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1 Comparison of Expander and Instant Controlled Pressure-Drop DIC 2 technologies as Thermomechanical Pretreatments in enhancing 3 solvent extraction of vegetal soybean oil

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13 **Running Head:**

14 DIC to enhance Solvent Extraction of vegetal oil

15 **Abstract**

16 As an intensification process of the unit operation of vegetable oil extraction, the cooking pre-
17 treatment systematically results in damaging thermal degradations. However, recently, two
18 thermomechanical expansion processes have been suggested and applied at industrial scales to
19 pre-treat oilseeds: expansion and instant controlled pressure drop DIC. In both cases, it is a flash
20 transformation from high-temperature/high-pressure to 100°C/atmospheric pressure and
21 33°C/vacuum phases, respectively. The use of DIC resulted in cumulative impact yields of 247
22 mg oil/g db, compared to 210, 230, and 224 for cracking, cracking/blocking, and expanding,
23 respectively. To achieve the same yields obtained in 160 min for cracked or cracked/flaked
24 soybean, the expander took 120 min against 35 min for DIC. Besides, thanks to the short heat
25 treatment and the induced instant cooling, DIC allowed the highest preservation of soybean oil
26 quality, with fatty acid concentrations almost identical to that of untreated seeds.

27 **Practical impacts**

28 In the sector of the vegetal oil industry, researchers and engineers mostly look for increasing the
29 extraction yields while preserving or improving the sensorial and biochemical quality of the oil.
30 Henceforth, the modification of the oleaginous seed texture may usually be the major factor

31 affecting and normally enhancing the capacity of extraction. Based on the improvement of
32 material structure through adequate expansion, the higher the volume of vapor issued from the
33 autovaporization throughout the instant controlled pressure-drop (DIC) technology, the higher
34 the oil yields. Moreover, using the instantaneous thermodynamic theory, the feasibility of
35 integrating DIC technology as an HTST treatment in the industrial-scale soybean extraction
36 process could be performed and optimized without any biochemical degradation.

37 **Keywords:** Texturing; Instant Controlled Pressure-Drop (DIC); Coupled Washing-Diffusion
38 Modeling; Effective Solvent Diffusion; Oil Quality Preservation.

39 1. Introduction

40 Engineers and researchers studying the processes of extraction of plant-based ingredients (active
41 molecules, fibers, vegetable oils, proteins, etc.) agree in the necessity of changing the natural
42 structure of the plant before performing the properly said solvent extraction. Extraction process
43 from beans and seeds, fruit and vegetables, leaves and roots, etc. normally requires significant
44 initial pretreatment operations, such as husking and cutting, crunching and grinding, cracking
45 and flaking, etc. Furthermore, the operations capable of increasing the porosity and tortuosity,
46 allow improving the technological ability for solvent extraction.

47 The extraction of vegetal oil is highly important in industrial processes. Likewise, for dozens of
48 years, soybean has become one of the strongest oleaginous seeds with significant amounts of
49 both oil and protein with 21.38% and 40.69% db (dry basis), respectively (Brumm et al., 2004).
50 Thus, soybean has become one of the most important food crops and the main lipid resource
51 consumed in the United States (57%) which, according to Cheng et al. (2018), is the world's
52 largest producer. The soybean oil is the largest vegetable oil used in industrial food applications
53 such as cooking, frying, etc., reaching 75% of the total human consumption of oil. With about
54 54% of the worldwide vegetal oil production, soybean is currently the main source of vegetable
55 oil (USDA, 2020) and protein-based meals (Britz et al., 2008). The adequate chromatography
56 analyses show that the polyunsaturated fatty acid fraction was the dominant compound with a
57 combination of linoleic acid C18:2, oleic acid C18:1, and linolenic acid C18:3 (Omega 3) with
58 percentages of 47.6%, 27.15%, and 6.7%, respectively (Cheng et al., 2018).

59 Commonly, the extraction of soybean oil is carried out by solvent, mainly n-hexane, which has
60 been preferred because of its high selectivity in dissolving the oil, its high volatility in removing
61 it from the meal, its high productivity and weak energy consumption, and environmental effects
62 (Hammond et al., 2005); (Rosenthal et al., 1996), and relatively low cost.

63 However, it is worth noticing that the modern industries of solvent extraction of oil, some initial
64 pre-treatment operations of oleaginous seeds have been used to render the structure and the sizes
65 more adapted to the process, whilst avoiding preheating stages that would induce thermal
66 degradations (Perez et al., 2011). Hence, normally successive mechanical pretreatments are used
67 such as i/ dehulling, ii/ grinding, and iii/ flattening. More recently, the use of expander has been
68 developed. It consists of short-term heat treatment at high steam pressure, followed by rapid
69 depressurization towards the atmospheric pressure. Expander treatment requires also a
70 subsequent cooling step to reach the ambient temperature level to stop thermal degradation and
71 meet consumer requirements of high-quality oil and meal. Expander removes the outer
72 membrane, reduces the surface resistance to mass transfer, and increases the porosity of the bean
73 matrix. Specifically, the solvent gets higher diffusivity within the expander-texture grain.
74 Therefore, a significant change in the structure of oleaginous material is essential to achieve
75 porosity (Zhang et al., 1994) (Hernandez E. 2019).

76 Nowadays, the pretreatment of seeds before the hexane extraction process and the degumming
77 and other refining operations are included in the industrial flow diagram Figure 1.

78 **Figure 1. Industrial flow-chart of the solvent extraction of soybean oil**

79 The dehulling process of removing the outer membrane allows more direct contact between the
80 solvent and the interaction bean surface. The cracking operation results in the breaking up into
81 small pieces of soybeans, which pass between two crenelated cylinders able to achieve the
82 distortion and breakage of the grain. Although such an operation implies no real modifications of
83 the diffusivity, the extraction is improved through the reduction of the transfer distance and the
84 increase of the interaction surface. Thus, grinding normally results in increasing the interaction
85 between the granules and the solvent. Thus, the crushing stage is generally followed by an
86 aspiration operation performed to purge the fine powder and eliminate the shell-forming about
87 8% of the weight of the seed. This operation allows a clear increase in protein in the cake.
88 Following cracking, the flaking of the soybeans is carried out using a passage in a succession of
89 rotating cylinders operating at high temperatures. The thickness of the flasks obtained ranges
90 from 0.2 to 0.3 mm, which results in a greater interaction surface area between the solvent and
91 the material, i.e. a greater starting accessibility. However, it is worth noticing that such pressing
92 usually implies a decrease in porosity and thus a lower diffusivity. Notwithstanding, chipping
93 generally has a positive impact on extraction with better yield and greater kinetics (Zhang et al.,
94 1994).

95 The study of extrusion and expander in the preparation of oleaginous materials started in the
96 1970s at a laboratory scale. Contrariwise, their use at **the** industrial-scale has not started before
97 the 1980s to impact oil extraction (Johnson and Lusas, 1983). The expander allows the soybeans
98 to be in close contact with a high-pressure saturated steam flow and move through a screw
99 within the high-temperature treatment vessel. This heating system converts soya flakes into
100 porous plugs employing the rapid passage of the material from the high-pressure zone (in the
101 internal part of the expander) towards the atmospheric pressure. Extrusion and expander mainly
102 differ from the heating energy source. Indeed, while the expander heats the seeds by
103 condensation/convection of saturated and super-heated steam, the extrusion heating source is
104 chiefly performed by the mechanical way of sharing between the screw and vessel-wall (Mat
105 Yusoff et al., 2015). **Allaf et al. (2014)** proved that the decompression towards the atmospheric
106 pressure systematically has a very limited impact in terms of expansion, whereas the instant
107 controlled pressure-drop towards a vacuum **has a** much higher capacity of texturing (swelling,
108 etc.). DIC can lead to a rupture of the cell walls. Indeed, the texturing effect normally depends on
109 the amount of vapor generated by autovaporization which is directly correlated with the
110 temperature drop ΔT_{pd} . **Thus, at a similar temperature and for different treatment-time, expander**
111 **impact should normally be much weaker than the Instant Controlled Pressure-Drop DIC.** Hence,
112 expander treatment of oleaginous grains, which is usually performed at 0.6 MPa as absolute
113 steam pressure with about 160 °C as treatment temperature, the decompression towards the
114 atmospheric pressure results in a temperature drop $\Delta T_{pd} = 60$ °C. With similar treatment
115 conditions of steam pressure and temperature, DIC flash-depressurization usually achieved
116 around a vacuum of 4 kPa, i.e. a final equilibrium temperature of about 30 °C, has a twice more
117 temperature drop $\Delta T_{pd} = 130$ °C.

118 Our present study deals with inspecting, defining, and evaluating the impacts the different
119 mechanical pretreatments and thermal/mechanical texturing ways on solvent extraction of
120 soybeans brought in terms of oil yields and quality. It aimed at relevantly measuring the effects
121 of the processing conditions and optimizing the specific case of Instant Controlled Pressure-Drop
122 DIC texturing for the most relevant industrial operation. Such a study should also allow
123 establishing empirical correlations between treatment conditions, structure, and functional
124 behavior of differently pretreated soybeans. More than a simple yield intensification and
125 preservation of both oil and meal quality, this study goes beyond the simple technical
126 comparison study of the texturing treatment. It was integrating the industrial processes and
127 identifying their impacts in terms of energy consumption, environmental impact, and total costs.

128 **2. Materials and methods**

129 **2.1. Raw materials**

130 American soybeans were provided by “Carthage Grains” corporation. They were pretreated at an
131 industrial scale before achieving the solvent extraction operation. The sequential pretreatments
132 were the cracking (C), cracking-flaking (CF), and cracking-flaking-expanding (CFEx) (Figure
133 2). **Afterward, these differently pretreated C, CF, and CFEx soybeans were textured by Instant
134 Controlled Pressure-Drop DIC** using the laboratory-scale equipment at different conditions of
135 saturated dry steam pressure P and treatment time t, as defined by the Design of Experiments
136 (DoE). **The differently pretreated soybeans were all adjusted in terms of moisture content using
137 an airflow drying at 40 °C and 3 m/s to reach 11% wet basis.**

138 **2.2. Chemical product**

139 For the extraction and analysis of soybean oils (fatty acids and tocopherols), the solvents used
140 (methanol, ethanol, toluene, dichloromethane, acetonitrile, chloroform, and hexane) were HPLC
141 and GC -grade solvents were supplied **from Thermo Fisher Scientific (Courtaboeuf, France)** by
142 UniLasalle Polytechnic Institute (Beauvais, France) and La Rochelle University (France).

143 **2.3. Experimental Methodology**

144 N-hexane solvent extraction of soybean oil was accomplished with differently pretreated samples
145 C; CF; and CFEx. To recognize the best manner of Instant Controlled Pressure-Drop DIC
146 texturing, we defined a specific Design of Experiment DoE integrating the main DIC operating
147 parameters. The responses in terms of yields and kinetics were measured as the most effective
148 way to assess the modification of the technological aptitude of the materials. Randall extraction
149 method was adapted to compare various yields while dynamic maceration allowed defining the
150 extraction kinetics. Additionally, chromatography analyses were used for measuring the
151 concentration of different fatty acids and specifying the extracted oil quality.

152 The protocol based on the integration of DIC texturing in the preparation stage of the soybean
153 seeds before solvent extraction.

154 **Figure 2. Diagram and experimental protocol of soybean oil extraction inserting a DIC texturing stage**
155 **Figure 2 presents different routes for vegetal oil extraction including the possible ways to**
156 **improve yields and kinetics of soybean oil extraction by adding DIC treatments** using a Design
157 of Experiment DoE on each type of pretreated seed at the industrial scale of the solvent
158 extraction line.

