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Multi-response surface optimisation of extrusion cooking to increase soluble dietary fibre and polyphenols in lupin seed coat

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Abstract
The seed coat of the legume lupin which is rich in insoluble dietary fibre is a major by-product in human food applications. Extrusion cooking has been demonstrated to increase desirable soluble dietary fibre in the Australian sweet lupin seed coat. In this study, processing condition of twin-screw extrusion cooking was optimised using a central composite rotatable design to increase soluble dietary fibre in lupin seed coat from 44.17 g/kg up to 113.69 g/kg dry basis. The high levels of polyphenols in the seed coat were retained. The optimal extrusion conditions which achieved maximum levels of soluble dietary fibre, total free phenolic content and total free individual phenolic content simultaneously were identified and validated. The extrusion cooking largely had no or slight effects on bioaccessibility and bioavailability of the selected minerals and individual polyphenol. The extrusion cooked lupin seed coat could be a natural antioxidant dietary fibre source for human consumption.

Keywords: lupin seed coat; extrusion cooking; response surface design; soluble dietary fibre; bioavailability
1. Introduction

The seed of legume lupin is a sustainable plant protein source for both livestock and humans (Johnson, Clements, Villarino, & Coorey, 2017). However, production of high-protein lupin kernel flour is accompanied by a large milling loss, with lupin seed coat being the main by-product. The seed coat accounts for 24% of the whole Australian sweet lupin seed (Lupinus angustifolius, ASL) and 18% of albus lupin (Lupinus albus) (Zhong et al., 2018). Nonetheless, the seed coat by-product could be an economical source of nutraceutical components, including natural dietary fibres and polyphenols (Zhong et al., 2018). Accordingly, several studies have developed alternative methods to utilise lupin seed coat into human foods, incorporating the seed coat into bakery food as dietary fibre ingredient for example (Tucek, 2009; Wandersleben et al., 2018).

Soluble dietary fibre (SDF) generally has more desirable health benefits compared with insoluble dietary fibre (IDF) (Elleuch et al., 2011). In this respect, over 80% of the whole lupin seed coat is IDF. Therefore, an extrusion cooking technology has been used to elevate the SDF level in the lupin seed coat (Zhong, Fang, Wahlqvist, Hodgson, & Johnson, 2019). In this previous study, fractional factorial design (FFD) was conducted as a first-order screening study to characterise those extrusion cooking parameters (independent factors) that significantly influenced SDF content. Extrusion temperature, total moisture in barrel and screw speed, were the main factors influencing the SDF increase (Zhong, Fang, et al., 2019). To obtain a more robust model to optimise the processing, a second-order study is required (Myers, Montgomery, & Anderson-Cook, 2016). There are multiple optimisation modelling designs, with central composite rotatable design which contains 5 factor levels, i.e. two factorial point (±1), two axial points (±α) and one center points (0), being most commonly used (Myers et al., 2016).

Lupin seed coat has considerable levels of minerals, with 67.5% of total calcium of the whole lupin seed being concentrated in the seed coat (Hung, Handson, Amenta, Kyle, & Yu, 1988). However, the total quantity of a nutrient does not necessarily reflect its available amount for human absorption (Ribas-Agusti, Martin-Belloso, Soliva-Fortuny, & Elez-Martinez, 2018). Karnpanit, Coorey, Clements, Benjapong, and Jayasena (2017) found that only 6% of calcium, 17% of iron, and 9% of zinc in whole ASL seeds were bioaccessible. Czubinski et al. (2019) indicated that up to 92% of the dominate polyphenol compound in lupin seed coat, apigenin-7-O-β-apiofuranosyl-6,8-di-C-β-glucopyranoside (Api-Apif-di-GlcP), could be release into digestion fluid from the raw whole ASL seed. The mineral content is expected to be unchanged before and after extrusion cooking. However, extrusion cooking disrupted lupin seed coat matrix, substantially decreased polyphenols that inhibit minerals absorption, modified dietary
fibre structures and converted IDF to SDF, as well as induced Maillard reaction, which can show positive or negative impacts on mineral absorption (Singh, Gamlath, & Wakeling, 2007; Zhong, Fang, et al., 2019).

The aim of the present study was to optimise extrusion cooking to simultaneously obtain high levels of soluble dietary fibre and polyphenols in lupin seed coat. A central composite rotatable design (CCRD) coupled with desirability function was used. Bioaccessibility and bioavailability of the selected individual polyphenols and minerals of the optimal extrusion cooked ASL seed coat were also investigated.

2. Materials and methods

2.1. Materials

The seed coat of Australian sweet lupin (Coromup, harvested in 2016) were provided by Coorow Seeds Company (Coorow, WA, Australia). About 10 kg of the seed coat was pre-dried in a 40 °C oven for 16 h to allow effective milling. The seed coat was milled using ZM 200 Retch Mill (Retsch Gmbh & Co, Haan, Germany) and passed through (>98 %) a 500 μm sieve. The seed coat flour was vacuum-packaged and stored at 4 °C before extrusion.

