Optimization of vacuum coating conditions to improve oil retention in Trout feed

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A B S T R A C T

This work describes a process approach to study the oil leakage phenomenon in feeds for large Trout. Using a pilot vacuum coater, four experimental coating parameters were studied (stirring speed, pressure, filling rate, and time needed to restore atmospheric pressure) through an experimental design. The properties measured on the coated pellets were the oil leakage rate and three other important usage properties, i.e. durability, hardness, and floatability. The coating conditions had a significant influence on oil leakage rate, varying at 40 °C from 2.7 % to 1.2 % depending on the coating conditions used. Finally, the vacuum level was the most effective factor in reducing leakage. Indeed, a progressive reduction in oil leakage rate was observed as the pressure inside the coater was reduced during coating.

1. Introduction

Since the last decade, aquaculture has expanded continuously to meet the increasing demand for fish for human consumption. According to the Food and Agriculture Organization of the United Nations (FAO) report dated 2018, global fish production has reached a record value of about 171 million tons in 2016. The aquaculture sector represented 47 % of the total and 53 % if we include the reduction to fishmeal and fish oil (non-food uses). The per-capita consumption of fish has doubled from about 9 kg per year in the 1960s to approximately 20.5 kg in 2017 (FAO, 2018). Consumers appreciate fish-related products thanks to their positive connotation (Olsen, 2003; Trondsen et al., 2004). They are rich in omega-3 and other fatty acids, calcium, proteins, and vitamins, which makes them a good alternative for a healthy life style. In particular, the distribution between saturated, mono-unsaturated and poly-unsaturated fatty acids inside fish oils is generally well balanced. This protein-rich meat can be a source of fewer calories in comparison with some terrestrial animals. For example, for Trout, the calorie level is about 132 kcal per 100 g (Frida, 2020a) instead of 211 kcal for beef (rump) (Frida, 2020b). However, it is very similar to chicken (128 kcal) (Frida, 2020c).

Eating fish has many health benefits and can even help to prevent some diseases. Twenty years ago, Connor (2000) had already demonstrated that a high consumption of fish oil is beneficial for health. For example, omega-3 fatty acids from cod liver oil or fatty fish are not only essential nutrients but they can also reduce the risk of many diseases (e.g. atherosclerosis, coronary heart disease, inflammatory disease, etc.). A very recent study also revealed that the role of omega-3 fatty acids in mental health is becoming increasingly obvious (Lange, 2020). All these reasons make aquaculture an attractive option for expanding animal protein and omega-3 fatty acid supply for consumers.

On the other hand, diets should supply all essential nutrients and energy in tune with animal requirements. It concerns essentially the maintenance of physiological functions like growth, reproduction, and health. For all animal production systems and particularly in aquaculture, the major issue is also to guarantee flesh and environmental quality, which are both related to nutrition (Kaushik, 2000).

Fish farming is a specialization of aquaculture. It refers to fish breeding in a natural environment, in an artificial pond or in RAS (Recirculating Aquaculture Systems). In particular, RAS is getting more and more attention, especially for salmon, yellowtail and, to a lesser extent, Trout. Pellets used to feed fish are considered as technological feed compared to other livestock animals (broilers, pigs, etc.). A complex technological procedure is required to produce such fish feed. This process involves the following operations, found in all fish feed

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producers: preparation of the raw materials (weighing, mixing, grinding, and sieving), conditioning, pellet forming, drying, fat coating, cooling, sieving, and lastly packaging. A simplified diagram of fish feed processing is given in Fig. 1.

Pellet forming (for example through pelleting/expansion, single-screw extrusion, or twin-screw extrusion) and coating are the main steps in the fish feed processing. Over the past forty years, extrusion technology has spread widely among the fish feed producers. The history of extrusion dates back to the end of the eighteenth century for metal forming, and it was then adapted for food processing around 1930−1940. Then, over the next thirty years, the number of applications of extrusion had expanded in several fields. In the early 1980s, extruders were introduced to fish feed processing, due to their ability to produce high-quality pellets with high lipid inclusion and tailored densities (Oliveira and De, 1990; Hilton et al., 1981; Huber, 2000). It is a thermal and mechanical treatment, in which food or feed material is forced to flow under elevated temperature, pressure, and shear, through a die. Many operating units can be performed like expansion, agglomeration of ingredients, starch gelatinization, protein denaturation, dehydration, pasteurization, mixing, texture alteration, product shaping, etc.

Compared to other thermal processes, a large variety of shapes, textures, colors, and appearances are now accessible through extrusion. Extrusion cooking at high-temperature helps with the destruction of anti-nutritional compounds, like trypsin inhibitors, microorganisms, and even undesirable enzymes, such as lipases and lipoxidases. In fact, extrusion is a high-temperature short-time heating process that contributes to minimizing food nutriment degradation while improving the digestibility of proteins and starch (by denaturation and gelatinization) from feedstuffs (Riaz and Rokey, 2011).

Thanks to its adaptability and versatility, twin-screw extrusion has become the primary processing tool to enhance the quality of feed. Particularly, even if the control of the density of fish feed pellets is also possible using a single-screw extruder, it is much easier in twin-screw extrusion to produce floating or sinking feed while ensuring consistent quality. Most fishes require floating pellets, whereas other species like shrimps need sinking ones (Craig, 2009). Salmons, seabasses, seabreams also prefer sinking feed. The same is true for Trouts, however, some farmers prefer the use of floating or semi-floating pellets as it is easier to check if the fishes are no longer hungry.

The addition of liquids, fats and oils in particular, is an important challenge in fish feed manufacturing. In aquaculture, fats (in their glyceride form) represent a rich source of energy for fish. Fats are also necessary to provide vitamins to fish, as they are soluble in them. To meet the nutritional requirements of fish, fish feed producers need to increase the amount of oil in the final feed pellet. However, this practice leads to serious technological problems (Jovanović et al., 2009).

The coating is the next operation after the pellet production (e.g. through extrusion) and the drying of pellets, in which liquid can be introduced subsequently. The traditional methods of oil application have commonly involved drum coaters, paddle mixers, and mist-coating units (Bortone, 2006). The major limitation of these types of coaters is the reduced amount of total fat they can apply. For aquatic species (especially salmonids), diets with high fat content are required to increase the dietary digestible energy (DE) level, with oil coatings exceeding 15 %. This task cannot be achieved with the equipment listed above.

More recently, the vacuum coating technology has been successfully used, even at industrial scale, for aquafeed products and pet foods, where the end-product quality and the fat level (up to 30 %) are in top priorities. It is now applied by almost all major manufacturers of feeds for salmonids. In parallel, over the past twenty years, to obtain a high level of fat inside the feed, the diets are mainly extruded (by means of single- or twin-screw extruders) rather than pelleted even if pelleted diets can be also vacuum coated when required. In fact, pellets and extruded feeds have different functional properties, due to the application of different processing conditions during their productions. Lamichhane et al. (2015) showed that vacuum coating is largely applied for high fat diets such as fish and pet extruded diets but there are also opportunities to apply vacuum coating to pelleted feed. In particular, this technology was successfully used to inject oil inside pellet pores for broilers. However, due to the lower porosity of pellets, maintaining their durability and reaching high uptakes of liquids from the vacuum technology is more delicate (Lamichhane et al., 2015).

For pelleted or extruded feed, vacuum coating is based on a simple physical exchange of air inside the feed pores with a liquid (Lamichhane et al., 2015). In the case of extruded feeds, a coating cycle starts by transferring dried pellets (approximately 8% moisture content) to the vacuum coater at a 70 °C preferred temperature (Perez, 2001), sealing the mixing vessel, and creating a vacuum. Extruded pellets are then gently mixed as the desired liquid is pumped into the coater chamber. After that, the vacuum is released. Initially, the air is removed from the inside of pellets, and vacuum release then causes the deep penetration of liquid in the pores of treated pellets. Lastly, coated pellets, at a temperature around 60 °C, are sent to a cooler prior to packaging (Perez, 2001). The volumes of both feed and liquid, the mixing intensity, and the vacuum conditions can be varied. However, they must be balanced with the maintenance of pellet quality and diet throughput. This principle described above applies to pelleted and extruded feeds.

Thanks to this technology, several applications have become possible for feed producers. Vacuum coating enables higher inclusion of fat in broiler diets without loss of the pellet durability, i.e. their resistance to fragmentation and abrasion during their distribution (Borquez and Perez, 2007). Besides, Strauch (2002) reported that the level of fat inclusion in the pelleted broiler diet was doubled to 100 g/kg by vacuum coating. Such fat addition level (i.e. 10 %) is particularly low in comparison to what is practiced in fish feeds. Depending on the feed type or

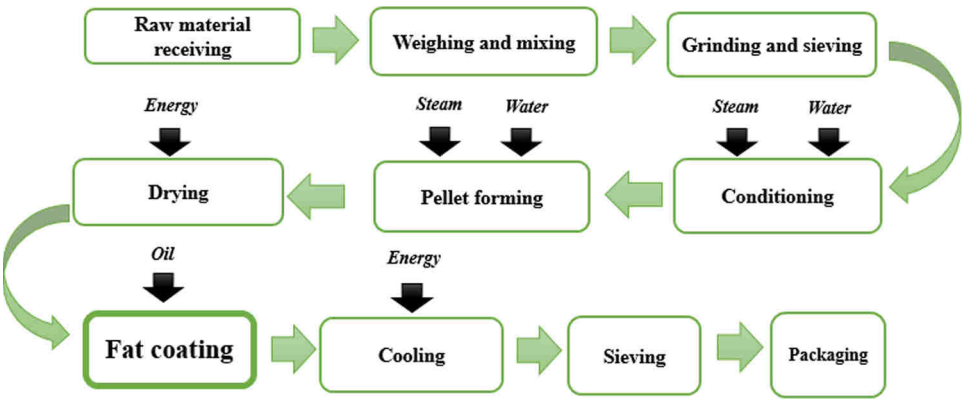

Fig. 1. Simplified fish feed diagram processing.

