



# Evaluation of crisps from maize-integral grape pomace flour mix manufactured by extrusion cooking

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**Abstract:** Integral grape pomace is a significant source on nutrients and can be valorized in maize crisps production industry. Being a fiber-rich ingredient, it is important to determine the optimal amount that can be included to keep acceptable quality parameters of the final product. Thus, this paper aims to investigate the impact of integral grape pomace (red and white) on maize crisps physical properties and starch digestibility. For this, a Response Surface Methodology design was applied and the optimization was performed by using desirability function. The increase of grape pomace dose led to the reduction of lightness, expansion index, crunchiness, and WAI and the increment of browning index, cutting, compression and puncture forces, crispness, and total starch. The optimal samples containing 20.50% white grape pomace and 18.28% red grape pomace respectively showed lower L\*, texture forces and functional properties compared to the control, while the porosity was enhanced. The molecular characteristics of the optimal samples were similar and some differences were found compared to the control. These results could be used for further researches on the chemical properties of the enhanced crisps and/or to develop this kind of products at an industrial level.

**Keywords:** integral grape pomace, corn expanded snacks, extruder, functional product, optimization.

## 1. Introduction

In the context of circular economy concept, more and more by-products are used in new and different processes. One of these is represented by the extrusion-cooking process, a continuous process that involves forcing a raw material mixture through a die under controlled conditions of temperature, pressure and shear [1,2]. New products with desired and unique characteristics, such as shape, color, texture, flavors etc. are outcomes of transformation of the raw material in this process that, in the same

time, reduced water consumption and minimized the energy needed [3]. The extrusion processing technology is recognized for its versatility, efficiency, and ability to change the functional and nutritional properties of raw materials through mixing, cooking, kneading, shearing, shaping, and forming [4,5]. Various chemical and structural transformations, such as starch gelatinization, protein denaturation, complex formation between amylose and lipids, and degradation reactions of vitamins and pigments take place during extrusion [6] influencing finite product quality.

Wine processing industry generally discharge high quantity of grape byproducts which include seeds, skin, pulps, and stems from grapes. These byproducts are rich in compounds that provide potential nutritional value and nutraceutical functionality for developing new and sustainable functional foods. Their used as functional ingredients in new food products represents a way to enhance nutritionally foods and, in the same time, a possible alternative to reduce byproducts negative impact on the environment by diminishing the carbon footprint.

Fiber-rich ingredients like fruit pomaces can exert different effects on the appearance and texture of the expanded products [7]. Even if the nutritional profile of the final product matters, the physical and sensory characteristics such as expansion index and texture of the extruded crisps have a great importance [8]. These characteristics depend on the quantity and type of the available starch which determines the amount and size of gas bubbles formed in the extrusion process [9]. The expansion index and the microstructure of crisps are dictated by starch gelatinization which depends on the extrusion cooking parameters and ingredients chemical profile [7,10].

Fruit pomaces powders can be considered natural sources of pigments. Kita et al. [11] reported that cranberry, chokeberry and blackcurrant juice powders incorporation in extruded crisps resulted in more pleasant color and increased hardness. Another study demonstrated that the incorporation of 11% apple, banana, strawberry and tangerine spray dried powders in extruded crisps determines considerable influence on expansion index and density of the final product [12]. The effects on fruits ingredients are different depending on the type used. Attenborough et al. [13] reported that freeze-dried ripe and unripe jackfruit enhanced the microstructure, aroma and nutritional profile of the extruded crisps at addition levels up to 30%.

To our knowledge, there is no paper presenting the combined effect of grape variety and dose (10-40%) on maize crisps quality. Considering these aspects, this paper aimed to investigate the effects of integral grape pomace (red and white) on maize

snacks physical, sensory and starch digestibility in order to establish the optimal dose.

## **2. Materials and methods**

### **2.1. Ingredients**

Whole grape pomace (red – GPR and white – GPW) was supplied by a research institute from Romania. Maize flour was acquired from a Romanian producer. After drying at 50 °C and grinding, grape pomace was sieved (0.2 mm mesh) and the feed mixtures were prepared by replacing maize with GPR or GPW (10–40%). The moisture content of the mixtures was adjusted to 15%.

