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### **ORIGINAL ARTICLE**

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## Effect of extrusion conditions on the physical and chemical properties of bean powders

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### Abstract

This study aimed to investigate the effects of three extrusion parameters (i.e., feed moisture, feed rate, and die temperature) on expansion volume, moisture, starch digestibility, pasting properties, and solvent retention capacities of two bean varieties (Fuji and Medalist). A Box-Behnken Design of Experiment and Response Surface Methodology were applied, considering three levels for each factor: 20, 27.5, 35 g/100 g for feed moisture; 2, 2.5, 3 kg/hr for feed rate; 70, 100, 130°C for die temperature. Feed moisture significantly affected (p < .05) all the measured characteristics of extruded bean powders, while feed rate resulted often not significant (p > .05). Genotype had a relevant effect on the considered properties; in fact, significant models fitted different response variables for Fuji and Medalist beans. The calculated models are a valid tool for the design of bean powders with specific qualities, to be used as new ingredients in food formulations.

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### **Practical applications**

This study investigates the effects of extrusion feed moisture, feed rate, and die temperature on some physicochemical properties of two bean varieties (Fuji and Medalist) largely available and characterized by a plain taste, thus appreciated by both children and elderly people. The results demonstrate that different starch digestibility, pasting properties, and solvent retention capacities can be obtained by modulating the extrusion conditions and taking into consideration the effect of bean genotype. The application of a Box-Behnken Design of Experiment elaborated by Response Surface Methodology allowed to calculate the reliable models to be used as valid tools for the design of extruded bean powders with specific qualities. Targeted extruded bean powders can be efficiently exploited as new ingredients in food formulations, with advantages for both consumers focused on healthier foods (e.g., diabetes, athletes, and children) and industries willing to better valorize their products.

### 1 INTRODUCTION

49 The regular consumption of pulses is promoted by health organizations 50 in order to contain obesity and its comorbidities (Luhovyy et al., 2015). 51 The possibility of developing new food ingredients and products using 52 pulses has been extensively discussed in the literature (Ai, Cichy, Harte, Kelly, & Ng, 2016; Cappa, Kelly, & Ng, 2018; Tiwari, Gowen,

& McKenna, 2011). According to Sozer, Holopainen-Mantila, and Poutanen (2017), the use of pulses will increase in the future, especially in combination with cereals, finding new applications able to satisfy both sensory and nutritional consumers' needs. Moriano et al. (2019) recently demonstrated that the use of extruded bean flour gives cookies with quality characteristics comparable to a traditional reduced-fat product but with improved nutritional profile in terms of higher level of



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slowly digestible starch. Kahraman, Harsa, Lucisano, and Cappa (2018) reported that the partial substitution of rice flour with chickpea flours in a gluten-free dough is a sustainable way to increase the protein content 4 and foaming capacity of the slurries, while reducing the retrogradation tendency. Indeed, the gluten-free market is one possible target sector 6 for the application of pulses, which are 2-3 times richer in proteins than cereals and contain high amounts of dietary fiber, minerals, vitamins, 8 and phytochemicals (Hayat, Ahmad, Masud, Ahmed, & Bashir, 2014).

9 The possibility of designing specific physicochemical properties 10 by rapid technologies such as extrusion could be useful to address pulse applications in foods. Extrusion is largely used in the production 11 12 of cereal products (Miller, 1994; Pardhi, Singh, Nayik, & Dar, 2019; 13 Rokey, 1994), as well as candies (Best, 1994) and products based on 14 texturized vegetable proteins (Lin, Huff, & Hsieh, 2000). During the 15 extrusion, sample is guickly treated under suitable processing condi-16 tions (e.g., feed moisture, feed rate, temperature, and screw speed) 17 and undergoes stress forces (e.g., friction and shear forces) inside 18 the extruder, before going through the extruder die, where a rapid 19 decrease in pressure causes a quick evaporation of the water, with 20 a consequent expansion of the structure. Extrusion is a complex pro-21 cess influenced by many processing variables, all potentially affecting the quality of the extrudates. Ilo, Liu, and Berghofer (1999) reported 23 that, during extrusion cooking, raw materials undergo many chemical 24 and structural transformations, such as starch gelatinization, protein 25 denaturation, complex formation between amylose and lipids, and degradation reactions of vitamins and pigments. Moreover, antinutri-26 27 tional factors of beans can be removed by extrusion cooking (Singh, 28 Gamlath, & Wakeling, 2007), while improving starch digestibility 29 (Singh, Dartois, & Kaur, 2010) and nutrient bioavailability (Brennan, 30 Brennan, Derbyshire, & Tiwari, 2011) compared to conventional cook-31 ing. However, the extrusion process is not being used widely for com-32 mercial value-addition for pulses (Berrios, Ascheri, & Losso, 2013) and more investigations are needed to promote its application.

34 This study investigated the main and interaction effects of three 35 extrusion parameters (i.e., feed moisture, feed rate, and die tem-36 perature) on bean powder physical and chemical properties, while 37 also taking into account possible genotype influence. Accordingly, 38 Design of Experiments (DoE) and Response Surface Methodology 39 (RSM) have been applied in order to model quality parameters of ex-40 truded powders obtained from two bean varieties, Fuji and Medalist, 41 belonging to Otebo and Navy market classes, respectively.

#### MATERIALS AND METHODS 2

### 2.1 | Materials

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48 Edible dry bean (Phaseolus vulgaris L.) seeds were provided by 49 Michigan State University Dry Bean Breeding and Genetics Program 50 (East Lansing, MI, USA). Both Fuji (F) and Medalist (M) beans were 51 grown under similar conditions at the Saginaw Valley Research and 52 Extension Center (Frankenmuth, MI, USA) in 2016. Using a Wiley 53 mill, dry beans were ground to powder (particle size  $\leq 1$  mm).