fish species, 4–40% of oil can be added to the extruded fish feed during the vacuum coating step. As an example, the salmon feed contains up to 40 % of oil (Bell and Koppe, 2010; Dethlefsen, 2017). The extrusion and expansion process creates enough pore space to infuse such a high level of fat. Also, it was reported that vacuum coating was used for vitamin addition, and exogenous enzyme supplementation (Maas et al., 2020). It helps protect these heat-sensitive liquid ingredients, from heat and oxidation by air (Li et al., 2003). In our case, for large Trout (i.e. weight of about 2.5 kg), the formulation has to contain up to 30 % of fat (Meng et al., 2019). When reaching these nutritional requirements, and even if vacuum coating of extruded feeds is something well established in industry, the obtained pellets may present a major quality defect: the oil leakage over time (Sørensen, 2012). For example, in the case of Atlantic salmon feeds with 31 % fat content, Pandey (2018) reported that oil leakage rates ranged from 1.7 % to 4.7 % (in proportion to the total weight of pellets), depending on the oily blend used, and the storage temperature (4 °C or 23 °C). Pellets tend to lose part of their oil under certain conditions, especially a notable increase in the ambient temperature. The high amounts of released oil can even result in the appearance of an oily film at the surface of fishponds. Oil leakage creates even bigger problems in RAS than in the natural environment, as the oil can negatively affect the microbiological filters cleaning the water in the recirculated facilities. In addition to this pollution, this released oil is lost for fish diets. Several studies have been conducted to solve this problem. A major recent advance (Dethlefsen, 2017) showed that pellets microstructure affects the physical quality of feed, in particular the oil leakage. It was found that the optimal pore structure is defined by a high pore area to volume ratio (Dethlefsen et al., 2016). To obtain this pore structure, a new generation of dies positioned at the outlet of both single- and twin-screw extruders was developed. They have shown a great ability not only for reducing significantly oil leakage but also for enhancing pellet strength. Much earlier, Nissinen and Sneddon (2000) patented a method for preparing fish feed pellets with high oil content. The latter consisted of extruding a mixture of basic components for forming a matrix of fish feed pellets together with an additive, which is solid under ambient temperature. Added during the extrusion treatment, this additional component can be a hydrogenated oil derived from animal or plant, or a lipid emulsifier such as mono-, di- or tri-glyceride. The resulting pellets released a lower oil amount during both storage and use while they may include up to 50 % (w/w) of oil.

In the literature, many coating methods are described, for example by conducting the coating operation into two successive steps: adding vegetable oil with low viscosity at the beginning of the vacuum release, and adding fish oil with higher viscosity or even hardened oils in a second time (Dethlefsen, 2017). Various release times and vacuum levels to ensure a deep distribution of oil into pores have been also investigated by the same author. For future development, another strategy could consist in conducting an additional over-coating step by applying hydrogenated oils at the pellet external surface to minimize oil leakage.

In this context, the present work aims to study the oil leakage phenomenon in fish feed, especially for large Trout. A process approach was conducted by optimizing the operating conditions of the vacuum coating step with the help of an experimental design. The first section of this work gives a brief overview of some parameters (i.e. temperature, oil viscosity, etc.) that could contribute to oil leakage, with a special focus on the mechanisms involved. In the second section, a systematic study was carried out to determine the influence of the main vacuum coating process parameters (i.e. the stirring speed, the vacuum level, the time to restore atmospheric pressure, and the filling rate of the coater) on the Oil Leakage Rate (OLR and OLR' values), and on the subsequent consequences on the pellets usage properties (durability, hardness, and floatability).

2. Materials and methods

2.1. Raw materials

2.1.1. Extruded pellets

Extruded pellets were obtained from Aqualia (Arue, France). The 9 mm pellets were prepared by using different ingredients (fishmeal, protein crops, products and by-products from cereals (wheat and corn) and oilseeds, vegetable and fish oils, vitamins, and minerals), added in proportions corresponding to the nutritional intake of Trouts. Pellets were produced through a Clextral (Firminy, France) Evolum 119+ twin-screw extruder, with a pre-cooking step using a pre-conditioner. Two different batches of pellets were used in this study: uncoated (i.e. just extruded) pellets and coated ones. These coated pellets were produced industrially using a 300 mbars pressure in the vacuum coater, and used as a reference. They were immediately stored at 5 °C after their reception and for two weeks before characterization. Uncoated pellets were maintained at ambient temperature (i.e. 20 °C).

2.1.2. Oils

Three commercial oils were involved in pellets formulation, and they were provided by Aqualia: two fish oils (FO1 and FO2), both obtained by wet pressing (0.92 g/cm^3 at 20 °C), and a crude rapeseed oil (RAO) obtained by cold mechanical pressing (0.91 g/cm^3 at 20 °C).

FO2 was added during the twin-screw extrusion process at a proportion of 2% (w/w) compared to the flour, meaning that it was already contained inside the uncoated pellets provided by Aqualia. RAO and FO1 oil batches added during pilot vacuum coating were the same as those used to produce at industrial scale the coated pellets from Aqualia. They were stored in a dry place away from light (especially for FO1 to avoid its possible degradation over time) before their use in the pellet coating step. For this, RAO and FO1 were previously combined in a 70/30 (w/w) mixture, called OCM, which was then added during vacuum coating to raise the energy content of the coated pellets.

2.2. Pellets characterization

2.2.1. Moisture content

Moisture contents of uncoated and coated pellets provided by Aqualia were determined according to ISO 665:2000 (ISO, 2000).

2.2.2. Protein content

Protein contents of uncoated and coated pellets provided by Aqualia were determined according to ISO 5983−1:2005 using the Kjeldahl method (ISO, 2005).

2.2.3. Fat content

Fat content in uncoated and coated pellets provided by Aqualia was determined according to ISO 659:2009 (ISO, 2009). This method was applied with some modifications, as the total crude fat extraction required three consecutive Soxhlet extractions, using cyclohexane as a solvent.

A first extraction was carried out on roughly crushed pellets. Then, the residual solid was recovered and subsequently finely ground using a Foss (Hillerød, Denmark) Cyclotec 1093 grinder fitted with a 2 mm sieve and then with a 1 mm one. After grinding, a second extraction step was conducted and a third one was required to ensure the complete recovery of fat. A 30–40 g test sample mass was used for the multi-stage extraction protocol.

2.2.4. NIR analysis

For NIR measurement, a Bruker Optik GmbH (MPA) (Billerica, United States) apparatus was used to determine moisture, fat, and protein contents in pellets. The infra-analyzer was calibrated with official methods. The measurements consisted of feeding the infra-analyzer with samples (about 400 g of pellets per sample). Each sample of pellets (i.e.

uncoated and coated pellets provided by Aqualia, plus extruded pellets coated using the pilot vacuum coater machine described below) was measured in duplicate and, for each repetition, two measurements were made. Then, the average NIR spectrum was calculated to deliver moisture, fat, and protein contents of the analyzed pellets. An Opus Lab software (Billerica, United States) was used to communicate with the NIR apparatus during analysis and to provide results.

Results obtained for moisture, protein and fat contents were then compared with those originating from ISO 665:2000, ISO 5983−1:2005 and ISO 659:2009 standards, respectively, for verification, in the case of the coated pellets provided by Aqualia.

2.2.5. Bulk density

The pellet bulk density was measured by filling the pellets in a measuring cylinder of known volume. A 1 L transparent cylinder was used to determine the bulk density, for the uncoated pellets and also for the coated ones provided by Aqualia. The pellet density was measured in triplicate by determining the mass per unit volume of the sample. The results were expressed in g/L (i.e. mass per volume) as mean values ± standard deviation.

2.3. Differential scanning calorimetry (DSC) measurements

Test samples were obtained from uncoated and coated pellets provided by Aqualia through a crushing step using a Nahita (Chapelle-Sur-Erdre, France) agate mortar with a 50 mm pestle. This operation was used to reduce the pellet size to be able to put samples inside the aluminum pans dedicated to such analysis. Samples were then analyzed with a Mettler Toledo (Columbus-Ohio, United States) DSC1 calorimeter under a constant flow of dry nitrogen. Two different values were determined: the starch gelatinization rate, and the glass transition temperature of gelatinized starch.

Firstly, about 10 mg sample was inserted inside a 100 μL hermetic steel sample pan. Then, 50 μL of Milli-Q water were added to samples into pans. A heating ramp at 5 °C/min from 5 °C to 100 °C was performed. The starch gelatinization rate in pellets was determined for each sample. It was also determined from the starting solid mixture.

Secondly, about 15 mg sample was inserted inside a 40 μL hermetic aluminum sample pan and was heated from 0 °C to 200 °C at a 10 °C/min heating ramp. The glass transition (T_g) of plasticized starch inside the analyzed pellet was determined.

2.4. Fatty acid composition of oils

Gas Chromatography (GC) was used to determine the fatty acid composition of oils. Oil extraction from commercial coated pellets provided by Aqualia (before and after the oil leakage test) was carried out by one cyclohexane Soxhlet extraction. Each sample was prepared as follows: 20 mg of oil was mixed with 1 mL TBME (tert-butyl methyl ether). In an insert, 100 μL of the previous solution was added to 50 μL TMSH (trimethylsulphonium hydroxide), 0.2 M in methanol. The whole mixture was gently stirred and analyzed by a Varian 3900 gaz chromatograph equipped with a FID detector (Varian) and a CP Select CB (Varian, USA) fused silica capillary column (50 m length, 0.25 mm internal diameter, and 0.25 μm film thickness). Temperature profile during the analysis was chosen as follows: 185 °C for 40 min, then 15 °C/min to 250 °C, and 250 °C for 10.7 min (for a 55.0 min total analysis duration). Temperatures of the injector and the detector were kept at 250 °C, and helium with a flow rate of 1.2 mL/min was used as the carrier gas. After analysis, the identification of fatty acids was conducted by comparison of their retention times with commercial standards (Supelco 37 Component FAME Mix, USA). The GC analysis for fatty acid relative composition was performed in duplicate for each sample. The results were expressed as mean values ± standard deviation.

2.5. Rheological analysis of oils

The rheological behavior of oils (RAO, FO1, FO2, and OCM) was evaluated using a Rheometer MCR 302 (Anton Paar, Graz, Austria), with a CP50−2 cone-plate geometry of 50 mm diameter and 2° angle. Tests were conducted in the rotation mode with 1 mL of oil sample deposed uniformly. The viscosity variation of each oil was determined at 20 °C, 40 °C, and 60 °C (three measurement repetitions per temperature). The minimum temperature of 20 °C for viscosity measurement was chosen because it is a storage temperature for coated pellets very frequently observed in fish farms during summer season. The rheoCompass software (Graz, Austria) was used for data acquisition. The results were expressed as mean values ± standard deviation.