### **2.2. Crisps production**

The extruded crisps were produced on a laboratory single-screw extruder (Kompakt extruder KE 19/25, Brabender, Duisburg, Germany) with a barrel diameter of 19 mm and a length-to-diameter ratio of 25:1. The diameter nozzle had 2 mm, the feeding rate was kept constant at 24 rpm, and the screw speed was established to 150 rpm. Four temperatures zones were set at 50°C, 95°C, 175°C, and 180°C. Equilibration of the crisps was done for 16 h before packing.

### **2.3. Expansion index and porosity**

For the Expansion Index (EI) determination the crisps were measured a digital caliper. Porosity was determined by displacement method: 15 crisps samples were put in a 250 mL cylinder filled with mustard seeds. The difference before and after inserting the crisps represented the bulk volume. After grinding the volume of the samples (powder) was recorded again and this represented the apparent volume. The difference between the bulk and apparent volumes estimated porosity [14].

### **2.4. Total starch and starch digestibility index (SDRI)**

The Megazyme kit (K-DSTRS; Megazyme, Bray, Ireland) was used for total starch determination, and the Starch Digestion Index (SDRI) was determined as the ratio of rapidly digestible starch (after 20 min of digestion) and total starch.

### **2.5. Chromatic properties**

Luminosity ( $L^*$ ), green to red nuance ( $a^*$ ), and yellow to blue nuance ( $b^*$ ) were determined using a CR-400 chromameter (Konica Minolta, Tokyo, Japan). The browning index was then calculated according to previous studies [15].

### **2.6. Texture**

A TVT 6700 texturometer (Perten Instruments, Hägersten, Sweden) was employed for texture forces determination. Compression force was measured by compressing 50% of the samples (1 cm length) using a 25 mm cylindrical probe. A V-shaped cutting blade was used for cutting force measurement on a sample with 4 cm length, and the crispness was determined as the slope of the first peak. The puncture force was measured with a 3 mm diameter puncture probe on samples of 1cm length. The test speed for all the methods was set to 1 mm/s.

**2.7.Crunchiness and chewiness**

A sensory analysis was performed for crunchiness and chewiness evaluation. The crisps were tasted by 65 panelists from the Food Engineering Faculty at Stefan cel Mare University of Suceava who were trained before. A 90-points scale was used.

**2.8.Water absorption (WAI) and solubility (WSI)**

WAI was measured by mixing 2.5 g of ground crisps with 25 mL of water for 30 min followed by centrifugation at 3000 rpm for 15 min [14]. The liquid part was separated and the solid was weighed. The liquid was evaporated to a constant weight to measure the WSI [14].

**2.9.Molecular properties evaluated by FT-IR**

Crisps molecular vibrations were investigated by using a Thermo Scientific Nicolet iS20 FT-IR spectrophotometer (Waltham, MA, USA) in attenuated total reflection (ATR) mode. The resolution was 4 cm<sup>-1</sup> and 32 scans were performed for each sample. The molecular vibrations were interpreted according to the literature [16,17].

**2.10.Data modelling and statistics**

The experimental design consisted of two factors: A – grape variety (white – GPW and red – GPR) and B – Dose (10, 20, 30, 40%). The real and coded values of the factors are presented in Table 1.

The polynomial cubic, quadratic or 2FI regression mathematical models were used for data fitting. The suitability of the models selected was established through sequential *F*-test, coefficients of determination (*R*<sup>2</sup>), adjusted coefficients of determination (*Adj.-*

*R*<sup>2</sup>), and significant probabilities. ANOVA was employed to evaluate the significance of the coefficients of the models and the results were considered significant of *p* < 0.05.

**Table 1.** Real and coded values of the factors

Run	Real		Coded	
	A-type	B-level (%)	A-type	B-level
1	GPW	30	{ -1 }	0.333
2	GPR	10	{ 1 }	-1.000
3	GPR	30	{ 1 }	0.333
4	GPR	40	{ 1 }	1.000
5	GPR	40	{ 1 }	1.000
6	GPW	10	{ -1 }	-1.000
7	GPW	20	{ -1 }	-0.333
8	GPW	30	{ -1 }	0.333
9	GPW	30	{ -1 }	0.333
10	GPW	40	{ -1 }	1.000
11	GPW	20	{ -1 }	-0.333
12	GPR	20	{ 1 }	-0.333
13	GPR	30	{ 1 }	0.333
14	GPW	40	{ -1 }	1.000
15	GPW	10	{ -1 }	-1.000
16	GPW	10	{ -1 }	-1.000
17	GPW	20	{ -1 }	-0.333
18	GPR	10	{ 1 }	-1.000
19	GPR	20	{ 1 }	-0.333
20	GPR	10	{ 1 }	-1.000
21	GPR	20	{ 1 }	-0.333
22	GPW	40	{ -1 }	1.000
23	GPR	40	{ 1 }	1.000
24	GPR	30	{ 1 }	0.333

