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Published in:
Animal Feed Science and Technology

Link to article, DOI:
10.1016/j.anifeedsci.2023.115752

Publication date:
2023

Document Version
Peer reviewed version

Link back to DTU Orbit

Citation (APA):
Understanding and modelling the aquafeed extrusion process for Atlantic salmon feed with an empirical model

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Abstract

An empirical model was applied to quantitatively model the effects of extrusion variables and recipe compositions on quality of feed pellets. Lab extrusion trials were performed with an Atlantic salmon feed recipe with using a central component design (CCD). Comparing with a coated commercial feed made with the same recipe, the lab extrusion pellets had similar bulk density, hardness, and durability properties. A Response Surface Method (RSM) was also used to regress the extrusion trial data and compared with the empirical model results. The respective Absolute Average Deviation (AAD%) values of the empirical model and RSM regression were 4.1% and 1.8% for pellet bulk density, 7.4% and 4.9% for pellet hardness, 10.4% and 10.0% for pellet oil absorption, 13.7% and 8.3% for pellet water stability, and 11.3% and 5.5% for pellet durability. Using the model coefficients obtained from the extrusion results, the empirical model can partly extrapolate the pellet qualities of an additional extruded recipe, which has a slightly adjusted wheat gluten and wheat. The model performance was evaluated based on $R^2$, RSME (root-mean-square deviation), AAD% of the empirical model was better than the RSM model to predict the effects of extrusion variables on pellet quality parameters. The effects of the extrusion process variables and
recipe composition on pellet quality parameters are predicted by the empirical model. Its
demonstrates that the empirical model is a new method to understand extrusion trial results in
different extrusion systems and recipes without changing its mathematical form of
expression, which gives a common reference to compare an extruded recipe in different
systems and has practical engineering applications.

**Keywords:**
Aquafeed, Atlantic salmon, Modelling, Extrusion, Response Surface Method,
Phenomenological model

**Abbreviations**

AAD%: absolute average deviation

CCD: central component design

RMSE: root-mean-square deviation

RSM: response surface method

RVA: rapid visco analyser

SPC: soy protein concentration

1. **Introduction**

Sustainable aquafeed production is in the scope of the core objectives for “Blue
Transformation” in aquaculture development, suggested by Food and Agriculture
Organization of the United Nations (FAO) in 2022 (FAO 2022). It is known that aquaculture
can help alleviate world food shortage, which can be achieved due to excellent growth
capacity and feed conversion of fish and thereby a fast production turnover. In 2020, 49% of the production of aquatic animals came from the aquaculture industry and 51% from capture fisheries (FAO 2022). As an important basis for sustainable aquaculture, sustainable aquafeed must be produced in terms of new raw materials or ingredients, energy efficiency, environmental and social compatibility.

For sustainable aquafeed development, one direction is to use alternative protein sources in diet formulations, such as but not limited to microalgae, insect meal, or single-cell proteins. Currently, one of the major protein ingredients found in diet formulations for fish is fishmeal derived from wild-caught forage fish (Hua et al. 2019). However, with the growth of the aquaculture industry, we cannot simply capture more wild forage fish to produce fishmeal as a major ingredient in terms of protecting the oceans natural resources and sustainable development. Many alternative ingredient sources have been investigated to replace fishmeal and other primary ingredients, such as the soybean protein concentrate with alternatives in aquaculture feeds (Mohan et al. 2022; Siva Raman et al. 2022; Ge et al. 2022; Rawski et al. 2020; Weththasinghe et al. 2022; Samuelsen et al., 2018).

Although many diverse ingredients can be used to replace fishmeal in terms of dietary nutrients, these alternative ingredients have also changed the processing properties of the diet in industrial-scale feed productions. In other words, one cannot be certain of the effects of inclusion level of a new ingredient on processing parameters which replace fishmeal and/or other ingredients in a new recipe for industrial scale feed production. Furthermore, differences in supplier, batch number, or seasons of harvest can negatively affect processing and pellet quality parameters due differences in nutrient compositions and therefore must be considered in industrial applications (Samuelsen and Oterhals, 2016). Currently, laboratory or pilot scale trials must be conducted before a new ingredient is implemented into an aquafeed recipe. For example, Weththasinghe et al. (2021; 2022) found that partial replacement of
fishmeal in an Atlantic salmon diet with full-fat black soldier fly larvae meal reduced feed pellet hardness, expansion, and water stability, but did not affect the growth performance of Atlantic salmon.

To understand the extrusion characteristics of a new recipe and the impact a new ingredient can have on pellet quality, a Response Surface Methodology (RSM) is often used to analyse the lab or pilot extrusion trial results. However, the RSM results are limited to the studied case (i.e., process conditions, extruders). This is because the RSM relays only on a statistically significant method rather than considering the physical and chemical mechanisms of the extrusion process. Therefore, an alternative method is needed. In the formulation of an aquafeed recipe, several combinations of ingredients can be used to meet a particular nutritional target profile for amino acids, lipids, or minerals. However, it is difficult to know if each recipe will produce the same finished feeds with the same pellet quality without the use of lab and/or pilot scale extrusion trials. To change the skill-based extrusion operation, it is important to quantitatively model the extrusion process from physical and chemical principles.