2.2. Experimental design

The central composite rotatable design (CCRD) was generated and analysed using the Design-Expert software (V11, Stat-Ease Inc. USA). Three independent factors which most significantly affected soluble dietary fibre content in the first-order model, namely extrusion temperature (°C), screw speed (rpm) and total moisture in barrel (%), and their actual levels were previously identified (Zhong, Fang, et al., 2019). Their ranges (i.e. factorial points) were 120/150 °C, 300/400 rpm and 30/40 % respectively (Table 1). The barrel has four independent temperature controlling zones (Figure S1). Unstable output and over-torque were observed when the temperature at zone 4 was higher than 130 °C. Therefore, the highest barrel temperature was set at zone 3. The barrel temperature profiles were 70/100/120/120°C (-1), 70/100/150/125°C (+1), 70/100/135/125°C (0), 70/100/110/110°C (-1.682), 70/100/160/125°C (+1.682), respectively. The experiments were conducted on two consecutive days.

Soluble dietary fibre (SDF), insoluble dietary fibre (IDF), total dietary fibre (TDF), total polyphenols content (TPC), and individual polyphenols of both free and bound polyphenol extracts were analysed. A polynomial quadratic regression equation (below) was used to describe the effects of the three factors on the responses:
\[ Y = \beta_0 + \sum_{i=1}^{3} \beta_i X_i + \sum_{i=1}^{3} \beta_{ii} X_i^2 + \sum_{i=1}^{2} \sum_{j=i+1}^{3} \beta_{ij} X_i X_j \]

Where \( Y \) represents the response; \( X_i \) and \( X_j \) are the selected independent factors (vary from 1 to 3); \( \beta_0 \) denotes the intercept coefficient, while \( \beta_i \), \( \beta_{ii} \), and \( \beta_{ij} \) are the first order, quadratic and interaction coefficients, respectively.

The global desirability function \((D)\) with relative “importance” degrees of the independent variables was performed on the Design-Expert software to simultaneously optimise the selected responses (Myers et al., 2016). Optimal solutions (Opti), which suggested the levels of independent variables to achieve the highest levels of responses, were generated by the design. Extrusion cooking was performed under the selected optimal solution, which had the highest \( D \) value, to validate the model.

2.3. Twin-screw extrusion operation

The extrusion cooking was performed on a co-rotating intermeshing twin-screw extruder (MPF 19:25, APV Baker Inc., England) as previously described (Zhong, Fang, et al., 2019). Dry feed rate was fixed at 4 kg per hour. About 400 g of extrudates of each run were collected. Real-time motor torque, barrel and die temperatures, and die pressure were recorded manually to calculate the specific mechanical energy (SME):

\[
SME \ (W \cdot h/kg) = \frac{\text{screw speed} \ (rpm) \times \text{torque} \ (%) \times 2200(W)}{500 \ (rpm) \times 100 \times \text{feed rate} \ (kg/h)}
\]

Where 500 rpm and 2200 W is the rated screw speed and motor power of the extruder respectively (Zhong, Fang, et al., 2019).

2.4. Extrudate analyses

2.4.1. Determination of dietary fibre composition

A K-TDFR kit (Megazyme Int., Wicklow, Ireland) was used to quantify the dietary fibre composition of the extrusion cooked lupin seed coats \((n = 20)\) and the raw seed coat (Zhong, Fang, et al., 2019).

2.4.2. Determination of total polyphenol content and individual polyphenols

Free and bound polyphenols were extracted as previously described (Zhong, Fang, et al., 2019). Total polyphenol content (TPC) in free (TFPC) and bound (TBPC) extracts were evaluated using Folin-Ciocalteu assay as described by Wu et al. (2017). The results were expressed as mg gallic acid equivalent per 100 gram dry sample (mg GAE/100 g db). Individual polyphenols in extracts were quantified following a validated HPLC-DAD method using authentic standards (Zhong, Wu, et al., 2019).
2.4.3. Mineral and polyphenol bioaccessibility and bioavailability of optimally extrusion cooked ASL seed coat

In the current study, bioaccessibility is defined as the percentage of the polyphenols/minerals which were released from the matrix and transferred into digestive fluid after ingestion. By contrast, bioavailability is the fraction of the compounds which is absorbed to the blood (dialysis tubing) comparing to the total amount in the raw sample (Versantvoort, Van de Kamp, & Rompelberg, 2004):

\[
\text{Bioaccessibility} (\%) = \frac{\text{Qty(tub)} + \text{Qty(sup)}}{\text{Qty(samp)}} \times 100
\]

\[
\text{Bioavailability} (\%) = \frac{\text{Qty(tub)}}{\text{Qty(samp)}} \times 100
\]

Where Qty (tub), Qty (sup) and Qty (samp) were the quantities of the compounds in dialysis tubing (i.e. bioavailable amount in the blood), supernatant (i.e. bioaccessible amount in digestive fluid) and the raw samples, respectively. Raw and extrusion cooked ASL seed coats under optimal (Opti) conditions were analysed.

