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RESEARCH ARTICLE

Influence of apple pomace inclusion on the process of animal feed pelleting

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Influence of apple pomace inclusion on the process of animal feed pelleting

Apple pomace (AP) is the main by-product of apple juice production. Large amounts of this material disposed to landfills can cause serious environmental problems. One of the solutions is to utilize AP as animal feed. The aim of this study was to investigate the impact of dried AP inclusion into model mixtures made from conventional feedstuffs, on pellet quality and pellet press performance. Three model mixtures, with different ratios of maize, sunflower meal and AP, were pelleted. The response surface methodology was applied in designing the experiment. The simultaneous and interactive effects of apple pomace share (APS) in the mixtures, die thickness (DT) of the pellet press and initial moisture content of the mixtures (M), on pellet quality and production parameters were investigated. Principal component analysis (PCA) and standard score analysis were applied for comprehensive analysis of the experimental data. The increase in APS led to an improvement of pellet quality parameters-pellet durability index (PDI), hardness (H), and proportion of fines in pellets. The increase in DT and M resulted in pellet quality improvement. The increase in DT and APS resulted in energy consumption of the pellet press. APS was the most influential variable for PDI and H calculation, while APS and DT were the most influential variables in the calculation of pellet press energy consumption. PCA showed that the first two principal components could be considered sufficient for data representation. In conclusion, addition of dried AP to feed model mixtures significantly improved the quality of the pellets.

Keywords: apple pomace, alternative feedstuff, pelleting, pellet quality, production parameters

Introduction

Nowadays, there is an increasing global trend in animal feed production towards more efficient utilization of alternative feedstuffs. Food industry by-products have a significant potential for utilization in animal nutrition. Apple pomace (AP) is the main by-product of apple juice production, generated during pressing of apples for juice extraction. Several million tonnes of AP are generated worldwide every year (Dhillon et
al. 2013). AP represents a heterogeneous mixture of apple peel, seeds, core, calyx, stems and exhausted soft tissue, and it accounts for about 30% of the weight of processed apples (Vendruscolo et al. 2008). Being an organic material with high moisture content (75–85%), it is susceptible to microbial contamination, uncontrolled fermentation and rapid spoilage (Bhushan and Gupta 2013). Direct disposal of large amounts of this material to landfills causes serious environmental pollution, but also a waste of resource (Wang et al. 2007). Although low in protein and fat content, AP still has considerable nutritional value, being good source of soluble carbohydrates (such as fructose, glucose, sucrose), polysaccharides referred as dietary fibre (cellulose, hemicellulose, pectin), polyphenols and minerals (Dílas et al. 2009; Abdollahzadeh et al. 2010; Parmar and Rupasinghe 2013). Since it is highly perishable, fresh AP has been used only at the local level as palatable feed for cattle and sheep (Crawshaw 2009). Preservation of AP can be achieved by ensiling or drying (Pirmohammadi et al. 2006). Several authors demonstrated that dried AP could be ground and used as an ingredient in rations for piglets (Gutzwiller et al. 2007), broilers (Ayhan et al. 2009), finishing lambs (Taasoli and Kafilzadeh, 2008) and lactating cows (Macgregor 2000).

In order to define dried AP as a new, alternative feedstuff, it is necessary to examine the possibility of its utilization in the industrial production of animal feed. Pelleting is a widely used thermal processing method in the animal feed industry. Pelleted feed provides numerous advantages compared to feed in mash form, such as: increased bulk density, enhanced handling and flow properties necessary for proper transportation in conveying equipment and discharging from silos, decreased dustiness of the material and feed wastage, decreased ingredient segregation and selective feeding, improved nutritional quality and animal performance (Thomas and van der Poel, 1996; Behnke 2001). On the other hand, pelleting process involves additional
equipment, energy consumption and maintenance costs. In order to actually use the advantages that pelleted feed provides, production of high quality pellets needs to be regarded as high priority. The term “pellet quality” most commonly refers to the physical quality of pellets, i.e. the ability of pellets to withstand attrition and fractures during handling and transportation in trucks and pneumatic conveying equipment (Abdollahi et al 2013). Poor quality pellets generate a high proportion of fines and dust, which causes segregation and arching in hoppers and a direct loss of feed (Aarsseth 2004). In addition, feeding low quality pellets can diminish the benefits of pelleting in terms of feeding efficiency (Behnke 2001).

Pelleting process and pellet quality are influenced by a number of factors, such as: feed ingredients, particle size, conditioning, die specifications, cooling and drying, which can be manipulated in order to achieve good quality pellets, with rational energy consumption. According to the research conducted by Maslovarić et. al. 2015, very good pellet quality was achieved when dried AP alone was pelleted. However, the impact of dried AP, as a feed ingredient in feed mixtures, on pellet quality and production parameters of the process has not been studied so far. The aim of the present study was to investigate the impact of dried AP inclusion into model mixtures made from conventional feedstuffs, on pellet quality and pellet press performance. It is important to point out that apple pomace used in this study corresponds to the apple pomace listed in the Catalogue of feed materials given by the Commission regulation (European Union) No 68/2013, which is another reason for better characterisation of this by-product in terms of its utilization as a feed ingredient.

The specific objective of this study was to examine the influence of the initial moisture content of the mixture and die thickness of the pellet press, on pellet quality and production parameters for each of the model mixtures. The response surface
methodology (RSM) was used in this study since it has been proven as a useful method for determining the influence of process variables on a group of dependent parameters. In order to provide a more comprehensive analysis of the obtained data, multivariate statistic techniques were applied, such as: principal component analysis (PCA) and standard score (SS) analysis.

**Materials and methods**

The experiments were conducted at the Feed to Food pilot-plant, at the Institute of Food Technology in Novi Sad (Serbia).

Three raw materials were used for the preparation of the model mixtures: maize and sunflower meal, as conventional feedstuffs, and dried AP, as an alternative feedstuff. All raw materials originated from the territory of the Republic of Serbia. AP was provided in the dry form by the fruit processing factory "Vino Župa" (Aleksandrovac). Drying was performed in a rotary drum dryer to decrease the moisture content to less than 100 g kg$^{-1}$, so that it could be safely stored until conducting the experiment.