Research on quantitative modelling has been carried out for the extrusion cooking process (Moraru and Kokini 2003; Kristiawan et al., 2020). Klein and Marshall (1966) suggested a basic framework of mathematical models for extrusion cooking. These modelling methods include 1) RSM (Altan et al., 2008; Chen et al. 2010), 2) numerical simulation through physical model (Alves et al., 2009; Gonzalez et al., 2001), 3) phenomenological or empirical method (Alvarez-Martinez et al., 1988; Cheng and Hansen 2016; Kristiawan et al., 2017), 4) genetic algorithm (Shankar and Bandyopadhyay 2007) and neural network method (Ganjyal et al., 2003). Although many of the methods (e.g., RSM) can describe the investigated processes, their results are often limited to the studied cases (experimental condition and range). From a physical principle and applied process engineering point of view, the
phenomenological or empirical method has the chance to capture the characteristics of the feed pellet extrusion process in terms of recipe composition and process variable changes in a process. Thus, a phenomenological or empirical method of Cheng et al. (2022) will be used in this study to analyse the feed pellet extrusion process.

The aim of this study was to investigate the effects of extrusion process variables on the physical quality of Atlantic salmon feed with a phenomenological model. The phenomenological model is also compared with the results from a RSM for the experimental data regression. In addition, the effects of extrusion process variables and recipe compositions on feed pellet qualities were also evaluated with both models.

2. Material and methods

2.1 Experimental ingredients and recipes

An industrial Atlantic salmon recipe meal mix without added oil (Diet-A in Table 1) and relevant ingredients were supplied by BioMar A/S (Brande, Denmark). Based on the Diet-A meal mix, a similar recipe, of 5 kg, was prepared for testing the extrusion process (“Extra” in Table 1) to observe the impact of a slightly changed recipe (adjust wheat gluten and wheat, see Table 1) on feed pellet quality. The ingredient content fluctuation often happens in practice, and this was one of the main aim of the investigation. In addition, 2 kg of commercially coated pellets (3 mm in diameter) produced by the same formulations as Diet-A (Table 1) were also obtained from LetSea AS (Dønna, Norway). In the investigation, the pellet quality parameters, such as bulk density and hardness, of the commercial product were used as a reference to evaluate the feed pellets produced in this extrusion trial. If the product quality parameters of the extrusion are similar to that commercial one, the extrusion trial data in the trials are valuable in terms of the observations of the effects of extrusion variables on
pellet quality. The recipe formulations and proximate chemical compositions used in the investigation are shown in Table 1.

2.2 Experimental design

A Central Composite Design (CCD) was used in the investigation, which included three factors and three levels. The three independent variables and their levels were temperature 100-125 °C, moisture content 23-28% (wt), and screw speed 250-400 rpm. For the design, the levels of the temperature, moisture, and screw speed were determined from initial trials for sinking pellet production and extruder limitation. The design results are shown in Table 2, where CTRL08 is the central point and was run twice.

2.3 Extrusion trial and feed pellet production

A twin-screw extruder (HAAKE Rheomex, PTW16, Thermo Scientific), having two co-rotating screws with three sets of kneading elements, was used. The recipe mixture was supplied into the extruder by a single-screw feeder controlled and the extruder control system (Polysoft, Thermo Scientific) at a constant feed flow rate 3 kg/h for all recipes. The extruder barrel consisted of 6 zones with independent heating capacities. A typical screw configuration was suggested by Thermo Scientific company and used for all recipe mixture extrusion. The temperature of zones 1, 2, and 3 near the feeding section were set to 25 °C and zones 4, 5, and 6 were set according to the experimental design values shown in Table 2. Water was injected in zone 2 of the extruder through a peristaltic pump (PERIMAX16, Spectec, GmbH, Germany). The water temperature was controlled through a water bath system (Julabo SW 20, moving-bed water bath, Germany). The discharge section of the extruder was equipped with a 3 mm diameter circular die without a cutter and extruded as ropes which were cut by hand to 4-5mm pellets.
The extrusion trials were carried out by following the experimental design shown in Table 2 by using the dry recipe mixture supplied by BioMar. During the extrusion, process variables were recorded every second and saved in the extruders computer system. Extruded feed products were collected after the extruder reached a steady state (about 5 minutes, calibrated by product weight measurement on a scale). After extrusion, all extruded products (ropes) were stored in sealed plastic bags and put into a storage room at 4 °C until manual cutting of the product to 4-5 mm pellets. After the pellets were manually cut, they were dried in a hot air oven (Rational, Combi-Dampfer, CCC 101/02, Germany) at 60 °C for 5 hours. After drying, the water content of the pellets was measured to ensure their water content was between 6-8% (wt). After drying, the pellets were stored in a cooling room at 4 °C until they could be coated with oil. Coating of the pellets was performed at room temperature (23-25 °C) using a home-made vacuum coating system comprised of a glass bottle and a vacuum pump (two-stage, VEVOR) with fish oil to reach the maximum oil absorption in the pellets. During the coating, 40 g of dried pellets were weighed and placed into the bottle with the vacuum pump connected to the bottle for 4-5 min to remove any air remaining in the bottle. Afterward, a 20-25% overdose of oil (over the oil percentage value given for Diet-A recipe, see Table 1) was introduced into the bottle to reach a saturation absorption. The vacuum bottle was then shaken for 3 minutes to ensure all the pellets absorbed the oil evenly. Any excess oil was removed from the pellets by paper towel and the pellets were stored at 4 °C in a storage room for further analysis. An addition, a second extrusion trial was performed with the recipe “Extra” shown in Table 1 with the relevant extrusion variables provided in Table 2 (Extra). In the second extrusion trial, the wheat gluten and wheat percentage were 15.2 and 18.0%, respectively. This adjustment was to observe the consequence of a slight recipe change on pellet quality, which is a common occurrence in industrial scale production.
2.4 Pellet quality measurement

Pellet hardness was measured using a texture analyser (TA-XT plus, Stable Micro System Ltd, UK) equipped with a 30 kg load cell. The pellet was placed upright on the plate and a 50 mm diameter cylinder probe was selected. The probe was set to a descent rate of 1 mm/s and lifted when the pellet was subjected to 60% deformation (Samuelsen et al. 2021). The peak force value of the graph obtained was the hardness result. For all the points in the experimental design (Table 2), the hardness measurement of pellets was repeated 15 times and an average value was taken.