159 2.4. Main intensification ways and technology

160 DIC texturing process (Figure 3) had different impacts depending on the operating parameters,
161 the characteristics of soybeans. It changed the porosity and the tortuosity of the treated samples
162 to, consequently, increase the solvent diffusivity within the bean. It also allowed breaking the
163 cell walls and increasing the oil availability in the grain (Jablaoui, 2018). The modifications of
164 oil diffusivity and availability allowed n-hexane extraction to revise Randall yields and dynamic
165 maceration kinetics, respectively.

166 Figure 4 shows that the difference in temperature just before and after DIC flash decompression
167 mainly alters its impacts. By using the saturated dry steam as the heating source, the treatment
168 temperature ranged from 152 to 180 °C depending on the pressure range between 0.5 and 1 MPa.
169 DIC processing time t usually ranged between 10 and 100 s, was defined to ensure adequate
170 thermal and moisture homogeneities within the seed. The pressure-drop is performed towards a
171 vacuum of 4 kPa, with an instantaneous mode in less than $20/40 \cdot 10^{-3}$ s. Usually, the
172 decompression rate $\Delta P/\Delta t$ should be higher than 500 kPa s^{-1} .

173 Figure 4 shows the DIC stage. It is worth noticing that a first vacuum phase is achieved to set a
174 close superficial interaction between the material and the saturated dry steam in the treatment
175 vessel. The high-temperature/high-pressure stage is usually defined to allow the grain to reach
176 suitable internal thermal and moisture homogeneities. By abruptly dropping the pressure towards
177 a vacuum, the autovaporization of some amount of the grain water instantaneously happens,
178 provoking an inevitable cooling and a possible texturing (Ben Amor and Allaf, 2009a).

$$m_v = \frac{m_d(c_{p,d} + W_{p,H2O}c_{p,w})(T_t - T_f)}{L} \quad \text{Eq 1}$$

179 The structure modification of the grains in terms of expansion ratio and tortuosity inevitably
180 depends on the quantity of the generated vapor m_v and more specifically its volume. For similar
181 treatment parameters of saturated steam pressure and temperature T_t , the autovaporized quantity
182 of m_v strictly depends on the final pressure, and, obviously the final temperature T_f . Since the
183 vacuum level of DIC was about 4-5 kPa, the temperature abruptly reached just after the instant
184 decompression an equilibrium level of about 30-33 °C. This means that m_v was about twice more
185 in DIC with $(T_t - T_f) = (152 - 33)^\circ\text{C}$ than in expander $(T_t - T_f) = (152 - 100)^\circ\text{C}$. It is well-
186 known that the higher the amount of vapor generated by autovaporization, the higher the
187 texturing effect (Ben Amor and Allaf, 2009b).

188 Subsequently, by instantaneously dropping the saturated dry steam towards a vacuum, DIC
189 incites a much higher possible expansion leading to much better porosity and greater tortuosity
190 than expander can do. The optimized DIC treatment should inexorably imply higher texturing
191 and tortuosity, hence possibly increasing the technological aptitudes of the product in terms of
192 effective diffusivity and starting accessibility with a higher interaction exchange surface.
193 Likewise, the autovaporization/texturing process may also lead to breaking cell walls, which
194 increases the availability of the oil and active molecules in the cells (Allaf and Allaf, 2014) and
195 (Jablaoui et al., 2019).

196 *Figure 3. Schematic diagram of DIC laboratory scale unit: 1/ Treatment vessel; 2/ Vacuum tank; 3/ Large section-*
197 *instant opening valve; 4/ water-ring vacuum pump; 5/ Saturated steam generator with condensed water trap.*

198 During the outstandingly short-time pressure-drop (dozens of milliseconds), only the
199 thermodynamics of instantaneity can adequately interpret this far-from the quasi-static process.
200 Since the temperature is fundamentally defined as the density per mole of the kinetic energy of
201 anisotropic translational random micro-agitation, the one-dimension organization of velocity
202 during such so short time decompression results in a precipitous cooling, easily crossing the
203 glass transition and greatly improving the vapor separation (Chemat et al., 2017).

204 *Figure 4. Stages of DIC and expander treatments, respectively: A/ Grains to set in the treatment vessel to*
205 *immediately connect with the vacuum tank (only for DIC); B/ Saturated dry steam to reach the desired pressure and*
206 *temperature treatment levels; C/ High pressure/high temperature to maintain for dozens of seconds (for DIC) and*
207 *some minutes (for expander); D/ Pressure to abruptly drop towards a vacuum (3-5 kPa for DIC), and towards the*
208 *atmospheric pressure (for expander); E/release from the vacuum towards the atmosphere pressure (for DIC) and a*
209 *cooling stage from 100 °C towards an almost ambient temperature (for expander).*

210 **2.4.1. Oil yield and solvent extraction of soybean seeds**

211 The impact of DIC treatments at different operating parameters was assessed by measuring the
212 oil yields and kinetics through n-hexane extraction. Each treated sample was crushed using a
213 knife-grinder for 15 s at 10000 t/min. Particles of 0.4 mm in diameter were selected for
214 determining the oil yield based on Eq. 2 (ISO 659:1998 - Oilseeds; Determination of oil content).

$$Y_{oil} = \frac{\text{mass of extracted oil}}{\text{mass of dry basis (db)}} \quad Eq. 2$$

215 **2.4.2. Solvent extraction through Randall method**

216 Similarly, the differently DIC-treated soybeans were crushed. 3 g of soybean powder at 11%
217 moisture were placed in a Randall velp-148 using n-hexane at 55°C, for 6-h. Then, the extract
218 was evaporated under vacuum using a 6-L rotary evaporator (Genser Scientific Instruments
219 91541 Rothenburg ob der Tauber; Germany) at 40 °C until constant mass to recover hexane
220 Micelle and residue under a dry air stream (Ben-Youssef et al., 2017).

221 **2.4.3. Dynamic Maceration (DM)**

222 3 g powder of DIC treated soybeans and a volume of 40 mL of n-hexane were added to a 100-ml
223 Erlenmeyer flask of. The mixture was stirred at 200 rpm for 12 h at 55 °C (slightly lower than
224 the boiling temperature of hexane). To study the maceration kinetics, the samples were collected
225 at different extraction times and filtered through a 0.2 µm filter syringe of (PTFE filters). The
226 extracted oil was separated from the solvent, and, then quantified. The filtrates were evaporated
227 to a constant weight under nitrogen flow at ambient temperature. The extraction yield was
228 defined as the amount of the oil extracted from 100 g of matter. Each experimental run was
229 conducted at least in duplicate.

230 **2.5. Study and modeling of the extraction kinetics**

231 After the starting stage of superficial interaction of the solvent, and since n-hexane is capable of
232 greatly dissolving the vegetable oils, the two phenomena of solvent-solute diffusion within the
233 grain, and mass convection at the exchange surface, simultaneously occur. Since the extraction
234 process was made under high-speed stirring conditions, the external superficial transport was so
235 intensified that the extraction process briefly became Negligible External Resistance (NER). The
236 internal effective diffusion of the solvent-solute was the limiting process.

237 Thus, the solvent extraction kinetics was studied through the phenomenological model of
238 Coupled Washing/Diffusion (CWD). In these conditions, the internal mass transfer revealed the
239 whole extraction process. Subsequently, it was modeled based on similar Fick's law with the
240 effective diffusivity D_{eff} ($\text{m}^2 \text{s}^{-1}$) to expose the global internal liquid transfer (Fernández et al.,
241 2012). The mathematical solutions change depending on the specific conditions of the shape and
242 average size of the granules. In the present case, we assumed D_{eff} as a constant, independently on
243 the position within the grain and operation time. Crank solutions can then be adopted as series or
244 error functions for short diffusion times, or trigonometric series or Bessel functions for larger
245 time (Lebovka et al., 2016).

$$\frac{(Y(t) - Y_{t_0})}{(Y_{\infty} - Y_{t_0})} = \sum_{i=1}^{\infty} A_i \exp(-q_i^2 \tau) \quad \text{Eq 3}$$

246 By limiting Crank series to its first term, the logarithm transformation allows linearizing **Eq 3**, to
247 identify the slope K. The experimental results to use must excluding the data ranged between the
248 initial time ($t=0$) and t_0 assumed as a time that let the extraction be controlled by the diffusion.
249 Thus, the effective diffusivity D_{eff} is estimated from Eq. 4:

$$\ln\left(\frac{Y - Y_{t_0}}{Y_{\infty} - Y_{t_0}}\right) = f(t) = Kt + a \quad \text{Eq 4}$$

250 It is noteworthy that only the experimental results for $t > t_0$ were used in pursuing D_{eff} . By
 251 extrapolating this theoretical model towards the origin ($t=0$), an initial yield Y_0 usually higher
 252 than $Y_i=0$ should reveal the starting accessibility δY_s yield (g oil/g db):

$$\delta Y_s = Y_0 - Y_i = Y_0 - 0 \quad \text{Eq.5}$$

253 the difference between the initial value of yield ($Y_i=0$) and the theoretical value calculated from
 254 the diffusion model divulges the starting accessibility to link with the initial superficial
 255 interaction (Bouallegue et al., 2015). The starting accessibility δY_s unfolds the oil quantity issued
 256 from the interaction between the solvent and the interaction surface of the product, normally
 257 carried out over a very short time.

258 **2.6. Assessments and Characteristics**

259 **2.6.1. Chromatography measurements**

260 Gas Chromatography GC was used to identify and quantify the free fatty acids. GC was Agilent
 261 Gas Chromatography 19091S-433 (Kyoto, Japan) well-appointed with a specific column (HP-
 262 5MS 30m*350 μ m*0.25 μ m). The oven followed a special temperature program starting at 155
 263 °C to rise 230 °C in 2 min to keep at a constant level of 240 °C for 50 min. Helium was the
 264 carrying gas used at 0.37 m/s as speed.

265 In an adapted vial, we set a sample of 15 μ l of oil, added 20 μ l of Tolbutamide Methyl-
 266 Hydroxylase (TMH) with 1 ml of solution (50/50) of methanol-chloroform. Successively,
 267 samples of 1 μ l each were automatically injected in a split mode of 1/200. The system was set to
 268 identify the fatty acids and their concentrations were measured through the integration of the
 269 peaks using an internal program library (de Carvalho Claudio Guilherme Portela et al., 2018).

270 High-Performance Liquid Chromatography HPLC we used for the quantification of tocopherols
 271 was a Shimadzu system fitted with two (LC-10AD) distribution pumps, an FL/FR-10AXL
 272 detector, an automatic injector of 25 μ l per sample, and, finally, an Altimma RPC-18 separation
 273 column of 250x4.6 mmx5 μ m (Associates Inc.). Acetonitrile/methanol solution of 75:25 was
 274 injected as the mobile phase at 1 ml/min as a flow rate. We stabilized the temperature of the
 275 column at 250 °C, set the fluorescence detector at 298 nm and 344 nm for excitation and
 276 emission wave, respectively, and selected the total separation time at 40 min. The α , β , and γ

277 tocopherol fractions were identified, and their concentrations were measured based on the
278 retention time set with a calibration range of the different standards.

