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Valérie Guénard-Lampron, Xincheng Liu, Marine Masson, David Blumenthal. Screening of different flours for 3D food printing: Optimization of thermomechanical process of soy and rye flour dough. *Innovative Food Science & Emerging Technologies / Innovative Food Science and Emerging Technologies*, 2023, 87, pp.103394. 10.1016/j.ifset.2023.103394 . hal-04133668

HAL Id: hal-04133668

<https://universite-paris-saclay.hal.science/hal-04133668v1>

Submitted on 20 Jun 2023

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Screening of different flours for 3D food printing: optimization of thermomechanical process of soy and rye flour dough

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Highlights

- Printability of the dough is optimized by water content and duration of the process
- The two parameters must be adjusted simultaneously due to their interactions
- Printing quality and stability of the products mainly depends on the water content
- Similar water content (~ 60%) optimizes printability of soy and rye flour dough
- Longer process time is needed to optimize soy flour dough

Abstract

3D food printing allows the development of personalized food, but it is necessary to diversify the printable recipes to propose a varied food offer. The objective of this project was to explore the printability potential of different flours and to optimize a thermomechanical process (mixing and heating of water and flour) to ensure good print quality. A bibliographic study of 25 flours and an experimental screening of five flours was conducted. Thanks to the previous steps, soy and rye flour were selected because of their interesting nutritional value and their printability potential. An experimental design (10 trials) for each flour showed a significant impact of the process parameters studied (percentage of water content and duration of the thermomechanical treatment) on print quality. The reverse engineering approach demonstrated that the optimal water content is similar for both flour (Soy: 59 %, Rye: 60 %), but the optimal duration of the thermomechanical treatment is quite different (Soy: 27 min, Rye: 22 min).

Industrial relevance text

Interest in 3D food printing has continued to grow in recent years. Products with different flavors, shapes and textures have been proposed in many studies. However, print quality is not always easy to predict and to obtain, especially when faced with nutritional or functional constraints. In our study, we developed a 3-steps approach (two screening steps including bibliographic and experimental method and then one optimization step) to propose new edible ink combining interesting nutritional composition and good print quality. To achieve this goal a design of experiments based on two process parameters (water content and duration of thermomechanical treatment) was conducted and a predictive model was established for soy and rye flours. The reverse-engineering method allowed determining the process parameters to use to ensure good print quality and stability of 3D-printed products. This approach could be applied for the development of new edible ink using other flours or integrating different ingredients.

Keywords : 3D food printing, dough, food processing, desirability function, reverse engineering

36 1. Introduction

37

38 3D printing using layer-by-layer deposition technology also called additive manufacturing are
39 increasingly studied for their potential in terms of food personalization. 3D extrusion printing offers the
40 possibility to design shape, texture, taste, and nutritional needs that meet consumers preferences and
41 the needs of specific populations (e.g., people with dysphagia, athletes, people with allergies or
42 following a diet, etc.). Different types of edible inks have been studied for 3D food printing such as
43 chocolate (Mantihal, Prakash, & Bhandari, 2019), gels system (Chen, Zhang, & Phuhongsung, 2021;
44 Wang, Zhang, Bhandari, & Yang, 2018; Yang, Zhang, Bhandari, & Liu, 2018), mashed potato (Liu,
45 Zhang, Bhandari, & Yang, 2018), ground meat (Dick, Bhandari, Dong, & Prakash, 2020; Dick,
46 Bhandari, & Prakash, 2019). Other studies have focused on cereal-based food like cookie dough
47 (Pulatsu, Su, Lin, & Lin, 2020) or snack (Derossi, Caporizzi, Oral, & Severini, 2020; Derossi,
48 Caporizzi, Paolillo, & Severini, 2020; Uribe-Wandurraga et al., 2020).

49

50 Wheat flour dough shown a strong interest in 3D food printing because of their structuring properties
51 of starch and proteins when using a thermomechanical treatment. A two-step thermomechanical
52 treatment firstly allows the hydration of the constituents of the flour during mixing, and then the
53 gelatinization of the starch as well as the denaturation of the proteins occurs during the mixing step
54 combined with heating (Masbernat et al., 2021). These changes make it possible to increase the viscosity
55 of the dough and thus make it printable. In the field of cereal products, few studies have focused on this
56 type of treatment, but the results of Champenois, Rao, & Walker (1998) and Masbernat et al. (2021)
57 demonstrated that hydrothermal transformations of starch and gluten proteins in hydrated doughs made
58 from wheat flour are impacted by water/flour ratio and process parameters (duration, intensity and
59 temperature). For example, if the amount of water available for starch gelatinization is not sufficient,
60 the dough obtained after the treatment will contain non-gelatinized or partially gelatinized starch
61 granules that are more rigid (higher G') than gelatinized starch and could lead to less dough sticky
62 particles. These properties of rigidity and stickiness could thus affect the printing quality of the doughs
63 (Masbernat, 2021).

64

65 More complex recipes with the addition of sugar and oil, dairy ingredients, fruits or vegetables puree to
66 the wheat flour dough have also been printed and validated (Guénard-Lampron, Masson, Leichtnam, &
67 Blumenthal, 2021; Masbernat, 2021). However, to diversify the tastes and textures and improve the
68 nutritional intake of these printable products made from flour, it is essential to explore matrices based
69 on other cereals, legumes, or nuts flours for example. Recent publications report on new food inks
70 integrating different flours with interesting nutritional properties (e.g., gluten-free, richer in fiber or
71 protein). Gluten-free snack bite (lupine or chickpea flour) (Agarwal et al., 2022), high fiber cookie (oat,
72 rye, rice, and carob flour) (Pavičić, Grgić, Ivanov, Novotni, & Herceg, 2021), protein and dietary fiber-
73 rich snack (wholegrain rye flour) (Lille, Kortekangas, Heiniö, & Sozer, 2020), gluten-free snack

74 (wholegrain buckwheat, proso millet, white corn, sweet potato or flax seed flour) (Radoš et al., 2022)
75 are some examples. However, the integration of these flours in food inks can affect printing quality and
76 stability of the printed product. For example, Agarwal et al. (2022) and Radoš et al. (2022) observed
77 that the print quality of the gluten-free snacks was strongly affected by the particle size of flour (e.g.
78 coarse filaments with lupine flour compared to smoother filaments with chickpea flour). It therefore
79 seems essential to study a formulation and process strategy that considers the printability of edible ink
80 prior to the development of these new 3D printed foods.

81

82 Godoi, Prakash, & Bhandari (2016) define the printability of a food material by its ability to maintain
83 its dimensional stability and support its own height. Nijdam, LeCorre-Bordes, Delvart, & Schon (2021)
84 summarized the quality of a print according to three main factors: printer capability (ex.: force required
85 to extrude food ink and accuracy of the displacement), filament quality (e.g.: rheological and
86 microstructural properties of the food ink) and dimensional stability (during and after printing). In
87 addition, several printing (e.g. printing speed, nozzle diameter, layer height, filling rate, fill pattern) and
88 post-processing parameters (e.g. cooking methods, temperature, and time) affect the quality and
89 stability of the 3D-printed products (Guénard-Lampron et al., 2021; Severini, Azzollini, Albenzio, &
90 Derossi, 2018; Severini, Derossi, & Azzollini, 2016).

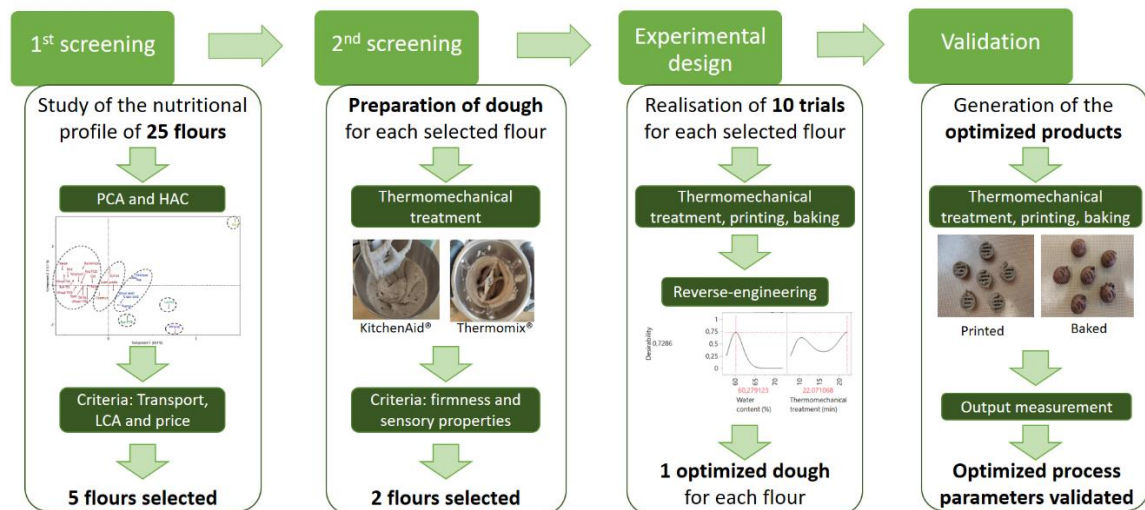
91

92 In this study, we developed a 3-step approach to answer our main objective, which was to know how to
93 ensure good printing quality of dough made from different flours with interesting nutritional qualities
94 by using a thermomechanical treatment similar to that developed by Masbernat et al., (2021) for wheat
95 flour dough. The first step of this study was to conduct a bibliographic screening of flours and to select
96 five flours according to three criteria (nutritional value, distance to major product region and price).
97 The second step was to select two flours from the previous selection according to their printability
98 potential by using an experimental screening. The last step was to optimize and validate the print quality
99 of the two flours that have demonstrated good printability potential during the previous step. The final
100 selection was limited to two flours due to the time required to conduct the tests and to validate the
101 potential for optimization by the process before applying this method to several flours. Finally, the aim
102 of our study was to propose a predictive model for each flour to obtain the specific thermomechanical
103 parameters to use for a good printing quality. The next sections are organized accordingly to our 3-step
104 approach.