Bulk density was measured by a beaker calibrated with distilled water at room temperature for its volume (V). The feed pellets were added to the beaker without external interference until it was overflowing. Then, a flat plate was used to scrape the pellets from the edge of the beaker and weighed (M). The bulk density was determined using the ratio of M/V, g/litre. The measurements were repeated three times.

Pellet oil absorption was measured by oil coating 40 g of pellets in the vacuum coating device as described in the previous section. After coating, the pellets were dried with 3 layers of tissue paper, and gently wiped to remove excess oil from the surface of the pellets and weighed. The pellet oil absorption rate was calculated according to equation 1.

\[
\text{Oil absorption capacity } \% = \frac{A_2 - A_1}{A_1} \times 100
\]

where \(A_1\) was the weight of the uncoated pellet (ca. 40g) and \(A_2\) was the weight of the coated pellet (g). Each measurement was repeated twice.

Water stability measurements were carried out by following the method of Baeverfjord et al. (2006). Pellets from the same trial were weighed in 3 groups, approximately 5 g per group. Then, the grouped pellets from the same run number were placed into three self-made metal
containers (modified by a pen holder). Nine containers were put into a moving-bed water bath (Julabo, SW20, moving-bed water bath, Germany) at 25 °C and shaken at 100 rpm shaking frequency for 30, 60, and 90 minutes. A total of nine containers were initially placed in a water bath. Over the course of 90 minutes, the nine containers were removed at different time intervals: three cages after 30 minutes, another three after 60 minutes, and the last three after 90 minutes. Pellets were carefully emptied from the container and placed into a tin foil cup and dried in a hot air oven for 2 hours at 135°C. Then, the dried pellet samples were cooled down to room temperature and were weighed. The water stability (WS, %) of the pellets was calculated using Eq. 2.

\[
WS\% = \frac{B_1(1-b)-B_2}{B_1(1-b)} \times 100
\]  

where \(B_1\) was the pellet weight before measurement (\(B_1=5\) g), \(B_2\) was the pellet weight after 90 min measurement (g), \(b\) was the water content of the sample pellet before measurement (wet basis). The moisture content of the pellets was measured by a moisture analyser (Buch & Holm A/S, AD-4714A, Japan) at 130 °C for 10 min under standard conditions by following the manual of the analyser. Each measurement was repeated three times.

Pellet durability was tested by a DORIS tester (AKVA smart ASA), where 350 g of uncoated pellets were taken into the device to carry out the test, as described in DORIS user’s manual. A 1 mm sieve was used to remove any dust from the collected product and was weighed as percent fines. The DORIS value was calculated by fine %, i.e., weight of collected fines (fine weight) divided by initial weight of the pellets (initial weight) as shown in equation 3.

\[
\text{DORIS (\% fine)} = \frac{\text{fine weight}}{\text{initial weight}}
\]

2.5 Pasting property measurement
The pasting properties of the ingredients and the recipe mixtures were measured using a Rapid Visco Analyser (RVA) (Newport Scientific, Australia). A 28 g solution was prepared in a canister by adding distilled water to 4 g of dry ingredients (wheat flour). The ingredients were first weighed in a disposable plastic dish and the distilled water added. The mixture was then mixed for 1 minute and put into the canister. The temperature and shear profiles used by Sørensen et al. (2011) were applied in the measurement, which is given in Table 3. In the RVA measurement, starch molecules in the ingredients and mixtures were gelatinized with water.

2.6 Model

In a feed pellet extrusion process, pellet physical quality parameters of an extruded recipe are determined by extruder geometric parameters, extrusion process operation variables, and the recipe composition. The extruder geometry is crucial for the pellet physical quality parameters before the start of the extrusion process. After starting the extruder, the pellet quality is controlled by process variables and recipe composition. In this study, the effects of extrusion variables and recipe properties other than extruder geometry were the investigation targets.

The method suggested by Cheng et al. (2022) was used to analyse the relationship between extrusion variables and feed pellet qualities. This method assumes that the pellet physical qualities are related to the viscous property of the melt flow in the die section of an extruder. The viscous property of the melt flow is related to extrusion process variables (screw speed, moisture content and die temperature) and recipe ingredients. Every individual ingredient in a recipe has its own contribution to the viscous property of the melt flow inside an extruder. Based on the assumptions and the work of Alvarez-Martinez et al. (1988), Harper and
Rhodes (1971) and Carley (1985), an empirical model was proposed by Cheng et al (2022) as:

\[ Y_{prop} = k_1 \exp(\sum x_i \ln \eta_i) N_s \alpha \exp \left( \frac{\Delta E}{RT} \right) \exp(k_2 M) \]  \hspace{1cm} (4)

where \( Y_{prop} \) is a feed pellet quality parameter that can be hardness, water solubility, durability, bulk density, and oil absorption, \( x_i \) is the ingredient composition, g/g, \( \Delta E \) is the energy of activation, J/mol, \( R \) is the gas constant, 8.314 J/mol·K, \( T \) is the absolute temperature, K, \( M \) is the moisture content, g/g, \( k_1, \eta_i, \alpha, \Delta E \) and \( k_2 \) are the experimental data determined parameters and can be obtained from experimental data regression. Equation (4) is the model to correlate an extrudate quality parameter with recipe ingredient compositions and extrusion variables. For different extrusion systems and recipes, the model will have different model coefficients that are determined by fitting experimental data. In equation (4), the term \( \exp(\sum x_i \ln \eta_i) \) represents the combination contribution of ingredients to pellet quality. The term \( N_s \alpha \exp \left( \frac{\Delta E}{RT} \right) \exp(k_2 M) \) is the contribution of extrusion process variables, i.e., screw speed \( (N_s) \), die temperature \( (T) \), and moisture content \( (M) \).

