

ACCEPTED ARTICLE

Orange tablet production enhancement by intervention of a dry method: A central composite strategy for experimental design and optimization

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Abstract

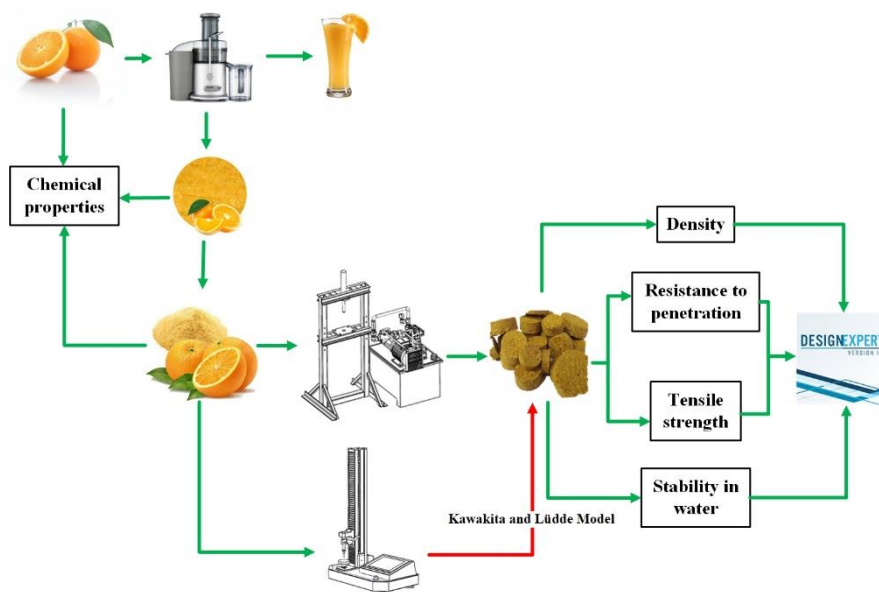
The present study aimed to develop orange pomace tablets as an additive with nutritional value. Orange pomace powder was converted into tablets by the drying method under the effect of compression force, diameter die and relaxation force changes. Some of the physicochemical properties (e.g., density, penetration resistance, tensile strength, stability in water, total phenol content, antioxidant capacity, and ascorbic acid) were investigated. The tableting process was optimized by central composite design. The Kawakita and Lüdde model with $R^2 = 0.995$ was used to describe the compressibility behavior of orange pomace powder. The effect of diameter die on the minimum tension of deformation changes was insignificant. The results proved that the changes in the value of the active ingredients were insignificant when the orange was dried, ground to powder, and converted to a tablet. Also, the effect of diameter dies changes on all of the responses was significant at 0.01, while the effect of compression force was insignificant just for penetration resistance. Effect of the relaxation force on the mechanical properties was significant (at level of 0.5 for Penetration resistance and 0.01 for Tensile strength) and on the density and stability in water was insignificant. Finally, the optimum point for tableting from orange pomace was suggested at the compression force of 8.6 kN, diameter die of 10 mm, and relaxation force of 1.14 kN for $1.13 \times 10^{-3} \text{ g mm}^{-3}$ of density, 111 N of Penetration resistance, 52.4 MPa of Tensile strength, and 51 g.s dl⁻¹ of Stability in water.

Practical applications

One of the most common processes in many chemical, food and pharmaceutical industries is the compression of powders to form solids due to its simplicity and low cost. on the other hand, tablets are the most common solid dosage forms for oral delivery of a specific amount of a nutrient. To reduce food waste and maximize the use of nutrition material, the orange pomace which was residual from juicing process orange pomace which are rich of different type of dietary fibers converted to the tablet by a dry method. Also, orange pomace tablets are more customer-friendly and can be stored for a long time.

35 **Keywords:** Orange, Tableting, Optimization, **Second compression**, Tablet formation
 36 threshold.

37 **Graphical abstract**



38
 39

Nomenclature	
a	Constant (rational number)
A	Diameter of the die
b	Constant (rational number)
B	Hight of the die
C	The degree of volume reduction
CF	Compression force (kN)
D	Tablet diameter (mm)
DD	Die diameter (mm)
F_{max}	Break force (N)
H	Tablet's height (mm)
K	The difference between the slop of every pair of consecutive points
M	Tablet's mass (g)
P	Pressure (pa)
RF	Relaxation force (kN)
V	Tablet's volume (mm^3)
V_0	The initial volume (mm^3)
ϵ	Strain (mm/mm)
ρ	Unit density (g/mm^3)
σ	Stress (kPa)
σ_t	Tablet tensile strength (MPa)