105

106 2. Material and Methods

107 Figure 1 presents the general organization of our study from the bibliographic research on 25 flours
108 to the validation of the optimized process parameters for 2 selected flours.



109

110 **Fig.1.** Experimental scheme of the two screening steps followed by the experimental design and the
 111 final validation of the process parameters studied.

112 2.1. First screening of different flours : Bibliographic research

113 To select the appropriate flours to be evaluated, three criteria were evaluated for 25 flours available in
 114 the market, including 13 cereal flours, 6 legume flours, 3 nut flours, 2 pseudo cereal flours and 1 root
 115 flour. The objective of this screening was to select 5 flours nutritionally better than wheat flour which
 116 was used in the reference recipe developed by [Masbernat, \(2021\)](#) and with various nutritious profile.
 117 To compare the potential of each flour, we defined three criteria: nutritional value, distance to major
 118 production regions and price, which helped to evaluate the flours on nutritional, ecological and
 119 economical aspects. Regarding the objective of this study the main criteria used is the nutritional aspect.
 120 For equal nutritional qualities, the economic and ecological aspects will be considered.

121 - Definition of the nutritional criteria

122 Nutritional value information was obtained in Ciqual (ANSES) and FoodData central (USDA) database.
 123 A total of 24 criteria was obtain on protein, starch, fibers, ash, mineral and vitamin content for each
 124 flour. To find the most relevant criteria to select good candidates, we look at the variation between
 125 flours for each nutritional aspect especially for minerals and vitamins, which were represented by many
 126 micronutrients. The coefficient of variation of each micronutrient was calculated by dividing the
 127 standard deviation by the average of the value of all the 25 flours. For example, the average calcium
 128 content for all the flours studied was 57.38 mg, the standard deviation was 62.41 and we obtained a
 129 coefficient of variation ($62.41/57.38 \times 100$) of 108.77 %. The coefficient of variation was much lower
 130 for other minerals (from 40 to 76 %). We decided to select micronutrients with a coefficient of variation
 131 superior of 100%. Calcium, vitamin E, vitamin K1, vitamin B2 and vitamin B9 were thus used in
 132 statistical analysis. From the criteria selected, Hierarchical Agglomerative Clustering (HAC) was used

133 to define clusters. Then, to diversify the nutritious profile of the 5 flours to be selected, only one flour
134 was chosen in each group of HAC.

135 - Definition of the ecological and economic criteria

136 The flours in the same group of HAC were compared according to ecological and economic criteria.
137 Firstly, the nearest production area to France was collected from FAOSTAT database for each flour.
138 This information allowed us to calculate the transport distance to France, which gives an indicator of
139 the impact of carbon footprint of the transport. The nearest production area was defined as the country
140 or region closest to France identified by the orange or darker color code in the international crop
141 production map of the FAOSTAT database, which means that has a medium to high production of the
142 corresponding crop. The transport distance was then calculated by measuring the distance from the
143 selected region to France. Secondly, according to the main environmental problems of agriculture
144 mentioned in FAO perspectives (FAO, 2003), 4 indicators of LCA (Life Cycle Assessment) were
145 collected from Agribalyse® database for flours for which information was available to evaluate the
146 environmental impact of the production of plants. The 4 indicators were: climate change (kg of CO₂
147 equivalent released/kg of product), eutrophication fresh water (kg of phosphorus equivalent released/kg
148 of product), land use (Pt/kg of product) and water scarcity (m³ water deprived/kg of product). The PEF
149 (Product Environmental Footprint) score, which gives an overall view of the impact of products on the
150 environment based on 16 environmental criteria, was also collected from Agribalyse® database. Then,
151 for an economical point of view, the price of each flour was compared (price/kg).

152 2.2. Second screening from an experimental approach with printing product

153 As the final objective is to optimize printing quality, we needed a second screening to determine the
154 two flours having the highest printability potential. For the flours selected by the bibliographic
155 approach, we compared the candidates after the preparation of the dough and their printing.

156 2.2.1. Preparation of the printable dough

157 The five flours used in this study were: rye flour (Type 170, Moulins de Versailles, France), chestnut
158 flour (Mon fournil, France), soy flour (Alnatura, Germany), lupine flour (Moulins de Versailles, France)
159 and chickpea flour (Mon fournil, France). The printing dough were prepared with one of these flours
160 and water. The water content of each flour (Table 1) was determined by weighing the dried matter after
161 90 min at 130 °C (Etuve EM10, Chopin Technologies, Villeneuve-la-Garenne, France) by a reference
162 method for cereals-based products (NF V03-707).

163 **Table 1.**

164 Water content measured for each flour.

Flours	Rye	Chestnut	Soy	Lupine	Chickpea
Water content (%)	11.2 ± 0.2	6.7 ± 0.3	7.4 ± 0.1	9.3 ± 0.1	9.5 ± 0.1

165 To compare the five flours, each of them was prepared using exactly the same formulation with 65 %
166 (w/w) total water content and the same thermomechanical treatment as described by [Guénard-Lampron,](#)
167 [Masson, Leichtnam, & Blumenthal \(2021\)](#). This thermomechanical process was developed by
168 [Masbernat et al.\(2021\)](#) to structure wheat dough by the combination of mixing and heating which led
169 to the formation of swollen gelatinized starch granules in a denatured proteins network. In our study, to
170 perform the thermomechanical treatment we used a planetary mixer with a leaf device (KSS45 EOB
171 CLASSIC, KitchenAid, USA) for the mechanical treatment (120 rpm, 12 min) and then a multifunction
172 robot (Vorwerk, Thermomix TM6, FR) for the thermomechanical treatment (85 °C, 100 rpm, 15 min).

173 2.2.2. 3D-Printing

174 The 3D food printer prototype built by Dagoma (Roubaix, France) and used by [Guénard-Lampron et](#)
175 [al. \(2021\)](#) was also used in our study. The dough (20 ± 1 °C) poached in the syringe of the prototype
176 was printed on silicone baking mat. Printing parameters were determined by preliminary tests and fixed
177 for all printing of this study. We used a nozzle diameter of 3.4 mm, a filling rate of 55 % and a printing
178 speed of $10 \text{ mm}\cdot\text{s}^{-1}$ to print six cylindrical model (diameter: 3 cm, height: 1.5 cm) for each dough.

179

180 2.2.3. Selection criteria

181 The five doughs made from each of the selected flour were compared accordingly to new criteria:
182 firmness and sensory aspect (visual and textural properties) of the dough. The nutritional composition
183 of the flours was again considered to decide between flours with a similar printability potential.

184

185 - Firmness of the dough

186 A texture analysis of the produced dough was performed as described by [Masbernat \(2021\)](#). The mean
187 force (N), which is an indicator of printability potential, was measured between 10 and 20 s of the back
188 extrusion method (piston probe with annulus gap of 1.5 mm, crosshead speed of 1mm/s, 30 mm of
189 depth) using a texture analyzer (TaHD, Stable Micro Systems, Surrey, UK). Triplicate were realized
190 for each dough of the second screening.

191 - Sensory aspect of the dough

192 Two experts in our laboratory evaluated visual and textural properties during consumption of the dough.
193 These aspects were important to assess consumer appreciation of the selected products during a future
194 project but also to ensure a good extrusion and printing of the dough avoiding very foamy or grainy
195 textures for example.

196 2.3. Final experimental design for the two selected flours with printed and baked products

197 Preliminary tests were conducted to determine the process parameters that can optimize printability of
198 the two selected flours and to define minimum and maximum limits of these parameters. The water
199 content was choose because ([Masbernat et al., 2021](#)) had already demonstrated that the water/flour ratio

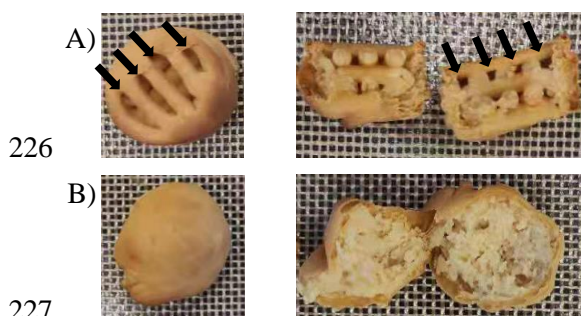
200 (w/w) had a significant impact on the printing quality of dough made from wheat flour. For the
201 thermomechanical treatment, we had the choice to study the temperature, the duration, or the speed.
202 Duration of the treatment was chosen because preliminary test demonstrated an impact of this parameter
203 on the firmness of the dough after the treatment. Temperature and speed of thermomechanical treatment
204 would also be interesting to study in a future project, but it is more complex to control these exact
205 parameters in our multifunction robot (Vorwerk, Thermomix TM6, FR) during the process. The
206 minimum and maximum limits of the parameters under study have been determined to obtain an
207 extrudable dough (not too firm) and which can be maintained during the stacking of the layers. Six
208 products were printed and baked directly after the printing for each trial of the experimental design. The
209 baking parameters were determined by preliminary tests and fixed at 185 ± 5 °C during 15 min.