#### Highlights

- Main and interaction effects of three extrusion parameters were assessed.
- Feed moisture was the most significant factor.
- · Feed rate and die temperature partially affected extruded bean powder quality.
- Genotype mainly affected physical and chemical properties of extruded bean powders.
- The calculated models can be used in designing valueadded bean powder ingredients.

#### 2.2 Design of experiments

To study simultaneously the main and interaction effects of three extrusion parameters on bean powder properties, a Box-Behnken DoE was developed for each bean variety (Design Expert software, v. 10.0.0.3, Stat-Ease Inc., Minneapolis, MN, USA). Three levels for each factor were considered: 20, 27.5, and 35 g/100 g for feed moisture; 2, 2.5, and 3 kg/hr for feed rate; 70, 100, and 130°C for die temperature. All levels were established based on preliminary extrusion trials. The DoE consisted of 15 experiments for each bean variety: 12 factorial runs and three replicates of the central point that crosses the intermediate levels of the three factors. The order of experiments was randomized to avoid the systematic errors and to minimize the effects of external factors. Each sample was identified by a code (Table 1) indicating the bean variety, the feed moisture level, the feed rate level, and the die temperature referred to as LT (low temperature, 70°C), MT (medium temperature, 100°C), and HT (high temperature, 130°C). Moreover, the three replicates of the central point were indicated with the letters a, b, or c at the end of the sample code.

#### 2.3 | Extrusion conditions

A corotating twin-screw extruder (MP19T2-25, APV Baker Inc., Grand Rapids, MI, USA) was used, with 19 mm barrel diameter, L/D ratio of 25:1, and 3 mm exit die diameter (Figure 1). Feed moisture was adjusted using a water injector (Brook Crompton E2 Metripump, Hudders Field, England). Feed rate was controlled using a volumetric feeder (K2V-T20, Coperion K-Tron Pitman Inc., Sewell, NJ, USA). Barrel temperatures were set to 40, 50, 65, 65, 70°C or 40, 55, 75, 95, 100°C or 40, 60, 80, 115, 130°C in order to produce LT, MT, or HT levels. Screw speed was set at 200 rpm and percentage motor load (%) was recorded in order to calculate the specific mechanical energy (SME; Köksel, Ryu, Basman, Demiralp, & Ng, 2004).

Extrudates were manually cut to 2 cm lengths and cooled down to room temperature before measuring diameters by a caliper (n = 10). Expansion value (%) was calculated with respect to extruder die opening (3 mm).

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TABLE 1         Extruded bean powder           sample identification, extrusion           parameters (feed moisture, feed rate, and           die temperature)           specific mechanical	Sample	Feed moisture (g/100 g)	Feed rate (kg/h)	Die temperature (°C)	SME (Wh/kg)	Expansi value (%
energy (SME), and expansion values	F20_2.5_LT	20	2.5	70	238	124
	F35_2.5_LT	35	2.5	70	92	25
	F20_2.5_HT	20	2.5	130	161	94
	F35_2.5_HT	35	2.5	130	82	56
	F20_2_MT	20	2	100	197	123
	F35_2_MT	35	2	100	104	36
	F20_3_MT	20	3	100	165	120
	F35_3_MT	35	3	100	84	66
	F27.5_2_LT	27.5	2	70	132	53
	F27.5_2_HT	27.5	2	130	131	51
	F27.5_3_LT	27.5	3	70	121	67
	F27.5_3_HT	27.5	3	130	106	71
	F27.5_2.5_MT	_a 27.5	2.5	100	129	66
	F27.5_2.5_MT	_b 27.5	2.5	100	128	65
	F27.5_2.5_MT	_c 27.5	2.5	100	131	64
	M20_2.5_LT	20	2.5	70	225	109
	M35_2.5_LT	35	2.5	70	90	19
	M20_2.5_HT	20	2.5	130	163	121
	M35_2.5_HT	35	2.5	130	80	55
	M20_2_MT	20	2	100	195	98
	M35_2_MT	35	2	100	99	19
	M20_3_MT	20	3	100	174	121
	M35_3_MT	35	3	100	82	46
	M27.5_2_LT	27.5	2	70	189	115
	M27.5_2_HT	27.5	2	130	118	43
	M27.5_3_LT	27.5	3	70	120	59
	M27.5_3_HT	27.5	3	130	106	83
	M27.5_2.5_M	T_a 27.5	2.5	100	122	49
	M27.5_2.5_M	T_b 27.5	2.5	100	121	50
	M27.5_2.5_M	T_c 27.5	2.5	100	121	50
	Note: <a c="" h="" ra<="" td=""><td>nlicates of the centr</td><td>al noint: E Euii</td><td>hean nowder: UT h</td><td>igh temperati</td><td>ure (130°C</td></a>	nlicates of the centr	al noint: E Euii	hean nowder: UT h	igh temperati	ure (130°C

LT, low temperature (70°C); M, Medalist bean powder; MT, medium temperature (100°C); relative standard deviations for expansion values were 14% (n = 10); SME, Specific mechanical energy.

Extrudates were conditioned (48 hr at 20-25°C, relative humidity 65%) and ground (particle size < 0.5 mm) using a Thomas Wiley mill (Model 4, Thomas Scientific Inc., Swedesboro, NJ, USA). For comparison, bean powders not subjected to extrusion (F\_raw and M\_raw) were ground down to the same particle size. All powders were stored at 4°C in sealed bags until analyses.

#### 2.4 **Proximate composition**

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Samples were analyzed for moisture (AACC Method 44-15A; American Association of Cereal Chemists [AACC], 2000), total nitrogen (AOAC 920.87; Association of Official Analytical Chemists, 1999), and total starch contents ("Total Starch Assay Kit"; Megazyme International Ireland Ltd., Bray, Wicklow, Ireland). The protein content was calculated adopting a conversion factor of 6.25. Results are expressed on a dry basis (db).

