



**HAL**  
open science

## Preconditioner influence on twin-screw extrusion cooking of starch-based feed pellets: The example of Fish Feed

Asma Chaabani, Laurent Labonne, Vanessa Durrieu, Antoine Rouilly, Fabien Skiba, Philippe Evon

### ► To cite this version:

Asma Chaabani, Laurent Labonne, Vanessa Durrieu, Antoine Rouilly, Fabien Skiba, et al.. Preconditioner influence on twin-screw extrusion cooking of starch-based feed pellets: The example of Fish Feed. *Aquacultural Engineering*, 2022, 98, pp.102268. 10.1016/j.aquaeng.2022.102268 . hal-03702896

HAL Id: hal-03702896

<https://hal.inrae.fr/hal-03702896>

Submitted on 22 Jul 2024

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



Distributed under a Creative Commons Attribution - NonCommercial 4.0 International License

1           **Preconditioner influence on twin-screw extrusion cooking of**  
2           **starch-based feed pellets: the example of *Fish Feed***

3  
4  
5           Asma Chaabani <sup>a,b</sup>, Laurent Labonne <sup>a</sup>, Vanessa Durrieu <sup>a</sup>, Antoine Rouilly <sup>a</sup>, Fabien Skiba <sup>b</sup>,  
6           Philippe Evon <sup>a,\*</sup>

7  
8  
9           <sup>a</sup> Laboratoire de Chimie Agro-Industrielle, LCA, Université de Toulouse, INRAE, Toulouse,  
10           France

11                                   <sup>b</sup> Nutricia, Haut-Mauco, France

12  
13  
14                                   \* Corresponding author.

15                           E-mail address: [Philippe.Evon@toulouse-inp.fr](mailto:Philippe.Evon@toulouse-inp.fr) (Ph. Evon).

16

## 17 **Abstract**

18 This work compares the performance of pellets for adult Trout obtained in two different  
19 processing conditions. The first one consists of adopting the traditional *Fish Feed* process  
20 based on a preconditioning steam treatment step before twin-screw extrusion. The second one  
21 was a simplified process based only on a twin-extruder ensuring both the cooking and the  
22 extrusion of fish pellets in a single step. Six formulations were obtained while-maintaining  
23 feed specifications (36 % of proteins and 30 % of lipids). The comparison between the two  
24 processing methodologies (*i.e.*, with or without preconditioning) was then based on two  
25 fundamental aspects. Firstly, the gelatinization rate of starch contained inside pellets was  
26 determined. Secondly, the pellet's characteristics (bulk density, expansion ratio, diameter and  
27 pellet's porosity) and their usage properties (oil leakage rate, durability, hardness and  
28 floatability) were evaluated. The present work has evidenced that it is possible to produce  
29 pellets without preconditioning while ensuring a total starch gelatinization and maintaining  
30 their usage properties. The *Fish Feed* process has thus been partially simplified.

31

## 32 **Keywords**

- 33 • *Fish Feed*
- 34 • Twin-screw extrusion process
- 35 • Preconditioning
- 36 • Moisture
- 37 • Expansion
- 38 • Starch gelatinization
- 39 • Physical properties

40

## 41 **1. Introduction**

42 Historically, *Fish Feed* has always been produced in a pelletized form. Using a simple  
43 technology such as a ring die pellet mill, pelletized feed is produced. It is rather stable and  
44 cheap in production cost. However, its main disadvantage is that there are limited possibilities  
45 to influence the physical properties of pellets, especially their shape and density (**Burel, 2017;**  
46 **Terpstra, 2015**). Yet, the diameter of commercial extruded *Fish Feed* can range from 0.5 to  
47 30 mm. In fact, the size of the pellet must adapt the fish size because this animal swallows the  
48 feed as an intact particle (**Terpstra, 2015**). Furthermore, thanks to the extrusion technology, it  
49 is possible to have a wide range of bulk densities (from 350 to 750 g/L). Both sinking and  
50 floating pellets can ensure satisfactory growth (**Craig, 2009**). However, some farmers prefer  
51 floating feed, which can help them to directly observe the feeding intensity and therefore to  
52 adjust feeding rates (**Khater et al., 2014**). The three main constituents inside pelletized feed  
53 are starch, proteins and lipids. If the last two ones are added for nutritional considerations, the  
54 main interest to add starch is to give cohesion to the pellet. However, starch is only partially  
55 cooked inside such pellets, making its digestion by fish more difficult. For the same reason,  
56 pellets become soft in the presence of water, thus contributing to oil leakage (**Terpstra,**  
57 **2015**). To overcome these limitations, extrusion has found its place in aquaculture for nearly  
58 fifty years, becoming nowadays the primarily used technique for aquafeeds ( **Kraugerud and**  
59 **Svihus, 2011; Rokey, 1994; Sørensen, 2012**). Thanks to this continuous technology,  
60 extruded feeds are of better quality with a clear improvement in some physical characteristics,  
61 e.g., a firm structure, the absence of dust, adjustable size and shape, and the stability in water  
62 (**Terpstra, 2015**). . Extrusion leads also to higher inclusion of lipids, and the density of the  
63 pellets is easily adjustable. This technique may be defined as a continuous cooking process in  
64 which a food or feed material is forced to flow, under elevated pressure, temperature and  
65 shear, through a die to give unique physical and chemical functionality to food materials  
66 (**Altan et al., 2009; Riaz and Rokey, 2011**). During this thermomechanical process, different  
67 functions can be performed such as ingredients agglomeration, starch gelatinization,  
68 expansion, dehydration, mixing, homogenization, protein denaturation, pasteurization,  
69 shearing, texturizing, and product shaping (**Riaz and Rokey, 2011**).

70 In *Fish Feed* manufacturing, the technological process applied nowadays involves the  
71 following operations: raw materials preparation (weighing, mixing, grinding and sieving),  
72 preconditioning, extrusion, drying, fat coating, cooling, sieving and packaging. An extrusion  
73 system consists of a barrel situated between the preconditioner and the pelletizing knife. This

74 key part of the extruder is made of one or two rotating screws for the cooking and raw  
75 materials processing. Extruders are classified in different ways, but the most common ones  
76 are based on the screw design (single- or twin-screw extruders), the source of heat and the  
77 level of moisture in the material during the process (dry and wet extruders) (**Riaz and Rokey,**  
78 **2011**). In *Fish Feed*, most of the extruders used are single-screw ones. However, and even if  
79 the twin-screw machines are much more expensive, these are known for their versatility (**Riaz**  
80 **and Rokey, 2011**).

81 Positioned between the dry mix feeder and the extruder barrel, the preconditioner is  
82 generally used in animal feed extrusion processing (*e.g.*, aquafeeds), especially when the  
83 moisture content exceeds 18 % (**Bortone, 2017; Riaz and Rokey, 2011**). During  
84 preconditioning, the raw material particles are mixed homogeneously and continuously with  
85 liquid water and steam for a given time before extrusion, to hydrate and heat the particles  
86 (**Riaz and Rokey, 2011**). Hydrothermal cooking of the raw material starts during this step,  
87 resulting in the swelling of flour particles as well as in the pre-gelatinization of starch  
88 granules. At this level, particles are heated to a temperature of 80 °C to 95 °C (**Rokey, 1994**),  
89 and hydrated to a moisture content ranging between 18 % and 30 % (w/w) (**Riaz and Rokey,**  
90 **2011; Strahm, 2000**). As pre-heating is much faster than pre-humidifying, the  
91 preconditioning step is thus firstly controlled by the transfer of moisture into the particles  
92 (**Lavocat and Raymond, 2018**). On the one hand, the hydration time is longer with a bigger  
93 size of the particles. On the other hand, it can be reduced thanks to a warmer environment. An  
94 efficient moistening and swelling of the particles can only be achieved thanks to a suitable  
95 residence time of the raw material in the preconditioner. In other terms, the preconditioner  
96 function is to provide the extruder with an evenly moistened and preheated mix. The quality  
97 of the preconditioning stage thus depends on the efficiency of heating, hydration, and mixing  
98 (**Riaz and Rokey, 2011**).

99 The first developed equipment was a single-shaft preconditioner. The latter ran at high  
100 speed to attain a good mixing but had a reduced retention time of only 30 s or less, which is  
101 now considered ineffective by today's standards (**Riaz, 2000**). This is why a double-shafted  
102 preconditioner was developed, and almost dominates nowadays the pellet feed industry  
103 (**Pandey, 2018**). Such preconditioner is made of a tank in which counter-rotating twin-shafts  
104 equipped with paddles are located. Water is injected with two nozzles located on both sides of  
105 the tank entrance. Steam is generally injected through two to six points on the first half of the  
106 tank. The spray nozzles help to distribute water homogeneously on the raw particles and thus

107 reduce the required mixing intensity (**Riaz, 2000**). The performance of the preconditioning  
108 step depends on several operating conditions, *e.g.*, the number and orientation of the paddles,  
109 the steam, and water flow rates in relation with the raw material one, and the rotation speed of  
110 the twin-shafts (**Riaz and Rokey 2011**). All these conditions have an impact on the efficiency  
111 of the heating and pre-humidifying process, as they influence the residence time of the raw  
112 material inside the preconditioner, its filling ratio, its mixing ability, and lastly the energy and  
113 mass transfers. Depending on the paddle configuration (mounted with direct or reverse pitch),  
114 the average filling ratio can be modulated and, consequently, the mean residence time of the  
115 mixture inside the preconditioner (**Riaz and Rokey, 2011**). The filling ratio is defined as the  
116 volume occupied by the raw material in relation to the total volume of the preconditioner.  
117 Increasing this parameter improves the hydration and temperature uniformity of the mix  
118 (**Bortone, 2017**). In addition, depending on the flow rate of steam injected, the starch  
119 gelatinization rate varies: the more steam injected, the more starch gelatinization occurs  
120 (**Lavocat and Raymond, 2018**). A reduced flow rate of the raw material also tends to  
121 increase its mean residence time in the preconditioner, thus resulting in improved  
122 gelatinization of starch (**Lavocat and Raymond, 2018**). Lastly, a reduced rotation speed of  
123 the twin-shafts increases the mean residence time in the preconditioner. However, a slight  
124 decrease in the gelatinization rate of starch may be evidenced in such a situation (**Lavocat**  
125 **and Raymond, 2018**).

126 In conclusion, the preconditioner (**Fig. 1**) is considered today as vital equipment in *Fish*  
127 *Feed* manufacturing. Its performance indeed directly influence the starch pre-gelatinization  
128 rate, which can reach 60-70 % at the preconditioner outlet, or even more (up to around 90 %)   
129 when working with a particularly low inlet flow rate of solid material (**Lavocat and**  
130 **Raymond, 2018**). **Riaz and Rokey (2011)** had reported that to improve the extrusion-  
131 cooking step and the final product quality, at least one-third of starch gelatinization must  
132 occur in the preconditioner. The latter contributes to an efficient heat transfer through friction  
133 inside the extruder barrel, just as a reduction in its energy consumption (**Sørensen, 2012**). It  
134 also lengthens the life of the extruder by reducing the abrasive wear of the barrel and screws  
135 (**Sørensen, 2012**). Lastly, the preconditioner is also interesting for sanitary purposes as this  
136 heating step can help to control or reduce salmonella levels in the raw materials (**Riaz and**  
137 **Rokey, 2011**). At an industrial scale, using a preconditioner can also lead to increasing the  
138 extruder productivity, and to an improvement in the final product quality, *e.g.* its water

139 stability (**Bortone, 2017**), density, and hardness. Recently, new preconditioner designs have  
140 even been developed for new ranges of aquafeed (**Riaz, 2019**).

141 Next to the preconditioner, the conditioned feed mixture is thus transferred to the extruder  
142 to be transported, melted, cooked, heated, and mixed at high temperature (120-130°C) and  
143 high pressure (20-30 bar) (**Sørensen, 2012; Terpstra, 2015**), and finally shaped through the  
144 die. This device contains as many holes as required. The die determines the extruded pellet's  
145 shape and size (**Riaz and Rokey 2011; Terpstra, 2015**). Finally, pellets are cut to the desired  
146 length, thanks to a rotating knife positioned immediately after the die. When the extrusion-  
147 cooked solid material leaves the die, an expansion phenomenon occurs through water  
148 evaporation. Small water droplets that were dispersed inside the solid pass from high to  
149 atmospheric pressure. Moisture vaporizes and expands pellets through the formation of pores  
150 (**Williams, 2000**). These small pores are then filled by oil at the moment of the coating extra-  
151 step in the case of the high-energy diets (**Sørensen et al., 2010**).

152 In the extrusion system, energy comes from two main sources, *i.e.*, mechanical, and  
153 thermal (**Sørensen, 2012**). The first one is known as the specific mechanical energy (SME)  
154 which is the amount of mechanical energy dissipated as heat inside the material (**Altan et al.,**  
155 **2009**). According to **Mercier et al (1989)**, SME is the product of torque and screw speed  
156 divided by mass flow rate. SME provides a good characterization of the extrusion process  
157 (**Altan et al., 2009**) and its value indicates the extent of molecular breakdown and eventually  
158 material degradation (**Godavarti and Karwe, 1997**).

159 The extrusion technology has many advantages among which versatility and its ability to  
160 obtain a high-quality product (**Gaosong and Vasanthan, 2000; Riaz and Rokey, 2011**).  
161 Given the fact that extrusion is a high-temperature short time (HTST) heating process,  
162 degradation of food nutrients is minimized, and the digestibility of dietary components is  
163 improved (**Riaz and Rokey, 2011**). In addition, it contributes to destroy the anti-nutritional  
164 compounds, some undesirable enzymes and microorganisms (**Riaz and Rokey, 2011**), just as  
165 it inactivates heat resistant toxins (*e.g.* aflatoxin) (**Saalia and Philips, 2011**). When extrusion  
166 conditions are not well optimized, it can nevertheless reduce the availability of some  
167 nutrients, generate the cross-linking of lysine, and also destroy some vitamins and other  
168 thermolabile substances (**Rokey, 1994**).