Three model mixtures, with different levels of AP were prepared in order to evaluate the effects of AP on pellet quality and the pelleting process. Model mixture AP 0 consisted only of maize (750 g/kg) and sunflower meal (250 g/kg). Model mixture AP 100 consisted of maize (650 g/kg), sunflower meal (250 g/kg) and 100 g/kg of AP, while model mixture AP 200 consisted of maize (550 g/kg), sunflower meal (250 g/kg) and 200 g/kg of AP.

**Chemical analysis**

Chemical composition of dried AP was determined within the previous research conducted by Maslovarić et al. (2015). Model mixtures were analysed for moisture,
crude ash, crude protein, crude fat, crude fibre and total sugar content. All analyses were performed in triplicate.

The moisture content was determined by vacuum oven drying at 100°C, in accordance with AOAC (Association of Official Analytical Chemists) Method 934.01 (AOAC 1998).

The crude ash content was determined according AOAC Method 942.05 (AOAC 2000) for animal feed.

The crude protein content was determined by Kjeldahl method according to the AOAC 2001.11 method (AOAC 2002). Digestion and distillation were performed in BÜCHI Labortechnik AG, Switzerland K-350 and K-436 units, respectively.

The crude fat content was determined accordance with in accordance with AOCS (American Oil Chemists Society) Method Ba 3–38 24. Soxhlet extraction was performed with a Büchi 810 Soxhlet fat extraction apparatus (Soxtect system HT, 1043 Extraction Unit, Foss Tecator AB, Höganäs, Sweden).

The crude fibre content was determined by filter bag technique, Ankom Technology Method 7, according to AOCS Approved Procedure Ba 6a-05.

Total sugars were determined according to the Luff–Schoorl method described in the Rulebook on methods for physical and chemical analyses used in quality control of cereals, milling products, pasta and deep freeze dough the Official Gazette of SFRY, No. 74/88 (Rulebook, 1988). The method is based on the reduction of CuSO4 from Luff solution to Cu2O in the presence of reducing sugars. The amount of the Cu2O is equivalent to the amount of reducing sugars. The unspent amount of cupric ion is titrated with sodium thiosulphate solution. The quantity of reducing sugar is estimated by the table, according to the difference between the consumption of sodium thiosulphate solution for the blank titration and sample titration. In order to determine
total sugars content, the sample is first subjected to hydrolysis, in order to decompose sucrose (non-reducing sugar) to glucose and fructose (reducing sugars).

The sample was dissolved in water and the solution was clarified with Carrez solutions I and II.

The following reagents were used:

- **Luff solution**: CuSO₄ 5H₂O (Lach-Ner, Czech Republic) - 25g dissolved in 100 ml water + citric acid C₆H₈O₇ H₂O (Merck Millipore, Germany) - 50 g dissolved in 50 ml water + Na₂CO₃ (Lach-Ner, Czech Republic) - 143 g dissolved in 300 ml warm water. These solutions were mixed together in a volumetric flask, and diluted to 1000 ml with water

- **Hydrochloric acid concentrate** (Sigma-Aldrich, Germany) (for hydrolysis)

- **Carrez I solution**: 21.95 g Zn(CH₃COOH)₂·2H₂O + 3g CH₃COOH 3H₂O (Lach-Ner, Czech Republic) dissolved in water and diluted to 100 ml

- **Carrez II solution**: 10.6 g K₄Fe(CH₆)₆ 3H₂O (Lach-Ner, Czech Republic) dissolved in water and diluted to 100 ml

- **Sodium thiosulphate solution**, 0.1mol/l (Lach-Ner, Czech Republic)

- **Potassium iodide solution**: 30 % (w/v) (Sigma-Aldrich, Germany)

- **Starch solution**: 5g soluble of starch dissolved in 30ml water and added to 1 l of boiling water (for titration)

- **Sodium hydroxide solution** 0.1 mol/l; Sodium hydroxide, min 98% (Lach-Ner, Czech Republic)

- **Hydrochloric acid solution** 0,1 mol/l; Hydrochloric acid concentrate (Sigma-Aldrich, Germany)
Preparation of the material for the pelleting process

The ingredients of the model mixtures were ground with a laboratory hammer mill (ABC Engineering, Pančevo, Serbia), with a 4 mm diameter of sieve openings.

A twin-shaft paddle mixer (SLJ070.2, Muyang, China) was used for mixing and conditioning the material. The model mixtures were conditioned only by water addition to achieve targeted moisture of 130, 150, and 170 g kg⁻¹, respectively. Water was added to the mixer throughout the nozzles positioned above the mixing pedals, which ensured uniform distribution of water.

Pelleting of the model mixtures

The prepared model mixtures were pelleted on a flat-die laboratory pellet press (Pellet Press 14-175; Amandus Kahl, Germany). Three different thicknesses of the pellet press die were used (18, 24, and 30 mm), while the diameter of die openings was 6 mm, regardless of the die thickness.

The temperature of the pellet press die was measured by a Pt 100 temperature sensor and read out from the pellet press display. The pellet press throughput had been set at 50 kg h⁻¹. Specific energy consumption of the pellet press (Esp), expressed as kilowatt-hours per tonne (kWh t⁻¹), was determined from pellet press energy consumption and press throughput. The specific energy consumption of pellet press (kWh t⁻¹) was calculated according to the equation (1):

\[
Esp = \frac{E - E_0}{Q} \cdot 1000
\]

(1)

Where E is the energy consumption during pelleting of the material (kW), E₀ is the energy consumption during the idle running of the pellet press (kW) and Q is material throughput (kg h⁻¹).
Immediately after the pelleting process, proportion of fines (FP) in the pelleted mixtures was determined by measuring the mass of the pellets before and after sieving on a 4.8 mm diameter sieve (sieve opening diameter = 0.8 x pellet diameter).

The pellets were cooled to room temperature with ambient air, in a vibrating drier/cooler (model FB 500 x 200, Amandus Kahl, Germany), and stored for 24 hours prior to determination of physical quality parameters.