2.6. Morphological characterization of pellets through tomography

Morphology of pellets, the internal structure (porosity), and oil distribution were characterized by tomography thanks to a RX Solutions Easy Tom (Chavanod, France) 3D X-ray laboratory tomography device. A 93 kV tension and a 282 μA intensity were the acquisition parameters chosen. Each pellet was positioned on a rotation stage. At each angular step of 0.25°, about 1,300 projections of transmitted X-ray intensity field were recorded through an X-ray detector and using a flat panel detector (1,920 × 1,536 pixels). Using a filtered back-projection algorithm, it was then possible to reconstruct a volume image from all radiographies, reflecting the variations of the linear attenuation coefficient in the sample with a spatial resolution of 8 μm.

2.7. Coating of pellets under vacuum

A Stolz (Paris, France) MRSV 100 pilot vacuum coater machine was used for the vacuum coating experiments. Before starting this study, some technical parameters were optimized. The flow meter accuracy was checked to ensure that it provided the required oil quantity. The most appropriate nozzle (i.e. diameter, length, exit angle, and flow rate) was also chosen to guarantee an efficient spraying system (APSIS 15/40). Thanks to this optimization, all pellets were exposed to sprayed liquid uniformly. Firstly, pellets and oils (RAO and FO1) were weighed using an OHAUS (USA) CD-11 digital floor balance. Here, the vacuum coating conditions used in industry (i.e. “vacuum coating on line”, right after the extrusion process) could not be mimicked perfectly. Indeed, the extruded pellets were coated fifteen days after their production through extrusion. They were cold (20 °C) and dry, and therefore not softened. Starch retrogradation was not to be excluded either. All these reasons could presumably affect negatively the efficiency of vacuum coating (unfavorable condition for oil penetration inside pellets).

Then, extruded pellets were loaded inside the vacuum coater tank, and the oil mixture was placed in the oil tank with an adequate volume of the oily blend associating RAO and FO1 oils in the right proportions (i.e. 70/30 (w/w)). Then, the oil mixture was heated to 40 ± 2 °C. As RAO and FO1 oils revealed both low and very close viscosities at 40 °C (Fig. 2), their homogeneous mixing was facilitated. The oil tank was pressurized up to 3 bars, and the vacuum was generated inside the mixing vessel using a Busch (Gerlingen, Germany) Mink MM 1104 BV 1.3 kW vacuum pump. After that, the oil blend was gradually added through the spray nozzle located at the top of the coater tank. Its injection was favored, due to its low viscosity at 40 °C (Fig. 2). Thanks to this spray, a proper dispersion was realized onto the feed while the two mixing blades were rotating. For all vacuum coating experiments, the proportion between extruded pellets and the OCM oil blend was 76.7/23.3 (w/w) to reach the required energy content of the coated pellets (i.e. 30 % (w/w) fat content). After complete oil distribution onto the surface of pellets, the pressure inside the coater was controlled back to atmospheric pressure. At the end of the coating operation, the overturning of the coater enabled the pellet discharge. A 3 kg representative sample was collected from each coating condition tested. Pellets were

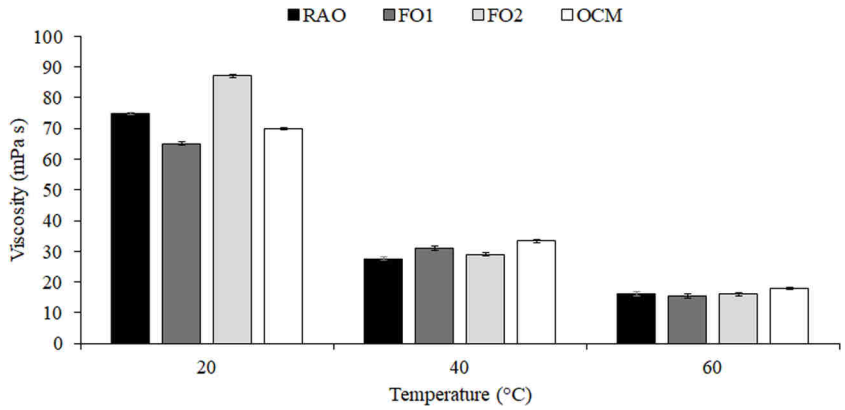

Fig. 2. Viscosity of oils at three different temperatures (20 °C, 40 °C, and 60 °C).

then stored at 5 °C to avoid their possible evolution over time before any further characterization. Although not determined at 5 °C, viscosity of oils was high enough at this temperature to avoid their leakage during storage. In particular, the oil leakage was measured from commercial coated pellets provided by Aqualia and stored at 5 °C, and the oil loss after two weeks storage was considered as negligible (less than 0.5 % (w/w) in proportion to the initial weight of fat inside pellets), presumably due to much higher viscosity of oils at this temperature. For all the experiments, fat content inside the coated pellets was measured by NIR to verify that it was always around 30 % (w/w).

2.8. Study of the influence of coating parameters

2.8.1. Experimental design

To study the influence of the coating parameters on oil leakage, sixteen coating experiments were conducted with the help of an experimental design having the form of a three-variable Doelhlert's matrix (Table 1). For those experiments, the coating parameters varied as follows: a stirring speed from 45 to 85 Hz, a pressure in the coater from 140 to 310 mbars, and a filling rate of the coater from 35 to 55 % in volume.

Statistical analysis of the results was carried out using the Nemrodw software (Marseille, France), which was also used to plot the isoresponse curves.

The best-fit second-order response (Y) obtained for each measured answer (essentially the usage properties of coated pellets) is given in Eq. (1).

$$\begin{aligned}\mathbf{Y} = {} & \mathbf{a_0} + (\mathbf{a_1} \times \mathbf{X_1}) + (\mathbf{a_2} \times \mathbf{X_2}) + (\mathbf{a_3} \times \mathbf{X_3}) \\ & + (\mathbf{a_{11}} \times \mathbf{X_1} \times \mathbf{X_1}) + (\mathbf{a_{22}} \times \mathbf{X_2} \times \mathbf{X_2}) + (\mathbf{a_{33}} \times \mathbf{X_3} \times \mathbf{X_3}) \\ & + (\mathbf{a_{12}} \times \mathbf{X_1} \times \mathbf{X_2}) + (\mathbf{a_{13}} \times \mathbf{X_1} \times \mathbf{X_3}) + (\mathbf{a_{23}} \times \mathbf{X_2} \times \mathbf{X_3})\end{aligned} \quad (1)$$

X_1, X_2, and X_3 correspond to the coding values of the experimental design, each varying from -1.0 to 1.0, and relating to the three tested coating parameters (i.e. the stirring speed, the pressure in the coater and the filling rate, respectively). a_i (i varying from 0 to 3), and a_{ij} (i and j varying from 1 to 3, and $i \leq j$) are the coefficients of the polynomial model.

2.8.2. Time to restore atmospheric pressure

In a second time, a fourth process parameter, the time to restore the atmospheric pressure after coating, was also studied. It was tested using a 65 Hz stirring speed, a 140 mbars pressure in the coater, and a 45 % in volume filling rate. For the experimental design, this time was set at 120 s. In that case, it was set at five different values (i.e. 60 s, 75 s, 90 s, 105 s, and 120 s).

2.9. Usage properties of coated pellets

2.9.1. Oil leakage rate (OLR)

Pellets OLR was evaluated using an unstandardized test developed for the purpose of the study, at four different experimental conditions: T1 (climatic chamber, 60 % relative humidity (RH), 20 °C), T2 (ventilated oven, 40 °C), T3 (water bath, 60 °C), and T4 (ventilated oven, 60 °C). T1 corresponded to the controlled storage of the coated pellets at room temperature. On the contrary, T2 to T4 consisted of simulating increasingly extreme conditions like the storage of the coated pellets in silos in direct sunlight during the summer.

For each tested condition, 130.0 ± 0.1 g of pellets were weighted. Besides, about 2 g of absorbent paper discs were put at the bottom of a 250 mL glass beaker, and the pellets were added inside.

After 24 h, the beakers were recovered and cooled at ambient tem-

Table 1
Process parameters of the sixteen coating experiments (Doehlert's experimental design).

Coating experiment number	X1	Stirring speed (Hz)	X2	Pressure in the coater (mbars)	X3	Filling rate (% in volume)
1	1.000	85	0.000	225.0	0.000	45.0
2	−1.000	45	0.000	225.0	0.000	45.0
3	0.500	75	0.866	298.6	0.000	45.0
4	−0.500	55	−0.866	151.4	0.000	45.0
5	0.500	75	−0.866	151.4	0.000	45.0
6	−0.500	55	0.866	298.6	0.000	45.0
7	0.500	75	0.289	249.6	0.816	53.2
8	−0.500	55	−0.289	200.4	−0.816	36.8
9	0.500	75	−0.289	200.4	−0.816	36.8
10	0.000	65	0.577	274.0	−0.816	36.8
11	−0.500	55	0.289	249.6	0.816	53.2
12	0.000	65	−0.577	176.0	0.816	53.2
13	0.000	65	0.000	225.0	0.000	45.0
14	0.000	65	0.000	225.0	0.000	45.0
15	0.000	65	0.000	225.0	0.000	45.0
16	0.000	65	0.000	225.0	0.000	45.0

perature when necessary (for T2, T3, and T4 conditions). The oil-soaked paper and pellets were separated. As part of the leaked oil could still be present on the pellet surface, the surface of all the pellets was then wiped off cautiously with the paper to ensure the complete absorption of leaked oil. Lastly, the oil-soaked paper and pellets were weighed, and the OLR value was determined according to Eq. (2). For each tested condition, experiments were made in triplicate. The results were expressed as mean values ± standard deviations.

$$\text{OLR } (\%) = \frac{\text{Weight of fat absorbed by the absorbent paper (g)}}{\text{Total weight of pellets before leakage (g)}} \times 100 \quad (2)$$

When expressed in proportion to the weight of fat inside pellets before leakage, the oil leakage rate was noted OLR', the latter possibly varying from 0% to 100 % (which was not the case for OLR). OLR' value was determined according to Eq. (3).

$$\text{OLR' } (\%) = \frac{\text{Weight of fat absorbed by the absorbent paper (g)}}{\text{Weight of fat inside pellets before leakage (g)}} \times 100 \quad (3)$$

To better visualize the effect of applied pressure on oil leakage phenomena, ΔOLR (%) was calculated, and it is defined as shown in Eq. (4). It represents the decrease (in relative value) of OLR as a function of the applied pressure in the coater (compared to the OLR value at P_{max} maximal pressure), for the four conditions of oil release test temperature.