169 For salmonids, it is mainly dietary proteins and lipids that allow them to cover their energy  
170 needs. These animals have limited capacity to efficiently use and process dietary

171 carbohydrates (**Médale and Guillaume, 1999**). This results in feed rich in proteins and lipids,  
172 and with low content in carbohydrates (**Burel, 2017; Terpstra, 2015**). Pellets with high-fat  
173 content are necessary to satisfy their nutritional requirements. However, during extrusion, the  
174 amount of fat that can be added is limited as it behaves as a lubricant, thus leading to a loss of  
175 mechanical shear (**Pandey, 2018**). To overcome this disadvantage of extrusion, oil is  
176 commonly added through a coating extrastep. Firstly, the fat coating was practiced with  
177 atmospheric systems like drum coaters, mist-coating units, and paddle mixers (**Bortone,**  
178 **2006**). However, for a special feed diet like high-energy feed for salmon, oil coating can only  
179 be successfully achieved with vacuum coaters. Under vacuum, the air inside the feed pores is  
180 first removed, and coating then allows the injected oil to fill these pores when the coater's  
181 pressure get back to the atmospheric pressure (**Lamichhane et al., 2015**). Dried pellets are  
182 transferred to the coater, which is subsequently placed under a vacuum. Then, oil is sprayed  
183 onto the extruded pellets and when the vacuum is released, the oil is forced into the pellet  
184 pores. Vacuum coating is an essential step to produce high-energy feed. It also enables the  
185 development of more nutritious feed containing functional additives (**Li et al., 2003**) like  
186 vitamins and exogenous enzymes that could not be added at the time of extrusion due to their  
187 thermolabile character (**Maas et al., 2020**). Thus, thanks to the extrusion and expansion  
188 process that allows the creation of enough pore spaces, it is now possible to infuse a high  
189 level of fat (*i.e.*, up to 40 %) during the vacuum coating step. This is especially required for  
190 salmon feed (**Bell and Koppe, 2010; Dethlefsen, 2017**).

191 The extrusion process allows *Fish Feed* producers to finely control the pellet density  
192 during production, the latter being easily adjusted to the eating habits of the farmed fishes  
193 (*i.e.*, sinking, semi-floating or floating pellets). Also, extrusion ensures greater feed stability  
194 in water with better production efficiency (**Clayton, 2002; Rokey, 2006**). In fish farming, in  
195 particular intensive aquaculture, the physical properties of feed are in top priority. A  
196 particularly important property is the ability of feed to be handled from production to the  
197 fishpond without creating an excessive amount of dust (**Sørensen, 2012**). Pellets need to  
198 maintain their integrity *via* high durability to minimize product loss during transport and  
199 pneumatic feeding. Thanks to its high versatility, the extrusion makes it possible.

200 The present work aims to develop an innovative twin-screw extrusion process to produce  
201 *Fish Feed* pellets for adult Trout. A twin-screw extruder is in fact capable of ensuring a wide  
202 range of functions like starch cooking, which is generally obtained partially in the  
203 preconditioner. In this context, the objective of this study is to highlight that the extruder



204 alone could allow for the future the realization of the whole *Fish Feed* process. This study  
205 mainly focuses on the comparison of extruded pellets obtained with and without a  
206 preconditioner. The first section concerns essentially the starch gelatinization phenomenon,  
207 which is of key importance in *Fish Feed* processes. In the second section, a comparison of the  
208 obtained pellets in terms of their usage properties (durability, hardness, floatability and oil  
209 leakage rate) has been conducted to understand specifically the role of the preconditioner and  
210 the challenges to be raised in its absence.

211

## 212 **2. Materials and Methods**

213

### 214 **2.1. Materials**

215 Various ingredients were used to produce the extruded pellets. Some of them were used for  
216 their protein intake, whether of animal (fish meal) or vegetable origin (*e.g.*, soy protein  
217 concentrate, etc.). Others were cereal's (*e.g.*, wheat, corn) products and by-products. Nutricia  
218 (France) provided all these ingredients.

219 Extruded pellets were coated using three commercial oils, that were provided as well by  
220 Nutricia: two fish oils (F1 and F2), both obtained by wet pressing (0.92 g/cm<sup>3</sup> at 20 °C), and a  
221 crude rapeseed oil (R) obtained by cold mechanical pressing (0.91 g/cm<sup>3</sup> at 20 °C). To avoid  
222 their oxidation, F1 and F2 were stored in a dry place away from light before their use during  
223 the coating step.

224 Extruded pellets were composed of 12.2% (w/w) corn gluten, 12.6% fish meal, 23.0%  
225 soybean meal, 3.7% soy protein concentrate, 1.3% wheat gluten, 17.2% shelled faba bean,  
226 3.0% wheat flour, 5.8% F1, 1.0% F2 and 20.2% R. Their chemical composition is  
227 summarized in **Table 1**. This composition was determined based on the chemical composition  
228 of each ingredient, and it has been developed to meet the nutritional requirements of adult  
229 Trout. In particular, the starch level was 11.1% (w/w) in proportion to the fresh matter (**Table**  
230 **1**), thus ensuring both expansion at the extruder die outlet, and cohesion for the final pellets.

231

### 232 **2.2. Feed recipe formulation**

233 To prepare the recipe, ingredients were finely ground by a Hosokawa Micron (Summit,  
234 New Jersey, USA) ZPS Zirkoplex industrial air classifier mill. After their reception, all the  
235 ingredients were weighed using an Ohaus (Parsippany-NJ, USA) CD-11 digital balance, and  
236 they were then introduced into an Electra (Poudenas, France) MH 400 horizontal mixer. This  
237 mixer is equipped with double hollow spirals with reverse pitch to improve the mixing. The  
238 mixing operation was carried out for 20 min by changing the direction of rotation every 5 min  
239 to obtain a homogeneous solid mixture. Six batches (each 120 kg) were prepared before  
240 extrusion, resulting in the production of six different formulations of extruded pellets named  
241 S1 to S6.

### 242 **2.3. Fish Feed production**

243 Extruded pellets were produced using a Cleextral (Firminy, France) Evolum HT 53 pilot-  
244 scale co-rotating and co-penetrating (*i.e.*, interpenetrating) twin-screw extruder. The S1 and  
245 S2 formulations were obtained according to the classical *Fish Feed* process presented in **Fig.**  
246 **2** whereas the S3 to S6 formulations were produced without the preconditioner.

247 The solid mixture was introduced at a 110 kg/h flow rate ( $Q_s$ ) in a Cleextral (Firminy,  
248 France) Evolum preconditioner with a capacity of 75 L for the pre-cooking step. This  
249 preconditioner was equipped with two counter-rotating shafts with variable speed and  
250 adjustable blades. At this level, liquid water ( $Q_{PREC}$  for the corresponding flow rate) and  
251 steam ( $Q_v$  for the corresponding flow rate) were added to partially cook and pre-humidify the  
252 initial dry solid mixture. To conduct this task, a Covemat (Trivier sur Moignans, France) GE  
253 490 steam generator and a Cleextral (Firminy, France) DKM Super KL CAMP piston pump  
254 were connected to the preconditioner. After a residence time of about 3-4 min, the pre-  
255 moistened and pre-heated mixture was fed into the Cleextral Evolum HT 53 twin-screw  
256 extruder. This machine has been designed to treat solid materials continuously at high  
257 pressure (from 1 to 275 bars) and high temperature (from 20 °C to 300 °C).

258 Extrusion parameters were the same for the six formulations produced. Only screw speed  
259 was changed: 425 rpm for the S1, S3 and S5 formulations, and 575 rpm for the S2, S4 and S6  
260 ones. The barrel was composed of nine modules with an internal diameter (D) equal to 53.15  
261 mm, each 4D in length. Screws had an outer diameter of 52.45 mm. The temperature profile  
262 was fixed as follows: 25 °C for module 1, 80 °C for modules 2 and 3, and 120 °C for modules  
263 4 to 9. The screw profile was kept the same, and it was especially composed of two  
264 successive pairs of reverse screw elements used here to apply an intense mixing and  
265 mechanical shearing to the solid mixture, thus resulting in a significant increase of its  
266 residence time. After its conveying from the module 1, the solid mixture was mixed with an  
267 additional amount of water ( $Q_{TSE}$  for the corresponding flow rate), which was injected at the  
268 end of module 3 using a Cleextral (Firminy, France) DKM Super KL-PP piston pump. Then,  
269 the reverse screw elements located at the end of module 7 were in fact the place where the  
270 solid mixture was compressed and plasticized, being converted into a dough-like state.  
271 Finally, the solid mixture was transported up to the die using conveying screws with  
272 decreasing pitch.

273 As the penultimate module was an open-top one, a Cleextral (Firminy, France) vent-stuffer  
274 (*i.e.*, a vertical stuffing screw) was positioned on its top to maintain the extruded mixture  
275 inside the barrel during the extrusion process. A die made of one conical insert having a 5.9  
276 mm outlet diameter was positioned at the end of the extruder barrel, and a Cleextral (Firminy,  
277 France) HC 45 granulator knife was installed immediately after the die. By regulating its  
278 rotation speed, the continuous cutting of the extruded pellets to the desired length (*i.e.*, around  
279 9.0 mm) was ensured.

280 For each formulation, the extrusion process was maintained for 10 to 15 min to reach a  
281 steady state before sampling the extruded pellets. Besides, samples were collected at the  
282 outlets of the preconditioner and the extruder to determine their moisture contents, and to  
283 carry out further characterizations. The process parameters (*i.e.*, the screw speed, motor  
284 current, barrel and material temperatures, and material pressure) were registered every 2 sec.  
285 Specific mechanical energy (SME) is directly related to the electrical power supplied by the  
286 motor (P), and it is expressed in W h/kg. In this case of the Cleextral Evolum HT 53 twin-  
287 screw extruder used in this study, the operating voltage of the motor (U) is equal to 454 V, its  
288 theoretical efficiency ( $\cos \varphi$ ) is 0.90, and the maximum screw speed ( $S_{\max}$ ) is equal to 800  
289 rpm. SME is therefore calculated according to Eq. (1).

290 
$$\text{SME} = \frac{P}{Q_S} \text{ with } P = U \times I \times \cos \varphi \times \frac{S_S}{S_{\max}} \quad (1)$$

291 With:

- 292 • P is the electrical power supplied by the motor (W).
- 293 • U is the operating voltage of the motor (V).
- 294 • I is the current consumed by the motor (A).
- 295 •  $\cos \varphi$  is the theoretical efficiency of the motor.
- 296 •  $S_S$  is the screw speed (rpm).
- 297 •  $S_{\max}$  is the maximum screw speed (rpm).
- 298 •  $Q_S$  is the inlet flow rate of the solid mixture (kg/h).

299 Extruded pellets were then collected and transferred to the dryer. This step was carried out  
300 using a continuous Cleextral (Firminy, France) Evolum 600 belt dryer. Drying was conducted  
301 at 120°C during 1800 sec for all the productions. The purpose of reducing moisture content  
302 was to make extruded pellets self-stable until the coating step. Pellets were then stored in a  
303 dry place (with final moisture content of around 6 % to 8 %).

304 Fat was exclusively added during the coating step, meaning that no oil was injected in the  
305 extruder at the moment of the extrusion cooking one. A Stolz (Paris, France) MRSV 100 pilot  
306 vacuum coater machine was used to conduct this task in order to raise the energy content of  
307 extruded pellets. An appropriate nozzle (APSYS 15/40) was chosen to guarantee an efficient  
308 spraying system. This configuration was optimal to spray the oily liquid uniformly. Firstly,  
309 the oil mixture composed of R, F1 and F2 oils (75/21/4 (w/w)) was prepared in a bucket and  
310 mixed intensely. The latter was then introduced into the oil tank and heated at  $40 \pm 2$  °C.  
311 During the oil mixture heating, the extruded pellets were loaded inside the vacuum coater  
312 tank. Simultaneously with the oil tank's pressurization up to 3 bars, a vacuum pump was used  
313 to generate vacuum in the inside of the mixing vessel. This vacuum pump was a Busch  
314 (Gerlingen, Germany) Mink MM 1104 BV 1.3 kW one. Then, a spray nozzle allowed the oily  
315 blend to be injected progressively. It was positioned in the top of the coater tank. Thanks to  
316 this spray, a proper dispersion was realized onto the feed while the two mixing blades were  
317 rotating. The proportion between extruded pellets and the oily blend was 73/27 (w/w), thus  
318 enabling to reach the targeted energy content of the coated pellets (*i.e.*, 30 % (w/w) fat  
319 content). The coater's pressure then got back to the atmospheric pressure once the oil was  
320 fully distributed. Lastly, the coater overturned, and the coated pellets could thus be recovered.  
321 The coating conditions used in the present work have been determined from a previous study  
322 (**Chaabani et al., 2020**). Identified as the optimal ones, they were as follows:

- 323 • 140 mbars for the vacuum coater's pressure at the moment of the oily blend addition.
- 324 • 65 Hz for the stirring speed.
- 325 • 45 % for the volumetric filling rate of the coater.
- 326 • 120 s for the time to restore the atmospheric pressure after the oily blend was added.

327 From each formulation, a sample of about 3 kg of coated pellets was collected after the  
328 coater's unloading, and these were then stored at 5 °C before their characterization. By taking  
329 such a precaution, the oil leakage has been avoided. NIR (Near-infrared) spectroscopy was  
330 used for all the experiments to ensure the 30 % (w/w) fat content in the coated pellets.

331 Considering that our objective was to understand specifically the role of the preconditioner  
332 in the *Fish Feed* process, extruded pellets were obtained as follows (**Table 2**):

- 333 • **Production of extruded pellets with preconditioner:** formulations S1 and S2, at 425  
334 rpm and 575 rpm, respectively.

335 • **Production of extruded pellets without preconditioner:** formulations S3 and S5 at  
336 425 rpm, and formulations S4 and S6 at 575 rpm. Additionally, two different amounts  
337 of water added in the twin-screw extruder were tested, *i.e.*, 26.5 kg/h for the S3 and S4  
338 formulations, and 30.0 kg/h for the S5 and S6 formulations, corresponding to water-to-  
339 solid ratios of 0.241 and 0.273, respectively. On the one hand, for the S3 and S4  
340 formulations, the 26.5 kg/h water flow rate corresponded to the sum of the  $Q_{PREC}$  and  
341  $Q_{TSE}$  water flow rates used for the S1 and S2 formulations. On the other hand, in the  
342 case of the S5 and S6 formulations, the additional amount of water added (*i.e.*, 3.5  
343 kg/h) corresponded to the amount of steam actually adsorbed by the solid mixture  
344 during the production of formulations S1 and S2.

345

## 346 **2.4. Fish Feed pellet's usage properties**

347

### 348 **2.4.1. Bulk density and expansion ratio (ER)**

349 Bulk density was determined by filling extruded pellets in a 1,000 mL cylinder and then  
350 determining the mass per unit volume of the sample. The pellets density was measured in  
351 triplicate and the result was expressed in g/L (*i.e.*, mass per volume) as mean values  $\pm$   
352 standard deviation. To standardize this measure, the cylinder was not tapped before weighing  
353 every sample (Aas et al., 2011).

354 The diameter of twenty uncoated extruded pellets was measured with an electronic digital  
355 sliding caliper. This diameter was used to calculate the expansion ratio according to Eq. (2).

$$356 \quad ER (\%) = \frac{\text{pellet diameter} - \text{die diameter}}{\text{die diameter}} \times 100 \quad (2)$$

357

### 358 **2.4.2. Durability**

359 Pellet durability was determined using a Doris tester (Durability on a Realistic Test) from  
360 the AKVA Group (Klepp, Norway). Initially, a sample mass of 350 g of coated pellets was  
361 sieved with 1.0 mm and 7.1 mm screens. After sieving, the material collected on each screen  
362 was weighed. Dust was the quantity of material collected under the finest screen. Small  
363 fractures from broken pellets were the quantity of material between the two tested screens.