2.4.3.1. In vitro digestion

A static in vitro digestion method was used (Minekus et al., 2014). Enzymes were α-amylase from Aspergillus oryzae (10065, 33.8 U/mg, Sigma-Aldrich, Australia), pepsin (PL082, 2500 U/mg, Chem-Supply, Australia) and pancreatin (PL378, Chem-Supply, Australia). NaHCO₃ (5.5 mL, 0.5 mol/L) and NaCl (5.5 mL, 9 g/kg) were added into a dialysis bag with a molecular mass cut off at 10 kDa (diameter 22 mm, 15 cm lengths, SnakeSkin, Thermo Fisher Australia), and the dialysis bag was put into the gastric chyme as described by Shumoy et al. (2017). Duplicate blank control was included during the in vitro digestion.

2.4.3.2. Mineral bioaccessibility and bioavailability

Dialysis tubing was collected, rinsed by ultrapure water, dried on an absorbent paper after digestion, then oven-dried to constant weight. Mineral content in the tubing was defined as Qty (tub). The resulting digestion mixture was centrifuged at 2750 × g at 4 °C for 5 min, yielding supernatant and pellet (Versantvoort et al., 2004). Supernatants from centrifuging were collected into tared glass tubes and freeze-dried to constant weight for determination of Qty (sup). Levels of calcium (Ca), sodium (Na), potassium (K), magnesium (Mg), zinc (Zn), iron (Fe) and copper (Cu) were determined using an inductively coupled plasma optical emission spectrometer (ICP-OES) (Varian, Palo Alto, USA) (Wu et al., 2016).
2.4.3.3. Polyphenol bioaccessibility and bioavailability

The suspension after digestion was acidified to pH 2 after intestinal digestion phase to stabilise the phenolic compounds and terminate the reactions (Pineda-Vadillo et al., 2016). Both the dialysis tubing and supernatant were freeze-dried, extracted and quantified as previously described (Section 2.5.2). The content of apigenin-7-O-β-apiofuranosyl-6,8-di-C-β-glucopyranoside (Api-Apf-di-Glc) was expressed as μg Vitexin equivalent (VE) /g db (Zhong, Fang, et al., 2019).

2.5. Statistical analysis

All results were presented on a dry basis (db) and expressed as mean ± standard deviation (n ≥ 2). The Design-Expert software (V11, Stat-Ease Inc. Minneapolis, MN, USA) was used to analyse the response data individually and obtain the most-fitting models. Each dependent variable (Section 2.2) was analysed separately, and their most-fitting models were established independently based on the following criteria: (1) model is significant with p being lower than 0.05; (2) lack-of-fit is insignificant with p > 0.10; (3) the adequate precision is greater than 4; (4) acceptable residual tests by examining diagnostic plots generated by the software (Myers et al., 2016). Independent-sample t-test was used to analyse the differences in responses between raw and extrusion cooked seed coat of each runs, as well as the mineral and polyphenol bioaccessibility and bioavailability between raw and optimally extrusion cooked samples. All statistics, including Pearson correlations, were performed using SPSS v23 (SPSS Inc., Chicago, Illinois, US). p < 0.05 was considered significant.

3. Results and discussion

3.1. Effect of extrusion cooking on lupin seed coat dietary fibre composition

3.1.1. Soluble dietary fibre (SDF)

As compared to raw lupin seed coat, the extrusion cooking significantly increased SDF content in the lupin seed coat, ranging from 67.29 to 103.78 g/kg db after extrusion cooking versus 44.17 g/kg db in the un-extruded sample (Table 1). A quadratic model which best fitted the criteria listed in Section 2.3.1 was “suggested” by the Design-Expert software to describe the collective impacts of the three independent variables on SDF (Table 2). The total moisture in the barrel had the most significant effects on SDF (p < 0.0001), followed by the barrel temperature (p = 0.0053). Barrel speed showed insignificant effects on SDF, but significant interactive effects of the screw speed and total moisture in the barrel were identified.

By examining the 3D surface plots and contour plots, moisture in barrel exhibited strong negative effects on SDF (Error! Reference source not found.A). Higher SDF levels were
obtained at high temperature (>132 °C) but low moisture content in the barrel (< 32%).

Moreover, a strong and positive correlation between SDF and SME \((r = 0.848, p < 0.0001)\) was found. A similar, albeit weaker, correlation between SDF and SME was observed in the previous screening study (Zhong, Fang, et al., 2019). The high temperature lowers the shear load in the barrel, reflecting as reduced torque (Alam, Kaur, Khaira, & Gupta, 2016; Duque, Manzanares, & Ballesteros, 2017; Zhong, Fang, et al., 2019). Likewise, high moisture in the barrel could moderate thermal energy exerted on the material, reduce shearing forces then the torque (Duque et al., 2017). The result is also supported by the strong negative correlation between moisture in the barrel and SME \((r = -0.895, p < 0.0001)\).

