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

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## Running Head:

DIC to enhance Solvent Extraction of vegetal oil

## Abstract

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

## Practical impacts

In the sector of the vegetal oil industry, researchers and engineers mostly look for increasing the extraction yields while preserving or improving the sensorial and biochemical quality of the oil. 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  $\Delta T_{pd}$ . **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  $500 \text{ kPa s}^{-1}$ .

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  $D_{\text{eff}}$  ( $\text{m}^2 \text{s}^{-1}$ ) 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  $D_{\text{eff}}$  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  $D_{\text{eff}}$  is estimated from Eq. 4:

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

250 It is noteworthy that only the experimental results for  $t > t_0$  were used in pursuing  $D_{\text{eff}}$ . 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  $\delta Y_s$  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  $\delta Y_s$  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 Agile  
 261 Gas Chromatography 19091S-433 (Kyoto, Japan) well-appointed with a specific column (HP-  
 262 5MS 30m\*350 $\mu$ m\*0.25 $\mu$ 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  $\mu$ l of oil, added 20  $\mu$ l of Tolbutamide Methyl-  
 266 Hydroxylase (TMH) with 1 ml of solution (50/50) of methanol-chloroform. Successively,  
 267 samples of 1  $\mu$ 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  $\mu$ l per sample, and, finally, an Altimma RPC-18 separation  
 273 column of 250x4.6 mmx5  $\mu$ m (Associates Inc.). Acetonitrile/methanol solution of 75:25 was  
 274 injected as the mobile phase at 1 ml/mm 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  $\alpha$ ,  $\beta$ , and  $\gamma$

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  $Y_{\infty}$ , time to reach the same yields as  
299 cracked Cr soybeans do in 160 min, starting accessibility  $\delta Y_s$ , and effective diffusivity  $D_{\text{eff}}$ .

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  $\delta Y_s$ ),  
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  $\delta Y_s$**  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  $\delta Y_s$  (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 ( $\delta$ ,  $\gamma$ , and  $\alpha$ ), show a slightly-significant variation according to the differently DIC treated  
435 soybeans. These modifications inside the values of tocopherol compounds;  $\delta$ ,  $\gamma$ , and  $\alpha$  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  $2085$   $\text{kJ kg}^{-1}$  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  $D_{\text{eff}}$  as well as the starting accessibility  $\delta Y_s$  of N-  
480 hexane solvent extraction of soybean oil in the different studied cases.  $D_{\text{eff}}$  and  $\delta Y_s$  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  $D_{\text{eff}}$  and  $\delta Y_s$  with  $0.065 \cdot 10^{-10}$   
484  $\text{m}^2/\text{s}$  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 ( $\text{m}^2\text{s}^{-1}$ )
DIC:	Instant Controlled Pressure-Drop
DM:	Dynamic Maceration
DoE:	Design of Experiments
$\Delta T_{pd}$ :	Variation of temperature through the pressure drop for Expander or DIC texturing processes
$\delta Y_s$ :	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 $T_t$ ( $\text{J kg}^{-1}$ )
$m_d$ :	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  $\text{H}_2\text{O}$ /kg db)  
 $X_{\text{wb}}$ : Percentage of Moisture content in soybean  
 $Y_{\infty}$ : 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  
 $Y_{\text{oil}}$ : 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

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## 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 %)		
<b>Untreated samples:</b>			0.20962 (100.00%)	0.23019 (100.00%)	0.2352 (100%)
<b>DIC-CPA</b>	0.45	70	0.2300± 0.0020 (109.71±0.95%)	0.2439± 0.0006 (105.95±0.25%)	-
<b>DIC2</b>	0.7	70	0.21597 (103.03%)	0.23352 (101.45%)	-
<b>DIC3</b>	0.45	120	0.24215 (115.52%)	0.24216 (105.20%)	-
<b>DIC5</b>	0.63	105	0.23145 (110.42%)	0.24713 (107.36%)	-
<b>DIC6</b>	0.63	35	0.22590 (107.77%)	0.23539 (102.26%)	-
<b>DIC8</b>	0.27	35	0.22839 (108.95%)	0.23559 (102.35%)	-
<b>DIC9</b>	0.27	105	0.22114 (105.49%)	0.23839 (103.56%)	-
<b>DIC11</b>	0.20	70	0.21719 (103.61%)	0.23505 (102.11%)	-
<b>DIC12</b>	0.45	20	0.22210 (105.95%)	0.23598 (102.52%)	-
<b>Optimal DIC texturing</b>			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  $D_{\text{eff}}$  and starting accessibility  $\delta Y_s$ , while TRY is the “Time needed to get the same reference of Yield from cracked beans”.

Type of pretreatment of soybeans	$D_{\text{eff}}$	$\delta Y_s$	TRY	$R^2$ of CWD model (%)
	Value ( $10^{-12} \text{ m}^2/\text{s}$ )	Value (mg oil/g db)	Value (min)	
<b>Cracked soybeans</b>	3.2 (100%)	124.2 (100%)	160 (100%)	86.57%
<b>Flaked soybeans</b>	1.7 (53%)	148.7 (120%)	160 (100%)	95.55%
<b>Expander textured by Flaked soybeans</b>	2.7 (84%)	166.3 (134%)	120 (75%)	98.64%
<b>DIC textured Flaked soybeans</b>	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.

