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1 **How Does Instant Autovaporization Deepen the Cold Press-Extraction Process** 2 **of Sunflower Vegetal Oil?**

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14 **Abbreviation list:**

15	DIC:	Instant Controlled Pressure-Drop
16	CCD:	Central Composite Design
17	RSM:	Response Surface Methodology.
18	K:	Matrix permeability coefficient (m ²)
19	v:	Kinematic viscosity (m ² s ⁻¹)
20	A:	Flux surface (m ²).
21	Pt:	Total pressure.
22	HTST:	High Temperature-Short Time
23	DoE:	Design of Experiments
24	Y:	Response
25	β_i , β_{ii} , and β_{ij} :	Regression coefficients
26	X _i :	Independent variables
27	ϵ :	Random error
28	i and j:	Factor indices.
29	HPLC:	High Performance Liquid Chromatography
30	RIE:	Ratio of Improvement of oil Extraction
31	Y _{press} :	Cold-press oil extraction yield (g Oil/g ddb).
32	Y _{cake} :	Residual cake oil (g Oil/g ddb).
33	Y _{whole seeds} :	Total sunflower seed yield (g Oil/g ddb).
34	ddb:	dry dry basis (mass basis of raw material devoid of water and oil).
35	db:	dry basis (mass basis of raw material devoid of water).
36	P:	Saturated dry steam pressure.
37	AOAC:	AOAC International (Standards of Association of Official Analytical Chemists).
38	t:	Heat treatment time.
39	R ² :	Regression coefficient.
40	RM:	Raw Material.
41	TMH:	2,3,5-trimethylhydroquinone.

42 Abstract

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

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

56 1. Introduction

57 Sunflower is a famous oleaginous plant widespread on all continents due to its hardiness and
58 low requirements for both inputs and water (Castro and Leite, 2018). Worldwide, its
59 cultivation is one of the largest in the world in Americas (Canada, Brazil, Argentina,
60 USA)(Heiser, 2008), Asia (China, India, Indonesia) and Europe in particular in France
61 (Gunstone, 2009). The sunflower seed consists of a shell and a kernel. The shell represents
62 70-90% of the dry matter formed from lignin, hemicellulose, cellulose fiber and a small
63 amount of lipids and proteins (Connor and Hall, 1997), while the kernel, with about 20% of
64 the dry matter of the seed, is the storage place of reserve for embryonic development
65 (Roche et al., 2004). Its lipid content is close to 44%, having a distribution of the main
66 saturated fatty acids: palmitic acid (C16: 0) of 5%-7%, stearic acid (C18: 0) of <10%, and
67 unsaturated: oleic acid (C18: 1) of 17%-20% and linoleic acid (C18: 2) of 50%-70% (Ayerdi
68 Gotor et al., 2016).

69 Diversification of sunflower varieties results in differences in the levels of linoleic and oleic
70 fatty acids. It generally meets the technological needs and requirements of the food and
71 non-food industries. For the food sector, sunflower has beneficial effects on human health.
72 It is a source of vitamin E, a dietary ingredient well-known for its anti-cholesterol properties
73 (Pelletier et al., 1995). It is also used in margarine (Moreau et al., 2002), canning, salad
74 dressings, table oil, and infant formula. Moreover, in the non-food sector, oleic sunflower oil
75 is valued because of its content of fatty acids as well as the presence of minor compounds
76 favorable to the manufacturing of pharmaceutical or cosmetic products, lubricants (Al
77 Mahmud et al., 2013), cleaning products, solvents in the paintings, etc. It can be used for the
78 production of both edible products and biofuel for diesel engines as a result of esterification
79 often with the methyl ester (Rashid et al., 2008) (Del Gatto et al., 2015), (Ayerdi-Gotor et al.,
80 200811-12).

81 As an edible product, and because of the negative health effects of solvent traces, it is
82 preferable that the industrial extraction of sunflower oil is obtained by pressing. However,
83 press-cake would generally have a high residual oil content of 15 to 20%, which is too high
84 for livestock feed or as a biomaterial (Isobe et al., 1992). This point also has a significant

85 economic impact due to the price difference between the oil and the residual solid cake.
86 Since the consumer demands to avoid the use of solvent extraction, an increase in the
87 extraction efficiency of the press becomes an essential objective in improving the
88 performance of the industrial process.