279 **2.6.2. Scanning Electron Microscopy**

280 The assessments of microstructural changes of each soybean material depending on its
281 pretreatment way were achieved through an environmental scanning electron microscopy (SEM)
282 (JSM—5410/5410 LV JEOL Ltd., Tokyo, Japan), using the method defined by Mounir et al.
283 (2015). Each type of soybean was positioned using carbon adhesive on covered support and the
284 measurements were performed under a vacuum of 7 Pa and an accelerating voltage of 20 kV.

285 **2.6.3. Design of Experiment DoE**

286 Among various possibilities, we opted for the Box Behnken as Design of Experimental DoE to
287 study the effect of DIC process parameters of saturated dry steam pressure (P) and treatment
288 time (t) on the oil extraction yields and kinetics. The RSM (response surface methodology) was
289 chosen as a nearly rotatable second-order design, with a five-level complete design consisting of
290 the center point, and the factorial and star points, normally identified as the points of the edges
291 from a cube. Indeed, while our objective was to study and optimize the DIC conditions, a 5-level
292 central composite design (CCD) was the most adequate design of experiment (DoE). This
293 implies a second-order empirical regression model to set the relationship between the controlled
294 independent variables of P and t input and the chosen responses. The selection of the
295 independent variables of the DIC processing parameters and their variation ranges was based on
296 the data issued from the literature and the accumulated 30-year know-how of our research team.
297 Additionally, some preliminary experiments were carried out in this objective. The dependent
298 variables chosen as responses were the following: oil yield Y_{∞} , time to reach the same yields as
299 cracked Cr soybeans do in 160 min, starting accessibility δY_s , and effective diffusivity D_{eff} .

300 To set the response surface methodology (RSM) to perform the statistical study of the data
301 issued from our experiments, Statgraphic software has been able to define the Analyses Of
302 Variance (ANOVA) and p-value of 5% probability level ($p < 0.05$), and display on Pareto charts
303 to highlight the impacts of independent variables on each response. We reviewed the fit-degree
304 of the model to quote the lack of correspondence, including Fisher-test value and p-value, and
305 also built up the response surface from a quadratic empirical equation, while defining R^2 .

306 **3. Results and discussion**

307 **3.1. Yield extraction versus DIC processing parameters**

308 The samples of different mechanically pretreated cracked (C), flaked (CF), and expanded
309 (CFEx) soybeans, as well as the respective DIC textured forms (C-DIC) and (CF-DIC) were all
310 submitted to the solvent extraction at the same conditions (Randall **velp-148** using n-hexane at
311 55 °C, for 6-h). The values of oil yields expressed in (g oil/g db) were shown in Table 1.

312 Table 1. Oil yields for Randall's 6-h extraction of soybean samples differently pretreated: (C) cracked,
313 (CF) flaked and expanded (CFEx) seeds, possibly followed by DIC texturing operation (C-DIC), (CF-
314 DIC) and (CFEx-DIC) soybeans. Where P and t are the DIC steam pressure (MPa) and treatment time (s),
315 respectively; Y_{oil} is Oil yields (g oil/g db: dry basis); RIE is the Ratio of Improvement of oil extraction=
316 $Y_{oil-DIC}/Y_{oil-RM}$ (%); CPA represents the Central Points (DIC 1, 4, 7, 10, and 13).
317

318 In the same solvent extraction conditions, following the two mechanical pretreatments of
319 cracking **C** and cracking-flaking **CF**, the soybeans had increased oil yields. However, as we
320 expected, DIC texturing systematically allows the oil availability and yields to be noticeably
321 greater than cracked **C** and flaked **CF** soybeans, by 15.55% and 7.34%, respectively.

322 **3.2. Statistical analysis of oil yields**

323 To express and evaluate the kinetic parameters of solvent oil extraction (yields, D_{eff} , and δY_s),
324 versus the DIC texturing parameters such as the saturated dry steam pressure and processing
325 time, we used Statgraphics software in analyzing the effect plots, estimating the surface
326 response, defining the variance, and fitting the empirical equation of the experimental results.

327 We also systematically introduced the normalized Pareto diagrams. It is a convenient way to
328 possibly recognize the significant effects of the linear, quadratic, and interaction DIC parameters
329 P and t. The statistical results are represented in Figure 5. In the case of oil yields, it confirmed
330 that P and t had the highest significant effects with a p-value of 0.05.

331 Similarly, Eq.6 gives the second-order empirical regression model of the extraction yields Y_{oil}
332 (expressed in g oil/g db) for cracked soybeans, versus the DIC processing parameters; P in MPa
333 and t in s with $R^2=81.72\%$.

$$Y_{oil_C} = 0.2088 + 0.1443P - 0.00041t - 0.218P^2 + 0.001Pt + 9.2 \cdot 10^{-7}t^2 \quad \text{Eq.6}$$

334 The DIC parameters for cracked and flaked soybeans were optimized at 0.48 MPa for 105 s and
335 0.49 MPa and 96 s, with oil yields of 0.2452 and 0.24215 g oil/g db, which presents an increase

336 of 0.033 and 0.00664 g oil/g db or about 14.56% and 3% higher than initial C and CF beans,
337 respectively.

338 Figure 5 also shows a higher significant effect of the DIC processing parameters on the
339 extraction yield of flaked soybeans CF with a regression coefficient $R^2=89.13$ and 0.05 as p-
340 value. Oil availability $Y_{oil,CF}$ was given as an empirical regression model in Eq.7:

$$Y_{oil_{CF}} = 0.2091 + 0.11P - 0.000015t - 0.15P^2 + 0.00035Pt + 1.7 \cdot 10^{-6}t^2 \quad \text{Eq.7}$$

341 By DIC texturing the flaked soybeans at 0.49 MPa for 96 s, oil yield reached 0.2452 g oil/g db
342 revealing an increase of 0.017 g oil/g db higher than the simply flaked soybeans.

343 In both cases C and CF, the optimal saturated steam pressure was identical (0.49 MPa).
344 However, the optimal treatment times were different (105 and 96 s, respectively). This is
345 obviously because the DIC treatment time depends on the thickness and the moisture and heating
346 diffusivities.

347 The study confirms an improvement in extraction yields and kinetic extraction essentially
348 generated by a change in the technological aptitude of the grains treated with DIC.

349 *Figure 5. Impact of DIC Parameters on oil yields in cracked C and flaked CF soybean seeds*

350 Based on the results of the statistical study (separate effects Figure 5), variation in extraction
351 efficiencies as a function of the calculated amount of self-sprayed steam (Eq 1), and for a
352 pressure variation interval of [0.27, 0.63] confirms a positive correlation represented in Figure 6
353 for both the crushed and crushed - flaked soybean study.

354 *Figure 6: Correlation of the amount of the autovaporized vapor and oil extraction yield*

355 **3.3. Statistical analyses of the impact of DIC on extraction kinetics**

356 To study the impact of various modifications of seed structure through the pretreatment
357 operations (cracking, flaking, and expansion) and DIC texturing technology on the kinetics of oil
358 extraction by n-hexane at each pretreated level of soybean, a study of the extraction yields as a
359 function of time was conducted. The purpose of the dynamic maceration extraction was to
360 compare the TRY (time required to achieve the same yields as cracked C beans) for CF beans
361 those from expander and suitable DIC-textured seeds (under optimal DIC conditions). The
362 experimental results (Figure 7) show that regardless of the level of pretreatment; Cracked,
363 flaked, or expanded, DIC texturing greatly accelerated the extraction process. Thus, while for
364 cracked soybeans, the oil yield of 0.207 g of oil/g db was reached in 160 min, a DIC-texturing

365 made it possible to obtain this level in only 35 min (TRY = 35 min), meaning three times faster
366 extraction capacity. For flaked soybean seeds, the value of **TRY** was almost similar to the
367 cracked beans, thus 160 min. The effect of expander was to reduce this **TRY** time to almost 120
368 min, which is about 1.33 times faster solvent extraction than for cracked or cracked/flaked beans.
369 It is worth highlighting that the optimized DIC texturing is about 4.7 times faster for solvent
370 extraction (TRY=35 min) (Table 2).

371 Based on preliminary results and data issued from literature, the operations of the kinetics were
372 considered for 160 min the differently treated seeds. In the cases of solvent extraction process of
373 soybean, the highly stirred dynamic maceration was defined as a **NER Negligible External**
374 **Resistance** process, which means that the resistance of the solute oil-in-solvent transport
375 compared to the internal diffusion transfer was so weak that the properly said unit operation was
376 methodically controlled by the internal mass transfer. We thus reviewed these different cases of
377 kinetics through the Coupled Washing Diffusion **CWD** phenomenological model. With beans
378 assumed to be spherical form, the slope K (s^{-1}) issued from Eq 4 was used to identify the
379 effective diffusivity D_{eff} (m^2/s). Figure 7 shows its evolution versus the different processing
380 parameters of DIC.

381 [Figure 7. Impact of Instant Controlled Pressure-Drop DIC texturing on oil extraction kinetics of different pretreated](#)
382 [soybeans.](#)

383 From the diffusion theoretical curve, the starting accessibility was estimated as the yield of
384 extracted oil before the diffusion process became the limiting phenomenon. It is projected by
385 subtracting the inferred theoretical diffusion value Y_0 and the experimental value Y_i at $t=0$ (Eq.
386 5). **The starting accessibility δY_s** divulges the oil extracted through a direct interaction between
387 the grain surface and the solvent.

388 [Table 2. Fundamental Parameters issued from the phenomenological model of Coupled Washing](#)
389 [Diffusion CWD: the effective diffusivity \$D_{eff}\$ and starting accessibility \$\delta Y_s\$, while TRY is the “Time](#)
390 [needed to get the same reference of Yield from cracked beans”.](#)

391 Cracked-flaked CF soybeans normally had lower effective diffusivity ($D_{eff}=1.7$ against $3.2 \cdot 10^{-12}$
392 m^2/s), and higher starting accessibility δY_s (0.1487 against 0.1242 g oil/g db) than only cracked
393 C seeds. Certainly because of the decrease in porosity and an increase in exchange surface that
394 flaking normally fetches.

395 Oppositely, by creating much higher porosity than expansion did, DIC-texturing resulted in
396 twice more effective diffusivity while the starting accessibility was 60% higher (Figure 7 and
397 Table 2). These experimental data demonstrate that DIC texturing is a crucial pretreatment,
398 which marks much better than expander, the higher diffusivity and apparent exchange surface.

399 Specifically, the experiments confirmed what was predicted from the fundamental instantaneous
400 thermodynamic analyses. Thus, although similar in terms of saturated steam pressure and
401 process temperature, the DIC texturing resulted in a greater intensification of the solvent
402 extraction. The greater the structure modification, the higher the porosity and tortuosity, and
403 thus, the better the technological capabilities of DIC textured soybeans. This was confirmed by
404 scanning electron microscopic (SEM) observations in (Figure 8) to better recognize the
405 biological microstructure and textural modifications in each pretreated group of seeds.

406 *Figure 8. Impact of expander and Instant Controlled Pressure-Drop DIC texturing on the structure of Crushed -*
407 *flaked soybeans by scanning electron microscopy SEM observation: a) nonthermal textured seeds; b) Expander*
408 *treated seeds; c) DIC textured seeds; d) DIC-textured of Expander treated seeds.*

409 *The microstructure of Crushed - flaked soybeans presents effectively organized cells, while*
410 *expander thermal texturing implies slight modifications of these cells. The impact of DIC was*
411 *much more important in opening the cells and, thus, increasing the oil kinetic extraction. The*
412 *most expansion impact became from coupling both expander and DIC treatments.*

413 Moreover, depending on the severity of each operation, possible broken-wall cells may occur.
414 However, the DIC stands out by reaching a more expanded structure, getting, just after
415 decompression, lesser equilibrium temperature (usually 33 °C instead of 100 °C) that is to say no
416 further cooling stage is needed and requiring much lower energy consumption. DIC allows
417 increasing oil yields, quality preservation, and effective kinetics.