<b>Fatty acids</b>	<b>Cracked bean oil</b>		<b>Flaked bean oil</b>		<b>Expander-bean oil</b>
<b>Treatments</b>	RM	DIC OPT	RM	DIC OPT	RM
<b>C16:0</b>	10.96±0.61	11.02±0.58	11.01±0.72	10.87±0.48	10.83±0.51
<b>C18:0</b>	4.55±0.02	4.5±0.02	4.51±0.01	4.53±0.02	4.46±0.01
<b>C18:1</b>	24.13±1.31	24.04±1.62	23.81±1.57	24.76±2.01	24.02±1.42
<b>C18:2</b>	52.99±2.55	52.85±3.01	53.11±3.12	52.37±2.88	53.1±3.15
<b>C18:3</b>	6.95±0.04	7.07±0.05	7.12±0.03	7.1±0.05	7.19±0.06
<b>C22:0</b>	0.42±0.01	0.52±0.01	0.44±0.01	0.39±0.01	0.41±0.01
<b>Delta (µg/g oil)</b>	191.39±9.52	177.82±8.02	175.90±10.85	208.95±11.30	162.55±8.95
<b>Gama (µg/g oil)</b>	341.21±10.20	378.08±11.33	327.00±10.01	284.99±09.84	308.78±11.32
<b>Alpha (µg/g oil)</b>	48.38±1.51	46.84±1.32	36.28±2.01	34.58±1.85	40.14±2.10
<b>Total Tocopherols (µg/g oil)</b>	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 <sup>-1</sup> oil)
	$m_o/m_d$	$m_d$	$m_o$	$m_w$	$T_a$	$T_t$	$T_f$	$T_c$	$\Delta T_{pd}$	$Q_h$	$Q_c$	$Q_t$	$m_v$	
			(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

