008). The value was calculated using the following formula:

$$AV = \frac{Sample\ titration\ value\ \times \mathrm{KOH\ normality} \times 56.1}{Sample\ Weight} \quad (5)$$

Table 3. Empirical models of oil yields Y_A, Y_B, and Y_C expressed in (g oil/g db), versus DIC operating parameters; saturated steam temperature (T) and total thermal processing time (t): (A) pressing extraction for DIC-treated seeds, (B) solvent extraction for pressing cake, and (C) solvent extraction for DIC treated seeds.

$Y_A = 0.2796 + 0.00384T - 0.0164t - 0.00003T^2 + 0.0002Tt + 0.00001t^2$	R^2=71.4%;	(14)
$Y_b = 0.2495 - 0.00288T + 0.0016t + 0.00003T^2 - 0.000007Tt - 0.00002t^2$	R^2=68.0%;	(15)
$Y_c = 0.3738 - 0.0008T + 0.0033t + 0.000005T^2 - 0.000004Tt - 0.00002t^2$	R^2 = 92.5%;	(16)

Where 56.1 = KOH molecular weight (g/mol)
The acid value was determined according to a reported procedure (Hou J.–F. Shaw, 2008), (Pomeranz and Meloan, 1994). Free Fatty Acid was calculated using the following formula:

$$\%FFA = \frac{Titration\ value\ \times \text{KOHNormality}\ \times 28.2}{Sample\ weight} \quad (6)$$

Where 28.2 is the oleic acid molar weight in g/mol.
Acid value and FFA% could be correlated as:

$$\frac{\text{AV}}{FFA} = \frac{56.1}{28.2} = 1.99 \quad (7)$$

$$\text{AV} = 1.99 * FFA \quad (8)$$

The percentage of free fatty acid is commonly calculated in expression of oleic acid, (1000 g) of sample contains 282 g of oleic acid (Dileesh et al., 2013), (O'Brien, 22008).
The Saponification value was determined according to a reported procedure (ASTM, 2006), Saponification value was calculated using the following formula:

$$SV = \frac{\text{blank titer value}\ \times \text{sample titer value}\ \times \text{M of HCl}\ \times M.Wt\ \text{o}}{Sample\ weight} \quad (9)$$

Determination of ester value and Percentage of Glycerol were determined according to a reported procedure from (Chan et al., 2010), (Japan Food Chem. Res. Found., 2009), (Ramos et al., 1997). Where:

$$Ester\,Value = Saponification\,Value - Acid\,Value \quad (10)$$

$$\%Glycerol = Ester\,Value * 0.0546 \quad (11)$$

The Iodine value was determined according to a procedure in (Barlow et al., 1997):

$$Iodine\ value = \frac{\text{blank titration} - \text{Sample titration} * 0.1 * 12.69}{Sample\ Weight} \quad (12)$$

The peroxide value PV was determined according to a reported procedure (ASTM international, 2005), (De Graaf et al., 2003) :

$$\text{PV} = \frac{(\text{Sample Titration} - \text{Blank Titration}) * Thiosulfate\ \text{Normality}}{\text{Sample weight}} \quad (13)$$

Where PV is expressed in milli-equivalents peroxide per 1000 g sample.

The values of all the properties were determined in triplicates. The standard deviation results are stated in Table 3.

RESULTS AND DISCUSSION

The results obtained from various experiments are expressed below:

Water content

Water content was measured in the different cases of DIC-textured and non-textured Camelina seeds by oven and IR moisture analyzer. After initial calibration (Chang, 1987), (Bogart, 2015) , the values were recorded in Table 2.

Extraction yield

The values of vegetal oil yield extracted from the seeds (by pressing and by solvent) and the cakes (by solvent) are presented in Table 2. Camelina S. seeds treated by DIC at (Steam Temperature T: 157.7°C and thermal treatment time: 40.6 s) give the highest value of extracting percentage of vegetal oil yield in both vegetal oil extraction methods, which reaches 0.3153 and 0.4490 g oil/g dry seeds for pressing and solvent extraction process respectively. Since the untreated seeds, gave only about (0.2277 and 0.3681g oil/ g dry seeds) for pressing and solvent extraction process respectively. Because the pressing leaves a substantial amount of residual vegetal oil in the oilcake/meal, the solvent extraction process was used to recover the residual vegetal oil from the resulting pressed cake (Sampath, 2009). The advantage of pre-pressing is that a press cake is formed from the small granules which should present a good solvent contact and percolation in the extractor. The 2-stage vegetal oil extraction operation of screw press and solvent extraction usually combines an economic advantages of higher yield and lower costs, better than single solvent extraction (Matthaus, 2012).