**Determination of the physical quality of pellets**

The cooled pellet samples from each treatment were used to determine bulk density (BD) (kg m\(^{-3}\)), pellet durability index (PDI\(_{24}\))\(^2\) and pellet hardness (H).

Bulk density (BD) of the pelleted mixtures was determined with a bulk density tester, Tonindustrie, West und Goslar, Germany.

Pellet durability was determined with a New Holmen Pellet Tester (NHP 100, TekPro Ltd, Norfolk, UK). In this test 100 g of sieved pellet samples were placed in a pyramid-shaped perforated chamber and introduced into a stream of air for 30 s, thereby bumping into each other and the walls of the chamber (Abdollahi, 2013). After the test, pellet samples were sieved again with a 4.8 mm diameter sieve. Pellet durability was expressed as pellet durability index (PDI\(_{24}\)), which represents the mass after the test to mass before the test ratio.

Pellet hardness (H) was determined with manually operated compression pellet tester “Pellet Hardness Tester“, AMANDUS KAHL GmbH & Co. KG, Germany, by measuring the force of the first fracture of individual pellets. The tester was equipped with 3.5 mm spring, which comprises the pressure range from 0 to 100 kg.

\(^2\) Number in subscript denotes time in hours between cooling and testing
All measurements were performed with three repetitions, except for pellet hardness which was measured for 20 pellet samples per treatment.

**Statistical analysis**

Descriptive statistical analysis of the data for each model mixture included determination of the mean and standard deviation (SD).

The experimental data used for the study of experimental results were obtained using a Box and Behnken’s fractional factorial (3 level-3 parameter) experimental design, with 15 runs (1 block), according to RSM, considering three factors (independent variables): apple pomace share (APS), die thickness of the pellet press (DT) and initial moisture content of the mixture (M). In this way, 15 different pellet samples, resulting from 15 experimental treatments, were obtained and submitted to further analysis. **Independent experimental factors for each of the samples are shown in Table 1.**

[Table 1 near here] rate

According to the available literature, dried apple pomace can be used in different rates as an ingredient in feed mixtures, e.g. 120 g kg\(^{-1}\) in dairy cows feed mixtures (Tiwari et al., 2008); 100 g kg\(^{-1}\) in broiler mixtures (Ayhan et al. 2009) or 200 g kg\(^{-1}\) when supplemented with enzymes (Matoo et al., 2001); 80 g kg\(^{-1}\) in mixtures for fattening pigs (Pieszek, 2017). The share of apple pomace in model mixtures (0 to 200 g kg\(^{-1}\)) was chosen in accordance with the aforementioned findings, so that the results of this study could be applied in practice.

Moisture content of 130 g kg\(^{-1}\) was the lowest possible in this investigation because this moisture content, in combination with low inclusion level of AP and low die thickness, resulted in very low quality of pellets. Further decrease of moisture content made pellet production impossible, i.e. there was no binding of particles during
pelletizing process. When pelleting the mixture with moisture content higher than 170 g kg\(^{-1}\) and no AP, the quality of the obtained pellets was very low. Additionally, moisture content higher than 16–17 g kg\(^{-1}\) is not recommendable because the produced pellets would have high moisture content and require drying, which would result in much higher energy consumption per unit mass of pellets. In this way, the highest possible range of moisture content was applied. Furthermore, the mean value of moisture content, (150 g kg\(^{-1}\)) was chosen according to the requirements of Box-Behnken’s experimental design that was used in this study.

Die thickness of 18 mm was the smallest available in our laboratory, while die thickness larger than 30 mm resulted in the extreme increase of specific energy consumption and in blocking of pellet press when model mixtures with low moisture content were pelleted. Additionally, the mean value of die thickness, i.e. 24 mm, was applied according to the requirements of Box-Behnken’s experimental design.

The RSM equations describe the effects of the test variables on the observed responses, determine test variables interrelationships and represent the combined effect of all test variables in the observed responses, enabling the experimenter to make efficient exploration of the process (Kuehl 2000; Madamba 2002; Brlek et al. 2013). The following second order polynomial (SOP) models were developed to relate six responses (Y) and three process variables (X) (equation (2)):

\[
Y_k = \beta_{k0} + \sum_{i=1}^{3} \beta_{ki} \cdot X_i + \sum_{i=1}^{3} \sum_{j=1}^{3} \beta_{kij} \cdot X_i^2 + \beta_{k12} \cdot X_1 \cdot X_2, \tag{2}
\]

Where: \(\beta_{k0}, \beta_{ki}, \beta_{kii}, \beta_{k12}, \) are constant regression coefficients; \(Y_k\), are either FP, PDI\(_{24}\), H, BD, Td or Esp; \(X_1\)–APS; \(X_2\)–DT and; \(X_3\)–M.
Analysis of variance (ANOVA) was conducted to show the significant effects of independent variables to the responses, and to show which responses were significantly affected by the different treatment combinations.

The SS analysis was introduced as a useful method for comparison different characteristics of various samples determined by multiple measurements, where samples are ranked based on the ratio of raw (particular) data and the extreme values of the measurements. This method enables comparison of data with different scales and units, by transforming them into normalised scores. The normalised scores represent dimensionless quantity derived by subtracting the minimum value from the raw data, or by subtracting the raw data from the maximum value, divided by the difference between maximum and minimum value, according to the equations (3) and (4) described by Brlek et al. (2013):

\[
x_i = \frac{x_i - \min_{i} x_i}{\max_{i} x_i - \min_{i} x_i}, \text{ in case of “the higher, the better” criteria, or}
\]

\[
x_i = \frac{\max_{i} x_i - x_i}{\max_{i} x_i - \min_{i} x_i}, \text{ in case of “the lower, the better” criteria}
\]

where \(x_i\) represents the raw data.

The sum of normalized scores of a sample for different measurements, when averaged, gives a single unit less value, termed as SS (Čolović et al. 2015). The higher, the better” criteria was applied to PDI, H and BD, while “the lower, the better” criteria was applied to FP, Esp and Td.
The principal component analysis (PCA) was used to discover the possible correlations between measured parameters and to classify and discriminate different samples.

