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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-04133668

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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- 6 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
- 12 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
- 17 experimental screening of five flours was conducted. Thanks to the previous steps, soy and rye flour
- 18 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.
- 21 The reverse engineering approach demonstrated that the optimal water content is similar for both flour
- 22 (Soy: 59 %, Rye: 60 %), but the optimal duration of the thermomechanical treatment is quite different
- 23 (Soy: 27 min, Rye: 22 min).
- 24 Industrial relevance text
- 25 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
- 27 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
- 31 (water content and duration of thermomechanical treatment) was conducted and a predictive model was
- 32 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
- 34 be applied for the development of new edible ink using other flours or integrating different ingredients.
- 35 Keywords: 3D food printing, dough, food processing, desirability function, reverse engineering

1. Introduction

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

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

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

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

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

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

2. Material and Methods

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

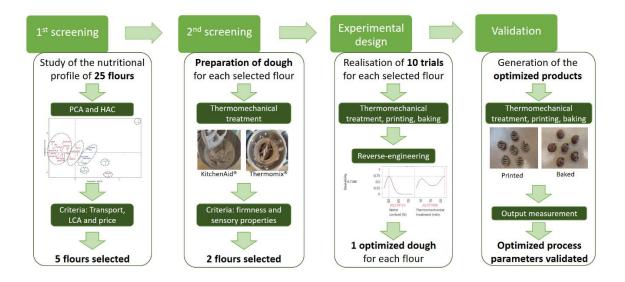


Fig.1. Experimental scheme of the two screening steps followed by the experimental design and the final validation of the process parameters studied.

2.1. First screening of different flours: Bibliographic research

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

- Definition of the nutritional criteria

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

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

- Definition of the ecological and economic criteria

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

2.2. Second screening from an experimental approach with printing product

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

2.2.1. Preparation of the printable dough

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

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

2.2.2. 3D-Printing

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

2.2.3. Selection criteria

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

- Firmness of the dough

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

- Sensory aspect of the dough

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

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

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

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

2.3.1. Analyzes of 3D-printed product

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

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

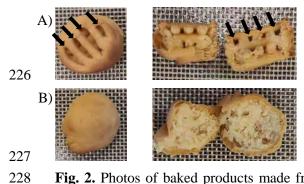


Fig. 2. Photos of baked products made from soy flour dough showing A) a product with all spaces retained (TMT of 25 min, water content of 60 %) and B) a product with no space left (TMT of 15 min, water content of 70 %).

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 ($P \le 0.05$) of the independent variables on each
- of the response variable were evaluated by a multiple regression analysis and were ranked according to
- their LogWorth (-log10(p value)).

236 3. Results and discussion

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3.1. Selection of 5 flours from the first screening

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

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

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

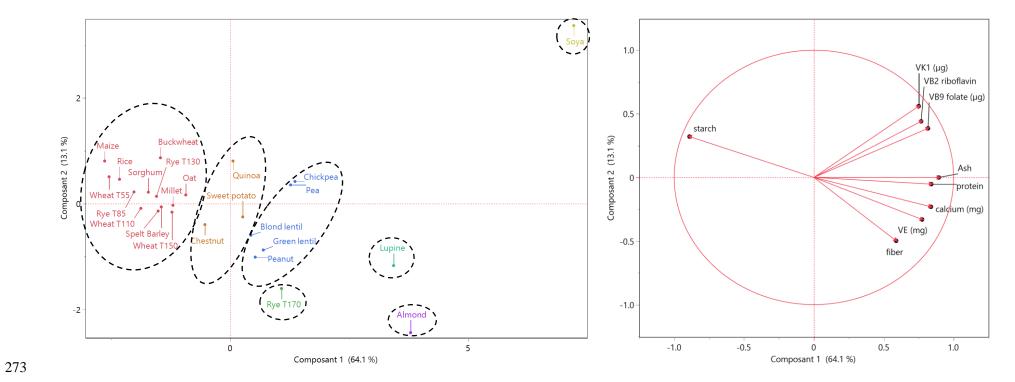


Fig. 3. Principal component analysis (PCA map of the flours and correlation circle of the nutritional components) of 25 flours. The dotted circles represent the 7 groups of flours obtained by a HAC analysis based on their similarities in terms of nutritional composition.