The cake from Camelina raw material has more percentage of vegetal oil (0.0929 g oil/g db cake) than all

Table 4. Chemical and physical properties of oil from raw material and DIC-treated Camelina seeds, in comparison with other researches.

Properties	Oil from Control Seeds	Oil from DIC treated seeds	Value in Other researches	References
Density at 15°C (g/cm³)	0.9205±0.2	0.9276±0.5	0.9228	[50]
Density at 25°C (g/cm³)	0.9182±0.07	0.9189±0.1	0.920; 0.9184	[48], [50]
Kinematic Viscosity at 40 °C (mm²/s)	27.86±0.8	28.07±0.3	30.9	[50]
Refractive Index at 25 °C	1.4776±0.003	1.4768±0.001	1.4756±0.0001	[54], **[50]**
Iodine Number (g I_2/100 g Oil)	112.08±0.5	86.28±0.6	127–155, 105, 104.7±0.3	[54], [55], [48], [50]
Acid number (mg KOH/g)	4.42±0.1	4.44±0.06	3.6 (1 to 5)	[65], [4]
Free Fatty Acids Content wt.%	2.22±0.1	2.23±0.3	0.2	[66]
Saponification Value (mg KOH/g Oil)	188.24±0.3	192.52±0.04	180-190, 187.8, 187.8±0.1	[54], [48], [50]
Ester value (mg KOH/g)	183.82	188.08	/	/
%glycerin	10.05	10.28	/	/
Peroxide Value, (m eq O_2/kg)	2.38±0.003	2.37±0.02	(2.38 ±0.01)	[50]

other DIC treated Camelina cakes which refers to that the mechanical pressing instruments has more efficiency with treated seeds than the raw materials, lower value was observed with cake from DIC treated number (5) which was (0.0585 g oil/g db cake).

Effect of DIC parameters on Solvent Extraction of Seeds.

In the experimental design, we used differently DIC-treated seeds in the estimation of vegetal oil yields (oil g/g seeds db) extracted by pressing and solvent and from pressing-cake from by solvent as the response parameters (or dependent variables). Figure 2 shows the effects of the operating parameters T and t on yield of vegetal oil; in terms of Pareto chart, Main effects, and response surface.

In the case of pressing of DIC-textured seeds shown in Figure 2(A), the yield Y_A shows that the most effective term was the interaction between T and t, while the linear and quadratic terms of each parameter individually were not significant. It was then possible to establish an empirical model of Y_A versus the DIC processing parameters. R^2 value = 71.4% proved that the empirical model (R^2=71.4%; Eq. 14) of yields versus DIC parameters was relevant. It is worth noting that the optimum value was achieved at T = 164 °C and t = 45 s. The highest value of pressing of DIC textured seeds Y_A = 0.3153 g oil/g seeds db was 38.5% greater than the raw material non-DIC treated seeds, which gave a yield of 0.2277 g oil/g seeds db.

Figures 2, 3 and 4 show that temperature T had non-significant effect while processing time t had a significant and negative effect on oil yield of solvent extraction of cake delivered from pressing of DIC-textured seeds. Thus, the lower the treatment time, the higher the residual oil in the cake. In the present case, the optimized DIC conditions should be operated for the lowest Y_B value. The optimum conditions were calculated to be T = 165°C and t = 15 s.

Moreover, it is worth comparing the evolutions of pressing yield Y_A and the residual oil in the meal Y_B. It was obvious that the higher the pressing effectiveness, the lower the residual oil in cake (Figure 5)..

Empirical model of solvent extraction yields from the DIC seeds cake was established versus the DIC processing parameters (Table 3).

The value of R^2=68.0%; Eq. 15 proved that this empirical model defining the impact of DIC treatment was moderately relevant for solvent extraction of pressing cake following DIC-texturing of Camelina seeds.