3. Results

3.1. Pasting property results

The RVA measurements were carried out for wheat, soy protein concentrate (SPC), wheat gluten, fishmeal, and Diet-A recipe mixture, respectively. The measurement results show the viscosity changes of an ingredient over time and are shown in Figure 1. The recipe for Diet-A includes commonly used protein sources such as fishmeal, wheat, SPC, and wheat gluten. In the recipe for Diet-A, the wheat and wheat gluten provided binding properties during extrusion. In Figure 1, the final viscosity of wheat may represent such binding characteristics.
with a high viscosity. The increase of final viscosity for SPC or gluten was established by the shear force during the RVA measurement. Lastly, it is clear that the fishmeal cannot build up viscous properties in the RVA measurement.

From the RVA results, it is assumed that the viscous property changes of the Diet-A recipe come from a summation of the contribution of each ingredient in the recipe. The summation calculation is based on a hypothesis to get the total viscosity of the Diet-A. In numerical calculation point of view, such summation can be seen in many circumstances, such as the total nutrition calculation, where a linear summation method is often used to calculate total crude protein, fat, and starch content from the information of each ingredient and the ingredient inclusion. For example, the total crude protein content of Diet-A can be calculated by the percentage of gluten, SPC, wheat, etc., and their protein content (see Table 1) by a linear summation of ingredient percentage times its protein content. In Figure 1, the viscosity of Diet-A, gluten, SPC, and wheat has different values. However, the relationship between the viscosity value of Diet-A and the viscosity values of different ingredients is not a simple linear summation by ingredient percentage times its viscosity value. The relationship between the viscosity values of Diet-A and ingredients is governed by a non-linear summation or combination.

In RVA measurement, these ingredients have been changed at a physical and chemical level, such that the starch molecules are broken-down through gelatinization, and protein molecules are re-organized. Thus, the combination rule for the viscosity of Diet-A mixture is not a simple linear summation of the viscosity of each ingredient. As the recipe mixture extrusion process is similar to the RVA in terms of shear force and temperature, the viscosity development of Diet-A in the extrusion process has a contribution of each ingredient. The viscosity of Diet-A in the extrusion process can also be calculated by a non-linear summation
rule. However, the non-linear summation rule is not available and must be developed for future use.

3.2. Extrusion trial and feed pellet quality parameter results

The minimum and maximum values of the pellet quality parameters are given in Table 4 and compared with the commercial feed produced using the same recipe. Feed provided by BioMar was used as a commercial control feed to which the quality parameters of the pellets produced in this study could be compared. In this case, the evaluation indicate that the feed pellets produced in this study are similar to the commercial control and are therefore valuable for model development and validation.

As can be seen from Table 4, that the pellet bulk density and durability from the extrusion trials were similar to that of the commercial feed. The pellet hardness was slightly higher than that of commercial feed and the pellet water stability (measurement of pellet solubility) from the extrusion trials was higher than the commercial feed. The received commercial feed was a coated feed pellet, but has the same recipe as Diet-A (see Table 1). In the commercial feed coating operation, 20% oil (including fish oil and rapeseed oil, see Diet-A in Table 1) was added into the pellets. After coating, in general, the pellet is more difficult to dissolve in fresh water because the pores or pore surfaces of the pellet are filled or covered with oil and therefore water cannot permeate it due to the hydrophobic properties of the oil or the changed surface tension in the porous structure. This can be seen from the comparison where the pellet quality from the extrusion trials was equivalent to that of the commercial feed pellet.

3.3. Modelling results
Equation (4) was used to fit the extrusion experimental data and to get the quantitative relationship for pellet quality parameters and extrusion variables. From the regression results, the model behaviours for correlation extrusion variables and pellet quality parameters were evaluated. In the evaluation, the regression parameters, i.e., absolute average deviation, $R^2$ value and RMSE (root-mean-square deviation) were calculated. The absolute average deviation (AAD%), $R^2$ value, and RMSE of the model regression are given in Table 5.

In Table 5, AAD% was calculated as

$$\text{AAD\%} = \frac{1}{n} \sum_{i=1}^{n} \left( \frac{|Y_{\text{exp}}^{\text{prop}} - Y_{\text{cal}}^{\text{prop}}|}{Y_{\text{exp}}^{\text{prop}}} \right) \times 100\%$$  \hspace{1cm} (5)

where $Y_{\text{exp}}^{\text{prop}}$ and $Y_{\text{cal}}^{\text{prop}}$ are the experimental and calculated pellet quality property, respectively, $n$ is the number of experimental runs.

In comparison, the RSM was also applied to analyse the same set extrusion data. The RSM analysis results are listed in Table 6. As can be seen from Table 6, the RSM can reach a good regression model for pellet physical properties. However, some RSM equations can only achieve a statistically significant, but cannot represent the impact of extrusion variables. For example, the model for pellet bulk density cannot describe the effect of moisture content, as the moisture content is not significant in data analysis.

In comparison with the RSM results, Tables 5 and 6 show that the $R^2$ value of equation (4) prediction ($R^2=0.51$) for pellet bulk density was a lower than that of the RSM ($R^2=0.94$), whereas the $R^2$ values of the equation (4) ($R^2=0.31$) and RSM prediction ($R^2=0.68$) were different to each other for pellet water stability. The equation prediction for pellet durability has a lower $R^2$ (0.53) than that of the RSM result ($R^2=0.92$). The pellet hardness prediction by the equation (4) gives also a lower $R^2$ (0.6) than that of the RSM result ($R^2=0.80$). For pellet oil absorption, the equation (4) prediction and RSM have a similar $R^2$, i.e., 0.89 and 0.92, respectively.
Figures 2-6 present the comparisons between the experimental and the fitted pellet quality parameters from equation (4) and the RSM. As can be seen from Figure 2, the errors for pellet bulk density prediction were not evenly distributed for the model calculation from equation (4). Figures 3-5 show that the prediction errors for pellet hardness, durability, and oil absorption were evenly distributed for equation (4) and the RSM. Figure 6 indicates that the prediction errors for pellet water stability were scattered and larger than that of other pellet quality parameters given in Figures 3-5.