364 The Doris test was then applied on the only preserved pellets. The objective of this test was  
365 to simulate the stresses to which pellets are exposed to in pneumatic feeding devices (Aas et  
366 al., 2011). Firstly, a known mass of preserved pellets was introduced on the Doris tester. The  
367 Doris device is made of an Archimede screw that feeds pellets from the inlet to the outlet of  
368 the tester. As the transportation can induce degradation by impact and shear, pellets were  
369 collected in a cup at the tester's outlet when the test was finished. Then, they were sieved with  
370 a 7.1 mm screen and the whole fraction of preserved pellets (collected on the upper screen)  
371 was weighed. The durability (expressed in % and possibly varying from 0% to 100%) was  
372 calculated as the ratio of the weight of preserved pellets to the initial weight of tested pellets.  
373 Extruded pellets of each formulation were analyzed in triplicate and results were expressed as  
374 mean values  $\pm$  standard deviations.

375

### 376 **2.4.3. Resistance to compression or hardness**

377 Pellet hardness (H) was measured using an Instron 33R4204 (Norwood-Massachusetts,  
378 United States) universal testing machine fitted with a 5 kN load cell. Two rigid plates were  
379 used during the test with the analyzed pellet positioned between them. The crosshead speed  
380 was 2 mm/min at the moment of the compression test, and the test result was the required  
381 force (F) to have the pellet broken. Hardness (H) was then expressed as the ratio of the  
382 maximum force (F) to the average diameter of the analyzed pellet (d). Proceeding in this way  
383 guarantees that dimensional variations of pellets were taken into account. The reported values  
384 are therefore the average of twenty replications and results were expressed as mean values  $\pm$   
385 standard deviations.

386

### 387 **2.4.4. Floatability**

388 The floatability of each formulation was tested using a large glass beaker (18.5 cm in  
389 diameter) filled with 4 L of fresh water at ambient temperature. Using a small plastic shovel,  
390 fifty pellets were counted and were then thrown softly into the water beaker. The number of  
391 floating pellets that remained suspended in the water after 30 s was determined. In  
392 aquaculture, *Fish Feeds* are intended to either sink or float (depending on the fish species).  
393 That is why depending on the obtained floatability, pellets are classified as follows: sinking  
394 pellets (floatability < 30 %), semi-floating pellets (between 30 % and 70 %), and finally  
395 floating pellets (over 70 %). For each condition, the measurements were carried out three

396 times and results were expressed as mean values  $\pm$  standard deviations. The water was  
397 changed at the end of each replication.

398

#### 399 **2.4.5. Oil leakage rate (OLR)**

400 Once coated, pellets tend to lose oil over time. The pellet's Oil Leakage Rate (OLR) was  
401 evaluated using a non-standardized method developed specifically for the purpose of this  
402 study. It was measured in two different conditions, *i.e.*, at 60 % relative humidity (RH) and 25  
403 °C on the one hand, and at 40 °C on the other hand. Conducted in a climatic chamber, the first  
404 incubation condition corresponded to pellets stored in a controlled manner whereas the second  
405 one had the objective to simulate the high temperature to which the pellets may be exposed  
406 when stored in silos in full sun during the summer season. The latter was conducted in a  
407 ventilated oven.

408 For both tested incubation conditions, the test sample mass of pellets was  $130.0 \pm 0.1$  g.  
409 These were then positioned in a glass beaker with a volume of 250 mL and at the bottom of  
410 which several layers of an absorbant paper, for a total mass of 2 g, have been placed. The  
411 incubation duration was chosen equal to 24 h. Specifically for the pellets incubated at 40 °C  
412 in a ventilated oven, the beaker has been left to cool to room temperature once the incubation  
413 period was over. Then, as part of the oil leaked during the test and has been absorbed by the  
414 paper, its weight was measured and this allowed determining the mass loss of the pellet in oil.  
415 Eq. (3) gives the formula used for the OLR calculation.

$$416 \quad \text{OLR (\%)} = \frac{\text{Weight of oil absorbed by the absorbent paper (g)}}{\text{Total weight of pellets before leakage (g)}} \times 100 \quad (3)$$

417 Experiments were carried out in triplicate. The results were expressed as mean values  $\pm$   
418 standard deviations.

419

#### 420 **2.5. Starch gelatinization (SG)**

421 To measure starch gelatinization rate, a Differential Scanning Calorimetry (DSC)  
422 apparatus was used. Firstly, test samples corresponding to the six uncoated pellets were  
423 obtained through a crushing step using a Nahita (La Chapelle sur Erdre, France) agate mortar  
424 with a 50 mm pestle. All the grinded powders were then conditioned in a climatic chamber  
425 (25 °C, 60 % RH) for three weeks to ensure an equivalent moisture content for all materials  
426 tested at the time of sampling. Lastly, samples were analyzed with a Mettler Toledo



427 (Columbus-Ohio, United States) DSC1 calorimeter under a constant flow of dry nitrogen. A  
428 hermetic steel sample pan with a volume of 100  $\mu\text{L}$  was used for the purpose of the DSC  
429 analysis. It contained the sample to be analyzed, and the test sample mass was chosen around  
430 10 mg. Then, the sample was completed with Milli-Q water (50  $\mu\text{L}$ ) and the analysis was  
431 realized after one night of incubation. After that, a heating ramp at 3  $^{\circ}\text{C}/\text{min}$  from 5  $^{\circ}\text{C}$  to 100  
432  $^{\circ}\text{C}$  was performed.

433 The starch gelatinization rate was also determined on the starting solid mixture (S0) and on  
434 the mixture collected at the output of the preconditioner (*i.e.*, after having added water and  
435 steam to the starting solid mixture, and before reaching the twin-screw extrusion machine).

436

## 437 **2.6. Morphological characterization of pellets through tomography**

438 To better understand differences between the obtained pellets (S1 to S6), their morphology  
439 (internal structure (porosity) and oil distribution) were determined by tomography. The  
440 tomography device used to perform this analysis was a RX Solutions Easy Tom (Chavanod,  
441 France) 3D X-ray laboratory tomograph. For the analysis, a tension of 114 kV and a current  
442 of 263  $\mu\text{A}$  were chosen. For each formulation, three pellets were analyzed. A rotating stage  
443 was used to support them. Approximately 1,440 projections of the transmitted X-ray intensity  
444 field were recorded for each angular step of  $0.25^{\circ}$ . This was made possible by the use of an X-  
445 ray detector and a flat panel detector ( $1,920 \times 1,536$  pixels). Then, a volume image of the  
446 analyzed sample could be reconstructed from all the radiographs using a filtered back-  
447 projection algorithm. This image allowed to observe, with a spatial resolution of 20  $\mu\text{m}$ , the  
448 variations of the linear attenuation coefficient inside the sample.

449 Three different zones were distinguished in this reconstruction: the starchy matrix (in light  
450 grey), the pores filled by the oil (in dark grey), and the empty pores (in black). The AVISO  
451 software enabled the determination of the volume occupied by these three zones inside the  
452 coated pellets ( $V_{\text{SM}}$ ,  $V_{\text{PO}}$  and  $V_{\text{EP}}$ , respectively), and the distribution in volume ( $\%$  (v/v) in  
453 proportion to the pellet's total volume) could then be calculated. For each formulation, this  
454 quantitative analysis was conducted three times, and results were then expressed as mean  
455 values  $\pm$  standard deviations.

456 From this distribution in volume, the pellet's porosity after extrusion-cooking and drying  
457 (*i.e.*, immediately before vacuum coating) was calculated according to Eq. (4).

458 
$$\text{Pellet's porosity (\%)} = \frac{(V_{PO} + V_{EP})}{(V_{SM} + V_{PO} + V_{EP})} \times 100 \quad (4)$$

459

## 460 **2.7. Water absorption and Water Solubility indices of extruded pellets**

461 The uncoated extruded pellets were ground in a fine powder using a Foss (Hillerød,  
462 Denmark) Cyclotec 1093 grinder fitted with a 1 mm screen. About 2.5 g sample was  
463 suspended in 30 mL of distilled water in a 50 mL centrifuge tube. The suspension was stirred  
464 intermittently, placed in an oven at 30°C for 30 min and then centrifuged at 1000 × g for 10  
465 min.

466 The supernatant liquid was decanted into an aluminium cup and then oven-dried for 2 h at  
467 135 °C (AACC method 44-19, 1995). The weight of the gel remaining in the centrifuge tube  
468 was measured.

469 Water absorption (WAI, %) and water solubility (WSI, %) indices were subsequently  
470 calculated as follows:

471

472 
$$\text{WAI} = \frac{W_g}{W_{ds}} \times 100 \quad (5)$$

473 Where:

474  $W_g$  is the weight of the gel (g).

475  $W_{ds}$  is the weight of the dry sample (g).

476

477 
$$\text{WSI} = \frac{W_s}{W_{ds}} \times 100 \quad (6)$$

478 Where:

479  $W_s$  is the weight of the dry matter in the supernatant (g).

480

## 481 **2.8. Statistical analyses**

482 Presented as mean values ± standard deviations, all data originated from triplicates, except  
483 hardness measurements that were performed on twenty pellets. The XLSTAT (Addinsoft,  
484 Bordeaux, France) software followed by a one-way analysis of variance (ANOVA) were used  
485 for analyzing the results in a statistical point of view so as to evaluate whether the difference  
486 between the pellet's physical properties is significant or not.  $P < 0.10$  was chosen for each  
487 variable to assess the significance of the differences. Tukey's test was used in the multiple  
488 comparison procedure.

489

### 490 **3. Results and discussion**

491

#### 492 **3.1. Thermal analysis of the extruded pellets (evaluation of starch** 493 **gelatinization)**

494 The DSC analysis was used to estimate the starch gelatinization rate inside the solid  
495 mixture at the preconditioner outlet just as inside the extruded pellets before coating.  
496 Although it is not the only analytical method known for measuring the gelatinization rate of  
497 starch, the DSC analysis is suitable when the fat content of the analyzed samples is low.  
498 Otherwise, interactions between starch and fat make it difficult to observe the gelatinization  
499 phenomenon. In the present study, as fish oils (F1 and F2) and rapeseed oil (R) were added  
500 exclusively during the coating step, the uncoated pellets only contained the oily molecules  
501 coming from the initial solid mixture at the moment of their DSC analysis. This content was  
502 only 4.2% of the fresh matter, which made DSC analysis appropriate in this case.

503 The results obtained from the extruded pellets processed at 575 rpm (formulations S2, S4  
504 and S6) are presented in **Fig. 3**, and particular attention has been focused on the temperature  
505 zone between 55°C and 80°C, where gelatinization of starch occurs. No thermal phenomenon  
506 was observed for these three formulations, and the same was true for the three others. This  
507 indicates that the starch gelatinization was complete during the extrusion process even in the  
508 case of formulations S3 to S6 for which no preconditioner was used. On the contrary, when  
509 conducted on the starting solid mixture (S0) or the preconditioned one (S<sub>2PREC</sub>), DSC  
510 thermograms revealed a specific endothermic peak from 60 °C to 80 °C, which was  
511 characteristic of the starch gelatinization and thus evidencing that gelatinization was not  
512 complete inside the preconditioned mixture (**Fig. 3**). **Table 3** gathers the starch gelatinization  
513 enthalpy ( $\Delta H$ ) for all the tested samples, and these enthalpy values were used to calculate the  
514 gelatinization rate (GR) of all processed mixtures (*i.e.*, the preconditioned ones and the  
515 extruded pellets) according to Eq. (7).  $\Delta H$  (expressed in J/g) was determined by calculating  
516 the area under the DSC normalized peak.

$$517 \quad \text{GR (\%)} = \frac{\Delta H (\text{initial mixture}) - \Delta H (\text{processed mixture})}{\Delta H (\text{initial mixture})} \times 100 \quad (7)$$

518 A gelatinization rate of about 60 % was observed for the two preconditioned mixtures at  
519 the origin of the S1 and S2 formulations. This result is in perfect agreement with data in the  
520 literature reported by **Lavocat and Raymond (2018)**, which confirms the preconditioner role

521 in aquafeed manufacturing. Raw materials (starch, protein, etc.) are hydrated and heated  
522 firstly in the preconditioner. A pre-cooking step is realized in the presence of water (in its  
523 steam and liquid forms), contributing to the loss of starch crystallinity and to the beginning of  
524 its gelatinization. Then, the pre-heated and pre-humidified mixture passes to the extruder,  
525 where the thermo-mechanical treatment is achieved by applying a temperature constraint  
526 along the barrel simultaneously with intensive shear stress resulting from the restrictive  
527 reverse elements positioned along the screw profile. In the literature, it was previously  
528 mentioned by **Riaz and Rokey (2011)** that the combination of generated heat, pressure and  
529 moisture inside the twin-screw extruder achieves cooking, starch gelatinization, and protein  
530 denaturation. **Hansen et al. (2010)** and **Kraugerud et al. (2011)** had reported that starch  
531 gelatinization varied between 73 % and 100 % in extruded *Fish Feed* and, this finding is in  
532 perfect accordance with the present DSC results that have revealed a complete gelatinization  
533 of starch for formulations S1 and S2.

534 Even without a preconditioning step, the extruder was still able to fully gelatinize starch,  
535 both with less (S3 and S4 formulations) or more (S5 and S6 formulations) water injected into  
536 the barrel. For aquafeed, starch acts as a natural binder contributing to the pellet's cohesion, a  
537 source of energy and a density control agent (**Bortone, 2017**). To make this last capacity  
538 effective, the starchy fraction must be well cooked and gelatinized during the extrusion  
539 process. At this level, all the extruded pellets produced in this study fulfill this objective.  
540 Nevertheless, their usage properties must be determined and discussed in a comprehensive  
541 manner before definitively validating that the production of *Fish Feed* pellets can be  
542 satisfactory ensured without using a preconditioner.

543

### 544 **3.2. Effect of the process parameters on pellet's qualities**

545 As described previously, S1 and S2 formulations were obtained *via* the traditional *Fish*  
546 *Feed* process including a preconditioning step before extrusion (**Fig. 2**). The related extrusion  
547 parameters, and pellet's characterizations and usage properties are summarized in **Table 4**.  
548 The moisture content is of critical importance to authorize efficient starch gelatinization and  
549 protein denaturation and, consequently, to maintain the nutritional quality during extrusion-  
550 cooking. It was measured at different levels during the *Fish Feed* process, *i.e.*, in the starting  
551 mixture, at the preconditioner outlet when used, at the extruder outlet and finally at the dryer  
552 outlet. The initial solid mixture had a moisture content equal to  $7.4 \pm 0.1$  %. The latter stayed

553 in the preconditioner for 3 to 4 min where it was pre-humidified *via* water and steam addition.  
554 This contributed to an increase in its moisture content to 19 % on average. In parallel, the  
555 particularly low associated standard deviation (0.2 % max) also indicates that water was  
556 uniformly distributed inside the solid mixture. The pre-humidified and pre-heated mixture left  
557 the preconditioner with an average temperature of  $75 \pm 2$  °C. Therefore, moisture and  
558 temperature data confirm the preconditioner role, which indeed consists in pre-heating and  
559 pre-humidifying the solid mixture, to ensure the starch pre-gelatinization.

