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Pressure, temperature and processing time in enhancing *Camelina sativa* oil extraction by Instant Controlled Pressure-Drop (DIC) texturing pre-treatment

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SUMMARY: Instant Controlled Pressure Drop (DIC) was evaluated as a texturing pre-treatment for the extraction of *Camelina sativa* (L.) oil. DIC was coupled to Accelerated Solvent Extraction (ASE), Pressing and Dynamic Maceration (DM). DIC optimization was performed by studying the effects of pressure, temperature and processing time on oil yield. DIC + ASE obtained seed-oil yields of 615.9±0.5 against 555.5±0.5 g oil/kg-ddb for untextured seeds (RM). Via pressing, oil yields were 490.9±0.5 and 444.7±0.5 g oil/kg-ddb for textured and untextured seeds, respectively. Through coupling DIC (P: 0.63 MPa and t: 105 s) to the pressing extraction (60 s) of seeds along with 2h of DM of meals, it was possible to reach 605.8 g oil/kg ddb of oil yield. The same results were not obtained for RM seeds, where after 24 h of DM extraction, the oil yield was 554.7 g oil/kg ddb. DIC allowed for an increase in Camelina oil yields, reduced extraction time and valorized pressing meals.

KEYWORDS: Camelina sativa*; "Instant Controlled Pressure Drop" DIC; Meal valorization; Oil pressing extraction; Oil solvent extraction; Texturing*

RESUMEN: ***Presión, temperatura y tiempo de procesamiento para mejorar la extracción de aceite de* Camelina sativa *mediante pretratamiento texturizado de descompresión instantánea controlada (DIC).*** La tecnología de Descompresión Instantánea Controlada (DIC) fue evaluada como un pretratamiento para la extracción de aceite de *Camelina sativa* (L.). El pretratamiento DIC fue acoplado a la Extracción Acelerada de Disolventes (ASE), al Prensado y a la Maceración Dinámica (DM). La optimización de DIC fue llevada a cabo a través del estudio de los efectos de presión, temperatura y tiempo de proceso en el rendimiento del aceite. ASE + DIC permitió alcanzar rendimientos de 615,9±0,5 comparado con 555,5±0,5 g aceite/kg-ddb (base seca) en el caso de las semillas sin texturización (RM). En el caso del prensado, los rendimientos fueron de 490,9±0,5 y 444,7±0,5 g aceite/kg-ddb para las semillas con y sin texturización, respectivamente. Al acoplar el tratamiento DIC (P: 0.63 MPa y t: 105 s) + la extracción por prensado de las semillas (60 s) + 2h de DM de las harinas, fue posible alcanzar un rendimiento de 606,7 g aceite/ kg ddb. No así para las semillas sin tratamiento, en las que posterior a 24 h de extracción por DM, el rendimiento fue de 554,7 g oil/kg ddb. La texturización DIC permitió incrementar los rendimientos del aceite de Camelina, reducir los tiempos de extracción y valorizar las harinas del prensado.

PALABRAS CLAVE: Camelina sativa*; "Descompresión Instantánea Controlada" DIC; Extracción de aceite por prensado; Extracción de aceite por solvente; Texturización; Valorización de la harina*

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1. INTRODUCTION

Native to different Romanian areas, northern Europe, the Mediterranean region, and Central Asia, Camelina (*Camelina sativa* L.*)* is an ancient annual oilseed crop that belongs to the *Cruciferae* family (Brassicaceae, Mustard) (Hurtaud and Peyraud, 2007; Carciumaru, 2007). Also named 'False flax' because its fruits resemble flax bolls (*Linum usitatisimum* L.), and 'Gold of Pleasure' a popular name invented by the Romans in the early centuries (Berti *et al.*, 2011), nowadays Camelina plays an important role in oilseed biotechnology. As an example, in 2008, for the first time, Chile introduced camelina seeds as a potential feedstock for biodiesel. It has also drawn the attention of the pharmaceutical industry thanks to its high omega-3-content. (Berti *et al.*, 2011).

Furthermore, thanks to its high oil content, with about 0.40 g oil/g db (dry matter basis), which is 0.67 g oil/g ddb (dry-dry matter basis: this means a mass basis of raw material rid of water and oil), and its healthy oil properties, with up to 90% of unsaturated fatty acid, camelina seeds have seen an increased interest from different industries (Budin *et al.*, 1995; Li *et al.*, 2014).

Camelina seed vegetable oil is extracted by cold mechanical pressing, by solvent extraction, or by a combination of both methods. In fact, combinations of both methods are most often used for economic reasons since the pressing process leaves a significant amount of residual oil in the oilcakes and meal, which can be extracted by solvent extraction (Gunstone, 2006). Indeed, camelina oilcakes/meal remaining from seed pressing typically contains 10 to 15% residual oil, 40% crude protein, about 13% fibers, 5% minerals, and minor amounts of other substances such as vitamins (Li *et al.*, 2014; Zubr, 1997). Moreover, thanks to its high crude protein content, oilcakes can be used as nutritive supplements in animal feed formulations (Moloney *et al.*, 2001).

Mechanical pressing and solvent extraction are the most commonly applied industrial techniques for camelina oil recovery, although they present some drawbacks such as low yields, long extraction periods, toxicological risks, excessive solvent residues and high costs (Belayneh *et al.*, 2015; Kartika *et al.*, 2010). To overcome these constraints, several oilseed pre-treatments have been evaluated at laboratory scale, such as pre-heating oilseeds using a hot air oven and microwaves (Yusuf, 2018). However, although these pre-treatments increase oil yield, their use at the industrial level remains difficult because of the drawbacks of their scaling-up and high costs, and the difficult preservation of the oil's quality. In this respect, the Instant Controlled Pressure Drop (DIC) and high-temperature short-time texturing pre-treatment makes the seed oil more readily available.

DIC technology is based on thermo-hydro-mechanical processing induced by subjecting a product to a rapid transition from high-steam pressure to a vacuum, triggering an instant autovaporization of a quantity of material water, a fast cooling and a well-controlled expansion of the product. The change in the structural characteristics of the product is generally revealed through the expansion rates of the product, which depend on the operating conditions. Various studies have shown that the most influential DIC operating parameters are saturated steam pressure, which is strictly correlated with temperature, and processing time (Mounir and Allaf, 2008; Allaf and Allaf, 2013).

On the other hand, previous studies on numerous crops (Allaf *et al.*, 2014; Bouallegue *et al.*, 2015), have shown that this innovative technology can intensify oil solvent extraction by giving higher yields in a shorter time, and can be easily scaled-up at industrial level by using the same processing conditions optimized at laboratory scale. Specifically, in the case of camelina seeds, it has been shown that DIC could be used to intensify the in-situ transesterification of seeds, leading to a significant increase in biodiesel production yield (Bamerni *et al.*, 2017). Moreover, the composition of camelina oil from DIC textured seeds was similar to raw material oil, which means a good preservation of the oil's quality (Bamerni, 2018).

Therefore, the goal of this study was to enhance the oil extraction of *Camelina sativa* seeds and meals by coupling Instant Controlled Pressure Drop (DIC) texturing pre-treatment to mechanical pressing and solvent extraction. To optimize oil yield extraction while preserving the quality of both oil and meals, the impacts of saturated steam pressure and processing time of DIC texturing pre-treatment were studied.