Comparing the AAD% and the RMSE of the data regression from equation (4) and the RSM, the RSM and Equation 4 has similar or different AAD% and RMSE for all studied pellet quality properties. However, the RSM model for pellet bulk density (see Table 6) has no extrusion moisture content term. The RSM model for hardness (see Table 6) has only extrusion temperature and no moisture and screw speed terms. The models are only obtained from statistically significant and have no physical explanations. Using the R², AAD%, RMSE values, Figures 2-6, and Table 6 as a measurement, it suggests that equation (4) can be used to replace the RSM to analyse the extrusion trial data in the study.

Taking the model coefficients from Table 7, equation (4) was also applied to predict the pellet quality parameters of extra recipe extrusion trial. The prediction results are given in Table 5. Table 5 shows that AAD% values of the equation (4) prediction for the extra recipe pellet bulk density and hardness are below 10% that is acceptable in industrial application. The AAD% values for the extra recipe pellet water stability and oil absorption are higher than 10%. From the prediction results for the extra recipe pellet qualities, it demonstrated that equation (4) can partly predict the pellet quality parameters for a recipe whose ingredient formulation was slightly changed from original recipe design table. On the other side, the RSM cannot predict pellet quality parameters in such a situation as the RSM model did not include the impact of recipe compositions, but the extrusion variables, i.e., temperature,
screw speed and moisture content. From the point of view of the understanding and prediction of recipe composition aspect, equation (4) is better than the RSM models in this study.

**3.4 Effects of extrusion variables on pellet bulk density and oil absorption from equation (4) prediction**

Higher bulk density pellets have a denser pellet structure and lower internal pore volume, which correlated to having a lower oil absorption capacity. Thus, pellet bulk density is related to pellet oil absorption. The effects of extrusion variables on pellet bulk density and oil absorption were calculated and presented in Figure 7. In Figure 7, a decrease in either moisture content from 28 to 23% or extrusion temperature gives an increase of pellet bulk density in the current trial, which is applicable for the production of feeds for Atlantic salmon.

Figure 8 shows that an increase of extrusion temperature gives more pellet oil absorption for the Diet-A recipe. Figure 7 shows that higher extrusion temperature produces lower bulk density pellets. And the lower bulk density pellets indicate more pores or higher pore volume, thus more oil absorption. For the Diet-A recipe, a decrease of moisture content (upper surface in Figure 8) increases the pellet oil absorption, especially at higher extrusion temperature. In the extrusion trials for Diet A, screw speed has no significantly impact on pellet bulk density or oil absorption, as shown in Figures 7-8.

**3.5 Effects of extrusion variables on pellet hardness, durability, and water stability equation (4) prediction**
Figs. 9-10 and 10 illustrate the effects of extrusion variables on pellet hardness and durability, respectively. From Figure 9, pellet hardness increases with decreasing of extrusion temperature, whereas the change of moisture content did not significantly influence pellet hardness. Figure 10 indicates that an increase of extrusion temperature increased pellet durability for the Diet-A recipe, resulting in more dust during pellet transportation and storage, which can lead to nutrient losses and water quality issues in recirculation aquaculture systems (leading to reduced performance). An increase of moisture content (lower surface in Figure 10) decreases pellet durability for the Diet-A recipe. Moreover, the screw speed or shear stress has no significant impacts on pellet hardness and durability in the Diet-A recipe extrusion trials, as shown in Figures 9-10.

From Figure 11 water stability increases with increasing of extrusion temperature, where pellets are more soluble in water. A decrease of moisture content from 28-23% gives a slightly higher water stability. An increase of screw speed increases pellet water stability.

4. Discussion

Based on the extrusion trial data, equation (4) was applied to correlate the relationship between the extrusion process variable and the pellet quality parameters. In feed pellet extrusion, changes in the material pasting or viscosity properties govern the physical properties of the final product. Equation (4) copes with some basic pellet production mechanisms such as viscosity development during processing. Therefore, equation (4) was used to calculate the effects of extrusion variables on pellet quality parameters in following sections. From such calculation, pellet quality parameter trends can be investigated with the changes of different extrusion variables.
4.1 Comparison of RSM and Equation (4)

The RSM model and the empirical model were different in terms of mathematical expression and physical explanations. The RSM models in Table 6 treated the extrusion trial data as a set of data without considering any physical and chemical aspects of the process and only applied statistical significances for the extrusion trial data analysis. Thus, the RSM models for different pellet property parameters had different mathematical expressions after optimizing its math expression. Table 6 shows that the RSM model for pellet bulk density has no extrusion moisture variable, and no extrusion moisture and screw speed for water stability. Thus, these modes have no physical explanations in terms of extruder operation. It has been known that RSM models can only be applied to the same experimental conditions and ranges as studied extrusion trials but cannot be used beyond these extrusion experimental boundaries (Kristiawan et al., 2020).