The evaluation of response surface methodology (RSM), ANOVA and PCA analyses was performed using (Data Analysis Software System), v. 10, StatSoft, Inc., Tulsa, OK, USA (2010, http://www.statsoft.com).

**Results and discussion**

Chemical composition of dried apple pomace and model mixtures is presented in Table 2. The increase in AP share caused a decrease in protein and fat content in the model mixtures. Such result was expected, given that AP is a poor source of these nutrients.

On the other hand, model mixtures AP 10 and AP 20 had higher sugar and fibre content compared to mixture AP 0, that consisted only of maize and sunflower meal.

[Table 2 near here]

**Descriptive statistics and SS analysis**

Descriptive statistics of the pellet press production parameters and parameters of pellet physical quality for each of the mixtures are presented in Table 3. It is obvious that the increase in AP share caused an improvement of pellet quality parameters. PDI value increased from 45.36% to 96.35% when the AP content increased from 0 to 200 g kg⁻¹, respectively.

[Table 3 near here]

These findings are in line with the results obtained in the pelleting process of each model mixture and calculated SS values (Table 4). According to the results given in Table 4, rather low pellet quality was obtained for the model mixture with no AP added (APS=0), especially regarding PDI values. On the other hand, all pellet samples of the model mixtures AP 100 and AP 200 had very good pellet quality, with PDI
values above 90% and low FP. Values of SS above 0.70 indicated high standard in the
desired production parameters (Esp, Td) and pellet quality parameters (PDI, H, FP,
BD). According to the SS analysis, the optimal values for pellet quality and production
parameters were obtained for sample No. 6 (highlighted in bold), i.e. model mixture AP
100 (PDI=97.78%; FP=0%; H=5.12; BD=589.60 kg m⁻³; Esp=17.60 kWh t⁻¹; Td=45.60
⁰C), for DT=30 mm and M=170 g kg⁻¹. Very good results were obtained for samples
No. 8 (model mixture AP 200, for DT=24 mm and M=170 g kg⁻¹; SS=0.72), No. 10,
(model mixture AP 200, for DT=30 mm and M=150 g kg⁻¹; SS=0.66) and No. 11
(model mixture AP 200, DT=24 mm and M=150 g kg⁻¹; SS=0.74), while pellets with SS
values below 0.50 were attributed with poorer physical properties and production
parameters. These results can be explained by higher fibre and sugar content in the
model mixtures containing AP.

[Table 4 near here]

Water-soluble fibres usually raise the viscosity of the feed. Viscous materials
may act as a coating around feed particles and enhance their binding, thus improving
pellet durability and hardness (Loar and Corzo 2011). According to Thomas et al.
(1998), insoluble plant fibres may have beneficial effect on the pelleting process since
they have the capability of entangling and folding between different particles or strands
of fibre. On the other hand, their stiffness and elasticity may impede good contact
between particles or fibres and induce weak spots in pellets, thus making them prone to
fractures. Zimonja et al. (2008) reported that inclusion of supplemental fibrous material
in the form of finely ground oat hulls (140 g kg⁻¹), to wheat and oat based broiler diets,
improved pellet durability. On the contrary, pellet durability decreased when coarsely
ground oat hulls were added to the same diets. Buchanan and Moritz (2009) recorded
that pellet durability decreased when 20 g kg⁻¹ and 40 g kg⁻¹ of oat hulls were added to
maize and soybean meal-based broiler diets. The addition of 50 g kg$^{-1}$ fibre in the form of cellulose to the same diet increased pellet durability by 8%, and decreased bulk density and percentage of fines in pellets. According to previously stated, it can be concluded that not only the amount, but also the form of supplemental fibre (pure fibre or fibrous material, finely or coarsely ground) can significantly affect pellet quality. In the present study, pellet durability was drastically increased by adding 100 and 200 g kg$^{-1}$ of dried AP to the model mixtures, even though it was not finely ground, which suggests that soluble sugars also contributed to the pellet quality improvement. Part of sugars may solubilize during conditioning and pelleting. Thus, sugars may have a binding effect within the pellet due to the formation of the solid bridges created by their re-crystallisation or glass transition after cooling/drying of pellets (Thomas et al. 1998).

According to the results presented in Table 3 and Table 4, addition of AP to the mixtures resulted in increased die temperature and increased energy consumption of the pellet press. When pelleting model mixtures with 100 and 200 g kg$^{-1}$ AP, energy consumption of the pellet press was increased by 17.5 and 26.7%, respectively, compared to energy consumption when pelleting model mixtures without AP 0. Such results were expected, due to the higher fibre content of AP 100 and AP 200 mixtures. Higher fibre content caused additional resistance to the compression in the die channels and, consequently, increased frictional heating of the die.

It can be observed that the best results in terms of pellet quality and production parameters were obtained for the samples produced with higher moisture contents and by using thicker dies (greater L/D ratio) (Table 4). The optimal result (sample No. 6) was obtained by using the thickest die and with the highest initial moisture content of the mixture. Greater die thickness resulted in improved pellet quality due to longer retention time of the material under elevated pressure, which enabled better bonding
between feed particles. The positive effect of increased die thickness on pellet durability was confirmed in the studies conducted by Buchanan (2008) and Miladinović and Svihus (2005). Čolović et al. (2010) reported increased pellet hardness, when increasing die thickness, while PDI was not significantly affected. In the research conducted by Fairchild and Greer (1999), an increase of the initial moisture content of the mash (finishing hog feed) from 120 to 150 g kg\(^{-1}\) by adding water into the mixer, resulted in significant improvement of pellet durability and decreased energy consumption of the pellet press. Similar effects were observed by Moritz et al. (2003), when 25 and 50 g kg\(^{-1}\) moisture was added to broiler feed prior to pelleting. Moreover, Fairchild and Greer (1999) reported that positive effect of moisture addition on PDI value was more pronounced when water was added in the liquid form compared to the addition of water in the form of steam, which supports the approach of the present study.