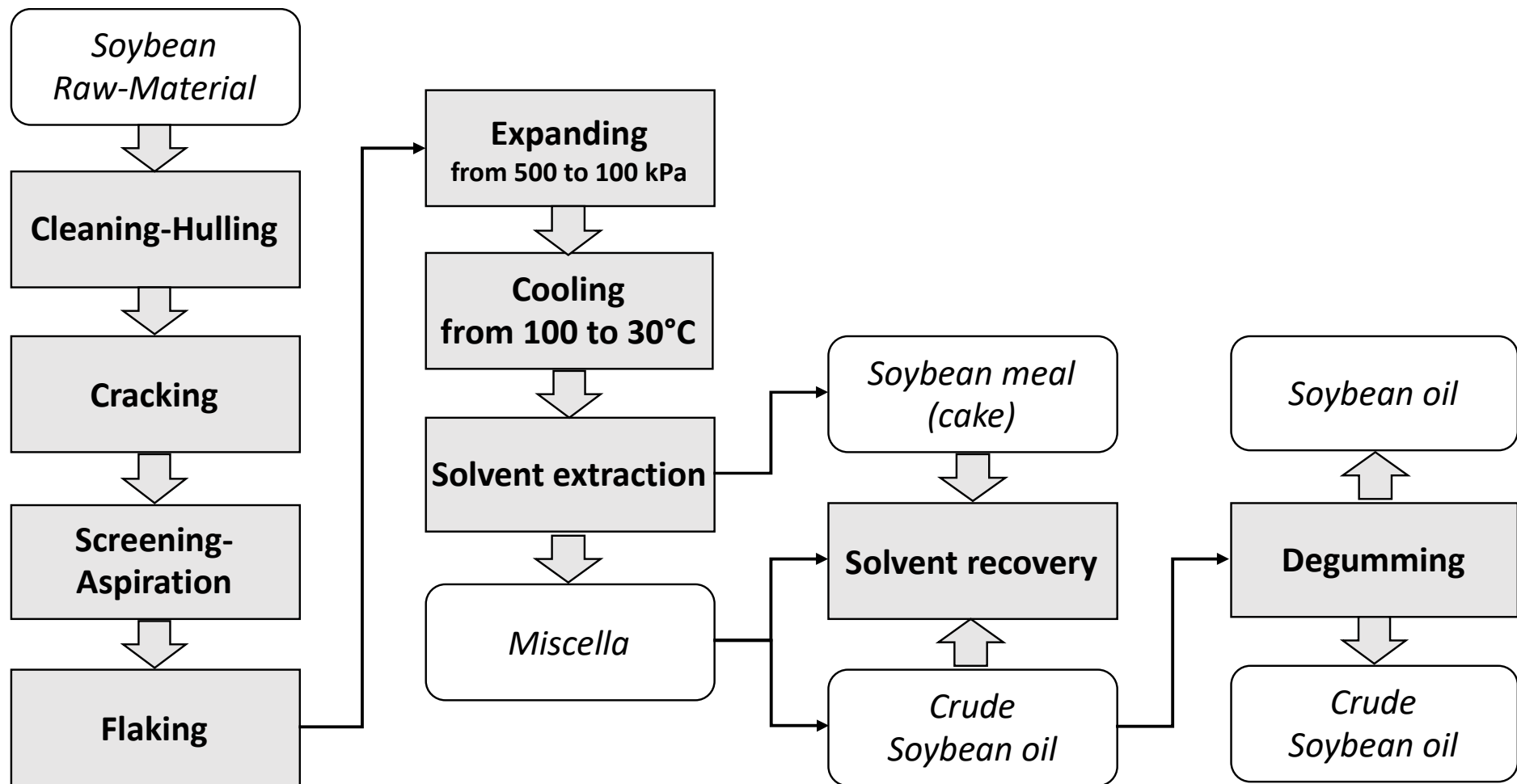


Figure 1. Industrial flow-chart of the solvent extraction of soybean oil

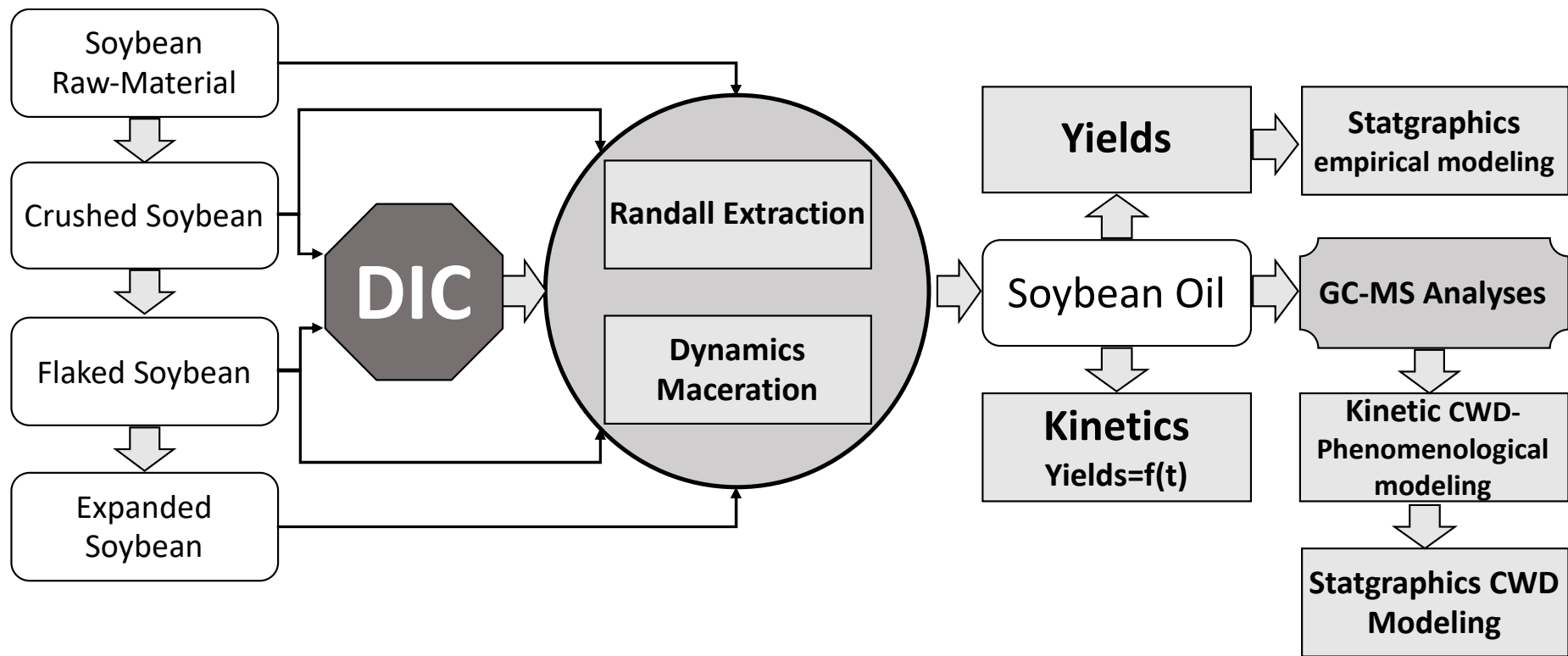


Figure 2. Diagram and experimental protocol of soybean oil extraction inserting a DIC texturing stage