Equation (4) had a same form of mathematical expression for all different pellet quality parameters, which was constructed from a widely accepted viscosity calculation model. The same mathematical expression can be applied to compare the effects of extrusion variables and recipe compositions at the same reference crossing different extruders and recipes. Since equation (4) had its physical explanations for each term, such as recipe composition impact, temperature, screw speed, and moisture content, it can be applied to predict the extra recipe pellet quality parameters with those model coefficients obtained from a similar recipe. In this study, equation (4) provided a good prediction of the pellet bulk density and hardness of the extra recipe using the model coefficients obtained from another extruded recipe. On the other hand, the same mathematical expression [equation (4)] for all different pellet quality parameters also reduces the accuracy of the equation (4). Further work is needed to improve the model prediction of each pellet quality parameter.
4.2 prediction of the effects of extrusion variables on pellet quality parameters by equation (4)

It had been indicated from the equation (4) prediction that extrusion temperature and moisture content can have significant influence on pellet qualities for the Diet-A recipe. The impact of extrusion screw speed on pellet quality was not strong. It indicated that when extruder screw speed is higher than 300 rpm, the shear force or mechanical energy input is enough for optimal extrusion. Similarly, Draganovic et al. (2011) investigated the effects of fish meal, wheat gluten, soy protein concentrate, and feed moisture on extruder system parameters using a constant extruder screw speed (650 rpm), where wheat gluten content was obtained from initial extrusion trials. Samuelsen et al. (2022) used a standard shaft speed (400 rpm) in their investigations for using tunicate (marine invertebrate) as a sustainable protein source in fish feed, where screw speed was not treated as a significant impact factor on pellet quality. However, when starch content or the starch source were changed in a recipe, the screw speed had a strong impact on pellet expansion and durability (Sørensen et al., 2011).

5. Conclusions

The feed pellet quality changes of an Atlantic salmon feed were investigated in a laboratory extrusion system with a central composition design. To quantitatively understand and model the feed extrusion process, a Response Surface Method (RSM) and an empirical model were applied to model the effects of extrusion variables on feed pellet qualities. Comparing with a coated commercial feed made by the same recipe, a lab-scale extrusion of pellets resulted in a similar bulk density, hardness, and durability properties. Using $R^2$, RSME, AAD%, experimental vs. fitted pellet quality parameter figures (Figs. 2-6), Table 6 and an extra recipe...
pellet quality prediction as a measurement, the empirical model [equation (4)], was better than the RSM model to predict the effects of extrusion variables on pellet quality parameters. The obtained empirical model well predicted the effects of the extrusion process variables and recipe composition on pellet quality parameters. It demonstrated that the empirical model is a new method to understand extrusion trial results in different extrusion systems and recipes without changing its mathematical expression, which provide a common reference to compare a recipe extrusion in different systems with practical engineering applications.

Acknowledgement

This work was supported by Innovation fund Denmark and Nordforsk project number 104310, BioMar A/S for supply of raw materials, LetSea for supply of a commercial feed (produced by BioMar A/S), and the support from all project partners.

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Declaration of Competing Interest
None

Figure 1. RVA results for wheat, SPC, wheat gluten, fishmeal and Diet-A recipe mixture
Figure 2. Experimental vs. model fitted pellet bulk density values. Eq.(4): equation (4) results, $R^2=0.51$, RSM: RSM model results, $R^2=0.84$
Figure 3. Experimental vs. model fitted pellet hardness values. Eq.(4): equation (4) results, $R^2=0.60$, RSM: RSM model results, $R^2=0.80$

Figure 4. Experimental vs. model fitted pellet durability values. Eq.(4): equation (4) results, $R^2=0.53$, RSM: RSM model results, $R^2=0.89$
Figure 5. Experimental vs. model fitted pellet oil absorption values. Eq.(4): equation (4) results, $R^2=0.89$, RSM: RSM model results, $R^2=0.92$
Figure 6. Experimental vs. model fitted pellet water stability values. Eq.(4): equation (4) results, $R^2=0.32$, RSM: RSM model results, $R^2=0.68$
Figure 7. Effects of extrusion variables on pellet bulk density. Three surfaces represent moisture content changes at 23% (upper surface), 25% (middle surface) and 28% (lower surface). Diet-A recipe.
Figure 8. Effects of extrusion variables on pellet oil absorption. Three surfaces represent moisture content changes at 23% (upper surface), 25% (middle surface) and 28% (lower surface). Diet-A recipe
Figure 9. Effects of extrusion variables on pellet hardness. Three surfaces represent moisture content changes at 23% (lower surface), 25% (middle surface) and 28% (upper surface). Diet-A recipe
Figure 10. Effects of extrusion variables on pellet durability. Three surfaces represent moisture content changes at 23% (upper surface), 25% (middle surface) and 28% (lower surface). Diet-A recipe
Figure 11. Effects of extrusion variables on pellet water stability. Three surfaces represent moisture content changes at 23% (upper surface), 25% (middle surface) and 28% (lower surface). Diet-A recipe

Table 1 Formulation and main nutrient composition of Atlantic salmon feed recipe used in the investigation (g/kg)

<table>
<thead>
<tr>
<th>No</th>
<th>Ingredient</th>
<th>Diet-A</th>
<th>Extra</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Fish meal</td>
<td>200</td>
<td>200</td>
</tr>
<tr>
<td>2</td>
<td>Soy protein concentrate</td>
<td>200</td>
<td>200</td>
</tr>
<tr>
<td>3</td>
<td>Wheat gluten</td>
<td>144.6</td>
<td>152.2</td>
</tr>
<tr>
<td>4</td>
<td>Wheat</td>
<td>187.6</td>
<td>180.0</td>
</tr>
<tr>
<td>5</td>
<td>Fish Oil</td>
<td>133.1</td>
<td>133.1</td>
</tr>
<tr>
<td>6</td>
<td>Rapeseed Oil</td>
<td>62.4</td>
<td>62.4</td>
</tr>
<tr>
<td>7</td>
<td>Other</td>
<td>72.4</td>
<td>72.4</td>
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Chemical composition*