560 At the extruder level, the screw speed was the only variable operating parameter, differing  
561 between the S1 and S2 formulations (425 rpm and 575 rpm, respectively). The moisture  
562 content was also determined at the extruder outlet, and it was equal to 25 % on average, due  
563 to the additional water injected into the extruder. In fact, a moisture content between 25 %  
564 and 30 % during wet extrusion of diets involving a mixture of cereal products, and vegetable  
565 and animal proteins has been recommended for aquafeed (**Rokey, 1994**). In such humidifying  
566 conditions, the formulation is better processed to obtain feed with high physical quality.  
567 Complete and uniform moisture penetration in the ingredient particles results in an increased  
568 heat transfer, allowing uniform gelatinization (**Rokey, 1994**).

569 During the extrusion process, the electrical energy is converted to mechanical energy  
570 through the screws, and it is then transferred to the solid mixture. Representing the amount of  
571 mechanical energy input per mass unit, SME values thus appear as a good way to characterize  
572 the efficiency of the extrusion process. In this study, increasing the screw speed from 425 rpm  
573 to 575 rpm (whatever the formulation) enhanced shear stress, increasing the energy input. At  
574 a constant temperature, SME is known to increase with the screw speed (**Baik et al., 2004**).  
575 An increased speed elevates the friction between the product and the screws: more mechanical  
576 energy is thus produced. Simultaneously, increasing the screw speed slightly promoted  
577 pellet's expansion observed through a decrease in its bulk density, confirming that it is an  
578 influential variable in twin-screw extrusion (**Rokey, 1994**). It should however be noted here  
579 that despite the increase in both pellet's diameter and expansion ratio from formulation S1 to  
580 formulation S2, the values obtained were not different in a statistical point of view (**Table 4**).  
581 Concerning the counter-pressure, which is also an important parameter that must be  
582 controlled during the extrusion process, and according to the values reported in **Table 4**, it  
583 decreased as the screw speed increased. The counter-pressure was in fact directly linked to the  
584 device's filling coefficient: the more the filling of the extruder, the more the counter-pressure.

585 With a reduced screw speed, the twin-screw machine was more filled and the counter-  
586 pressure logically increased.

587 Differences were found between the S1 and S2 formulations concerning their usage  
588 properties. Although slightly lower, the expansion level generated in S1 was a little more  
589 favorable to better retain oil in these pellets as confirmed by the lower OLR values obtained at  
590 25 °C and 40 °C as compared to S2 (**Table 4**). On the other hand, as S2 had a greater  
591 expansion ratio (55 % instead of 51 %), its durability and hardness were slightly reduced. In  
592 aquafeed, the expansion is one of the most important factors as it affects the density, hardness,  
593 brittleness and oil holding capacity (**Rosentrater et al., 2009**). A large number of studies had  
594 reported that an inverse relationship existed between the pellet's expansion and physical  
595 quality parameters like durability and hardness (**Aarseth et al., 2006a; Hansen and**  
596 **Storebakken, 2007; Morken et al., 2012; Sørensen et al., 2009, 2010, 2011**).

597 *Fish Feed* pellets must adapt to the species, and they are intended to either sink or float  
598 (**Dethlefsen, 2017**). In our case, S1 gave semi-floating pellets while S2 gave fully floating  
599 ones. In fact, floatability was directly linked to the pellet's density after vacuum coating: the  
600 lower the density, the higher the floatability. This is the reason why the bulk density of coated  
601 pellets is also of key importance with regard to their floatability (**Chevanan et al., 2007,**  
602 **2009**). Bulk density is directly linked to the expansion degree of pellets generated during the  
603 extrusion process (**Glencross et al., 2011**). In aquaculture, bulk density is not only adjusted  
604 according to the feeding habits of the targeted fish species but also to fish farmers feeding  
605 practices (**Sørensen, 2012**).

606 The quality of pellets was also investigated for the S3 to S6 formulations obtained without  
607 the preconditioner. All the tested conditions allowed to obtain extrudable, pelletable and  
608 coatable *Fish Feed* (up to 30% (w/w) fat). According to the results presented in **Table 4**, S3  
609 and S4 were the formulations with the lowest moisture contents at the extruder outlet (19.8%  
610 in average) of the entire study, and this had led to the pellets with the poorest usage  
611 properties. Firstly, both S3 and S4 pellets had a rough surface. This was the consequence of  
612 their low moisture contents, causing more difficulty for the extruded material to pass through  
613 the die orifice. In such conditions, and as water is known to act as a plasticizer for starch and  
614 proteins, the reduced moisture content increased the material viscosity, rendering less  
615 favorable its flowing. In addition, bonds (*i.e.*, hydrogen ones and/or van der Waals forces)  
616 may also appear between particles, due to moisture acting as a binder (**Pietsch, 2002**). This

617 property was, therefore, less favored in the case of the S3 and S4 formulations given their low  
618 moisture content at the extruder outlet. This resulted also in higher SME values (**Table 4**). It  
619 should be remembered here that water is the most important and widely used plasticizer in  
620 extrusion processing (**Alvarez-Martinez et al., 1988; Blanche and Sun, 2004**). S3 and S4  
621 formulations had so higher bulk densities and reduced expansion ratios of only 32% and 36%,  
622 respectively. In fact, the expansion is highly affected by the flour moisture (**Draganovic et**  
623 **al., 2011**) and the driving force in the pellet's expansion is the vaporization of water (**Cheng**  
624 **and Friis, 2010**). Thus, it is reasonable to deduce that a high content of moisture boosts the  
625 expansion potential. As the expansion affects the oil holding capacity (**Rosentrater et al.,**  
626 **2009**), higher values of OLR at 25 °C and 40 °C were logically obtained for the less expanded  
627 pellets of the S3 and S4 formulations. Broken pellets and dust (**Table 4**) were also more  
628 important with S3 and S4. Low moisture content during extrusion was responsible of poorer  
629 cooking quality, therefore resulting in more fragile pellets (*i.e.*, with little cohesion and  
630 binding strength). These results are in perfect agreement with those reported by **Rokey**  
631 **(1994)**.

632 On the other hand, higher durability was obtained with S3 in comparison to S1. With less  
633 water and a low screw speed, S3 had a high density reflecting a less porous structure. It is  
634 reasonable to assume that this configuration explains the high durability of the S3 pellets. On  
635 the contrary, pellets from S4 had lower durability than those from S2 despite their lower  
636 moisture content, and this was probably the consequence of the higher screw speed chosen  
637 (575 rpm instead of 425 rpm). S3 and S4 had also lower hardness values in comparison with  
638 S1 and S2. The pellets obtained in that case were less homogeneous with non-uniform  
639 cooking, due to less water during the extrusion process, probably leading to more fragile  
640 pellets from a mechanical point of view. **Pietsch (1983)** had reported that water in pellets  
641 bound particles together by a “liquid bridge”. This deficit in water during the extrusion of  
642 pellets from the S3 and S4 formulations could be responsible for their brittleness as the  
643 particles inside the mixture were less bound together during the extrusion process. Finally, S3  
644 and S4 pellets were both sinking ones. Even increasing the screw speed (case of the S4  
645 formulation) was not sufficient to have at least semi-floating pellets.

646 To conclude, S3 and S4 pellets did not meet the specifications of adult Trout feed. Even by  
647 increasing the screw speed (S4 formulation), such setting was not enough to obtain a floating  
648 pellet (floatability of 25% for a recommendation of at least 70 %). This difficulty to obtain

649 pellets with the targeted properties can be explained by the fact that S3 and S4 were obtained  
650 in non-optimal moisture level conditions as found in the literature (25-30 %).

651 In addition to these poor usage properties, pellets from S3 and S4 could present other  
652 disadvantages due to their low moisture content. For example, **Rokey (1994)** had reported  
653 that the palatability was reduced when lower moisture levels were applied. This condition can  
654 even contribute to the destruction of heat-labile nutrients like lysine and ascorbic acid. The  
655 same author recommended a moisture content of 250-300 g/kg during the extrusion of fish  
656 diets to prevent losses of nutrients. In our case, this moisture content was only 241 g/kg for  
657 both S3 and S4 formulations (**Table 2**). **Finley (1989)** and **Opstvedt et al. (1984)** have also  
658 found that when low moisture content occurred in combination with temperature higher than  
659 100 °C, the cross-linking between amino acids could occur inside the fish proteins.  
660 Consequently, digestibility was reduced for almost all amino acids, especially cysteine  
661 (**Andorsdottir, 1985; Ljøkjel et al., 2000**). The same phenomenon was observed with  
662 rainbow Trout when water added to the extruder was restricted in comparison with diets  
663 obtained at more elevated moisture contents (**Sørensen et al., 2002**). In other terms, in the  
664 case of the S3 and S4 formulations and apart from the fact that they were obtained without  
665 preconditioner, their processing with low moisture content could affect negatively their  
666 digestibility.

667 Since S3 and S4 resulted in pellets with unsatisfactory properties, corrective measures  
668 were applied by injecting more water inside the extruder barrel (30.0 kg/h instead of 26.5  
669 kg/h) when extruding the S5 and S6 formulations without using the preconditioner. In fact,  
670 this correction was made by taking into account the water that was absorbed by the solid  
671 mixture in the preconditioner in the case of formulations S1 and S2. This adjustment was  
672 evidenced by higher moisture content values at the extruder outlet for S5 and S6 in  
673 comparison with S3 and S4: 24 % instead of 20 % (**Table 4**). The most important finding is  
674 that adding more water had significantly improved the pellet's usage properties, which  
675 became then similar to S1 and S2, especially with the highest screw speed (575 rpm) (**Table**  
676 **4**), confirmed by the statistical analysis. Adding more water was necessary for the viscoelastic  
677 properties of the extruded mixture and to guarantee complete hydration. S5 and S6 densities  
678 (both at the extruder outlet and at the coater one) were reduced and became much closer to S1  
679 and S2 ones. In this case, water injected in the extruder barrel can be used to control the  
680 extruded pellet's density (**Bortone, 2017**). Consequently, more water helped to generate more  
681 expanded pellets with a larger diameter that met the expansion level of S1 and S2 (with no



682 statistical difference). This improvement contributed also to better oil retention, which  
683 resulted in lower OLR values at 25 °C, compared to S3 and S4, in the range to those measured  
684 for S1 and S2.

685 At 40 °C, no statistical difference was observed for OLR of pellets extruded at 575 rpm  
686 (comparison between S2 and S6). In fact, the combined action of an additional amount of  
687 water and an increase in the screw speed allowed a further improvement on the pellet's usage  
688 properties, explaining why the results for S6 were so close to those for S2. Additionally,  
689 floating pellets were obtained with S6 (86 %). Despite a slight floatability reduction in  
690 comparison with S2, S6 still met the feed specifications for large Trout (*i.e.*, floating pellet  
691 type). Dust generation was also not statistically different between S2 and S6, and particularly  
692 limited. These results demonstrate the beneficial effect of adequate moisture content during  
693 extrusion to generate desirable usage properties even without the preconditioning step.

694 Overall, durability and hardness were improved with S5 and S6 compared to S1 and S2.  
695 Such improvement in the pellet's usage properties is often desired in aquaculture. In this  
696 situation, the ability of the feed to be handled while maintaining its integrity (fewer dust  
697 amount) is guaranteed (**Sørensen et al., 2012**). In intensive aquaculture (*e.g.*, big and modern  
698 Trout farming), feeds should be resistant to a multitude of constraints such as handling,  
699 mechanical stress during transport and pneumatic feeding devices (**Aarseth, 2004; Aarseth et**  
700 **al., 2006b**). That is why the durability measurements made at lab scale (use of a Doris tester)  
701 tend to mimic the stress applied to the pellets in real conditions. On the other hand, hardness  
702 assesses the resistance to breakage when pellets are exposed to external pressure. It is often  
703 used to mimic the force applied on pellets when they are stacked on top of each other during  
704 storage in silos or bins, the mechanical solicitation in a screw conveyor, and the compressing  
705 force between animal teeth (**Kaliyan and Morey, 2009**).

706 In the specific case of diets for salmonids, starch is kept at a lower concentration than  
707 proteins, due to their low capacity to digest and metabolize starch (**Hemre et al., 2002**). As  
708 native proteins generally have a stronger water affinity than starch (**Semenova et al., 2006;**  
709 **Yahata et al., 2006**), it is reasonable to assume that water will be more easily absorbed by the  
710 proteins inside the extruder when no preconditioner is used. Additionally, and for the same  
711 reason, adding more water to the mixture in the extruder (S5 and S6 formulations in  
712 comparison with the S3 and S4 ones) must undoubtedly have contributed to an increased  
713 absorption of water by the proteins and thus their improved denaturation, which must be the  
714 reason why the pellet's durability and hardness were improved for the S5 and S6

715 formulations. These results underline the twin-screw extruder ability to ensure the entire *Fish*  
716 *Feed* process, *i.e.*, both the starch gelatinization as previously evidenced by the DSC results  
717 (**Table 3** and **Fig. 3**) and the protein denaturation.

718 Besides, it is well known that high water content during extrusion contributes to higher  
719 nutrient availability (**Sørensen, 2012**). For example, when shrimp were fed by extruded diets  
720 at high moisture content rather than from dry extrusion conditions, the results showed an  
721 improvement in growth performance that emphasizes the importance of moisture during  
722 processing (**Obaldo et al., 2000**). Given these results, it appears therefore necessary to  
723 complete the present study by *in vivo* feeding trials on adult Trout in order to check if those  
724 findings will still be valid in the absence of preconditioner. As a more elevated water content  
725 during extrusion was used for S5 and S6, these two formulations should be the most  
726 interesting ones in terms of palatability and nutrient digestibility, especially since the pellets  
727 from these two formulations have a common appearance once coated. Indeed, **Fig. 4** shows  
728 the extruded pellets obtained at a screw speed of 575 rpm before and after coating. No visual  
729 difference was observed between S2 and S6. On the contrary, the S4 pellets were  
730 distinguishable from the two others as they revealed a less uniform appearance and a rougher  
731 surface.

732 To conclude, a complete starch gelatinization can occur along the twin-screw extruder  
733 barrel alone without any preconditioning treatment. In addition, the same level of expansion  
734 can be reached at the die outlet but on the express condition that the liquid water injected in  
735 the extruder barrel is the sum of both liquid water flow rates (*i.e.*, the one added in the  
736 preconditioner and the second in the twin-screw extruder) and the part of injected steam really  
737 absorbed by the flours along the preconditioner when the latter is implemented. For future  
738 work, it will be however necessary to ensure that not using a preconditioner will not lead to a  
739 premature wear of both the screw elements and the barrel in the twin-screw machine.