$$\Delta\text{OLR } (\%) = \frac{(OLR\ (Pmax) - OLR\ )}{OLR\ (Pmax)} \times 100 \quad (4)$$

2.9.2. Durability

350 g of coated pellets were initially sieved with 1.0 mm and 7.1 mm screens. After sieving, the material collected on each screen was weighed. Dust was the quantity of material collected under the finest screen. The broken pellets were the quantity of material between the two tested screens. For all realized tests, these two fractions represented less than 0.1 % (w/w) of the test sample mass. This was thus considered as negligible. The preserved pellets on the upper screen were then introduced on a Doris (Durability on a Realistic Test) tester from AKVA Group (Klepp, Norway). Their mass corresponded to the initial weight of tested pellets in Eq. (5). The tester is designed to mimic the pellet degradation during pneumatic feeding (Aas et al., 2011). It consists of an Archimede screw conveying pellets from the inlet to the outlet of the tester. At the end of the test, pellets were collected in a cup, and they were sieved with a 7.1 mm screen. After sieving, the weight of preserved pellets on the top of this screen was determined. The durability result was calculated according to Eq. (5). Each pellet type was analyzed in triplicate. The results were expressed as mean values ± standard deviations.

$$\text{Durability } (\%) = \frac{\text{Weight of preserved pellets on the upper screen after sieving}}{\text{Initial weight of tested pellets}} \times 100 \quad (5)$$

2.9.3. Resistance to compression or hardness

The resistance to compression of pellets was measured thanks to an Instron 33R4204 (Norwood-Massachusettes, United States) universal testing machine fitted with a 5 kN load cell. Uncoated and coated pellets were analyzed and, for each condition, pellets (one by one) were positioned between two rigid plates. The compression test was conducted using a crosshead speed of 2 mm/min. During the test, the maximum force (F) obtained for each analyzed pellet was registered. In order to take into consideration the dimensional variations of pellets, hardness (H) was expressed as the ratio of the maximum force (F) divided by the average diameter of the analyzed pellet (d). The reported values of hardness were the averages of eighteen replications.

2.9.4. Floatability

A large glass beaker (18.5 cm in diameter) was filled with 4 L of fresh water. Fifty pellets were counted in a small plastic shovel, and they were then thrown into the water beaker softly. Floating pellets were counted after 30 s. Depending on the obtained floatability value, samples were classified as follows: less than 30 % (sinking pellets), between 30 % and 70 % (semi-floating pellets), and over 70 % (floating pellets). For each pellet type, this test was conducted in triplicate. The results were expressed as mean values ± standard deviations. The water was changed at the end of each replication.

2.10. Statistical analyses

Data are presented as mean values ± standard deviations for triplicate experiments, with the exception of the hardness measurement which was carried out eighteen times. Statistical analyses were performed with XLSTAT software (Addinsoft, Bordeaux, France). One-way ANOVA test was used to assess the significance of differences for each variable at $P < 0.10$. The multiple comparison procedure used Tukey's test.

3. Results and discussion

3.1. Characterization of extruded pellets

As an introduction, the determination of fat content in coated pellets was consistent between the two methods used, i.e. the multi-stage Soxhlet extraction procedure and NIR measurement. For commercial coated pellets (9 mm in diameter) from Aqualia company, fat content was 31.52 ± 1.31 % and 31.47 ± 1.42 %, respectively, for these two analytical methods. Faster to implement, NIR was thus chosen as a routine tool in this study to check the final fat content after coating experiments. Otherwise, the Soxhlet protocol was useful to analyze the lipid fractions contained in pellets and in the soaked paper after the oil leakage test. In the same way, NIR measurements for moisture and protein contents were also consistent with those obtained from ISO standards: 5.15 ± 0.06 % and 5.04 ± 0.05 %, respectively, for moisture content, and 36.60 ± 0.41 % and 36.68 ± 0.51 %, respectively, for protein content.

Table 2 summarizes the most important physicochemical characterizations carried out on uncoated and coated pellets provided by Aqualia. Uncoated pellets initially contained about 50.4 % proteins (w/w) and 5.8 % fat (w/w), the latter originating naturally from the raw

Table 2
Characterization of uncoated and coated pellets provided by Aqualia (contents of chemicals are expressed in % of the fresh matter).

Characteristic	Uncoated pellets	Coated pellets
Humidity (%)	6.80 ± 0.04	5.04 ± 0.05
Fat (%)	5.79 ± 0.07	31.52 ± 1.31
Protein (%)	50.35 ± 0.60	36.68 ± 0.51
Bulk density (g/L)	452.4 ± 2.8	580.5 ± 8.1

Results in the table correspond to the mean values ± standard deviations.

materials used in their formulation, especially soybean meal, fishmeal, and FO2. During coating, the addition of oil blend (RAO and FO1) raised the fat content of final pellets to reach 31.5 % (w/w). In parallel, the protein content inside coated pellets decreased to 36.7 %. The density of uncoated pellets was also determined to adjust the filling rate (in volume) of the pilot vacuum coater for every coating condition tested. It was 452 g/L. Subsequently, the determination of batch weight needed to be loaded into the coater for each trial could be determined. The density of coated pellets provided by Aqualia was higher (580 g/L) than that of the uncoated ones, due to the fat addition. The characterization of coated pellets obtained in this study, i.e. using the pilot vacuum coater, will be discussed in the section dedicated to the study of the influence of the vacuum coating process parameters (see paragraph 3.6).

3.2. Thermal analysis of extruded pellets

The DSC analysis of uncoated and coated pellets from Aqualia company was conducted to characterize the starch gelatinization rate and the glass transition temperature (T_g) of amorphized starch.

3.2.1. Starch gelatinization rate

No thermal phenomenon was observed for both samples. This result clearly indicates that the starch gelatinization was complete during the pellet manufacture. On the contrary, when conducted from the starting solid mixture, DSC revealed a specific endothermic peak, from 55 °C to 80 °C, which is characteristic of starch gelatinization. In the fish feed process, the raw materials (starch, proteins, etc.) are hydrated and heated firstly in the pre-conditioner (pre-cooking in the presence of water, in its vapor and liquid forms), where starch loses crystallinity and starts to gelatinize. The mixture passes then into the twin-screw extruder, where the thermal treatment is completed with intensive shear stress. The heat generated, in conjunction with pressure and moisture, achieves cooking, protein denaturation, and starch gelatinization (Riaz and Rokey, 2011).

3.2.2. Glass transition temperature (T_g) of amorphized starch

T_g of amorphized starch inside uncoated and coated pellets was determined using the midpoint temperature of the baseline shift observed on the thermograms. The starch glass transition temperature was approximately equal for both analyzed pellets (i.e. 61 °C), meaning that the oil has no plasticizing effect on starch.

In the literature, this temperature varies for different starch types, especially due to the variation in the amylose/amylopectin ratio according the starch origin. For example, the glass transition of equilibrated wheat starch takes place at 61 °C (Riaz and Rokey, 2011) or, in the case of corn starches, it varies between 52 and 60 °C for samples containing around 13 % moisture (Liu et al., 2010). This matched perfectly the obtained results as the starch contained in pellets comes predominantly from wheat. In addition, because water acts as a plasticizer for starch (Rouilly and Rigal, 2002), T_g value can also be modified according to the relative humidity of the environment where pellets are stored: the more moisture, the lower the T_g. In consequence, when starch glass transition occurs (from a glassy to a rubbery state), the structure of the whole material is modified. Subsequently, it may affect the pellets cohesion and thus their ability to properly retain oil over time.

3.3. Fatty acid composition of released oil

Table 3 shows the fatty acid composition of all fats analyzed through GC. First, individual oils (RAO, FO1, and FO2) were analyzed. Then, they were compared to the other oil fractions: oil inside commercial coated pellets from Aqualia, before (PBOL) and after (PAOL) oil leakage test, and oil lost during that test (OABP). Here, the oil leakage test was conducted during 24 h, at 60 °C in a water bath (T3 experimental condition). This test resulted in 3.4 ± 0.1 % and 10.8 ± 0.4 % OLR and OLR' values, respectively. As the oil partly leaked from the commercial coated pellets during the test, their chemical composition (Table 2) has evolved. After the test, their moisture and protein contents increased to 5.37 ± 0.04 % and 38.36 ± 0.44 %, respectively, and their fat content decreased to 28.92 ± 0.37 % (contents expressed in % of the fresh matter).

RAO was particularly rich in oleic acid (60 %) and, in a smaller amount, in linoleic acid (19 %), whereas FO1 and FO2 fish oils revealed large amounts of eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA) (13 % and 8–10 %, respectively). Those results were in perfect

Table 3
Fatty acid composition of oils (%).