418 **3.4. Gas Chromatography analyses and soybean oil quality versus DIC treatment** 419 **conditions**

420 *Gas chromatography was used to identify and quantify the fatty acid compounds of the different*
421 *oil samples.* Table 3 presents the two groups of saturated and unsaturated lipids. The amounts of
422 the palmitic acid (C16:0), stearic acid (C18:0), and arachidic acid (C20:0), which are the
423 saturated fatty acids, were preserved after DIC treatment at their initial percentages of 11%,
424 4.5%, and 0.4%, respectively. The same behavior was observed for the unsaturated fatty acids
425 with oleic acid (C18:1), linoleic acid (C18:2), and linoleic (C18:3) whose percentages were
426 24.1%, 53%, and 7%, respectively. In optimized DIC texturing conditions, the yield
427 enhancement was accompanied by substantial preservation of the oil quality.

428 *Table 3. Gas Chromatography analyses of Fatty acid distribution and tocopherol compounds and contents*
429 *for raw material, Expander-treated, and differently DIC textured soybeans.*

430 Such a similarity in the lipid profiles of oil extracted from raw material (RM) and DIC-textured
431 soybeans allowed increasing yields, enhancing the kinetics, and preserving the oil and cake
432 quality.

433 Moreover, the data (Table 3) based on a comparative determination of the tocopherols content
434 (δ , γ , and α), show a slightly-significant variation according to the differently DIC treated
435 soybeans. These modifications inside the values of tocopherol compounds; δ , γ , and α occurred
436 but without any negative impact on total tocopherol content. Moreover, as usual, optimized DIC
437 treatment allows preserving or even increasing the antioxidant availability in various types of
438 plant-based materials, because of the new porous texture performed at high temperature for a
439 very short time (High-Temperature/Short-Time HTST DIC process). The fact that Expander
440 treatment implies much larger thermal treatment time and resulting in much lower porosity than
441 DIC perfectly explains this behavior.

442 As confirmed in the work of (Mannai et al., 2019) and (Jablaoui et al., 2018), a high-
443 temperature/short-time treatment, a suitably optimized DIC involves significant conservation of
444 the biochemical composition, even for thermal sensitive molecules. Indeed, since the high-
445 temperature treatment is achieved in a very short time and the final low temperature is
446 immediately reached thanks to the vacuum level, DIC treatment didn't imply any notable
447 biochemical degradation.

448 **3.5. Industrial application: Energy balance and environmental impact**

449 The integration of DIC in the industrial process of solvent extraction of soybean oil results in a
450 great reduction of the operation time and an overall increase in oil yield with preservation of the
451 quality. Thus, for a total quantity of one ton (1000 kg) of raw material, the oil yields become 177
452 kg for DIC-soybeans in about 35 min and 163 kg for expander-soybeans in about 120 min
453 against 154 kg in 160 min for nonthermal textured cracked soybeans (Table 4).

454 The “Expander” and DIC operations need energy consumptions, which can be issued from Eq. 1.
455 They both depend on the treatment temperatures and oil and water contents:

$$Q = m(a c_{p,d} + X_{wb} c_{p,w} + Y_o c_{p,o})(T_t - T_f) \quad \text{Eq.8}$$

456 where m is the total considered weight of soybeans, a , X_{wb} , and Y_o are the relative percentages of
457 dry material, moisture content, and oil, respectively; their values depend on the pretreatment
458 types (nonthermal textured, and Expander and DIC textured beans), the specific heats are $c_{p,d}=$
459 1.050 , $c_{p,w}= 4.180$, and $c_{p,o}=2.365$ $\text{kJ kg}^{-1} \text{K}^{-1}$ of dry material, water, and oil, respectively; and
460 latent evaporation values are 2107 and 2085 kJ kg^{-1} at 152 and 159 $^{\circ}\text{C}$, respectively.

461 Table 4. Main data of improvement of oil yields and energy consumption using DIC and expander texturing ways

462 This table allows justifying the interest of using DIC texturing as an intensification way of
463 solvent extraction of soybean oil. The environmental impact is directly correlated with energy
464 consumption.

465 **4. Conclusion**

466 It is well known that the natural structure of plant organs (fruit, vegetables, leaves, roots,
467 seeds...) has a too weak technological aptitude versus numerous unit operations of extraction.
468 Adequate pretreatments of the natural structure may, if well-defined, lead to increasing the yields
469 and enhancing the operation kinetics while preserving the final oil and cake quality. Hence, in
470 this way for the specific case of solvent extraction of soybean oil, a new trend was introduced at
471 the industrial scale; this has been the pretreatment through the texturing of cracked and/or
472 cracked/flaked beans. Another texturing process was also separately studied; the Instant
473 Controlled Pressure-Drop DIC. The current research aimed to mainly compare the impact of
474 these both texturing operations of expander and DIC before the n-hexane solvent extraction
475 resulted in so greater kinetics. The “expander” allowed the oil yield of cracked soybean to
476 increase from 209.6 in 160 min to 224.4 mg oil/g db in 120 min, while DIC texturing resulted in
477 a yield of up to 248.95 mg oil/ g db in about 35 min.

478 The phenomenological modeling of coupled washing diffusion CWD allowed defining and
479 measuring the effective solvent diffusivity D_{eff} as well as the starting accessibility δY_s of N-
480 hexane solvent extraction of soybean oil in the different studied cases. D_{eff} and δY_s were
481 recognized as $0.022 \cdot 10^{-10} \text{ m}^2/\text{s}$ and 124.2 mg oil/g db, respectively for the non-textured cracked
482 beans. After the expander treatment, they increased until $0.027 \cdot 10^{-10} \text{ m}^2/\text{s}$ and 166.3 mg oil/g db,
483 respectively. The specific texturing by DIC resulted in much higher D_{eff} and δY_s with $0.065 \cdot 10^{-10}$
484 m^2/s and 199.6 mg oil/g db, respectively.

485 GC and HPLC assessments of the oil established that the fatty acid profiles and tocopherol
486 contents for the DIC-textured soybeans were almost similar to the untextured seeds. This reveals
487 that optimized DIC texturing is correlated with the absence of any significant biochemical
488 degradation. Indeed, although DIC texturing implies the use of high-temperature saturated steam,
489 it is performed in so specific short time that it results in the preservation of the oil quality in
490 terms of fatty acids' compounds and tocopherol content. This study proves the ability of DIC
491 treatment to preserve the extracted oil quality.

492 The present technical and scientific study as well as the first approach of the economic analysis
493 shows that DIC can advantageously replace the mechanical flaking and thermal texturing
494 Expander. The well-optimized DIC allows reducing the extraction time, increasing yield, and
495 preserving the quality of finished oil and cake products with low energy consumption and
496 positive environmental impact.

497 **Nomenclatures and abbreviations:**

a	Percentage of dry matter in soybean
C:	Soybean cracking (C),
CCD:	Central composite design of experiments
CF:	Soybean cracking-flaking (CF),
CFEx:	Soybean cracking-flaking-expanding (CFEx)
$c_{p,d}$:	Specific heat capacity of dry matter ($c_{p,d} = 1050 \text{ J kg}^{-1}\text{K}^{-1}$)
$c_{p,o}$:	Specific heat capacity of soybean oil ($c_{p,o} = 2385 \text{ J kg}^{-1}\text{K}^{-1}$)
$c_{p,w}$:	Specific heat capacity of water ($c_{p,w} = 4180 \text{ J kg}^{-1}\text{K}^{-1}$)
CPA:	Central Point Average,
CWD:	Coupled Washing/Diffusion phenomenological kinetic modeling where washing is the interaction between the solvent and surface.
D_{eff} :	Effective solvent diffusivity (m^2s^{-1})
DIC:	Instant Controlled Pressure-Drop
DM:	Dynamic Maceration
DoE:	Design of Experiments
ΔT_{pd} :	Variation of temperature through the pressure drop for Expander or DIC texturing processes
δY_s :	Value of starting accessibility expressed en (kg oil/kg dry basis db)
GC:	Gas chromatography
HPLC:	High performance liquid chromatography
HTST:	High-Temperature/Short-Time
L:	Latent heat of vaporization at T_t (J kg^{-1})
m_d :	Amount of dry matter (kg)
m_v :	Amount of vapor generated by autovaporization (kg) through the pressure drop for Expander or DIC texturing processes
NER:	Negligible External Resistance hypothesis for phenomenological modeling.
P:	Saturated dry steam pressure (MPa) in DIC treatment
PTFE:	Polytetrafluoroethylene membrane Filters
Q_c	Amount of cooling energy for Expander or DIC texturing processes
Q_h	Amount of heating energy for Expander or DIC texturing processes
$q_i^2\tau$:	$= \pi^2 \frac{D_{eff}}{4L^2} (t - t_0)$ for infinite plate and $= -\frac{\pi^2 D_{eff}}{L^2} (t - t_0)$ for spherical form
Q_t	The total amount of energy used with Expander or DIC texturing processes
RM:	Raw Material,
RSM:	Response surface methodology
t:	DIC treatment time (s)
T:	Temperature ($^{\circ}\text{C}$),
T_a :	Ambient temperature
T_c	Post cooling temperature after Expander or DIC texturing processes
T_f	Final Equilibrium temperature of Expander (100 kPa) or DIC texturing process (5 kPa)
TRY:	The time required to reach the same cracked beans C as the reference Yield (min)

T_t : Treatment temperature just before dropping the saturated steam pressure ($^{\circ}\text{C}$)
 $W_{p,\text{H}_2\text{O}}$: Moisture content dry basis (kg H_2O /kg db)
 X_{wb} : Percentage of Moisture content in soybean
 Y_{∞} : Oil yield at infinite time
 $Y(t)$: Oil yields versus extraction time
 Y_0 : The calculated value of oil yield at $t=0$
 Y_i : Value of oil yield at $t=0$; here $=0$
 Y_o : Percentage of oil in soybean
 Y_{oil} : Oil yield= Extract Oil Mass/Dry Basis (db) Mass (kg Oil/kg db)
 Y_{i0} : Oil yield at a time t_0