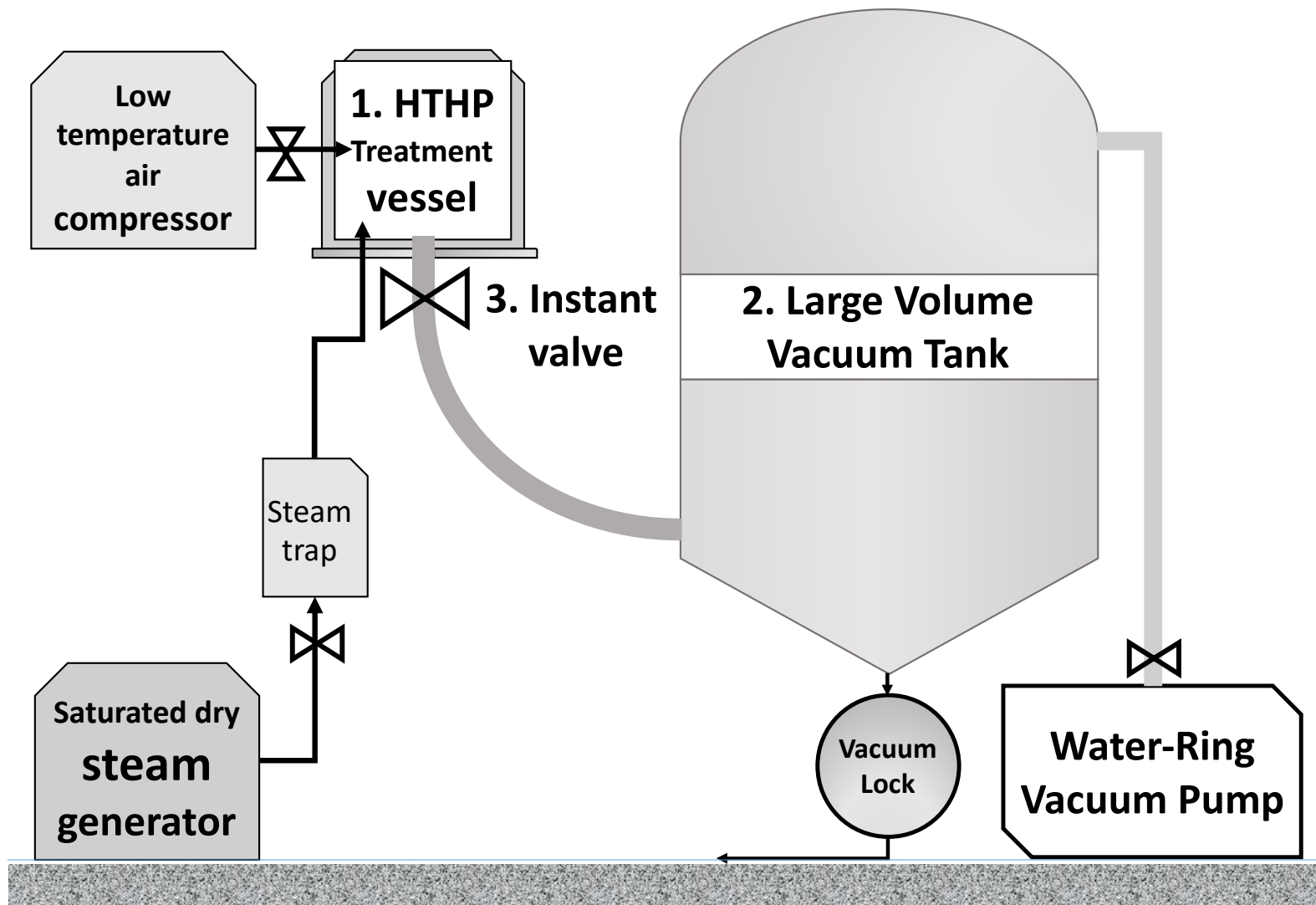


Figure 3. Schematic diagram of DIC laboratory scale unit: 1/ Treatment vessel; 2/ Vacuum tank; 3/ Large section-instant opening valve; 4/ water-ring vacuum pump; 5/ Saturated steam generator with condensed water trap.