40 INTRODUCTION

41 Citrus fruits are among the favorite fruit products regarding their pleasant flavor and
42 nutritional value. The total world citrus production in 2020 was about 158 million tons, more
43 than half of which were produced by oranges (FAO, 2020). About one-third of the annual world
44 orange production is used for orange juice production (Pan *et al.*, 2019). Most of the horticulture
45 output of oranges is used to prepare juice and concentrates because they are a suitable
46 replacement for different beverages such as coffee, tea, and carbonated soft drinks (Oduntan
47 and Arueya, 2019). The juicing process usually produces a large amount of waste such as peel,
48 pomace, cores, unripe, and damaged fruits (Bozkir *et al.*, 2020; Gomez and Martinez, 2018),
49 leading to a serious environmental issue (Badaoui *et al.*, 2019). The residual solids contain
50 around 50% of the whole fruit weight. Therefore, several thousand tons of pomace are produced
51 as an agricultural by-product. Some post-harvest processing can convert these “waste”
52 materials into high-value products (Pan *et al.*, 2019; Huang and Ma, 2016). One of these
53 processes is making tablets from powders produced from food. In this study, unlike other
54 studies, the powder is converted into tablets by a dry method (Naji-Tabasi *et al.*, 2021a). In
55 addition to the fact that tableting by dry method is more applicable to industries, this method is
56 cheaper, faster and easier. On other hand, tableting is the one of the most usual methods for
57 preparation specific amount of food material. because production and using of them are easy
58 (Gaikwad and Kshirsagar, 2020).

59 Dietary fiber sources have multiple physiological advantages. Dietary fiber may affect bile
60 acid metabolism by adsorbing bile salts and their metabolites, resulting in a higher excretion of
61 bile acids in feces (Dai and Chau, 2017; Shahwar *et al.*, 2017). Based on solubility, dietary fiber
62 is divided into two categories: insoluble and soluble dietary fiber. The most common type of
63 dietary fiber is insoluble, which is found in fruits and vegetables (Huang *et al.*, 2019). The
64 recommended fiber intake for adults is 25 g d⁻¹ (Slavin, 2013). By-products from orange juice
65 extraction also are among the pectin-rich dietary fiber sources. Some orange varieties, like
66 Valencia, have a considerable dietary fiber of 64.3 g 100 g_{dry matter}⁻¹ (Quiles *et al.*, 2016). In
67 addition orange pomace have different usages such as source of essential oil, maize replacement
68 in broiler diet, pectinase enzyme refining of sunflower oil, input in the production of citric acid,
69 gluten-free bread, bakery products and extrudates (Oduntan and Arueya, 2019).

70 The high moisture content of by-product agriculture leads to short self-life and high storage
71 costs. By-products are perishable and more susceptible to rapid microorganism growth (Masud
72 *et al.*, 2020). Therefore, providing appropriate processing methods for this product is highly
73 important to reduce these costs and increase shelf life (Rashidi *et al.*, 2021). Drying by-product

74 is one of the most widely used methods to process and preserve this agricultural waste (Wang
75 *et al.*, 2021). The by-product in powder form has potential applications as food ingredients or
76 even biomass in food waste management (Rashidi *et al.*, 2021).

77 Fruits powders have high potential usage as food material in preparing different processed
78 food products (Romani *et al.*, 2018; Shaari *et al.*, 2018; Karam *et al.*, 2016). Like other kinds
79 of food powders, fruit powders are generally bulky and need more space for storage and
80 transportation. Compressing the powder up to a specific volume into a tablet form decreases
81 the fruit powder's volume and surface area, thereby lowering the chances of rehydration and
82 quality degradation (Aziz *et al.*, 2018). Ready-to-eat or ready-to-serve food and drinks are
83 becoming more popular every day. In this respect, fruit powder tablets (used as drinks or
84 chewed) should meet consumer demand (Saifullah *et al.*, 2016b).

85 In recent years, biomass has been one of the most useable renewable energy sources. The
86 initial material often has high water content, which decreases its energy potential. Therefore, it
87 is necessary to perform some pretreatment, such as drying (Mudryk and Werle, 2018).

88 In addition, **second compression**, also known as compression coating or press coating, is used
89 in pharma tablet production to improve some mechanical characteristics (Ascani *et al.*, 2019;
90 Foppoli *et al.*, 2017). In this study, the second compression was applied to investigate the effect
91 of **second compression** on the mechanical characteristics of the food or by-product tablets.