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501

502 **References**

- 503 Allaf, T. and Allaf, K. (Eds.), 2014. Instant Controlled Pressure Drop (D.I.C.) in Food
504 Processing: From Fundamental to Industrial Applications, Food Engineering Series.
505 Springer-Verlag, New York.
- 506 Allaf, T., Zougali, B.B., Van Nguyen, C., Negm, M., Allaf, K., 2014. DIC Texturing for Solvent
507 Extraction, in: Allaf, T., Allaf, K. (Eds.), Instant Controlled Pressure Drop (D.I.C.) in
508 Food Processing: From Fundamental to Industrial Applications, Food Engineering Series.
509 Springer New York, New York, NY, pp. 127–149. https://doi.org/10.1007/978-1-4614-8669-5_7
- 511 Ben Amor, B., Allaf, K., 2009a. Impact of texturing using instant pressure drop treatment prior
512 to solvent extraction of anthocyanins from Malaysian Roselle (*Hibiscus sabdariffa*). *Food*
513 *Chem.* 115, 820–825. <https://doi.org/10.1016/j.foodchem.2008.12.094>
- 514 Ben Amor, B., Allaf, K., 2009b. Impact of texturing using instant pressure drop treatment prior
515 to solvent extraction of anthocyanins from Malaysian Roselle (*Hibiscus sabdariffa*). *Food*
516 *Chem.* 115, 820–825. <https://doi.org/10.1016/j.foodchem.2008.12.094>
- 517 Ben-Youssef, S., Fakhfakh, J., Breil, C., Abert-Vian, M., Chemat, F., Allouche, N., 2017. Green
518 extraction procedures of lipids from Tunisian date palm seeds. *Ind. Crops Prod.* 108,
519 520–525. <https://doi.org/10.1016/j.indcrop.2017.07.010>
- 520 Bouallegue, K., Allaf, T., Besombes, C., Younes, R.B., Allaf, K., 2015. Phenomenological
521 modeling and intensification of texturing/grinding-assisted solvent oil extraction: case of
522 date seeds (*Phoenix dactylifera* L.). *Arab. J. Chem.*
523 <https://doi.org/10.1016/j.arabjc.2015.03.014>
- 524 Britz, S.J., Kremer, D.F., Kenworthy, W.J., 2008. Tocopherols in Soybean Seeds: Genetic
525 Variation and Environmental Effects in Field-Grown Crops. *J. Am. Oil Chem. Soc.* 85,
526 931–936. <https://doi.org/10.1007/s11746-008-1286-y>
- 527 Brumm, T., Hurburgh, C., Rippke, G., 2004. Quality of the 2004 Soybean Crop from the United
528 States. *Agric. Biosyst. Eng. Tech. Rep. White Pap.*
- 529 Chemat, F., Rombaut, N., Meullemiestre, A., Turk, M., Perino, S., Fabiano-Tixier, A.-S., Abert-
530 Vian, M., 2017. Review of Green Food Processing techniques. Preservation,
531 transformation, and extraction. *Innov. Food Sci. Emerg. Technol.* 41, 357–377.
532 <https://doi.org/10.1016/j.ifset.2017.04.016>
- 533 Cheng, M.-H., Sekhon, J.J.K., Rosentrater, K.A., Wang, T., Jung, S., Johnson, L.A., 2018.
534 Environmental impact assessment of soybean oil production: Extruding-expelling
535 process, hexane extraction and aqueous extraction. *Food Bioprod. Process.* 108, 58–68.
536 <https://doi.org/10.1016/j.fbp.2018.01.001>
- 537 de Carvalho Claudio Guilherme Portela, da Silva Matheus Felipe, Mandarin José Marcos
538 Gontijo, Grunvald Anna Karolina, Ramos Nilza Patrícia, Ribeiro José Lopes, de Paulo
539 Campos Godinho Vicente, 2018. Fatty Acid Profiles in Sunflower Grains During Storage
540 in Different Environments. *J. Am. Oil Chem. Soc.* 95, 61–67.
541 <https://doi.org/10.1002/aocs.12007>
- 542 Fernández, M.B., Perez, E.E., Crapiste, G.H., Nolasco, S.M., 2012. Kinetic study of canola oil
543 and tocopherol extraction: Parameter comparison of nonlinear models. *J. Food Eng.* 111,
544 682–689. <https://doi.org/10.1016/j.jfoodeng.2012.01.036>

- 545 Hammond, E.G., Johnson, L.A., Su, C., Wang, T., White, P.J., 2005. Soybean Oil, in: Bailey's
546 Industrial Oil and Fat Products. American Cancer Society.
547 <https://doi.org/10.1002/047167849X.bio041>
- 548 **Hernandez E. 2019. Production, Processing, and Refining of Oils. In Healthful lipids/Editors,**
549 **Casimir C. Akoh and Oi-Ming Lai. Taylor & Francis Group ISBN 13: 978-1-893997-51-**
550 **6 (hbk).ISO 659:1998 - Oilseeds -- Determination of oil content (Reference method)**
551 **[WWW Document], n.d. URL <https://www.iso.org/standard/23297.html> (accessed**
552 **4.5.18).**
- 553 Jablaoui, C., 2018. La texturation par détente instantanée [sic] contrôlée DIC dans le
554 développements [sic] de nouvelles opérations d'extraction d'huiles des graines
555 oléagineuses (phdthesis). Université de La Rochelle ; Université de Carthage (Tunisie).
- 556 Jablaoui, C., Besombes, C., Jamoussi, B., Ismail, H.B., Rhazi, L., Allaf, K., 2018. Phytosanitary
557 Decontamination of Crude Rapeseed Oil by Instant Multi-Flash Autovaporization (MFA)
558 3, 13.
- 559 Jablaoui, C., Zeaiter, A., Bouallegue, K., Jemoussi, B., Besombes, C., Allaf, T., Allaf, K., 2019.
560 8 - Instant controlled pressure drop as new intensification ways for vegetal oil extraction,
561 in: Chemat, F., Vorobiev, E. (Eds.), Green Food Processing Techniques. Academic Press,
562 pp. 221–244. <https://doi.org/10.1016/B978-0-12-815353-6.00008-2>
- 563 Johnson, L.A., Lusas, E.W., 1983. Comparison of alternative solvents for oils extraction. J. Am.
564 Oil Chem. Soc. 60, 229–242. <https://doi.org/10.1007/BF02543490>
- 565 Lebovka, N., Vorobiev, E., Chemat, F., 2016. Enhancing Extraction Processes in the Food
566 Industry. CRC Press.
- 567 Mannai, A., Jableoui, C., Hamrouni, L., Allaf, K., Jamoussi, B., 2019. DIC as a pretreatment
568 prior to ultrasonic extraction for the improvement of rebaudioside A yield and
569 preservation of vitamin B1 and B6. J. Food Meas. Charact. 13, 2764–2772.
570 <https://doi.org/10.1007/s11694-019-00197-2>
- 571 **Mark Ash and Dana Golden, July 2020. Oil Crops Outlook. USDA (United States Department of**
572 **Agriculture) No. (OCS-20g) 7 pp.**
- 573 Mat Yusoff, M., Gordon, M.H., Niranjana, K., 2015. Aqueous enzyme assisted oil extraction from
574 oilseeds and emulsion de-emulsifying methods: A review. Trends Food Sci. Technol. 41,
575 60–82. <https://doi.org/10.1016/j.tifs.2014.09.003>
- 576 Perez, E.E., Carelli, A.A., Crapiste, G.H., 2011. Temperature-dependent diffusion coefficient of
577 oil from different sunflower seeds during extraction with hexane. J. Food Eng. 105, 180–
578 185. <https://doi.org/10.1016/j.jfoodeng.2011.02.025>
- 579 Rosenthal, A., Pyle, D.L., Niranjana, K., 1996. Aqueous and enzymatic processes for edible oil
580 extraction. Enzyme Microb. Technol. 19, 402–420. [https://doi.org/10.1016/S0141-](https://doi.org/10.1016/S0141-0229(96)80004-F)
581 [0229\(96\)80004-F](https://doi.org/10.1016/S0141-0229(96)80004-F)
- 582 Zhang, F., Koseoglu, S.S., Rhee, K.C., 1994. Effects of expander process on the phospholipids in
583 soybean oil. J. Am. Oil Chem. Soc. 71, 1145–1148. <https://doi.org/10.1007/BF02675910>
- 584

Tables

Table 1. Oil yields for 6-h Randall extraction of different-pretreated soybean samples; cracked (C), flaked (CF), and expanded (CFEx) seeds, and their DIC textured forms (C-DIC), (CF-DIC), and (CFEx-DIC) soybeans; where P and t are the DIC steam pressure (MPa) and treatment time (s), respectively; Y_{oil} is Oil yields (g oil/g db: dry basis); RIE is the Ratio of Improvement of oil extraction = $Y_{oil-DIC}/Y_{oil-RM}$ (%); CPA represents the Central Points (DIC 1, 4, 7, 10, and 13).

.	DIC parameters		Cracked C seeds	Flaked CF seeds	Expanded CFE seeds
	P (MPa)	t (s)	Yield in g oil/g db (RIE %)		
Untreated samples:			0.20962 (100.00%)	0.23019 (100.00%)	0.2352 (100%)
DIC-CPA	0.45	70	0.2300± 0.0020 (109.71±0.95%)	0.2439± 0.0006 (105.95±0.25%)	-
DIC2	0.7	70	0.21597 (103.03%)	0.23352 (101.45%)	-
DIC3	0.45	120	0.24215 (115.52%)	0.24216 (105.20%)	-
DIC5	0.63	105	0.23145 (110.42%)	0.24713 (107.36%)	-
DIC6	0.63	35	0.22590 (107.77%)	0.23539 (102.26%)	-
DIC8	0.27	35	0.22839 (108.95%)	0.23559 (102.35%)	-
DIC9	0.27	105	0.22114 (105.49%)	0.23839 (103.56%)	-
DIC11	0.20	70	0.21719 (103.61%)	0.23505 (102.11%)	-
DIC12	0.45	20	0.22210 (105.95%)	0.23598 (102.52%)	-
Optimal DIC texturing			0.24221 (115.53%)	0.24520 (106.52%)	-

P: DIC saturated dry steam pressure (MPa); t: DIC processing time (s); Y_{oil} : n-hexane Oil yields (g oil/g db: dry basis); RIE (Ratio of Improvement of oil Extraction) = $Y_{oil-DIC}/Y_{oil-RM}$ (%); CPA: Central Points (DIC1, 4, 7, 10, and 13).

Table 2. Fundamental Parameters issued from the phenomenological model of Coupled Washing Diffusion CWD: the effective diffusivity D_{eff} and starting accessibility δY_s , while TRY is the “Time needed to get the same reference of Yield from cracked beans”.

Type of pretreatment of soybeans	D_{eff}	δY_s	TRY	R^2 of CWD model (%)
	Value ($10^{-12} \text{ m}^2/\text{s}$)	Value (mg oil/g db)	Value (min)	
Cracked soybeans	3.2 (100%)	124.2 (100%)	160 (100%)	86.57%
Flaked soybeans	1.7 (53%)	148.7 (120%)	160 (100%)	95.55%
Expander textured by Flaked soybeans	2.7 (84%)	166.3 (134%)	120 (75%)	98.64%
DIC textured Flaked soybeans	6.5 (203%)	199.6 (161%)	37 (22%)	98.84%

Table 3. Gas Chromatography analyses of Fatty acid distribution and tocopherol compounds and contents for raw material, Expander-treated, and differently DIC textured soybeans.

Fatty acids Treatments	Cracked bean oil		Flaked bean oil		Expander-bean oil
	RM	DIC OPT	RM	DIC OPT	RM
C16:0	10.96±0.61	11.02±0.58	11.01±0.72	10.87±0.48	10.83±0.51
C18:0	4.55±0.02	4.5±0.02	4.51±0.01	4.53±0.02	4.46±0.01
C18:1	24.13±1.31	24.04±1.62	23.81±1.57	24.76±2.01	24.02±1.42
C18:2	52.99±2.55	52.85±3.01	53.11±3.12	52.37±2.88	53.1±3.15
C18:3	6.95±0.04	7.07±0.05	7.12±0.03	7.1±0.05	7.19±0.06
C22:0	0.42±0.01	0.52±0.01	0.44±0.01	0.39±0.01	0.41±0.01
Delta (µg/g oil)	191.39±9.52	177.82±8.02	175.90±10.85	208.95±11.30	162.55±8.95
Gama (µg/g oil)	341.21±10.20	378.08±11.33	327.00±10.01	284.99±09.84	308.78±11.32
Alpha (µg/g oil)	48.38±1.51	46.84±1.32	36.28±2.01	34.58±1.85	40.14±2.10
Total Tocopherols (µg/g oil)	580.98±14.32	602.74±15.12	539.18±13.83	528.52±14.12	511.47±15.03

Table 4. Main data of improvement of oil yields and energy consumption using Expander and DIC texturing ways for a basis of m=1000 kg of soybeans; m_d , m_o , and m_w are the weight values of the dry material, oil content and moisture content, respectively.