2. MATERIALS AND METHODS

2.1. Materials

Camelina seeds (*Camelina sativa* L. Crantz) were provided by "Sanctum Méditerranée"; 30250 JUNAS (France), and conserved at room temperature (~24 °C) before extraction and assessments. All reagents used were of analytical grade.

2.2. Methods

2.2.1. Moisture content determination

Camelina moisture content was determined by the ISO 6540:2010 gravimetric method (ISO, 2010). Samples (2 g) were placed on an oven (Air Concept-Fir Labo, AC 60) at 105 °C for 24 h, and constant weight was determined by an analytical balance (OHAUS MB23, accuracy of ±0.0001 g). All moisture contents were expressed on dry basis (db).

2.2.2. Solvent and pressing extraction of Camelina sativa *oil*

Camelina sativa seeds were sorted, cleaned and divided into two groups: 1) Raw material seeds, and 2) DIC-textured seeds. For oil extraction, four individual operations were studied: 1) Cold Pressing Extraction; 2) n-hexane Accelerated Solvent Extraction ASE; 3) Dynamic-Maceration extraction DM extraction, and 4) a coupled cold pressing of seeds and DM solvent extraction of meals. The processing protocol of DIC pre-treatment of *Camelina sativa* seeds and the mechanical and solvent extractions of oil are illustrated in Figure 1.

Solvent extraction. Two methods were used for solvent extraction; Accelerated Solvent Extraction (ASE) and Dynamic Maceration Method (DM). Before any solvent extraction, camelina seeds were ground at the rate of 10000 rpm for 15 s (Grindomix, GM200 - F. Kurt Retsch GmbH & Co. KG, Haan, Germany), and the obtained powders were sieved (Vibratory Sieve Shaker, Fritsch, Germany) into 0.4 mm particle size fractions. Sieving was carried out for 10 min under an amplitude of 1.5 mm and the camelina powders were kept at 4 °C in the dark until analysis.

To define ASE suitable conditions for camelina, preliminary tests based on the study of Kraujalis *et al.*, (2013) were performed on a Dionex ASE 350 system (Thermo Fisher scientifique, Sunnyvale, CA, USA). 7 g of camelina seed powder were mixed with 1 or 2 g of diatomaceous earth and placed in a 34 ml Dionex stainless-steel cell (2.9 cm diameter). Hexane was used as a solvent, which represented 60% of the cell volume. ASE was initiated by 5 min pre-heating time to reach 100 ºC at 10 MPa, followed by 4 cycles of extraction of 10 min each. Then, cell content was purged during 150 s with nitrogen to remove impurities and the extracts were collected in a vial. Hexane was removed by a rotary vacuum evaporator under reduced pressure (10 mbar; 40 °C), and the extracted oils were dried under a stream of nitrogen. Extractions were conducted in triplicate. Finally, the oil extracts were weighed and stored at 4 °C for subsequent chemical analysis. Oil yield was expressed in g oil/kg ddb ($Y_{ASE;}$ seeds); ddb concerns material which excludes both water and oil contents.

DM was performed with extraction batches of 2 g of grain powder with 20 ml of n-Hexane. To assure a good contact between the phases, the entire extraction operation was conducted under magnetic stirring at 400 rpm. The extraction was carried out at ambient temperature (25 °C), in triplicate. To evaluate the extraction performance, oil yield was determined at different interval times (0, 15, 30, 45, 60, 90, 120, 150, 180, 240, 300, 360, 420, 480 and 1440 min). To measure the oil contents, the extracts were filtered with 0.2 μm PTFE filters (Sartorius Stedim Biotech GmbH/Germany), and the mixtures (hexane/oil solutions) were separated under vacuum by nitrogen flow (Liebisch Mini evaporator, Germany). The extracted oil was dried until constant weight. Oil extraction yield ($Y_{DM;}$ seeds) was calculated as shown in equation 1. Oil yield was expressed in g oil/kg ddb.

$$Y_{DM;seeds} = \frac{\begin{array}{c}\textit{weight of oil extract obtained after}\\ \textit{solvent extraction}\end{array}}{\begin{array}{c}\textit{weight of concerned dried}\\ \textit{dried powder}\left(ddb\right)\end{array}} \qquad \text{Eqn.1}$$

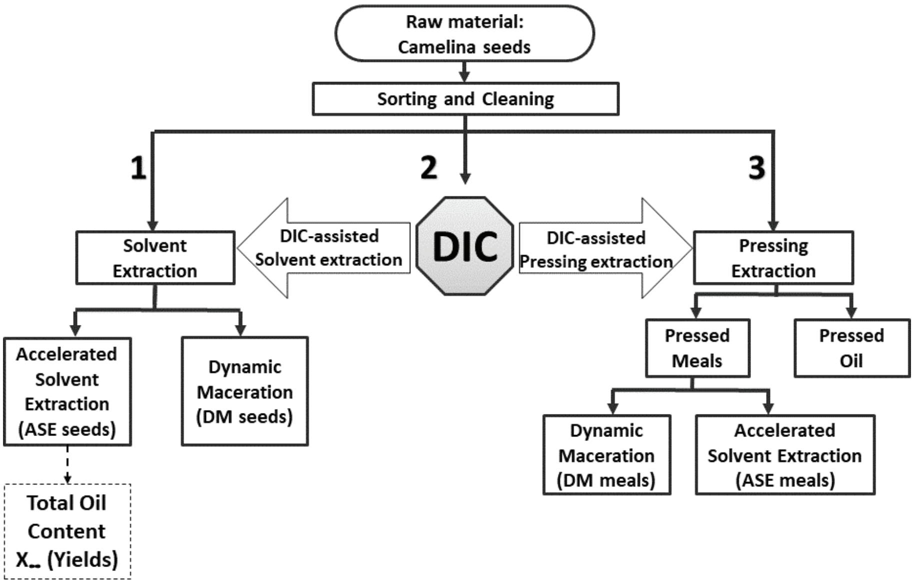

FIGURE 1. Study protocol of oil extraction from *Camelina sativa* seeds and meals.

Pressing extraction. Pressing extraction was carried in a single-screw press machine ("OMEGA 20" type "Taby Orebro", Germany). First, without any sample, the screw-press barrel was heated by an electrical resistance to 80 °C. Subsequently, 300 g of camelina seeds were placed in the screw press, and oil and meals were recovered after 30 to 60 s of pressing. The diameter of pressing nozzle was 8 mm, and fine particles in the expressed oil were separated by filtration. Oil content was gravimetrically determined ($Y_{pressing\ 1}$), and it was expressed as g oil/kg of dried seeds (ddb):

$$Y_{pressing} = \frac{\textit{weight of oil extract obtained after pressing extraction}}{\textit{weight of dried dried seeds}\left(dbb\right)} \qquad \text{Eqn. 2}$$

To measure the total residual oil contents of the meals, the ASE method was applied by using 10 g of meal powder ($Y_{ASE;}$ meals), and the recovered oils were stored at 4 °C for further analysis. To establish the extraction kinetics of camelina pressed meals, the DM method was applied as described previously ($Y_{DM;}$ meals).