210 2.3.1. Analyzes of 3D-printed product

211 Seven dependent variables (*Y*) were analyzed. One variable on the firmness of the dough (as described
212 in section 2.2.3), four variables on the 3D-printed product before baking (height, diameter, number of
213 unaligned layers and number of cuts visible in the printed layers) and two on the 3D-printed product
214 after baking (number of spacing kept on and inside the product due to the filling rate of 55%). Triplicate
215 were realized for the mean force measurement and the other variables were measured on the 6 printed
216 products for each trial.

217 For the evaluation of the qualitative characteristics of the 3D-printed products, a ScanCube ® and a
218 Canon EOS 750D ® camera were used to standardize the pictures. Pictures were taken from different
219 angles to get an overview of the product. Two qualitative characteristics of the visual aspect before
220 baking were evaluated from the pictures obtained: the number of unaligned layers (stacking of the
221 printed layers) and the number of cuts visible in the printed layers (discontinuous printing). After
222 baking, new pictures were taken and the number of spacing kept after baking on and inside the product
223 were evaluated (Figure 2). The method consists of counting the spacing clearly shown on the surface of
224 the product baked and on at least one of the two sides of the product cut.

225



228 **Fig. 2.** Photos of baked products made from soy flour dough showing A) a product with all spaces
229 retained (TMT of 25 min, water content of 60 %) and B) a product with no space left (TMT of 15 min,
230 water content of 70 %).

231 2.4. Statistical analysis

232 The experimental design and statistical analysis were performed using JMP software (version 16.0, SAS
233 Institute Inc., Cary, SC, USA). The significant effects ($P \leq 0.05$) of the independent variables on each
234 of the response variable were evaluated by a multiple regression analysis and were ranked according to
235 their LogWorth ($-\log_{10}(p \text{ value})$).

236 3. Results and discussion

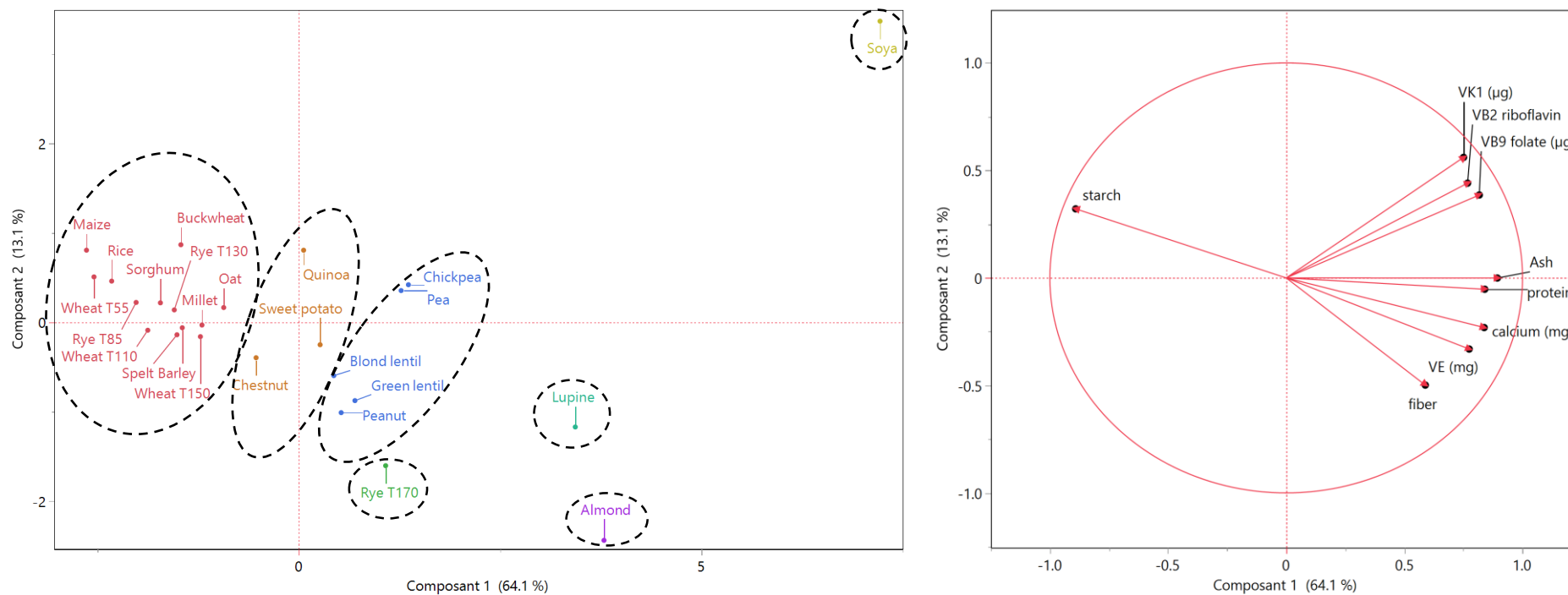
237
238 3.1. Selection of 5 flours from the first screening

239 The objective of this screening was to select 5 flours nutritionally better than wheat flour which was
240 used in the reference recipe developed by Masbernat, (2021). The results of the PCA (Figure 3) shows
241 7 groups of flours classified by the HAC analysis according to the distance between them, which means,
242 the similarity of their nutritional composition. Components 1 and 2 of the PCA explain 77.2 % of the
243 total variance. Most of the variance was explained by component 1 (64.1 %) and was related to all
244 nutritional variables studied. Fiber and vitamins also contributed to the variance explained by
245 component 2 (13.1 %). Flours on the left of the plane are high in starch, while those on the right are
246 lower in starch and high in ash, protein, and fiber, especially those on the bottom right, such as flours
247 of almond, lupine and rye T170. For vitamins and minerals, flours relatively rich in vitamins K1, B2
248 and B9 are distributed in the upper right part (typically soy flour), and flours containing more calcium
249 and vitamin E than the others are drawn to the right and down.

250 The first group on the left of the plane including maize, rice, sorghum, oat, rye T85, rye T130, spelt,
251 barley, millet, buckwheat was eliminated because wheat T55, T110 and T150 were in this same group
252 which meant that the nutritional composition of these flours and that of wheat flours was similar based
253 on our criteria. Regarding the group containing chestnut, quinoa and sweet potato flours, the selection
254 was based on the transport distance because the price was similar for these three flours and the LCA
255 data was only available for the chestnut flour (Table 2). The regions producing quinoa (North America,
256 Peru) and sweet potato (Spain, Italy) are not in France, while chestnuts are relatively abundant in France
257 (8860 tons produced in 2020) compared to the other two crops (FAO, 2022; Ruiz et al., 2014). More
258 transportation would be necessary to supply these flours (quinoa: 6000 km and sweet potato: 1000 km),
259 so they have been eliminated (Table 2). For the next group including chickpea, pea, peanut, blond and
260 green lentil, the peanut was eliminated due to its high transport distance (2000 km). The chickpea flour
261 was selected for its lower cost compared to other flours in this group and was also approved by its
262 relatively low PEF score (0.15) among the 25 flours, even if the data of other flours in the same group
263 wasn't available. Finally, the almond flour was rejected due to its high price and the lupine, rye T170
264 and soy flour were selected mainly due to their nutritional composition. Regarding the environmental
265 criteria, rye flour T170 obtains a relatively low PEF score (0.13) and LCA criteria among all the flours
266 studied, while soy flour was still selected despite its higher impact on the environment. Environmental

267 criteria were not available for lupine flour. Considering all these criteria, soya, lupine, chickpea,
268 chestnut and rye T170 were chosen mainly because of their appreciated nutritional profile and the
269 absence of transport or price issues. This selection contained 3 legume flours (soya, lupine and
270 chickpea), 1 cereal flour (rye) and 1 nut flour (chestnut), which also allowed a diversification of the
271 type of the plants chosen.

272



273

274 **Fig. 3.** Principal component analysis (PCA map of the flours and correlation circle of the nutritional components) of 25 flours. The dotted circles represent
275 the 7 groups of flours obtained by a HAC analysis based on their similarities in terms of nutritional composition.

