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Abbreviation list:

DIC: Instant Controlled Pressure-Drop
CCD: Central Composite Design
RSM: Response Surface Methodology.
K: Matrix permeability coefficient (m^2)
ν: Kinematic viscosity (m^2 s^{-1})
A: Flux surface (m^2).
Pt: Total pressure.
HTST: High Temperature-Short Time
DoE: Design of Experiments
Y: Response
β_i, β_{ii}, and β_{ij}: Regression coefficients
X_i: Independent variables
ε: Random error
i and j: Factor indices.
HPLC: High Performance Liquid Chromatography
RIE: Ratio of Improvement of oil Extraction
Y_{press}: Cold-press oil extraction yield (g Oil/g ddb).
Y_{cake}: Residual cake oil (g Oil/g ddb).
$Y_{whole\ seeds}$: Total sunflower seed yield (g Oil/g ddb).
ddb: dry dry basis (mass basis of raw material devoid of water and oil).
db: dry basis (mass basis of raw material devoid of water).
P: Saturated dry steam pressure.
AOAC: AOAC International (Standards of Association of Official Analytical Chemists).
t: Heat treatment time.
R^2: Regression coefficient.
RM: Raw Material.
TMH: 2,3,5-trimethylhydroquinone.

Abstract

Sunflower oil industry is interested in both increasing yield and preserving quality of the extracted oil. The current work aimed to investigate the effect of texturing by instant controlled pressure-drop (DIC) on oil cold-press extraction. DIC texturing consists of heating at saturated dry-steam pressure P (200-700 kPa or 110-160 °C), for a short processing time t, followed by an instantaneous pressure-drop (20-40 ms) towards a vacuum (3-5 kPa). This resulted in increasing oil pressing-yield up to 46% and 33%, for linoleic and oleic varieties, respectively. It also increased the whole-oil availability by 11%, with a conservation of the biochemical composition of both oil and residual cake. A multi-criteria optimization of the DIC operating parameters; P and t, was performed through a 5-level Central Composite Design (CCD) and statistical analyses. This highlighted the direct significant effect of t on the oil yield while greatly preserving the quality of the final products.

Keywords: Sunflower Seeds; Instant Controlled Pressure-Drop (DIC); Cold-Press Extraction; oil quality; Central Composite Design (CCD), Response Surface Methodology (RSM).

1. Introduction

Sunflower is a famous oleaginous plant widespread on all continents due to its hardiness and low requirements for both inputs and water (Castro and Leite, 2018). Worldwide, its cultivation is one of the largest in the world in Americas (Canada, Brazil, Argentina, USA)(Heiser, 2008), Asia (China, India, Indonesia) and Europe in particular in France (Gunstone, 2009). The sunflower seed consists of a shell and a kernel. The shell represents 70-90% of the dry matter formed from lignin, hemicellulose, cellulose fiber and a small amount of lipids and proteins (Connor and Hall, 1997), while the kernel, with about 20% of the dry matter of the seed, is the storage place of reserve for embryonic development (Roche et al., 2004). Its lipid content is close to 44%, having a distribution of the main saturated fatty acids: palmitic acid (C16: 0) of 5%-7%, stearic acid (C18: 0) of <10%, and unsaturated: oleic acid (C18: 1) of 17%-20% and linoleic acid (C18: 2) of 50%-70% (Ayerdi Gotor et al., 2016).

Diversification of sunflower varieties results in differences in the levels of linoleic and oleic fatty acids. It generally meets the technological needs and requirements of the food and non-food industries. For the food sector, sunflower has beneficial effects on human health. It is a source of vitamin E, a dietary ingredient well-known for its anti-cholesterol properties (Pelletier et al., 1995). It is also used in margarine (Moreau et al., 2002), canning, salad dressings, table oil, and infant formula. Moreover, in the non-food sector, oleic sunflower oil is valued because of its content of fatty acids as well as the presence of minor compounds favorable to the manufacturing of pharmaceutical or cosmetic products, lubricants (Al Mahmud et al., 2013), cleaning products, solvents in the paintings, etc. It can be used for the production of both edible products and biofuel for diesel engines as a result of esterification often with the methyl ester (Rashid et al., 2008) (Del Gatto et al., 2015), (Ayerdi-Gotor et al., 200811-12).

As an edible product, and because of the negative health effects of solvent traces, it is preferable that the industrial extraction of sunflower oil is obtained by pressing. However, press-cake would generally have a high residual oil content of 15 to 20%, which is too high for livestock feed or as a biomaterial (Isobe et al., 1992). This point also has a significant economic impact due to the price difference between the oil and the residual solid cake. Since the consumer demands to avoid the use of solvent extraction, an increase in the extraction efficiency of the press becomes an essential objective in improving the performance of the industrial process.

From fundamental and phenomenological point of view (Bouallegue et al., 2015), press-extraction is based on the gradient of total-pressure as driving force. This similar Darcy-system can be presented as following:

$$\dot{m}_{oil} = -\frac{K}{\nu}\vec{A}.\overrightarrow{grad}(P_t) \quad \textit{Eq. 1}$$

Where K is the oil-matrix permeability coefficient expressed in m^2, ν the kinematic viscosity expressed in $m^2\ s^{-1}$, A the normal oil-flux surface expressed in m^2, and P_t the total pressure. A hydrostatic liquid behavior of the material, through for example high water content, induces a more homogeneous total pressure and, then, a too weak gradient of total pressure. Hence, the use of dried seeds is an inexorable condition in press-extraction operation (Bouallegue et al., 2016). Therefore, to increase the pressed oil yield, one has to spread the pressure gradient, reduce both viscosity and product thickness. In addition, heating is well known to be capable of inducing increased yields through some thermal degradation of the cell walls. However, this operation inevitably induces a certain degradation of the main molecules and ingredients, many users recommend not to insert it. Hence, the objective of increasing the oil extraction yield must be coalesced with a perfect preservation of quality, and specialists advise against the use of heating as pretreatment and opt for a cold-press method to preserve the quality of both extracted oil and cake (Boutin and Badens, 2009).

A specific intensification of the process can be obtained by texturing by Instant Controlled Pressure-Drop (DIC), which normally increases the value of the initial permeability coefficient through higher porosity and better tortuosity of the matrix. Moreover, DIC texturing may also result in breaking the cell-walls, thus allowing the oil availability to be higher. However, once the consolidation stage of pressing is reached, porosity disappears and only some tortuosity effect and, much more, the oil availability issued from broken cell-walls persist. However, the DIC technique proposed for texturing sunflower seeds may possibly affect the functional properties of sunflower seeds, while probably preserving the quality of the oil extracted and the residual solid cake (Van, 2010).

Indeed, the challenge of using instant pressure-controlled DIC coupled with cold press to well extract the sunflower seed oil must is worth to be studied to obtain the benefits of increasing oil yields and preserving the high quality of the oil (Ixtaina *et al.*, 2011), (Cường, 2013). Thus, the oil from DIC-assisted cold pressing extraction must be analyzed to prove that it has the same quality as conventional cold-pressed oil, in terms of biochemical composition, sensory characteristics, and color. The oil must meet to all international oil standards.