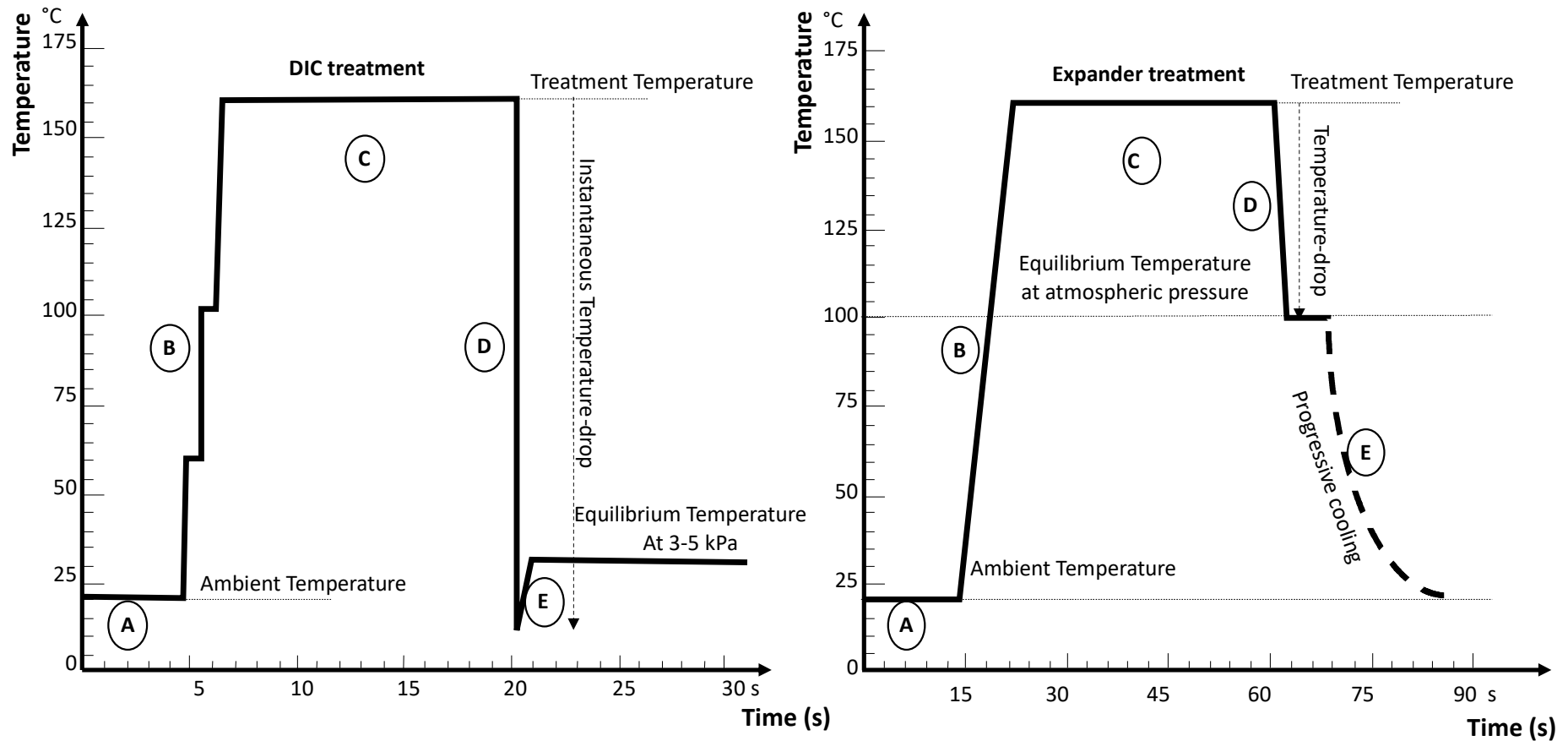


Figure 4. Stages of DIC and expander treatments, respectively: A/ Grains to set in the treatment vessel to immediately connect with the vacuum tank (only for DIC); B/ Saturated dry steam to inject till reaching the desired pressure and temperature treatment levels; C/ High pressure/high temperature to 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).

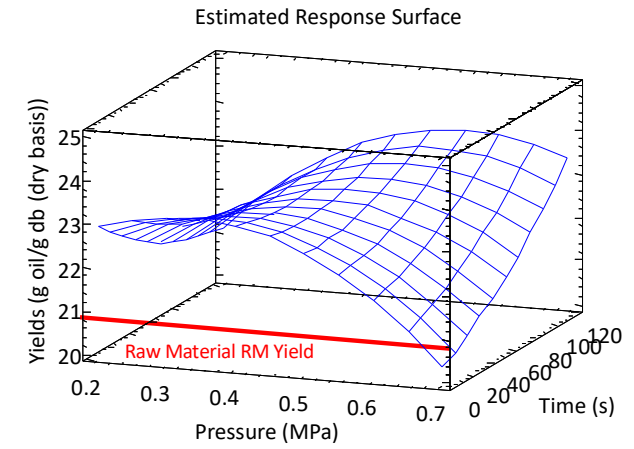
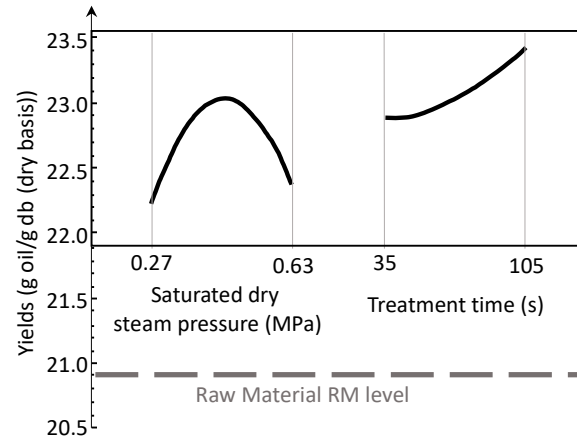
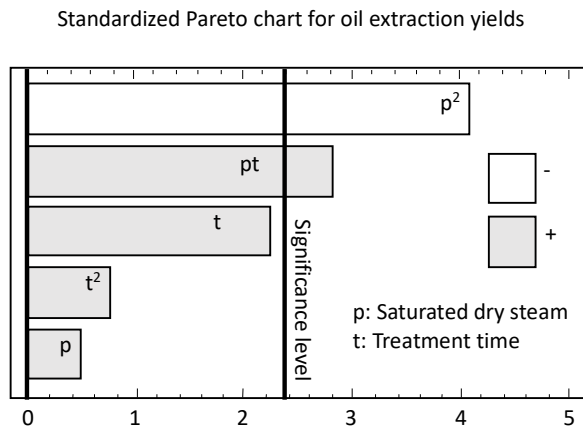