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

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

92 Where K is the oil-matrix permeability coefficient expressed in m², ν the kinematic viscosity
93 expressed in m² s⁻¹, A the normal oil-flux surface expressed in m², and P_t the total pressure. A
94 hydrostatic liquid behavior of the material, through for example high water content, induces
95 a more homogeneous total pressure and, then, a too weak gradient of total pressure. Hence,
96 the use of dried seeds is an inexorable condition in press-extraction operation (Bouallegue et
97 al., 2016). Therefore, to increase the pressed oil yield, one has to spread the pressure
98 gradient, reduce both viscosity and product thickness. In addition, heating is well known to
99 be capable of inducing increased yields through some thermal degradation of the cell walls.
100 However, this operation inevitably induces a certain degradation of the main molecules and
101 ingredients, many users recommend not to insert it. Hence, the objective of increasing the
102 oil extraction yield must be coalesced with a perfect preservation of quality, and specialists
103 advise against the use of heating as pretreatment and opt for a cold-press method to
104 preserve the quality of both extracted oil and cake (Boutin and Badens, 2009).

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

114 Indeed, the challenge of using instant pressure-controlled DIC coupled with cold press to
115 well extract the sunflower seed oil must be studied to obtain the benefits of
116 increasing oil yields and preserving the high quality of the oil (Ixtaina *et al.*, 2011), (Cường,
117 2013). Thus, the oil from DIC-assisted cold pressing extraction must be analyzed to prove
118 that it has the same quality as conventional cold-pressed oil, in terms of biochemical
119 composition, sensory characteristics, and color. The oil must meet to all international oil
120 standards.

121 2. Materials and methods

122 2.1. Raw materials and chemicals

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

125 Two varieties of sunflower: linoleic and oleic types were used. They were transported to the
126 laboratory and well packaged, and stored at room temperature. The initial moisture content
127 dry basis (db) (W_i) was about 0.07 ± 0.005 g H₂O/g db for the linoleic sample and $0.09 \pm$
128 0.005 g H₂O/g db for the oleic sample. Our study on cold-pressing extraction has concerned
129 the whole sunflower seeds, thus maintaining their hulls.

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

136 **2.2. Treatment process and Experimental Protocol**

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

141 *Figure 1.*

142 Some part of the experiments and assessments required a laboratory-scale n-hexane solvent
143 extraction. Following the DIC texturing treatment and prior to the properly-said pressing, a
144 drying step was performed. The sunflower seed samples underwent an airflow drying at 40
145 $^\circ\text{C}$ and 1 m/s, until reaching a final moisture content of 0.060 ± 0.005 g H₂O/g db. This level is
146 well-known as the most appropriate moisture content for the cold press-extraction. For an
147 assessment requiring a solvent extraction process, the seeds were ground using a laboratory
148 knife mill (RETSCH, Grindomix model GM 200-F; Kurt Retsch GmbH & Co. KG, Haan,
149 Germany) at 8000 rpm for 40 seconds.

150 **2.2.1. DIC treatment**

151 The DIC unit was presented in various articles (Figure 2) (Allaf and Allaf, 2014; Sabah Mounir
152 et al., 2011; and Albitar et al., 2011). The research works carried out on instant controlled
153 pressure-drop technology (DIC) at both fundamental levels and industrial applications have
154 the specificity of a theoretical foundation based on the thermodynamics of instantaneity
155 (Allaf et al., 2014): far from the equilibrium, processes occur between the two-asymptotic
156 behaviors of quasi-static (classical thermodynamics) and instantaneous limits. Processes and
157 transformations occurring in a theoretically zero time (transport of molecules in dozens of
158 milliseconds), generally imply a decrease in the fluctuation degree of the random
159 translational movement of the particles, at the same time an equally instantaneous decrease
160 of the temperature. The relaxation time (Δt of dozens of ms) is an essential parameter in the
161 considered processes; it implies a very high pressure-drop rate $\Delta P/\Delta t$ often greater than 5
162 10^5 Pa s⁻¹. The cooling rate can reach exceptional levels of 1500 to 2000 kW/m² of the
163 product (Allaf et al., 2018b).