276

277 **Table 2**

278 Comparison of ecological and economic criteria of 25 flours available in the market in France

Flour	Transport distance to France (km)	Price (€/kg)	PEF (mPt/kg of product)	Climate change (kg CO ₂ eq/kg of product)	Eutrophication freshwater (kg P eq/kg of product)	Land use (Pt/kg of product)	Water scarcity (m ³ depriv./kg of product)
Almond	0	28.55	NA	NA	NA	NA	NA
Barley	0	2.2	0.17	0.9	0.26	122.12	1.55
Blond lentil	0	10.5	NA	NA	NA	NA	NA
Buckwheat	0	3.93	0.16	0.88	0.38	149.3	2.46
Chestnut	0	13.86	0.58	2.29	0.61	8.48	29.61
Chickpea	1000	4.99	0.15	0.8	0.46	198.09	1.25
Green lentil	0	8	NA	NA	NA	NA	NA
Lupine	0	5.22	NA	NA	NA	NA	NA
Maize	0	2.14	0.17	0.59	0.36	149.1	5.81
Millet	0	5.88	0.18	1.04	0.28	124.63	1.61
Oat	0	4.2	NA	NA	NA	NA	NA
Pea	0	6.8	NA	NA	NA	NA	NA
Peanut	2000	10	NA	NA	NA	NA	NA
Quinoa	6000	9.975	NA	NA	NA	NA	NA
Rice	0	4.5	0.37	2.42	0.43	117.23	16.46
Rye T130	0	3.42	0.13	0.8	0.26	53.27	0.46
Rye T170	0	3.35	0.13	0.8	0.26	53.27	0.46
Rye T85	0	2.99	0.13	0.8	0.26	53.27	0.46
Sorghum	0	4.08	NA	NA	NA	NA	NA
Soya	0	5.6	0.25	4.47	0.56	216.68	0.47
Spelt	500	2.88	0.16	0.88	0.38	149.3	2.46
Sweet potato	1000	12	NA	NA	NA	NA	NA
Wheat T110	0	1.08	0.09	0.51	0.15	80.99	0.3
Wheat T150	0	1.83	0.09	0.51	0.15	80.99	0.3
Wheat T55	0	0.99	0.09	0.51	0.15	80.99	0.3

279 NA: PEF score and LCA data not available for almond, blond lentil, green lentil, lupine, oat, pea, peanut, quinoa, sorghum, and sweet potato flours.











280 3.2. Selection of 2 flours from the second screening

281 The objective of this second screening was to select only two flours from the previous selection to
282 implement a print quality optimization strategy. [Table 3](#) shows the three main nutritional criteria
283 considered for this second screening, the visual aspect after each treatment, the mean force, and the
284 main negative sensory aspect of doughs after thermomechanical treatment.

285 As [Guénard-Lampron et al. \(2021\)](#) demonstrated with 3D-printed food product based on wheat flour
286 that maximum force between 23 to 37 N allowed a good printing quality, it is clear based on the mean
287 force of each dough that lupine (18.1 N) and rye (13.8 N) flour dough had a better printability potential
288 then chestnut (3.5 N), soy (5.2 N) and chickpea (1.6 N). However, the lupine flour dough showed the
289 most important negative sensory aspect with a very pronounced bitterness (higher than for chickpea and
290 chestnut) and a very unpleasant sandy texture in the mouth. This texture could be explain by the
291 presence of bigger particle size in lupine flour which impact negatively printing quality ([Agarwal et al.,](#)
292 [2022](#)) and sensory properties of food ([Villarino, Jayasena, Coorey, Chakrabarti-Bell, & Johnson, 2015](#)).
293 These reasons as well as the fact that rye flour have the highest fiber content (23.8 %) explains why we
294 decided to keep rye flour and to eliminate lupine flour. The chickpea flour seemed to have good
295 potential in terms of physico-chemical and sensory properties as demonstrated by [Miñarro, Albanell,](#)
296 [Aguilar, Guamis, & Capellas \(2012\)](#) during the preparation of proofed batter to obtain gluten-free bread.
297 Nevertheless, in our study the chickpea flour dough was immediately rejected due to its bitterness and
298 its very low firmness and foamy texture that are not compatible with a precise and smooth printing. The
299 important foaming capacity and stability of the chickpea flour has already been demonstrated by [Yadav,](#)
300 [Yadav, & Dhull \(2012\)](#) and could be due to its high protein content. The firmness of the chickpea flour
301 dough could be increased by combining chickpea flour with another protein source such as a pea protein
302 isolate and the foamy texture could be decreased by avoiding mechanical processing as proposed by
303 [Agarwal et al. \(2022\)](#). Finally, we had to choose between chestnut and soy flour. We selected the soy
304 flour because its dough had a slightly higher firmness than that made from chestnut, did not present a
305 negative sensory aspect, and had a higher protein content (35.8 % comparatively to 5.7 % for chestnut
306 flour). The low firmness of the chestnut may be surprising since this flour has a high starch content
307 (46.9 %) but this is still lower than that for wheat flour T55 (69.3 %). It would be interesting to learn
308 more about the behavior of the chestnut flour in a future 3D printing project since this flour demonstrates
309 strong potential as novel ingredient in cereal products ([Allouch, Sfayhi, Doggui, & Debbabi, 2022;](#)
310 [Dall'Asta et al., 2013](#)).

311

312 **Table 3**
 313 Comparison of the 5 flours selected for the second screening after mechanical and thermomechanical treatments.

	Rye (T170)	Chestnut	Soy	Lupine	Chickpea
Protein (%)	15.9	5.7	35.8	36.2	22.4
Starch (%)	18.7	46.9	5.4	2.6	25.3
Fiber (%)	23.8	12.6	10	18.9	10.8
Mechanical treatment					
Thermo-mechanical treatment					
Mean force (N)	13.8 ± 0.7	3.5 ± 0.3	5.2 ± 0.2	18.1 ± 0.2	1.6 ± 0.1
Negative sensory aspect		Bitter (+)		Bitter (+++) Sandy	Bitter (++) Foamy

314 *The intensity of the negative sensory aspect perceived is represented by the symbol “+”.

315

316

317 3.3. Generation of different doughs from the 2 selected flours

318 The objective of this last step was to optimize the print quality of the soy and rye flours that have
 319 demonstrated good printability potential during the previous step. Table 4 shows the two Central
 320 composite designs of 10 trials determined for each flour. The water content varied from 58 to 72 % for
 321 soy and rye flour and the duration of the thermomechanical treatment was studied from 13 to 27 min
 322 for soy flour and from 8 to 22 min for rye flour. These experimental designs were realized to propose a
 323 predictive model for each flour and to obtain the specific thermomechanical parameters to use for a
 324 good printing quality.

325 **Table 4**

326 Experimental design of 10 trials for A) soy and B) rye flour

A)			B)		
Trial	Water content (%)	Thermomechanical treatment (min)	Trial	Water content (%)	Thermomechanical treatment (min)
1	70	15	1	60	10
2	72	20	2	72	15
3	65	20	3	65	22
4	65	13	4	58	13
5	70	25	5	65	15
6	65	27	6	70	20
7	60	15	7	65	15
8	58	20	8	65	8
9	65	20	9	60	20
10	60	25	10	70	10

327
 328 Table 5 and 6 shows respectively the evolution of rye and soy dough after the thermomechanical
 329 treatment (visual observation and mean force measured) and then after printing and baking for each of
 330 the 10 trials.

331 The pictures after the thermomechanical treatment and the mean force (N) of the dough clearly show
 332 the broad range of dough texture obtained for each of the experimental plan. The differences of mean
 333 force between each trial were significant ($P < 0.05$) and ranges from 1.5 to 31.5 N for rye flour dough
 334 and from < 1 (force not detected) to 36.6 N for soy flour dough.

335 All the other variables studied (height, diameter, number of unaligned layers and number of spacing
 336 kept after baking on and inside the printed product) also demonstrated significant differences ($P < 0.05$)
 337 between the trials except for the number of cuts visible in the printed layers of 3D-printed product. This
 338 variable was not considered for the following analyses.

339 The post-print pictures provide a quick view of print quality and the dough's ability to hold the weight
 340 of the top layers. These pictures show that most of the trials allowed obtaining an acceptable stacking
 341 of the printed layers except for the following trials: 65 % - 8 min and 70 % - 10 min for rye flour dough
 342 and 72 % - 20 min for soy flour dough. The mean force could explain these results since these trials

343 represent the weakest forces obtained within each of the experimental plans. However, the stacking was
344 clearly not perfect for all the other trials, and we can observe that generally the printing quality increase
345 with higher mean force of the dough.

346 The pictures after baking show the ability of the paste to maintain the shape of the printed model under
347 the effect of oven heat (15 min at 185 °C). For each of the experimental plans, only three trials obtained
348 a final 3D-printed product similar to the print model: trials 60 % - 10 min, 58 % - 13 min, 60 % - 20
349 min for rye flour dough and trials 60 % - 15 min, 58 % - 20 min, 60 % - 25 min for soy flour dough.
350 The 3D-printed products obtained with these process parameters clearly show the cylindrical shape of
351 the model (without sagging) as well as the space between the printing lines on the surface of the product.
352 For the other trials, a deformation (spreading of the dough) of the products is observed. Overall, doughs
353 with higher water content seems to spread out more during baking. As demonstrated by [Masbernat et](#)
354 [al. \(2021\)](#) for wheat flour dough, the water/flour ratio is a crucial parameter to control the rheological
355 properties of the dough as well as their printability. Our results confirm that this water/flour ratio is also
356 important when preparing dough made from rye or soy flour.