92 Considering the nutritious advantages of fruit pomace, some published studies have focused
93 on understanding and optimizing the tableting processes. Some other studies have considered
94 various usages of the compression properties of different agricultural wastes (Banožić *et al.*,
95 2021; Naji-Tabasi *et al.*, 2021b; Aziz *et al.*, 2018; Osorio-Fierros *et al.*, 2017; Saifullah *et al.*,
96 2016a; Saifullah *et al.*, 2016b; Gallo *et al.*, 2015; Etti *et al.*, 2014; Saifullah *et al.*, 2014; Taufiq
97 *et al.*, 2014; He *et al.*, 2013; Klein *et al.*, 2013; Mesnier *et al.*, 2013; Zea *et al.*, 2013; Yusof *et*
98 *al.*, 2012).

99 **Most of quality attributes of tablets manufacturing process such as appearance, content**
100 **uniformity, hardness, thickness, friability, Disintegration time and Dissolution time are affected**
101 **of Tablet compression process. The weight effect of different parameters on the tableting**
102 **process is different. So, parameters optimization is necessary to find a balance between the**
103 **effect of different parameter on the compression process (Jongwuttanaruk and Thavornwat,**
104 **2022; Garlapati and Roy, 2017). There are numerus method to processes optimization that**
105 **central composite design (CCD) is the one of the most popular method for optimization**
106 **(Homayounfar *et al.*, 2023; Chen *et al.*, 2022). Some researchers tried to optimize tablet**
107 **processing by CCD (Rashidi *et al.*, 2021; Ghasemi and Chayjan, 2018; Ghasemi *et al.*, 2018).**

108 Consumers usually like fruit tablets which dissolve in water quickly and used them in the form
109 of juice. fruit Tablets which can dissolve very quickly are more acceptable (Naji-Tabasi *et al.*,
110 2021a), so weight of stability in water consider as the most important response. On the other
111 hand, tablet density affect the dissolution of orange pomace powder tablet (Roslan *et al.*, 2021),
112 also high density is more suitable for transportation (Rashidi *et al.*, 2021). Although high value
113 of the penetration resistance and tensile strength are too suitable for transportation, a tablet with
114 high penetration resistance and tensile strength is not customer friendly.

115 To our knowledge, no research has been conducted on evaluating and optimizing the orange
116 pomace tableting process. Therefore, in this paper, the effect of pressure, diameter die, and the
117 second compression on some of the physical properties of orange tablets (i.e., density,
118 penetration resistance, tensile strength, and stability in water) was investigated and optimized
119 by central composite design (CCD).

120

121 MATERIALS AND METHOD

122 Powder preparation

123 The powder was prepared from orange (*Valencia*) pomace. For this purpose, fresh orange
124 juice was extracted by an electric juicer, and pomace as residuals was oven dried at 70°C for 24
125 h (Alaei and Amiri Chayjan, 2015). Next, the dried pomace was ground, and the powder was
126 sifted with mesh #40. The prepared powder was kept at -20°C for the following experiments.

127

128 Chemical properties

129 Some of the chemical properties were measured by converting the fresh orange into a tablet.
130 For this purpose, total phenol content (*TPC*), antioxidant capacity (*AC*), and ascorbic acid (*AA*)
131 were measured for fresh orange, dried orange, and orange powder.