DIC texturing pre-treatment. The DIC (French for Détente Instantanée Contrôlée) is a thermo-mechanical process induced by subjecting the product to a saturated steam pressure (about 0.05 – 1 MPa, according to the product) for a short period of time (some seconds), followed by an abrupt pressure drop towards a vacuum (up to 1.5 kPa). This abrupt pressure drop ($\Delta P/\Delta t > 0.2$ MPa s^{-1}) triggers an autovaporization of volatile molecules which induces a cooling and texturing effect (Allaf and Allaf, 2013). Figure 2A shows the schematic time-temperature-pressure profiles of a DIC processing cycle.

DIC texturing pre-treatment was carried on 300 g of camelina seeds, and selected DIC processing parameters were saturated steam pressure (from 0.2 to 0.7 MPa) and treatment time (from 20 to 120 s). The DIC equipment was composed of three main elements: i) the processing vessel (1), where samples were treated; ii) the vacuum system, which consists of a vacuum tank (2) with a volume 130 times greater than the processing reactor, and an adequate vacuum pump (3) and iii) the pneumatic valve (V2) with a large diameter (more than 200 mm). To ensure an abrupt/instant connection between the vacuum tank and the processing reactor, V2 was opened in a very short time (less than 0.2 s). Figure 2B presents the schematic diagram of the DIC equipment.

To determine the impact of the DIC treatment on the oil extraction yield and quality, treated samples were submitted to both extraction methods: a) Solvent Extraction (ASE and DM) and b) Pressing Extraction. Untreated raw material was used as a control (RM), and in both cases oil yields were expressed in g oil/kg ddb.

2.2.3. Experimental design and statistical analysis

Through previous preliminary studies, saturated steam pressure (P) and process heating time (t) were identified as the most important independent variables in the DIC treatment of camelina seeds (n=2). Then to determine the impact of these independent variables, a five-level central composite rotatable design was employed. The studied design included $2^n=2^2=4$ (-1/-1; -1/+1; +1/-1 and +1/+1) factorial trials, 2*n=2*2=4 (-α/0; + α/0; 0/-α and 0/+α) star trials; and five repetitions of the central point (0,0). The total trials were 13. The value of α (axial distance) depending on the number (n) of operating parameters was calculated as $\alpha = \sqrt[4]{2^n} = 1.4142$. Applied operative DIC parameters are shown in Table 1.

The experiments were run randomly to minimize the effects of unexpected variability in the observed responses due to extraneous factors.

To identify the significant differences of the effects between independent variables, the analysis of variance (ANOVA) was performed ($p < 0.05$). Moreover, a second-order polynomial function was employed to relate each response variable (Y) to the operating parameters (χ) as shown in equation 3:

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i \chi_i + \sum_{i=1}^{k} \beta_{ii} \chi_i^2 + \sum_{i=1}^{k-1}\sum_{j=2}^{k} \beta_{ij} \chi_i \chi_j + \mathcal{E} \qquad \text{Eqn. 3}$$

Where Y is the response, β_0,β_i,β_{ii} and β_{ij}, are the regression coefficients, χ_i and χ_j are the independent variables of DIC, ε is random error and i and j are the indices of factors.

The significance and the adequacy of the model were interpreted by estimating the lack of correspondence, R^2 and Fisher test value (F-value). The Pareto chart was used to determine the effects that were statistically significant with a p-value of 0.05. Response surface methodology (RSM) was used to analyze the experimental design results and optimize the treatment parameters through a multi-criteria procedure. Experimental results were statistically analyzed by Statgraphics Centurion Software (MANUGISTICS Inc., Rockville, USA).

3. RESULTS

3.1. Moisture content of camelina seeds

The moisture content of raw camelina seeds was 5.5 ±0.2 g H_2O/100 g db %. After DIC treatment, among the 13 experimental points, the results varied between 4.9 to 6.0 ±0.02 g H_2O/100 g db.

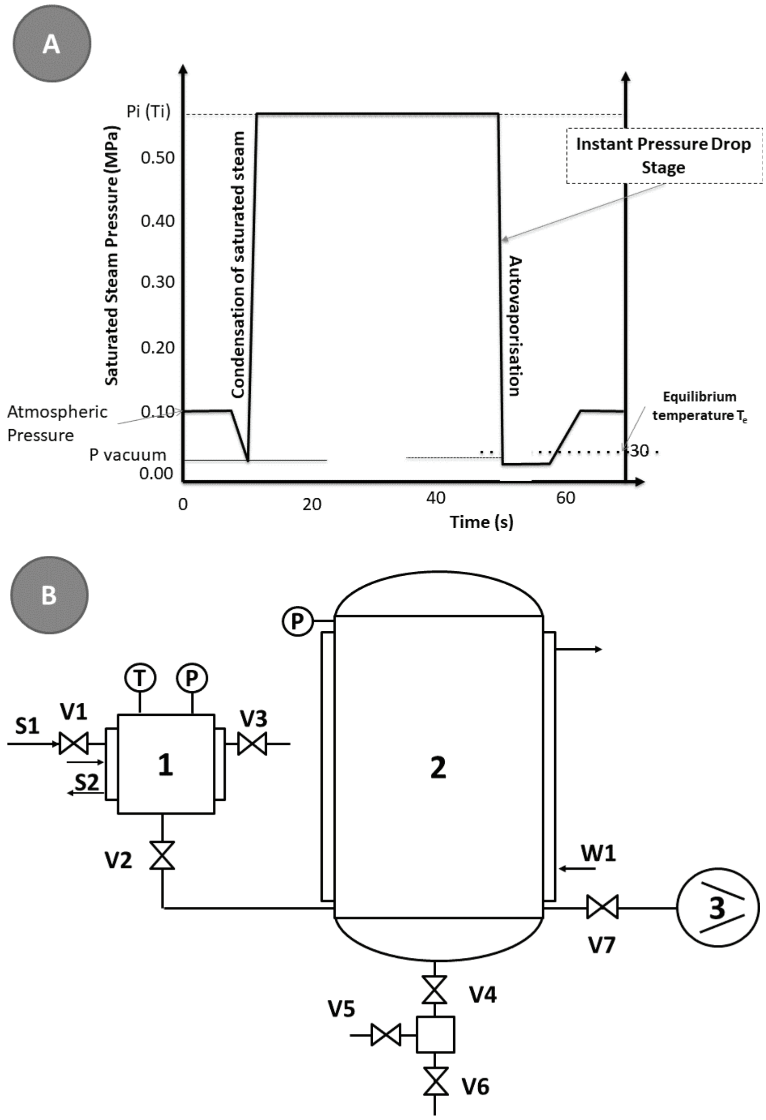

FIGURE 2. A) DIC treatment cycle: pressure evolution vs time and B) Schematic diagram of DIC equipment: (1) DIC Reactor, (2) Vacuum tank, (3) Vacuum pump, V1-V7-valves, S1 and S2- saturated steam injection, W1- cooling water.

TABLE 1. Coded and real levels of independent variables used in the experimental design

	Coded level				
Independent Variables	**-α**	**-1**	**0**	**+1**	**+α**
Saturated Steam pressure (MPa)	0.2	0.27	0.45	0.63	0.7
Saturated Steam Temperature (°C)	120.2	129.9	147.9	160.7	164.9
Processing time (s)	20	35	70	105	120

3.2. Solvent extraction of camelina seeds

3.2.1. Accelerated solvent extraction of non-treated (RM) and treated (DIC) camelina seeds

The averages oil yields of treated and non-treated *C. sativa* seeds are shown in Table 2. For RM, the average oil content obtained after ASE was 555.5 g oil/kg ddb, and in the case of DIC-treated seeds, the results varied from 569.4 to 615.9 g oil/kg ddb. The highest oil yield value for DIC samples corresponded to the experimental point DIC 5 (P: 0.63 MPa and t: 105 s) and the lowest to DIC 8 (P: 0.27 MPa and t: 35 s).