As presented in Table 1, along with the increases of SDF after extrusion, insoluble dietary fibre (IDF) level significantly decreased and the total dietary fibre (TDF) level remained constant compared to raw seed coat. The same pattern, which indicates the redistribution from IDF to SDF, was widely found in extruded cereal brans, pea and soybean seed coats (Zhong et al., 2018). Multiple mechanisms are established to explain the transformation, like particle size reduction, degradation of fibres and increased enzymatic digestibility (Zhong et al., 2018). Furthermore, in agreement with the previous screening study (Zhong, Fang, et al., 2019), IDF data generated a model with only the total moisture content in barrel showing significant effects (Table 2, Figure S2), but no significant model was found for TDF.

3.2. Effect of extrusion cooking on total polyphenol content (TPC)

Total polyphenol content in free phenolic extracts (TFPC) was increased by extrusion cooking under some conditions, while significant decreases were also observed in comparisons with raw seed coat (Table 3). A similar pattern in TFPC of extrusion cooked wheat bran was found by Ramos-Enriquez et al. (2018). According to the predictive equation in terms of coded factors of the selected linear model for TFPC, the moisture content in the barrel exerted the most significant and negative effects on TFPC, followed by screw speed which showed significant positive impacts. In contrast, extrusion cooking reduced total polyphenol content in bound extracts (TBPC), maximally by 33.2% (Table 3), but no significant model for TBPC was obtained. Taken together, TFPC made up over 65% of TPC in all samples, therefore, TFPC played a predominant role in the ANOVA analysis of TPC, giving a very similar linear model as the one of TFPC (Table 2, Figure S3 &S4). In addition, a strong correlation between TFPC and SME \((r = 0.909, p < 0.0001)\) was found. Besides, the negative correlations between TBPC and SME \((r = -0.504, p = 0.024)\) implied that extrusion cooking may liberate bound polyphenols to be free forms.
Extrusion cooking is found to reduce TPC of wheat bran, rice bran, barley bran and oat bran, up to by 73.38% (Kaur, Sharma, Singh, & Dar, 2015). Conversely, the increases in TPC are also widely reported in extruded food material, such as wheat bran (Ramos-Enriquez et al., 2018) and sorghum bran (Salazar Lopez et al., 2016). Wang, He, and Chen (2014) and Brennan, Brennan, Derbyshire, and Tiwari (2011) summarised some conflicting results in this aspect. Thermal degradation, decarboxylation and polymerisation of polyphenols during extrusion may contribute to the decreases (Altan, McCarthy, & Maskan, 2009). In contrary, the increases are primarily explained by the release of polyphenols from the cell wall matrix, depolymerisation of high molecular weight polyphenols (like condensed tannins) and Maillard reaction products (Brennan et al., 2011; Wang et al., 2014).

3.3. Effect of extrusion cooking on individual polyphenols

The influences of extrusion cooking on TPC of different food material have been extensively studied but less with respect to individual phenolics (Brennan et al., 2011). In agreement with the previous study, this study found that around 90% of total individual phenolics (TIPC) were found in free extracts (Table 3). Moreover, apigenin-7-O-β-apiofuranosyl-6,8-di-C-β-glucopyranoside (Api-Apif-di-Glc) was the dominant individual polyphenol (Zhong, Wu, et al., 2019). In this regard, only total phenolic individual content in free phenolic extracts (TFIPC) and Api-Apif-di-Glc will be further discussed in detail.

The impacts of extrusion cooking on the concentration of individual phenolics were not consistent. Extrusion cooking has been reported to decrease the content of heat-sensitive extractable phenolic acids (Altan et al., 2009) and flavonoids (Khanal, Howard, & Prior, 2009). Nonetheless, extrusion cooking also is indicated to increase some phenolic individuals by increasing their extractability (Wang et al., 2014), or lowering molecular weight of tannins (Khanal et al., 2009). In the current study, extrusion cooking decreased TFIPC values regardless of the extrusion conditions, mainly owing to the overwhelming losses of Api-Apif-di-Glc which accounted for more than 70% of the total individual polyphenols (Table 3). Moreover, all three independent variables in the quadratic model for TFIPC showed significant effects (Table 2). A quadratic model was suggested for Api-Apif-di-Glc, with barrel temperature and screw speed showing significant effects (Table 2). The model meets all the criteria as mentioned in Section 2.5, however, it is worthy to note that the difference between adjusted and predicted $R^2$ is greater than 0.2, indicating problems with the model (Stat-Ease Inc., 2018). According to the 3D surface, higher levels of Api-Apif-di-Glc and thus TFIPC was observed under higher screw speed, high temperature but low moisture (Figure 1). This may be attributed to the shorter residence time and lower SME (Kazemzadeh, 2011).
3.4. Optimisation and validation of the extrusion cooking

Given the current study initially focused on SDF level, only SDF, total free phenolic content (TFPC) and total free individual phenolic content (TFIPC) were selected as the responses. The corresponding optimal solution generated by the software (1 of 79 solutions), which aimed to achieve the highest levels of SDF and phenolic content simultaneously, had the highest desirability \(D = 0.849\) (Table 4). As shown in Table 4, the actual values of the responses at the optimal settings were comparable to the predicted values \((p > 0.05)\). The findings confirmed that the selected RSM models are capable of predicting the three responses after extrusion cooking.