164 *Figure 2.*

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

168 (Berka-Zougali et al., 2010) (Albitar et al., 2011). Practical treatment with DIC involves a
169 vacuum stage of 5 kPa, followed by a HTST (High Temperature-Short Time) type process
170 generally using saturated dry steam absolute pressure (usually ranged between 0.05 and 1
171 MPa), i.e. temperature of 60 °C to 170 °C (Besombes et al., 2010). The short treatment time
172 (some dozens of seconds) includes a steam injection step (lasting less than 3 s) involving
173 vapor condensation on the product surface (moisture content may increase by ΔW of almost
174 0.1 g H₂O/g db, resulting in soaring heating coefficient up to tens of kW m⁻² K⁻¹), followed by
175 a homogenization step of both thermal and condensed water within the product.
176 Succeeding this roughly balance step, the pressure abruptly drops>> towards the vacuum
177 (steam tank maintained at 3 to 5 kPa) (Allaf and Allaf, 2013).

178 Instantaneous-type of coupled thermomechanical transformations of DIC is: i/ an
179 autovaporization of a certain quantity of volatile molecules, mainly water to reach the new
180 thermodynamic equilibrium; (the moisture content undergoes a decrease ΔW of
181 approximately -0.11 g H₂O/g db), ii/ mechanical stresses within the material resulting in a
182 new structure and texture, iii/ a cooling of the material possibly involving an overshoot
183 beyond of the glass transition. Thus, the new often expanded structure of the matrix is
184 preserved (Haddad et al., 2008), and iv/ while preserving a perfect stop of the thermal
185 degradation.

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

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

202 **2.2.2. Extraction process: Mechanical cold-press extraction**

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

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

210 In our laboratory-scale study, the cold-pressing device is based on single-screw extrusion
211 which operates according to a continuous process (p500R, Anton Fries Maschinenbau GmbH,

212 Germany). It operates at a frequency of 50 Hz, a voltage of 400 V and a power of 1.5 kW.
 213 This device comprises a stainless-steel cylindrical hopper of 35 mm diameter. In the feeding
 214 zone, the seeds are pressed and freely transported under the effect of the mono-screw
 215 towards the bottom of a fixed tube to create a high-pressure cold-compression. This fixed
 216 tube contains holes along its length through which the oil is recovered at the bottom of the
 217 press by a flow through calibrated 8 mm hole called die or nozzle, positioned at cylinder end
 218 to remove the cakes in granular forms. This separation is done in an area called the filtration
 219 zone.

220 Following filtration, the extracted oil was measured while the cake was stored in poly-bag at
 221 a temperature of 4 °C. The oil yield is expressed in g oil/g ddb (dry dry matter, which means
 222 a mass basis of raw material rid of water and oil) with an error estimated at 0.05%. Cold-
 223 press oil extraction was successively performed on the raw-material (untreated) and DIC-
 224 textured sunflower seeds after adjusting the moisture content of each sample to $0.06 \pm$
 225 $0.005 \text{ g H}_2\text{O/g db}$.

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

226 2.2.3. Design of Experiments DoE

227 2.2.3.1. Experimental Protocol

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

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

$$Y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{i=1}^{k-1} \sum_{j=2}^k \beta_{ij} x_i x_j + \varepsilon \quad \text{Eq. 3}$$