Stat-Ease Design-Expert software (trial version) was used for data modelling. The optimization of the dose added for each variety of grape was made through multiple responses analysis and desirability function. The following constraints were applied: total starch, SDRI, L\*, and BI were kept in range, porosity, EI, crispness and WSI were maximized, and the compression force, cutting force, puncture force, and WAI were minimized.

Means comparisons was done by applying Tukey test (*p* < 0.05) by using XL STAT 2024 version.

**3. Results and discussion**

The experimental data was fitted to the quadratic, cubic or 2FI models which showed acceptable adequacy (*R*<sup>2</sup> > 0.68). The *adj.-R*<sup>2</sup> values were close enough to *R*<sup>2</sup> to confirm models' suitability, and all models were significant at *p* < 0.01. The effects of factors on the studied variables are displayed in Table 2.

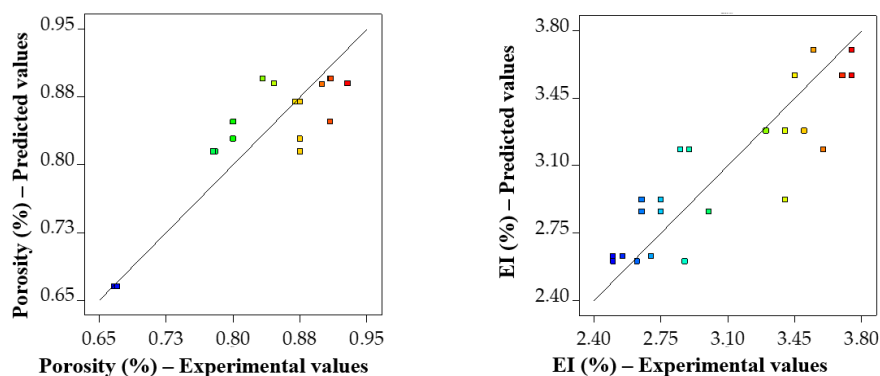
**Table 2.** Mathematical modelling of data

Factor	Porosity (%)	EI (%)	Total starch (%)	SDRI	L*	BI	Cut force (N)	Crispness (N/s)	Compression force (N)	Puncture force (N)	Crunchiness	Chewiness	WAI (%)	WSI (%)
Const.	0.89	3.05	41.71	0.82	53.50	29.67	7.75	5.10	16.51	8.57	51.79	42.21	4.73	23.20
A-grape variety	0.02	-0.04	2.95**	-0.03**	-2.41**	-4.56**	0.49**	0.56**	3.60**	2.12**	-1.87**	-0.34*	-0.02	-3.51**
B-Dose	-0.04	-0.51**	7.46**	0.01	-5.31**	3.02**	0.87**	0.86**	3.41**	1.90**	1.70*	1.92**	-0.49**	-0.37
AB	0.02*	0.03	2.46**	0.03**	-1.26**	-1.06**	1.08**	-0.08	2.10**	0.41*	3.11**	0.93**	-0.04	1.49
B <sup>2</sup>	-0.09**	0.07	3.61**	-0.01*	1.95**	-0.62*	-1.01**	-0.48*				-0.04	-0.01	-6.65**
AB <sup>2</sup>	-0.07**			0.03**										
B <sup>3</sup>	0.09*			0.01										
p-value	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
R <sup>2</sup>	0.81	0.76	0.96	0.87	0.99	0.99	0.86	0.84	0.94	0.93	0.68	0.91	0.79	0.70
Adj.-R <sup>2</sup>	0.75	0.72	0.95	0.83	0.98	0.98	0.84	0.80	0.93	0.93	0.63	0.89	0.75	0.64

\* - significant at  $p < 0.05$ , \*\* - significant at  $p < 0.01$

Crisps expansion index was affected significantly ( $p < 0.05$ ) by grape pomace dose (Table 2). Porosity was affected only by the linear and quadratic terms of factors interaction and by the quadratic and cubic terms of dose ( $p < 0.05$ ). Both porosity and expansion index decreased with dose increase. Giannini et al. [18] reported smaller porosity and expansion index of corn snacks with apple and oat fibers. The results of the present study

may be related to the fat and fiber content of the integral grape pomace used [18]. Expansion ratio depends on the size, quantity and position of the air bubbles surrounded by the cooked network. It was demonstrated that fiber-rich ingredients decrease cell size, possibly due to the premature break of gas bubbles, which consequently diminished snacks expansion [19]. The predicted vs. experimental values are displayed in Figure 1.