### 2.5 | Starch digestibility

Starch digestibility of samples was determined according to the Englyst method (Englyst, Kingman, & Cummings, 1992), quantifying the released glucose using the "D-Glucose Assay Kit" (Megazyme

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27		2.6   Pasting prope	rties		
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32		lowing indices were extrap	olated from	n the RVA patterns: visc	ositv at
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38		2.7   Water and lac	tic acid r	etention capacities	;
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48 49 Water retention capacity (WRC) and lactic acid retention capacity (LARC) of bean powders were determined following AACC Method 56-11 (AACC, 2000), with minor modifications (Cappa et al., 2018). As extruded samples showed a high solvent affinity, the amount of solvent was increased to 30 ml for 3 g of sample. Results were calculated on db.

### 2.8 | Statistical analysis

If not differently specified, all measurements were run in triplicate 51 and mean values were used as responses for the elaboration of DoE 52 by RSM (Design Expert v. 10.0.0.3, Stat-Ease Inc., Minneapolis, MN, 53 USA). The following second order polynomial model containing where y is the value of the considered response variable,  $x_1$ ,  $x_2$  and  $x_3$  are the values of feed moisture, feed rate, and temperature, respectively,  $\beta_0$  is the constant value,  $\beta_1$ ,  $\beta_2$ , and  $\beta_3$  are the linear coefficients,  $\beta_{12},\beta_{13},$  and  $\beta_{23}$  are the interaction coefficients,  $\beta_{11},\beta_{22},$  and  $\beta_{33}$  are the quadratic coefficients, and  $\varepsilon$  is the random error. The significance of each coefficient was determined by one-way analysis of variance. If some of the coefficients were not significant, the model was simplified, paying attention to support hierarchy.

Correlation analysis among variables was performed following the Pearson approach (Statgraphics Centurion 18, Statpoint Technologies Inc., The Plains, VA, USA). The Pearson product moments of correlation (r) were calculated, together with the p values of statistical significance.

#### **RESULTS AND DISCUSSION** 3

#### 3.1 **Expansion values**

Extrudates obtained from Fuji had slightly higher expansion values compared to Medalist samples (25%-124% and 19%-121%, respectively), indicating that genotype somehow affected the sample expansion (Table 1); this can be partially attributed to the different chemical composition (e.g., the amylose content) of the two bean varieties (Cappa et al., 2018). The extrusion conditions greatly affected the sample expansion: different expansion values

n of the twin-screw extruder used. <sup>1</sup>D, screw diameter (19 mm); FP, forwarding paddle; P, kneading paddle n, twin lead screw; Single, single lead screw; Die: <u>3 mm single round-type exit die</u>

0

0

Type<sup>1</sup>

8D Twin

7 FP

8D Twin

3 FP

3 RP

2D Single

4 FP

3 RP

2D Single

Die

III 

0

倡

linear, interaction, and tors was applied (Mon

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_2 + \beta_{23} x_2 x_3 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2 + \epsilon$$
(1)

Extrusion die

IV

Flight angle (°)

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60

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1 were achieved according to the extrusion factor levels. Expansion 2 values of the samples belonging to different bean varieties (F or 3 M) and extruded under the same conditions were significantly cor-4 related (r = .78; p < .001). In general, higher expansion values were 5 obtained using the lowest feed moisture (see samples F20\_2.5\_LT, 6 F20\_2\_MT, M20\_2.5\_HT and M20\_3\_MT). Actually, the lower 7 the feed moisture, the higher the viscosity of material inside the 8 extruder barrel (Köksel et al., 2004), and the higher the friction 9 and shear forces, as confirmed by the high SME values (Table 1). 10 A higher SME value inside the barrel results in a higher pressure 11 drop at the extruder exit die (Ai et al., 2016), thus promoting a higher expansion value. Similarly, the increase in die temperature 12

and feed rate resulted in an increase in expansion values for both bean varieties in about 80% of samples. A better understanding of the main and interaction effects of the considered extrusion parameters was accomplished through elaboration of the DoE (Section 3.5).

#### 3.2 | Proximate composition and starch digestibility

Different moisture content values were obtained according to the process conditions used (Table 2), ranging from 8.4 (F20\_2.5\_HT) to 9.9 g/100 g (F20\_3\_MT) for Fuji and from 8.5 (M27.5\_2\_LT) to

 TABLE 2
 Moisture, protein, total starch, rapidly digestible starch (RDS), slowly digestible starch (SDS), and resistant starch (RS) of Fuji (F) and Medalist (M) bean powders. For sample identification see Table 1