<table>
<thead>
<tr>
<th></th>
<th>Diet-A</th>
<th>Extra</th>
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<tbody>
<tr>
<td>Crude protein</td>
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<td></td>
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<tr>
<td>Lipid</td>
<td>234.3</td>
<td></td>
</tr>
<tr>
<td>Carbohydrate</td>
<td>233.8</td>
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</table>

*Calculated based on raw material analyses.
Table 2 Code, actual values of experimental design variables, and extrusion results of Diet-A(*) and extra (***) recipes

<table>
<thead>
<tr>
<th>Code</th>
<th>Moisture %wt</th>
<th>SS rpm</th>
<th>Temp °C</th>
<th>Hardness N</th>
<th>BD(^1) g/L</th>
<th>OAC(^2) %</th>
<th>WST-90(^3) %</th>
<th>DORIS % fine</th>
</tr>
</thead>
<tbody>
<tr>
<td>CTRL 01*</td>
<td>23</td>
<td>250</td>
<td>100</td>
<td>97.96</td>
<td>655</td>
<td>6.44</td>
<td>17.18</td>
<td>1.43</td>
</tr>
<tr>
<td>CTRL 02*</td>
<td>23</td>
<td>250</td>
<td>125</td>
<td>69.09</td>
<td>582</td>
<td>14.25</td>
<td>15.03</td>
<td>1.62</td>
</tr>
<tr>
<td>CTRL 03*</td>
<td>23</td>
<td>325</td>
<td>112.5</td>
<td>98.77</td>
<td>644</td>
<td>9.75</td>
<td>13.14</td>
<td>1.55</td>
</tr>
<tr>
<td>CTRL 04*</td>
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<td>400</td>
<td>100</td>
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<td>615</td>
<td>10.00</td>
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<td>250</td>
<td>112.5</td>
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<td>612</td>
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<td>1.36</td>
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<tr>
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<td>100</td>
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<td>6.00</td>
<td>19.06</td>
<td>1.14</td>
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<tr>
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<td>325</td>
<td>112.5</td>
<td>101.56</td>
<td>622</td>
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<td>18.97</td>
<td>1.75</td>
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<tr>
<td>CTRL 09*</td>
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<td>325</td>
<td>125</td>
<td>75.02</td>
<td>574</td>
<td>16.00</td>
<td>15.82</td>
<td>1.61</td>
</tr>
<tr>
<td>CTRL 10*</td>
<td>25</td>
<td>400</td>
<td>125</td>
<td>89.15</td>
<td>628</td>
<td>9.88</td>
<td>19.06</td>
<td>1.38</td>
</tr>
<tr>
<td>CTRL 11*</td>
<td>28</td>
<td>250</td>
<td>100</td>
<td>92.16</td>
<td>694</td>
<td>7.25</td>
<td>15.08</td>
<td>0.76</td>
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<tr>
<td>CTRL 12*</td>
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<td>250</td>
<td>125</td>
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<td>11.94</td>
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<td>1.65</td>
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<tr>
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<td>325</td>
<td>112.5</td>
<td>89.81</td>
<td>602</td>
<td>6.25</td>
<td>16.84</td>
<td>1.61</td>
</tr>
<tr>
<td>CTRL 14*</td>
<td>28</td>
<td>400</td>
<td>100</td>
<td>101.56</td>
<td>637</td>
<td>5.87</td>
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<td>0.93</td>
</tr>
<tr>
<td>CTRL 15*</td>
<td>28</td>
<td>400</td>
<td>125</td>
<td>83.61</td>
<td>568</td>
<td>11.63</td>
<td>24.34</td>
<td>1.49</td>
</tr>
<tr>
<td>Extra**</td>
<td>23</td>
<td>325</td>
<td>125</td>
<td>83.92</td>
<td>569</td>
<td>11.25</td>
<td>16.1</td>
<td></td>
</tr>
</tbody>
</table>

\(^1\) Bulk density, \(^2\) Oil absorption capacity, \(^3\) Water stability at 90min, DORIS: DORIS value

*: CTRL 01-15: diet-A recipe trials

**: Extra: extra recipe trial, Temp: temperature, SS: screw speed

*4*: CTRL08 is the central point in the experimental design

Table 3 Temperature and shear profile used in RVA measurement

<table>
<thead>
<tr>
<th>Time (min:sec)</th>
<th>Type (Temp or Speed)</th>
<th>Value (°C) or (RPM)</th>
</tr>
</thead>
<tbody>
<tr>
<td>00:00</td>
<td>Temp</td>
<td>50°C</td>
</tr>
<tr>
<td>00:00</td>
<td>Speed</td>
<td>960rpm</td>
</tr>
<tr>
<td>00:10</td>
<td>Speed</td>
<td>160rpm</td>
</tr>
<tr>
<td>01:00</td>
<td>Temp</td>
<td>50°C</td>
</tr>
<tr>
<td>04:30</td>
<td>Temp</td>
<td>95°C</td>
</tr>
<tr>
<td>07:00</td>
<td>Temp</td>
<td>95°C</td>
</tr>
<tr>
<td>Time</td>
<td>Temp</td>
<td>°C</td>
</tr>
<tr>
<td>--------</td>
<td>------</td>
<td>----</td>
</tr>
<tr>
<td>11:00</td>
<td></td>
<td>50</td>
</tr>
<tr>
<td>13:00</td>
<td></td>
<td>50</td>
</tr>
</tbody>
</table>