**Figure 1.** Predicted vs. experimental data for the expansion properties

Total starch content was influenced by grape variety, the linear interaction between factors and by the linear and quadratic term of dose ( $p < 0.05$ ). SDRI was affected significantly ( $p < 0.05$ ) by grape variety, the quadratic term of dose and by the linear and quadratic terms of factors interactions (Table 2). Total starch and SDRI increased with dose increment.

Lou et al. [20] obtained a high digestibility of cookies with grape pomace, with a predicted glycemic index  $> 90$  and recommended *in vivo* studies for clarifications. Predicted vs. experimental values for SDRI and total starch are showed in Figure 2.

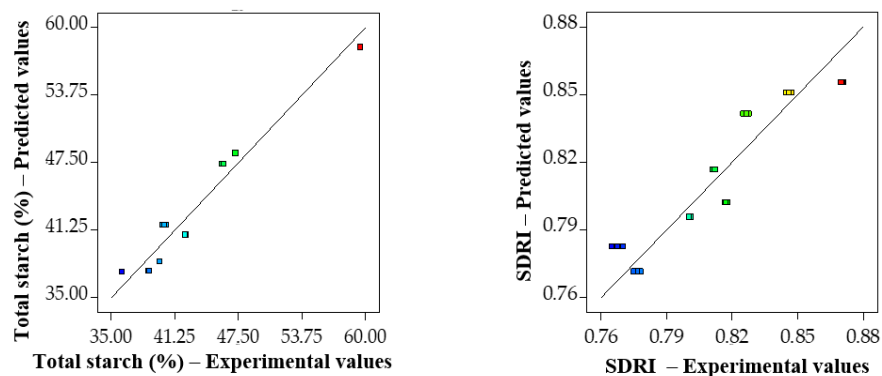


Figure 2. Predicted vs. experimental data for starch digestibility properties

Both chromatic parameters ( $L^*$  and BI) were significantly influenced ( $p < 0.05$ ) by grape variety, and the linear and quadratic terms of dose (Table 2). The interaction between factors also had a significant effect on the chromatic properties.  $L^*$  decreased and BI increased proportional with dose increment. Similar to our results, Marcos et al. [21] observed a reduction of  $L^*$  and nuance and

different hue angle values of crackers depending on grape pomace variety (white or red), confirming thus the significant impact of both addition level and type of grape pomace on final product chromatic properties. Experimental data vs. predicted values of chromatic properties are shown in Figure 3.

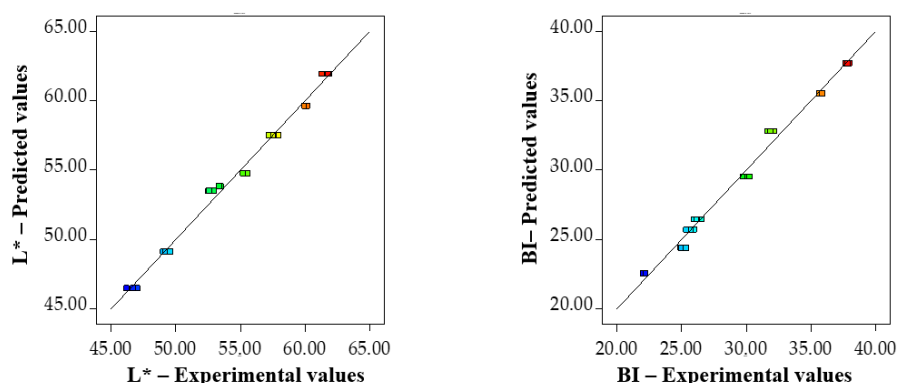


Figure 3. Predicted vs. experimental data for chromatic properties

Texture forces (cut, compression and puncture) and crispness were significantly affected by both factors and their interactions ( $p < 0.05$ ). Cut force and crispness were also influenced by the quadratic terms of dose (Table 2). Cut, compression, and puncture forces showed an increasing trend with dose increment. Navarro Cortez [22] also reported lower peak force values of blue corn snacks with orange bagasse. The maximum force is a measure of cells resistance to breakage, which is actually influenced by the number of cells per volume and their structure [22].