Furthermore, to evaluate the impact of DIC parameters on ASE oil yield, the Analysis of Variance (ANOVA) and the response surface methodology (RSM) were applied. Table 3 and Figures 3a and 3b illustrate the impact of P (steam pressure) and t (treatment time) on the ASE oil yield of camelina seeds. The obtained results showed that under the selected domain values, the pressure (P), the time (t) and the interaction of both variables (P and t), presented positive effects on the increase in oil yield; the higher P and t, the higher the oil yield.

By expressing the "P" in MPa and "t" in s, the statistical analysis allowed to obtain a regression model for the ASE oil yield ($Y_{\text{ASE seeds}}$), with R^2 of 89.8%:

$$Y_{ASE\ seeds} = 578.43 - 1.78\text{P} - 0.44\text{t} - 67.15P^2 + 1.74\text{Pt} + 0.000025t^2 \qquad \text{Eqn. 4}$$

To optimize (maximize) the ASE oil yield of seeds, the optimal conditions for DIC treatment for this response were P =0.7 MPa and t = 120 s (638.49 g of oil/100 g dry-dry basis).

3.2.2. Dynamic maceration extraction of non-treated (RM) and treated (DIC) camelina seeds

For RM, the average oil content obtained after 2, 8 and 24 h of DM were 284.8, 405.2 and 550.0 g oil/kg ddb, respectively. In the case of DIC-treated seeds, after 1, 2 and 8h of DM yields were 407.8, 462.8 and 520 g oil/kg ddb, respectively. All these results corresponded to experimental point DIC 5 (P: 0.63 MPa and t: 105 s). In the specific case of DM after 2h ($\text{DM}_{2\text{h}}$), DIC-treated seeds varied from 439.5 to 462.8 g oil/kg ddb. At this extraction time (2 h), in any of the selected DIC-treatment conditions, the oil yield kinetics of camelina seeds were always better than that of untreated samples. Figure 4A presents the extraction kinetics of seed oil by DM of RM and DIC 5 (0.63 MPa and 105 s).

To evaluate the impact of DIC parameters on $\text{DM}_{2\text{h}}$ oil yield, the ANOVA and the RSM were applied. Table 3 and Figures 3C and 3D illustrate the impact of P (steam pressure) and t (treatment time) on the $\text{DM}_{2\text{h}}$ oil yield of camelina seeds. The results showed that under the selected domain values, the pressure (P) and the quadratic effect of the time (t^2) had a significant effect on the oil yield. The higher the pressure, the higher the $\text{DM}_{2\text{h}}$ oil yield.

Equation 5 shows the regression model for the $\text{DM}_{2\text{h}}$ oil yield ($\text{DM}_{\text{2h; seeds}}$), with R^2 of 83%:

$$Y_{DM2h\ seeds} = 433.3 - 12.4\text{P} + 0.31\text{t} + 22.9P^2 + 0.38\text{Pt} - 0.0032t^2 \qquad \text{Eqn. 5}$$

P: Saturated steam pressure in MPa
t: Thermal treatment time in s

To optimize (maximize) the $\text{DM}_{2\text{h}}$ oil yield of seeds, the optimal conditions for DIC treatment for this response were P =0.7 MPa and t = 91 s (462.68 g of oil/100 g dry-dry basis).

3.3. Pressing extraction of camelina seeds

3.3.1. Pressing extraction of non-treated (RM) and treated (DIC) camelina seeds

The average camelina seed oil pressing yield (Y_{pressing}) from RM was 444.7 g oil/kg ddb. And in the case of DIC-treated seeds Y_{pressing} values varied from 449.8 to 490.9 g oil/kg ddb. The highest oil yield value for the DIC samples corresponded to experimental point DIC 5 (P: 0.63 MPa and t: 105 s) and the lowest to DIC 8 (P: 0.27 MPa and t: 35 s).

The ANOVA and the RSM allowed to evaluate the effect of DIC parameters on oil Y_{pressing} and the results showed that under the selected domain

TABLE 2. Oil yields from camelina seeds and pressing-meals

Run no	**$\text{DIC}_{1;\,4;\,7;\,10;\,13}$**	**DIC_2**	**DIC_3**	**DIC_5**	**DIC_6**	**DIC_8**	**DIC_9**	**DIC_{11}**	**DIC_{12}**	**RM**
Saturated Steam Pressure (MPa)	0.45	0.70	0.45	0.63	0.63	0.27	0.27	0.20	0.45	-
Processing Time (s)	70	70	120	105	35	35	105	70	20	-
$Y_{\text{ASE;seeds}}$	588.0±6.0	603.0	610.4	615.9	570.0	569.4	571.4	573.9	575.3	555.50± 4.00
$Y_{\text{2h-DM;seeds}}$	451.0±1.0	455.8	442.6	462.8	453.6	439.9	439.5	446.5	441.0	284.80±0.05
Y_{pressing} (g oil/kg ddb)	475.0±1.0	484.7	485.5	490.9	463.3	449.8	481.7	472.1	451.8	444.70± 4.00
$Y_{\text{ASE;meals}}$	131.8±0.2	133.3	132.4	133.4	133.0	131.0	131.2	130.7	131.7	110.00±0.25
$Y_{\text{2h-DM;meals}}$	113.0±4.0	114.1	113.8	115.8	114.5	111.4	112.5	111.9	113.6	51.60±0.27

$Y_{\text{ASE; seeds}}$: Accelerated solvent extractionoil yield faraom camelina seeds assisted by DIC
$Y_{\text{2h-DM;seeds}}$: Dynamic Maceration 2h oil yield from camelina seeds assisted by DIC
Y_{pressing}: Pressing extraction 1 oil yield from camelina seeds assisted by DIC
$Y_{\text{ASE;meals}}$: Accelerated Solvent Extraction oil yield from camelina meals assisted by DIC
$Y_{\text{2h-DM;meals}}$: Dynamic Maceration 2h oil yield from camelina meals assisted by DIC

TABLE 3. Analysis of Variance (ANOVA) of the camelina oil yields from the different studied extraction methods