	Pre-thermal texturing data													Energy Cost/Added kg oil (kWh kg ⁻¹ oil)
	m_o/m_d	m_d	m_o	m_w	T_a	T_t	T_f	T_c	ΔT_{pd}	Q_h	Q_c	Q_t	m_v	
			(kg)		(°C)					(kWh)			(kg)	
Non-textured	0.210	735.78	154.22	110	/	/	/	/	/	/	/	/	/	
Expander	0.224	727.89	163.11	110	20	159	100	33	59	62	30	92	46	10.35
DIC	0.249	712.60	177.40	110	20	152	33	33	119	60	0	60	92	2.57

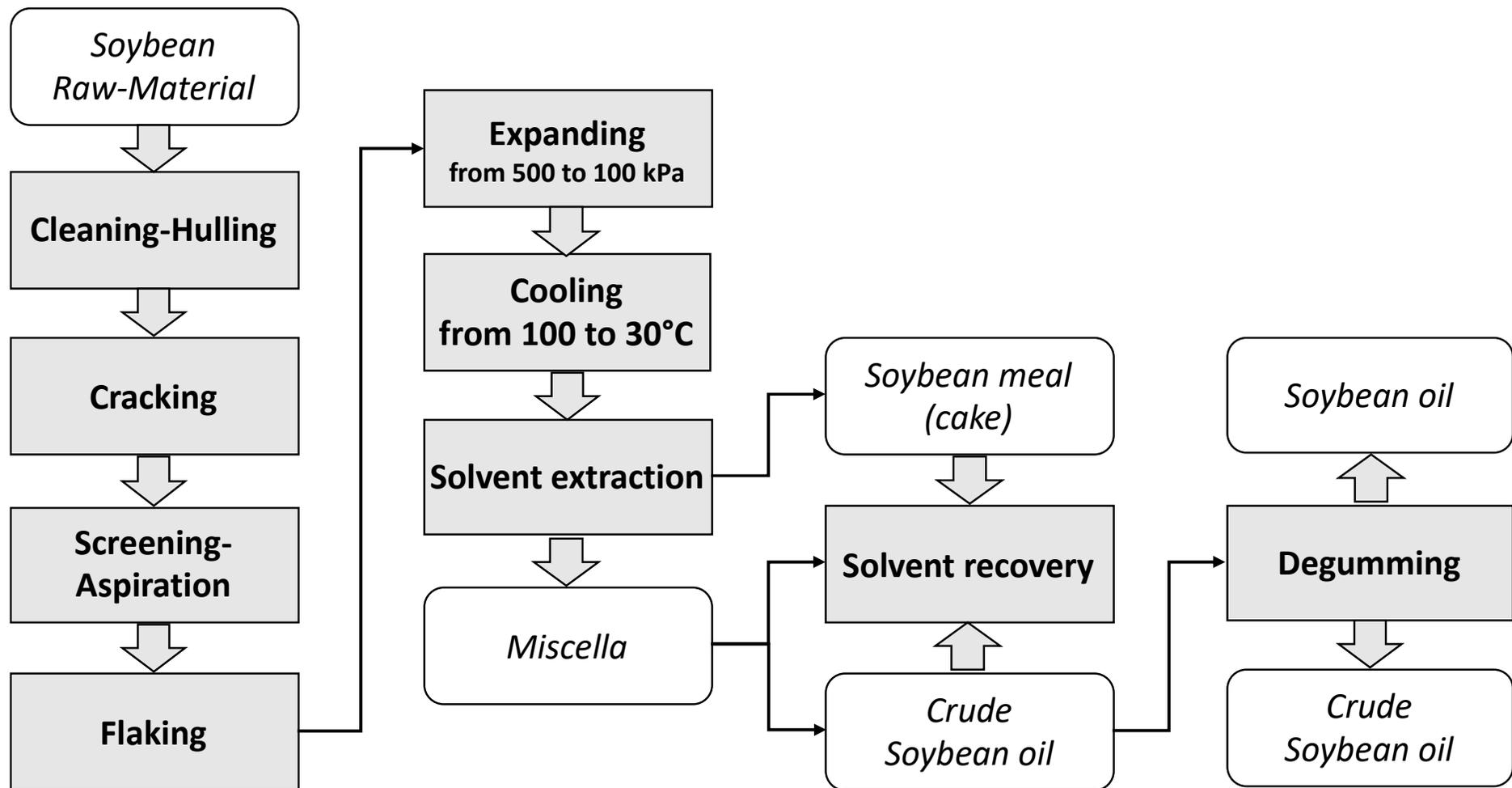


Figure 1. Industrial flow-chart of the solvent extraction of soybean oil

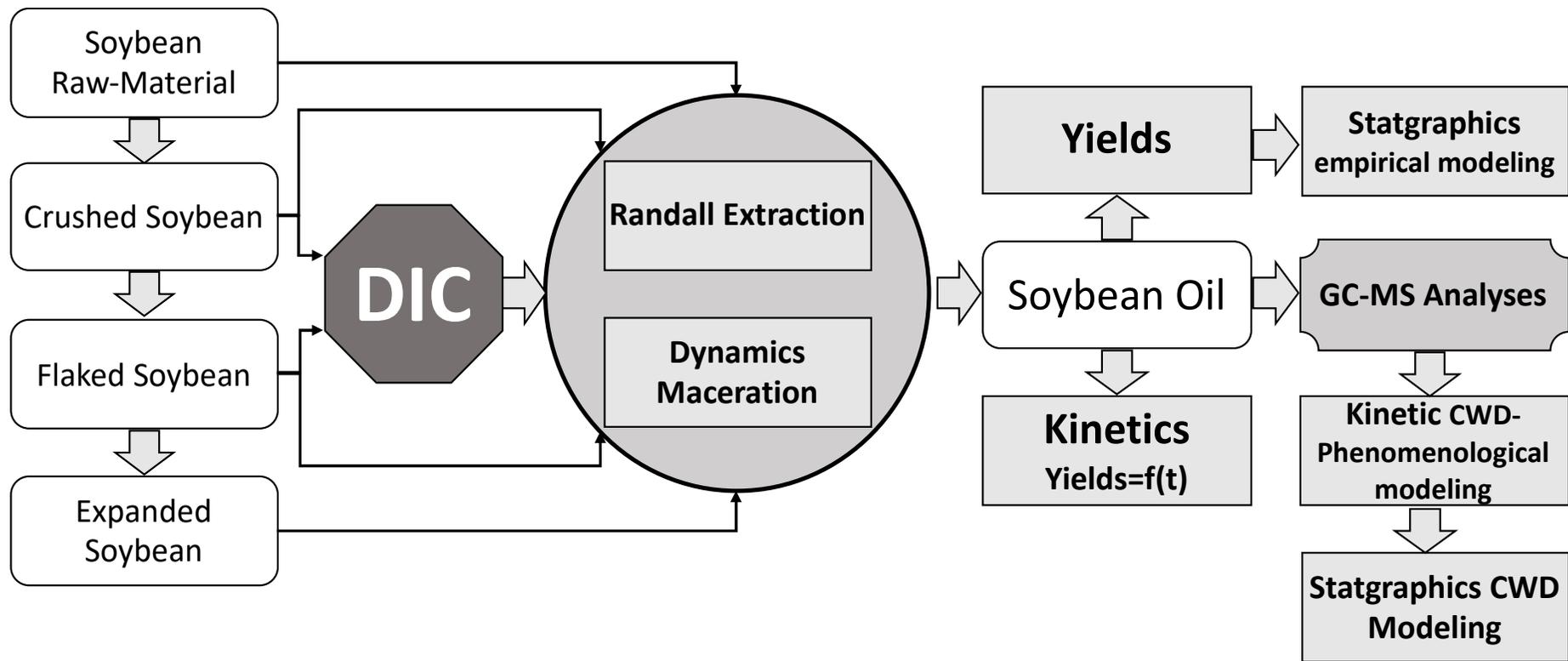


Figure 2. Diagram and experimental protocol of soybean oil extraction inserting a DIC texturing stage

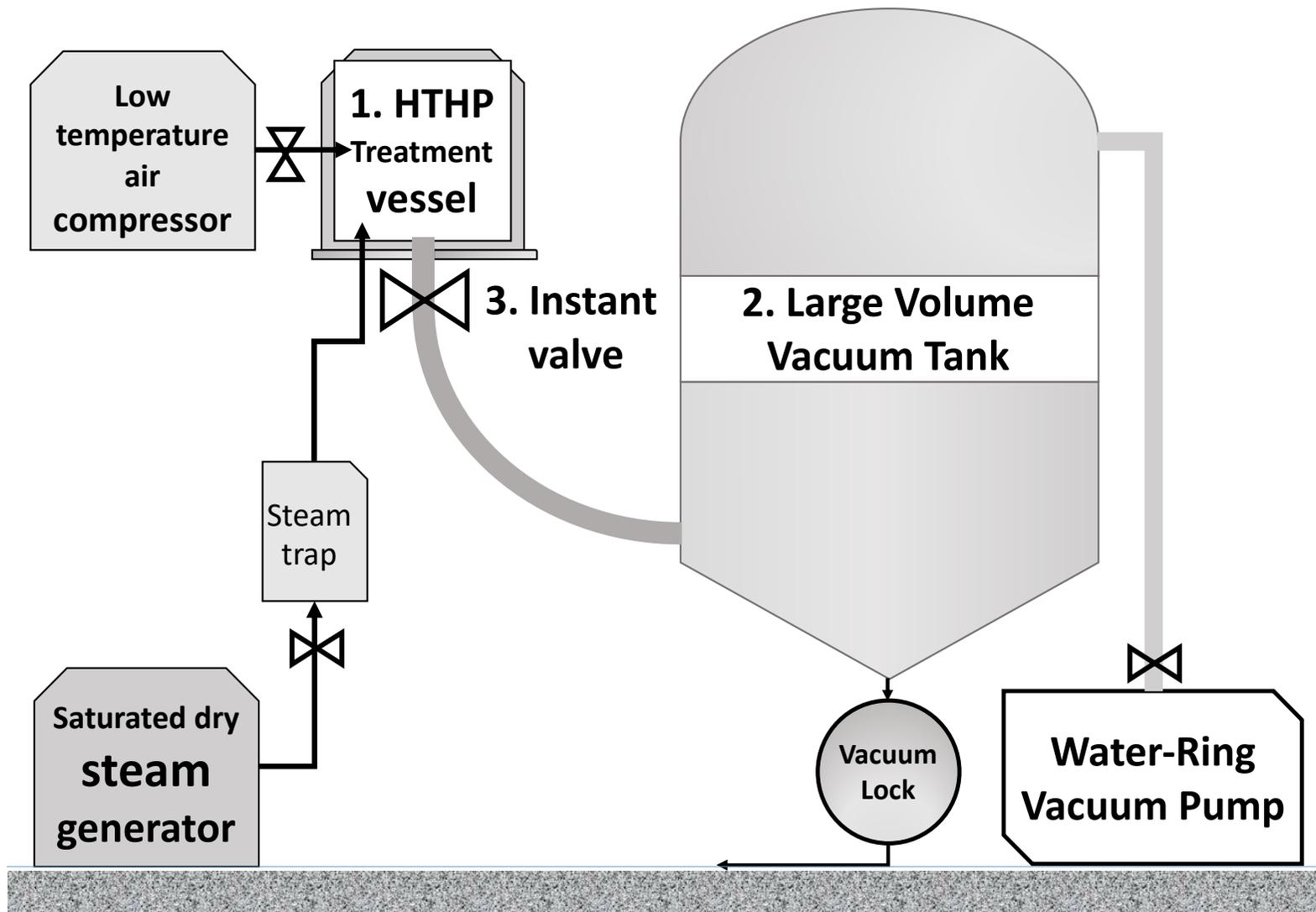


Figure 3. Schematic diagram of DIC laboratory scale unit: 1/ Treatment vessel; 2/ Vacuum tank; 3/ Large section-instant opening valve; 4/ water-ring vacuum pump; 5/ Saturated steam generator with condensed water trap.