Crispness raised as the dose of grape pomace was higher. Wójtowicz et al. [23] obtained higher crispness of gluten free snacks with chokeberry and elderberry powders as the dose increased. It was reported that the raise of sugars level up to a certain level increases crispness [24]. Thus, the presence of sugars from grape pomace could explain the results obtained for crispness.

Figure 4 shows the fitting between the experimental and predicted values for the texture and sensory parameters.

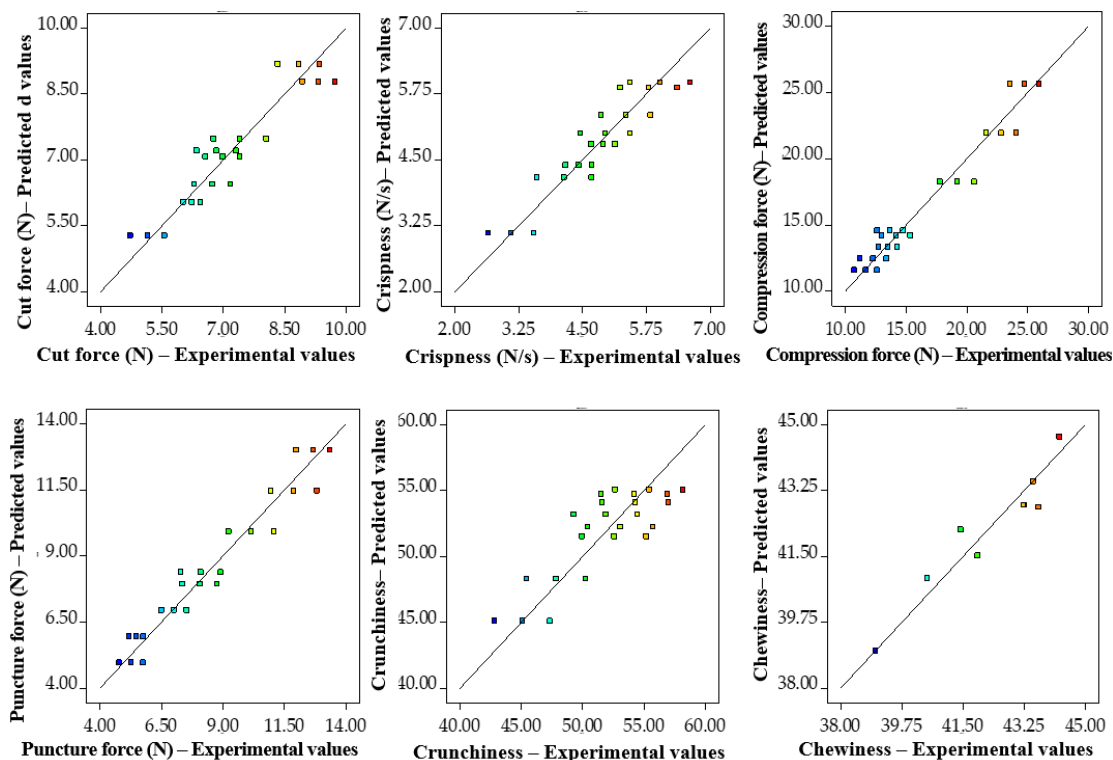


Figure 4. Predicted vs. experimental data for texture and sensory properties

Crunchiness and chewiness sensory properties were significantly influenced by both factors and their linear interaction ( $p < 0.05$ ). A raise of crunchiness was obtained as the dose of grape pomace increased, and the same trend was found also for chewiness. Kloepfer [25] also reported increasing chewiness and decreasing crunchiness of corn extruded snacks with apple or cranberry

pomace as the amount added was higher. Crisp structure plays a crucial role in crunchiness determination because it is affected by the size and number of pores [24]. Fibers, especially the insoluble ones, are one of the main components affecting product chewiness. The dependence between the experimental and the predicted values for WSI and WAI are displayed in Figure 5.