Source	Sum of Squares	Degree of freedom	Mean Square	F-Ratio	P-Value
Oil seeds extraction assisted by DIC texturing pre-treatment					
1. Analysis of variance of Accelerated Solvent Extraction oil yield from camelina seeds assisted by DIC ($Y_{ASE;\ seeds}$)					
A: Pressure	9.32118	1	9.32118	13.59	**0.0078**
B: Time	11.9513	1	11.9513	17.43	**0.0042**
AA	0.0843607	1	0.0843607	0.12	0.7361
AB	4.84	1	4.84	7.06	**0.0326**
BB	0.769668	1	0.769668	1.12	0.3246
Total error	4.80019	7	0.685741		
Total (corr.)	31.7138	12			
2. Analysis of variance of Dynamic Maceration 2 h oil yield from camelina seeds assisted by DIC ($Y_{2h\text{-}DM;\ seeds}$)					
A: Pressure	3.14405	1	3.14405	22.92	**0.0020**
B: Time	0.152975	1	0.152975	1.12	0.3260
AA	0.0384822	1	0.0384822	0.28	0.6127
AB	0.2304	1	0.2304	1.68	0.2361
BB	1.0751	1	1.0751	7.84	**0.0265**
Total error	0.960233	7	0.137176		
Total (corr.)	5.67451	12			
3. Analysis of variance of pressing extraction oil yield of camelina seeds assisted by DIC ($Y_{pressing}$)					
A: Pressure	2.05225	1	2.05225	38.79	**0.0004**
B: Time	14.3538	1	14.3538	271.29	**0.0000**
AA	0.092801	1	0.092801	1.75	0.2270
AB	0.046225	1	0.046225	0.87	0.3811
BB	0.96265	1	0.96265	18.19	**0.0037**
Total error	0.370361	7	0.0529087		
Total (corr.)	17.9757	12			
Oil meals extraction assisted by DIC texturing pre-treatment					
4. Analysis of variance of Accelerated Solvent Extraction oil yield from camelina meals assisted by DIC ($Y_{ASE;\ meals}$)					
A: Pressure	0.077558	1	0.077558	195.65	**0.0000**
B: Time	0.0037471	1	0.0037471	9.45	**0.0180**
AA	0.00126782	1	0.00126782	3.20	0.1169
AB	0.0001	1	0.0001	0.25	0.6309
BB	0.00238088	1	0.00238088	6.01	**0.0441**
Total error	0.00277491	7	0.000396416		
Total (corr.)	0.0874308	12			
5. Analysis of variance of Dynamic Maceration 2 h oil yield from camelina meals assisted by DIC ($Y_{2h\text{-}DM;\ meals}$)					
A: Pressure	0.11308	1	0.11308	30.21	**0.0009**
B: Time	0.00899735	1	0.00899735	2.40	0.1650
AA	0.00668517	1	0.00668517	1.79	0.2232
AB	0.0001	1	0.0001	0.03	0.8748
BB	0.0303029	1	0.0303029	8.10	**0.0249**
Total error	0.0262023	7	0.00374318		
Total (corr.)	0.182231	12			

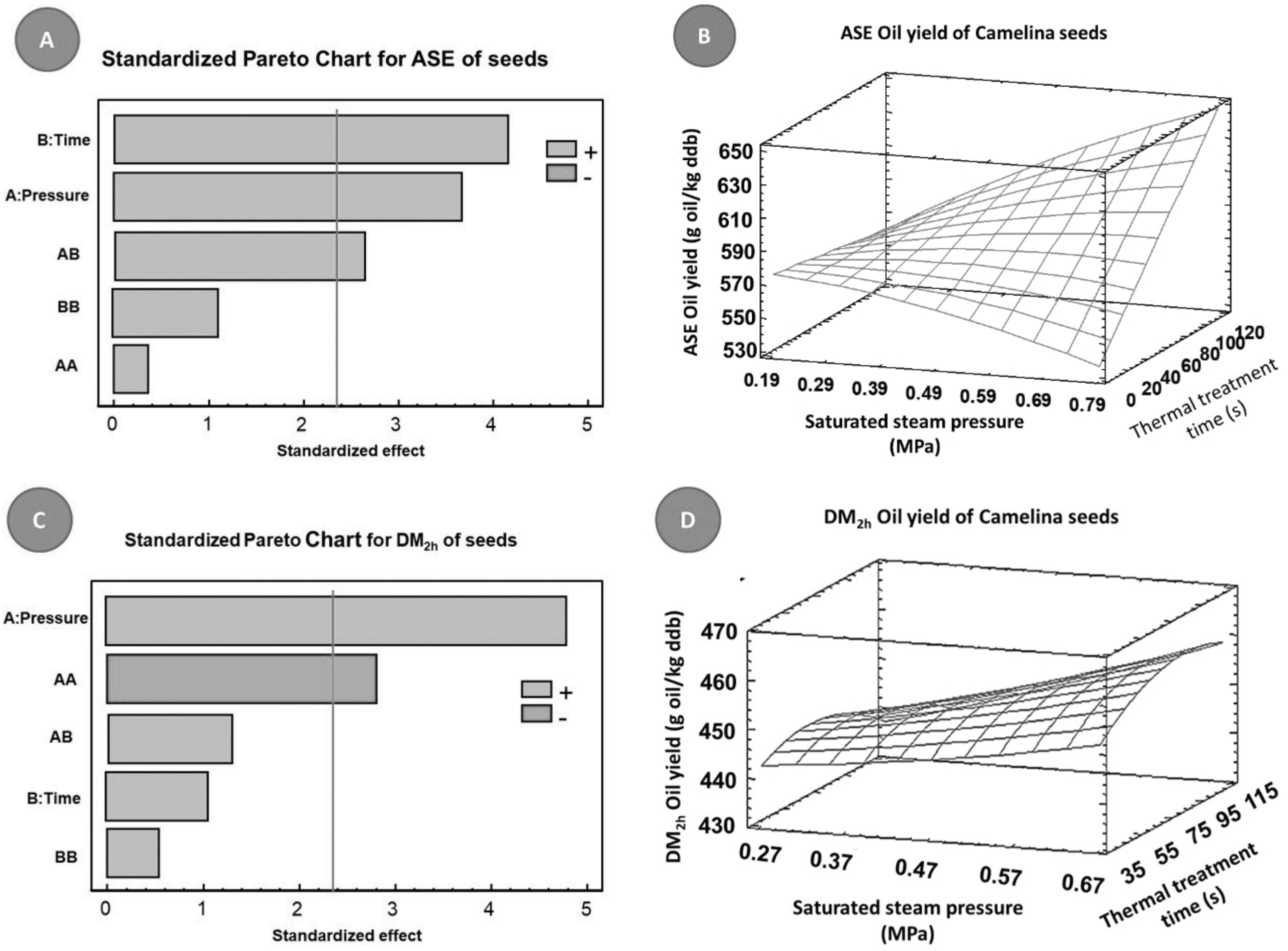

FIGURE 3. Effect of DIC parameters on the solvent extraction yields from camelina seed powder: A) Standardized Pareto Chart of ASE $_{seeds}$; B) Estimated Response Surface of ASE $_{seeds}$; C) Standardized Pareto Chart of DM$_{2hseeds}$ and D) Estimated Response Surface of DM$_{2hseeds.}$ ASE: Accelerated Solvent Extraction and DM$_{2h}$: Dynamic Maceration 2 h.

values, the pressure (P), time (t) and quadratic effect of this factor (t^2) had a significant effect on the pressing oil yield. The higher the pressure and the time, the higher the oil $Y_{pressing}$. Table 3 and Figure 5 illustrate the impact of P (steam pressure) and t (treatment time) on oil $Y_{pressing}$ of camelina seeds.

Equation 6 shows the regression model for the oil $Y_{pressing}$, with R^2 of 97%:

$$Y_{pressing} = 422.5 + 7.9\mathrm{P} + 0.88\mathrm{t} + 35.6P^2 - 0.17\mathrm{Pt} - 0.0030t^2 \quad \text{Eqn. 6}$$

P: Saturated steam pressure in MPa
t: Thermal treatment time in s

To optimize (maximize) the oil $Y_{pressing}$ of seeds, the optimal conditions for DIC treatment for this response were P =0.7 MPa and t = 120 s (493.59 g of oil/100 g dry-dry basis).