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

246 **2.3. Assessment methods and processes**

247 **2.3.1. Measurement of moisture content**

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

250 **2.3.2. Oil extraction by Randall**

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

263 In the present case of sunflower seeds, various differently processed, un-textured and DIC-
264 textured seeds, and cakes were studied. Before starting the properly said operation, Velp
265 148/3 parameters were checked out and corrected for compatibility. In the current case,
266 since the solvent used was n-hexane, and the plate temperature was 180 °C, Viton seals
267 were used. A sample of 5 to 7 g of crushed sunflower was placed in the porous cartridge to
268 receive, by washing, almost 40 ml n-hexane. This operation was carried out in 6 h. For
269 removing the remainder traces of n-hexane solvent from the evaporator, the cartridge
270 contents were deposited in 18 ml Pyrex tubes and put under Sorbonne with airflow heated
271 at 65 °C for 1 h. This extraction operation is faster, ampler, and more effective than a simple
272 maceration extraction. The extracts were weighed using an analytical high-precision balance
273 (with an accuracy of 0.001 g) and then stored at -4 °C for biochemical analyses. Oil yields
274 were calculated with an estimated error of ± 0.0005 g oil/g ddb.

275 **2.3.3. Determination of the free fatty acids**

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

279 The chromatography device comprised a HP-5MS (5% Phenyl Methyl Siloxane) capillary type
280 column (30 m \times 350 μ m \times 0.25 μ m), a furnace with initial temperature ranged from 155 to
281 230 °C with a speed of 45 °C/min. The final phase reached a maximum level of 240 °C and
282 the oven run time was 10 min for each sample.

283 The protocol of the fatty acid analysis was to consider for each sample 15 μ l of sunflower oil
284 mix with 20 μ l of the TMH, and 800 μ l of the isooctane and, after stirring for a few seconds in
285 a vortex, it was ready for analysis. A volume 0.8 μ l of the assembly was eluted and injected
286 using a syringe in split mode and the temperature of the injector was maintained at 270 °C.
287 Once vaporized by the injector, the compounds were entrained in the column by hydrogen

288 as carrier gas. Peak integration and identification of fatty acids were based on the use of the
289 mass spectral MS database of the NIST'98 National Library (National Institute of Standards
290 and Technology of the United States (NIST), Gaithersburg, MD, USA).

291 **2.3.4. Determination of the protein content**

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

296 DUMAS method, which is performed under oxygen and at high temperature does not
297 directly measure the protein content. This allows quantification of the total nitrogen that is
298 expressed as a percentage and using an internationally recognized conversion factor of 6.25
299 which differs from protein to protein depending on its amino acid composition, equivalent to
300 0.16g of nitrogen per gram of protein is required to convert the measured nitrogen content
301 to a protein content. It's an easy and fast analysis.

302 **2.3.5. Determination of tocopherol content**

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

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

313 In the current case, the mobile phase was composed of a mixture (75:25 v/v) of acetone and
314 methanol with a flow rate of 1 cm^3/min , provided by the HPLC Delta Chrom SDS 030 pump.
315 The tocopherols were separated by the reversed phase C18 column at 30°C, 250 mm * 4.6
316 mm, and 5 μm as particle size (Macherey-Nagel Ltd., Düren, Germany). Quantification of the
317 results was evaluated and integrated by comparison with the retention time collected with
318 those of the respective standard surfaces using the chromatographic station software CSW
319 32 (Data Apex Ltd., Prague, Czech Republic).

320 **3. Results and Discussions**

321 **3.1. Effect of DIC-texturing on cold-press extraction**

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

$$Y_{\text{press}} = Y_{\text{whole-seeds}} - Y_{\text{cake}} \quad \text{Eq. 4}$$

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

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

329 *Table 1.*

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

339 *Figure 3.*

340 For both technologic and economic reasons, industries require reaching the highest cold-
341 press extraction yields Y_{press} . DIC-texturing at 0.2 MPa for 50 s resulted in dramatically
342 increasing of the cold-press yield of linoleic of 146% RIE, which means 0.3462 against
343 0.5060 g oil/g ddb for non-treated seeds. Similar results were obtained for oleic seeds DIC-
344 treated at 0.5 MPa for 50 s, with RIE=132% and cold press yield of 0.5206 against 0.3950
345 g oil/g ddb for non-treated seeds. It is worth highlighting that DIC-texturing pretreatment
346 allows increasing the cold press yields respect to the whole seed yields by about 20%, from
347 53.11% to 73.56% for linoleic variety and 12%, from 59.50% to 71.64%, for oleic variety
348 (Table 1).