Table 2
 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 CO2 eq/kg of product)	Eutrophication freshwater (kg P eq/kg of product)	Land use (Pt/kg of product)	Water scarcity (m3 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

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

3.2. Selection of 2 flours from the second screening

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

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

Table 3Comparison 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

^{*}The intensity of the negative sensory aspect perceived is represented by the symbol "+".

3.3. Generation of different doughs from the 2 selected flours

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

Table 4Experimental design of 10 trials for A) soy and B) rye flour

A)		Water	Thermomechanical	B) _		Water	Thermomechanical
	Trial	content	treatment		Trial	content	treatment
_		(%)	(min)			(%)	(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

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

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

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

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

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

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

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

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

Table 5Impact 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		900
65	15		12.0 ± 0.5		200
65	8		3.5 ± 0.2		300
60	20		22.9 ± 1.1		
70	10		1.5 ± 0.1		

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

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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		

^{*} Force below the detection level of the texture analyzer (< 1 N).

Water content and thermomechanical treatment had a significant impact ($P \le 0.05$) on all dependent variables except on the number of cuts visible in the printed layers (P = 0.1 for rye flour products and P = 0.4 for soy flour products) which was not included in the subsequent statistical analyses. In addition, water content had a lower P value (LogWorth = 18.356 for rye flour product and 21.813 for soy flour product) than that thermomechanical treatment (LogWorth = 2.560 for rye flour product and 3.641 for soy flour product) which indicates the higher importance of the total water content. The thermomechanical treatment does not seem to explain the differences in quality and stability of 3Dprinted products for both flours (Fig. 4). However, the study of the interactions between the water content and the thermomechanical treatment demonstrates the importance of considering these two process parameters simultaneously. The interaction between these parameters had a significant impact $(P \le 0.05)$ on diameter (for both flours), height (only for rye flour), mean force and spacing after baking inside the 3D-printed product (only for soy flour). Figure 5 shows the impact of this interaction on diameter (target of 3 cm) and height (target of 1.5 cm) for 3D-printed product made from rye flour. When the water content is higher (> 65 %), the duration of the thermomechanical treatment has a greater impact on the diameter and height of the 3D printed model. A too short treatment (~ 8 min) will lead to a more collapsed product (larger diameter and smaller height). While a too long treatment (~ 22 min) will lead to a more compact product (narrower diameter) but with a good height. For the soy flour products, a similar interaction on diameter was observed. Figure 6 shows the significant interactions $(P \le 0.05)$ between process parameters on mean force measured and on number of spacing kept after baking inside the soy flour products. For recipes with a water content of less than 70 %, we observe an impact of the duration of the thermomechanical treatment on these variables. For example, a longer treatment (~27 min) always allows a better preservation of the spaces between the printing lines as well as a higher mean force. These results suggest that a longer thermomechanical treatment would allow better print quality, but that this effect is limited when the water content becomes higher (nearly 70%). Our results also demonstrated that the print quality and stability of 3D-printed products made from rye or soy flours could be controlled by the simultaneous adjustment of the water content and the duration of the thermomechanical treatment. The importance of these parameters on the rheological properties (e.g. viscosity, G' and elasticity) on highly hydrated doughs (60 to 90 % of water content) made from wheat flour has already been demonstrated and was mainly explained by hydrothermal transformations of starch and gluten proteins (Champenois et al., 1998; Masbernat et al., 2021). However, in our study, we can suppose that other transformations occurs in the proteins network since we used rye flour (which contains lower starch and gluten then wheat flour) and soy flour (which is gluten-free and contains very little starch). Grossmann & Koehler, (2016) also suggested that non-gluten proteins would be more involved in rye protein functionalities and technological properties of rye flour than gluten proteins.