740

### 741 **3.3. Morphological characterization of pellets through tomography**

742 Results obtained for the pellet's expansion ratios, and their density, OLR values and  
743 floatability were completed by their morphological characterization through 3D X-ray  
744 tomography. **Fig. 5** presents one example of tomography image taken from all coated pellets  
745 analyzed (S1 to S6). Thanks to tomographic reconstructions of the extruded pellets, it was  
746 possible to carry out a quantitative analysis that enabled to determine the distribution in

747 volume relative to the three different zones inside the coated pellets (*i.e.*, the starchy matrix,  
748 the oil-filled pores and the empty ones).

749 **Table 5** shows significant differences in the volume distribution between these three  
750 different zones when comparing the pellets produced with preconditioner (S1 and S2) and  
751 those without (S3 and S4). In particular, the S3 and S4 formulations produced without  
752 preconditioner and with a reduced amount of water revealed a reduced proportion of oil-filled  
753 and empty pores. On the contrary, when more water was added in the extruder (S5 and S6),  
754 these differences were largely reduced and the volumes occupied by the oil-filled and empty  
755 pores were not statistically different from those inside the S1 and S2 pellets. These  
756 tomography results are very relevant since the same trend was observed previously in terms of  
757 usage properties. With the lowest moisture content (S3 and S4), results in **Table 4** showed  
758 that expansion was unfavored, resulting in higher OLR values (especially at 25 °C). The  
759 obtained pellets were therefore less porous (higher density), logically explaining at the same  
760 time the much higher values for  $V_{SM}$ . However, even without a preconditioner but with the  
761 express condition of adding higher proportion of water in the extruder barrel, the obtained  
762 pellets (S5 and S6) presented an internal structure similar to those obtained with the  
763 preconditioned mixtures. It is thus reasonable to assume that the expansion phenomenon  
764 generated at the die outlet was equivalent between the S1 and S2 formulations on the one  
765 hand, and the S5 and S6 ones on the other hand.

766 **Pastor-Cavada et al. (2011)** had reported that bulk density and expansion ratio were the  
767 best properties to describe product porosity. In this context and to verify the previous  
768 hypothesis, the sum of  $V_{PO}$  and  $V_{EP}$  was used to determine the pellet's porosity (**Table 4**). The  
769 results revealed no statistical difference between S1, S2, S5 and S6 pellets. These findings are  
770 in perfect agreement with the previously discussed results related to the expansion ratio. On  
771 the other hand, S3 and S4 had the lowest pellet's porosity, reflecting their lower expansion  
772 ratio, which was the consequence of the reduced water amount injected inside the extruder  
773 barrel. These results highlight the clear correlation existing between the pellet's expansion  
774 ratio and their porosity.

775 To conclude, the expansion ratio generated during extrusion strongly influences the usage  
776 properties and final appearance of the product (**Moraru and Kokini, 2003**). In the literature,  
777 the screw speed, the shape and the diameter of the die are known to influence expansion. In  
778 this study, even without a preconditioning step, it has been shown that expansion could be

779 also adjusted thanks to the judicious choice of the quantity of added water. Without  
780 preconditioner, an adequate optimization of the extrusion-cooking conditions can thus  
781 perfectly lead to pellets as efficient as those obtained through a classical *Fish Feed* process.  
782 The key element is the expansion phenomenon that dictates the usage properties (*i.e.*, density,  
783 OLR and floatability) of the final extruded pellets.

784

### 785 **3.4. WAI and WSI values**

786 In several studies, WAI and WSI indices are often used to provide further information  
787 about the cooking rate of starch. This method is particularly justified at a practical level, as it  
788 does not require significant time, biochemical extraction, and isolation and characterization of  
789 a complex mixture (**Kim et al., 1989; Kirby et al., 1988**). The values of WAI and WSI  
790 measured on the preconditioned mixture and extruded pellets are summarized in **Table 6**.

791 **Kirby et al. (1988)** indicated that a degradation of molecules could be illustrated through  
792 WSI values. As starch degrades, the quantity of soluble matters increases, and this results in  
793 modifications in the WSI value (**Guha et al., 1997**). According to **Table 6**, no statistical  
794 difference in terms of WSI values was evidenced for the six extruded formulations and even  
795 for the preconditioned mixture. Alternatively, **Seker (2005)** and **Van den Einde et al. (2003)**  
796 have reported that high WSI values are the indication of effective starch gelatinization. In our  
797 case, this information means that starch gelatinization was the same for S1 to S6 formulations.  
798 This finding is consistent with the previous DSC results. And, although the differences were  
799 not significant in a statistical point of view, the lowest WSI value was obtained with the  
800 preconditioned mixture for which the gelatinization rate was estimated through DSC analysis  
801 to only 60% (**Table 3**). This result can be explained as the latter had only undergone  
802 preconditioning while pellets have been extruded, with much higher mechanical shear  
803 applied.

804 In an aqueous dispersion, **Mason and Hosney (1986)** indicated that it is possible to  
805 estimate the volume of gelled and swollen particles that retain their integrity *via* the WAI  
806 value. According to **Table 6**, no statistical difference was found between the S1 and S2  
807 pellets (with preconditioner), and the S5 and S6 ones (without preconditioner and with a  
808 higher amount of water added). **Bortone (2004)** and **Gomez and Aguilera (1984)** had  
809 reported that WAI is related to starch gelatinization extent followed by degradation and  
810 dextrinization. These phenomena are responsible for water absorption. On the other hand,

811 WAI values relative to pellets S3 and S4 were statistically different in comparison with those  
812 of S5 and S6. When the moisture content in pellets was reduced (S3 and S4), starch swelling  
813 was therefore limited in comparison with formulations also obtained without preconditioner  
814 but with more added water during the process (S5 and S6). This result is consistent with  
815 previous studies conducted by **Anderson et al. (1970)** and **Oikonomou and Krokida (2012)**,  
816 where WAI had decreased when the moisture content was reduced, but reported as well that  
817 WAI decreased with an increase in screw speed (no correlation established in this work).

818 **Ravindran et al. (2011)** have reported that WSI reflects the pellet's hydrophobicity while  
819 WAI indicates their hydrophilic capacity. These two parameters are useful as they can  
820 therefore influence the pellet's usage properties. In the present study, WAI and WSI values  
821 relative to S1 and S2 were not statistically different from those of S5 and S6. This finding was  
822 confirmed by the results presented in **Table 4** where most of the pellet's characteristics were  
823 kept without preconditioner with the precaution to work with adequate moisture content in the  
824 extruder.

825 The starch contained in pellets consists of two chains of glucose molecules, namely  
826 amylose and amylopectin. The first one is a linear polymer with a few branches while the  
827 second one is a highly branched polymer (**Tharanathan, 1995**). The extrusion process of  
828 pellets causes substantial changes in the physical constitution of the starchy fraction  
829 (**Davidson et al., 1984**). Those changes confer specific functional properties to the *Fish Feed*  
830 pellets. Among the most important practical properties of starch, its ability to swell,  
831 contributes to viscous paste, either in hot water or when treated with chemicals (**Knight,**  
832 **1969**). It is also important to specify that other components like proteins and fats can also  
833 influence the starch conversion during extrusion (**Lin et al., 1997**). To conclude this part, it  
834 should be kept in mind that results concerning WAI and WSI are not based on fundamental  
835 structural characteristics of the material. This is the reason why discussing them has been  
836 done cautiously in the present study.

837 In aquaculture, *Fish Feed* pellets are very often produced using a preconditioner that  
838 precooks the feed (pre-gelatinization of starch). Not using it requires consequently subtle  
839 changes, specifically at the extruder level. The present work demonstrated that an  
840 optimization was necessary to achieve the desired product attributes while maximizing its  
841 usage properties. In our case, as pellets are designed to feed large Trout, it was necessary to  
842 manufacture them at high moisture content and high screw speed in the process without

843 preconditioning. The adjustment of other extrusion parameters (e.g., screw profile,  
844 temperature profile, etc.) would also possibly enable to reach the pellet's targeted properties.  
845 Further research will be required to determine the importance of each of them.

#### 846 **4. Conclusion**

847 In this work, *Fish Feed* pellets for adult Trout were successfully manufactured at pilot  
848 scale through extrusion cooking without a preconditioner. The gelatinization of starch can be  
849 entirely conducted inside the twin-screw machine, thus reducing the investment cost of the  
850 industrial lines. The simplification of the process results as well in pellets with the same usage  
851 properties as those originating from a classical *Fish Feed* process including both a  
852 preconditioner and a twin-screw extruder. A final check of screw elements and extruder barrel  
853 wear would however be necessary to ensure the industrial gain of this simplified process over  
854 the long term. Indeed, even if the specific mechanical energy required was almost the same  
855 for both processes, the counter-pressure was significantly higher without the preconditioner.  
856 For future work, as the process without preconditioner has been validated in terms of pellet's  
857 usage properties, it will be necessary to carry out *in vivo* feeding trials on adult Trout to  
858 evaluate the pellet's palatability and nutrient digestibility, as these two characteristics are the  
859 most reliable barometers of product quality.

860

#### 861 **CRedit authorship contribution statement**

862 Asma Chaabani: Investigation, Writing – original draft, Writing – review &  
863 editing, Visualization. Laurent Labonne: Investigation. Vanessa Durrieu:  
864 Formal analysis, Writing – original draft, Writing – review & editing.  
865 Antoine Rouilly: Writing – original draft, Writing – review & editing.  
866 Fabien Skiba: Resources, Supervision, Project administration. Philippe  
867 Evon: Conceptualization, Methodology, Validation, Formal analysis,  
868 Investigation, Writing – original draft, Writing – review & editing,  
869 Visualization, Supervision, Project administration.

870

#### 871 **Declaration of Competing Interest**

872 The authors report no declarations of interest.

873

874 **Acknowledgements**

875 This research was granted by ANRT (National Association for Research and  
876 Technology in France). We would like to thank also Dr. Marina Fazzini  
877 (Laboratoire Génie de Production, ENI, Tarbes, France) for conducting the  
878 morphological characterization of pellets through tomography.

879

## References

- 880  
881  
882 AACC. 44-19. 1995. Moisture-air oven method, drying at 135 °C. AACC Approved methods  
883 (9th ed.). St. Paul, MN, USA.
- 884 Aarseth, K.A., 2004. Attrition of feed pellets during pneumatic conveying: the influence of  
885 velocity and bend radius. *Biosyst. Eng.* 89, 197-213.  
886 <https://doi.org/10.1016/j.biosystemseng.2004.06.008>
- 887 Aarseth, K.A., Sørensen, M., Storebakken, T., 2006a. Effects of red yeast inclusions in diets  
888 for salmonids and extrusion temperature on pellet tensile strength: Weibull analysis. *Anim.*  
889 *Feed Sci. Tech.* 126, 75-91. . <https://doi.org/10.1016/j.anifeedsci.2005.06.005>
- 890 Aarseth, K.A., Perez, V., Bøe, J.K. Jeksrud, W.K., 2006b. Reliable pneumatic conveying of .  
891 *Aquacult. Eng.* 35, 14-25. . <https://doi.org/10.1016/j.aquaeng.2005.06.006>
- 892 Aas, T. S., Oehme, M., Sørensen, M., He, G., Lygren, I., Åsgård, T., 2011. Analysis of pellet  
893 degradation of extruded high-energy fish feeds with different physical qualities in a  
894 pneumatic feeding system. *Aquacult. Eng.* 44, 25-34.  
895 <https://dx.doi.org/10.1016/j.aquaeng.2010.11.002>
- 896 Altan, A., McCarthy, K.L., Maskan, M., 2009. Effect of screw configuration and raw material  
897 on some properties of barley extrudates. *J. Food Eng.* 92, 377-382.  
898 <https://doi.org/10.1016/j.jfoodeng.2008.12.010>
- 899 Alvarez-Martinez, L., Kondury, K.P., Harper, J.M., 1988. A general model for expansion of  
900 extruded products. *J. Food Sci.* 53, 609-615. [https://doi.org/10.1111/j.1365-](https://doi.org/10.1111/j.1365-2621.1988.tb07768.x)  
901 [2621.1988.tb07768.x](https://doi.org/10.1111/j.1365-2621.1988.tb07768.x)
- 902 Anderson, R. A., Conway, H., Peplinski, A. J., 1970. Gelatinization of corn grits by roll  
903 cooking, extrusion cooking and steaming. *Starch-Stärke* 22 (4), 130-135.  
904 <https://doi.org/10.1002/star.19700220408>
- 905 Andorsdottir, G., 1985. PhD. Protein quality, methionine supplementation and fat levels in  
906 starter diets for salmon fry. *Cand. Scient. University of Oslo*, p. 109.
- 907 Baik, B. K., Powers, J., Nguyen, L. T., 2004. Extrusion of regular and waxy barley flours for  
908 production of expanded cereals. *Cereal Chem.* 81, 94-99.  
909 <https://doi.org/10.1094/CCHEM.2004.81.1.94>
- 910 Bell, G.J., Koppe, W., 2010. Lipids in Aquafeeds. In: Turchini, G.M., Ng, W.K., Tocher, D.  
911 R. (Eds.), *Fish Oil Replacement and Alternative Lipid Sources in Aquaculture Feeds*. CRC  
912 Press, Boca Raton, pp. 21-59.
- 913 Blanche, S. & Sun, X., 2004. Physical characterization of starch extrudates as function of  
914 melting transitions and extrusion conditions. *Adv. Polym. Technol.* 23, 277-290.  
915 <https://doi.org/10.1002/adv.20017>
- 916 Bortone, E. J., 2004. Extrusion processing part I. Ingredient functionality of formulas. *Global*  
917 *Aquaculture Advocate.* 7, 69-72. Retrieved from  
918 <https://www.aquaculturealliance.org/advocate/extrusion-processing-of-aquatic-feeds-part-1/>  
919 (accessed 25 January 2021).



920 Bortone, E., 2006. Vacuum Process Supports High Oil Content in Feed Coatings. Global  
921 Aquaculture Advocate, July/August 2006, pp. 68-69. Retrieved from:  
922 [https://www.aquaculturealliance.org/advocate/vacuum-process-supports-high-oil-content-in-](https://www.aquaculturealliance.org/advocate/vacuum-process-supports-high-oil-content-in-feed-coatings/)  
923 [feed-coatings/](https://www.aquaculturealliance.org/advocate/vacuum-process-supports-high-oil-content-in-feed-coatings/) (accessed 30 January 2020).

924 Bortone, E., 2017. Performance of aquafeed ingredients in extrusion cooking. Global  
925 Aquaculture Advocate, October 2017. Retrieved from  
926 [https://www.aquaculturealliance.org/advocate/preconditioning-crucial-aquafeed-](https://www.aquaculturealliance.org/advocate/preconditioning-crucial-aquafeed-manufacturing/)  
927 [manufacturing](https://www.aquaculturealliance.org/advocate/preconditioning-crucial-aquafeed-manufacturing/) (accessed 14 March 2021).