Fig.5-a: Cracked soybean seeds (C)

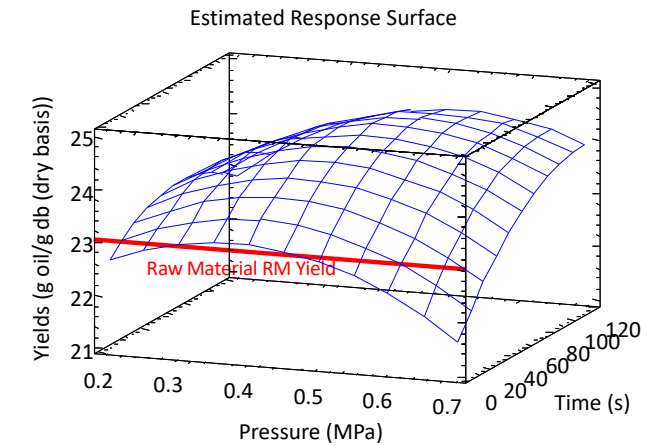
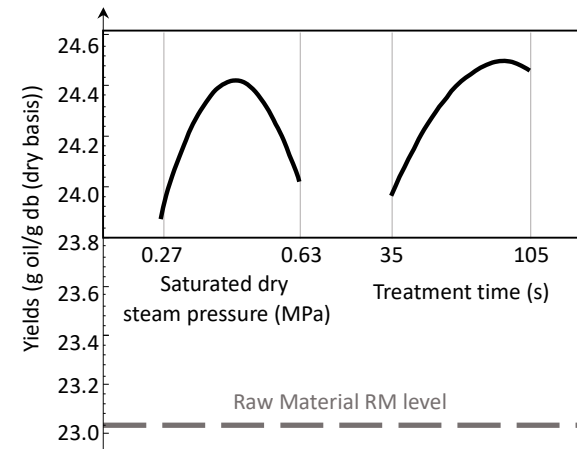
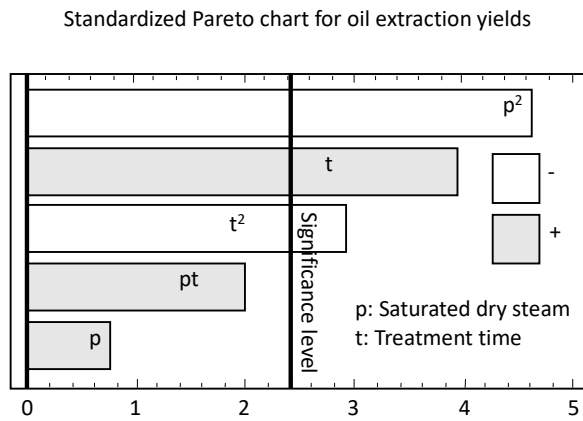


Fig.5-b: Flaked soybean seeds (CF)