Fatty acid (biochemical nomenclature / name)		RAO			FO1			FO2			PBOL			PAOL			OABP		
C14:0	Myristic acid	n.i.			6.2	±	0.0	6.3	±	0.0	1.4	±	0.0	1.4	±	0.0	1.4	±	0.0
C16:0	Palmitic acid	4.6	±	0.0	21.1	±	0.0	22.7	±	0.1	9.0	±	0.0	9.0	±	0.1	9.0	±	0.0
C18:0	Stearic acid	1.7	±	0.0	5.7	±	0.0	5.9	±	0.0	2.8	±	0.1	2.8	±	0.1	2.8	±	0.0
C20:0	Arachidic acid	0.5	±	0.0	0.6	±	0.0	0.4	±	0.0	0.5	±	0.0	0.5	±	0.0	0.5	±	0.0
C22:0	Behenic acid	0.8	±	0.0	n.i.			n.i.			0.8	±	0.1	0.8	±	0.0	0.8	±	0.0
Total saturated fatty acids (SFA)		7.5	±	0.0	33.7	±	0.1	35.4	±	0.1	14.4	±	0.1	14.3	±	0.1	14.5	±	0.0
C16:1n-7	Palmitoleic acid	0.2	±	0.0	7.2	±	0.0	7.3	±	0.0	1.6	±	0.0	1.6	±	0.0	1.5	±	0.0
C18:1n-9	Oleic acid	60.1	±	0.0	20.7	±	0.0	18.4	±	0.1	49.4	±	0.2	49.0	±	0.3	50.3	±	0.1
C18:1n-7c	trans-Vaccenic acid	3.1	±	0.0	3.8	±	0.0	3.6	±	0.0	2.8	±	0.2	2.9	±	0.1	3.0	±	0.2
C20:1n-9	Gondoic acid	1.1	±	0.0	1.8	±	0.0	2.2	±	0.0	1.8	±	0.0	1.8	±	0.0	1.8	±	0.0
C22:1n-11	Erucic acid	n.i.			1.2	±	0.0	1.7	±	0.0	0.7	±	0.0	0.7	±	0.0	0.7	±	0.0
Total monounsaturated fatty acids (MUFA)		64.6	±	0.0	34.6	±	0.0	33.2	±	2.1	56.4	±	0.4	56.1	±	0.3	57.4	±	0.1
C18:2n-6	Linoleic acid	19.3	±	0.0	4.6	±	0.0	2.7	±	0.0	17.1	±	0.0	17.0	±	0.1	16.5	±	0.1
C18:3n-3	Linolenic acid	8.6	±	0.0	1.4	±	0.0	1.0	±	0.0	6.3	±	0.0	6.3	±	0.0	6.2	±	0.0
C18:4n-3	Stearidonic acid	n.i.			1.9	±	0.0	2.0	±	0.0	0.4	±	0.0	0.4	±	0.0	0.4	±	0.0
C20:4n-6	Eicosatetraenoic acid	n.i.			0.9	±	0.0	1.0	±	0.0	0.1	±	0.2	0.2	±	0.2	0.1	±	0.1
C20:5n-3	Eicosapentaenoic acid (EPA)	n.i.			13.2	±	0.1	12.6	±	0.1	1.7	±	0.0	1.8	±	0.0	1.6	±	0.0
C22:5n-3	Docosapentaenoic acid (DPA)	n.i.			1.7	±	0.1	1.7	±	0.0	0.3	±	0.2	0.4	±	0.2	0.4	±	0.0
C22:6n-3	Docosahexaenoic acid (DHA)	n.i.			7.9	±	0.1	10.4	±	0.1	3.3	±	0.0	3.4	±	0.1	3.0	±	0.0
Total polyunsaturated fatty acids (PUFA)		27.9	±	0.0	31.7	±	0.0	31.4	±	0.1	29.1	±	0.2	29.6	±	0.2	28.1	±	0.1

Results in the table correspond to the mean values ± standard deviations.
n.i., not identified.
RAO, crude rapeseed oil; FO1 and FO2, fish oils; PBOL, oil inside commercial coated pellets from Aqualia before oil leakage; PAOL; residual oil inside commercial coated pellets from Aqualia after oil leakage; OABP, oil lost during the oil leakage test conducted from the commercial coated pellets from Aqualia.
The oil leakage test conducted for the generation of PAOL and OABP oil fractions was as follows: 60 °C temperature in a water bath during 24 h.

accordance with the literature data (Miller et al., 2010; National Research Council, 2011). Also, PBOL revealed without surprise a fatty acid profile which fitted quite well with the distribution between RAO and FO1 inside the oil mixture (70/30 (w/w)), and FO2 added during extrusion (2% (w/w) in proportion to the flour), with 49 % oleic acid, 17 % linoleic acid, 2% EPA, and 3% DHA.

The fatty acid composition of released oil (OABP) extracted from the absorbent paper used during the oil leakage test revealed the same distribution in fatty acids as for the oil inside coated pellets, whether before (PBOL) and also after (PAOL) the leakage test. Indeed, for these three oils, saturated fatty acids (SFA) represented 14.4–14.5 % of total fatty acids, with palmitic acid as the main one (9.0 %). Monounsaturated fatty acids (MUFA) represented 56.1–57.4 % of total fatty acids, oleic acid being the most represented monounsaturated fatty acid (49.0–50.3 %) in these three cases. Lastly, the content in polyunsaturated fatty acids (PUFA) was quite similar (from 28.1%–29.6%), with 16.5–17.1 % linoleic acid.

As the same distribution of fatty acids was observed for the pellet fat fraction before and after oil leakage, no preferential release of one of the three oils added to the fish feed, whether during twin-screw extrusion (FO2) and at coating (RAO and FO1), was observed.

Given the high temperature (40 °C) and pressure involved during vacuum coating, oxidative damage to the lipid fractions could occur. For future work, it would thus be useful to assess peroxidation characteristics (i.e. peroxide value) of oils before and after coating, which could provide pertinent information on possible biological impacts.

3.4. Oil rheology

The rheology of RAO, FO1, FO2, and OCM oils was studied at 20 °C, 40 °C, and 60 °C (Fig. 2) to understand more about the oil leakage observed for fish feed pellets. They all revealed a Newtonian behavior.

Oil viscosity is very sensitive to temperature. At 20 °C, OCM viscosity value (70 mPa. s) was situated between RAO (75 mPa. s) and FO1 (65 mPa. s) ones, as it was a blend of these two oils. Even if FO1 and FO2 were two fish oils, they revealed large differences in their viscosity values at 20 °C. In fact, FO2 was much more viscous than FO1 (i.e. 87 mPa.s instead of 65 mPa.s). This difference might be due to their slightly different fatty acid compositions, especially DHA (C22:6n-3). According to Table 3, FO2 contained 10.4 % of DHA, instead of 7.9 % only for FO1. One other reason could be also the difference in their contents in saturated fatty acids (SFA), especially palmitic acid, with 33.7 % total SFA for FO1 and 35.4 % for FO2. Fish oils are largely used in aquaculture and for human health as they are a primary source in providing health beneficial long-chain polyunsaturated fatty acids, especially omega-3 fatty acids (e.g. EPA and DHA) (National Research Council, 2011). All oil viscosities were much lower and closer one from another at a higher temperature (40 °C and especially 60 °C): around 30 mPa.s and 16 mPa. s, respectively. This is the reason why the oil leakage phenomenon is favored at higher temperature for the three oils, thus explaining also the previously mentioned non-selectivity of oil leakage between RAO, FO1, and FO2 oils.

Reducing oil leakage in coated pellets is a key issue for fish feed producers. The temperatures to which coated pellets may be exposed from their manufacture to their distribution to fish may affect their quality, especially their ability to retain oil over time. High temperatures (around 60 °C) affect the structure of the pellet (softened material), due to the starch glass transition that can occur in such conditions, thus resulting in pellet softening and lower oil viscosity. These combined effects thus facilitate the observed oil leakage.

3.5. Morphological characterization of pellets

Fig. 3 shows the tomographic reconstructions of the uncoated pellets, and those produced from the pilot vacuum coater. The starchy matrix appeared in light grey and white, and empty pores in black. The oil was also visible inside the coated pellets (b and c), appearing in dark grey (i. e. slightly darker than the starchy matrix). In general, the oil filled well the porosities near the surface. At 151 mbars (image c), the high applied vacuum level in the pilot vacuum coater favored undoubtedly a better oil penetration to the middle of the pellet compared to pellets produced with only a 299 mbars vacuum (image b). It is thus reasonable to assume that the lowering of the pressure in the coater favored the subsequent reduction in the oil leakage. The study of the influence of the vacuum coating process parameters on OLR presented in the next paragraph will try to validate this hypothesis.

3.6. Influence of the vacuum coating process parameters

3.6.1. Process parameters choice

To begin, sixteen coating experiments were carried out using the vacuum coater in different conditions, by varying the three following parameters: the stirring speed, the pressure in the coater, and the filling rate (in volume) of the coater tank (Table 1), as they were susceptible to have a significant influence on oil leakage. Thanks to preliminary work, their ranges of variation were chosen as follows: 45−85 Hz, 150–300 mbars, and 35–55 % in volume, respectively. Those choices took into account technical constraints of the pilot vacuum coater and some characteristics of the feed to be tested (especially its density). In parallel, the time to restore the atmospheric pressure after coating was always 120 s for those sixteen experiments.

Pandey (2018) reported that pressure in the coating vessel reduced down to 200 mbars was more efficient to remove air from the feed pores, leaving them as open cells. As the extruded feed tested in this study was strong enough, higher vacuum levels were tested. Thus, the pressure applied in the coater varied in this study from 140 to 310 mbars.

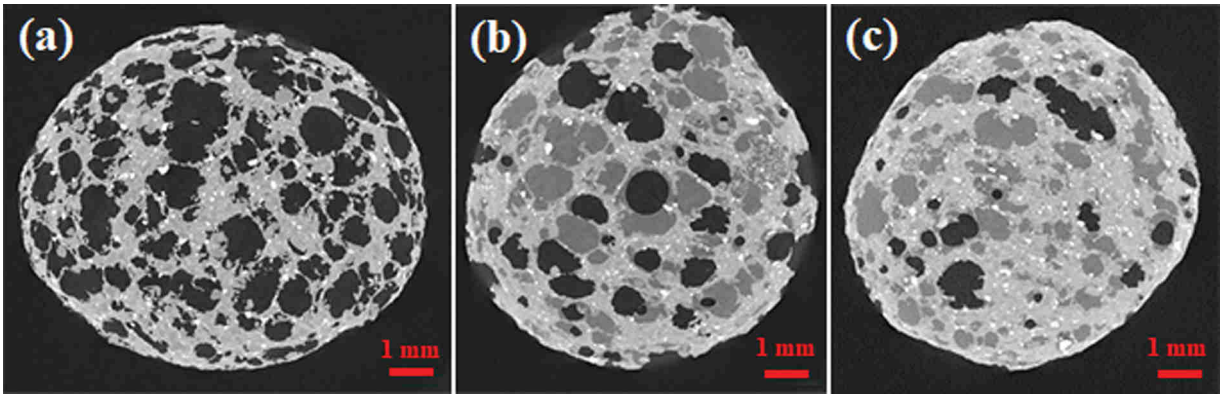

Fig. 3. Tomography images of fish feed pellets: (a) uncoated pellet, (b) coated pellet produced using a 299 mbars pressure in the pilot vacuum coater, and (c) coated pellet produced using a 151 mbars pressure in the pilot vacuum coater (55 Hz stirring speed and 45 % filling rate of the coater during vacuum coating).

Table 4
Chemical characterization of the coated pellets produced using the pilot vacuum coater from the sixteen coating conditions of the Doehlert's experimental design (% of the fresh matter).