3.5. Mineral and Api-Apif-di-Glep bioaccessibility and bioavailability

3.5.1. Mineral bioaccessibility and bioavailability

As presented in Table S2, calcium was the main minerals of the raw lupin seed coat (6.40 g/kg db), followed by K (3.10 g/kg db) and Mg (1.50 g/kg db). In fact, 67.5% of the calcium in the ASL seed was indicated to locate in the seed coat (Hung et al., 1988). Calcium chelates with pectins in the cell wall and plays a critical role in cell wall growth and stabilisation of cell wall structures (Moïse, Han, Gudynaitė-Savitch, Johnson, & Miki, 2005). The mineral content was stable after extrusion except that iron was more than doubled (Table S2). Extrusion cooking is reported to increase contents of iron and copper in both extruded whole ASL seed flour and ASL kernel flour (Suliburska, Krejpcio, Lampart-Szczapa, & Wojciak, 2009). Similar iron increases were found in extruded pea and kidney bean seed meals (Alonso, Rubio, Muzquiz, & Marzo, 2001), legume flours (Lombardi-Boccia, Lullo, & Carnovale, 1991) and maize-based snack food (Hazell & Jolinson, 1989). The increases are explained by contaminations during extrusion cooking, mainly from wearing of screws.

As presented in Figure 2, extrusion showed no significant effects on bioaccessibility and bioavailability of Cu, Fe, Mg and Zn. In terms of iron bioavailability, however, given the occurrence of exogenous iron from extrusion cooking, the non-significant difference between before and after the processing indicated that iron from contamination could not solubilise during digestion (Lombardi-Boccia et al., 1991). In contrast, extrusion cooking significantly lowered the Ca bioaccessibility (Figure 2).

Extrusion cooking was extensively indicated to improve mineral absorption directly (e.g. disruption of food matrix and enhanced enzyme digestibility) or indirectly (e.g. reduction of mineral chelating compounds like phytates and condensed tannins) (Alam et al., 2016; Nikmaram et al., 2017). However, no significant changes in mineral absorption after extrusion cooking were also reported (Drago, Velasco-Gonzalez, Torres, Gonzalez, & Valencia, 2007).
In this study, extrusion cooking was demonstrated to modify the composition and structure of lupin seed coat dietary fibre. However, both IDF and SDF showed mineral binding/physical trapping capacity (Baye, Guyot, & Mouquet-Rivier, 2017). Moreover, SDF can serve as a thickening agent that affects digesta viscosity such that show negative influences on nutrient infusibility. SDF of extrusion cooked seed coat primary contain s pections and hemicelluloses (Zhong, Fang, et al., 2019), while Ca can chelate with pectins (Moïse et al., 2005). Therefore, the reduced Ca bioaccessibility could be explained by the increased SDF level. In this regard, the positive effects on mineral bioaccessibility, bioavailability and dialysability caused by extrusion cooking may be compensated, partly at least, by the potential negative effects, resulting in conflicting overall effects (Baye et al., 2017).

3.5.2. Api-A pijf-di-Glc p bioaccessibility and bioavailability

The Api-A pijf-di-Glc p content was reduced from 271.50 μg vitexin equivalent (VE)/g db to 165.54 μg VE/g db. Czubinski et al. (2019) suggested that the in vitro digestion procedure could release up to 92% of the initial Api-A pijf-di-Glc p from whole ASL seed four. However, the authors did not quantify Api-A pijf-di-Glc p in the digest fluid (supernatant). In this study, only around 50% of Api-A pijf-di-Glc p in the raw seed coat were found in the digest fluid.

As shown in Figure 2, significantly higher levels of bioaccessible Api-A pijf-di-Glc p were found in extrusion cooked sample compared to the respective raw seed coat, but its bioavailability was not affected ($p > 0.05$). In contrast, extrusion was reported to improve the bioavailability of sorghum catechins (Gu, House, Rooney, & Prior, 2008), phenolic acids of barley and oat (in vivo experiment using pigs as a model system) (Hole et al., 2013). Dietary fibre in the lupin seed coat can interact with Api-A pijf-di-Glc p chemically and physically, reducing its absorption. Therefore, increased SDF hamper the diffusion and thus bioavailability of Api-A pijf-di-Glc p (Ribas-A gusti et al., 2018).