3.4. Solvent extraction of camelina meals from pressing

3.4.1. Accelerated solvent extraction of non-treated (RM) and treated (DIC) camelina meals

To determine the performance of pressing extraction, the residual oil yields of the meals were determined through ASE ($Y_{ASE;\ meals}$). The average oil meal content for RM was 110 g oil/kg ddb, while for DIC-treated samples, yields varied from 130.7 to 133.4 g oil/kg ddb. The highest oil yield value for DIC samples corresponded to experimental point DIC 5 (P: 0.63 MPa and t: 105 s) and the lowest to DIC 11 (P: 0.20 MPa and t: 70 s).

To evaluate the effect of DIC parameters on $Y_{ASE;\ meals,}$ the ANOVA and the RSM were applied. The results showed that under the selected domain values, the pressure (P), the time (t) and

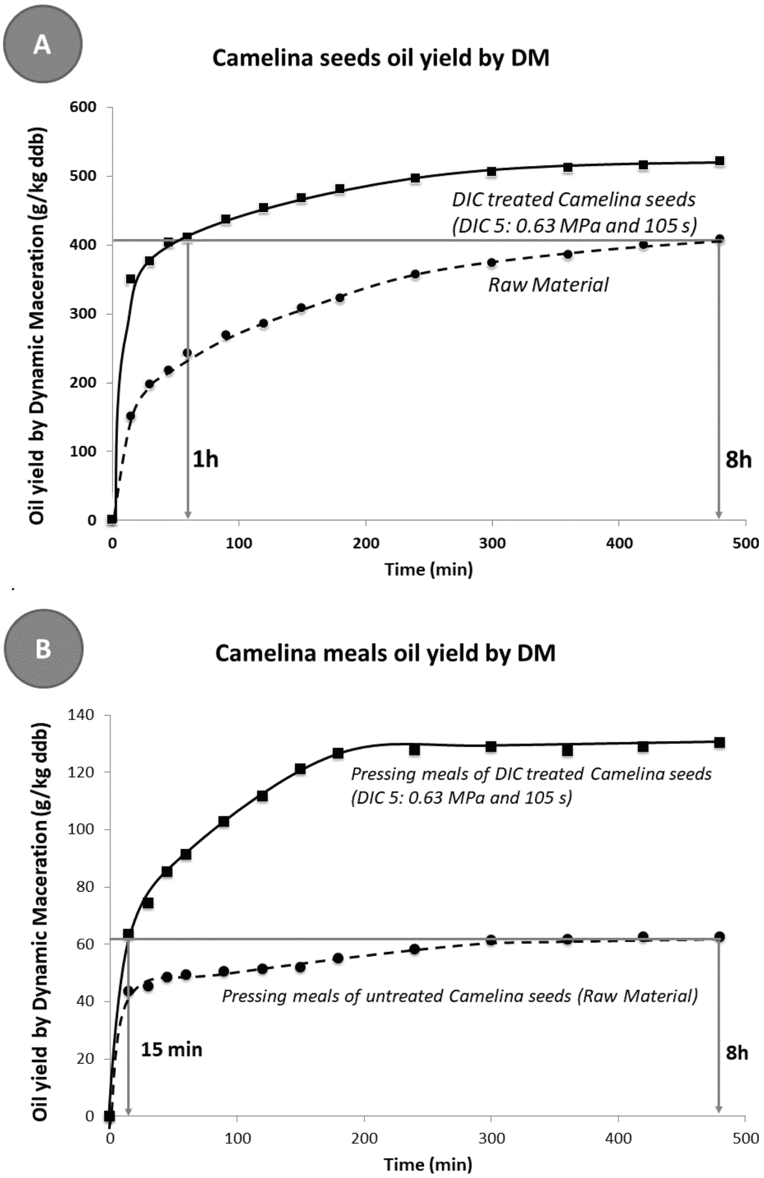

FIGURE 4. Extraction kinetics by dynamic maceration (DM) of camelina oil from RM and DIC-treated seeds (A) and meals (B).

the quadratic effect of this factor (t^2) had a significant positive effect on the pressing oil yield. The higher the pressure and the time, the higher the oil $Y_{ASE;\,meals}$. Table 3 and Figures 6A and 6B illustrate the impact of P (steam pressure) and t (treatment time) on oil $Y_{ASE;\,meals}$.

Equation 7 shows the regression model for the oil $Y_{ASE;\,meals}$, with R^2 of 96.8%:

$$Y_{ASE\,meals} = 130.7 + 1.1\mathrm{P} - 0.18\mathrm{t} + 4.1P^2 + 0.008\mathrm{Pt} + 0.0015t^2 \qquad \text{Eqn. 7}$$

P: Saturated steam pressure in MPa
t: Thermal treatment time in s

To optimize (maximize) the oil $Y_{pressing}$ of seeds, the optimal conditions of DIC treatment for this response were P =0.7 MPa and t = 120 s (134.15 g of oil/100 g dry-dry basis).

3.4.2. Dynamic maceration extraction of non-treated (RM) and treated (DIC) camelina meals

The average oil contents obtained after 2, 8 and 24 h of dynamic maceration of untreated camelina meals were 51.6, 63.5 and 110 g oil/kg ddb, respectively. In the case of DIC-treated meals, the best performance was shown by DIC 5 (0.63 MPa and 105 s). For this treatment, after 1, 2 and 8 h of DM, oil extraction oil yields were 91.3, 115.8 and 130 g oil/kg