357 3.3.1. Impact of the process parameters and their interactions on print quality and stability of
358 3D-printed products.

359 [Figure 4](#) shows results of principal component analysis (PCA) performed to present a map of 3D-printed
360 products from rye (Fig.3a) and soy (Fig.3b) flour depending on their print quality and the stability of
361 the model after printing and baking (principal variables) and the process parameters (explanatory
362 variables). The PCA shows that components 1 and 2 explain 79 % for rye flour products and 88.1 %
363 for soy flour products of the total variance. For each PCA, the component 1 explained the majority
364 (54% for rye flour and 62.5% for soy flour products) of the variables related to the quality and stability
365 (number of unaligned layers, number of spacing kept and mean force) of the printed model and was
366 associated to the water content. A lower water content is associated with a higher mean force and a
367 better stability of the 3D printed model (better preservation of spaces between printing lines and better
368 stacking). The dimensional profile (diameter and height) of 3D-printed products was supported by
369 component 2 (25% for rye flour and 25.6% for soy flour products). A higher diameter means that the
370 product has collapsed and is therefore negatively correlated with the height of the product.






371

372
373
374

Table 5
Impact of water content (%) and time of thermomechanical treatment (TMT) on mean force (N) of rye flour dough and on the visual observations after TMT, printing, and baking (15 min, 185°C).

Water (%)	TMT (min)	TMT	Mean force (N)	Printing	Baking
60	10		18.4 ± 0.5		
72	15		3.9 ± 0.2		
65	22		10.9 ± 0.2		
58	13		31.5 ± 0.5		
65	15		17.1 ± 1.9		
70	20		5.7 ± 0.1		
65	15		12.0 ± 0.5		
65	8		3.5 ± 0.2		
60	20		22.9 ± 1.1		
70	10		1.5 ± 0.1		

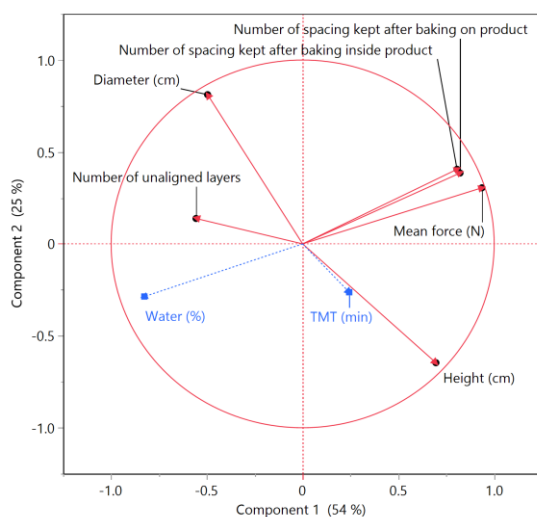
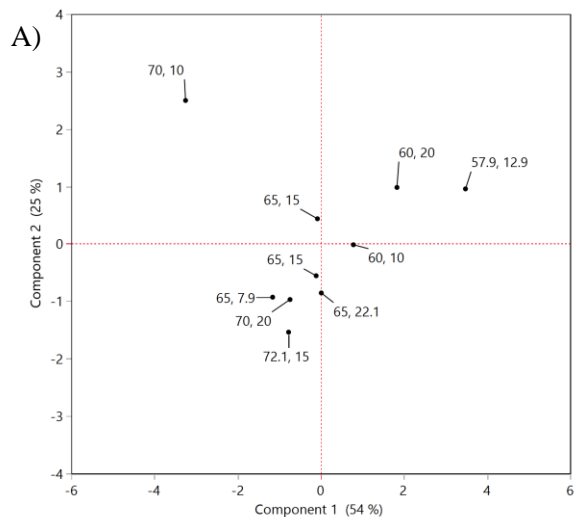
375 **Table 6**
 376 Impact of water content (%) and time of thermomechanical treatment (TMT) on mean force (N) of
 377 soy flour dough and on the visual observations after TMT, printing, and baking (15 min, 185°C).

Water (%)	TMT (min)	TMT	Mean force (N)	Printing	Baking
70	15		2.4 ± 0.2		
72	20		< 1 *		
65	20		5.8 ± 0.2		
65	13		4.9 ± 0.2		
70	25		3.1 ± 0.2		
65	27		11.0 ± 0.3		
60	15		23.2 ± 0.2		
58	20		36.6 ± 0.8		
65	20		9.4 ± 0.6		
60	25		31.1 ± 0.6		

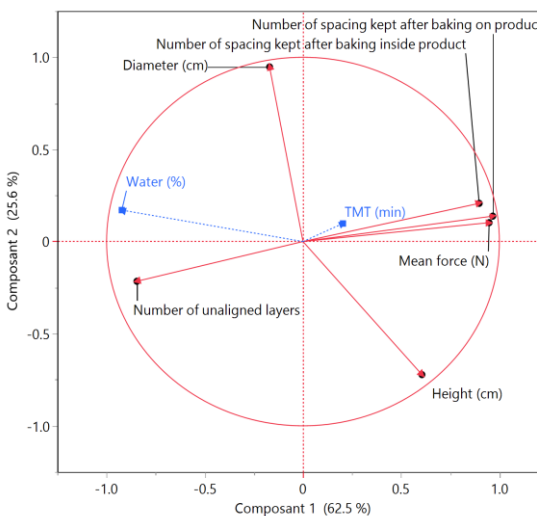
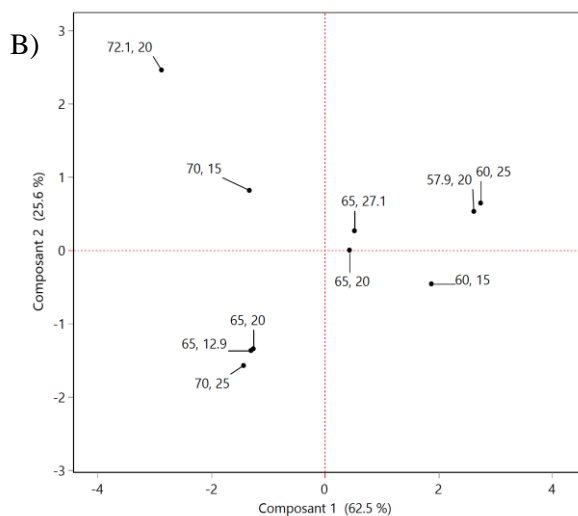
378 * Force below the detection level of the texture analyzer (< 1 N).

379 Water content and thermomechanical treatment had a significant impact ($P \leq 0.05$) on all dependent
380 variables except on the number of cuts visible in the printed layers ($P = 0.1$ for rye flour products and
381 $P = 0.4$ for soy flour products) which was not included in the subsequent statistical analyses. In addition,
382 water content had a lower P value (LogWorth = 18.356 for rye flour product and 21.813 for soy flour
383 product) than that thermomechanical treatment (LogWorth = 2.560 for rye flour product and 3.641 for
384 soy flour product) which indicates the higher importance of the total water content. The
385 thermomechanical treatment does not seem to explain the differences in quality and stability of 3D-
386 printed products for both flours (Fig. 4). However, the study of the interactions between the water
387 content and the thermomechanical treatment demonstrates the importance of considering these two
388 process parameters simultaneously. The interaction between these parameters had a significant impact
389 ($P \leq 0.05$) on diameter (for both flours), height (only for rye flour), mean force and spacing after baking
390 inside the 3D-printed product (only for soy flour). Figure 5 shows the impact of this interaction on
391 diameter (target of 3 cm) and height (target of 1.5 cm) for 3D-printed product made from rye flour.
392 When the water content is higher ($> 65\%$), the duration of the thermomechanical treatment has a greater
393 impact on the diameter and height of the 3D printed model. A too short treatment (~ 8 min) will lead to
394 a more collapsed product (larger diameter and smaller height). While a too long treatment (~ 22 min)
395 will lead to a more compact product (narrower diameter) but with a good height. For the soy flour
396 products, a similar interaction on diameter was observed. Figure 6 shows the significant interactions
397 ($P \leq 0.05$) between process parameters on mean force measured and on number of spacing kept after
398 baking inside the soy flour products. For recipes with a water content of less than 70% , we observe an
399 impact of the duration of the thermomechanical treatment on these variables. For example, a longer
400 treatment (~ 27 min) always allows a better preservation of the spaces between the printing lines as well
401 as a higher mean force. These results suggest that a longer thermomechanical treatment would allow
402 better print quality, but that this effect is limited when the water content becomes higher (nearly 70%).

403 Our results also demonstrated that the print quality and stability of 3D-printed products made from rye
404 or soy flours could be controlled by the simultaneous adjustment of the water content and the duration
405 of the thermomechanical treatment. The importance of these parameters on the rheological properties
406 (e.g. viscosity, G' and elasticity) on highly hydrated doughs (60 to 90% of water content) made from
407 wheat flour has already been demonstrated and was mainly explained by hydrothermal transformations
408 of starch and gluten proteins (Champenois et al., 1998; Masbernat et al., 2021). However, in our study,
409 we can suppose that other transformations occurs in the proteins network since we used rye flour (which
410 contains lower starch and gluten than wheat flour) and soy flour (which is gluten-free and contains very
411 little starch). Grossmann & Koehler, (2016) also suggested that non-gluten proteins would be more
412 involved in rye protein functionalities and technological properties of rye flour than gluten proteins.