The results of the present study suggest that higher moisture content provided better sugar and soluble fibres solubilisation, as well as disruption of the rigid structure of insoluble fibres, which significantly contributed to the quality of pellets.

On the other hand, it can be observed that production of pellets containing AP by using thicker dies required higher energy consumption and caused higher die temperatures. This is due to the combination of the effects of the longer press way and higher fibre content, which caused more friction between the material and die channel walls. These results are in line with those obtained by Čolović et al. (2010). Increased moisture content diminished these phenomena due to its lubricating effect and softening the mash (Moritz et al. 2003).
**Principal component analysis (PCA)**

The PCA allows a considerable reduction in a number of variables in a complex data set to and the detection of the structure in the relationship between measured (examined) parameters and different process variables that give complimentary information (Cvetković et al., 2015).

All the samples had different APS, DT and M, described according to the experimental design, as predicted by the PCA score plot (Figure 1).

[Figure 1 near here]

The full auto-scaled data matrix presented in Table 4, for variables APS (0, 100 or 200 g kg\(^{-1}\)), DT (18, 24 or 30 mm) and M (130, 150 or 170 g kg\(^{-1}\)), and response variables FP, PDI\(_{24}\), H, BD, Td and Esp were submitted to PCA. For visualizing data trends and the discriminating efficiency of the used descriptors, a scatter plot of samples using the first two principal components (PCs) issued from PCA of the data matrix was obtained (Figure 1). As it can be seen, there is a neat separation of the 15 samples of apple pellets with differentiation of production parameters, according to the experimental design. Quality results show that the first two principal components, accounting for 88.07% of the total variability can be considered sufficient for data representation. Concerning parameters of quality, FP (which contributed 16.2% of the total variance, based on correlations) was the strongest positively influential variable for the first principle component calculation, while PDI (16.7%), H (17.3%), Td (12.9%), Esp (15.2%) and SS (12.7%) had a negative effect in the formulation of the first principal component. The most positively influential parameter in the calculation of the second principal component was SS (which explained 23.2% of the total variance), while the most negative influence was noticed by BD (23.9%), Td (21.8%) and Esp (14.0%).
PCA biplot (Figure 1) clearly shows the influence of independent processing variables on pellet quality and parameters of the pelleting process. As it can be seen, an increase in the initial moisture content of the mixtures (M), die thickness (DT) and apple pomace share (APS), resulted in increased values of PDI and H and BD (positioned on the same side of the biplot in relation to M, DT and APS), and decreased FP (positioned on the opposite side of the biplot). Along with an increase in DT and APS, the parameters of the pelleting process, Td and Esp, also increased. These effects were discussed within previous sections of the paper.

PCA biplot showed good discrimination between the samples of the model mixture with no AP added (AP 0, positioned on the right side of the biplot) and the samples of the mixtures containing AP (AP 100 and AP 200, positioned on the left side of the biplot). This came as a result of significant differences between these groups of samples in terms of pellet quality.

The final score increased with the increase in M and APS, which is generally demonstrated by better SS results. The best scores have been obtained for sample No. 6 (with APS=100 g kg\(^{-1}\), DT=30 mm, M=170 g kg\(^{-1}\)), followed by samples No. 8, 10 and 11 (Table 4). These results coincide well with the PCA analysis, where these points are located in the upper left part of the graph, where SS increases.

**ANOVA and RSM analysis**

In this study, ANOVA was conducted in order to show the significant effects of the independent variables (APS, DT and M) on the observed response variables (FP, PDI, H, BD, Td and Esp). The investigated samples are characterized by relatively good pellet quality, especially in case of increased DT and M. According to ANOVA, the evaluation of the SOP model for FP is mostly affected by the linear terms of DT and M (statistically significant at p<0.05 level) (Table 5). The calculation of PDI and H was
affected the most by APS (statistically significant at p<0.01 level), and then by the linear terms of DT and M (p<0.05). The linear terms of DT and M were found to be most influential for mathematical model developing of BD (statistically significant at p<0.01 level). The linear terms of APS, DT and M were the most influential variables in the SOP model for the calculation of Td (statistically significant at p<0.01 level), while APS and DT were the most influential variables for the Esp calculation (p<0.01 level).

The predicted values were found to be in good agreement with the experimental values. The average error between the predicted values and experimental values was under 10%. According to Madamba (2002) the average error below 10% indicates an adequate fit for practical purposes. To verify the significance of the models, ANOVA was conducted and the results indicate that all the models are significant, with a minor lack of fit, suggesting that they adequately represent the relationship between the responses and the factors.

**Conclusions**

The results of the study on adding dried apple pomace to the maize and sunflower meal model mixtures, lead to the following conclusions:

- Adding 100 and 200 g kg⁻¹ apple pomace to the model mixtures significantly increased pellet quality in terms of durability (PDI-pellet durability index), hardness, percentage of fines and bulk density. Specific energy consumption of the pellet press was increased by 17.5 and 26.7%, but it was associated with over 40% and 50% increase in pellet durability, respectively.

- Increased die thickness and initial moisture content of the mixtures improved pellet quality. Increased die thickness resulted in higher die temperature and specific energy consumption of the pellet press.
Standard score analysis showed that the optimal process parameters (in terms of pellet quality and energy consumption) were as follows: dried apple share in the model mixtures—100 g kg\(^{-1}\), die thickness—30 mm, initial moisture content—170 g kg\(^{-1}\). Very good results were obtained with 200 g kg\(^{-1}\) dried apple pomace share, 24 mm die thickness and 170 g kg\(^{-1}\) initial moisture content.

A drastic improvement in pellet quality as a result of adding dried apple pomace to the model mixtures suggests that this by-product could be also used as a binder in pelleted feed production.

**Acknowledgements**

This article is a result of the research within the project III 46012, funded by the Ministry of Education, Science and Technological Development of the Republic of Serbia.
Table and figure captions

Table 1. Independent experimental factors and their levels

Table 2. Chemical composition of dried AP and model mixtures, (g kg⁻¹ dry matter basis)

Table 3. Descriptive statistics of the production parameters and pellet physical quality parameters.