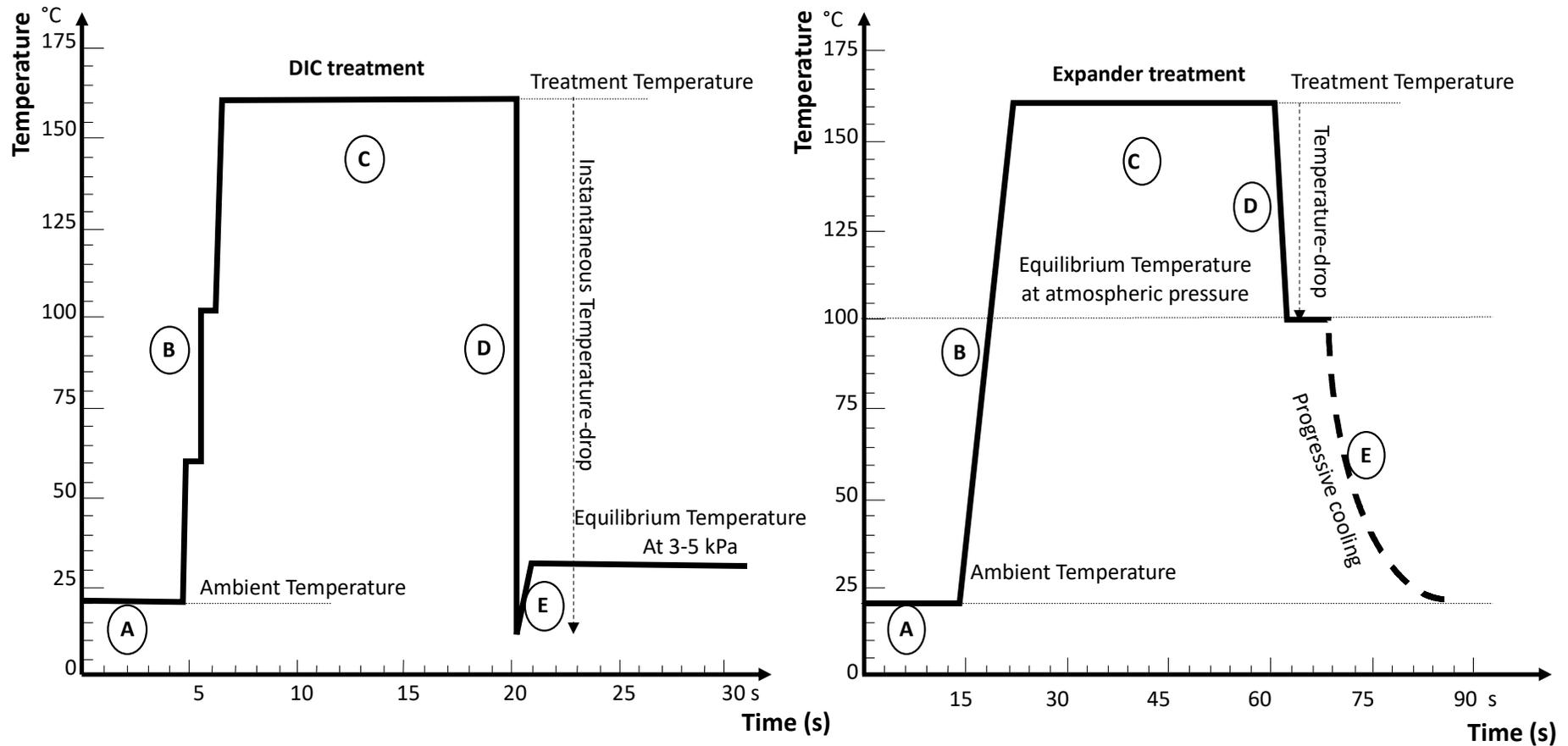


Figure 4. Stages of DIC and expander treatments, respectively: A/ Grains to set in the treatment vessel to immediately connect with the vacuum tank (only for DIC); B/ Saturated dry steam to inject till reaching the desired pressure and temperature treatment levels; C/ High pressure/high temperature to maintain for dozens of seconds (for DIC) and some minutes (for expander); D/ Pressure to abruptly drop towards a vacuum (3-5 kPa for DIC), and towards the atmospheric pressure (for expander); E/ release from the vacuum towards the atmosphere pressure (for DIC) and a cooling stage from 100 °C towards an almost ambient temperature,(for expander).

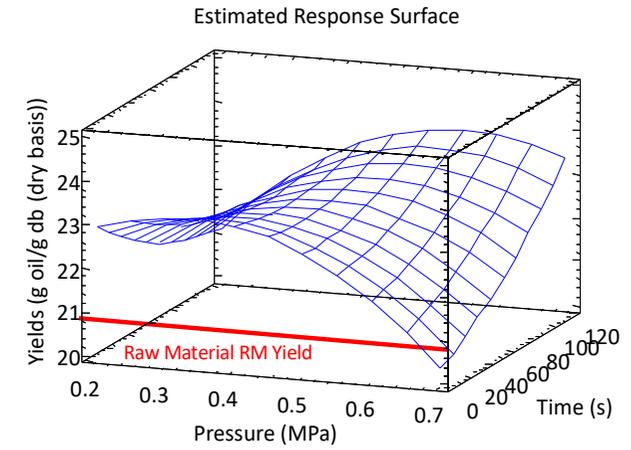
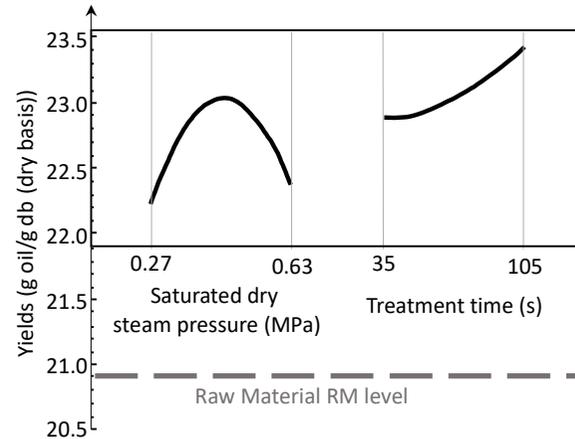
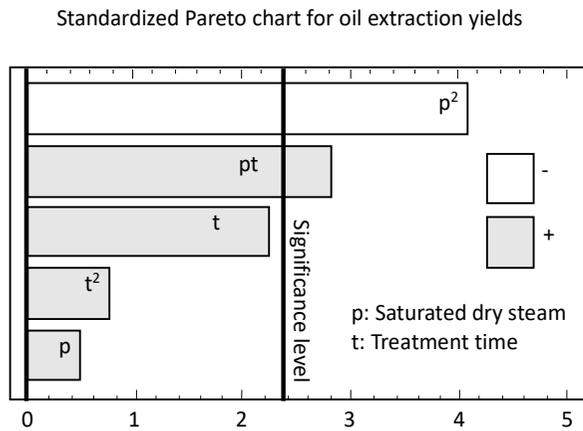


Fig.5-a: Cracked soybean seeds (C)

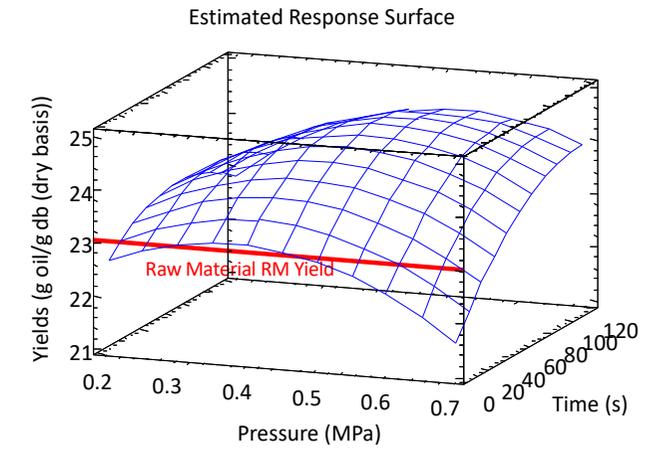
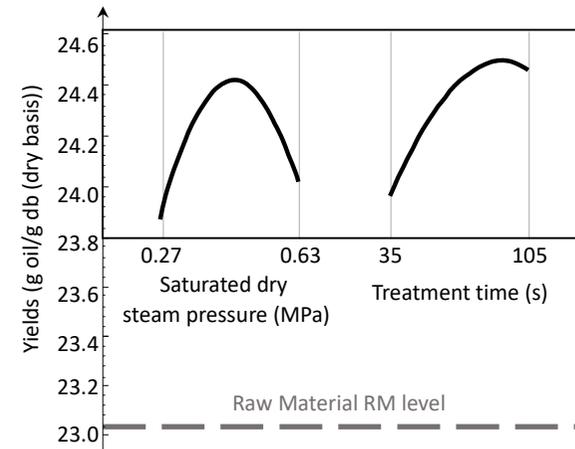
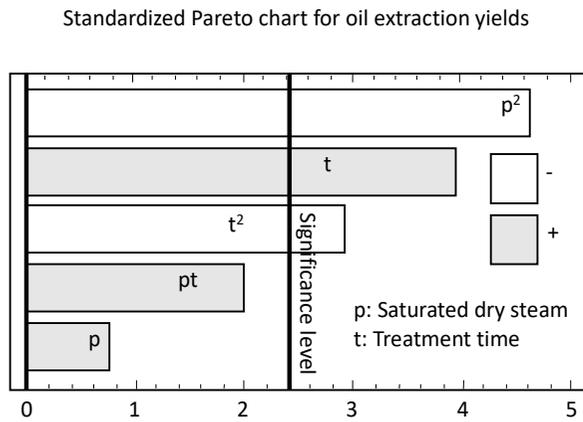


Fig.5-b: Flaked soybean seeds (CF)

Figure 5. Impact of DIC Parameters on oil yields in cracked C and flaked CF soybean seeds