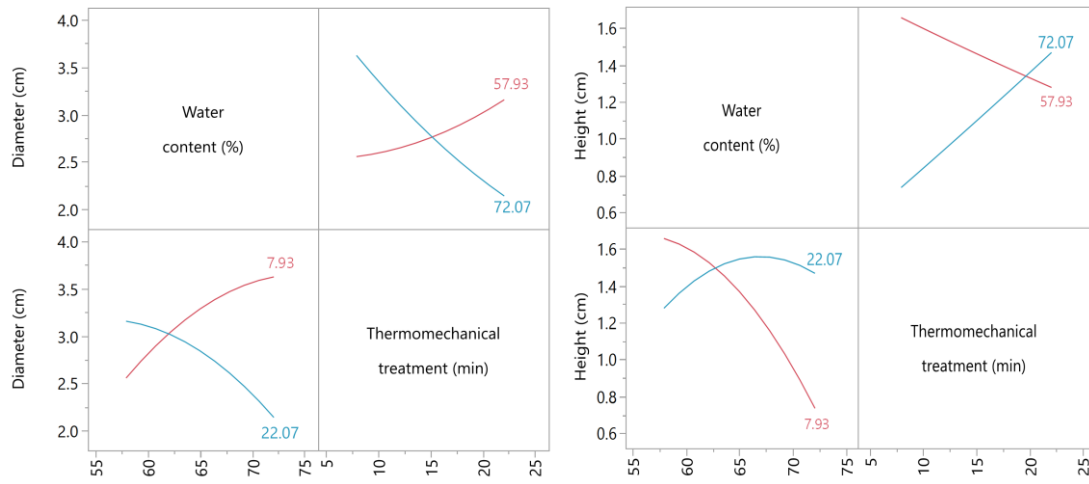


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414

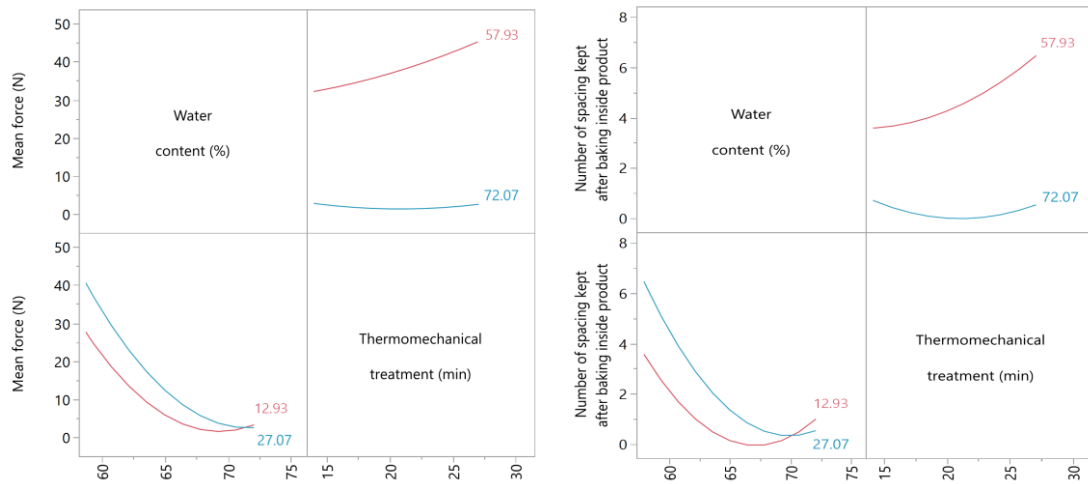
415 **Fig. 4.** Principal component analysis (PCA map and correlation circle) of 3D-printed products obtained
 416 from (A) rye flour dough and (B) soy flour dough. Each trial is identified by the process parameters of
 417 the experimental plan (water content (%), thermomechanical treatment (min)). Principal variables (red
 418 lines) were mean force (N), height (mm), diameter (mm), number of unaligned layers and number of
 419 spacing kept after baking (on surface and inside the printed product). Explanatory variables were
 420 process parameters (blue dashed lines).



421

422 **Fig. 5.** Significant interactions ($P \leq 0.05$) between process parameters (water content and
 423 thermomechanical treatment) on diameter (A) and on height (B) of 3D-printed products made from
 424 rye flour.

425



426

427 **Fig. 6.** Significant interactions ($P \leq 0.05$) between process parameters (water content and
 428 thermomechanical treatment) on mean force measured (A) and on number of spacing kept after baking
 429 inside the product (B) for soy flour dough.

430

431 3.3.2. Reverse-engineering approach to optimize thermomechanical process of rye and soy 432 dough

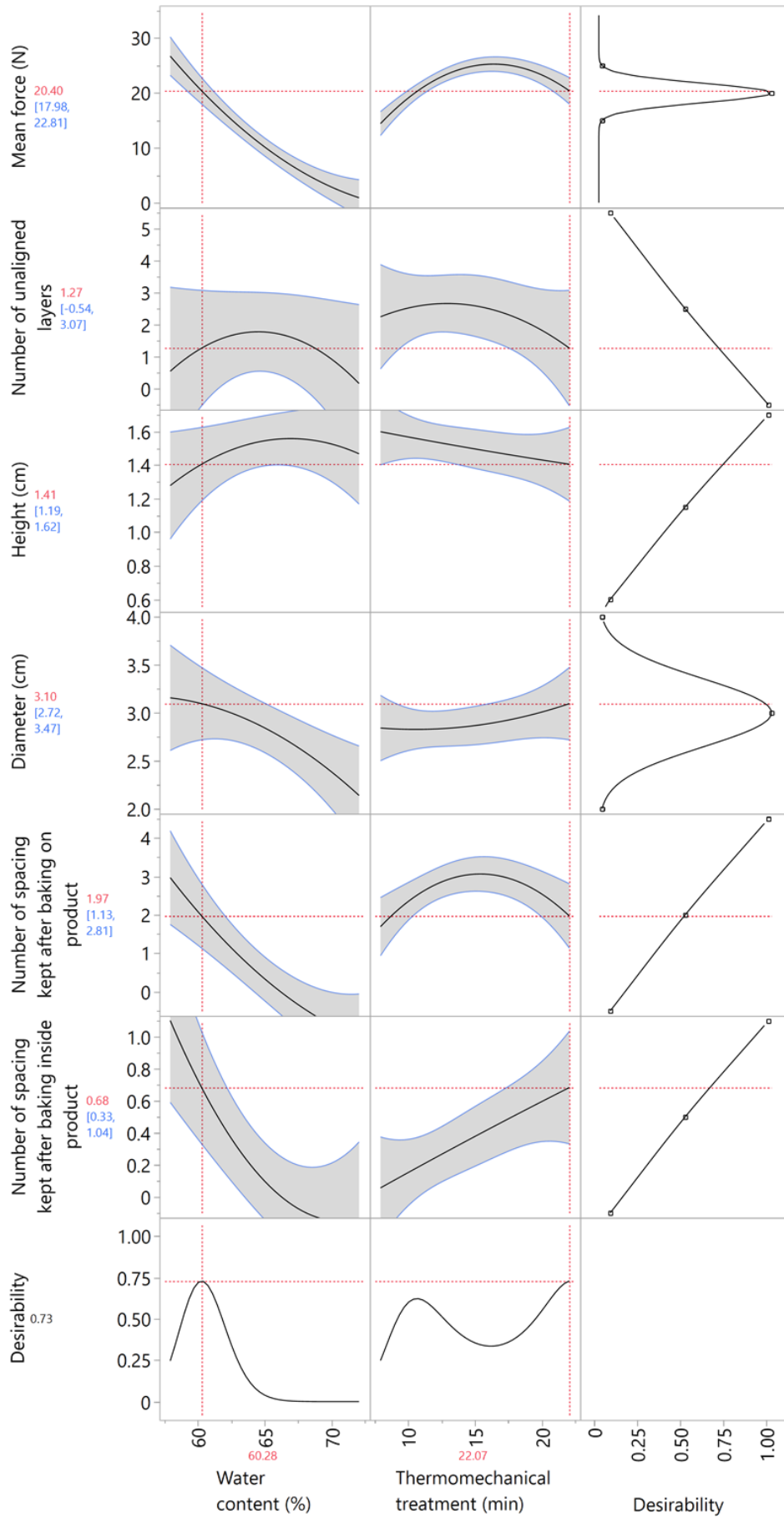
433 In order to optimize the print quality of the rye and soy dough, a reverse-engineering approach was
 434 applied using the desirability function as used by (Monnet et al., 2021). This method models the results
 435 of the experimental design and optimizes the different input variables (in our case the water content and
 436 the thermomechanical treatment time) by considering the performance of all output variables.