2. Materials and methods

2.1. Raw materials and chemicals

The sunflower seeds were supplied by the company "Presse de Gascogne" in Cologne (France). This company specializes in the field of organic French-origin sunflower food oil.

Two varieties of sunflower: linoleic and oleic types were used. They were transported to the laboratory and well packaged, and stored at room temperature. The initial moisture content dry basis (db) (W_i) was about 0.07 ± 0.005 g H_2O/g db for the linoleic sample and 0.09 ± 0.005 g H_2O/g db for the oleic sample. Our study on cold-pressing extraction has concerned the whole sunflower seeds, thus maintaining their hulls.

The chemicals such as 99.99% purity n-hexane solvent used for oil extraction were provided by the University of La Rochelle-France. While for fatty acid analysis, 2,3,5-trimethylhydroquinone (*TMH*) and iso-octane used to prepare the stock solution for the standards: α, β, and γ-Tocopherols for the calibration range of tocopherol analysis with acetone as solvent, were provided by the Institut Polytechnique UniLasalle Beauvais-(France), and kept at -40°C for chromatography analyses.

2.2. Treatment process and Experimental Protocol

The treatment protocol of oil press-extraction process was achieved at laboratory-scale (Figure 1). In the first part of our experimental study, sunflower seeds were subjected to an instant controlled pressure-drop (DIC) treatment aiming at greatly intensifying oil cold press-extraction.

Figure 1.

Some part of the experiments and assessments required a laboratory-scale n-hexane solvent extraction. Following the DIC texturing treatment and prior to the properly-said pressing, a drying step was performed. The sunflower seed samples underwent an airflow drying at 40 °C and 1 m/s, until reaching a final moisture content of 0.060 ± 0.005 g H_2O/g db. This level is well-known as the most appropriate moisture content for the cold press-extraction. For an assessment requiring a solvent extraction process, the seeds were ground using a laboratory knife mill (RETSCH, Grindomix model GM 200-F; Kurt Retsch GmbH & Co. KG, Haan, Germany) at 8000 rpm for 40 seconds.

2.2.1. DIC treatment

The DIC unit was presented in various articles (Figure 2) (Allaf and Allaf, 2014; Sabah Mounir et al., 2011; and Albitar et al., 2011). The research works carried out on instant controlled pressure-drop technology (DIC) at both fundamental levels and industrial applications have the specificity of a theoretical foundation based on the thermodynamics of instantaneity (Allaf et al., 2014): far from the equilibrium, processes occur between the two-asymptotic behaviors of quasi-static (classical thermodynamics) and instantaneous limits. Processes and transformations occurring in a theoretically zero time (transport of molecules in dozens of milliseconds), generally imply a decrease in the fluctuation degree of the random translational movement of the particles, at the same time an equally instantaneous decrease of the temperature. The relaxation time (Δt of dozens of ms) is an essential parameter in the considered processes; it implies a very high pressure-drop rate ΔP/Δt often greater than 5 10^5 Pa s^{-1}. The cooling rate can reach exceptional levels of 1500 to 2000 kW/m^2 of the product (Allaf et al., 2018b).

Figure 2.

DIC has been inserted as relevant process or effective intensification mean for many unit operations of decontamination, drying, extraction, chemical or enzymatic reactive transformations, etc. (Allaf et al., 2014) (Sabah Mounir et al., 2011) (Mounir et al., 2012)

(Berka-Zougali et al., 2010) (Albitar et al., 2011). Practical treatment with DIC involves a vacuum stage of 5 kPa, followed by a HTST (High Temperature-Short Time) type process generally using saturated dry steam absolute pressure (usually ranged between 0.05 and 1 MPa), i.e. temperature of 60 °C to 170 °C (Besombes et al., 2010). The short treatment time (some dozens of seconds) includes a steam injection step (lasting less than 3 s) involving vapor condensation on the product surface (moisture content may increase by ΔW of almost 0.1 g H_2O/g db, resulting in soaring heating coefficient up to tens of kW m^{-2} K^{-1}), followed by a homogenization step of both thermal and condensed water within the product. Succeeding this roughly balance step, the pressure abruptly drops>> towards the vacuum (steam tank maintained at 3 to 5 kPa) (Allaf and Allaf, 2013).

Instantaneous-type of coupled thermomechanical transformations of DIC is: i/ an autovaporization of a certain quantity of volatile molecules, mainly water to reach the new thermodynamic equilibrium; (the moisture content undergoes a decrease ΔW of approximately -0.11 g H_2O/g db), ii/ mechanical stresses within the material resulting in a new structure and texture, iii/ a cooling of the material possibly involving an overshoot beyond of the glass transition. Thus, the new often expanded structure of the matrix is preserved (Haddad et al., 2008), and iv/ while preserving a perfect stop of the thermal degradation.

Following a few preliminary tests, two operating parameters of DIC treatment of sunflower seeds, namely the dry saturated steam absolute pressure (P) ranged between 0.2 and 0.7 MPa, and the treatment time (t) ranged between 15 and 85 s were selected. DIC-texturing begins by introducing into the treatment vessel a quantity of 150 - 200 g of sunflower seeds at 7 ± 0.2% db (dry basis) water content. An initial vacuum makes it possible to greatly reduce the diffusion resistance between the exchange surface and the steam, thus inducing an improvement of the heat transfer.

Hence, after closing the pneumatic valve, the high-pressure steam is injected to reach the desired pressure level (and temperature). It is maintained throughout the treatment until reaching a level of thermal homogeneity and humidity within the material. An abrupt pressure-drop towards the vacuum results in an autovaporization of the water, an instantaneous cooling of the solid material and a possible more or less marked expansion of the product (Mounir et al., 2011). DIC-textured oleaginous seeds have better technological aptitude regarding cold-press extraction with higher oil yield. In order to evaluate and analyze the biochemical composition, DIC-textured seeds are often crushed before an extraction operation with n-hexane.

2.2.2. Extraction process: Mechanical cold-press extraction

The present research aims to study the variation of the yield of sunflower oil from the two oleic and linoleic varieties and to compare the results obtained from the treated and untreated matrices. The industrial procedure for extracting sunflower oil is based on the cold-mechanical way (press, cavitator, centrifuge systems…).

At this level, the extraction of oils is based on the mechanical action of press compression. The pressing operation allows the oleaginous seed to liberate the oily liquid contained in the kernel cells and separate it from the residual solid called “cake”.

In our laboratory-scale study, the cold-pressing device is based on single-screw extrusion which operates according to a continuous process (p500R, Anton Fries Mashinenbau GmbH, Germany). It operates at a frequency of 50 Hz, a voltage of 400 V and a power of 1.5 kW. This device comprises a stainless-steel cylindrical hopper of 35 mm diameter. In the feeding zone, the seeds are pressed and freely transported under the effect of the mono-screw towards the bottom of a fixed tube to create a high-pressure cold-compression. This fixed tube contains holes along its length through which the oil is recovered at the bottom of the press by a flow through calibrated 8 mm hole called die or nozzle, positioned at cylinder end to remove the cakes in granular forms. This separation is done in an area called the filtration zone.