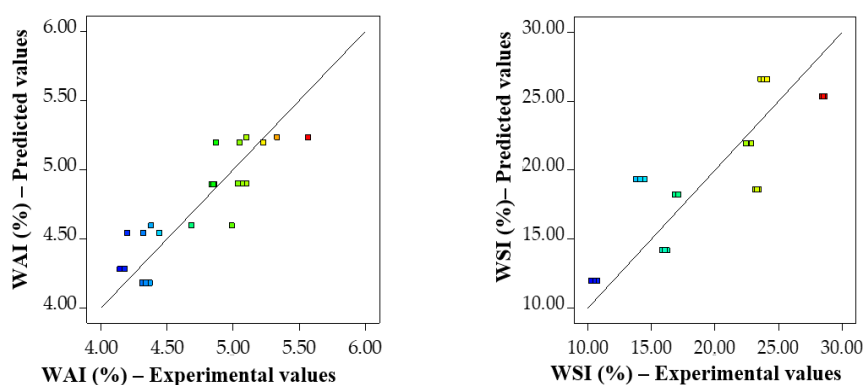


Figure 5. Predicted vs. experimental values for functional properties

WAI was significantly influenced ( $p < 0.05$ ) only by grape pomace dose, while WSI was affected by grape variety and the quadratic term of dose (Table 2). Both WAI and WSI decreased with the dose of grape pomace increase. Navarro Cortez et al. [22] found that

the addition of orange pomace in corn extrudates resulted in lower WAI and WSI. The high amount of fibers from grape pomace could be responsible for WSI decrease because this compounds tend to link to the starch chains and to disturb the

dextrinization which led to changes in snacks solubility [26].

Pearson correlations between crisps characteristics are showed in Figure 6. Total starch content and expansion index was positively correlated with texture parameters. L\* was negatively correlated with texture forces and crispness. EI was negatively correlated with puncture force, which was in line with the findings of Navarro Cortez [22] for blue corn snacks with orange bagasse.

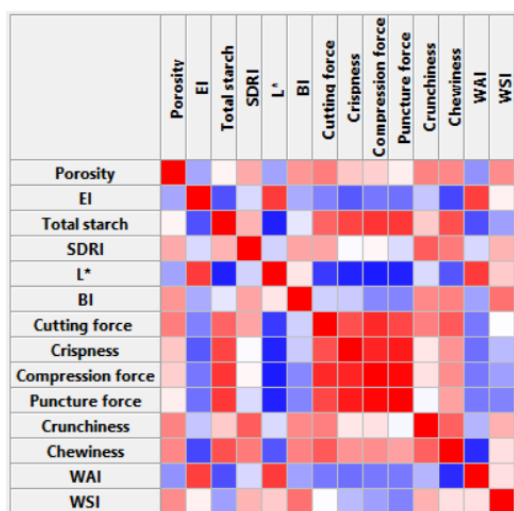


Figure 6. Pearson correlations coefficients

### Optimization

The optimal dose for white grape pomace was established to 21.50% (GPW). The optimal crisp with red grape pomace contains 18.28% (GPR). It was found that the expansion index of the optimal samples was lower than of the control probably due to the high intake of fibers that disturbed gas cells formation. GPR and GPW exhibited higher total starch content. The lowest SDRI was observed in GPR crisp (Table 3). L\* and BI were lower in GPW and GPR compared to the control and this was expected because of the presence of pigments from grape pomaces. Furthermore, the presence of sucrose in grape pomace promotes caramelization and Maillard reactions which determine differences in chromatic parameters [27].

The compression, puncture, and cutting forces, as well as the crispness of the control were higher compared to the optimal samples. This could be explained by the

weakening effect of fibers on crisps structure [8].

Table 3. Comparison between the optimal (predicted values) and control crisps properties