437 **Figure 7** shows the desirability function of the T170 rye flour recipes. Different optimization targets
 438 were selected for each textural and visual characteristic. We chose a mean force of 20 N (± 5 N) based
 439 on our observations during the trials of the experimental design. For example, during the TMT of the
 440 rye flour dough with a water content of 58 % and a duration of TMT of 13 min a higher resistance of
 441 the dough during the mixing and a blockage of the blades were observed possibly related to a too firm

442 (mean force of 31.5 N after TMT) or sticky dough. Further characterization of the textural and
443 rheological properties of this dough would be necessary. This targeted mean force allows us to obtain
444 a repeatable and homogeneous treatment without the blockage of the blades. For the diameter, 3 cm
445 was targeted because it corresponds to the value of the model, and that a too small diameter may mean
446 a non-optimal print quality and a too high diameter means a too liquid dough that does not hold the
447 shape. The height of the 3D-printed product was maximized because although the model targeted a
448 height of 1.5 cm, it was not a disadvantage to have a slight swelling during baking. The most important
449 was to avoid being under this value which would rather indicate a sagging of the product due to a bad
450 hold of the dough. For the others visual characteristics, we chose to minimize the number of unaligned
451 layers and maximize the number of spacing's kept on the surface and inside the product after baking to
452 get closer to the 3D-model. Optimizing all these outputs results in a desirability of 72.9 % when the
453 water content of the dough is 60.3 % and the TMT is 22.1 min. We also observed a 2nd peak for the
454 duration of the thermomechanical treatment at about 11 min, but the selection of this value leads to a
455 lower desirability (61 %). For soy flour dough, we only modified the targeted mean force and chose to
456 maximize this variable because during the trials of the experimental design we did not observe any
457 problem during the thermomechanical treatment or during the printing for high value of firmness (up
458 to 36.6 N). The optimized recipe reached a desirability of 83.7 % with 59.3 % of water in the dough
459 and a TMT of 27.1 min (Figure 8).

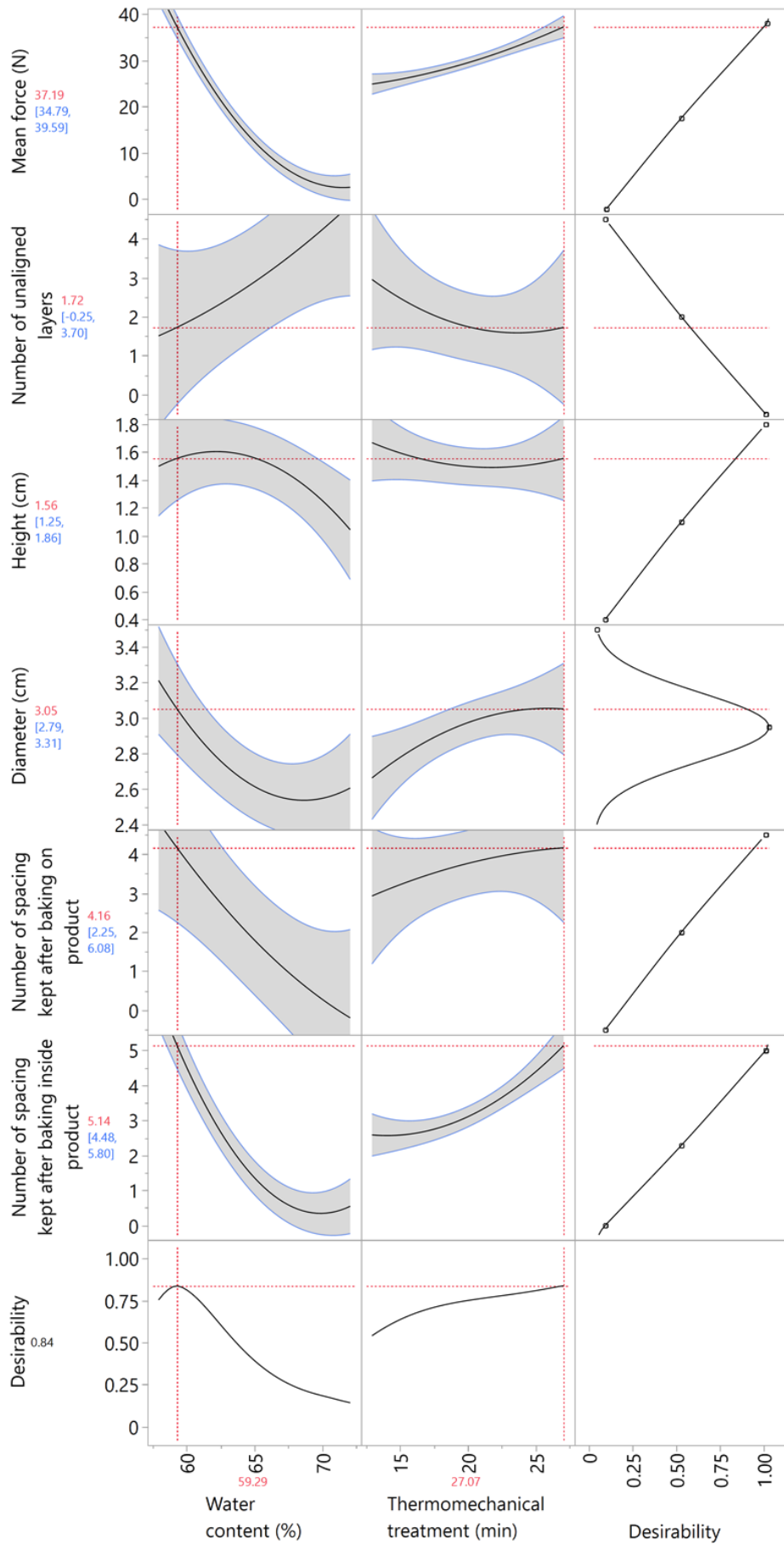
460 For each flour, the results of *F*-test and *R*² obtained for each variable measured are presented in Table
461 7 and 8. This additional information shows a significant effect ($p \leq 0.05$) of all the response variables.
462 For rye and soy flour products, a higher *F*-value (159.86 and 439.83 respectively) for the mean force
463 was observed indicating greater differences between the trials for this variable. For each flour, 2
464 variables reached a high *R*² value (≥ 0.85) while the *R*² varies between 0.39 and 0.70 for the other
465 variables. These results also explain why the desirability of each flour is not higher.

466 These parameters obtained with the reverse-engineering method show that for soy and rye flour, less
467 water and more time of thermomechanical treatment are needed to obtain an ideal product compared to
468 wheat flour (15 min and 65 % of water content) (Guénard-Lampron et al., 2021; Masbernat et al., 2021).
469 This can be explained by the difference in the starch content of these flours: wheat flour contains more
470 starch and needs sufficient hydration during the thermomechanical treatment to allow swelling and
471 gelatinization of the starch granules, which leads to an increase in viscosity (Doublier, 1990). Also,
472 Grossmann & Koehler, (2016) demonstrated that the presence of co-constituents in the rye flour
473 increase the temperature of the starch gelatinization which is in agreement with the longer
474 thermomechanical treatment needed to optimize the printability of our rye flour dough. However, the
475 two peaks of desirability depending on the TMT for rye flour are more difficult to explain. The quite
476 different compositions in terms of protein and fiber could possibly lead to a different network formation
477 mechanism. Future studies will be necessary to better understand how these new printable doughs are
478 structured (rheological and microstructural properties) following the thermomechanical treatment.



479

480 **Fig. 7.** Maximization of desirability to obtain a good printing quality of rye flour dough and a stable
 481 and consistent 3D model after baking.



482

483 **Fig. 8.** Maximization of desirability to obtain a good printing quality of soy flour dough and a consistent
 484 3D model after baking.

485 **Table 7**

486 Results of the F -test and R^2 for each variable measured on 3D-printed product made from rye flour
 487 dough.

	F -test (DF: 5, 24)	R^2
Mean force (N)	$F = 159.86$ $p > F \leq 0.0001$	0.97
Number of unaligned layers	$F = 3.74$ $p > F \leq 0.01$	0.44
Height (cm)	$F = 6.93$ $p > F \leq 0.004$	0.59
Diameter (cm)	$F = 4.70$ $p > F \leq 0.004$	0.49
Number of spacing kept after baking on product	$F = 27.90$ $p > F \leq 0.0001$	0.85
Number of spacing kept after baking inside product	$F = 5.69$ $p > F \leq 0.001$	0.54

488

489 **Table 8**

490 Results of the F -test and R^2 for each variable measured on 3D-printed product made from rye flour
 491 dough.

	F -test (DF = 5, 22)	R^2
Mean force (N)	$F = 439.83$ $p > F \leq 0.0001$	0.99
Number of unaligned layers	$F = 2.82$ $p > F \leq 0.04$	0.39
Height (cm)	$F = 10.19$ $p > F \leq 0.0001$	0.70
Diameter (cm)	$F = 8.12$ $p > F \leq 0.0002$	0.65
Number of spacing kept after baking on product	$F = 9.42$ $p > F \leq 0.0001$	0.69
Number of spacing kept after baking inside product	$F = 99.13$ $p > F \leq 0.0001$	0.96