Following filtration, the extracted oil was measured while the cake was stored in poly-bag at a temperature of 4 °C. The oil yield is expressed in g oil/g ddb (dry dry matter, which means a mass basis of raw material rid of water and oil) with an error estimated at 0.05%. Cold-press oil extraction was successively performed on the raw-material (untreated) and DIC-textured sunflower seeds after adjusting the moisture content of each sample to 0.06 ± 0.005 g H_2O/g db.

$$Y_{huile}(g\ oil/g\ ddb) = \frac{oil\ mass}{dry\ dry\ seed\ mass\ (ddb)} \qquad \text{Eq. 2}$$

2.2.3. Design of Experiments DoE

2.2.3.1. Experimental Protocol

From literature and previous experience of our research team, succeeding few preliminary tests, a Design of Experiments DoE was defined. In the current case, since our objective was not a simple approach of operational trends, but especially to carry out a relevant optimization study of the main operating parameters of the new industrial treatment DIC operation, we opted for a 5-level 2-parameter Box-Wilson Central Composite Design (CCD). For DIC-texturing, the two operating parameters of the absolute value of saturated dry steam pressure (P) and the treatment time (t) were ranged from 0.2 to 0.7 MPa, and from 15 to 85 s, respectively.

The experimental responses were statistically processed using Statgraphics plus software (Statgraphics Centurion XV, StatPoint Technologies, Inc., Rockville, USA). This Response Surface Methodology (RSM) allows highlighting for each dependent variable (Y) the significance level of the DIC factors as independent variables x_i (P and t) through ANOVA, p-value ($P \leq 0.05$), and Pareto Charts, as well as the general trends, response surfaces, and iso-response curves, issued from the empirical second-order polynomial model Y versus the independent variables x_i, and R^2 whose value reveals the degree of validity of this empirical model:

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

Where Y is the response, β_i, β_{ii}, and β_{ij} are the regression coefficients, x_i the independent variables, ε is a random error, i and j are the factor indices.

2.3. Assessment methods and processes

2.3.1. Measurement of moisture content

The water content was determined according to the AOAC method (2005) at 105 °C for 24 hours until a constant weight is obtained.

2.3.2. Oil extraction by Randall

Usually, solvent extraction of both whole seed and residual cake (before and after the cold-press extraction, respectively), is carried out as an initial assessment stage to measure the composition of plant-base materials. Frequently, oil solvent extraction system used is “Randall's SER 148/3 model equipment from Velp Scientifica” (Italy). This is a closed loop cycle of oil solvent extraction procedure; i/ starting by **bringing to boil** solvent in container using a hot plate, ii/ following by solvent **vapor condensation** through an adequate cooling system, in the same container to wash the product placed in porous cartridge in this container (**reflux washing** where the solvent regenerated by condensation, flows and washes the sample contained in the cartridge), iv/ ending by a “**recovery phase**" during which the majority of the solvent is removed and the extracted oil remains in the containers with traces of solvent. Finally, under Sorbonne, the contents are deposited in Pyrex tubes to evaporate and remove the remainder traces of solvent from the evaporator.

In the present case of sunflower seeds, various differently processed, un-textured and DIC-textured seeds, and cakes were studied. Before starting the properly said operation, Velp 148/3 parameters were checked out and corrected for compatibility. In the current case, since the solvent used was n-hexane, and the plate temperature was 180 °C, Viton seals were used. A sample of 5 to 7 g of crushed sunflower was placed in the porous cartridge to receive, by washing, almost 40 ml n-hexane. This operation was carried out in 6 h. For removing the remainder traces of n-hexane solvent from the evaporator, the cartridge contents were deposited in 18 ml Pyrex tubes and put under Sorbonne with airflow heated at 65 °C for 1 h. This extraction operation is faster, ampler, and more effective than a simple maceration extraction. The extracts were weighed using an analytical high-precision balance (with an accuracy of 0.001 g) and then stored at -4 °C for biochemical analyses. Oil yields were calculated with an estimated error of ± 0.0005 g oil/g ddb.

2.3.3. Determination of the free fatty acids

Fatty acid separation was performed using the Agilent 19091S-43 GC Gas Chromatography method (Kyoto, Japan). It is a qualitative and quantitative analysis method that serves to separate a very complex mixture of gaseous samples.

The chromatography device comprised a HP-5MS (5% Phenyl Methyl Siloxane) capillary type column (30 m × 350 µm × 0.25 µm), a furnace with initial temperature ranged from 155 to 230 °C with a speed of 45 °C/min. The final phase reached a maximum level of 240 °C and the oven run time was 10 min for each sample.

The protocol of the fatty acid analysis was to consider for each sample 15 µl of sunflower oil mix with 20 µl of the TMH, and 800 µl of the isooctane and, after stirring for a few seconds in a vortex, it was ready for analysis. A volume 0.8 µl of the assembly was eluted and injected using a syringe in split mode and the temperature of the injector was maintained at 270 °C. Once vaporized by the injector, the compounds were entrained in the column by hydrogen as carrier gas. Peak integration and identification of fatty acids were based on the use of the mass spectral MS database of the NIST'98 National Library (National Institute of Standards and Technology of the United States (NIST), Gaithersburg, MD, USA).

2.3.4. Determination of the protein content

The determination of the protein content was based on the classical DUMAS method carried out by a nitrogen/protein analyzer LECO FP-528, which consists of a total combustion of 100 mg of homogeneous crushed dried sunflower seeds (24-h oven at 110°C). A double repetition was required for analysis for each sample.

DUMAS method, which is performed under oxygen and at high temperature does not directly measure the protein content. This allows quantification of the total nitrogen that is expressed as a percentage and using an internationally recognized conversion factor of 6.25 which differs from protein to protein depending on its amino acid composition, equivalent to 0.16g of nitrogen per gram of protein is required to convert the measured nitrogen content to a protein content. It's an easy and fast analysis.

2.3.5. Determination of tocopherol content

The determination of the tocopherol content of the sunflower oils was performed using High Performance Liquid Chromatography HPLC (SpectraPhysics, Thermo Separation Products, USA) in 40 min. The multi-λ French standard measurement ISO 9936, 1997 implies a complete separation of the 4 forms of tocopherol α, β, γ, and δ using a fluorescence detector with 298 nm as excitation wavelength, and 344 nm as emission wavelength (Velasco and Dobarganes, 2002).

The protocol of the tocopherol analysis was to consider a sample of 1 g of sunflower oil, and add 1 ml of acetone in a Vial; then the whole was vortexed a few seconds and directly injected into the HPLC system for analysis. The sum of the concentrations α-, β-, γ-, and δ-tocopherol is used to determine the total tocopherol expressed in µg/g oil.