Sample	Moisture (g/100 g)	Protein (g/100 g db)	Total starch (g/100 g db)	RDS (g/100 g db)	SDS (g/100 g db)	RS (g/100 g db)
F20_2.5_LT	8.83 ± 0.01	21.66 ± 0.19	38.96 ± 0.27	25.8 ± 0.7	-	9.7 ± 0.7
F35_2.5_LT	9.23 ± 0.05	21.63 ± 0.08	37.57 ± 0.79	20.7 ± 0.1	5.2 ± 0.2	11.7 ± 0.1
	8.44 ± 0.09	21.76 ± 0.13	37.94 ± 0.67	19.2 ± 0.3	$14.0 \pm 0.2$	$4.8 \pm 0.5$
-35_2.5_HT	8.80 ± 0.14	$21.83 \pm 0.20$	37.61 ± 0.77	23.7 ± 0.3	5.2 ± 0.3	8.6 ± 0.1
20_2_MT	9.14 ± 0.05	21.74 ± 0.13	37.58 ± 0.77	22.2 ± 0.0	$15.8 \pm 0.1$	-
-35_2_MT	9.59 ± 0.09	20.66 ± 0.08	35.19 ± 0.47	30.1 ± 0.2	-	5.1 ± 0.2
20_3_MT	9.91 ± 0.05	21.79 ± 0.22	36.92 ± 0.18	12.9 ± 0.1	13.9 ± 0.8	$10.1 \pm 0.8$
35_3_MT	9.21 ± 0.08	21.91 ± 0.09	36.69 ± 0.39	19.9 ± 1.1	$3.0 \pm 0.4$	13.8 ± 0.7
27.5_2_LT	9.80 ± 0.16	21.54 ± 0.14	37.93 ± 0.63	18.9 ± 0.1	$13.4 \pm 0.2$	5.6 ± 0.2
27.5_2_HT	8.88 ± 0.13	$21.80 \pm 0.14$	38.04 ± 0.55	24.1 ± 0.4	1.6 ± 0.2	$12.3 \pm 0.2$
27.5_3_LT	9.11 ± 0.04	21.60 ± 0.08	38.14 ± 0.86	17.8 ± 0.4	6.5 ± 0.3	13.8 ± 0.7
27.5_3_HT	8.57 ± 0.01	20.54 ± 0.22	37.64 ± 0.34	14.5 ± 0.5	10.8 ± 0.2	$12.4 \pm 0.7$
27.5_2.5_MT_a	8.94 ± 0.07	$21.44 \pm 0.21$	35.89 ± 0.40	20.8 ± 0.5	13.5 ± 0.5	3.7 ± 0.1
27.5_2.5_MT_b	8.96 ± 0.03	21.86 ± 0.19	37.94 ± 0.27	21.6 ± 1.1	11.6 ± 1.1	2.7 ± 0.1
27.5_2.5_MT_c	9.24 ± 0.02	20.29 ± 0.07	37.16 ± 0.61	20.5 ± 0.4	12.5 ± 0.2	$3.2 \pm 0.5$
_raw	11.19 ± 0.02	22.06 ± 0.02	38.53 ± 0.67	3.4 ± 0.5	$6.1 \pm 0.8$	29.8 ± 0.4
120_2.5_LT	8.98 ± 0.01	20.92 ± 0.06	38.13 ± 0.59	23.7 ± 0.6	$4.4 \pm 0.4$	10.1 ± 1.0
135_2.5_LT	9.86 ± 0.20	20.92 ± 0.01	37.89 ± 0.21	18.5 ± 1.9	6.6 ± 0.4	12.9 ± 1.5
120_2.5_HT	9.06 ± 0.04	20.96 ± 0.16	35.95 ± 0.70	17.5 ± 0.8	10.4 ± 1.2	8.0 ± 0.3
435_2.5_HT	9.17 ± 0.02	23.01 ± 0.03	36.32 ± 0.19	22.9 ± 0.1	5.1 ± 0.5	8.3 ± 0.4
120_2_MT	9.96 ± 0.03	$21.27 \pm 0.15$	38.97 ± 0.59	21.5 ± 0.3	6.8 ± 0.9	$10.7 \pm 0.5$
135_2_MT	10.25 ± 0.16	20.86 ± 0.01	36.76 ± 0.45	19.0 ± 0.4	7.1 ± 0.2	10.7 ± 0.6
120_3_MT	9.45 ± 0.14	19.61 ± 0.14	36.89 ± 0.32	23.8 ± 0.1	$3.3 \pm 0.3$	9.7 ± 0.3
M35_3_MT	9.80 ± 0.08	20.90 ± 0.21	36.34 ± 0.27	19.7 ± 0.2	$12.1 \pm 0.3$	4.5 ± 0.1
427.5_2_LT	8.53 ± 0.16	$20.42 \pm 0.07$	38.62 ± 0.74	21.5 ± 0.4	$12.3 \pm 0.4$	$4.8 \pm 0.1$
/127.5_2_HT	8.87 ± 0.03	19.97 ± 0.01	36.86 ± 0.39	25.5 ± 0.1	8.7 ± 0.3	2.6 ± 0.3
427.5_3_LT	9.20 ± 0.18	21.49 ± 0.14	38.94 ± 0.67	21.9 ± 0.5	14.6 ± 0.3	$2.4 \pm 0.1$
M27.5_3_HT	8.71 ± 0.16	$21.13 \pm 0.07$	36.77 ± 0.72	20.8 ± 0.7	$4.4 \pm 0.2$	11.6 ± 0.6
M27.5_2.5_MT_a	9.88 ± 0.02	20.96 ± 0.02	37.47 ± 0.63	23.7 ± 0.3	8.3 ± 0.1	5.5 ± 0.3
427.5_2.5_MT_b	9.84 ± 0.07	20.90 ± 0.07	38.47 ± 0.70	25.4 ± 0.8	7.9 ± 0.6	5.2 ± 0.3
M27.5_2.5_MT_c	9.82 ± 0.14	20.91 ± 0.07	38.45 ± 0.80	25.5 ± 0.9	7.7 ± 0.6	5.3 ± 0.3
M_raw	13.48 ± 0.02	22.40 ± 0.07	38.34 ± 0.74	2.2 ± 0.1	3.0 ± 0.1	33.2 ± 0.1

53 Note: db, dry basis; -, not detectable; F\_raw, nonextruded Fuji powder; M\_raw, nonextruded Medalist powder.

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10.3 g/100 g (M35\_2\_MT) for Medalist, while the raw samples had 1 2 values of 11.2 and 13.5 g/100 g, respectively. As expected, the extrusion process did not affect protein and total starch contents. Extruded 4 and raw powders had protein contents of 20.3-21.9 g/100 g db and 5 22.4 g/100 g db, respectively, for Fuji, and of 19.6-23.0 g/100 g db 6 and 22.1 g/100 g db, respectively, for Medalist. Total starch ranged 7 from 35.2 (F35\_2\_MT) to 39.0 g/100 g db (F20\_2.5\_LT and M20\_2\_ 8 MT), for both Fuji and Medalist extrudates, whereas the raw samples 9 had a total starch content of 38.5 g/100 g db and 38.3 g/100 g db, 10 respectively. The slight effect of the extrusion on total starch con-11 tent was previously reported by Ai et al. (2016).