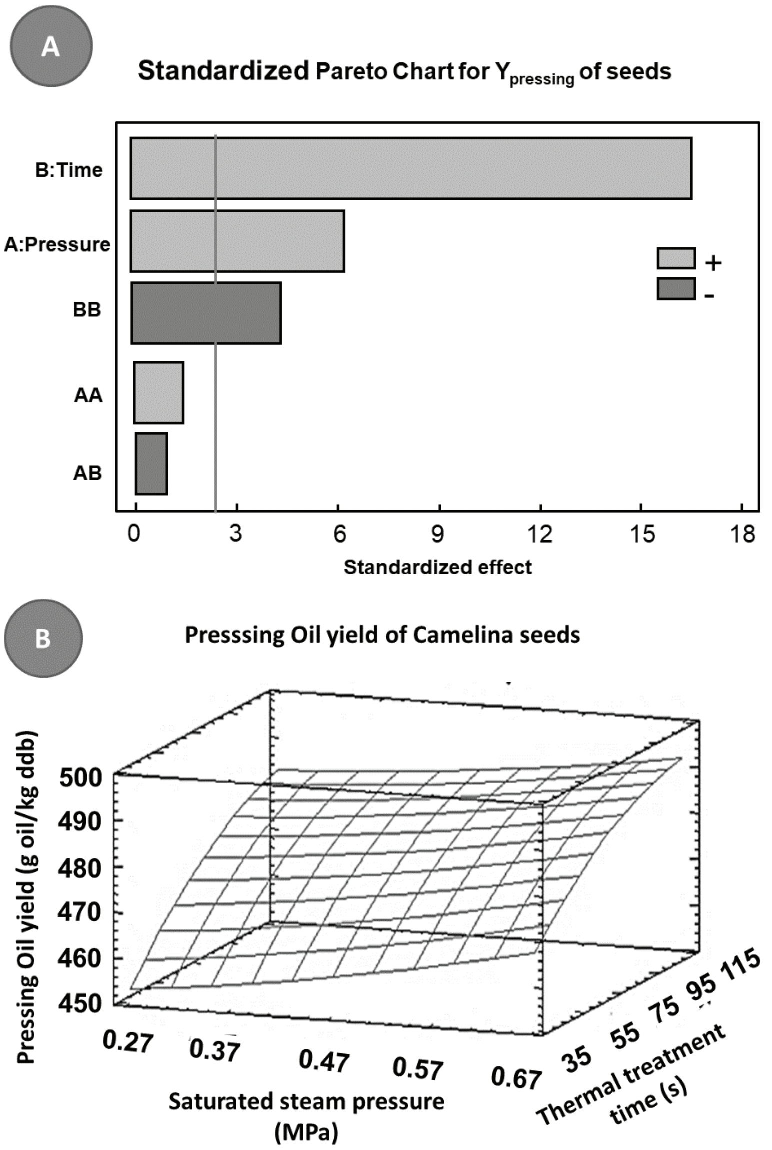

FIGURE 5. Effect of DIC parameters on the oil $Y_{pressing}$ from camelina seeds: A) Standardized Pareto Chart; B) Estimated Response Surface.

ddb, respectively. As it can be observed in Table 2, in only two hours all DIC-treated samples achieved the same oil yield as RM after 24 h. DM oil yields from DIC meals varied from 111.4 to 115.8 g oil/kg ddb. Figure 4B presents the extraction kinetics of meal oil by DM of RM and DIC 5 (0.63 MPa and 105 s).

To evaluate the effect of DIC parameters on the oil yield of meals after 2h of DM ($DM_{2h;\ meals}$), the ANOVA and the RSM was applied. The results showed that under the selected domain values, the pressure (P) and the quadratic effect of the time (t^2) had a significant positive effect on the pressing oil yield. The higher the pressure and the time, the higher the $DM_{2h;\ meals}$ oil $yield_s$. Table 3 and Figures 6C and 6D illustrate the impact of P (steam pressure) and t (treatment time) on oil $DM_{2h;\ meals}$.

Equation 8 shows the regression model for $DM_{2h;\ meals}$, with R^2 of 85.6%:

$$Y_{DM2h;\ meals} = 113.6 + 2.5\mathrm{P} - 0.06\mathrm{t} + 9.5P^2 + 0.007\mathrm{Pt} + 0.00053t^2 \qquad \text{Eqn. 8}$$

P: Saturated steam pressure in MPa

t: Thermal treatment time in s

To maximize the of Y_{DM2h} of meals, the optimal conditions of DIC treatment were P =0.7 MPa and t = 120 s (121.02 g of oil/100 g dry-dry basis).

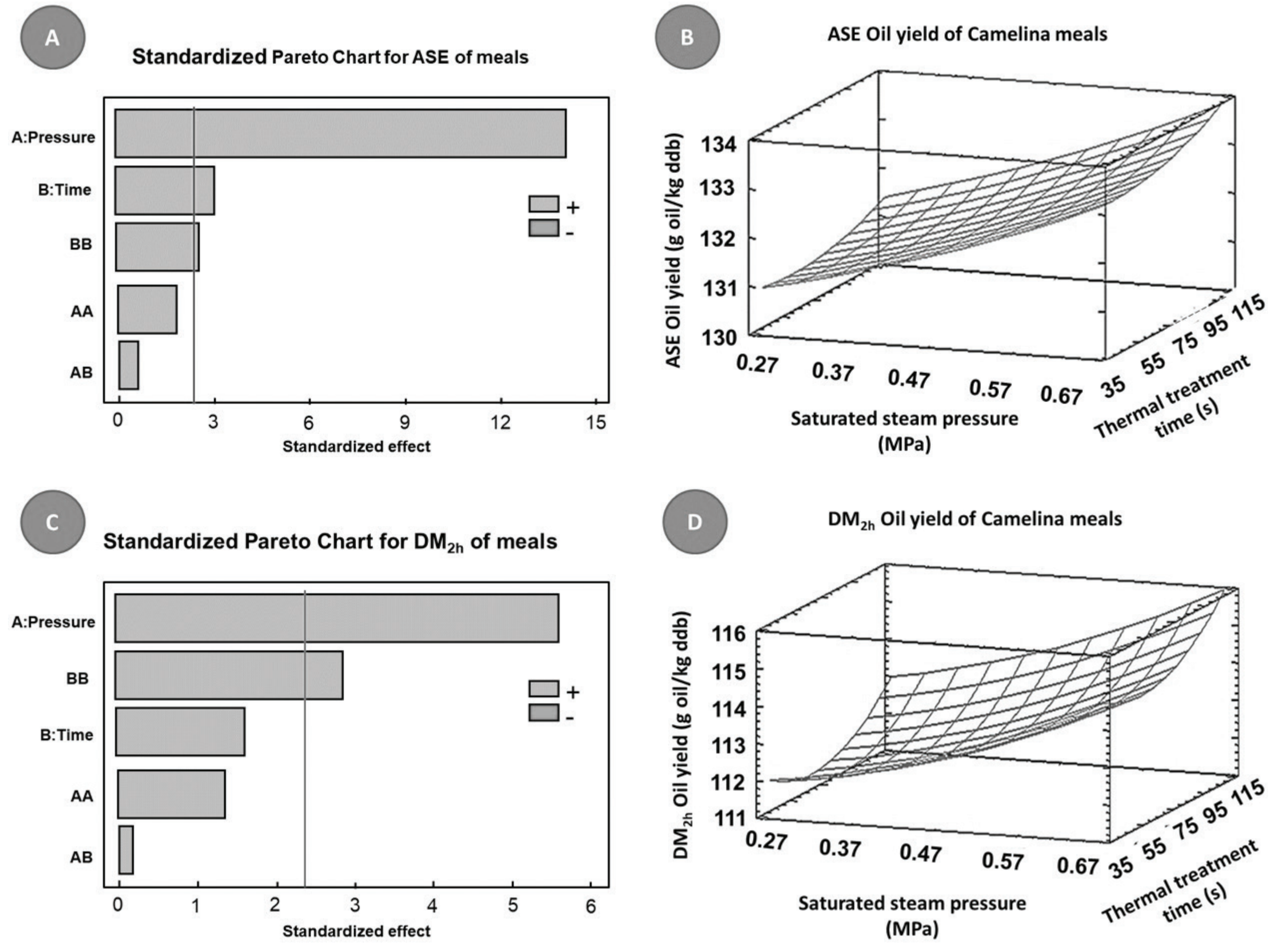

FIGURE 6. Effect of DIC parameters on the oil yield solvent extraction from camelina meals: A) Standardized Pareto Chart of ASE $_{\text{meals}}$; B) Estimated Response Surface of ASE $_{\text{meals}}$; C) Standardized Pareto Chart of $\text{DM}_{\text{2hmeals}}$ and D) Estimated Response Surface of $\text{DM}_{\text{2hmeals}}$.ASE: Accelerated Solvent Extraction and DM_{2h}: Dynamic Maceration 2h.