Conclusions

Dietary fibre is an essential component of a healthy diet, with soluble dietary fibre (SDF) having a more profound effect on health. In the current study, extrusion cooking is used to process the lupin seed coat. A central composite rotatable design coupled with global desirability function is performed to optimise the extrusion cooking to achieve high SDF and polyphenols content simultaneously. Optimal extrusion conditions, namely barrel temperature at 141°C, total moisture in the barrel at 30% and a screw speed of 400 rpm are obtained from the design. The seed coat SDF content is increased from 44.17 g/kg db up to 101.47 g/kg db, while high polyphenol content is maintained. Using an in vitro digestion model, this study
reveals that the extrusion shows no effects on the mineral bioaccessibility and bioavailability but increases Api-Api/f-di-Glcp bioaccessibility. In summary, this study suggests that extrusion cooking could be a promising technology to increase the biofunctional properties of the lupin seed coat as a high fibre antioxidant food ingredient. Nevertheless, a higher SDF level is still desired. For example, combinations of extrusion cooking and other pre- and post-treatments to further increase SDF content merits further investigation.

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Conflict of interest

The authors declare that they have no conflict of interest.

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FIGURES

Figure 1. 3D surface SDF (A) and TFIPC (B) as a function of (1) barrel temperature and total moisture in barrel at a constant screw speed of 350 rpm; (2) barrel temperature and screw speed at moisture of 35%; (3) screw speed and total moisture in barrel at barrel temperature of 135 °C.

Figure 2. Bioaccessibility and bioavailability of selected minerals and apigenin-7-O-β-apiofuranosyl-6,8-di-C-β-glucopyranoside (Api-Api'f-di-Glc) in raw and extruded lupin seed coats under optimal conditions
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* $p \leq 0.05$; ** $p \leq 0.01$
TABLES

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Table 2. Coded and actual predictive equations for each response using their corresponding actual coefficients acquired in the models

Table 3. Total polyphenol content and total individual polyphenol content of each extrusion run of the central composite rotatable design compared to the raw lupin seed coat

Table 4. Numerical optimisation criteria using desirability analysis of the multi-responses
Table 1. Coded and actual experimental levels in the central composite rotatable design and experimental results for SME, dietary fibre composition compared to the raw lupin seed coat

<table>
<thead>
<tr>
<th>Run</th>
<th>X₁</th>
<th>X₂</th>
<th>X₃</th>
<th>SME (W·h/kg)</th>
<th>Dietary fibre (g/kg db)</th>
</tr>
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<tbody>
<tr>
<td></td>
<td>Barrel temperature (°C)</td>
<td>Screw speed (rpm)</td>
<td>Total moisture in barrel (%)</td>
<td></td>
<td>SDF</td>
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<tr>
<td>Raw</td>
<td>135 (0)</td>
<td>350 (0)</td>
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<td>44.17±0.56</td>
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<td>3</td>
<td>135 (0)</td>
<td>350 (0)</td>
<td>43.409 (±1.682)</td>
<td>135.52</td>
<td>90.46±2.26</td>
</tr>
<tr>
<td>4</td>
<td>135 (0)</td>
<td>350 (0)</td>
<td>43.409 (±1.682)</td>
<td>135.52</td>
<td>90.43±4.93</td>
</tr>
<tr>
<td>5</td>
<td>135 (0)</td>
<td>350 (0)</td>
<td>43.409 (±1.682)</td>
<td>135.52</td>
<td>78.64±4.65</td>
</tr>
<tr>
<td>6</td>
<td>135 (0)</td>
<td>350 (0)</td>
<td>43.409 (±1.682)</td>
<td>135.52</td>
<td>89.85±2.07</td>
</tr>
<tr>
<td>7</td>
<td>135 (0)</td>
<td>350 (0)</td>
<td>43.409 (±1.682)</td>
<td>135.52</td>
<td>82.04±1.94</td>
</tr>
<tr>
<td>8</td>
<td>135 (0)</td>
<td>350 (0)</td>
<td>43.409 (±1.682)</td>
<td>135.52</td>
<td>87.17±1.15</td>
</tr>
<tr>
<td>9</td>
<td>135 (0)</td>
<td>350 (0)</td>
<td>43.409 (±1.682)</td>
<td>135.52</td>
<td>77.46±0.12</td>
</tr>
<tr>
<td>10</td>
<td>135 (0)</td>
<td>350 (0)</td>
<td>43.409 (±1.682)</td>
<td>135.52</td>
<td>89.85±2.07</td>
</tr>
<tr>
<td>11</td>
<td>135 (0)</td>
<td>350 (0)</td>
<td>43.409 (±1.682)</td>
<td>135.52</td>
<td>82.04±1.94</td>
</tr>
<tr>
<td>12</td>
<td>135 (0)</td>
<td>350 (0)</td>
<td>43.409 (±1.682)</td>
<td>135.52</td>
<td>87.17±1.15</td>
</tr>
<tr>
<td>13</td>
<td>135 (0)</td>
<td>350 (0)</td>
<td>43.409 (±1.682)</td>
<td>135.52</td>
<td>77.46±0.12</td>
</tr>
<tr>
<td>14</td>
<td>135 (0)</td>
<td>350 (0)</td>
<td>43.409 (±1.682)</td>
<td>135.52</td>
<td>89.85±2.07</td>
</tr>
<tr>
<td>15</td>
<td>135 (0)</td>
<td>350 (0)</td>
<td>43.409 (±1.682)</td>
<td>135.52</td>
<td>82.04±1.94</td>
</tr>
<tr>
<td>16</td>
<td>135 (0)</td>
<td>350 (0)</td>
<td>43.409 (±1.682)</td>
<td>135.52</td>
<td>87.17±1.15</td>
</tr>
<tr>
<td>17</td>
<td>135 (0)</td>
<td>350 (0)</td>
<td>43.409 (±1.682)</td>
<td>135.52</td>
<td>77.46±0.12</td>
</tr>
<tr>
<td>18</td>
<td>135 (0)</td>
<td>350 (0)</td>
<td>43.409 (±1.682)</td>
<td>135.52</td>
<td>89.85±2.07</td>
</tr>
<tr>
<td>19</td>
<td>135 (0)</td>
<td>350 (0)</td>
<td>43.409 (±1.682)</td>
<td>135.52</td>
<td>82.04±1.94</td>
</tr>
<tr>
<td>20</td>
<td>135 (0)</td>
<td>350 (0)</td>
<td>43.409 (±1.682)</td>
<td>135.52</td>
<td>87.17±1.15</td>
</tr>
</tbody>
</table>