Table 4. Pellet quality and production parameters of pelleting AP model mixtures.

Table 5. ANOVA table-effects of process parameters on of model mixtures pellet physical quality and production parameters of pelleting.

Figure 1. PCA biplot for pelleting parameters and pellet quality.

References


AOCS (2006). Official Method Ba 6a-84, AOAC 962.09. Approved Procedure Crude Fiber Analysis in Feeds by Filter Bag Technique


Madamba PS. 2002. The response surface methodology: an application to optimize dehydration operations of selected agricultural crops. LWT--Food Sci. and Technol. 35:584-592.


Rulebook on methods for physical and chemical analyses used in quality control of cereals, milling products, pasta and deep freeze dough, the Official Gazette of SFRY, No. 74/88, (1988) / Pravilnik o metodama fizičkih i hemijskih analiza za kontrolu...
kvaliteta žita, mlinskih i pekarskih proizvoda, testenina i brzo smrznutih testa "Službeni list SFRJ" br. 74/88, 1988 (in Serbian)


Tables:

Table 1. Independent experimental factors and their levels

<table>
<thead>
<tr>
<th>Experimental factor</th>
<th>Symbol</th>
<th>Low</th>
<th>Centre</th>
<th>High</th>
</tr>
</thead>
<tbody>
<tr>
<td>APS, g kg⁻¹</td>
<td>X₁</td>
<td>0</td>
<td>100</td>
<td>200</td>
</tr>
<tr>
<td>DT, mm</td>
<td>X₂</td>
<td>18</td>
<td>24</td>
<td>30</td>
</tr>
<tr>
<td>M, g kg⁻¹</td>
<td>X₃</td>
<td>130</td>
<td>150</td>
<td>170</td>
</tr>
</tbody>
</table>

APS—dried apple pomace share in the model mixture

DT—die thickness

M—initial moisture content of the model mixture

Table 2. Chemical composition of dried AP and model mixtures, (g kg⁻¹ dry matter basis)

<table>
<thead>
<tr>
<th></th>
<th>AP</th>
<th>AP 0</th>
<th>AP 10</th>
<th>AP 20</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture</td>
<td>79.6±0.10</td>
<td>107.1±0.21</td>
<td>103.6±0.23</td>
<td>100.1±0.13</td>
</tr>
<tr>
<td>Protein</td>
<td>63.2±0.26</td>
<td>169.5±0.01</td>
<td>164.9±0.15</td>
<td>157.5±0.13</td>
</tr>
<tr>
<td>Fat</td>
<td>24.5±0.12</td>
<td>42.9±0.02</td>
<td>39.9±0.03</td>
<td>38.8±0.02</td>
</tr>
<tr>
<td>Fibre</td>
<td>225.5±0.27</td>
<td>79.3±0.04</td>
<td>99.7±0.04</td>
<td>119.9±0.04</td>
</tr>
<tr>
<td>Ash</td>
<td>22.0±0.04</td>
<td>28.1±0.03</td>
<td>29.5±0.02</td>
<td>29.6±0.01</td>
</tr>
<tr>
<td>Starch</td>
<td>NA</td>
<td>557.8±0.44</td>
<td>481.5±0.46</td>
<td>405.9±0.53</td>
</tr>
<tr>
<td>Total soluble sugars, g kg⁻¹</td>
<td>319.5±0.23</td>
<td>43.3±0.10</td>
<td>73.7±0.19</td>
<td>103.7±0.04</td>
</tr>
</tbody>
</table>

AP—dried apple pomace

AP 0, AP 100, AP 200—model mixtures made from maize, sunflower meal and 0, 100 and 200 g kg⁻¹ dried apple pomace

The results are presented as mean ± standard deviation (SD)

NA—not analysed
Table 3. Descriptive statistics of the production parameters and pellet physical quality parameters

<table>
<thead>
<tr>
<th>Model mixture</th>
<th>Descriptive statistics</th>
<th>$T_d$ (°C)</th>
<th>$E_{sp}$ (kWh t$^{-1}$)</th>
<th>FP (%)</th>
<th>BD (kg m$^{-3}$)</th>
<th>H</th>
<th>PDI$_{24}$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AP 0</td>
<td>Average</td>
<td>43.70</td>
<td>14.27</td>
<td>4.61</td>
<td>580.44</td>
<td>2.50</td>
<td>45.36</td>
</tr>
<tr>
<td></td>
<td>SD</td>
<td>2.46</td>
<td>2.42</td>
<td>0.68</td>
<td>16.78</td>
<td>0.88</td>
<td>21.74</td>
</tr>
<tr>
<td>AP 100</td>
<td>Average</td>
<td>45.92</td>
<td>17.31</td>
<td>3.39</td>
<td>589.23</td>
<td>4.47</td>
<td>85.73</td>
</tr>
<tr>
<td></td>
<td>SD</td>
<td>2.72</td>
<td>2.57</td>
<td>2.40</td>
<td>30.65</td>
<td>0.90</td>
<td>17.15</td>
</tr>
<tr>
<td>AP 200</td>
<td>Average</td>
<td>47.25</td>
<td>19.48</td>
<td>1.63</td>
<td>593.97</td>
<td>4.75</td>
<td>96.35</td>
</tr>
<tr>
<td></td>
<td>SD</td>
<td>2.45</td>
<td>2.15</td>
<td>1.40</td>
<td>22.25</td>
<td>0.74</td>
<td>1.97</td>
</tr>
</tbody>
</table>

AP 0, AP 100, AP 200–model mixtures made from maize, sunflower meal and 0, 100 and 200 g kg$^{-1}$ dried apple pomace