928 Burel, C., 2017. Bases de la nutrition et formulation en aquaculture. Durabilité des aliments  
929 pour le poisson en aquaculture : réflexions et recommandations sur les aspects  
930 technologiques, économiques, sociaux et environnementaux, UICN - Union Internationale  
931 pour la Conservation de la Nature; Comité Français de l'UICN, 296 p., 2017,  
932 9782831718316. Hal-01607152. <https://doi.org/10.2305/IUCN.CH.2017.02.fr>

933 Chaabani, A., Labonne, L., Tercero, C. A., Picard, J. P., Advenier, C., Durrieu V., Rouilly,  
934 A., Skiba, F., Evon, Ph., 2020. Optimization of vacuum coating conditions to improve oil  
935 retention in trout feed. Aquacult. Eng. 91, 102-127.  
936 <https://doi.org/10.1016/j.aquaeng.2020.102127>

937 Cheng, H., Friss, A., 2010. Modelling extrudate expansion in a twin-screw food extrusion  
938 cooking process through dimensional analysis methodology. Food Bioprod. Process. 88, 188-  
939 194. <https://doi.org/10.1016/j.fbp.2010.01.001>

940 Chevanan, N., Muthukumarappan, K., Rosentrater, K.A. & Julson, J.L., 2007. Effect of die  
941 dimensions on extrusion processing parameters and properties of DDGS-based aquaculture  
942 feeds. Cereal Chem. 84(4), 389-398. <https://doi.org/10.1094/CCHEM-84-4-0389>

943 Chevanan, N., Muthukumarappan, K. & Rosentrater, K.A., 2009. Extrusion studies of  
944 aquaculture feed using distillers dried grains with solubles and whey. Food Bioprocess.  
945 Technol. 2(2), 177-185. <https://doi.org/10.1007/s11947-007-0036-8>

946 Clayton, G., 2002. Better product density control. Feed International, 23(11), 4.

947 Craig, S., 2009. Understanding fish Nutrition, Feeds, and Feeding. VCE Publications,  
948 Virginia Tech. Retrieved from  
949 [https://vtechworks.lib.vt.edu/bitstream/handle/10919/48950/420-256\\_pdf.pdf?sequence=1](https://vtechworks.lib.vt.edu/bitstream/handle/10919/48950/420-256_pdf.pdf?sequence=1)  
950 (accessed 2 January 2020).

951 Davidson, V. J., Paton, D., Diosady, L. L., Larocque, G. J., 1984. Degradation of wheat starch  
952 in a single screw extruder: Characteristics of extruded starch polymers. J. Food Sci. 49, 453-  
953 458. <https://doi.org/10.1111/j.1365-2621.1984.tb12441.x>

954 Dethlefsen, M.W., 2017. PhD. Die Hard - improving the physical quality of extruded fish,  
955 feed pellets. Kgs. Lyngby: National Food Institute, Technical University of Denmark.  
956 [https://backend.orbit.dtu.dk/ws/files/138706490/\\_public.pdf](https://backend.orbit.dtu.dk/ws/files/138706490/_public.pdf) (accessed 26 January 2021).

957 Draganovic, V., Van der Goot, A.J., Boom, R., Jonkers, J., 2011. Assessment of the effects of  
958 fishmeal, wheat gluten, soy protein concentrate and feed moisture on extruder system  
959 parameters and the technical quality of fish feed. Anim. Feed Sci. Technol. 165, 238-250.  
960 <https://doi.org/10.1016/j.anifeedsci.2011.03.004>

961 Finley, J.W., 1989. Effects of processing on proteins: an overview. In: Protein Quality and the  
962 Effects of Processing (Phillips, R.D. & Finley, J.W. eds), pp. 1-8. Dekker, New York.

963 Gaosong, J., Vasanthan, T., 2000. Effect of Extrusion Cooking on the Primary Structure and  
964 Water solubility of b-Glucans from Regular and Waxy Barley. Cereal Chem. 77, 396-400.  
965 <https://doi.org/10.1094/CCHEM.2000.77.3.396>

966 Glencross, B., Rutherford, N. & Hawkins, W., 2011. A comparison of the growth  
967 performance of rainbow trout (*Oncorhynchus mykiss*) when fed soybean, narrow-leaf or  
968 yellow lupin meals in extruded diets. Aquacult. Nutr. 17, e317-e325.  
969 <https://doi.org/10.1111/j.1365-2095.2010.00765.x>

970 Godavarti, S., Karwe, M.V., 1997. Determination of specific mechanical energy distribution  
971 on a twin-screw extruder. J. Agr. Eng. Res. 67, 277-287.  
972 <https://doi.org/10.1006/jaer.1997.0172>

973 Gomez, M. H., Aguilera, J. M., 1984. A physicochemical model for extrusion of cornstarch. J.  
974 Food Sci. 49, 40-43. <https://doi.org/10.1111/j.1365-2621.1984.tb13664.x>

975 Guha, M., Ali, S. Z., Bhattacharya, S., 1997. Twin-screw extrusion of rice flour without a die:  
976 Effect of barrel temperature and screw speed on extrusion and extrudates characteristics. J.  
977 Food Eng. 32, 251-267. [https://doi.org/10.1016/S0260-8774\(97\)00028-9](https://doi.org/10.1016/S0260-8774(97)00028-9)

978 Hansen, J.Ø., Storebakken, T., 2007. Effects of dietary cellulose level on pellet quality and  
979 nutrient digestibilities in rainbow trout (*Oncorhynchus mykiss*). Aquaculture 272 (1-4), 458-  
980 465. <https://doi.org/10.1016/j.aquaculture.2007.09.005>

981 Hansen, J.Ø., Penn, M., Øverland, M., Shearer, K.D., Krogdahl, Á., Mydland, L.T.,  
982 Storebakken, T., 2010. High inclusion of partially deshelled and whole krill meals in diets for  
983 Atlantic salmon (*Salmo salar*). Aquaculture 310, 164-172.  
984 <https://doi.org/10.1016/j.aquaculture.2010.10.003>.

985 Hemre, G.I., Mommsen, T.P., Krogdahl, Á., 2002. Carbohydrates in fish nutrition: effects on  
986 growth, glucose metabolism and hepatic enzymes. Aquacult. Nutr. 8, 175-194.  
987 <https://doi.org/10.1046/j.1365-2095.2002.00200.x>

988 Kaliyan, N., Morey, R.V., 2009. Factors affecting strength and durability of densified  
989 products. Biomass Bioenerg. 33, 337-359. <https://doi.org/10.1016/j.biombioe.2008.08.005>

990 Khater, E.S.G., Bahnasawy, A.H., Ali, S.A., 2014. Physical and mechanical properties of fish  
991 feed pellets. J. Food Process. Technol. 5(10), 1000378. <https://doi.org/10.4172/2157-7110.1000378>

992 Kim, C. H., Maga, J. A., Martin, J. T., 1989. Properties of extruded blends of wheat dried  
993 distiller grain flour with other flours. Int. J. Food Sci. Tech. 24, 373-384.  
994 <https://doi.org/10.1111/j.1365-2621.1989.tb00657.x>

995 Kirby, A. R., Ollett, A. L., Parker, R., Smith, A. C., 1988. An experimental study of screw  
996 configuration effects in the twin-screw extrusion cooking of maize grits. J. Food Eng. 8(4),  
997 247-272. [https://doi.org/10.1016/0260-8774\(88\)90016-7](https://doi.org/10.1016/0260-8774(88)90016-7)

998 Knight, J. W., 1969. The starch industry (1st ed.). New York, NY, USA: Pergamon.  
999 <https://www.worldcat.org/title/starch-industry/oclc/706868822>

- 1000 Kraugerud, O.F., Jørgensen, H.Y., Svihus, B., 2011. Physical properties of extruded fish feed  
1001 with inclusion of different plant (legumes, oilseeds, or cereals) meals. Anim. Feed Sci.  
1002 Technol. 163(2-4), 244-254. <https://doi.org/10.1016/j.anifeedsci.2010.11.010>
- 1003 Kraugerud, O.F., Svihus, B., 2011. Effects of online pretreatment of plant ingredients on  
1004 processing responses and physical properties in extruded fish feed. Anim. Feed Sci. Tech.  
1005 168, 250-256. <https://doi.org/10.1016/j.anifeedsci.2011.04.089>
- 1006 Lamichhane, S., Sahtout, K., Smillie, J., Scott, T.A., 2015. Vacuum coating of pelleted feed  
1007 for broilers: opportunities and challenges. Anim. Feed Sci. Technol. 200, 1–7.  
1008 <https://doi.org/10.1016/j.anifeedsci.2014.11.015>
- 1009 Lavocat, E., Raymond, S., 2018. Preconditioning in Animal Feed Extrusion Processing.  
1010 Animal Feed Extrusion Course organized by Cleextral at Saint Etienne (LYON), October 16<sup>th</sup>,  
1011 2018.
- 1012 Li, Y., Li, J., Liu, Z., Ruan, R., Mao, Z., 2003. Vacuum coating of heat-sensitive liquid  
1013 ingredients onto feed pellets. Trans. ASAE. 46 (2), 383-387.
- 1014 Lin, S., Hseih, F., Huff, H. E., 1997. Effect of lipids and processing conditions on degree of  
1015 starch gelatinization of extruded dry pet food. LWT-Food Sci. Technol. 30, 754-761.  
1016 <https://doi.org/10.1006/fstl.1997.0271>
- 1017 Ljøkjel, K., Harstad, O.M., Skrede, A., 2000. Effect of heat treatment of soybean meal and  
1018 fishmeal on amino acid digestibility in milk and dairy cows. Anim. Feed Sci. Technol. 84, 83-  
1019 95. [https://doi.org/10.1016/S0377-8401\(00\)00104-8](https://doi.org/10.1016/S0377-8401(00)00104-8)
- 1020 Maas, R.M., Verdegem, M.C.J., Stevens, T.L., Schrama, J.W., 2020. Effect of exogenous  
1021 enzymes (phytase and xylanase) supplementation on nutrient digestibility and growth  
1022 performance of Nile tilapia (*Oreochromis niloticus*) fed different quality diets. Aquaculture  
1023 529, 735723. <https://doi.org/10.1016/j.aquaculture.2020.735723>
- 1024 Mason, W. R., Hosney, R. C., 1986. Factors affecting the viscosity of extrusion cooked  
1025 wheat starch. Cereal Chem. 63, 436-441.
- 1026 Médale, F., Guillaume, J.C., 1999. Nutrition énergétique. In : Guillaume, J., Kaushik, S.,  
1027 Bergot, P., Métailler, R. (Eds), Nutrition des poissons et des crustacés, INRA, Paris, France :  
1028 87-111.
- 1029 Mercier, C., Linko, P., Harper, J.M., 1989. Extrusion Cooking, p. 471. American Association  
1030 of Cereal Chemists, St Paul, MN. <https://doi.org/10.1002/star.19900420115>.
- 1031 Moraru, C.I., Kokini, J.L., Kokini 2003. Nucleation and expansion during extrusion and  
1032 microwave heating of cereals foods. Compr. Rev. Food Sci. Food Saf. 2, 147-165.  
1033 <https://doi.org/10.1111/j.1541-4337.2003.tb00020.x>
- 1034 Morken, T., Kraugerud, O.F., Sørensen, M., Storebakken, T., Hillestad, M., Christiansen, R.,  
1035 Øverland, M., 2012. Effects of feed processing conditions and acid salts on nutrient  
1036 digestibility and physical quality of soy-based diets for Atlantic salmon (*Salmo salar*).  
1037 Aquacult. Nutr. 18(1), 21-34. <https://doi.org/10.1111/j.1365-2095.2011.00872.x>

- 1038 Obaldo, L.G., Dominy, W.G., Ryu, G.H., 2000. Extrusion processing and its effect on  
1039 aquaculture diet quality and shrimp growth. *J. Appl. Aquacult.* 10(2), 41-54.  
1040 [https://doi.org/10.1300/J028v10n02\\_04](https://doi.org/10.1300/J028v10n02_04)
- 1041 Oikonomou, N. A., Krokida, M. K., 2012. Water absorption index and water solubility index  
1042 prediction for extruded food products. *Int. Food Prop.* 15, 157-168.  
1043 <https://doi.org/10.1080/10942911003754718>
- 1044 Opstvedt, J., Miller, R., Hardy, R.W., Spinelli, J., 1984. Heat-induced changes in sulfhydryl  
1045 groups and disulfide bonds in fish protein and their effect on protein and amino acid  
1046 digestibility in rainbow trout (*Salmo gairdneri*). *J. Agric. Food. Chem.* 32(4) 929-935.
- 1047 Pandey, B., 2018. Pellet Technical Quality of Feeds for Atlantic Salmon. Master's Thesis.  
1048 Norwegian University of Life Sciences. Ås.-96.
- 1049 Pastor-Cavada, E., Drago, S., González., R., Juan, R., Pastor, J., Alai., M., Vioque, J., 2011.  
1050 Effects of the addition of wild legumes (*Lathyrus annuus* and *Lathyrus clymenum*) on the  
1051 physical and nutritional properties of extruded products based on whole corn and brown rice.  
1052 *Food Chem.* 128, 961-967. <https://doi.org/10.1016/j.foodchem.2011.03.126>
- 1053 Pietsch, W., 1983. Agglomerate bonding and strength. In: *Elements II. Briquetting and*  
1054 *Agglomeration. Proceedings of the Institute for Briquetting and Agglomeration, Manitowish*  
1055 *Waters, WI, USA (ed. by R.M. Koerner & J.A.MacDougall), pp. 9-40. Engineering*  
1056 *Information, Inc., Hoboken, NJ, USA.*
- 1057 Pietsch, W., 2002. *Agglomeration Processes: Phenomena, Technologies, Equipment (p. 622).*  
1058 *Weinheim: Wiley-VCH.* <https://dx.doi.org/10.1002/9783527619801>
- 1059 Ravindran, G., Carr, A., Hardacre, A., 2011. A comparative study of the effects of three  
1060 galactomannans on the functionality of extruded pea-rice blends. *Food Chem.* 124, 1620-  
1061 1626. <https://dx.doi.org/10.1016/j.foodchem.2010.08.030>
- 1062 Riaz, M.N., 2000. *Extruders in Food Applications.* CRC Press.
- 1063 Riaz, M.N., 2019. Food extruders. In *Handbook of farm, dairy and food machinery*  
1064 *engineering (pp. 483-497).* Academic Press. [https://doi.org/10.1016/B978-0-12-814803-](https://doi.org/10.1016/B978-0-12-814803-7.00019-1)  
1065 [7.00019-1](https://doi.org/10.1016/B978-0-12-814803-7.00019-1)
- 1066 Riaz, M.N., Rokey, G.J., 2011. *Extrusion Problems Solved: Food, Pet Food and Feed.*  
1067 *Woodhead Publishing Limited, Sawston.*
- 1068 Rokey, G.J., 1994. Pet food and fish food extrusion. In: *The Technology of Extrusion*  
1069 *Cooking (Frame, N.D. ed.), 1<sup>st</sup> edn, pp. 144-189.* Blackie Academic & Professional, Glasgow,  
1070 UK.
- 1071 Rokey, G.J., 2006. *Increasing Aquatic Feed Production through Plant Optimization,* Wenger  
1072 *Manufacturing Inc. Publication, pp. 83-88.*
- 1073 Rosentrater, K.A., Muthukumarappan, K., Kannadhasan, S., 2009. Effects of ingredients and  
1074 extrusion parameters on aquafeeds containing DDGS and potato starch. *J. Aquacult. Feed Sci.*  
1075 *Nutr.* 1, 22-38. <https://medwelljournals.com/abstract/?doi=joafsnu.2009.22.38>