Factor levels are expressed as actual levels (coded levels).
SME, specific mechanical energy; SDF, soluble dietary fibre; IDF, insoluble dietary fibre; TDF, total dietary fibre.

† The highest barrel temperature set at zone 3, temperature profiles were 70/100/120/120°C (-1), 70/100/150/125°C (+1), 70/100/135/125°C (0), 70/100/110/110°C (-1.682), 70/100/160/125°C (+1.682), respectively.

‡ Raw was un-extrusion cooked lupin seed coat, and it was not part of the design.

§ Rounded to integer in actual operation.

¶ Rounded to one decimal in actual operation

Means assigned with different letters in the same column indicate significant differences as compared to raw seed coat (p < 0.05).
Table 2. Coded and actual predictive equations for each response using their corresponding actual coefficients acquired in the models

<table>
<thead>
<tr>
<th>Source</th>
<th>SDF</th>
<th>IDF</th>
<th>TFPC</th>
<th>TPC</th>
<th>TFIPC</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Coded</td>
<td>Actual</td>
<td>Coded</td>
<td>Actual</td>
<td>Coded</td>
</tr>
<tr>
<td>Intercept</td>
<td>89.36</td>
<td>-545.3663</td>
<td>853.03</td>
<td>790.569</td>
<td>37.09</td>
</tr>
<tr>
<td>A- Barrel Temperature</td>
<td>2.2</td>
<td>4.9419</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>B- Screw Speed</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>1.30</td>
</tr>
<tr>
<td>C- Moisture in Barrel</td>
<td>-7.92</td>
<td>10.2282</td>
<td>11.91</td>
<td>2.3824</td>
<td>-4.24</td>
</tr>
<tr>
<td>AB</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>AC</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>BC</td>
<td>-4.05</td>
<td>-0.0162</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>A²</td>
<td>-3.04</td>
<td>-0.0135</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>B²</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>C²</td>
<td>-1.53</td>
<td>-0.061</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

*p*-value (Model)  
< 0.0001  
< 0.0001  
< 0.0001  
< 0.0001  
0.0033

*p*-Lack of Fit  
0.3066  
0.0666  
0.5391  
0.202  
0.8857

Fit statistics  
R² 0.9645  
0.7666  
0.8677  
0.8052  
0.8806

Adjusted R² 0.9289  
0.7199  
0.8413  
0.7662  
0.7612

Predicated R² 0.7741  
0.5664  
0.7406  
0.6081  
0.5406

Adequate Precision 19.14  
15.6236  
18.4315  
15.273  
9.7193

SDF, soluble dietary fibre; IDF, insoluble dietary fibre; TFPC, Total polyphenol content in free phenolic extracts; TPC, Total polyphenol content; TFIPC, total individual phenolics in free phenolic extracts.
Table 3. Total polyphenol content and total individual polyphenol content of each extrusion run of the central composite rotatable design compared to the raw lupin seed coat