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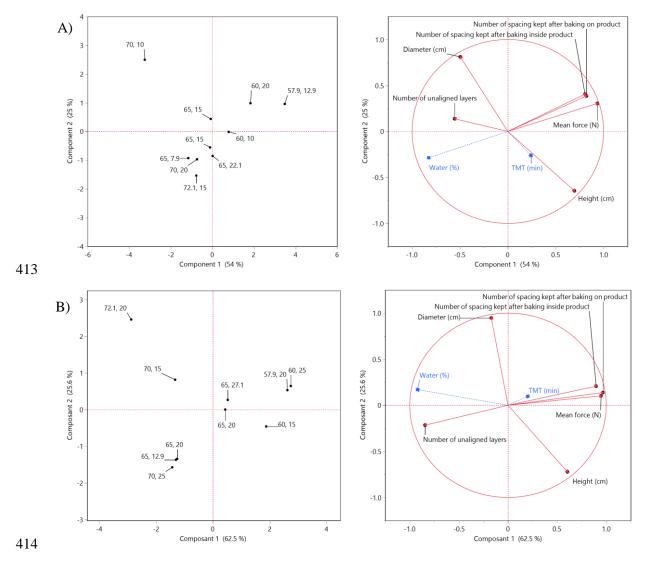


Fig. 4. Principal component analysis (PCA map and correlation circle) of 3D-printed products obtained from (A) rye flour dough and (B) soy flour dough. Each trial is identified by the process parameters of the experimental plan (water content (%), thermomechanical treatment (min)). Principal variables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

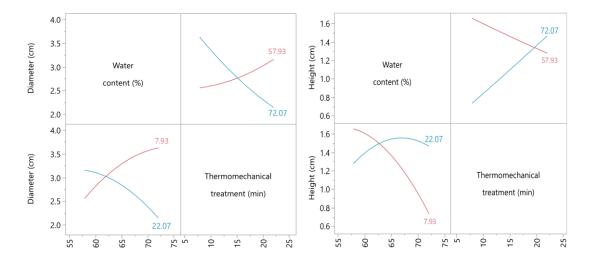


Fig. 5. Significant interactions ($P \le 0.05$) between process parameters (water content and thermomechanical treatment) on diameter (A) and on height (B) of 3D-printend products made from rye flour.

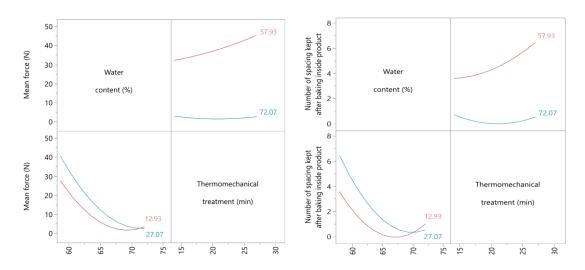


Fig. 6. Significant interactions ($P \le 0.05$) between process parameters (water content and thermomechanical treatment) on mean force measured (A) and on number of spacing kept after baking inside the product (B) for soy flour dough.

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

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

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

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

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

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

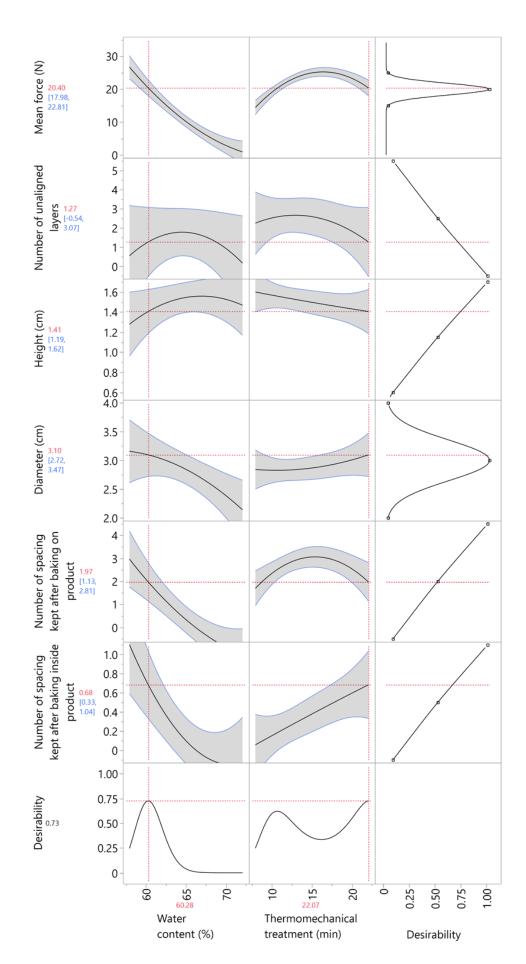


Fig. 7. Maximization of desirability to obtain a good printing quality of rye flour dough and a stable and consistent 3D model after baking.