Coating experiment number	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Humidity (%)	5.8 ± 0.1	5.8 ± 0.1	5.9 ± 0.1	5.9 ± 0.1	5.9 ± 0.2	5.6 ± 0.1	5.8 ± 0.1	5.6 ± 0.1	5.7 ± 0.1	5.7 ± 0.2	5.8 ± 0.1	5.7 ± 0.2	6.0 ± 0.3	5.5 ± 0.1	5.6 ± 0.1	5.4 ± 0.1
Fat (%)	29.6 ± 0.7	30.1 ± 0.3	29.3 ± 0.4	30.7 ± 0.3	29.6 ± 0.6	29.9 ± 0.3	29.7 ± 0.3	29.8 ± 0.2	30.0 ± 0.4	29.6 ± 0.2	29.9 ± 0.8	29.5 ± 0.4	30.6 ± 0.2	29.9 ± 0.2	30.4 ± 0.4	29.7 ± 0.5
Proteins (%)	36.7 ± 0.2	36.4 ± 0.3	37.2 ± 0.6	36.0 ± 0.2	36.6 ± 0.4	36.9 ± 0.7	37.4 ± 0.8	35.8 ± 0.4	36.8 ± 1.0	36.2 ± 0.1	37.5 ± 1.0	36.7 ± 0.3	35.9 ± 0.8	36.7 ± 0.6	36.2 ± 0.8	36.9 ± 0.1

Results in the table correspond to the mean values ± standard deviations.

Considering the limited oil tank capacity (10 kg) and the density of uncoated pellets (452 g/L, Table 2), the maximal filling rate of the coater tank to guarantee a 30 % (w/w) fat content after vacuum coating was 55 % (V/V). Finally, the stirring speed varied from 45 to 85 Hz to maintain the integrity of the pellets.

3.6.2. *Pellets composition*

Usually, the fish feed contains 10 % max of moisture once coated and dried (FAO, 2020). In this study, moisture content was a little lower (between 5.4 % and 5.9 %) (Table 4), presumably due to an excessive drying after extrusion or to their storage in a dry place. Besides, protein and fat contents were verified for the sixteen experiments thanks to NIR measurements. Protein and fat contents were 36.6 % (w/w) and 29.9 % (w/w) in average, respectively. The fat content of coated pellets was in perfect accordance with initial needs for every coating experiment conducted.

3.6.3. *Pellets characterization*

Table 5 provides the results of the characterization of coated pellets, i.e. OLR, OLR', durability, hardness, and floatability, for the different experimental design coating conditions. OLR was evaluated for four different incubation conditions (T1 to T4). This rate was always higher when temperature increased (from T1 to T3-T4). Indeed, at 20 °C (T1 condition), OLR varied between 0.6%–1.2% whereas, at 60 °C in the ventilated oven (T4 condition), it was between 3.8 % and 5.8 %. Comparing T3 and T4 conditions (both at 60 °C), the ventilated oven, closer to the real conditions of storage silos, favored the oil leakage in comparison with a water bath.

High temperatures (around 60 °C) may affect the structure of the pellet (softened material), due to the glass transition of starch that can occur in such conditions. At such temperature, the viscosity of all oils was also largely decreased (Fig. 2).

The coefficients of the best-fit second-order response for each usage property, and the corresponding correlation coefficient (R^2) are presented in Table 6. For OLR values measured at the four incubation conditions tested, a_2 coefficient (first-order coefficient relative to the coater pressure) was always positive and had a much higher absolute value than the two other first-order coefficients, relative to the stirring speed (i.e. a_1 coefficient) and to the filling rate of the coater (i.e. a_3 coefficient), respectively. In parallel, the R^2 correlation coefficient was at least 0.91, proving that the polynomial models represented in a satisfactory manner the oil leakage phenomenon for every tested incubation mode. The pressure in the coater was then the most influential parameter on OLR. As an example, the reading of the isoresponse curves (Fig. 4) for oil leakage at 60 % RH and 20 °C in climatic chamber (T1 incubation condition) perfectly illustrates the importance of the coater pressure on OLR variation, with a progressive reduction in the oil leakage phenomenon as the coater pressure was reduced (Fig. 4a and b).

The reduction of pressure in the coater allowed deeper penetration of oil in the pellet core during coating and lower oil leakage. The 3D X-ray tomography radiographs, taken from coated pellets originating from conditions number 4 (i.e. 151 mbars pressure) and number 6 (i.e. 299 mbars pressure), both using the same stirring speed (55 Hz) and the same filling rate (45 %), perfectly illustrate that tendency (Fig. 3).

Conversely, the stirring speed and the coater filling rate demonstrated a slighter effect on OLR. To better visualize the effect of pressure on OLR reduction, ΔOLR was calculated according to Eq. (4). Since stirring speed and filling rate were less influencing on OLR, they were chosen in that case in the middle of the experimental domain, i.e. 65 Hz stirring speed ($X_1 = 0$) and 45 % in volume filling rate ($X_3 = 0$). OLR could be halved at 20 °C (T1 condition) and 40 °C (T2 condition) when the coater pressure was minimal (Fig. 5), which indicated that a reduced pressure (140 mbars) could lead to the filling of almost all pores, and a significant reduction in the OLR value. This reduction was less significant for T3 and T4 conditions: -34 % and -38 %, respectively, at minimal coater pressure.

Such a finding may have interesting consequences on an industrial scale. Indeed, as the filling rate of the coater does not affect OLR, the optimization of the coating process will not disturb the industrial productivity of the vacuum coating operation. Thanks to this systematic study, 140 mbars pressure in the coater was also definitely identified as the optimal condition for an efficient coating and therefore for a reduced OLR.

An experimental study was conducted in parallel to this work during the year 2019 with the main objective to verify the real temperature values observed in a storage silo in real condition (fish farm located in Saint-Julien en Born, South West of France). Temperature sensors were positioned inside the silo, measuring continuously during a whole year the temperature of pellets in three feed storage silos oriented differently from the sun. The maximal temperature value recorded was 45 °C, reached during the hottest days of the year (summer 2019). This finding comforted the chosen experimental conditions to evaluate the oil leakage rate. As a reminder, at 45 °C, the glass transition temperature of the starch matrix is not reached. In addition, as the temperature inside silos can reach high values in summer, this would be necessary to analyze for future work the possible oxidative damage of the feeds occurring during their storage.

The durability of coated pellets from the different coating experiments was also determined (Table 5). Indeed, high nutrient dense diets have been developed for rainbow Trout since a long time, and reducing the waste production from these feeds has always been a major concern (Robert et al., 1993; Cho and Bureau, 1997). The polynomial model associated with durability revealed a satisfactory 0.89 correlation coefficient (Table 6). The positive a_2 coefficient showed that a reduced pressure in the coater during vacuum coating affected the pellet durability. When a high vacuum was applied during coating, the pellet was weakened and its durability was reduced. Good durability for such feed (i.e. extruded pellets, 9 mm in diameter) is defined by values exceeding strictly 90 % according to the requirements from Aqualia company. In fact, with reduced pressure (i.e. 151 mbars), durability was at its lowest values (i.e. 85–86 %). In parallel, the a_3 coefficient relative to the filling rate of the coater was negative, and its absolute value was much lower than that of the a_2 coefficient. Two observations can thus be deduced. On the one hand, the effect of the coater filling rate on pellet durability is

Table 5
OLR and OLR' oil leakage rates, durability, hardness, and floatability of the sixteen coated pellets obtained through the experimental design.