In the current case, the mobile phase was composed of a mixture (75:25 v/v) of acetone and methanol with a flow rate of 1 cm^3/min, provided by the HPLC Delta Chrom SDS 030 pump. The tocopherols were separated by the reversed phase C18 column at 30°C, 250 mm * 4.6 mm, and 5 µm as particle size (Macherey-Nagel Ltd., Düren, Germany). Quantification of the results was evaluated and integrated by comparison with the retention time collected with those of the respective standard surfaces using the chromatographic station software CSW 32 (Data Apex Ltd., Prague, Czech Republic).

3. Results and Discussions

3.1. Effect of DIC-texturing on cold-press extraction

In the current study, since the yields are expressed in terms of g Oil/g ddb (dry dry basis), we estimated the cold-press extraction yield by subtracting the oil content of the residual cake oil content $\mathbf{Y_{cake}}$ from the initial sunflower seed $\mathbf{Y_{whole\text{-}seed}}$, both obtained by performing solvent extraction (n-hexane) using the Randall velp-148 for 6 h:

$$Y_{press} = Y_{whole-seeds} - Y_{cake} \qquad \textit{Eq. 4}$$

To better recognize the comparative aspect of DIC processing in terms of yield enhancement, we used the ratio of improvement of oil extraction (RIE) expressed as a percentage, between DIC-textured and raw-material seeds:

$$RIE = \frac{Y_{randall;DIC}}{Y_{randall;RM}} \qquad \textit{Eq. 5}$$

Table 1.

Table 1 and Figure 3 show the impact of DIC-texturing under different treatment conditions on cold-press oil yields expressed in (g oil/g ddb dry dry basis) of linoleic and oleic sunflower seeds. Thus, the whole DIC-textured linoleic seeds had an increased oil content $Y_{whole\text{-}seeds}$ from 0.6519 g oil/g ddb for raw non-textured seeds up to 0.7208 g oil/g ddb, which means RIE of 111% RIE. Similar behavior was observed for oleic seeds; the increasing of the oil content of DIC-textured seeds was from 0.6639 g oil/g ddb for the raw non-textured seeds up to 0.7363 g oil/g ddb (or RIE=111%) of the whole DIC-textured oleic seeds. This should be due to an increasing of availability resulting from the higher porosity and possibly the broken cell walls by DIC-texturing triggered on the matrix.

Figure 3.

For both technologic and economic reasons, industries require reaching the highest cold-press extraction yields $\mathbf{Y_{press}}$. DIC-texturing at 0.2 MPa for 50 s resulted in dramatically increasing of the cold-press yield of linoleic of 146% RIE, which means 0.3462 against 0.5060 g oil/g ddb for non-treated seeds. Similar results were obtained for oleic seeds DIC-treated at 0.5 MPa for 50 s, with RIE=132% and cold press yield of 0.5206 against 0.3950 g oil/g ddb for non-treated seeds. It is worth highlighting that DIC-texturing pretreatment allows increasing the cold press yields respect to the whole seed yields by about 20%, from 53.11% to 73.56% for linoleic variety and 12%, from 59.50% to 71.64%, for oleic variety (Table 1).

Moreover, the effects of operating parameters of DIC treatment such as saturated vapor pressure (P) and heat treatment time (t) on the yields of oil, and the contents in the cake and in the whole seeds ($\mathbf{Y_{press}}$; $\mathbf{Y_{cake}}$; $\mathbf{Y_{whole\text{-}seeds}}$) were statistically analyzed by the statgraphic software (Figure 3).

Table 2.

Table 3.

It is worth noticing that the standardized Pareto Chart (Figure 3.A) shows a significant effect of saturated dry steam pressure (P) and treatment time (t) on the cold-press oil yield $\mathbf{Y_{press}}$, residual oil content $\mathbf{Y_{cake}}$, and oil content in whole seeds $\mathbf{Y_{whole\text{-}seeds}}$ with p value of 0.05. The **mean absolute error** (MAE) is computed as the sum of the squared error values and is the most commonly used **lack-of-fit** indicator in statistical fitting procedures.

Thus, the empirical second-order regression models of $\mathbf{Y_{press}}$, $\mathbf{Y_{cake}}$, and $\mathbf{Y_{whole\text{-}seeds}}$ versus the DIC processing parameters were computed. For linoleic variety, they had regression coefficients R^2 of 83.66%, 87.78%, and 90.62%, respectively, and MAE of 0.01395, 0.01313, and 0.00308, respectively (Table2). Similar study was performed in oil extraction from the oleic variety. They had regression coefficients R^2 of 69.62%, 67.02% and 91.14%, respectively, and the MAE were 0.03575, 0.03692, and 0.00395, respectively (Table 3).

3.2. Impact of DIC on the quality of the final sunflower seed cake

Despite the great positive incidence of DIC-texturing on technological aptitudes of cold-oil pressing, it was noteworthy to study the impact of such DIC-texturing on the quality of both extracted oil and cake of sunflower. The specificity of great enhancement of oil yields by DIC-texturing would not be so interesting without the perfect preservation of the main compounds and active molecules of sunflower seeds. Indeed, the conventional heating of oilseeds often leads to an increase in oil yields, but it also causes a great deterioration in the quality of both extracted oil and cake. As HTST (high temperature/short time) treatment, DIC often allows the concerned material to preserve its quality. Therefore, it was essential to carry out a detailed study of the impact of DIC-texturing on the composition of cake in proteins, and the oil in fatty acids and the availability of tocopherol.

3.2.1. Evolution of protein content of the sunflower whole seeds and press-cake vs DIC treatment conditions.

Table 4.

Figure 4.

The analyses of the total proteins were performed by the classical DUMAS method in the cases of the whole sunflower seeds and cakes at different conditions of DIC treatments, in comparison with the untreated seeds for both linoleic and oleic varieties.

Table 4 illustrates the results of this study and shows that the total protein content had no (or weak) change between the DIC treated and untreated samples; Figure 4 expresses a significantly absence of any effect.

3.2.2. Evolution of fatty acid contents of sunflower oil versus DIC treatment conditions

The composition on saturated and unsaturated fatty acids of the whole sunflower seeds and meal, mainly includes palmitic acid (C16:0), stearic acid (C18:0), oleic acid (C18:1), and linoleic acid (C18:2) of the two linoleic and oleic varieties are presented in Table 5.

The GC (Gas Chromatography) analyses proves that, whatever the processing conditions were, DIC texturing triggered no significant change in the fatty acid composition compared to the non-textured (RM) seeds. On the other hand, this observation was confirmed by the statistical analyses through Pareto Charts which reveal the analysis of the variance (ANOVA). Figure 5 highlights that, whatever the fatty acid compounds were, there was a significantly no effect for both whole seeds and cake of the two linoleic and oleic varieties.

Table 5.

Figure 5.