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

353 *Table 2.*

354 *Table 3.*

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

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

366 **3.2. Impact of DIC on the quality of the final sunflower seed cake**

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

377 **3.2.1. Evolution of protein content of the sunflower whole seeds and press-cake vs DIC**
378 **treatment conditions.**

379 *Table 4.*

380 *Figure 4.*

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

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

387 **3.2.2. Evolution of fatty acid contents of sunflower oil versus DIC treatment conditions**

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

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

397 *Table 5.*

398 *Figure 5.*

399 **3.2.3. Tocopherol content of sunflower oil versus DIC treatment conditions**

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

404 The weak variation in tocopherol contents in both seed oil and cake, indicates that there was
405 no change between the DIC-textured and non-textured samples. Moreover, the statistical

406 analyses represented by Pareto Charts show a significantly no effect of the DIC parameters
407 of the two linoleic and oleic varieties. This confirms that there was no degradation of the
408 tocopherol contents (Figure 6).

409 *Table 6.*

410 *Figure 6.*

411 **4. Conclusion**

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

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

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- 544

545 **Table captions**

546 *Table 1. Cold-press Y_{press} , solvent extracted Residual cake oil Y_{cake} , and total whole-seed oil yields $Y_{whole-seeds}$, expressed in g Oil/g db (dry basis: material mass*
547 *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*
548 *and differently DIC-treated sunflower seeds.*

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

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

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

552 *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*
553 *conditions*

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

555 **Figure captions**

556 Figure 1. Applied protocol of sunflower seed

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

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

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

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

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

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Table 1.

Linoleic variety			Y _{press}			Y _{cake}			Y _{whole seeds}			Y _{press} /Y _{whole-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-seeds}			Y _{press} /Y _{whole-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%

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571

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

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

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

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

577

578 *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

579 <RM>: Average of Raw Material samples

580 <DIC>: Average of DIC treatments

581 *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*

Trial n°	Linoleic variety							
	whole seed of sunflower				cake of sunflower			
	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

582 <RM>: Average of Raw Material samples

583 <DIC>: Average of DIC treatments

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

Trial n°	Linoleic variety			TTP
	β	γ	α	
	(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