<table>
<thead>
<tr>
<th>Run†</th>
<th>Total polyphenol content (mg GAE/100 g db)</th>
<th>Total individual polyphenols (μg/g db)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Free(TFPC)</td>
<td>Bound(TBPC)</td>
</tr>
<tr>
<td>Raw‡</td>
<td>36.11±0.31(^a)</td>
<td>18.14±1.00(^a)</td>
</tr>
<tr>
<td>1</td>
<td>32.02±0.54(^b)</td>
<td>14.63±0.02(^b)</td>
</tr>
<tr>
<td>2</td>
<td>32.30±1.71(^b)</td>
<td>15.25±0.36(^b)</td>
</tr>
<tr>
<td>3</td>
<td>41.21±0.69(^b)</td>
<td>13.72±0.70(^b)</td>
</tr>
<tr>
<td>4</td>
<td>41.84±1.13(^b)</td>
<td>15.17±0.93(^b)</td>
</tr>
<tr>
<td>5</td>
<td>34.30±1.13(^b)</td>
<td>16.74±0.67(^a)</td>
</tr>
<tr>
<td>6</td>
<td>36.99±0.30(^a)</td>
<td>13.29±0.53(^a)</td>
</tr>
<tr>
<td>7</td>
<td>37.93±0.36(^a)</td>
<td>13.93±0.08(^a)</td>
</tr>
<tr>
<td>8</td>
<td>32.27±1.31(^b)</td>
<td>16.76±0.11(^a)</td>
</tr>
<tr>
<td>9</td>
<td>36.78±0.55(^a)</td>
<td>15.44±0.70(^b)</td>
</tr>
<tr>
<td>10</td>
<td>30.59±0.92(^b)</td>
<td>15.61±0.21(^b)</td>
</tr>
<tr>
<td>11</td>
<td>40.51±0.59(^a)</td>
<td>12.12±0.31(^b)</td>
</tr>
<tr>
<td>12</td>
<td>45.70±0.50(^b)</td>
<td>12.96±1.11(^b)</td>
</tr>
<tr>
<td>13</td>
<td>33.67±1.31(^b)</td>
<td>16.65±0.18(^b)</td>
</tr>
<tr>
<td>14</td>
<td>36.39±1.51(^a)</td>
<td>13.13±0.77(^b)</td>
</tr>
<tr>
<td>15</td>
<td>36.92±0.67(^a)</td>
<td>13.91±0.95(^b)</td>
</tr>
<tr>
<td>16</td>
<td>37.22±1.99(^a)</td>
<td>13.80±0.99(^b)</td>
</tr>
<tr>
<td>17</td>
<td>43.48±1.25(^b)</td>
<td>13.96±1.86(^b)</td>
</tr>
<tr>
<td>18</td>
<td>36.93±0.93(^a)</td>
<td>12.90±0.93(^b)</td>
</tr>
<tr>
<td>19</td>
<td>39.22±0.44(^b)</td>
<td>12.81±1.43(^b)</td>
</tr>
<tr>
<td>20</td>
<td>37.41±2.56(^a)</td>
<td>13.69±0.01(^b)</td>
</tr>
</tbody>
</table>

† See Table 1 for complete experimental levels.
‡ Raw was un-extrusion cooked lupin seed coat, and it was not part of the design.
TF/BPC, Total polyphenol content in free/bound phenolic extracts; TPC, Total polyphenol content (i.e. TFPC+TBPC); TF/BIPC, total individual phenolics in free/bound phenolic extracts; TIPC, Total individual phenolics content (i.e. TFIPC+TBIPC). Means assigned with different letters in the same column indicate significant differences as compared to raw seed coat (p < 0.05).
Table 4. Numerical optimisation criteria using desirability analysis of the multi-responses

<table>
<thead>
<tr>
<th>Factors</th>
<th>Goal</th>
<th>Goal limits</th>
<th>Importance level (r)</th>
<th>Weight</th>
<th>Optimal solution</th>
<th>Actual value</th>
<th>95% PI range</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Independent factors</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Barrel temperature (°C)</td>
<td>In range</td>
<td>120-150</td>
<td>-</td>
<td>-</td>
<td>141.1</td>
<td>141</td>
<td></td>
</tr>
<tr>
<td>Total moisture in barrel (%)</td>
<td>In range</td>
<td>30-40</td>
<td>-</td>
<td>-</td>
<td>30</td>
<td>30</td>
<td></td>
</tr>
<tr>
<td>Screw speed (rpm)</td>
<td>In range</td>
<td>300-400</td>
<td>-</td>
<td>-</td>
<td>400</td>
<td>400</td>
<td></td>
</tr>
<tr>
<td><strong>Responses</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SDF (g/kg db)</td>
<td>Maximize</td>
<td>6.73-10.38</td>
<td>++++++ (5)</td>
<td>1</td>
<td>99.06±0.22a</td>
<td>101.47±2.50a</td>
<td>95.83-102.29</td>
</tr>
<tr>
<td>TFPC (mg GAE/100 g db)</td>
<td>Maximize</td>
<td>30.6-50.8</td>
<td>+++ (3)</td>
<td>1</td>
<td>42.92±0.15a</td>
<td>42.90±0.30a</td>
<td>40.13-45.74</td>
</tr>
<tr>
<td>TIFPC (µg/g db)</td>
<td>Maximize</td>
<td>264-313.3</td>
<td>++++ (4)</td>
<td>1</td>
<td>307.01±5.80a</td>
<td>309.08±1.89a</td>
<td>294.44-319.59</td>
</tr>
<tr>
<td><strong>Desirability</strong></td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td>0.849</td>
<td></td>
</tr>
</tbody>
</table>

SDF, soluble dietary fibre; TFPC, Total polyphenol content in free phenolic extracts; TIFPC, total individual phenolics in free phenolic extracts;

PI, prediction interval.

Means assigned with different letters in the same raw indicate significant differences (p < 0.05).