3.2.3. Tocopherol content of sunflower oil versus DIC treatment conditions

Table 6 presents the contents of total tocopherol and its forms (β, γ and α) of sunflower seed oils of the two linoleic and oleic varieties. These values were assessed by HPLC high performance liquid chromatography, for differently DIC textured and non-textured (RM) processing conditions.

The weak variation in tocopherol contents in both seed oil and cake, indicates that there was no change between the DIC-textured and non-textured samples. Moreover, the statistical analyses represented by Pareto Charts show a significantly no effect of the DIC parameters of the two linoleic and oleic varieties. This confirms that there was no degradation of the tocopherol contents (Figure 6).

Table 6.

Figure 6.

4. Conclusion

The great interest of industry manufacturing the sunflower oil in increasing the yields while preserving the quality of both oil and cake, as well as defining a new deodorization process was the basis of our investigation on the effect of texturing by instant controlled pressure-drop (DIC) on the press extraction of sunflower oil, respectively. DIC-texturing has allowed great impact on cold-press oil yields, reaching an increasing press-oil yields by about 46% and 32%, for linoleic and oleic varieties, respectively. Since DIC is a HTST (High Temperature/Short Time) operation, it normally results in the conservation of the biochemical composition of the material and a total preservation of the biochemical composition of both final products; oil and cake. The multi-criteria optimization of the DIC operating parameters, namely the saturated dry steam pressure (P) and the processing time (t), using a 5-level Central Composite Design (CCD) of Experimental Design (DoE) and prominently statistical analyses of the experimental results, highlighted the direct significant effect of the operating parameters P and t on the DIC performance. The highest yield of cold-press oil extraction has corresponded to 50 s of treatment time, with 0.2 and 0.5 MPa as saturated dry steam pressure, for linoleic and oleic varieties, respectively.

Thus, the Instant Controlled Pressure-Drop DIC texturation as a means of possible modification of the raw material structure appears getting great impact on increasing yields of the cold-press extraction of oil, on preserving the quality of both oil and residual cake with no change between the DIC textured and non-textured samples.

5. Reference

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Table captions

Table 1. ***Cold-press*** *Y_{press}**, solvent extracted Residual cake oil*** *Y_{cake}, and total* ***whole-seed oil yields*** *$Y_{whole\text{-}seeds}$, expressed in g Oil/g db (dry basis: material mass devoid from water content) and g Oil/g ddb (dry dry basis: material mass devoid from water and oil contents) of the linoleic and oleic variety for untreated and differently DIC-treated sunflower seeds.*

Table 2. The empirical models retained from the second-order regression of linoleic variety

Table 3. The empirical models retained from the second-order regression of oleic variety

Table 4. Protein contents of whole seeds and cake of sunflower versus different DIC treatment conditions.

Table 5. Saturated and unsaturated fatty acid amount of the whole seed and of the cake of sunflower for Linoleic and oleic variety of different DIC treatment conditions

Table 6. Tocopherol contents of the sunflower oil for the Linoleic and Oleic varieties of different Instant Controlled Pressure-Drop DIC treatment conditions.

Figure captions

Figure 1. Applied protocol of sunflower seed

Figure 2. Instant controlled pressure-drop (DIC) laboratory-scale unit.

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Figure 6. Standardized Pareto Charts for Tocopherol contents of whole sunflower seeds for linoleic and oleic variety versus Instant Controlled Pressure-Drop DIC processing parameters P (saturated dry steam) and t (processing time).

Table 1.

Linoleic variety			Y_{press}			Y_{cake}			$Y_{whole\ seeds}$			$Y_{press}/Y_{whole\text{-}seeds}$
Run no	P(MPa)	t (s)	g Oil/g db	g Oil/g ddb	RIE%	g Oil/g db	g Oil/g ddb	RIE%	g Oil/g db	g Oil/g ddb	RIE%	
RM	-	-	0.2096	0.3462	100%	0.2341	0.3057	100%	0.3946	0.6519	100%	53.11%
DIC 1	0.5	50	0.2147	0.3649	105%	0.2508	0.3348	110%	0.4117	0.6997	107%	52.16%
DIC 2	0.7	50	0.2527	0.4317	125%	0.2167	0.2767	91%	0.4146	0.7084	109%	60.94%
DIC 3	0.5	85	0.2382	0.4057	117%	0.2292	0.2974	97%	0.4128	0.7030	108%	57.70%
DIC 4	0.5	50	0.2002	0.3418	99%	0.2676	0.3654	120%	0.4142	0.7071	108%	48.33%
DIC 5	0.6	74.7	0.2488	0.4281	124%	0.2264	0.2927	96%	0.4189	0.7208	111%	59.40%
DIC 6	0.6	25.3	0.2125	0.3571	103%	0.2443	0.3233	106%	0.4049	0.6804	104%	52.48%
DIC 7	0.5	50	0.2632	0.4471	129%	0.2011	0.2517	82%	0.4114	0.6989	107%	63.98%
DIC 8	0.3	25.3	0.2689	0.4522	131%	0.1866	0.2294	75%	0.4053	0.6816	105%	66.34%
DIC 9	0.3	74.7	0.2622	0.4411	127%	0.1944	0.2413	79%	0.4056	0.6824	105%	64.64%
DIC 10	0.5	50	0.2572	0.4346	126%	0.2033	0.2552	83%	0.4082	0.6898	106%	63.01%
DIC 11	0.2	50	0.2998	0.5060	146%	0.1539	0.1819	60%	0.4076	0.6879	106%	73.56%
DIC 12	0.5	15	0.2733	0.4569	132%	0.1769	0.2149	70%	0.4019	0.6718	103%	68.01%
DIC 13	0.5	50	0.2564	0.4342	125%	0.2058	0.2591	85%	0.4094	0.6933	106%	62.62%

Oleic variety			Y_{press}			Y_{cake}			$Y_{whole\text{-}seeds}$			$Y_{press}/Y_{whole\text{-}seeds}$
Run no	P(MPa)	t (s)	g Oil/g db	g Oil/g ddb	RIE%	g Oil/g db	g Oil/g ddb	RIE%	g Oil/g db	g Oil/g ddb	RIE%	
RM	-	-	0.2374	0.395005	100%	0.2119	0.2688745	100%	0.3990	0.6638795	100%	59.50%
DIC 1	0.5	50	0.2436	0.4175983	106%	0.2288	0.2966805	110%	0.4167	0.7142788	108%	58.46%
DIC 2	0.7	50	0.1266	0.2183322	55%	0.3361	0.5062509	188%	0.4201	0.7245832	109%	30.13%
DIC 3	0.5	85	0.2989	0.5189654	131%	0.1785	0.2172855	81%	0.4240	0.7362508	111%	70.49%
DIC 4	0.5	50	0.3015	0.5206118	132%	0.1709	0.2061271	77%	0.4209	0.7267389	109%	71.64%
DIC 5	0.6	75	0.2908	0.5028691	127%	0.1846	0.226392	84%	0.4217	0.7292611	110%	68.96%
DIC 6	0.6	25	0.2683	0.4539809	115%	0.1923	0.2380834	89%	0.4090	0.6920643	104%	65.60%
DIC 7	0.5	50	0.265	0.454888	115%	0.2074	0.2616705	97%	0.4174	0.7165584	108%	63.48%
DIC 8	0.3	25	0.2668	0.4530433	115%	0.1968	0.2450199	91%	0.4111	0.6980632	105%	64.90%
DIC 9	0.3	75	0.2802	0.4789306	121%	0.1872	0.230315	86%	0.4149	0.7092456	107%	67.53%
DIC 10	0.5	50	0.2814	0.4840479	123%	0.191	0.2360939	88%	0.4187	0.7201419	108%	67.22%
DIC 11	0.2	50	0.2649	0.4468739	113%	0.1936	0.2400794	89%	0.4072	0.6869533	103%	65.05%
DIC 12	0.5	15	0.2648	0.4484239	114%	0.1968	0.2450199	91%	0.4095	0.6934439	104%	64.67%
DIC 13	0.5	50	0.2865	0.4932339	125%	0.1859	0.2283503	85%	0.4191	0.7215842	109%	68.35%