Table 4 Minimum and maximum values of feed pellet quality parameters

<table>
<thead>
<tr>
<th></th>
<th>Diet-A recipe</th>
<th>Commercial feed$^1$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>min</td>
<td>max</td>
</tr>
<tr>
<td>Bulk density, g/l</td>
<td>568</td>
<td>694</td>
</tr>
<tr>
<td>Durability, %fine</td>
<td>0.76</td>
<td>1.74</td>
</tr>
<tr>
<td>Hardness, N</td>
<td>62.74</td>
<td>104.28</td>
</tr>
<tr>
<td>Oil absorption, %</td>
<td>5.87</td>
<td>17.13</td>
</tr>
<tr>
<td>Water stability, %</td>
<td>13.14</td>
<td>24.34</td>
</tr>
</tbody>
</table>

1. commercial feed: a commercial feed product, 3mm diameter, oil coated, the same recipe as Diet A

Table 5 Absolute Average deviation (AAD%), $R^2$ value and RMSE of equation (4) in regression of Diet-A recipe extrusion data and prediction of pellet quality parameter for extra recipe extrusion

<table>
<thead>
<tr>
<th></th>
<th>Diet-A</th>
<th>Extra</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>AAD%</td>
<td>$R^2$</td>
</tr>
<tr>
<td>Bulk density, g/l</td>
<td>4.13</td>
<td>0.51</td>
</tr>
<tr>
<td>Water stability, %</td>
<td>13.67</td>
<td>0.32</td>
</tr>
<tr>
<td>Hardness, N</td>
<td>7.41</td>
<td>0.60</td>
</tr>
<tr>
<td>Oil absorption, %</td>
<td>10.43</td>
<td>0.89</td>
</tr>
<tr>
<td>Table 6</td>
<td>RSM model results with significant terms for pellet physical quality parameters. Diet-A recipe extrusion *</td>
<td></td>
</tr>
<tr>
<td>---------</td>
<td>---------------------------------------------------------------------------------------------------------------</td>
<td></td>
</tr>
<tr>
<td>Response</td>
<td>Equation</td>
<td></td>
</tr>
<tr>
<td>Bulk density¹, g/l</td>
<td>[ Y = 617.3 - 9.51 SS - 37.3 T + 10.31 SS \times T ]  &lt;0.00  0  16.5  1.8</td>
<td></td>
</tr>
<tr>
<td>Water stability², %</td>
<td>[ Y = 16.75 - 0.610 M - 1.42 SS - 1.617 T - 1.225 M \times SS + 1.9 M ]  0.53</td>
<td>0.18</td>
</tr>
<tr>
<td>Hardness, %</td>
<td>[ Y = 98.38 - 12.529 T - 9.284 T^2 ]  0.06153</td>
<td>11.11</td>
</tr>
<tr>
<td>Oil absorption, %</td>
<td>[ Y = 92.25 - 14.625 M + 35.375 T + 5.875 SS - 10.156 M \times SS + 21.25 T^2 ]  0.06153</td>
<td>11.11</td>
</tr>
<tr>
<td>HDurability, % fine</td>
<td>[ Y = 1.581 - 0.105 M + 0.225 T + 0.14 \times MT - 0.132 SS^2 - 0.048 SS \times T - 0.123 T^2 ]  0.06153</td>
<td>11.11</td>
</tr>
</tbody>
</table>

* all the values in the equation are based on the normalized or coded values (M, SS and T), which can be calculated/converted using, \[ M = \frac{m - (m_{\text{max}} + m_{\text{min}})}{(m_{\text{max}} - m_{\text{min}})/2} \] \(SS = \frac{ss - (ss_{\text{max}} + ss_{\text{min}})}{(ss_{\text{max}} - ss_{\text{min}})/2} \) and \(T = \frac{t - (t_{\text{max}} + t_{\text{min}})}{(t_{\text{max}} - t_{\text{min}})/2} \), respectively. Where \( m \): moisture content, g/g, \( ss \): screw speed, rpm, \( t \): temperature, °C, are actual values. Subscripts min and max are the minimum and maximum values of process conditions (experimental ranges). RMSE: root mean square error

<table>
<thead>
<tr>
<th>Table 7</th>
<th>Equation (4) coefficients obtained from regression extrusion trial data</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coefficient</td>
<td>Hardness N</td>
</tr>
<tr>
<td>(k_1) (Fishmeal)</td>
<td>0.06153</td>
</tr>
<tr>
<td>(\eta_1) (SPC)</td>
<td>40.58</td>
</tr>
<tr>
<td>(\eta_2) (wheat gluten)</td>
<td>21.00</td>
</tr>
<tr>
<td>(\eta_3) (wheat)</td>
<td>19.01</td>
</tr>
<tr>
<td>(\eta_4) (other)</td>
<td>20.40</td>
</tr>
<tr>
<td>(\Delta E)</td>
<td>13.83</td>
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</table>

<p>| | 1571.75 | 117.82 | -4493.61 | - | -1850.19 | - |</p>
<table>
<thead>
<tr>
<th>$a$</th>
<th>$\alpha$</th>
<th>$\eta_1$</th>
<th>$\eta_5$</th>
<th>$\eta_6$</th>
<th>$\eta_7$</th>
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<tbody>
<tr>
<td>$\alpha$</td>
<td>7.440E-3</td>
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<td>0.2671</td>
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<td>$k_2$</td>
<td>0.3662</td>
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<td>-6.0581</td>
<td>1.554</td>
<td>-</td>
</tr>
</tbody>
</table>

$^1$ Bulk density, $^2$ Oil absorption capacity, $^3$ Water stability at 90min, DORIS: DORIS value.

*$\eta_1$-$\eta_7$: viscous coefficients for fishmeal, SPC, wheat gluten, wheat and other, respectively

**Highlights**

1. Lab extrusion trials for Atlantic salmon feed
2. Pellet qualities of the lab extrusion trials were like a commercial feed
3. An empirical model was better than Response Surface Method in data analysis
4. An empirical model can predict pellet qualities of a similar recipe extrusion