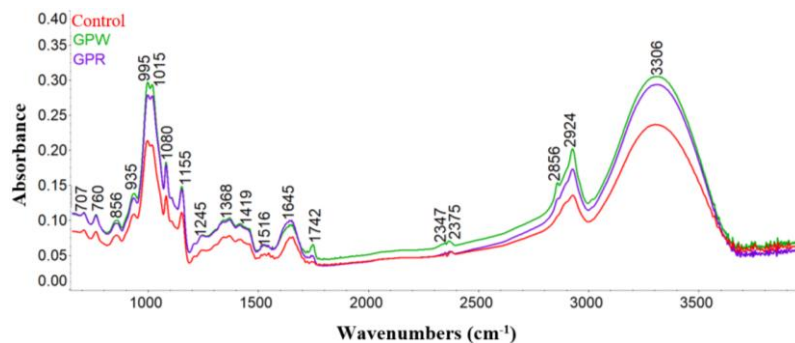
Property	GPW		GPR		Control	
	Mean	SD	Mean	SD	Mean	SD
Level (%)	21.50	-	18.28	-	0.00	-
Porosity (%)	0.89 <sup>a</sup>	0.04	0.88 <sup>a</sup>	0.04	0.73 <sup>b</sup>	0.02
EI (%)	3.22 <sup>b</sup>	0.24	3.25 <sup>b</sup>	0.24	3.70 <sup>a</sup>	0.03
Total starch (%)	37.83 <sup>b</sup>	1.52	40.95 <sup>a</sup>	1.52	35.90 <sup>b</sup>	0.00
SDRI	0.86 <sup>a</sup>	0.01	0.78 <sup>c</sup>	0.01	0.84 <sup>b</sup>	0.00
L*	56.90 <sup>b</sup>	0.55	54.43 <sup>c</sup>	0.55	78.48 <sup>a</sup>	0.12
BI	33.29 <sup>a</sup>	0.59	24.11 <sup>b</sup>	0.59	31.98 <sup>a</sup>	0.40
Cut force (N)	7.25 <sup>b</sup>	0.54	7.17 <sup>b</sup>	0.54	14.61 <sup>a</sup>	1.77
Crispness (N/s)	4.30 <sup>b</sup>	0.43	5.22 <sup>b</sup>	0.43	7.23 <sup>a</sup>	0.77
Compression force (N)	12.63 <sup>b</sup>	1.23	17.64 <sup>a</sup>	1.23	20.09 <sup>a</sup>	1.46
Puncture force (N)	6.12 <sup>b</sup>	0.71	9.66 <sup>a</sup>	0.71	10.10 <sup>a</sup>	1.13
Crunchiness	53.98 <sup>a</sup>	2.45	47.77 <sup>b</sup>	2.45	-	-
Chewiness	42.33 <sup>a</sup>	0.56	40.59 <sup>b</sup>	0.56	-	-
WAI (%)	4.85 <sup>b</sup>	0.21	4.95 <sup>a</sup>	0.21	4.52 <sup>c</sup>	0.19
WSI (%)	26.79 <sup>b</sup>	3.44	17.86 <sup>c</sup>	3.44	31.80 <sup>a</sup>	0.30

Mean values followed by different letters are significantly different ( $p < 0.05$ )

GPW was crispier and chewier compared to GPR. WSI values were found to be higher in control compared to GPW and GPR, which was in agreement with the data obtained by Navarro Cortez et al. [22] and can be due to the higher content of gelatinized starch of the control.

### Optimal crisps characteristics

GPW and GPR presented similar FT-IR spectra and intensities of the absorbances, but with considerable differences compared to the control. GPW and GPR presented an absorption band at 2856  $\text{cm}^{-1}$  which was not observed in control crisps (Figure 7). The absorption band observed at 3306  $\text{cm}^{-1}$  is given by the O-H stretching vibration, indicating the presence of water and/or cellulose and hemicellulose [28]. The presence of methylene group of polysaccharides was indicated by the bands at 2856 and 2924  $\text{cm}^{-1}$ , while the band observed at 1742  $\text{cm}^{-1}$  given by the C=O stretching vibration indicated the presence of uronic acid [28] or lipids from grape pomace [29]. The enrichment of the nutritional profile of maize crisps was confirmed by the molecular characteristics which indicated the presence of biological active compounds from grape pomace.



**Figure 7.** FT-IR characteristics of the optimal and control crisps

#### 4. Conclusion

This paper highlighted the impact of integral grape pomace on maize extruded crisps properties. The results showed that grape pomace dose increment resulted in lower  $L^*$ , porosity, EI, crunchiness, and WAI, and higher BI, cut, compression and puncture forces, and chewiness. The optimal doses for white and red grape pomace were found to be 21.50 and 18.28% respectively. Changes of the chromatic properties were observed in the optimal samples compared to the control. Furthermore, the optimal crisps exhibited smaller cut, compression, and puncture forces, crispness, and WSI values compared to maize control crisp. The expansion index was lower in the optimal samples, but the values were not highly affected. Consequently, the use of integral grape pomace from red and white varieties to create novel functional crisps is feasible since the properties of the final product were acceptable.

#### Compliance with Ethics Requirements

Authors declare that they respect the journal's ethics requirements. Authors declare that they have no conflict of interest.

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