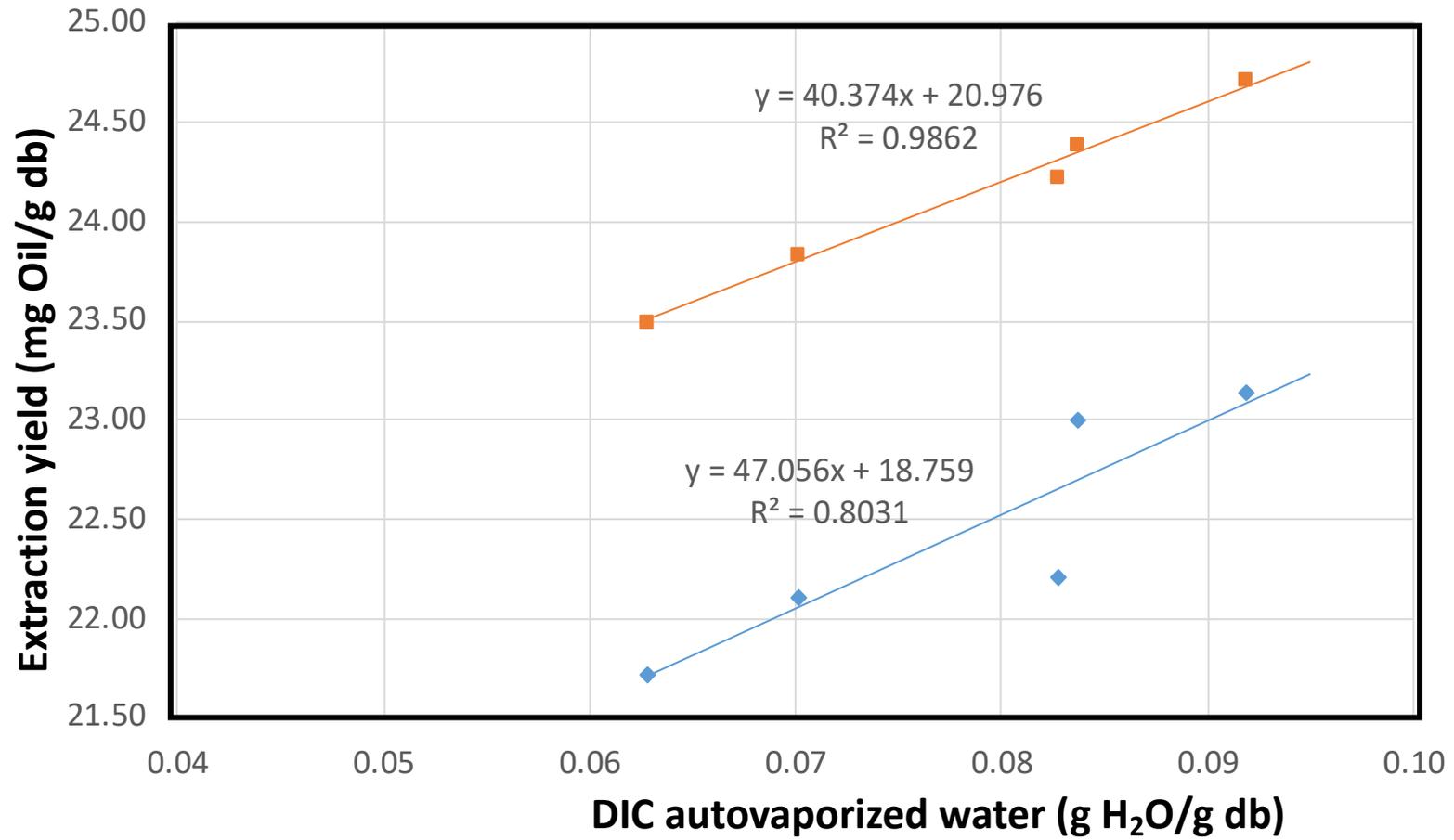


Figure 6: Correlation of the amount of the autovaporized vapor and oil extraction yield

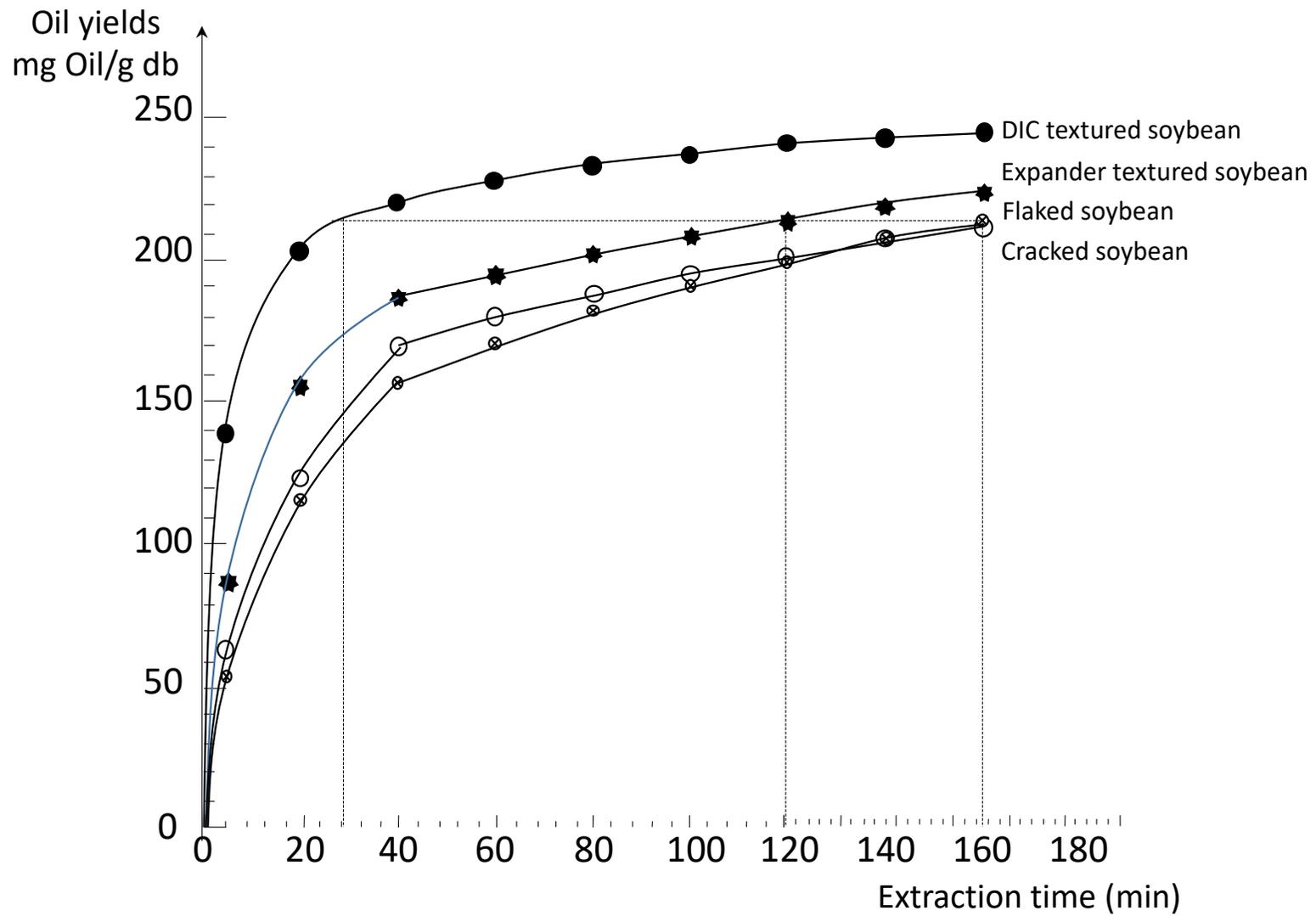


Figure 7. Impact of Instant Controlled Pressure-Drop DIC texturing on oil extraction kinetics of different pretreated soybeans.

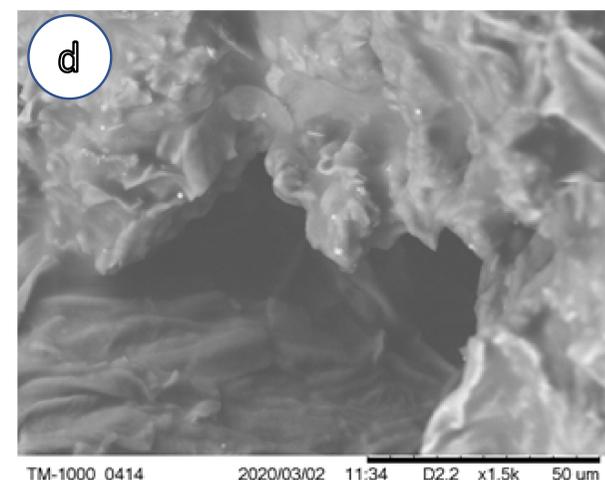
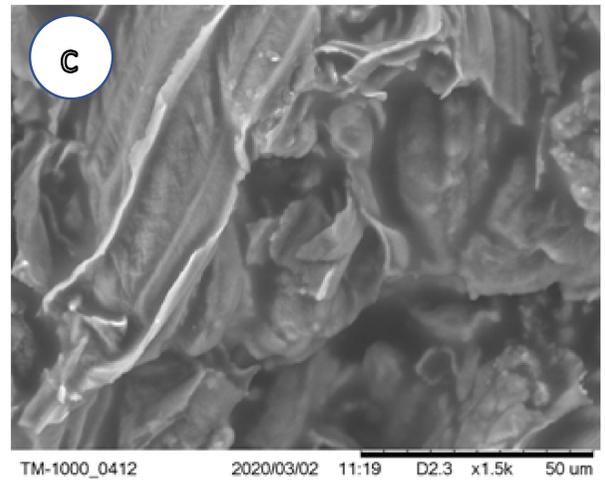
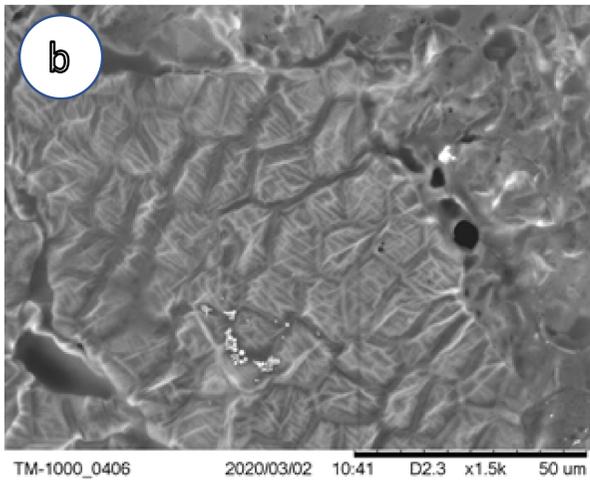
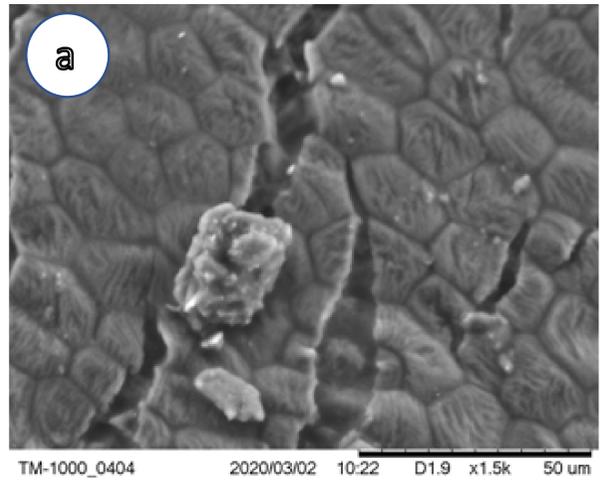


Figure 8. Impact of expander and Instant Controlled Pressure-Drop DIC texturing on structure of Crushed - flaked soybeans by scanning electron microscopic SEM observation: a) non thermal textured seeds; b) Expander treated seeds; c) DIC textured seeds; d) DIC-textured of Expander treated seeds.