12 Conversely, the extrusion highly affected the starch digestibility 13 of bean powders. The amount of RDS of the extrudates was more 14 than four times higher than that of raw sample for both Fuji (12.9-15 30.1 vs. 3.4 g/100 g db) and Medalist (17.5-25.5 vs. 2.2 g/100 g db) 16 samples. The RDS increase in extruded samples was related to a 17 partial starch gelatinization occurring during the extrusion cooking 18 process and it is an advantage for people needing food easily and 19 rapidly digestible (e.g., athletes and children), while it is less desirable 20 for people requiring slow rises in glycaemia (e.g., diabetics).

21 F raw powder SDS value was in the range of those of extruded samples (6.1 vs. 1.6-15.8 g/100 g db), while for Medalist extruded 22 23 samples the SDS values were always higher than that of M raw (3.3-24 14.6 vs. 3.0 g/100 g db). The differences between Fuji and Medalist 25 samples proved that bean genotype can affect the starch digestibility properties, probably due to the organization and amylose content 26 27 of starch granules (Hoover, Hughes, Chung, & Liu, 2010). According 28 to Cappa et al. (2018), Fuji and Medalist had amylose contents of 29 approximately 37 and 42 g/100 g on a dry starch basis, respectively, 30 but some differences can be found according to the crop year.

31 For both bean varieties, RS values of extruded samples were 32 much lower than those of the corresponding raw samples: 2.7-13.8 versus 29 g/100 g db for Fuji and 2.4-12.9 versus 33.2 g/100 g db 34 for Medalist. The reduction of RS content is consistent with litera-35 ture data (Alonso, Aguirre, & Marzo, 2000; Ma, Wang, Wang, Jane, 36 & Du, 2017); in fact, the extrusion partially cooks samples, making 37 starch more easily available to the enzymatic attack. Ma et al. (2017) 38 reported that RDS contents of cooked legume starches are signifi-39 cantly higher than those of raw starches, while SDS and RS contents 40 are significantly lower, attributing this to the fact that raw legume 41 starches with intact starch granules have a concentrically arranged 42 structure in amorphous and crystalline regions, resulting in high re-43 sistance to enzymatic digestion.

### 3.3 | Pasting properties

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RVA curves of raw samples showed a low viscosity at the beginning of the test, followed by a rapid increase due to swelling and
gelatinization of starch granules at temperatures higher than 60°C.
Extruded bean powders showed a higher viscosity at the beginning
of the RVA-testing (temperature < 80°C), indicating that starch was</li>
already partially gelatinized as an effect of extrusion: gelatinized

powders are more readily hydrated and able to contribute to viscosity development at low temperatures. Then, during the heating phase and holding time at 95°C, a slight increase in viscosity was evidenced for all extruded samples (Table 3); moreover, lower FVs were achieved after the cooling phase compared to raw powders. FVs of extruded samples ranged from 444 (F20\_2.5\_LT) to 807 cP (F35\_2.5\_HT) for Fuji and from 474 (M20\_2.5\_HT) to 962 cP (M35\_2.5\_HT) for Medalist samples, while F\_raw and M\_raw powders showed values of 1,642 and 1,602 cP, respectively. Similar findings were reported by Ai et al. (2016) who attributed the lower FVs of extruded bean powders to the loss of integrity of starch granules and the degradation of starch molecules during extrusion cooking.

With respect to raw samples, extruded samples had lower SB values (37%–59% lower for Fuji; 42%–59% lower for Medalist), confirming that starch is partially gelatinized during extrusion and thus less rearrangement takes place during RVA-testing. The lower SB values can be an advantage when the extruded powders are intended as new ingredients for staling-sensitive products.

#### 3.4 | Water and lactic acid retention capacities

Interactions among bean powders and other ingredients (especially liquid ones) during food production can play a crucial role in rheological behavior (Cappa, Kelly, & Ng, 2020). Extruded powders exhibited higher WRC and LARC values in comparison to raw powders (Table 3): WRC and LARC values of Fuji extruded samples were, respectively, 107%–190% and 11%–85% higher than the values of raw powder; for Medalist, WRC and LARC values were 82%-174% and 24%-77% higher after extrusion. This behavior could be ascribed partly to starch gelatinization caused by extrusion cooking, which led to the exposition of more hydroxyl groups and enhanced the hydrophilic properties of extruded bean powders with respect to the raw ones. The capacity of extruded samples to interact with lactic acid solution more than raw samples indicates that not only starch but also proteins are affected by extrusion, due to thermal and mechanical stresses involved in the process, which somehow denature proteins (Chen, Wei, & Zhang, 2011).

#### 3.5 | Extrusion modeling

The RSM allowed modeling the characteristics of extruded powders as a function of the considered processing parameters; significant (p < .05) models are reported in Table 4. Besides the coefficient values (in terms of coded factors), several figures of merit were calculated: determination coefficient ( $R^2$ ), adjusted  $R^2$ , predicted  $R^2$ , and lack of fit (LOF, p value). For some response variables it was not possible to obtain a significant model, meaning that experimental factors outside of the considered ones affect those properties. Bean genotype had a relevant effect on the considered physical and chemical features, since significant models fitted different response variables for Fuji and Medalist.