$T_d$–die temperature, $E_{sp}$ - specific energy consumption, FP–proportion of fines, BD –bulk density of the pellets, H–the hardness of the pellets, PDI$_{24}$–pellet durability index determined 24h after cooling
<table>
<thead>
<tr>
<th>No.</th>
<th>APS (g kg(^{-1}))</th>
<th>DT (mm)</th>
<th>M (g kg(^{-1}))</th>
<th>FP (%)</th>
<th>PDI(_{24}) (%)</th>
<th>H kg m(^{-3})</th>
<th>BD</th>
<th>T(_d) (°C)</th>
<th>E(_{sp}) (kWh t(^{-1}))</th>
<th>SS</th>
<th>Polarity</th>
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</thead>
<tbody>
<tr>
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<td>170</td>
<td>3.25</td>
<td>78.20</td>
<td>4.21</td>
<td>555.97</td>
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<td>14.08</td>
<td>0.61</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>20</td>
<td>18</td>
<td>150</td>
<td>3.38</td>
<td>95.90</td>
<td>4.18</td>
<td>572.41</td>
<td>44.90</td>
<td>18.46</td>
<td>0.59</td>
<td>+</td>
</tr>
<tr>
<td>3</td>
<td>170</td>
<td>150</td>
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<td>42.50</td>
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<tr>
<td>5</td>
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<td>1.98</td>
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<tr>
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<td>150</td>
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<td>2.50</td>
<td>580.44</td>
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<td>14.27</td>
<td>0.47</td>
<td>-</td>
</tr>
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<td>200</td>
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<td>130</td>
<td>1.88</td>
<td>93.80</td>
<td>4.04</td>
<td>605.22</td>
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</tr>
<tr>
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<td>100</td>
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<td>150</td>
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<td>4.36</td>
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<td>0.65</td>
<td>+</td>
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<td>18</td>
<td>130</td>
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<td>50.76</td>
<td>3.03</td>
<td>568.20</td>
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<td>14.89</td>
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</table>

Polarity: ‘+’ = the higher the better criteria, ‘−’ = the lower the better criteria.

AP-dried apple pomace; APS—dried apple pomace share, DT—die thickness, M—initial moisture content of the mixture, FP—proportion of fines, PDI\(_{24}\)—pellet durability index determined 24h after cooling, H—pellet hardness, BD—bulk density of the pellets, T\(_d\)—die temperature, E\(_{sp}\)—specific energy consumption of the pellet press, SS—standard score.
Table 5. ANOVA table—effects of process parameters on of model mixtures pellet physical quality and production parameters of pelleting (sum of squares)

<table>
<thead>
<tr>
<th>Term</th>
<th>FP</th>
<th>PDI_{24}</th>
<th>H</th>
<th>BD</th>
<th>T_d</th>
<th>Esp</th>
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</thead>
<tbody>
<tr>
<td>APS</td>
<td>9.115</td>
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<td>450.000</td>
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<td>APS²</td>
<td>0.025</td>
<td>169.223</td>
<td>1.520</td>
<td>63.450</td>
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</tr>
<tr>
<td>DT²</td>
<td>3.386</td>
<td>212.140</td>
<td>0.496</td>
<td>175.430</td>
<td>0.103</td>
<td>2.038</td>
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<tr>
<td>M</td>
<td>10.773</td>
<td>441.847</td>
<td>2.101</td>
<td>2155.290</td>
<td>14.311</td>
<td>3.255</td>
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<tr>
<td>M²</td>
<td>2.260</td>
<td>2.201</td>
<td>0.293</td>
<td>520.052</td>
<td>0.410</td>
<td>10.121</td>
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<tr>
<td>APS × DT</td>
<td>0.950</td>
<td>145.625</td>
<td>0.002</td>
<td>490.623</td>
<td>0.302</td>
<td>0.016</td>
</tr>
<tr>
<td>APS × M</td>
<td>0.011</td>
<td>6.954</td>
<td>0.053</td>
<td>46.104</td>
<td>3.062</td>
<td>2.179</td>
</tr>
<tr>
<td>DT × M</td>
<td>0.567</td>
<td>368.870</td>
<td>0.176</td>
<td>22.043</td>
<td>2.560</td>
<td>2.061</td>
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<tr>
<td>Error</td>
<td>6.990</td>
<td>330.929</td>
<td>1.428</td>
<td>560.420</td>
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<td>4.381</td>
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<td>r²</td>
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<td>0.921</td>
<td>0.902</td>
<td>0.925</td>
<td>0.980</td>
<td>0.958</td>
</tr>
</tbody>
</table>

APS—apple pomace share (g kg⁻¹) in the model mixture, DT—die thickness (mm), M—initial moisture content of the model mixture (g kg⁻¹), FP—proportion of fines (%), PDI_{24}—pellet durability index determined 24 h after cooling (%), H—pellet hardness, BD—bulk density of the pellets (kg m⁻³), T_d—die temperature (°C), Esp—specific energy consumption of the pellet press (kWh t⁻¹), r²—coefficient of determination

*Significant at p<0.05 level, **Significant at p<0.10, level 95% confidence limit, error terms were found statistically insignificant
Figures:

Figure 1. PCA biplot for pelleting parameters and pellet quality

Td–pellet press die temperature (°C), Esp–specific energy consumption during pelleting of experimental mixtures (kWh t⁻¹), FP–proportion of fines (%), H–pellet hardness, PDI–pellet durability index determined 24 h after cooling (%), BD – bulk density of the model mixture (kg m⁻³), APS–apple pomace share in the model mixture (g kg⁻¹), DT–die thickness (mm), M–initial moisture content of the model mixture (g kg⁻¹)
<table>
<thead>
<tr>
<th>Experimental factor</th>
<th>Symbol</th>
<th>Low</th>
<th>Centre</th>
<th>High</th>
</tr>
</thead>
<tbody>
<tr>
<td>APS, g kg⁻¹</td>
<td>X₁</td>
<td>0</td>
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<td>200</td>
</tr>
<tr>
<td>DT, mm</td>
<td>X₂</td>
<td>18</td>
<td>24</td>
<td>30</td>
</tr>
<tr>
<td>M, g kg⁻¹</td>
<td>X₃</td>
<td>130</td>
<td>150</td>
<td>170</td>
</tr>
</tbody>
</table>

APS—dried apple pomace share in the model mixture

DT—die thickness

M—initial moisture content of the model mixture
Table 2. Chemical composition of dried AP and model mixtures, (g kg\(^{-1}\) dry matter basis)