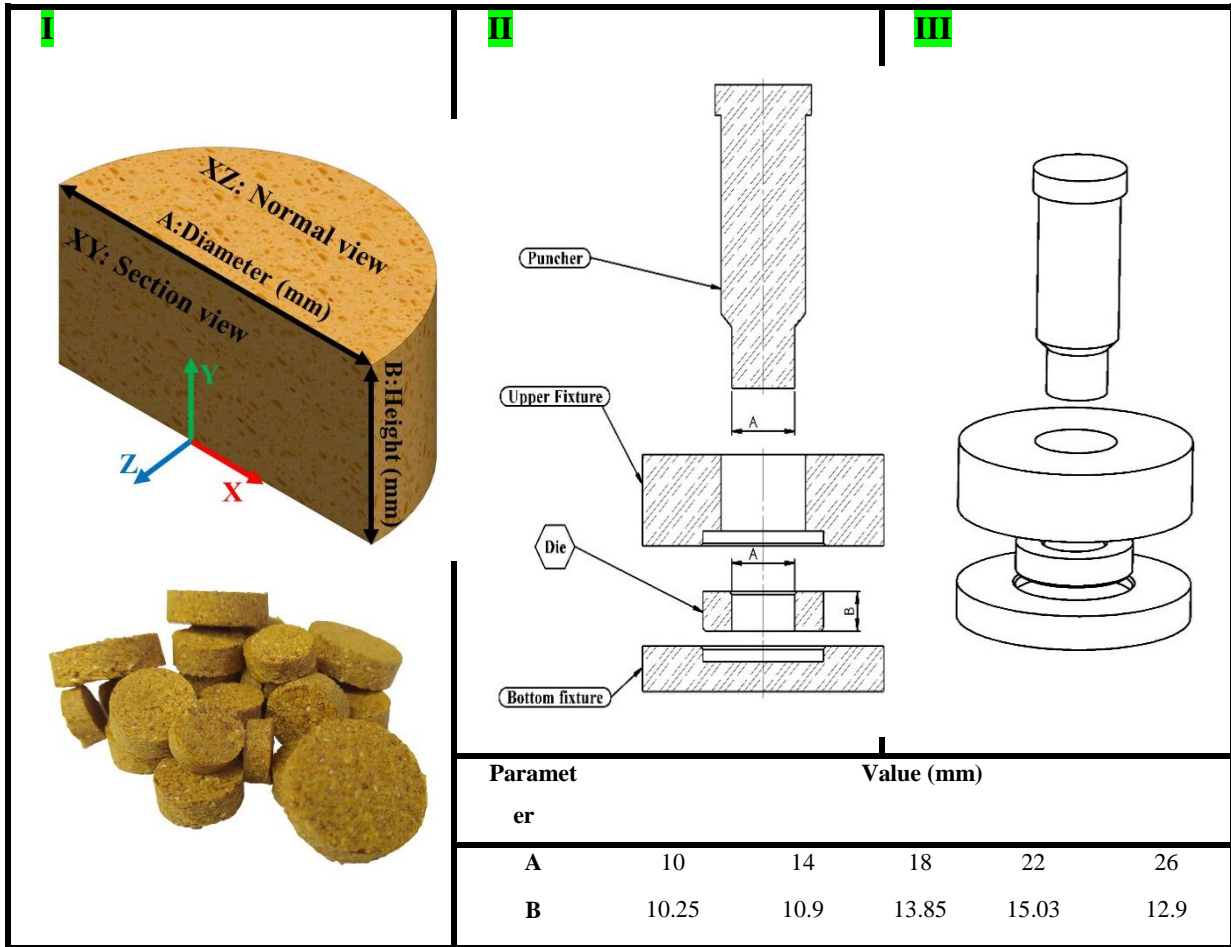
132 The Folin-Ciocalteu method was used to determine the *TPC* (Alwazeer and Ors, 2019;
133 Singleton and Rossi, 1965). *AC* was measured by radical scavenging activity with the 1,1
134 diphenyl-2 picrylhydrazyl (*DPPH*) (Değirmencioglu *et al.*, 2016; Brand-Williams *et al.*, 1995),
135 and titration method by 2,6-dichlorophenol indophenol was used to evaluate the *AA* (Lu *et al.*,
136 2018).

137

138 Tableting process

139 The prepared powder was compressed into a closed-end die by a hydraulic press (Ghasemi
140 and Chayjan, 2018), and the orange powder formed into a flat round tablet (Yohannes and
141 Abebe, 2021). Figure 1B shows the schematic diagram of the tablet dies. Based on the die
142 diameter (*DD*), it was classified into different ranges of 10 to 26 mm. A hydraulic press

143 equipped with pressure control, flow control, and jack position control systems pushes the
 144 puncher. Then, the material was compressed into the die, formed as the tablet (Figure 1.I), and
 145 the tablet was extruded. For studying the effect of **second compression** on mechanical
 146 characteristics, **the second compression was applied when the compressive force was relaxed**
 147 **around a specific value (Relaxation force or *RF*).** for example, when the compressive force
 148 **relaxed to 1, 2, 3 or 4 kN, compressive force was applied again and 0 relaxation force means**
 149 **second compression was not applied.**



150 **Figure 1.** Section I: Orange tablet perspective, Section II: and Section III: Schematic view of
 151 **hydraulic press and tablet die.**
 152

153 The recompression effect on the mechanical properties of tablets was studied after relaxing
 154 the compression force (*CF*) to 0, 1, 2, 3, or 4 kN. Each sample was compressed in triplicate.

155 **Unit density**

157 The unit density of every tablet was determined by measuring the volume and mass of the
 158 tablet. The density was calculated as Eq. (1):

$$\rho = \frac{M}{V} = \frac{M}{A \times H} = \frac{4M}{\pi D^2 H} \quad (1)$$

159 Where ρ , M , V , D , and H are unit density (g mm^{-3}), tablet mass (g), tablet volume (mm^3),
160 tablet diameter (mm), and tablet height (mm), respectively.