DIC-texturing seeds resulted in greatly increasing vegetal oil solvent extraction with a deeply high yield with 0.4490 against 0.3681 g oil/g db for raw material non-textured Camelina seeds; it means texturing by DIC increased the solvent yield by about 22%. Figures 2, 3 and 4 show that the most effective terms of DIC parameters were separately processing time and temperature, with positive impacts, which means the higher the DIC treatment time and temperature, the higher the yield of vegetal oil issued from solvent extraction of textured seeds. An empirical model of the DIC seeds-solvent extraction yields versus the DIC processing parameters was also created (Table 3). The value of R^2=92.5%; Eq. 16 and the levels of t and T parameters support that the DIC treatment was highly relevant with solvent extraction process of DIC-textured Camelina seeds. The optimum value was calculated to be T=165 °C and t = 45 s.

Finally, it is worth noting that the optimum of DIC treatment, which induced the highest pressing yield, the lowest presence of residual vegetal oil in pressing meal, and the highest yield of solvent extraction, was obtained at T=165 °C for 45 s.

Physical and chemical characteristics of vegetal Oil

Table 4 presents some physical and chemical characteristics of vegetal oil raw material Camelina and values issued from DIC-treated Camelina vegetal oil are also presented.

Determination of Density

The values of the density of Camelina vegetal oil raw material and treated by DIC at two different temperatures are present in Table 4. At 15 °C, the density value was 0.9205±0.2 and 0.9276±0.5 g/cm^3, and at 25°C the density was (0.9182±0.07and 0.9189±0.1g/cm^3), obtained from raw material non-treated Camelina and DIC-treated Camelina, respectively. These values were similar to other research's (Abramovič and Abram, 2005), (Zahir et al., 2014). Thus, vegetal oil issued from DIC-treated Camelina seeds was slightly denser than the control vegetal oil but still lower than the other research results. This slightly increasing refers to the fact that the vegetal oil from DIC-treated seeds had slightly higher degree of unsaturation than the vegetal oil from raw material seeds.

Determination of Kinematic Viscosity

Table 4 shows that the kinematic viscosity at 40 °C of raw and treated materials was (27.86±0.8 and 28.07±0.3 mm^2/s), respectively. There was no significant change in the value, and that proved the DIC treatment did not make any decomposition for the vegetal oil. From another study done on Camelina, it was shown a slight increasing of Camelina vegetal oil viscosity from 30.7 to 31.8 mm^2/s happened when Camelina seeds were heating at 180°C for one day. So it strongly emphasizes that a short-time heating doesn't affect the viscosity (Zahir et al., 2014), (Crowley and Frohlich, 1998).

Determination of Refractive Index

Refractive index (RI) of vegetal oil is one of the most crucial quality attributes, which would imply deception or reveal purity of vegetal oil (Yunus et al., 2009). RI is a parameter that relates to molecular weight, FA chain length, degree of unsaturation, and degree of conjugation (Gunstone, 2011).

In Table 4, Refractive Index RI measured at 25°C was 1.4776±0.003 and 1.4768±0.001 for vegetal oil obtained from raw material and vegetal oil acquired from DIC-treated Camelina seeds, respectively. This is comparable with the values from other studies. Since refractive index increases with additional double bonds, and particularly conjugated double bonds, the stability of RI proves that DIC treatment did not affect this aspect of vegetal oil.

Determination of Iodine number

The iodine number IN indicates the unsaturation degree of a vegetable oil. This is an important parameter in the study of the oxidative rancidity of vegetable oils;

the higher the degree of unsaturation, the greater the possibility of rancidity of vegetable oils (Sadasivam and Manickam, 2005). Low iodine values lead to greater oxidative storage stability. During storage, the chemical changes and oxidative of vegetal oils are characterized by a decrease in the total unsaturation degree of vegetal oils and an increase in free fatty acid contents (Zahir et al., 2014). Table 5 indicates that the iodine value for raw material and DIC-treated Camelina were (112.08±0.5 and 86.28±0.6 g I_2/100 g Oil), respectively. These results indicate that the DIC-treated seeds had less double bonds and then better oxidative stability.

Free Fatty Acid or Acid Value

Free fatty acid, frequently stated as acid value, is defined as the amount of KOH (expressed in mg) capable of neutralizing 1.0 g of the oil sample (Salhin et al., 2013). The fatty acid composition in the seeds should be correlated to the functional properties, nutritional value, shelf-life, and flavor of the food products resulting from them (Berry and Pertanika, 1982).