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

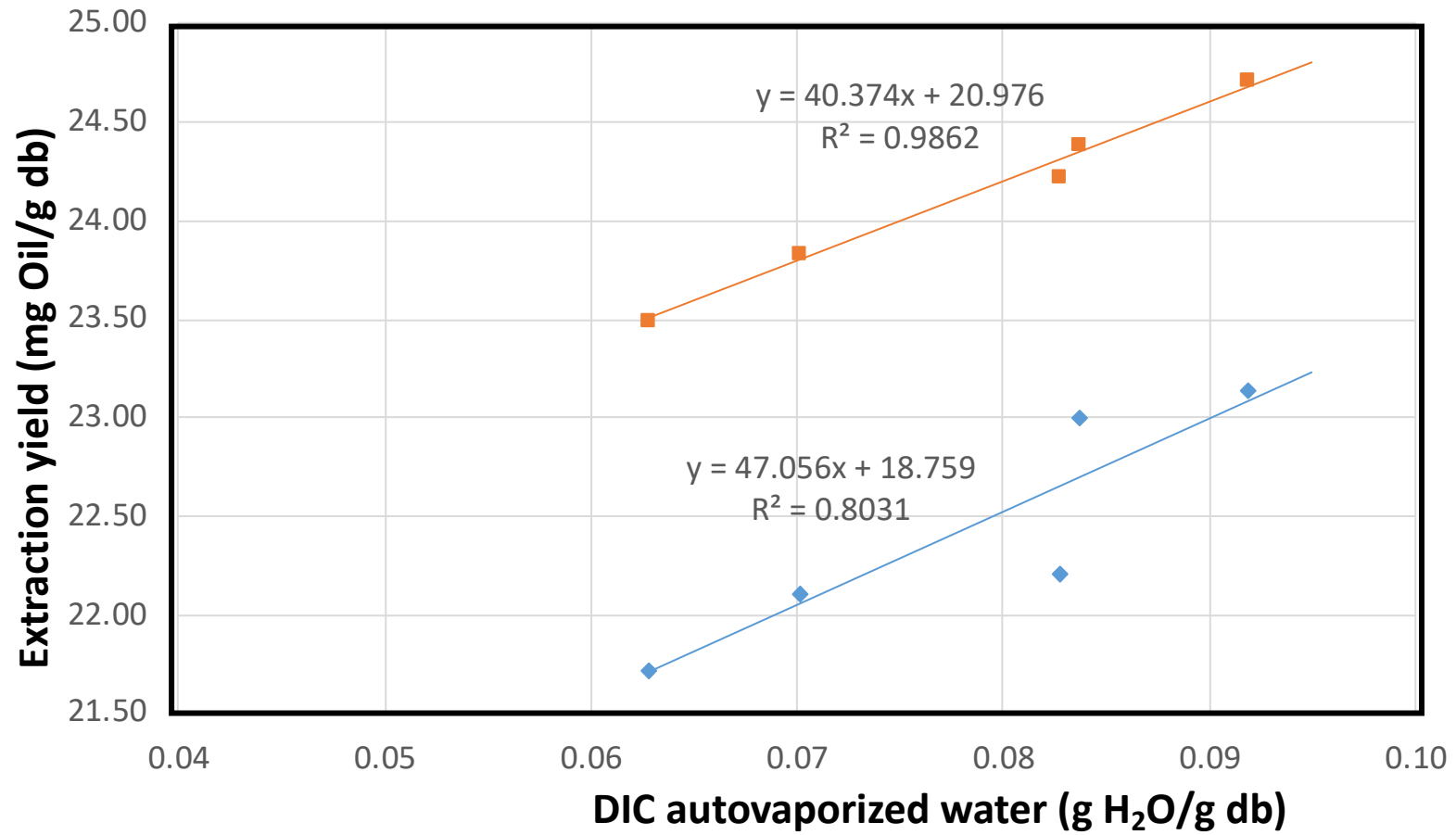


Figure 6: Correlation of the amount of the autovaporized vapor and oil extraction yield