587 <RM>: Average of Raw Material samples

588 <DIC>: Average of DIC treatments

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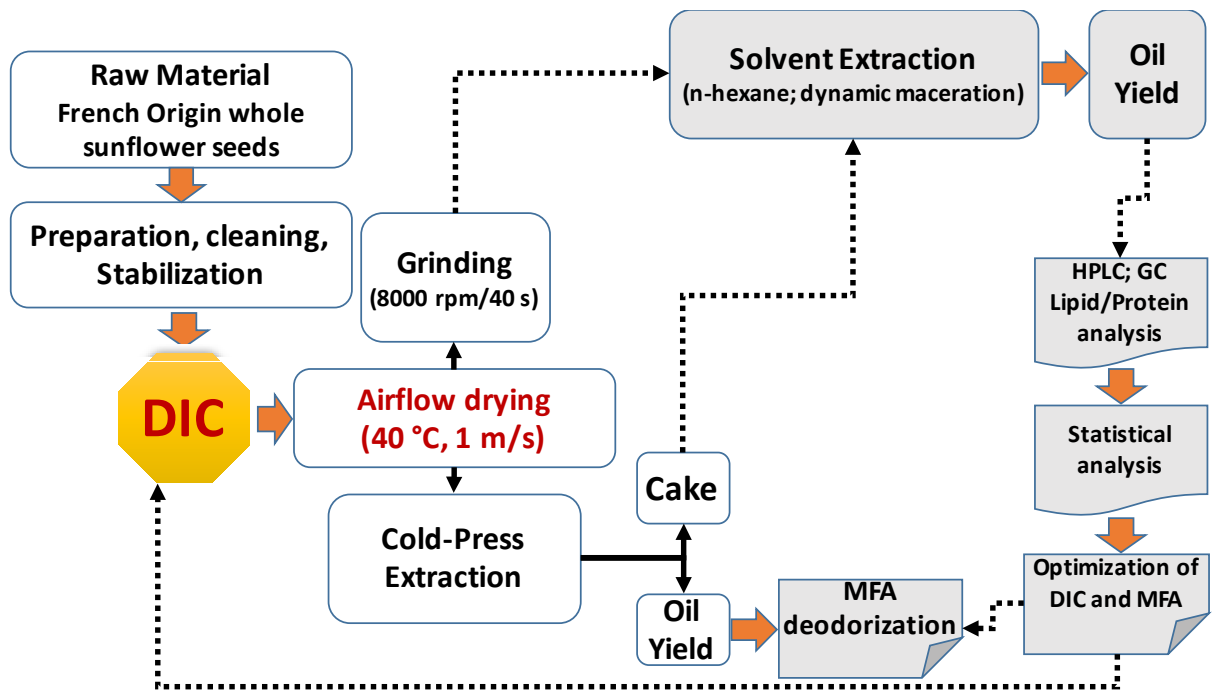
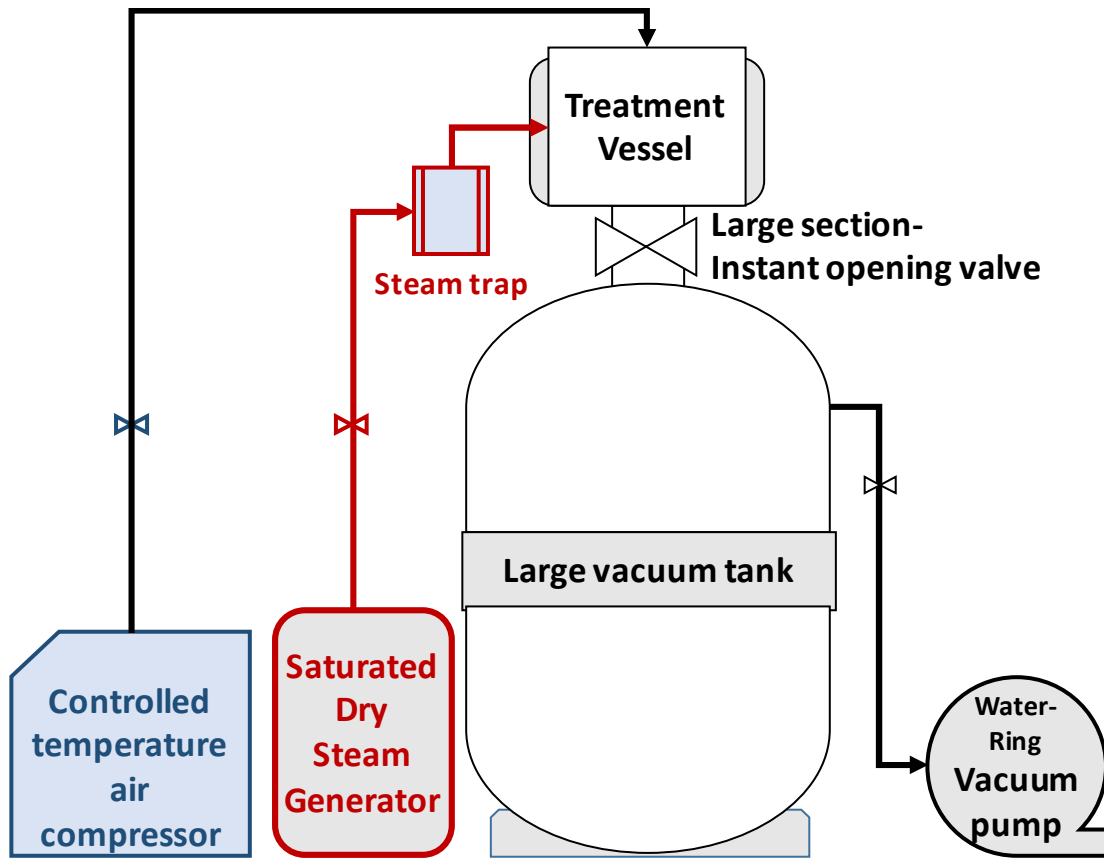