Coating experiment number	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
OLR (%) for the four tested conditions																
T1	$0.90^{cd} \pm 0.07$	$0.81^{cde} \pm 0.01$	$1.21^{a} \pm 0.06$	$0.67^{de} \pm 0.03$	$0.71^{de} \pm 0.07$	$1.04^{ab} \pm 0.04$	$0.88^{bcd} \pm 0.03$	$0.72^{de} \pm 0.09$	$0.62^{e} \pm 0.08$	$1.03^{ab} \pm 0.05$	$0.96^{bc} \pm 0.07$	$0.59^{e} \pm 0.08$	$0.69^{de} \pm 0.01$	$0.78^{cde} \pm 0.11$	$0.74^{de} \pm 0.11$	$0.71^{de} \pm 0.13$
T2	$1.61^{e} \pm 0.07$	$1.51^{fgh} \pm 0.06$	$2.70^{a} \pm 0.12$	$1.48^{fgh} \pm 0.03$	$1.32^{hi} \pm 0.03$	$2.43^{b} \pm 0.10$	$1.74^{de} \pm 0.04$	$1.42^{ghi} \pm 0.07$	$1.37^{hi} \pm 0.07$	$2.14^{c} \pm 0.03$	$1.94^{cd} \pm 0.08$	$1.20^{i} \pm 0.08$	$1.69^{ef} \pm 0.12$	$1.60^{efg} \pm 0.07$	$1.65^{efg} \pm 0.08$	$1.49^{fgh} \pm 0.09$
T3	$3.11^{ef} \pm 0.03$	$3.19^{de} \pm 0.00$	$4.08^{a} \pm 0.05$	$2.88^{gh} \pm 0.01$	$2.73^{i} \pm 0.01$	$4.03^{a} \pm 0.08$	$3.28^{d} \pm 0.01$	$3.18^{de} \pm 0.03$	$2.91^{gh} \pm 0.00$	$3.74^{b} \pm 0.06$	$3.46^{c} \pm 0.00$	$2.74^{hi} \pm 0.11$	$3.10^{ef} \pm 0.07$	$3.10^{ef} \pm 0.05$	$3.13^{de} \pm 0.03$	$2.96^{fg} \pm 0.04$
T4	$4.40^{e} \pm 0.02$	$4.65^{de} \pm 0.05$	$5.89^{a} \pm 0.03$	$4.01^{f} \pm 0.04$	$3.83^{fg} \pm 0.09$	$5.81^{a} \pm 0.09$	$4.49^{e} \pm 0.03$	$4.03^{f} \pm 0.15$	$3.78^{g} \pm 0.07$	$5.07^{b} \pm 0.07$	$4.92^{bc} \pm 0.09$	$3.47^{h} \pm 0.08$	$4.84^{cd} \pm 0.04$	$4.55^{e} \pm 0.04$	$4.47^{e} \pm 0.06$	$4.57^{e} \pm 0.03$
OLR' (%) for the four tested conditions																
T1	$3.04^{bc} \pm 0.23$	$2.68^{cde} \pm 0.03$	$4.13^{a} \pm 0.19$	$2.17^{e} \pm 0.11$	$2.40^{de} \pm 0.23$	$3.48^{ab} \pm 0.12$	$2.96^{bcd} \pm 0.11$	$2.42^{de} \pm 0.30$	$2.07^{e} \pm 0.26$	$3.49^{ab} \pm 0.18$	$3.19^{bc} \pm 0.24$	$2.00^{e} \pm 0.28$	$2.25^{de} \pm 0.04$	$2.59^{cde} \pm 0.36$	$2.43^{de} \pm 0.38$	$2.38^{de} \pm 0.43$
T2	$5.46^{ef} \pm 0.23$	$5.03^{fgh} \pm 0.19$	$9.20^{a} \pm 0.42$	$4.84^{fgh} \pm 0.09$	$4.47^{hi} \pm 0.11$	$8.11^{b} \pm 0.32$	$5.85^{de} \pm 0.14$	$4.77^{ghi} \pm 0.22$	$4.57^{hi} \pm 0.24$	$7.25^{c} \pm 0.09$	$6.47^{d} \pm 0.26$	$4.07^{i} \pm 0.29$	$5.53^{ef} \pm 0.39$	$5.35^{efg} \pm 0.22$	$5.42^{efg} \pm 0.25$	$5.02^{fgh} \pm 0.32$
T3	$10.52^{def} \pm 0.09$	$10.60^{def} \pm 0.00$	$13.92^{a} \pm 0.18$	$9.38^{i} \pm 0.02$	$9.21^{j} \pm 0.02$	$13.47^{a} \pm 0.28$	$11.03^{d} \pm 0.03$	$10.67^{de} \pm 0.09$	$9.70^{hi} \pm 0.01$	$12.64^{b} \pm 0.21$	$11.55^{c} \pm 0.01$	$9.29^{ij} \pm 0.36$	$10.12^{fgh} \pm 0.23$	$10.36^{efg} \pm 0.18$	$10.31^{efg} \pm 0.09$	$9.95^{gh} \pm 0.14$
T4	$14.88^{e} \pm 0.08$	$15.47^{d} \pm 0.18$	$20.07^{a} \pm 0.09$	$13.09^{fg} \pm 0.11$	$12.94^{fg} \pm 0.29$	$19.40^{a} \pm 0.29$	$15.12^{de} \pm 0.10$	$13.53^{f} \pm 0.51$	$12.60^{g} \pm 0.23$	$17.16^{b} \pm 0.22$	$16.46^{c} \pm 0.30$	$11.76^{h} \pm 0.26$	$15.81^{cd} \pm 0.13$	$15.21^{de} \pm 0.14$	$14.71^{e} \pm 0.19$	$15.36^{de} \pm 0.10$
Durability (%)	$88.3^{bc} \pm 1.5$	$89.8^{ab} \pm 0.5$	$89.7^{ab} \pm 0.9$	$85.6^{cd} \pm 0.1$	$85.4^{cd} \pm 2.3$	$90.8^{ab} \pm 0.6$	$90.6^{ab} \pm 0.6$	$91.0^{a} \pm 0.4$	$90.5^{ab} \pm 0.8$	$91.3^{a} \pm 0.6$	$89.8^{ab} \pm 1.2$	$90.2^{ab} \pm 0.6$	$90.3^{ab} \pm 0.8$	$91.6^{a} \pm 0.5$	$91.5^{a} \pm 0.5$	$91.0^{ab} \pm 0.4$
Hardness (N/mm)	$14.8^{cde} \pm 2.5$	$12.3^{e} \pm 2.4$	$17.4^{ab} \pm 3.9$	$13.1^{de} \pm 2.3$	$14.7^{cde} \pm 3.2$	$15.8^{bcd} \pm 2.9$	$15.6^{bcd} \pm 2.3$	$14.1^{cde} \pm 2.0$	$16.5^{abc} \pm 3.3$	$18.2^{a} \pm 3.6$	$17.3^{ab} \pm 3.5$	$17.5^{ab} \pm 3.5$	$16.8^{abc} \pm 2.9$	$17.0^{abc} \pm 4.8$	$17.6^{ab} \pm 3.1$	$18.6^{a} \pm 2.9$
Floatability (%)	$7.0^{de} \pm 1.0$	$24.0^{a} \pm 3.0$	$10.0^{cde} \pm 3.0$	$7.0^{de} \pm 1.0$	$10.6^{cde} \pm 4.0$	$23.0^{ab} \pm 1.0$	$4.5^{e} \pm 4.0$	$11.0^{cde} \pm 4.0$	$16.0^{c} \pm 0.0$	$23.0^{ab} \pm 4.0$	$11.0^{cde} \pm 1.0$	$7.0^{de} \pm 1.0$	$11.0^{cde} \pm 1.0$	$14.0^{cd} \pm 4.0$	$14.0^{cd} \pm 0.0$	$10.0^{cde} \pm 3.0$

Results in the table correspond to the mean values ± standard deviations.
Means in the same line with the same superscript letter (a–j) are not significantly different at $P < 0.10$.
OLR was measured for four different conditions after 24 h of incubation:T1: climatic chamber, 60 % RH, 20 °C; T2: ventilated oven, 40 °C; T3: water bath, 60 °C; T4: ventilated oven, 60 °C.
OLR, oil leakage rate in proportion to the total weight of pellets before leakage; OLR', oil leakage rate in proportion to the weight of fat inside pellets before leakage.

Table 6
Coefficients of the best-fit second-order response for each usage property of the coated pellets obtained, and corresponding correlation coefficient (R^2).

Coefficient	a_0	a_1	a_2	a_3	a_{12}	a_{13}	a_{23}	a_{11}	a_{22}	a_{33}	R^2
OLR (%) for the four tested conditions											
T1	0.730	0.026	0.288	0.012	0.075	−0.014	0.003	0.125	0.195	0.025	0.91
T2	1.608	0.008	0.704	−0.011	0.248	−0.180	0.125	−0.048	0.516	−0.076	0.98
T3	3.073	−0.089	0.733	−0.072	0.115	0.014	0.086	0.077	0.451	0.087	0.98
T4	4.610	−0.160	1.182	0.000	0.150	−0.163	0.219	−0.085	0.395	−0.553	0.97
Durability (%)	91.102	−0.510	2.108	−0.460	−0.521	0.957	−0.898	−2.060	−3.595	0.614	0.89
Hardness (N/mm)	17.508	1.091	1.484	0.212	−0.063	−2.376	−2.274	−4.002	−1.689	−0.040	0.95
Floatability (%)	12.250	−5.604	4.799	−5.619	−9.623	−3.638	−7.527	3.250	−0.528	−0.929	0.90

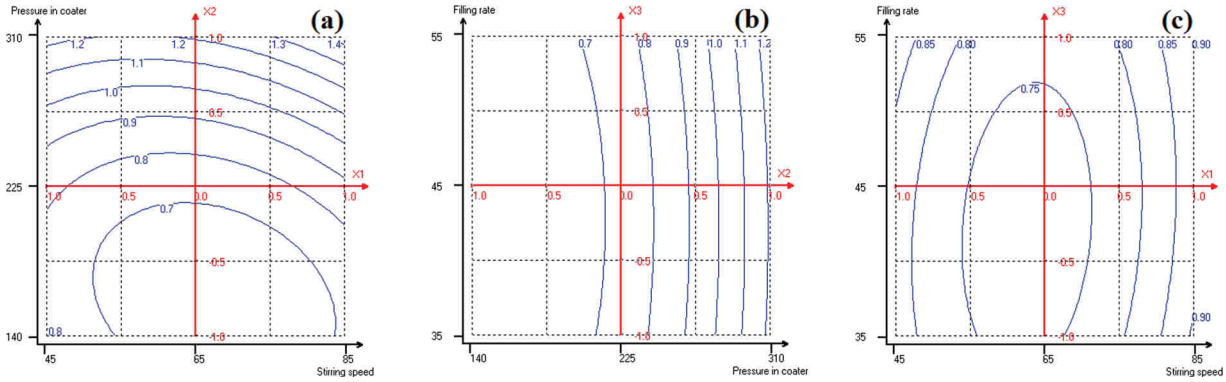

Fig. 4. Isoresponse curves for OLR at 60 % RH and 20 °C in a climatic chamber (T1 incubation condition), at a 45 % filling rate of the coater (a), at a 65 Hz stirring speed (b), and at a 225 mbars pressure applied in the coater (c).

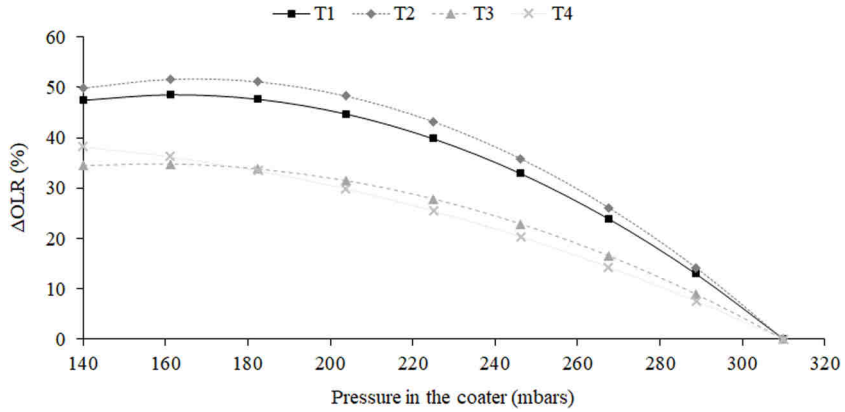

Fig. 5. Predicted reduction in OLR for the four incubation conditions tested, calculated from the corresponding polynomial models according to Eq. (4), at 65 Hz stirring speed and 45 % in volume filling rate.

rather limited in comparison with pressure. On the other hand, with higher filling rate values, the pellet durability would tend to slightly decrease. For such setting, the number of pellets inside the coater tank is higher, and the pellets' tendency to collide each other is therefore stronger. However, no more broken pellets were observed for the highest filling rate tested, and durability remained high (i.e. 90–91 %). This is proof that the influence of the coater filling degree was indeed negligible here. For the range of variation of the three vacuum coating parameters tested during this study, only the coater pressure had a predominant influence on durability.