In our work, Camelina vegetal oils issued from raw material and DIC-treated seeds had acid value recorded to be 4.42±0.1 and 4.44±0.06, respectively. They also contained degrees of free fatty acids of 2.22±0.1 and 2.23±0.3, respectively. Although DIC-treatments exposed the seeds to high temperature (115 to 165°C), which normally should cause increasing in FFA%, DIC treatment had no effect because such high temperature was done in very short time (maximum time was at 45 seconds). Research performed on Palm vegetal oil revealed that heating did not significantly affect FFA content and the percentage of free fatty acid varied from 3.500±0.200 in a raw material to 3.533±0.215 after exposition to 180°C for 14 h (Dongho-Dongmo et al., 2014). Additionally, research done on Camelina sativa revealed there was no significant effects of short-time heating on Camelina vegetal oil FFA%, which varied from FFA%=3.00 for Camelina raw material to 3.25 after heating at 180 °C for 24 h, and 3.50 after 5 days (Crowley and Frohlich, 1998).

Determination of Saponification Value SV

SV is expressed as the quantity in mg of KOH required for the saponification of the esters and the neutralization of the free acids of 1 g of the vegetal oil [33]**.** In Table 4, the saponification value SV is recorded for raw material and DIC treated Camelina to be (188.24±0.3 and 192.52±0.04 mg KOH/g Oil) respectively.

Low refractive index and high saponification value indicate low average molecular weight of fatty acids. Saponification value was used to predict the type of glycerides in vegetal oils and fats by measured the alkali-reactive groups in the samples. Saponification values of glycerides are as high as the chain fatty acids are short (O'Brien, 2008).

From our results, we can predict that the vegetal oil from DIC treated seeds has very slightly shorter-chain fatty acids, accordingly lower molecular weight than the vegetal oil produced from raw material seeds.

Determination of Ester Value and Percentage of Glycerol

Ester value is a measurement of the saponifiable amount of glyceride present in a sample of vegetal oil, (Dileesh et al., 2013). In Table 4, the ester value calculated for raw material and DIC-treated Camelina seeds was (183.82 and 188.08 mg KOH/g), respectively. The percentage of glycerol was also calculated for vegetal oil from raw material and DIC-treated Camelina to be 10.05 and 10.28, respectively. This means that the glycerol in DIC vegetal oil was founded to be slightly more than in raw material oil, and, hence, with higher FFA percentage. This slightly higher percentage of glycerol gives the seeds a higher glass transition temperature due to greater polarity of glycerol than triglyceride (de Graaf et al., 2003).

Determination of Peroxide Value PV

The Peroxide Value PV is the amount of peroxide contained in 1000 g of the substance; it is expressed in mg of active oxygen. PV is a relevant parameter able to reveal the quality and stability of the vegetal oil recognizing the range where rancidity reactions happen during storage. It is worth mentioning that oil PV increases with the storage time, temperature and contact with air (Zahir et al., 2014).

Peroxide Value measures primary oxidation reactions of the oil, which mainly happen from hydro-peroxides. In general, the lower the Peroxide Value, the better the quality of the oil. Thus, when the oil has high levels of peroxides, it may remain odorless as long as the secondary oxidation has not started. The secondary stage of oxidation happens when the hydro-peroxides decompose into carbonyls and other compounds, in particular aldehydes. These quality aspects measured by the Acid Value, are the reason of the vegetal oil to get a rancid smell (Moser, 2010). In Table 5, Peroxide value for vegetal oil from raw materials and DIC-treated seeds were 2.38±0.003 and 2.37±0.02 respectively, these two results were very close to each other's.

CONCLUSION

The data issued from this research work have evidenced that DIC-assisted pressing or solvent extraction processes has positive impacts on the yield of vegetal oil extraction from the dried Camelina sativa seeds. Among the different DIC conditions, the experiment 5 (time: 40.6 s; Temperature: 157.7 °C) gave the highest vegetal oil yield of pressing (0.3153 g oil/g db) and solvent extraction (0.4490 g oil/g db). In order to exam the suitability of DIC treatment with Camelina seed properties, the determination of some chemical and physical properties, which were done and compared with other research results proved that DIC treatment does not affect the oil properties. Thus, the vegetal oil did not suffer from any decomposition during the DIC processing. All Camelina vegetal oil properties had values close to raw materials. Only exception was the iodine value, which had lower value for DIC-treated oil than that of both oils from Camelina raw material and in other references. This indicates that there were lower double bonds in vegetal oil from DIC-treated seeds than the vegetal oil from untreated seeds. This property will serve a lot in using Camelina vegetal oil for biodiesel manufacturing.

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Added Graphic.