Table 2. The empirical models retained from the second-order regression of linoleic variety

$Y_{press} = 1.063 - 1.918p - 0.0078t + 1.744p^2 + 0.00384pt + 0.000059t^2$	R^2=83.658%
$Y_{cake} = -0.39267 + 1.884p + 0.00830t - 1.79356p^2 - 0.00127pt - 0.0000712t^2$	R^2=87.784%
$Y_{whole-seeds} = 0.7011 - 0.1225p + 0.00005t + 0.04908p^2 + 0.002562Pt - 0.0000084t^2$	R^2=90.623%

Table3. The empirical models retained from the second-order regression of oleic variety

$\boldsymbol{Y_{press} = 0.1728 + 1.825p - 0.00201t - 2.412p^2 + 0.00173pt + 0.000021t^2}$	***R^2=69.617%***
$\boldsymbol{Y_{cake} = 0.478833 - 1.67217p + 0.00226992t + 2.20435p^2 + 0.0000565pt - 0.000026t^2}$	***R^2=67.018%***
$\boldsymbol{Y_{whole-seeds} = 0.65164 + 0.1524p + 0.0002576t - 0.2079p^2 + 0.00179Pt - 0.0000056t^2}$	***R^2=91.137%***

Table 4. Protein contents of whole seeds and cake of sunflower versus different DIC treatment conditions.

run n°.	linoleic variety	
	whole seeds	sunflower cake
DIC 1	17.87	22.91
DIC 2	18.11	22.56
DIC 3	17.31	22.22
DIC 4	17.71	23.36
DIC 5	17.61	23.65
DIC 6	18.01	22.53
DIC 7	18.51	23.81
DIC 8	20.47	25.91
DIC 9	17.55	25.46
DIC 10	18.80	25.03
DIC 11	17.77	25.66
DIC 12	17.67	25.91
DIC 13	17.71	23.22
<RM>	18.06 ± 0.80	25.59 ± 0.66
<DIC>	18.08 ± 0.82	24.02 ± 1.38

<RM>: Average of Raw Material samples

<DIC>: Average of DIC treatments

Table 5. Saturated and unsaturated fatty acid amount of the whole seed and of the cake of sunflower for Linoleic and oleic variety of different DIC treatment conditions

	Linoleic variety							
	whole seed of sunflower				cake of sunflower			
Trial n°	C16:0	C18:0	C18:1	C18:2	C16:0	C18:0	C18:1	C18:2
DIC 1	6.79	4.24	23.08	64.12	6.68	4.29	25.89	62.49
DIC 2	6.65	4.17	22.91	64.69	6.78	4.33	24.13	64.11
DIC 3	6.71	4.27	23.54	63.77	6.88	4.38	24.37	63.72
DIC 4	6.60	4.14	23.84	63.68	6.83	4.47	24.71	62.99
DIC 5	6.58	4.27	23.25	64.09	6.83	4.47	24.77	63.26
DIC 6	6.51	4.17	23.25	64.34	6.83	4.43	24.38	62.97
DIC 7	6.45	4.21	23.47	63.98	6.89	4.54	25.14	62.37
DIC 8	6.44	4.18	23.28	63.98	6.88	5.00	24.53	63.09
DIC 9	6.49	4.18	23.68	63.74	6.95	4.52	24.63	63.22
DIC 10	6.48	4.11	23.10	64.43	6.96	4.66	25.12	62.20
DIC 11	6.52	4.25	23.55	63.97	7.12	4.57	24.70	62.92
DIC 12	6.65	4.38	24.04	63.29	6.96	4.80	25.18	61.95
DIC 13	6.56	4.16	23.70	63.78	6.96	4.74	25.45	61.76
<RM>	6.75 ±0.02	4.354 ±0.06	24.14 ±0.16	63.28 ±0.17	6.73 ±0.07	4.41 ±0.24	24.23 ±0.71	63.75 ±1.34
<DIC>	6.57 ±0.11	4.209 ±0.07	23.45 ±0.33	63.99 ±0.36	6.89 ±0.11	4.56 ±0.15	24.85 ±0.49	62.85 ±0.68

<RM>: Average of Raw Material samples

<DIC>: Average of DIC treatments

Table 6. Tocopherol contents of the sunflower oil for the Linoleic and Oleic varieties of different Instant Controlled Pressure-Drop DIC treatment conditions.

	Linoleic variety			
Trial n°	β	γ	α	TTP
	(µg/g oil)			
DIC-1	11.64	12.86	166.74	191.24
DIC-2	12.16	13.97	216.98	243.1
DIC-3	10.63	13.69	188.26	212.59
DIC-4	9.93	12.12	166.74	188.79
DIC-5	10.05	11.93	156.86	178.84
DIC-6	12.68	13.86	174.37	200.91
DIC-7	8.87	12.4	175.62	196.9
DIC-8	10.96	14.22	201.29	226.46
DIC-9	9.12	11.95	162.08	183.15
DIC-10	9.66	14.86	224.01	248.54
DIC-11	0	14.6	154	168.6
DIC-12	9.03	13.76	180.42	203.22
DIC-13	9.16	13.07	166.12	188.35
Optimum	9.76	13.13	165.68	188.58
<RM>	12.8±3.8	14.3±0.7	195.4±30.5	222.6±26.1
<DIC>	9.5±3.1	13.3±1.0	179.5±22.3	202.4±24.3