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an powders. For sa	ample identificati	UT SEE TADIE 1				
Sample	V1 (cP)	V2 (cP)	FV (cP)	SB (cP)	WRC (g/g db)	LARC (g/g db)
F20_2.5_LT	231 ± 23	236 ± 27	444 ± 10	197 ± 21	3.66 ± 0.01	3.73 ± 0.04
F35_2.5_LT	278 ± 6	347 ± 9	651 ± 11	305 ± 2	$3.28 \pm 0.04$	2.58 ± 0.07
F20_2.5_HT	259 ± 8	283 ± 16	492 ± 18	209 ± 3	4.09 ± 0.05	4.23 ± 0.03
F35_2.5_HT	565 ± 35	559 ± 18	807 ± 26	248 ± 8	$3.95 \pm 0.05$	3.73 ± 0.11
F20_2_MT	286 ± 1	294 ± 1	523 ± 18	229 ± 17	3.92 ± 0.08	3.88 ± 0.04
F35_2_MT	314 ± 3	400 ± 7	655 ± 13	255 ± 6	$3.15 \pm 0.05$	3.17 ± 0.09
F20_3_MT	279 ± 1	293 ± 2	521 ± 1	228 ± 1	3.67 ± 0.01	$3.75 \pm 0.01$
F35_3_MT	407 ± 25	494 ± 27	767 ± 31	273 ± 4	$3.46 \pm 0.02$	$3.65 \pm 0.09$
F27.5_2_LT	390 ± 3	409 ± 6	631 ± 10	222 ± 4	3.67 ± 0.06	$3.86 \pm 0.06$
F27.5_2_HT	425 ± 15	426 ± 52	640 ± 10	215 ± 42	4.13 ± 0.05	4.17 ± 0.08
F27.5_3_LT	355 ± 4	355 ± 1	553 ± 13	198 ± 12	3.70 ± 0.07	$3.80 \pm 0.11$
F27.5_3_HT	394 ± 16	401 ± 30	639 ± 42	238 ± 12	$4.41 \pm 0.06$	4.28 ± 0.11
F27.5_2.5_MT_a	349 ± 8	370 ± 4	612 ± 2	242 ± 1	3.79 ± 0.08	3.76 ± 0.09
F27.5_2.5_MT_b	404 ± 7	405 ± 3	642 ± 6	237 ± 3	$3.82 \pm 0.07$	3.70 ± 0.09
F27.5_2.5_MT_c	347 ± 12	350 ± 10	564 ± 13	214 ± 4	$3.81 \pm 0.00$	3.67 ± 0.10
F_raw	656 ± 15	1,159 ± 12	1,642 ± 28	484 ± 16	$1.52 \pm 0.02$	$2.32 \pm 0.03$
M20_2.5_LT	238 ± 12	275 ± 26	494 ± 36	219 ± 22	$3.33 \pm 0.05$	$3.23 \pm 0.10$
M35_2.5_LT	310 ± 24	401 ± 2	719 ± 8	319 ± 10	$2.88 \pm 0.03$	2.98 ± 0.09
M20_2.5_HT	259 ± 21	266 ± 18	474 ± 40	208 ± 1	$3.93 \pm 0.07$	$4.02 \pm 0.01$
M35_2.5_HT	606 ± 3	674 ± 11	962 ± 2	288 ± 3	$3.80 \pm 0.11$	$3.42 \pm 0.07$
M20_2_MT	297 ± 13	319 ± 23	544 ± 23	225 ± 5	$3.67 \pm 0.01$	$3.85 \pm 0.06$
M35_2_MT	270 ± 10	362 ± 9	627 ± 6	266 ± 4	$3.08 \pm 0.06$	$2.91 \pm 0.02$
M20_3_MT	274 ± 1	290 ± 1	516 ± 4	226 ± 20	$3.91 \pm 0.04$	$3.91 \pm 0.03$
M35_3_MT	385 ± 15	490 ± 13	776 ± 11	286 ± 14	$3.05 \pm 0.01$	$3.28 \pm 0.01$
M27.5_2_LT	290 ± 9	309 ± 6	532 ± 8	223 ± 13	$3.53 \pm 0.03$	$3.66 \pm 0.01$
M27.5_2_HT	494 ± 23	493 ± 6	745 ± 8	252 ± 40	$3.82 \pm 0.06$	3.83 ± 0.12
M27.5_3_LT	437 ± 30	447 ± 23	690 ± 16	243 ± 7	$3.51 \pm 0.03$	3.60 ± 0.02
M27.5_3_HT	450 ± 10	470 ± 12	763 ± 50	293 ± 9	4.33 ± 0.01	4.13 ± 0.02
M27.5_2.5_MT_a	406 ± 8	449 ± 22	690 ± 27	242 ± 11	$3.50 \pm 0.06$	3.52 ± 0.09
M27.5_2.5_MT_b	423 ± 5	465 ± 22	713 ± 3	248 ± 15	3.58 ± 0.02	3.74 ± 0.01
M27.5_2.5_MT_c	430 ± 6	476 ± 3	725 ± 17	249 ± 3	$3.50 \pm 0.04$	3.45 ± 0.02
M raw	637 ± 25	1 099 + 7	1 602 + 21	503 + 13	1 58 + 0.02	2 34 + 0 03

39 Note: db, dry basis; F\_raw, nonextruded Fuji powder; FV, final viscosity achieved at the end of the cooling cycle; LARC, lactic acid retention capacity; M\_raw, nonextruded Medalist powder; SB, setback; V1, viscosity at the beginning of the holding period at 95°C; V2, viscosity reached at the end of 40 the holding period at 95°C; WRC, water retention capacity. 41

43 Thus, the calculated models should be considered reliable only for 44 the corresponding bean variety. In general, Fuji models showed 45 higher significance levels and a nonsignificant LOF that is related to an adequate fitting of the design space. In contrast, most of the 46 Medalist models showed a significant LOF, and predicted R<sup>2</sup> gen-47 erally lower than those of Fuji models, meaning a lower prediction 48 49 ability. Nevertheless, the adequate precision for all the reported 50 models ranged from 7 to 90 (data not shown), indicating a high 51 signal-to-noise ratio in the design space.