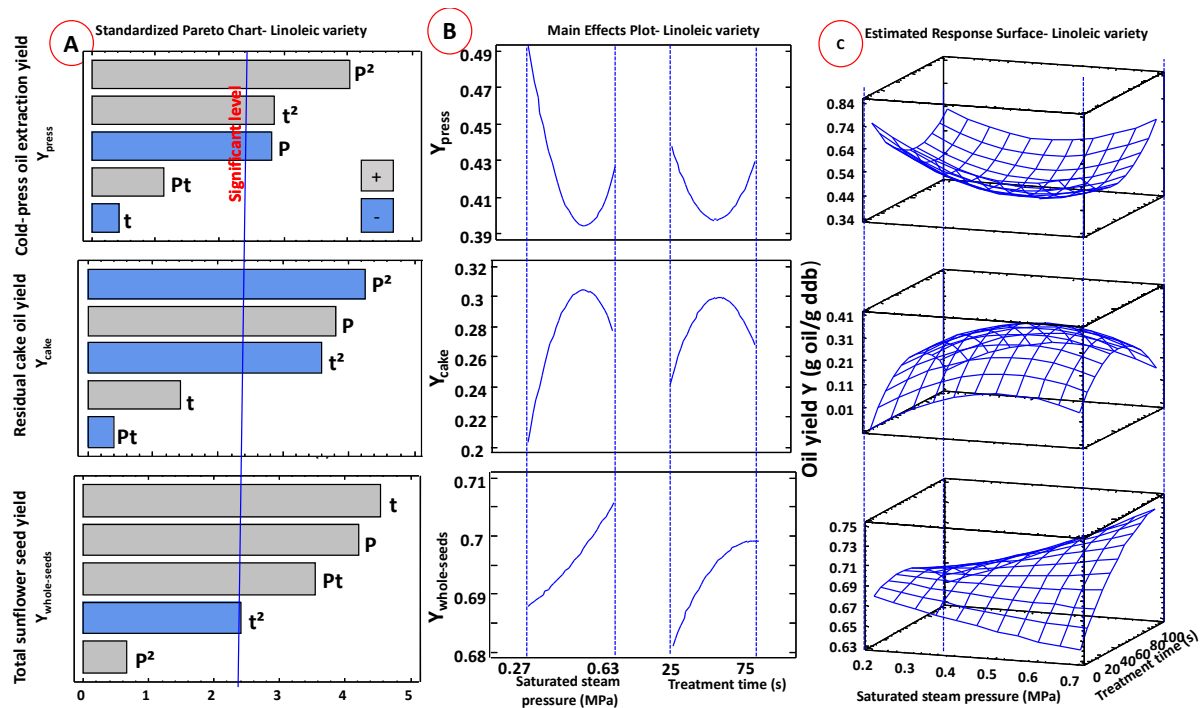
Figure 1. Applied protocol of sunflower seed

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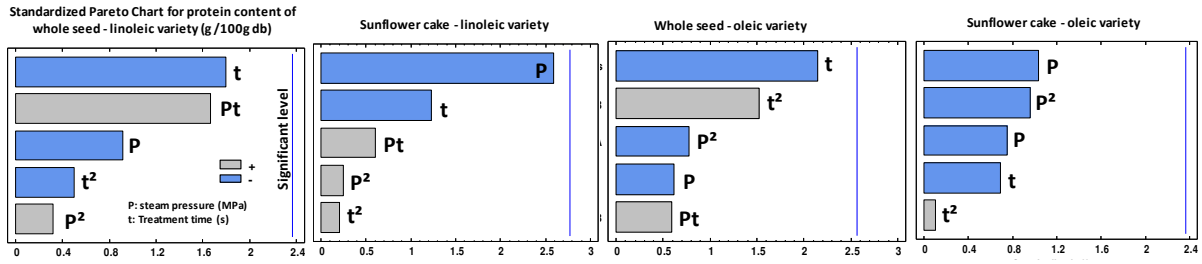
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Figure 2. Instant controlled pressure-drop (DIC) laboratory-scale unit.