- 1076 Saalia, F. K., Phillips, R. D., 2011. Degradation of aflatoxins by extrusion cooking: Effects on  
1077 nutritional quality of extrudates. *Lwt-Food Sci. Technol.* 44, 1496-1501.  
1078 <https://doi.org/10.1016/j.lwt.2011.01.021>
- 1079 Seker, M., 2005. Selected properties of native or modified maize starch/soy protein mixtures  
1080 extruded at varying screw speed. *J. Sci. Food Agr.* 85, 1161-1165.  
1081 <https://doi.org/10.1002/jsfa.2086>
- 1082 Semenova, M.G., Belyakova, L.E., Polikarpov, Y.N., Il'in, M.M., Istarova, T.A., Anokhina,  
1083 M.S., Tsapkina, E.N., 2006. Thermodynamic analysis of the impact of the surfactant–protein  
1084 interactions on the molecular parameters and surface behavior of food proteins.  
1085 *Biomacromolecules* 7(1), 101–113. <https://doi.org/10.1021/bm050455m>
- 1086 Sørensen, M., 2012. A review of the effects of ingredient composition and processing  
1087 conditions on the physical qualities of extruded high-energy fish feed as measured by  
1088 prevailing methods. *Aquacult. Nutr.* 18, 233-248. <https://doi.org/10.1111/j.1365-2095.2011.00924.x>
- 1090 Sørensen, M., Ljøkjel, K., Storebakken, T., Shearer, K.D. & Skrede, A., 2002. Apparent  
1091 digestibility of protein, amino acids and energy in rainbow trout (*Oncorhynchus mykiss*) fed a  
1092 fishmeal based diet extruded at different temperatures. *Aquaculture* 211, 215-225.  
1093 [https://doi.org/10.1016/S0044-8486\(01\)00887-0](https://doi.org/10.1016/S0044-8486(01)00887-0)
- 1094 Sørensen, M., Stjepanovic, N., Romarheim, O.H., Krekling, & Storebakken, T., 2009.  
1095 Soybean meal improves the physical quality of extruded fish feed. *Anim. Feed Sci. Technol.*  
1096 149(1-2), 149-161. <https://doi.org/10.1016/j.anifeedsci.2008.05.010>
- 1097 Sørensen, M., Luyen, G.Q.N., Storebakken, T. & Øverland, M., 2010. Starch source, screw  
1098 configuration and injection of steam into the barrel affect physical quality of extruded fish  
1099 feed. *Aquacult. Res.* 41(3), 419-432. <https://doi.org/10.1111/j.1365-2109.2009.02346.x>
- 1100 Sørensen, M., Morken, T., Kosanovic, M., Øverland, M., 2011. Pea and wheat starch possess  
1101 different processing characteristics and affect physical quality and viscosity of extruded feed  
1102 for Atlantic salmon. *Aquacult. Nutr.* 17, e326-e336. <https://doi.org/10.1111/j.1365-2095.2010.00767.x>
- 1104 Strahm, B.S., 2000. Preconditioning. In: *Extruders in Food Applications* (Riaz, M.N. ed.), pp.  
1105 115-126. Technomic Publishing Company Inc., Lancaster, PA.
- 1106 Terpstra, A. H., 2015. *The Composition and Production of Fish Feeds, An Overview of Data*  
1107 *from the Literature and the Internet. The Netherlands, Anno Domini MMXV.* Pdf. Retrieved  
1108 from [https://vdocuments.mx/the-composition-and-production-of-fish-ahm-terpstra-2015-de-](https://vdocuments.mx/the-composition-and-production-of-fish-ahm-terpstra-2015-de-alimenti-piscibus.html)  
1109 [alimenti-piscibus.html](https://vdocuments.mx/the-composition-and-production-of-fish-ahm-terpstra-2015-de-alimenti-piscibus.html) (accessed 15 January 2021).
- 1110 Tharanathan, R. N., 1995. Starch: The polysaccharides of high abundance and usefulness. *J.*  
1111 *Sci. Ind. Res. (India)* 54, 452-458. <http://ir.cftri.res.in/id/eprint/5486>
- 1112 Van den Einde, R. M., Van der Goot, A. J., Boom, R. M., 2003. Understanding molecular  
1113 weight reduction of starch during heating-shearing processes. *J. Food Eng.* 68, 2396-2404.
- 1114 Williams, M.A., 2000. Interrupted-Flight Expanders-Extruders. *Extruders in Food*  
1115 *Applications*, 63-79.

1116 Yahata, Y., Fukuoka, M., Mihori, T., Watanabe, H., 2006. Water can be made to diffuse  
1117 against the water content gradient in starchy foods. J. Food Eng. 76, 490-499.  
1118 <https://doi.org/10.1016/j.jfoodeng.2005.05.050>  
1119

1120 **Tables and figures captions**

1121

1122 **Table 1**

1123 Composition of the final extruded pellets (contents of chemicals are expressed in % (w/w) in  
1124 proportion to the fresh matter).

1125

1126 **Table 2**

1127 Operating conditions applied to produce the extruded pellets (S1, S2, S3, S4, S5 and S6).

1128

1129 **Table 3**

1130 Starch gelatinization enthalpy and gelatinization rate for the initial mixture, the  
1131 preconditioned ones and all the extruded pellets before coating.

1132

1133 **Table 4**

1134 Twin-screw extrusion parameters, pellets characterizations and pellets usage properties for the  
1135 S1 to S6 formulations.

1136

1137 **Table 5**

1138 Distribution in volume of the starchy matrix, the oil-filled pores and the empty ones inside the  
1139 coated pellets.

1140

1141 **Table 6**

1142 WSI and WAI results measured on the preconditioned mixture and extruded pellets.

1143

1144 **Fig. 1.** General view (on the left) and inside (on the right) of a preconditioner (illustrations  
1145 reproduced with the authorization of the Clextral Company).

1146

1147 **Fig. 2.** Simplified diagram of the *Fish Feed* process used to produce the extruded pellets of  
1148 formulations S1 and S2.

1149

1150 **Fig. 3.** DSC analysis of the initial mixture, the preconditioned one and the extruded uncoated  
1151 pellets obtained at 575 rpm.

1152

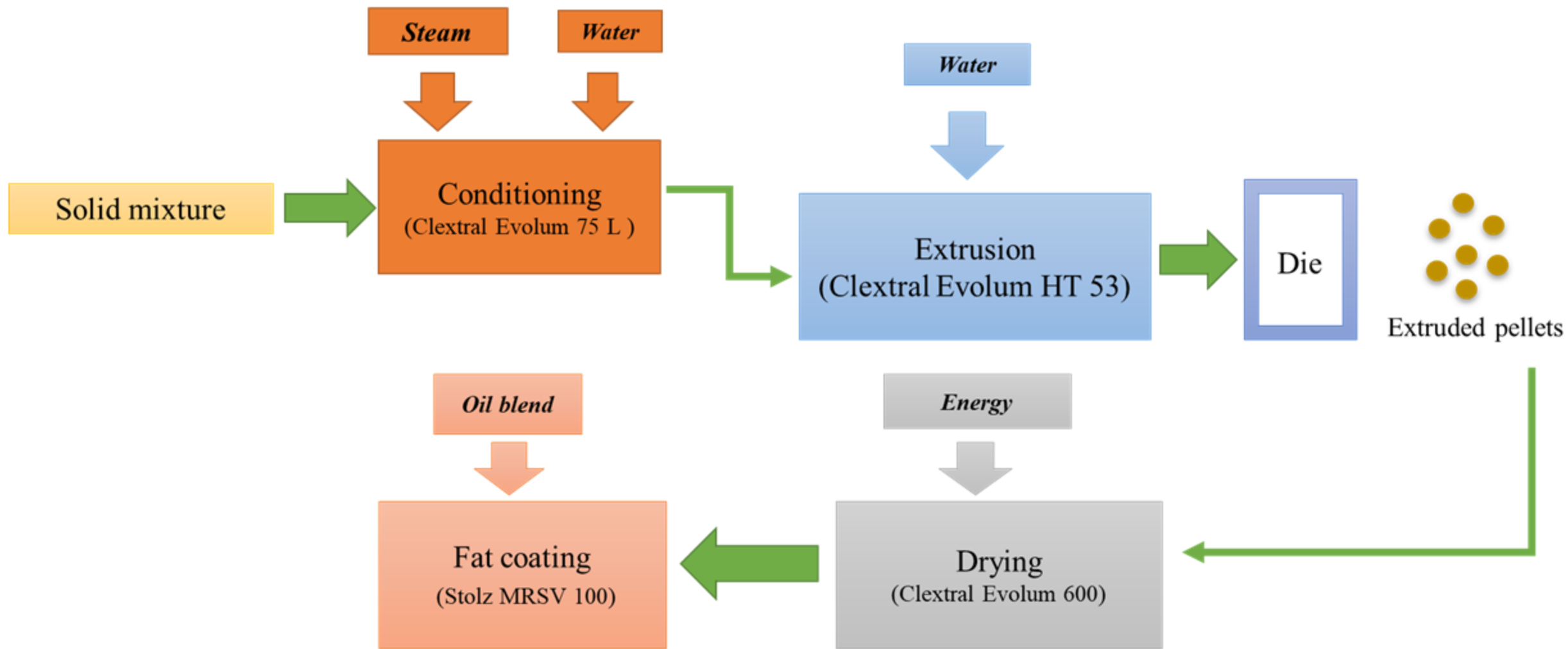
1153 **Fig. 4.** Photographs of the extruded pellets obtained at a screw speed of 575 rpm (uncoated  
1154 pellets (dryer outlet) are presented at the top and the coated ones at the bottom; from left to  
1155 right: S2, S4 and S6) (8.5 cm for the Petri dish diameter).

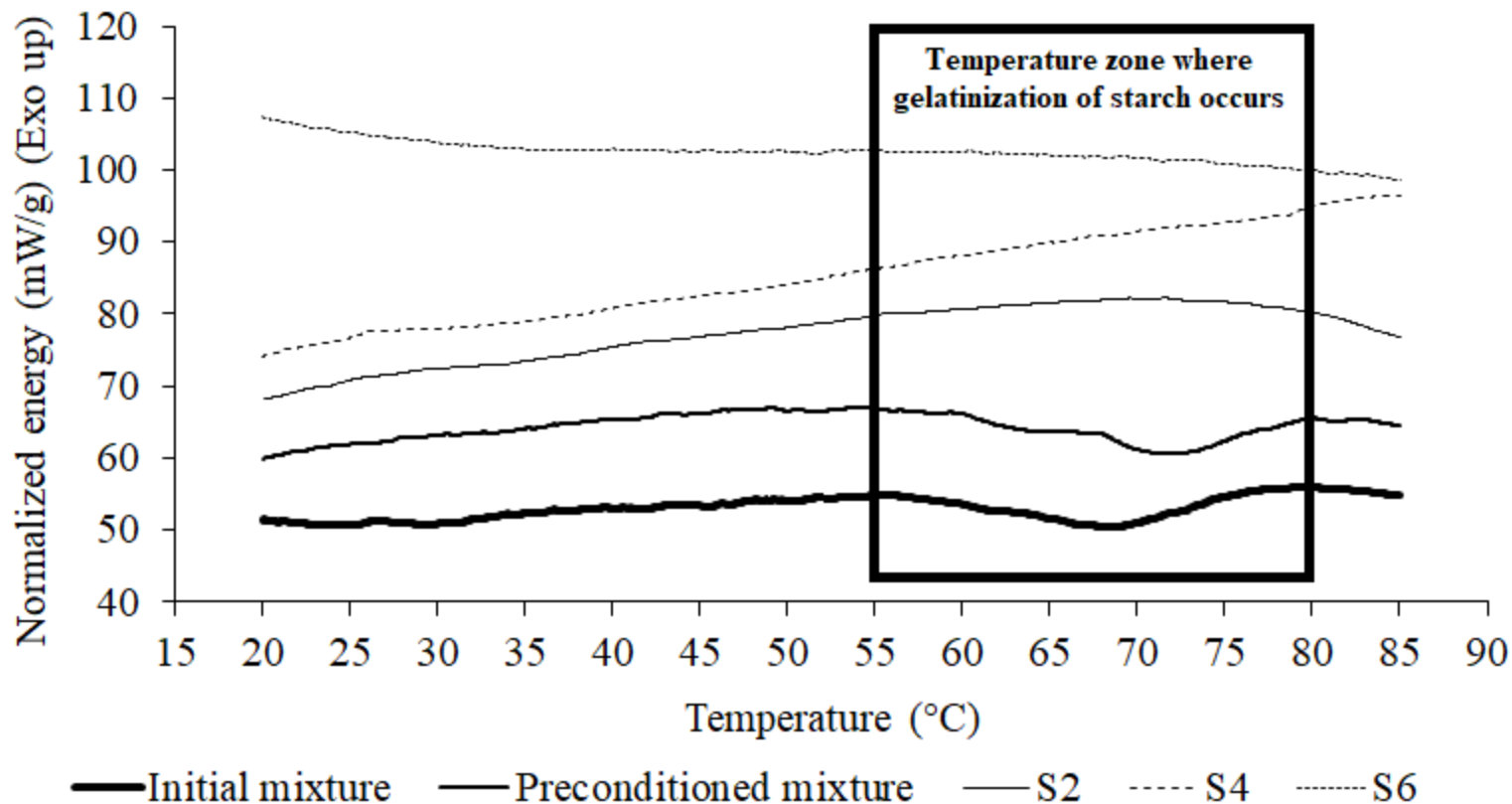
1156

1157 **Fig. 5.** Tomography images of *Fish Feed* pellets from formulations (a) S1, (b) S2, (c) S3, (d)  
1158 S4, (e) S5, and (f) S6 (425 rpm for the screw speed for the three images on the top, and 575  
1159 rpm for the three ones below). The volume occupied by the starchy matrix appears in light  
1160 grey and white, and the empty pores in black. The oil is also visible inside the coated pellets,  
1161 appearing in dark grey (*i.e.*, slightly darker than the starchy matrix).