<RM>: Average of Raw Material samples

<DIC>: Average of DIC treatments

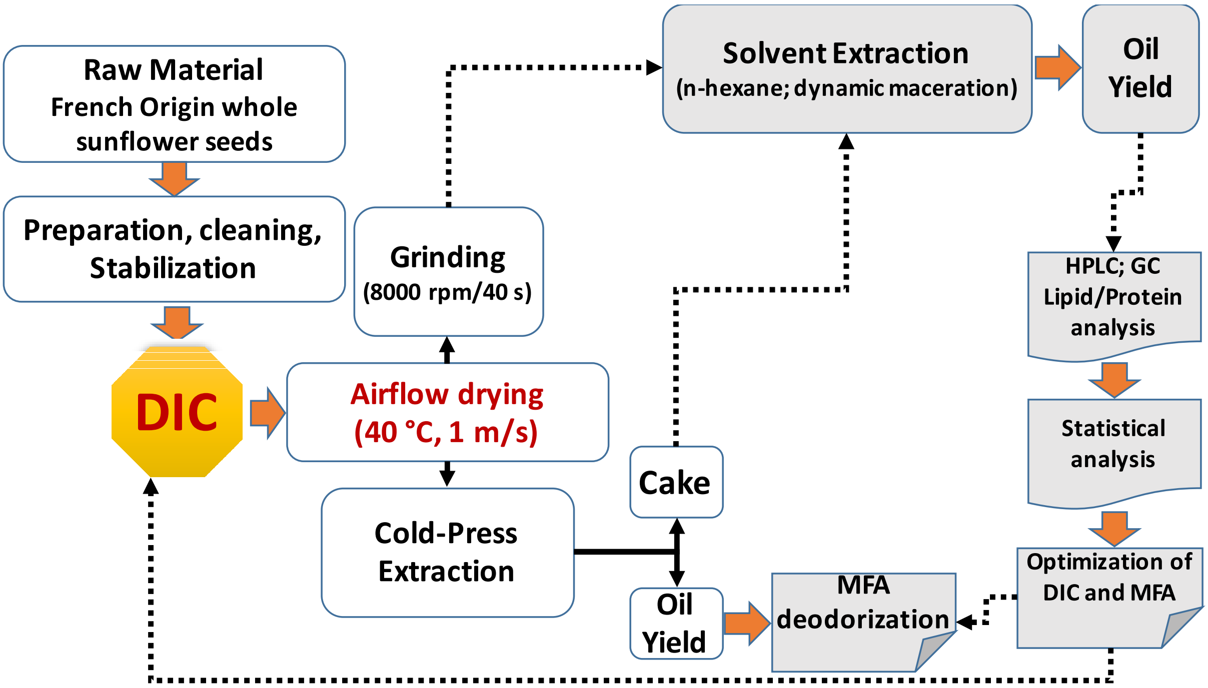

Figure 1. Applied protocol of sunflower seed

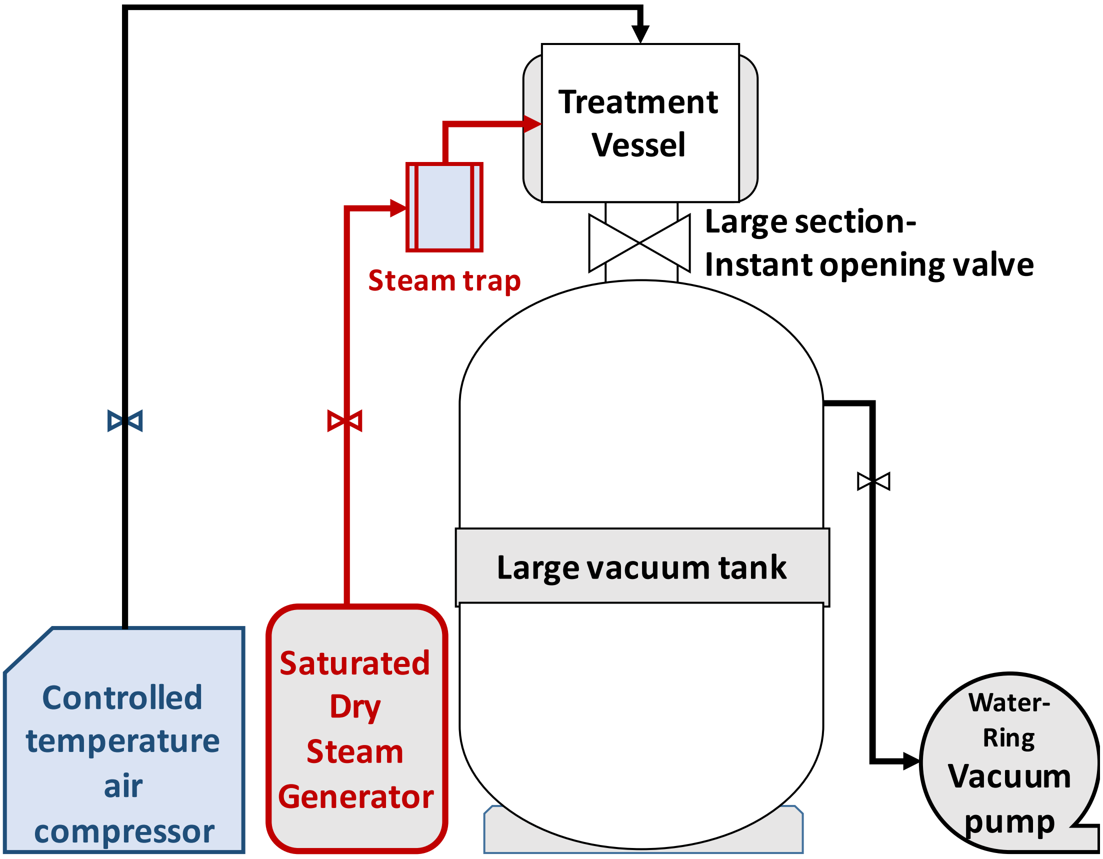

Figure 2. Instant controlled pressure-drop (DIC) laboratory-scale unit.