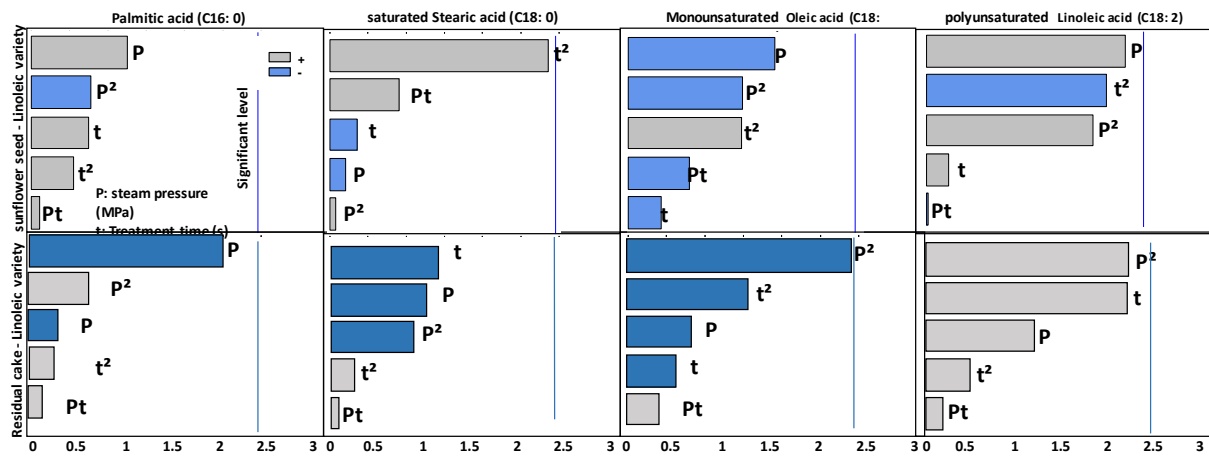
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Figure 3. Effect of Instant Controlled Pressure-Drop DIC parameters on oil yields by cold-press extraction, cake oil concentration and whole oil concentration of differently DIC-textured sunflower seeds: A) Standardized Pareto Chart; B) Separated direct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



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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).



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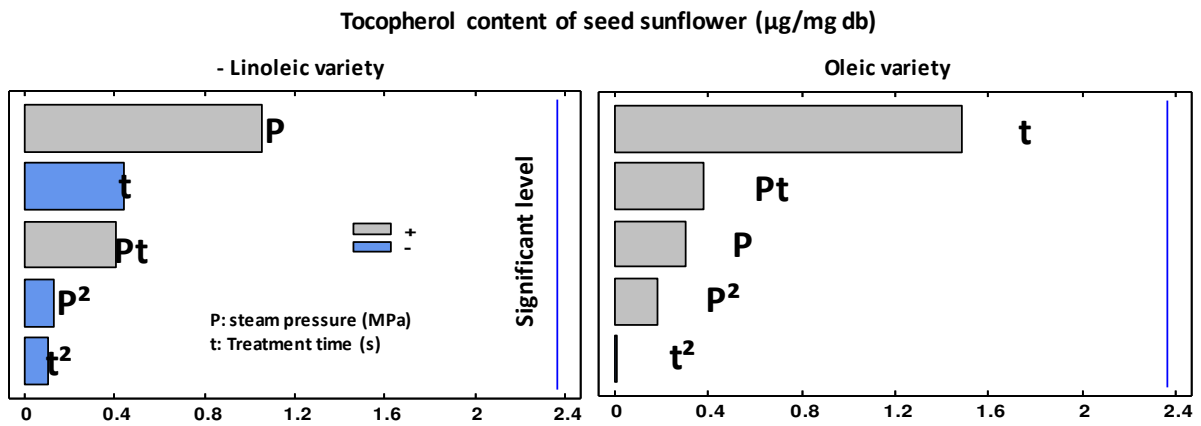
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Figure 5. Standardized Pareto Charts of DoE of Instant Controlled Pressure-Drop DIC for fatty acid amounts of sunflower whole seeds and cakes for linoleic varieties versus DIC processing parameters P (saturated dry steam) and t (processing time).



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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).