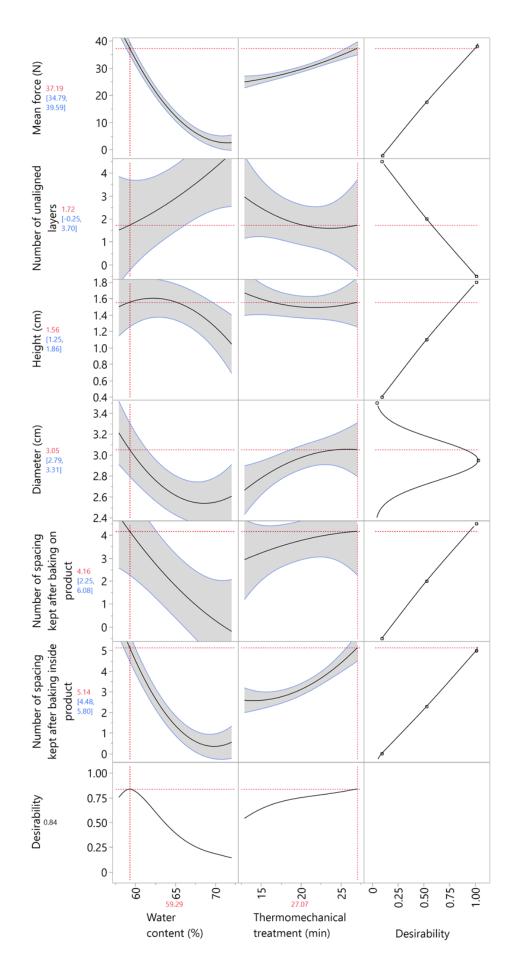


Fig. 8. Maximization of desirability to obtain a good printing quality of soy flour dough and a consistent 3D model after baking.

Table 7
 Results of the *F*-test and R² for each variable measured on 3D-printed product made from rye flour dough.

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

Table 8

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

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

3.3.3. Validation of the optimized thermomechanical process parameters

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

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

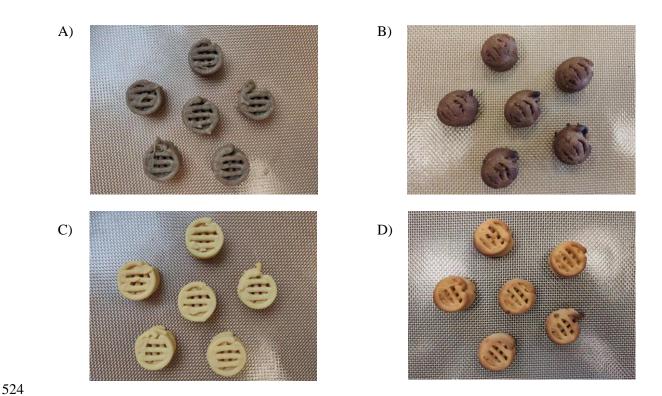


Fig. 9. Photos of optimal recipes with rye flour before (A) and after (B) baking and with soy flour before (C) and after (D) baking.

Table 9Comparison between the output average of the optimized products obtained from rye flour 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

Table 10Comparison between the output average of the optimized products obtained from soy flour 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

4. Conclusions

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

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

Regarding the printability, soy and rye flour dough showed a good potential for 3D printing and the reverse-engineering approach allowed to optimize the water content and the duration of the thermomechanical treatment for each flour to obtain a good printing quality. The water content explained the main problems in terms of printing quality and stability of the products. A similar water content was obtained for the optimization of both flour dough (60 % for rye flour and 59 % for soy flour). However, interactions were observed between the two process parameters studied indicating that it is necessary to adjust simultaneously the water content and the duration of the thermomechanical 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 thermomechanical treatment. The usefulness of this treatment would also be questionable when using 563 low-starch flour since this treatment is based on the structuring of the dough by the gelatinization of the 564 starch. A sensory characterization and a consumer study should also be realized on 3D-printed and 565 566 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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Conflict of Interest

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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