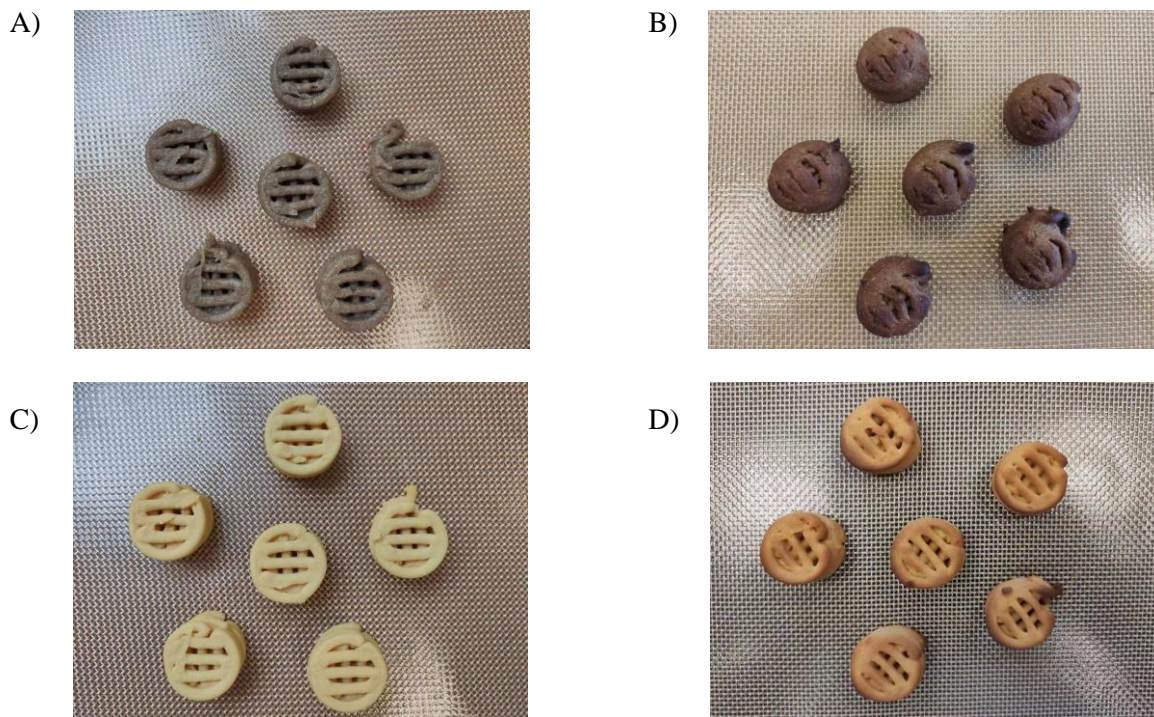
492

3.3.3. Validation of the optimized thermomechanical process parameters

493
494 The recipes optimized by the reverse-engineering approach needed validation. Thus, two samples were
495 made with soy flour and rye flour respectively with the optimized parameters (Figure 9). We were able
496 to confirm that these recipes are indeed optimal in terms of print quality before and after baking by the
497 visual observations and the dimensional measurements (height: 1.5 ± 0.1 cm and diameter: 3.0 ± 0.1
498 cm) of the 3D-printed products. The mean force obtained was 35.9 ± 1.7 N and 21.3 ± 0.6 N respectively
499 for soy and rye flour dough and allowed a good printing quality. However, for soy flour dough it was
500 observed that the firmness of the dough should not be higher for the 3D printer prototype used in this
501 study because the extrusion was sometimes more difficult (requires a lot of force to extrude the dough
502 through the nozzle). Table 9 and 10 show the comparison between the output average of the optimized
503 products obtained from rye and soy flours and the confidence interval of the desirability function. All
504 the outputs are in the confidence interval except the number of spacing kept after baking on product for
505 rye flour (3.83 for the optimized products while [1.13, 2.81] for the confidence interval) and the number
506 of spacing kept after baking inside product for soy flour (4 for the optimized products while [4.48, 5.8]
507 for the confidence interval). However, with our 3D print model, the maximum number of spaces
508 generated by the printer is 4 because of the size of the model (diameter of 3 cm), the nozzle diameter
509 (3.4 mm) and the filling rate (55 %). We can consider these values (3.83 and 4) as well optimized. These
510 results shows that the outputs of the optimized products obtained are consistent with the desirability
511 model.

512 The reverse-engineering method is appreciated when we deal with new printable recipes using flours
513 less studied than wheat flour. Soy and rye flours have quite different and little-known characteristics in
514 terms of water absorption, printability potential or deformation of the dough during baking, for example.
515 The reverse-engineering method associated with a design of experiments makes it possible to model
516 these characteristics with a limited number of experiments, considering several parameters at the same
517 time. Once the model is established, the characteristics included in this model can be predicted and the
518 ideal conditions can be determined according to the desired results, without the need to proceed with a
519 trial-and-error approach or to evaluate these specific conditions. For the optimization of other flours, it
520 would be necessary to start with the pre-tests to define the limits of the parameters to be varied, but this
521 approach remains applicable even for flours or recipes with more complex properties.

522



524

525 **Fig. 9.** Photos of optimal recipes with rye flour before (A) and after (B) baking and with soy flour before
 526 (C) and after (D) baking.

527 **Table 9**
 528 Comparison between the output average of the optimized products obtained from rye flour
 529 and the confidence interval of the desirability function.

	Lower bound of the confidence interval	Output average of the optimized products	Upper bound of the confidence interval
Mean force (N)	17.98	21.28	22.81
Number of unaligned layers	-0.54	0.67	3.07
Height (cm)	1.19	1.50	1.62
Diameter (cm)	2.72	2.95	3.47
Number of spacing kept after baking on product	1.13	3.83	2.81
Number of spacing kept after baking inside product	0.33	0.67	1.04

530

531 **Table 10**
 532 Comparison between the output average of the optimized products obtained from soy flour
 533 and the confidence interval of the desirability function.

	Lower bound of the confidence interval	Output average of the optimized products	Upper bound of the confidence interval
Mean force (N)	34.81	35.95	39.61
Number of unaligned layers	-0.25	3.50	3.70
Height (cm)	1.25	1.67	1.86
Diameter (cm)	2.79	3.08	3.31
Number of spacing kept after baking on product	2.25	3.83	6.08
Number of spacing kept after baking inside product	4.48	4.00	5.80

534

535 4. Conclusions

536 Thanks to our 3-steps approach including two screening steps (from a bibliographic study to an
 537 experimental strategy) and one optimization step based on experimental designs and reverse-
 538 engineering method, we were able to propose two new edible inks with interesting nutritional
 539 composition and good print quality.

540 Regarding the bibliographic study on the 25 flours, our main criteria was the nutritional aspect. The
 541 protein and fiber contents were considered as a factor to classify the different flours. This classification
 542 could be further developed in future studies by including more indicators related to protein quality such
 543 as the content of essential amino acids, digestibility and bioavailability of proteins as discussed by
 544 [Floret, Monnet, Micard, Walrand, & Michon \(2021\)](#). Fiber quality could also be represented with the
 545 content of different fiber types such as dietary fiber and FODMAPs ([Atzler, Sahin, Gallagher, Zannini,
 546 & Arendt, 2021](#)). Economic and environmental criteria were also considered for the selection of flours.
 547 However, it was difficult to select the flours according to their environmental criteria (PEF score and
 548 LCA data) since these were not available for all the flours studied. In future studies, these criteria should
 549 still be considered when formulating new innovative 3D printed foods to offer a more sustainable diet.

550 Regarding the printability, soy and rye flour dough showed a good potential for 3D printing and the
 551 reverse-engineering approach allowed to optimize the water content and the duration of the
 552 thermomechanical treatment for each flour to obtain a good printing quality. The water content
 553 explained the main problems in terms of printing quality and stability of the products. A similar water
 554 content was obtained for the optimization of both flour dough (60 % for rye flour and 59 % for soy
 555 flour). However, interactions were observed between the two process parameters studied indicating that
 556 it is necessary to adjust simultaneously the water content and the duration of the thermomechanical
 557 treatment. A longer treatment was needed to optimize printing quality and stability of soy flour dough

558 (27 min) compared to rye flour dough (22 min). This reverse-engineering approach was therefore
559 conclusive in determining the specific parameters to be use for the preparation of these two printable
560 doughs and would be relevant to use for the development of new printable matrices from other flours.

561 In future research, additional instrumental measurements (e.g., rheological, and microstructural
562 properties) would be necessary to better understand the structuring of dough following
563 thermomechanical treatment. The usefulness of this treatment would also be questionable when using
564 low-starch flour since this treatment is based on the structuring of the dough by the gelatinization of the
565 starch. A sensory characterization and a consumer study should also be realized on 3D-printed and
566 baked product.

567

568 Declaration of interests

569 The authors declare that they have no known competing financial interests or personal relationships that
570 could have appeared to influence the work reported in this paper.

571

572 Acknowledgements

573 This work was supported by a single interministerial fund (FR) and by regional co-funding (Hauts de
574 France et Auvergne Rhône Alpes) through a collaborative R&D project certified by Vitagora,
575 Euramaterials and Cimes clusters.

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Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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