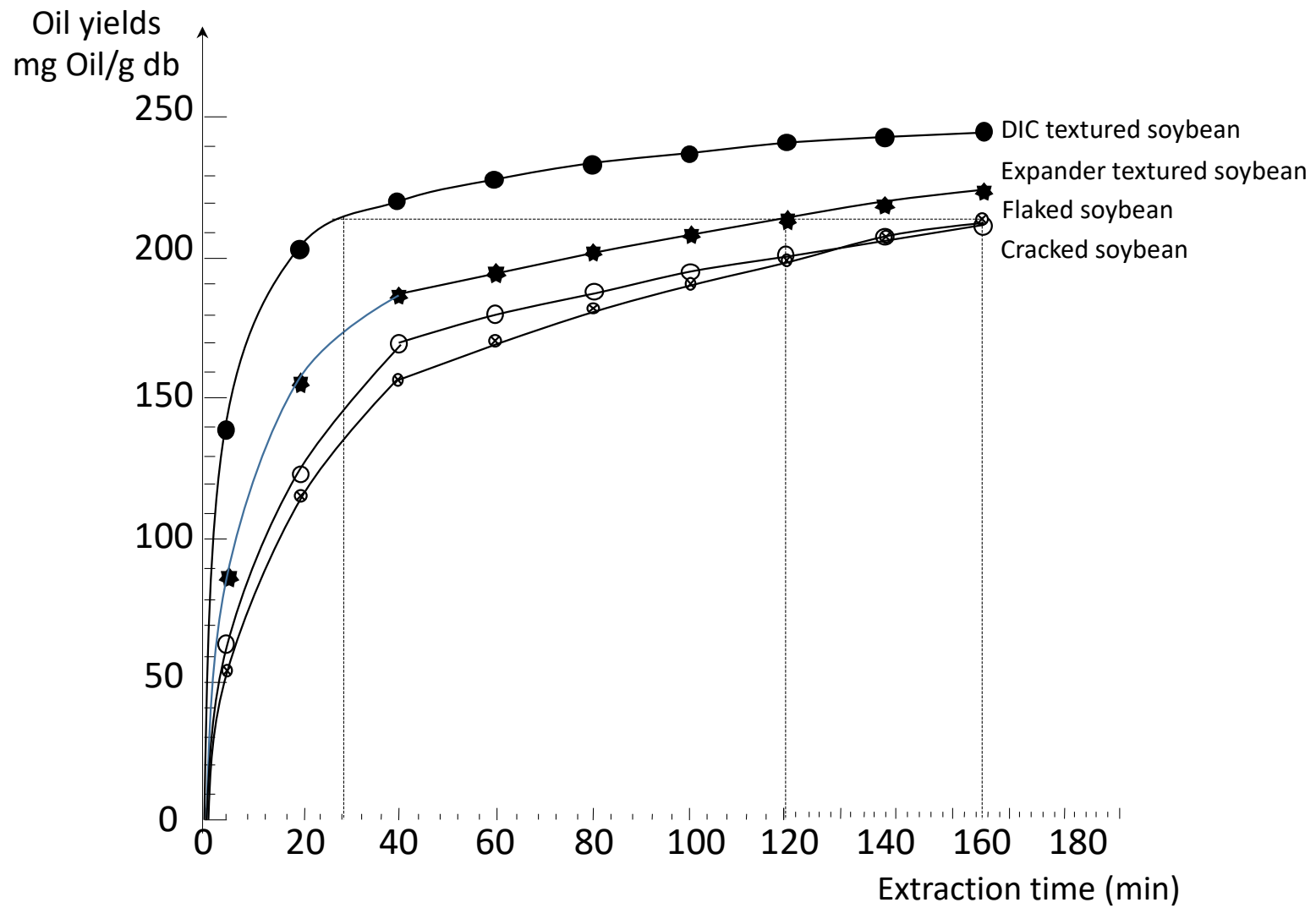


Figure 7. Impact of Instant Controlled Pressure-Drop DIC texturing on oil extraction kinetics of different pretreated soybeans.



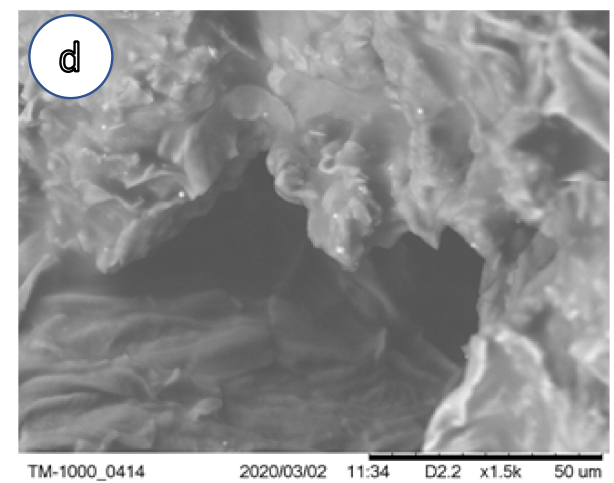
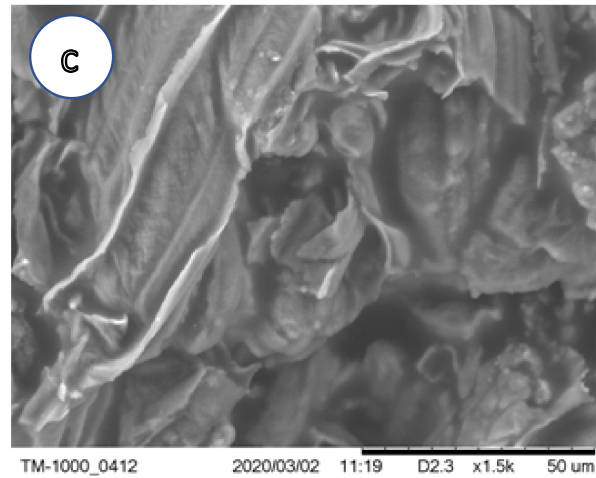
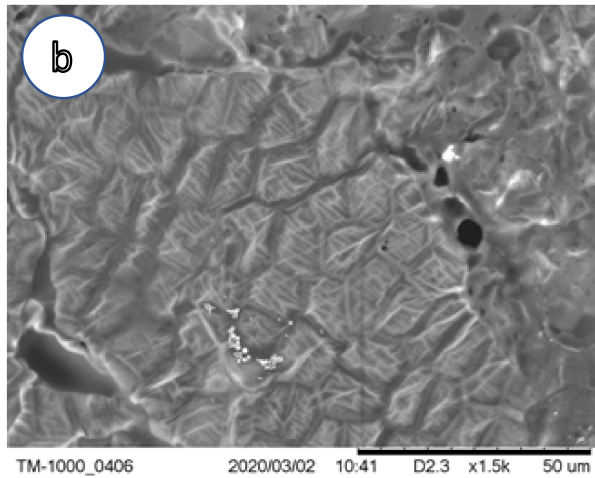
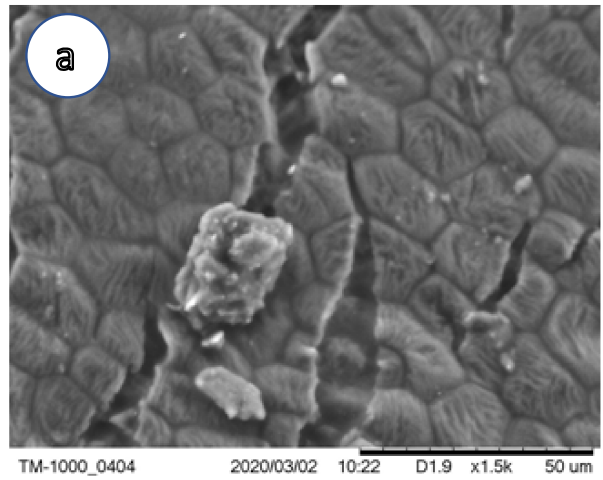


Figure 8. Impact of expander and Instant Controlled Pressure-Drop DIC texturing on structure of Crushed - flaked soybeans by scanning electron microscopic SEM observation: a) non thermal textured seeds; b) Expander treated seeds; c) DIC textured seeds; d) DIC-textured of Expander treated seeds.