52 Feed moisture was the most significant factor, affecting all the 53 considered characteristics of extruded powders, while feed rate was

the least significant one. Many interaction and quadratic effects were significant, suggesting that the effect of one factor is dependent on the other experimental conditions. In particular, nine models of the 18 considered showed quadratic effects, mainly of feed moisture and die temperature, indicating a nonlinear relationship among the response variable and the experimental factors. Indeed, response surfaces for those characteristics presented a concavity or a convexity, depending on the sign of the quadratic effect, and the maximum or minimum value of the response variable was inside the experimental range.

The best models for Fuji were obtained for expansion value and RS, which showed the highest values of  $R^2$ , adjusted  $R^2$ , and

48 49 50 51 52 53 <b>T ABLE 4</b> 50 51 52 53	45 46 47	41 44 44 44 44 44 44 44 44 44 44 44 44 4	oded factor 85	s) of significa	32 33 34 tu	28 29 29 20 29 20 20 20 20 20 20 20 20 20 20 20 20 20	26 pour leimo	22 23 24 25 25 26 25 26 26 26 26 26 26 26 26 26 26 26 26 26	19 <b>20 21 2 3 4 5 5 1 1 1 1 1 1 1 1 1 1</b>	15 16 17 18 18 18	12 12 13 14 14 variance fo	9         9           10         10           11         11	6 6 7 8 8 8	1 2 2 3 3 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9
for the extrusion of Fu	iji and M "	edalist bean p ″	owders "	0	9	0	0	0	0	0	<b>n</b> 2	A.1: D <sup>2</sup>	D1 D2	
kesponse variable	p <sub>0</sub>	P1	p <sub>2</sub>	p <sub>3</sub>	$p_{12}$	P <sub>13</sub>	P <sub>23</sub>	P <sub>11</sub>	P <sub>22</sub>	P 33	¥	AdJ. K	Pred. K	LUF (p-value)
Fuji Otebo	,													
Expansion value (%)	65.3	-34.9***	7.7***	0.5 n.s.	8.1***	$15.1^{***}$		17.4***	3.5**	-8.0***	.999***	.996	.989	.174 n.s.
RDS (g/100 g db)	20.8	2.8**	-4.6***	-0.1 n.s.							.898***	.864	.741	.109 n.s.
SDS (g/100 g db)	12.1	-6.4***	0.2 n.s.	-2.0**		2.2*	4.0***		-3.8**		.974***	.947	.860	.441 n.s.
RS (g/100 g db)	3.2	2.0***	4.4***	-1.2**				1.9**	2.5***	3.1***	.994***	.986	.957	.510 n.s.
V1 (cP)	380	17 n.s.	-13 n.s.	17 n.s.				-99***			.894***	.842	.781	.826 n.s.
V2 (cP)	375	87***	2 n.s.	40**							.755**	.688	.491	.263 n.s.
FV (cP)	609	$112^{***}$	4 n.s.	37*							.847***	.806	.697	.531 n.s.
SB (cP)	234	$21^{**}$	8 n.s.	-1 n.s.							.658*	.543	.261	.660 n.s.
WRC (g/g db)	3.80	-0.19***	0.05 n.s.	0.28***	0.14*			-0.24***		0.18**	.954***	.920	.764	.029*
LARC (g/g db)	3.76	-0.21***	0.05 n.s.	0.20***	0.15*			-0.18**		0.23**	.947***	.901	.705	.179 n.s.
Medalist														
Expansion value(%)	66.5	-38.8***	11.2 n.s.	10.6 n.s.							.900***	.863	.807	.174***
Moisture (g/100 g)	9.9	0.2***	-0.2***	-0.2***		-0.3***				-0.7***	.999***	766.	.996	.773 n.s.
V1 (cP)	418	63*	24 n.s.	67*		69 n.s.				-89*	.780**	.658	.138	.031*
V2 (cP)	412	97**	27 n.s.	59*							.637**	.538	.270	.027*
FV (cP)	664	132***	37 n.s.	64*							.737**	.665	.462	.043*
SB (cP)	249	29***	10*	$11^{**}$							.840***	.792	.637	.084 n.s.
WRC (g/g db)	3.68	-0.26**	0.09 n.s.	0.33***				-0.23*			.816**	.742	.563	.047*
LARC (g/g db)	3.61	-0.37***	0.08 n.s.	0.17*							.795***	.434	.558	.427 n.s.
Note: $\beta_0$ , intercept; $\beta_1$ , c of the interaction betw coefficient of the quad Abbreviation: db, dry b.	oefficient een feed atic term asis; FV, f	of feed moist moisture and $c$ of feed rate, $\beta$ inal viscosity a	are; $β_2$ , coeffi lie temperatu $_{33}$ , coefficien chieved at th	cient of feed r rre; $\beta_{23}$ , coeffic t of the quadr e end of the c	ate; $\beta_{3}$ , coef cient of the i atic term of ooling cycle;	ficient of die nteraction b die tempera	temperatu etween fee ture; R <sup>2</sup> , co c acid reter	Ire; $\beta_{12}$ , coeffierd and diversify the set of diversify of the difficient of the distribution capacity of the set of the distribution capacity of the set of the distribution capacity of the set	cient of the e temperatu eterminatior ; RDS, rapidl	interaction be re; $\beta_{11}$ , coeffic r; Adj. $\mathbb{R}^2$ , adju y digestible s	etween feed i cient of the quisted R <sup>2</sup> ; Precent tarch; RS, res	moisture and uadratic terr 1. R <sup>2</sup> , predict iistant starch	d feed rate; m of feed m ted R <sup>2</sup> ; LOF, h; SB, setbao	<ul> <li>3<sub>13</sub>, coefficient</li> <li>bisture; β<sub>22</sub>,</li> <li>lack of fit.</li> <li>k; SDS, slowly</li> </ul>
digestible starch; V1, vi Significance levels: n.s.,	scosity at not signi	the beginning ficant.	of the holdir	ig period at 95	5°C; V2, visc	osity reache	d at the en	d of the holdir	ng period at	95°C; WRC, v	vater retentio	on capacity.		
*p < .05; **p < .01; ***p	< .001.												K	