There is a lack of standardization of equipment and measurement for this durability parameter (Sørensen et al., 2010), and these results must be discussed cautiously. In fact, in big and modern Trout farming, pellets are commonly transported to the cages using a pneumatic conveying system. That is why the measurement of durability tends to mimic the stresses applied to pellets during storage, transportation, and pneumatic feeding. Therefore, pellets should resist to these different solicitations (Pandey, 2018). In fact, extruded pellets are considered as brittle materials (Aarseth et al., 2006a), and are susceptible to generate fines and fractures upon handling. Aarseth (2004); Aarseth et al. (2006b) and Salman et al. (2002) have reported that during pneumatic conveying, especially for large pellet sizes (> 8 mm), collisions between the pellets and eventually with pipe walls may damage the pellets during their distribution. Generally, in extruded pelleted feed, two types of wear can occur: fragmentation and abrasion. The first involves the breakage of the extruded pellet into big particles and usually occurs during storage and transportation. On the other hand, abrasion involves the fracture on the edges of feed, generating fine particles from the pellet side. This wear is mainly caused by friction during transport and handling. Abrasion is considered as the most damaging type of wear from the nutritional and environmental point of view (Pandey, 2018). Aas et al. (2011) have also reported that the pneumatic feeding system was highly challenging, as it is also co-responsible for the creation of dust by abrasion and attrition of the feed pellets. Additionally, when oil leakage occurs, the combination

of free oil and dust inevitably leads to serious problems (claims, blocked pipes, and time wasted in cleaning). Besides, the generated dust and undersized particles have no feed value and, consequently, they are considered as feed loss and increase production cost (Aas et al., 2011).

These results should encourage fish feed producers to anticipate the possible reduction in the durability of their coated pellets when optimizing the vacuum coating conditions with the objective of a reduction in OLR. Because the high vacuum is necessary at coating for reducing the leakage phenomenon, it could thus be suggested to adapt also the operating conditions implemented during the twin-screw extrusion process to maintain durability as high as possible after coating. For future work, reducing the amount of FO2 fish oil added during twin-screw extrusion could be a solution to produce more durable pellets, thus ensuring their better holding during vacuum coating and possibly preserving a higher durability value for the end product. Indeed, even if adding FO2 fish oil in the twin-screw machine has a beneficial lubrication effect during the extrusion of the solid mixture in the die, decreasing the resulting specific mechanical energy, this addition also contributes to a reduction in the consistency of the mixture during its granulation, and therefore to a deterioration in the durability of the extruded pellets.

Considering resistance to compression, higher values were observed for coated pellets. For the uncoated ones, the mean value of hardness was about 11.0 ± 2.0 N/mm, which is lower than the measured values obtained for the totality of the coated pellets (Table 5). This result could be explained by the filling of some pellet pores by oils during coating, which may strengthen their internal structure. There was also a good correlation between hardness and durability for the different vacuum coating conditions tested. Indeed, a higher vacuum applied during coating reduced these two properties at the same time (positive value for the two a_2 coefficients), and the coated pellets thus became more brittle. As previously reported, because a physical exchange of air inside the feed pores occurs during vacuum coating, when a higher vacuum is applied, more air must be removed when vacuuming and then needs to go back inside the coater tank to restore the atmospheric pressure at the end of the coating cycle. It is thus reasonable to assume that this could cause more fractures within the pellets. The two other coating parameters also slightly influenced the pellet hardness. The positive values of a_1 and a_3 coefficients of the corresponding polynomial model revealed an improved hardness at higher stirring speed and, to a much lesser extent, at higher coater filling rate. In particular, even if the filling rate was expected to affect the mechanical constraint during stirring, its increase finally appeared as slightly positive on the pellet hardness.

Sørensen (2012) has reported that pellet hardness depends on several other parameters such as the degree of expansion, the used raw materials, and the processing parameters. Pillay and Kutty (2005) have demonstrated that pellet hardness also affects fish digestibility. According to Baeverfjord et al. (2006), hard pellet is more difficult to assimilate, and it can lead to fermentation and gas production in the fish stomach, which may result in inflammation and rupture. On the contrary, soft pellets may cause further complications like osmoregulatory stress and abdominal distension syndrome in rainbow Trout, due to the oil separation inside the stomach.

Fish feed pellets are, depending on the fish species, intended to either sink or float (Dethlefsen, 2017). Table 5 shows the results of floating properties for the sixteen coating trials. All floatability values were under 30 %, which indicated that all pellets sank, regardless of the vacuum coating conditions used. The associated polynomial model revealed a satisfactory correlation coefficient (i.e. 0.90), and the positive a_2 coefficient indicated that increased pressure in the vacuum coater tended to increase the pellet floatability (Table 5). On the contrary, the two other first-order coefficients (a_1 and a_3) were both negative, meaning that higher floatability could be obtained by reducing the stirring speed and the coater filling rate. Uncoated pellets (452 g/L bulk density) were manufactured as semi-floating ones. After vacuum coating, the addition of fat resulted in an increased pellet density while the pellet pores became filled with oil. Dethlefsen (2017) reported that, for semi-floating feed, the twin-screw extruder conditions helped to control this floatability parameter. Twin-screw extrusion is ideal to produce pellets with specific density. The mechanical shear applied to the solid mixture can be adjusted precisely through the extrusion parameters (e.g. the screw rotation speed, the number of holes at the die, their geometry, etc.). The amounts of liquid water and water vapor added during extrusion are also two other parameters to control expansion at the machine outlet. To guarantee uniformity in the specific pellet density within the same batch, it is above all essential that all the holes at the die have the same geometry. Their drying can thus be uniform, just as their coating.

Lastly, the time to restore the atmospheric pressure at the end of the vacuum coating process was also studied for its potential impact on OLR. Fig. 6 shows a decreasing trend of OLR when this time is increased from 60 s to 120 s, either at 20 °C (T1 incubation condition) or at 60 °C (T3 incubation condition). In other words, better oil penetration into the core of pellets was ensured when the time to restore the atmospheric pressure was longer (i.e. 120 s). In fact, when the oil addition sequence was completed under vacuum, the vacuum pressure was slowly released up to the atmospheric pressure (1.015 bars). This phenomenon created a pressure differential that forced the oil into the voids of the pellets. When the pressure equilibration was too fast, the oil film coating could break, allowing air back into the open pores (Bortone, 2006). This explained why OLR was reduced when the time to restore the atmospheric pressure was increased from 60 s to 120 s (as an example, -23 % at 20 °C), even if this could be problematic for productivity at an industrial scale.

To conclude, the most important finding of this study is that the control of pressure at coating is the major factor to decrease OLR, and 140 mbars pressure in the vacuum coater was identified as the optimal condition for an efficient coating and therefore for a greater reduction in OLR. These particularly promising results were obtained from cold pellets, vacuum coated fifteen days after being extruded. It is still interesting to note here that a reduction in OLR has also been observed at reduced pressure on the Aqualia industrial line for which the extruded pellets were still hot at vacuum coating (65 ± 2 °C), as this unit operation was conducted continuously after extrusion. Nonetheless, this reduction in OLR was found to be slightly lower at industrial scale (for T3 incubation condition, -27 % instead of -35 % at lab scale), due to a less intense vacuum (200 mbars instead of 140 mbars) and, for productivity reasons, to a faster time to restore atmospheric pressure (75 s instead of 120 s).

This study was successful also in proving that pellet integrity was practically conserved for all the experiments conducted with the experimental design. Pellets were subjected to various mechanical stresses during vacuum coating, especially depending on the filling degree used, resulting in a potential generation of fines. However, the amount of fines was generally negligible whatever the applied conditions. In aquaculture, the generation of fines can eventually lead to water pollution, and also reveal a negative effect on the oxygen supply, and filter capacity (All about feed, 2012).

4. Conclusion

The present paper aimed to provide fish feed producers technical data on the coating operation of high-fat pellets. Storage temperature increase favored oil leakage from the pellets, due to lower oil viscosity and pellet structure alteration when reaching the glass transition temperature of their starchy phase. One solution to solve this problem is to act at the process level. The results of the experimental design showed that the pressure inside the coater was the predominant parameter: a reduced applied pressure guaranteed deep penetration of oil inside the pellets, contributing to a reduction of more than 50 % in the oil leakage rate at 40 °C (from 2.8 % max to 1.4 % min for OLR, and from 9.5 % max to 4.7 % min for OLR'). However, the pellet durability was negatively impacted at the same time. The reduction in oil leakage rate was only

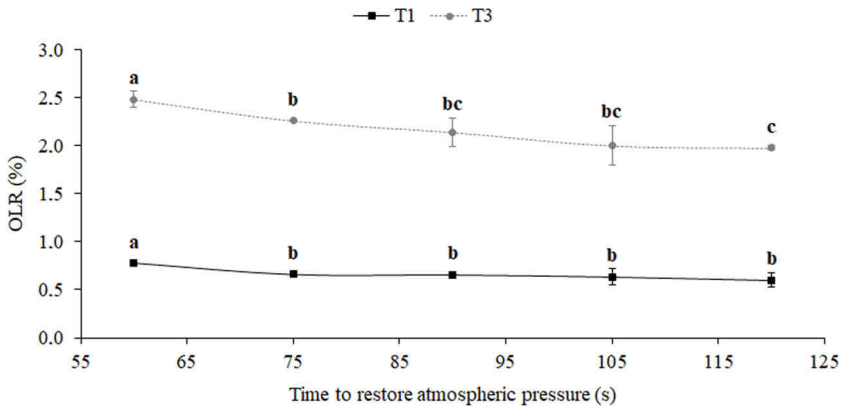

Fig. 6. Effect of time to restore the atmospheric pressure on OLR at 60 % RH and 20°C in a climatic chamber (T1 incubation condition), and at 60°C in a water bath (T3 incubation condition), for the next coating conditions: 65 Hz stirring speed, 140 mbars pressure in the coater, and 45 % in volume filling rate (means in the same curve with the same superscript letter (a–c) are not significantly different at $P < 0.10$).

partial and further work needs to be done, e.g. techniques to generate more expansion during twin-screw extrusion to enhance oil uptake while preserving high durability level, or to treat pellet surface after vacuum coating (e.g. over-coating extra-step).

CRediT authorship contribution statement

Asma Chaabani: Investigation, Writing - original draft, Writing - review & editing, Visualization. **Laurent Labonne:** Investigation. **Carlos Alburez Tercero:** Investigation. **Jean-Pierre Picard:** Resources. **Catherine Advenier:** Resources. **Vanessa Durrieu:** Formal analysis, Writing - original draft, Writing - review & editing. **Antoine Rouilly:** Writing - original draft, Writing - review & editing. **Fabien Skiba:** Resources, Supervision, Project administration. **Philippe Evon:** Conceptualization, Methodology, Validation, Formal analysis, Investigation, Writing - original draft, Writing - review & editing, Visualization, Supervision, Project administration.

Declaration of Competing Interest

The authors report no declarations of interest.

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