Uncoated extruded pellets



Coated extruded pellets



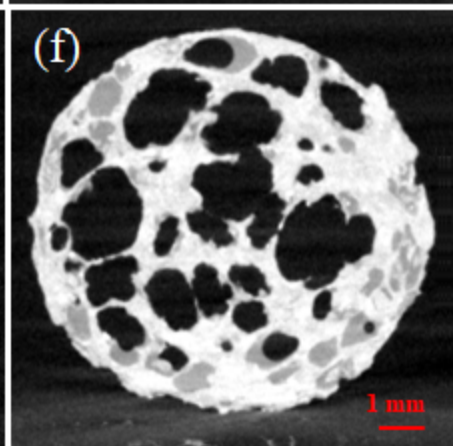
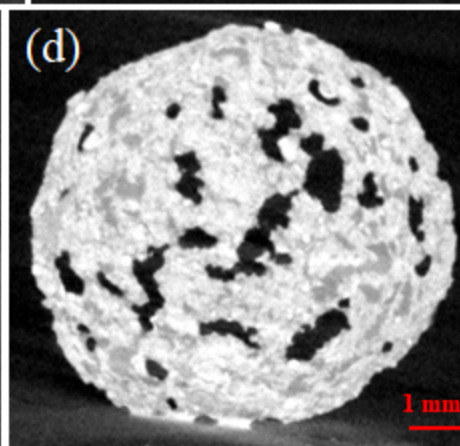
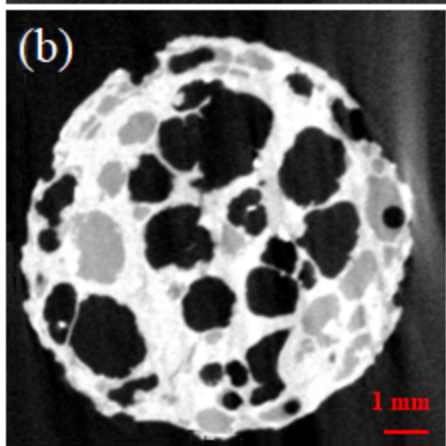
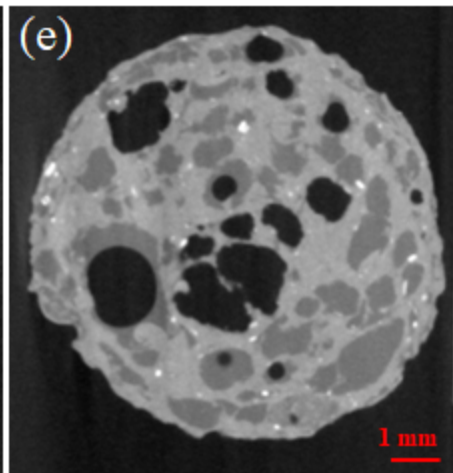
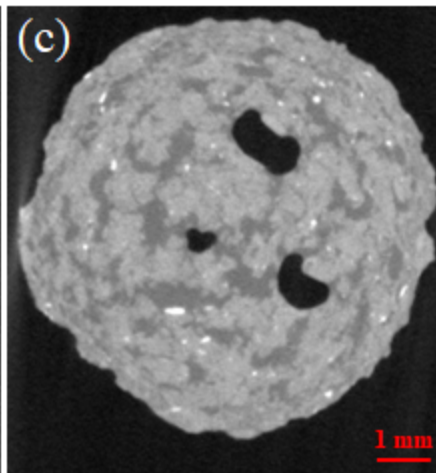
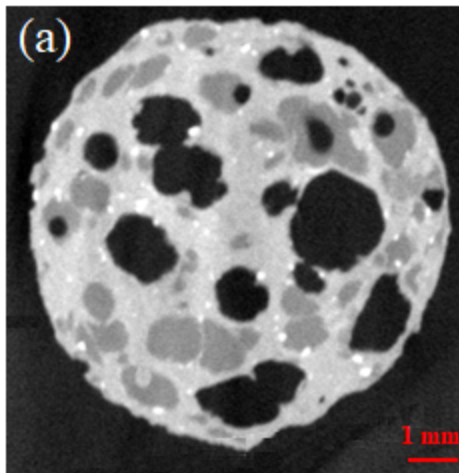
S2



S4



S6



**Table 1**

Composition of the final extruded pellets (contents of chemicals are expressed in % (w/w) in proportion to the fresh matter).

<b>Chemical</b>	<b>Content (% (w/w))</b>
<b>Crude protein</b>	35.5
<b>Fat</b>	30.0
<b>Starch</b>	11.1
<b>Crude cellulose</b>	2.9
<b>Ash</b>	4.9
<b>Moisture</b>	7.0

**Table 2**

Operating conditions applied to produce the extruded pellets (S1, S2, S3, S4, S5 and S6).

Operating conditions	With preconditioner			Without preconditioner		
	S1	S2	S3	S4	S5	S6
<b>Solid inlet flow rate (kg/h)</b>	110	110	110	110	110	110
<b>Screw speed (rpm)</b>	425	575	425	575	425	575
<b>Device's filling coefficient (kg/h rpm)</b>	0.258	0.191	0.258	0.191	0.258	0.191
<b><math>Q_V</math> (kg/h)</b>	13.2	13.2	0.0	0.0	0.0	0.0
<b><math>Q_{PREC}</math> (kg/h)</b>	10.9	10.9	0.0	0.0	0.0	0.0
<b><math>Q_{TSE}</math> (kg/h)</b>	15.6	15.6	26.5	26.5	30.0	30.0
<b>Water-to-solid ratio</b>	0.361	0.361	0.241	0.241	0.273	0.273

*The device's filling coefficient is defined as the ratio of the solid inlet flow rate to the screw speed.  $Q_V$  is the steam flow rate in the preconditioner;  $Q_{PREC}$  is the water flow rate in the preconditioner;  $Q_{TSE}$  is the water flow rate in the twin-screw extruder.*

**Table 3**

Starch gelatinization enthalpy and gelatinization rate for the initial mixture, the preconditioned ones and all the extruded pellets before coating.

<b>Code</b>	<b>S0</b>	<b>S1</b>	<b>S2</b>	<b>S1</b>	<b>S2</b>	<b>S3</b>	<b>S4</b>	<b>S5</b>	<b>S6</b>
		<b>PREC</b>	<b>PREC</b>						
<b>Material</b>	Initial mixture	Preconditioned mixtures		Powder from extruded pellets (dryer outlet)					
	1.14	0.45	0.47						
<b><math>\Delta H</math> (J/g)</b>	±	±	±	0.00	0.00	0.00	0.00	0.00	0.00
	0.03	0.01	0.01						
<b>GR (%)</b>	n.a*	60.5	58.8	100.0	100.0	100.0	100.0	100.0	100.0

\* *n.a.*: not applicable; *GR*: gelatinization rate (%).



**Table 4**

Twin-screw extrusion parameters, pellets characterizations and pellets usage properties for the S1 to S6 formulations.

Formulations	With preconditioner			Without preconditioner		
	S1	S2	S3	S4	S5	S6
<b>Screw speed (rpm)</b>	<b>425</b>	<b>575</b>	<b>425</b>	<b>575</b>	<b>425</b>	<b>575</b>
<b>Twin-screw extrusion parameters</b>						
<b>Moisture at preconditioner outlet (%)</b>	18.9 <sup>a</sup> ± 0.1	19.0 <sup>a</sup> ± 0.2	n.a	n.a	n.a	n.a
<b>Material temperature at preconditioner outlet (°C)</b>	76 <sup>a</sup> ± 1	73 <sup>b</sup> ± 2	n.a	n.a	n.a	n.a
<b>I (A)</b>	39 <sup>d</sup> ± 2	39 <sup>d</sup> ± 1	50 <sup>a</sup> ± 1	44 <sup>b</sup> ± 1	40 <sup>c</sup> ± 1	37 <sup>e</sup> ± 1
<b>SME (W h/kg)</b>	76 <sup>e</sup> ± 3	102 <sup>b</sup> ± 3	97 <sup>c</sup> ± 2	117 <sup>a</sup> ± 4	78 <sup>d</sup> ± 3	98 <sup>c</sup> ± 4
<b>Counter-pressure (bar)</b>	14 <sup>e</sup> ± 3	9 <sup>f</sup> ± 3	35 <sup>a</sup> ± 1	29 <sup>b</sup> ± 2	23 <sup>c</sup> ± 4	19 <sup>d</sup> ± 3
<b>Pellets characterizations</b>						
<b>Moisture at extruder outlet (%)</b>	25.4 <sup>a</sup> ± 0.1	24.7 <sup>b</sup> ± 0.1	19.8 <sup>c</sup> ± 0.2	19.8 <sup>c</sup> ± 0.1	24.3 <sup>b</sup> ± 0.3	24.4 <sup>b</sup> ± 0.3
<b>Bulk density (extruder outlet) (g/L)</b>	396 <sup>c</sup> ± 1	361 <sup>e</sup> ± 5	486 <sup>a</sup> ± 8	377 <sup>d</sup> ± 3	434 <sup>b</sup> ± 6	386 <sup>cd</sup> ± 2
<b>Bulk density (coater outlet) (g/L)</b>	589 <sup>c</sup> ± 1	534 <sup>e</sup> ± 2	687 <sup>a</sup> ± 2	596 <sup>c</sup> ± 4	649 <sup>b</sup> ± 2	565 <sup>d</sup> ± 3
<b>Pellet diameter (dryer outlet) (mm)</b>	8.9 <sup>ab</sup> ± 0.3	9.2 <sup>a</sup> ± 0.3	7.8 <sup>c</sup> ± 0.2	8.0 <sup>c</sup> ± 0.3	8.8 <sup>b</sup> ± 0.3	9.0 <sup>ab</sup> ± 0.3
<b>Expansion ratio (%)</b>	51 <sup>ab</sup> ± 5	55 <sup>a</sup> ± 5	32 <sup>c</sup> ± 3	36 <sup>c</sup> ± 5	49 <sup>b</sup> ± 5	52 <sup>ab</sup> ± 5
<b>Pellet's porosity (%)</b>	47 <sup>a</sup> ± 5	49 <sup>a</sup> ± 1	14 <sup>b</sup> ± 1	23 <sup>b</sup> ± 5	43 <sup>a</sup> ± 3	46 <sup>a</sup> ± 4
<b>Pellets usage properties</b>						
<b>OLR (%) at 25 °C</b>	0.66 <sup>d</sup> ± 0.04	0.83 <sup>c</sup> ± 0.01	1.67 <sup>a</sup> ± 0.08	1.04 <sup>b</sup> ± 0.04	0.75 <sup>cd</sup> ± 0.06	0.83 <sup>c</sup> ± 0.09
<b>OLR (%) at 40 °C</b>	1.37 <sup>d</sup> ± 0.01	1.56 <sup>bc</sup> ± 0.07	2.17 <sup>a</sup> ± 0.00	1.48 <sup>cd</sup> ± 0.00	1.52 <sup>bc</sup> ± 0.08	1.64 <sup>b</sup> ± 0.02
<b>Pellet breakage (%)</b>	0.40 <sup>bc</sup> ± 0.10	0.10 <sup>b</sup> ± 0.00	0.55 <sup>ab</sup> ± 0.05	0.60 <sup>a</sup> ± 0.00	0.19 <sup>d</sup> ± 0.06	0.25 <sup>cd</sup> ± 0.10

<b>Dust (%)</b>	0.10 <sup>a</sup> ± 0.00	0.01 <sup>c</sup> ± 0.00	0.12 <sup>a</sup> ± 0.02	0.07 <sup>b</sup> ± 0.00	0.00 <sup>c</sup> ± 0.00	0.00 <sup>c</sup> ± 0.00
<b>Durability (%)</b>	78 <sup>cd</sup> ± 0	76 <sup>d</sup> ± 1	81 <sup>ab</sup> ± 0	72 <sup>e</sup> ± 1	84 <sup>a</sup> ± 1	80 <sup>bc</sup> ± 2
<b>Hardness (N/mm)</b>	17 <sup>b</sup> ± 3	13 <sup>d</sup> ± 2	14 <sup>cd</sup> ± 2	10 <sup>e</sup> ± 2	22 <sup>a</sup> ± 4	16 <sup>bc</sup> ± 3
<b>Floatability (%)</b>	65 <sup>c</sup> ± 2	100 <sup>a</sup> ± 0	0 <sup>e</sup> ± 0	25 <sup>d</sup> ± 3	5 <sup>e</sup> ± 1	86 <sup>b</sup> ± 2

---

*Means in the same line with the same superscript letter (a-f) are not significantly different at  $P < 0.10$ .*

**Table 5**

Distribution in volume of the starchy matrix, the oil-filled pores and the empty ones inside the coated pellets.

	<b>S1</b>	<b>S2</b>	<b>S3</b>	<b>S4</b>	<b>S5</b>	<b>S6</b>
<b>V<sub>SM</sub> (%)</b>	53 <sup>b</sup> ± 5	51 <sup>b</sup> ± 1	86 <sup>a</sup> ± 1	77 <sup>a</sup> ± 5	57 <sup>b</sup> ± 3	54 <sup>b</sup> ± 4
<b>V<sub>PO</sub> (%)</b>	29 <sup>a</sup> ± 3	24 <sup>a</sup> ± 1	11 <sup>bc</sup> ± 1	9 <sup>c</sup> ± 2	29 <sup>a</sup> ± 8	20 <sup>ab</sup> ± 1
<b>V<sub>EP</sub> (%)</b>	18 <sup>bc</sup> ± 2	25 <sup>ab</sup> ± 2	3 <sup>d</sup> ± 1	14 <sup>c</sup> ± 4	14 <sup>c</sup> ± 5	26 <sup>a</sup> ± 2

*V<sub>SM</sub> is the volume occupied by the starchy matrix. V<sub>PO</sub> is the volume occupied by the oil-filled pores. V<sub>EP</sub> is the volume occupied by empty pores. Means in the same line with the same superscript letter (a-d) are not significantly different at P < 0.10.*

**Table 6**

WSI and WAI results measured on the preconditioned mixture and extruded pellets.

<b>Formulation</b>	<b>WSI</b>	<b>WAI</b>
<b>Preconditioned mixture</b>	10.9 <sup>a</sup> ± 0.9	200 <sup>c</sup> ± 5
<b>S1</b>	12.3 <sup>a</sup> ± 1.1	259 <sup>ab</sup> ± 6
<b>S2</b>	12.7 <sup>a</sup> ± 0.2	253 <sup>ab</sup> ± 1
<b>S3</b>	12.7 <sup>a</sup> ± 1.0	242 <sup>b</sup> ± 2
<b>S4</b>	12.3 <sup>a</sup> ± 0.8	243 <sup>b</sup> ± 3
<b>S5</b>	11.6 <sup>a</sup> ± 0.6	274 <sup>a</sup> ± 12
<b>S6</b>	11.7 <sup>a</sup> ± 0.7	277 <sup>a</sup> ± 17

*Means in the same column with the same superscript letter (a-c) are not significantly different at  $P < 0.10$ .*