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predicted R<sup>2</sup>. All experimental factors significantly affected these responses, also with interaction and quadratic effects creating curvatures and torsions in the corresponding response surfaces (Figure 2). 4 According to the calculated model, a higher expansion value can be obtained with low feed moisture levels and a medium temperature range, in agreement with previous discussion (Section 3.1). The feed rate effect on expansion value was more important at high feed 8 moisture levels (Table 4; Figure 2a). RS was highly and positively affected by feed rate and moisture (Figure 2b), and negatively re-10 lated to die temperature (Table 4). Thus, the highest RS values can be obtained with extrusion performed at high feed rate and moisture levels, but at low temperatures. These processing conditions correspond to lower thermal and mechanical stresses, causing a lower degree of starch gelatinization and, consequently, a lower accessibility of starch to enzyme digestion (i.e., higher RS). For starch fractions of

Fuji samples, quite good models were obtained, indicating a major and negative role of feed rate on RDS and of feed moisture on SDS. Actually, low feed rates imply a higher thermal stress, accounting for higher starch disorganization and higher RDS content. In fact, low feed rates infer a lower amount of material inside the extruder and potentially a longer residence time. In contrast, low levels of feed moisture mean a higher solid-like behavior of the melt and higher stresses due to frictional forces and longer residence times; thus, the native organization of starch can be lost. High SDS levels were found for Fuji samples treated at low levels of feed moisture, suggesting an increase of starch granule compactness. This is in agreement with Cappa, Lucisano, Barbosa-Cánovas, and Mariotti (2016) who reported the formation of a more compact structure in starchy powders treated at high hydrostatic pressure in the presence of low amount of water.



FIGURE 2 Response surfaces for: (a) expansion value and (b) resistant starch (RS) of Fuji extruded bean powders; (c) moisture and (d) 52 expansion value of Medalist extruded bean powders. The experimental factor not shown is always set at the intermediate level (i.e., feed 53 moisture = 27.5 g/100 g; feed rate = 2.5 kg/hr; die temperature = 100°C)

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1 No significant models were obtained for starch fractions of 2 Medalist powders, suggesting that bean genotype affects starch granule organization and susceptibility to extrusion. The best 4 Medalist models were obtained for powder moisture and expansion 5 value (Figure 2c,d), even if the latter model presented a significant LOF (Table 4). Moisture variations were small, but the model indi-6 cated that higher values were obtained when feed moisture, feed 8 rate, and die temperature ranges were 33-35 g/100 g, 2-2.2 kg/hr, 9 and 80-105°C, respectively. A linear model was obtained for expan-10 sion value, in which only feed moisture had a significant and negative effect (Table 4; Figure 2d): the lower the feed moisture, the higher 11 12 the friction forces inside the extruder and greater the pressure drop 13 across the extruder die opening.

14 Significant models were obtained for all the considered pasting 15 properties, with a better performance for Fuji powders (Table 4). 16 Quadratic models were calculated for V1, while the other indices 17 showed linear models. Feed moisture was the most significant fac-18 tor: with an increase in feed moisture, increases in all RVA-indices 19 were measured, suggesting a partial disorganization of starch gran-20 ules during extrusion, due to the lower residence time in the extruder caused by the more liquid-like behavior of the melt. In fact, when na-21 tive starch is subjected to heating and cooling phases in the presence of water, such as in the RVA test, starch gelatinizes, and retrogrades, 23 24 resulting in higher values of PV and FV. However, if starch is already 25 partially or totally gelatinized and retrograded, no further increases in viscosity are measured during the RVA test. Accordingly, bean 26 27 powders extruded under higher feed moisture levels showed higher 28 PV and FV values, denoting that a lower starch gelatinization had 29 occurred during extrusion. In contrast, feed rate did not significantly 30 affect the pasting properties, with the exception of SB of Medalist 31 powders. When significant, die temperature had a direct effect on 32 RVA indices. Actually, the highest values of V1, V2, FV, and SB in Fuji powders were obtained with the higher levels of both feed moisture 34 and die temperature. These treatment conditions implicate a more 35 liquid-like behavior of the melt (and potentially less friction stress) 36 inside the extruder, causing fewer changes in starch, which, in con-37 trast, swells, gelatinizes and retrogrades more during the RVA test.

Both feed moisture and die temperature resulted in statistically 39 significant WRC and LARC models of both bean varieties (Table 4), 40 but with opposite effects: retention capacity values increased with decreasing feed moisture but with increasing extrusion temperature, 41 42 due to a higher disorganization of starch granules and a higher exposition of hydrophilic groups, as already reported by Ai et al. (2016). 43 44 Feed rate was not associated with significant changes in retention 45 capacities of extruded powders, but its interaction with moisture was significant for Fuji samples, indicating a higher effect of feed 46 47 rate at the highest values of feed moisture.

#### CONCLUSIONS 4

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52 Results of this study demonstrated the suitability of DoE techniques 53 and RSM for the multivariate study of effects of extrusion cooking

on bean powder physical and chemical properties. Bean genotype had a relevant effect on sample characteristics, thus the obtained models should be considered reliable only for the corresponding bean variety. In further researches, the calculated models can be applied to define the appropriate extrusion conditions to design tailor-made bean powders to be used as new ingredients in food formulations. In such studies, the evaluation of the sensory properties of the final products should be included.

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#### CONFLICT OF INTEREST

The authors have declared no conflicts of interest for this article.

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