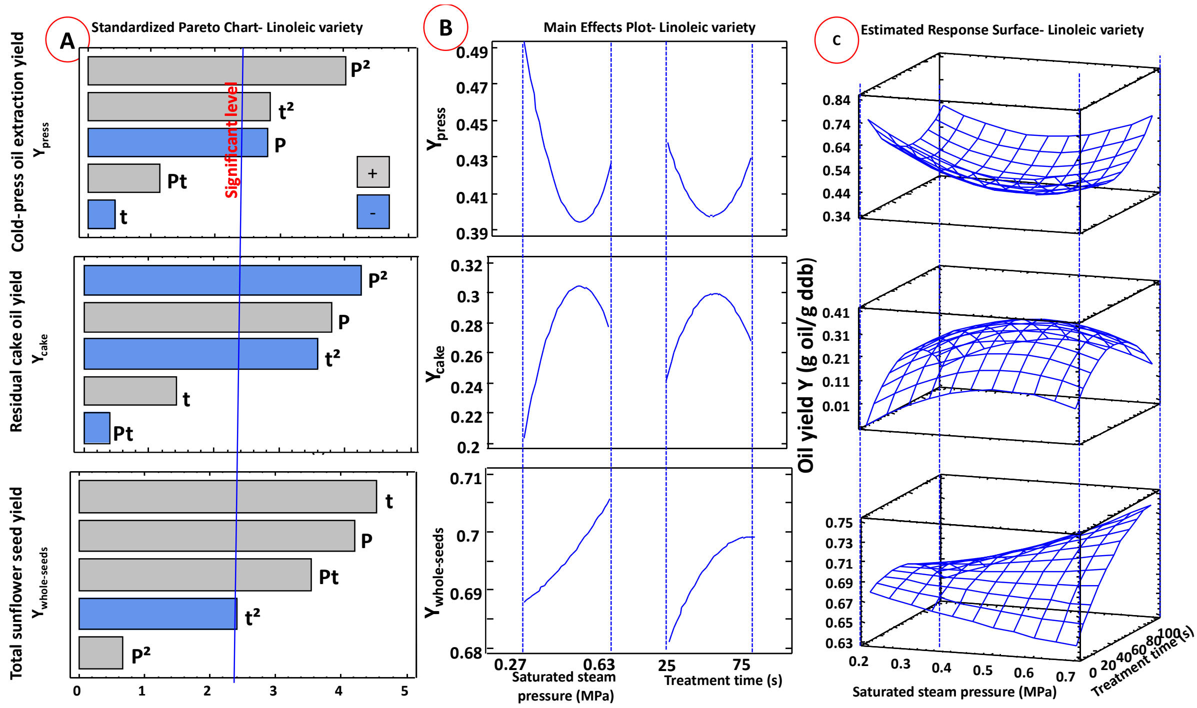

Figure 3. Effect of Instant Controlled Pressure-Drop DIC parameters on oil yields by cold-press extraction, cake oil concentration and whole oil concentration of differently DIC-textured sunflower seeds: A) Standardized Pareto Chart; B) Separated direct effects and C) Estimated response area.

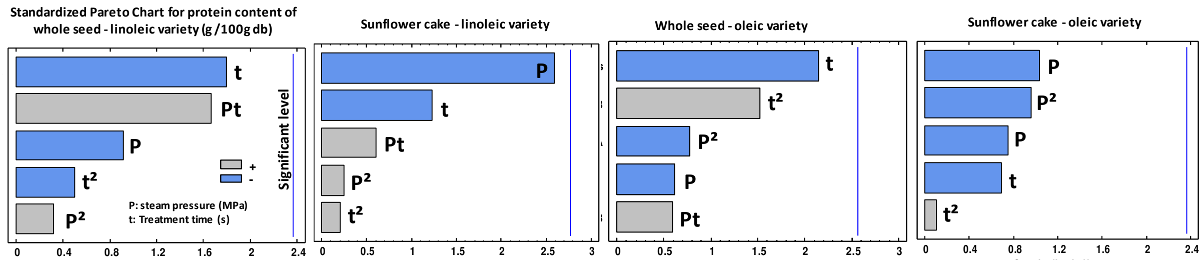

Figure 4. Standardized Pareto charts of Instant Controlled Pressure-Drop DIC pretreatment for protein content of the sunflower whole seeds and cake for linoleic and oleic varieties versus DIC processing parameters P (saturated dry steam) and t (processing time).

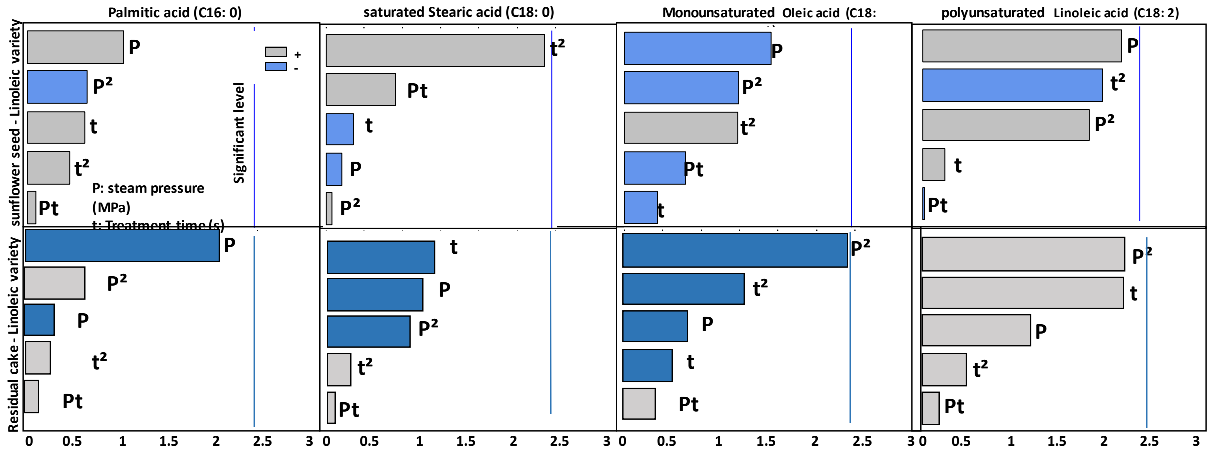

Figure 5. Standardized Pareto Charts of DoE of Instant Controlled Pressure-Drop DIC for fatty acid amounts of sunflower whole seeds and cakes for linoleic varieties versus DIC processing parameters P (saturated dry steam) and t (processing time).

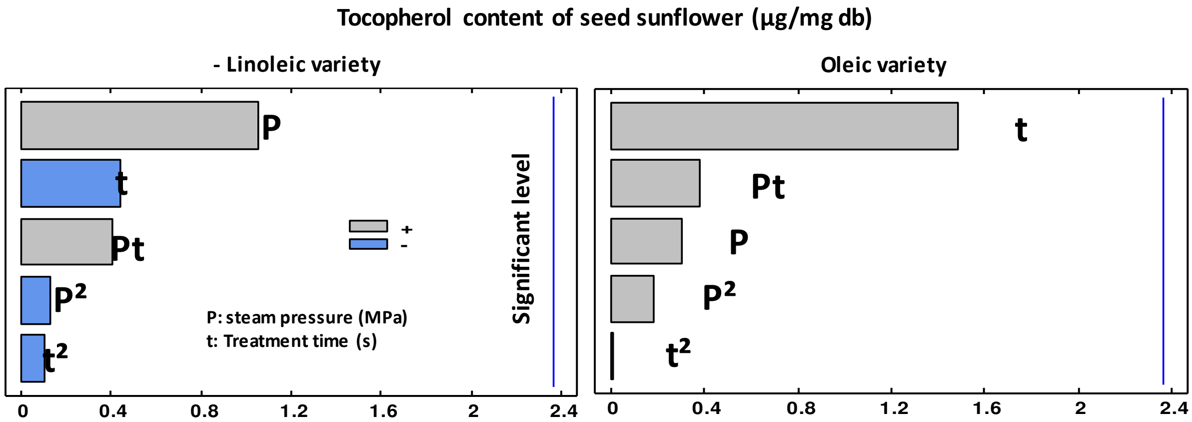

Figure 6. Standardized Pareto Charts for Tocopherol contents of whole sunflower seeds for linoleic and oleic variety versus Instant Controlled Pressure-Drop DIC processing parameters P (saturated dry steam) and t (processing time).