<table>
<thead>
<tr>
<th></th>
<th>AP</th>
<th>AP 0</th>
<th>AP 10</th>
<th>AP 20</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture</td>
<td>79.6±0.10</td>
<td>107.1±0.21</td>
<td>103.6±0.23</td>
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<td>63.2±0.26</td>
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</tr>
<tr>
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<td>103.7±0.04</td>
</tr>
</tbody>
</table>

AP—dried apple pomace
AP 0, AP 100, AP 200—model mixtures made from maize, sunflower meal and 0, 100 and 200 g kg\(^{-1}\) dried apple pomace
The results are presented as mean ± standard deviation (SD)
NA—not analysed
Table 3. Descriptive statistics of the production parameters and pellet physical quality parameters

<table>
<thead>
<tr>
<th>Model mixture</th>
<th>Descriptive statistics</th>
<th>$T_d$ (°C)</th>
<th>$E_{sp}$ (kWh t$^{-1}$)</th>
<th>FP (%)</th>
<th>BD (kg m$^{-3}$)</th>
<th>H</th>
<th>PDI$_{24}$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AP 0</td>
<td>Average</td>
<td>43.70</td>
<td>14.27</td>
<td>4.61</td>
<td>580.44</td>
<td>2.50</td>
<td>45.36</td>
</tr>
<tr>
<td></td>
<td>SD</td>
<td>2.46</td>
<td>2.42</td>
<td>0.68</td>
<td>16.78</td>
<td>0.88</td>
<td>21.74</td>
</tr>
<tr>
<td>AP 100</td>
<td>Average</td>
<td>45.92</td>
<td>17.31</td>
<td>3.39</td>
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<td>4.47</td>
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<td>2.57</td>
<td>2.40</td>
<td>30.65</td>
<td>0.90</td>
<td>17.15</td>
</tr>
<tr>
<td>AP 200</td>
<td>Average</td>
<td>47.25</td>
<td>19.48</td>
<td>1.63</td>
<td>593.97</td>
<td>4.75</td>
<td>96.35</td>
</tr>
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<td></td>
<td>SD</td>
<td>2.45</td>
<td>2.15</td>
<td>1.40</td>
<td>22.25</td>
<td>0.74</td>
<td>1.97</td>
</tr>
</tbody>
</table>

AP 0, AP 100, AP 200—model mixtures made from maize, sunflower meal and 0, 100 and 200 g kg$^{-1}$ dried apple pomace

$T_d$—die temperature, $E_{sp}$—specific energy consumption, FP—proportion of fines, BD—bulk density of the pellets, H—hardness of the pellets, PDI$_{24}$—pellet durability index determined 24h after cooling
Table 4. Pellet quality and production parameters of the AP model mixtures’ pelleting

<table>
<thead>
<tr>
<th>No.</th>
<th>APS (g kg⁻¹)</th>
<th>DT (mm)</th>
<th>M (g kg⁻¹)</th>
<th>FP (%)</th>
<th>PDI₂₄ (%)</th>
<th>H</th>
<th>BD kg m⁻³</th>
<th>T₉₀ (°C)</th>
<th>Eₛₛ (kWh t⁻¹)</th>
<th>SS</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0</td>
<td>24</td>
<td>170</td>
<td>3.25</td>
<td>78.20</td>
<td>4.21</td>
<td>555.97</td>
<td>43.20</td>
<td>14.08</td>
<td>0.61</td>
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<tr>
<td>2</td>
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| Polarity | - | + | + | + | - | - |

AP-dried apple pomace; APS—dried apple pomace share, DT—die thickness, M—initial moisture content of the mixture, FP—proportion of fines, PDI₂₄—pellet durability index determined 24h after cooling, H—pellet hardness, BD—bulk density of the pellets, T₉₀—die temperature, Eₛₛ—specific energy consumption of the pellet press, SS—standard score.

Polarity: ‘+’ = the higher the better criteria, ‘-’ = the lower the better criteria.
Table 5. ANOVA table - effects of process parameters on of model mixtures pellet physical quality and production parameters of pelleting (sum of squares)

<table>
<thead>
<tr>
<th>Term</th>
<th>FP</th>
<th>PDI_{24}</th>
<th>H</th>
<th>BD</th>
<th>T_d</th>
<th>E_{sp}</th>
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<tr>
<td>APS</td>
<td>9.115**</td>
<td>1817.050+</td>
<td>4.898+</td>
<td>450.000**</td>
<td>25.205+</td>
<td>40.257+</td>
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<td>APS^2</td>
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<td>169.223</td>
<td>1.520**</td>
<td>63.450</td>
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<td>DT^2</td>
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<td>212.140</td>
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<td>175.430</td>
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<td>M</td>
<td>10.773*</td>
<td>441.847*</td>
<td>2.101*</td>
<td>2155.290+</td>
<td>14.311+</td>
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<td>M^2</td>
<td>2.260</td>
<td>2.201</td>
<td>0.293</td>
<td>520.052**</td>
<td>0.410</td>
<td>10.121*</td>
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<td>APS × DT</td>
<td>0.950</td>
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<td>0.002</td>
<td>490.623**</td>
<td>0.302</td>
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<td>APS × M</td>
<td>0.011</td>
<td>6.954</td>
<td>0.053</td>
<td>46.104</td>
<td>3.062*</td>
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<td>DT × M</td>
<td>0.567</td>
<td>368.870**</td>
<td>0.176</td>
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</table>

APS—apple pomace share (g kg^{-1}) in the model mixture, DT—die thickness (mm), M—initial moisture content of the model mixture (g kg^{-1}), FP—proportion of fines (%), PDI_{24} - pellet durability index determined 24 h after cooling (%), H—pellet hardness, BD—bulk density of the pellets (kg m^{-3}), T_d—die temperature (°C), E_{sp}—specific energy consumption of the pellet press (kWh t^{-1}), r^2—coefficient of determination

*Significant at p<0.05 level, **Significant at p<0.10, level 95% confidence limit, error terms were found statistically insignificant