4. DISCUSSION

Thanks to its environmental adaptability, its satisfactory seed yields, and its multiple oil applications (i.e, biofuels, oleochemical compounds, animal feed, and food applications), *Camelina sativa* has attracted the attention of both research and industry (Zanetti *et al.*, 2017). Besides, as showed in this study, camelina seeds contains more than 50% oil, 555.5 g oil/kg ddb for ASE of RM, which increase its feasibility for use in several industries. Similar results were found by Moslavac *et al.*, (2014) after 8 h of Soxhlet extraction, where they determined 42.40 ± 0.73% of oil from RM camelina seeds. Then, to extract the total oil content of camelina seeds, a variety of different extraction methods were applied, being the most used techniques: a) mechanical pressing, b) solvent extraction, c) or a combination of both methods (Avram *et al.*, 2015; Moslavac *et al.*, 2014; Stroescu *et al.*, 2015). However, the main drawback of mechanical pressing is the high percent of residual oil in the cakes. Moreover, even if solvent extraction helped to recover the remaining oil from the cakes, long extraction periods would be needed to perform at appropriate efficiency.

Then, to make the extraction of camelina oil more affordable, it is therefore important to redefine industrial methods that allow for recovering the largest amount of this oil in the shortest time. In this study, DIC texturing pre-treatment was applied to camelina seeds before mechanical pressing and solvent extraction, and the results showed that this technology systematically enhanced the oil extraction (Table 2).

Camelina oil solvent extraction was studied through ASE and DM, the first one aimed to determine the total oil content of seeds and meals, and the second one to study the different transfer mechanisms during oil solvent extraction. In the case of ASE of seed oil, DIC treatment allowed to extract 10.8% (615.9 g oil/kg ddb) more than RM samples, which means that through ASE it was not feasible to obtain the total oil amount from the seeds. In fact, DIC treatment allowed the seeds to attain higher porosity which triggered the rupture of the oil-containing glands. Moreover, in figure 4B, it can be observed that the DIC treatment of seeds produced a clear improvement in the reduction of extraction time by DM, reaching (DIC 5: 410.5 g oil/kg ddb) a higher yield in 1 h than RM after 8 h of extraction (RM 8h: 405.2 g oil/kg ddb). According to Allaf and Allaf, (2013), most of the kinetics are highly dependent on the porosity and tortuosity of the material. Then, the structure modification of seeds generated by DIC enhanced the solute-in-solvent transfer within the holes of the solid matrix, which allowed to reduce the extraction period and to increase oil yield.

In the specific case of 2h of DM of seeds, DIC 5 presented 1.6 times (462.8 g oil/kg ddb) better oil yield than RM (284.8 g oil/kg ddb); which meant that thanks to the DIC treatment more than 80% of the total RM seed oil (555.5 g oil/kg ddb) could be obtained after 2 h of DM extraction. Furthermore, in both cases, ASE and DM, it was observed that the higher the pressure of the DIC treatment, the higher the improvement in the seed oil yield; the optimum experimental pressure value was 0.63 MPa.

Due to the fact that conventional solvent extraction methods represent 80% of the total processing time, 90% of the required energy, and more than 99% of the solvent used for the whole analysis procedure, pressing extraction has become an interesting solvent-free extraction technique to study and to ameliorate (Chemat *et al.*, 2015). In this work, seed pressing was carried in a single-screw pressing machine, and results showed that compared to the total RM seed oil, this technique allowed for the recovery of a little more than 80% of camelina seed oil in a few minutes, 444.7 g oil/kg ddb in the case of RM seeds and 490.9 g oil/kg ddb for DIC 5. Just the same as in solvent extraction, DIC treatment conditions allowed for the disruption of cell membranes and the cell walls of seeds, which increased the efficiency of pressing extraction though the new porous matrix.

Though pressing extraction allowed for obtaining good oil yields, at best there was around 20% of seed oil that remained in meals. For this reason, in order to attain total oil recovery, industries apply a second solvent extraction step (Uitterhaegen and Evon, 2017). In this study, to determine the residual oil in pressed meals, ASE was applied. The results showed that by comparing the total oil amount from RM seeds (555.5 g oil/kg ddb) to recovered meal oil, it was possible to recover 19.8% of the oil (110 g oil/kg ddb) in the case of RM, and up to 24% from DIC-treated meals (133.4 g oil/kg ddb). On the other hand, by looking at the results of DM from camelina meals, we could point out that after 2 h of extraction, DIC 5 treatment allowed for obtaining 20.8% oil (115.8 g oil/kg ddb), which represented 2.24 times more oil than from RM meals (51.6 g oil/kg ddb). It should be noted that even after 24 of DM for RM meals (110 g oil/kg ddb), DIC 5 showed the highest oil yield. Similar results were found by Moslavac *et al.*, (2014), who, after 8h of Soxhlet extraction of camelina pressed meals, obtained 15.7% of recovered oil. Furthermore, in all cases, it was observed that for pressed seeds and meals, the higher the pressure and the time of DIC treatment, the higher the improvement in oil yield. In both cases, optimum experimental values were found at P= 0.63 MPa and t= 105 s.

When comparing the performance of the different studied extraction methods, it can be concluded that by coupling DIC treatment (P: 0.63 MPa and t: 105 s) to pressing extraction followed by the DM of meals for 2 h it was possible to reach 606.7 g oil/kg ddb of oil yield, which meant that thanks to the DIC treatment it was possible to obtain 9.21% more than the initial total oil content recovered by ASE of camelina RM seeds (555.5 oil/kg ddb of oil yield). Moreover, by comparing these results to RM oil yield after pressing coupled to DM for 2 h (496.3 g oil/kg ddb), we could highlight an improvement of 22% in the final oil yield as a result of the DIC pre-treatment.

DIC is a convenient texturing pre-treatment to increase the oil yield from seeds, to reduce the oil extraction time, to ensure the final oil quality, to valorized pressing meals, and to increase the industrial processing capacities. Moreover, one of the main advantages of the DIC process is its ease of use at industrial level. In fact, the obtained optimal laboratory parameters could be scaled up without any problem. DIC reactors are currently operating at laboratory, pilot, and industrial scales. Nowadays, different DIC reactors are operating worldwide, e.g., in France, Spain, Italy, Mexico, Malaysia, and China.

5. CONCLUSIONS

Camelina sativa L. Crantz is a reemerging oilseed crop with a high oil content. This work focused on the enhancement of the oil extraction of *Camelina sativa* seeds and meals through the Instant Controlled Pressure Drop (DIC) texturing pre-treatment. Results showed that compared to RM, DIC pre-treatment coupled to solvent and pressing extraction increased the oil yields of camelina seeds and meals. In the case of solvent extraction, DIC pre-treatment coupled to ASE allowed for obtaining 10.8% more oil than from untreated camelina seeds. Furthermore, through coupling DM to DIC it was possible to reduce the extraction time of oil seeds from 8 h to only 1 h. On the other hand, compared to raw seeds, DIC coupled to pressing allowed for a 10.3% increase in oil yields. Additionally, DIC improved extraction from oil meals by recovering 2.2 times more oil than untreated meals after two hours of DM. The optimal experimental DIC treatment conditions were 0.63 MPa and 105 s. DIC pretreatment allowed for increasing camelina oil yields, reducing extraction time and valorizing pressing meals, which makes it the ideal process for camelina oil extraction.

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