161 The mass of every tablet was measured by an electronic balance with 0.001g precision (AND,
162 Japan). Also, the volume of each tablet was calculated as Eq. (1), where a digital caliper was
163 used to measure the diameter (D) and height of the tablets.

164

165 **Food texture**

166 The texture properties of tablets were measured by a universal test machine (HAK S250-B1,
167 Iran). For this purpose, two types of tests were conducted: 1) the puncher test to determine the
168 penetration resistance and 2) the pressure test to determine the break force.

169 **Puncture test**

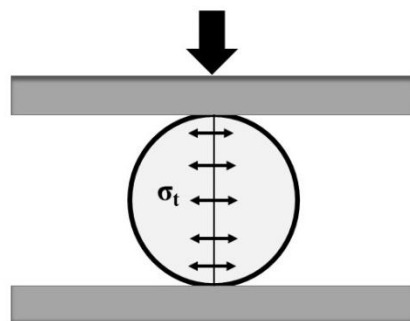
170 This test was conducted using a 5 mm diameter probe that penetrated the orange tablet at 10
171 mm/min speed. In this test, the maximum force was recorded as penetration resistance (known
172 as firmness of texture). The treatments were performed in triplicate (Gholami *et al.*, 2020).

173 **Uniaxial tensile strength (UTS)**

174 The UTS test was conducted by a diametric compression test method (Sun *et al.*, 2020;
175 Mitchell *et al.*, 2017). For this purpose, tablets were placed between two solid plates, one
176 moving down at a speed of 0.2 mm s^{-1} constant. In this condition, the tablet's tensile strength
177 acts outward along the vertical axis (Figure 2). The force with which the tablet breaks along its
178 central axis is known as the maximum force for the break (F_{max}). Consequently, the tablet' UTS
179 was calculated with the following equation:

$$\sigma_t = \frac{2F_{\text{max}}}{\pi DH} \quad (2)$$

180 where σ_t and F_{max} are tablet tensile strength (MPa) and break force (N), respectively. Also,
181 F_{max} is recorded by the food texture machine.



182

183 **Figure 2.** Schematic illustration of force acting on a cylindrical tablet during the diametral
184 compression test.

185 **Stability in water**

186 This test was carried out based on a new method on the real usage of tablets. In this method,
187 1g of the orange tablet was put in 0.1 L of distilled water, and the beaker was stirred at a rate
188 of 90 cycles/min. The time taken to collapse an orange tablet into a beaker containing distilled
189 water was recorded (Sun *et al.*, 2020). Finally, stability in water for an orange tablet was
190 presented as gram second per deciliter (g.s dL⁻¹) (1 deciliter = 100 mL).

191
192 **Tablet formation threshold**

193 The tablet formation threshold was determined from the stress-strain curve analysis of the
194 tablet formation. For this purpose, the point of maximum curvature was analyzed based on an
195 algorithm developed numerically by Dawidowski and Koolen (1994). The line slope (m_q) at
196 each point of the curve is expressed as Eq. (3):

$$m_q = \frac{\varepsilon_{j+1} - \varepsilon_j}{\sigma_{j+1} - \sigma_j} \quad (3)$$

197 where ε_{j+1} and ε_j are strain (mm mm⁻¹) of two consecutive points and σ_{j+1} and σ_j are
198 corresponding stress (kPa). The difference between the slop of every pair of consecutive points
199 was equal to K (Eq. 4):

$$K = m_{q+1} - m_q \quad (4)$$

200 Curvature was maximum when K was maximum (Naderi-Boldaji *et al.*, 2018).

201
202 **Compaction model**

203 The Kawakita and Lüdde model was used to describe the compressibility behavior of orange
204 powder (Roslan *et al.*, 2021). The constants (rational number) a and b were calculated by linear
205 fitting on the P/C versus P graph.

$$\frac{P}{C} = \frac{1}{ab} + \frac{P}{a} \quad (5)$$

$$C = \frac{V_0 - V}{V_0} \quad (6)$$

206 where P is pressure (Pa) and C is the degree of volume reduction that depends on the density.
207 Also, V_0 and V are the initial volume (mm³) and volume of the powder bed under pressure or
208 tablet volume (mm³).

209
210 **Optimization of tableting**

211 In this study, CCD was used to evaluate the effect of three independent variables, CF (kN),
212 DD (mm), and RF (kN), on four responses of density (g mm⁻³), penetration resistance (N), break

213 force (N), and stability in water (g.s dl⁻¹). To this end, 34 experiments were implemented based
 214 on CCD (Table 2). Finally, the results were analyzed by Design-Expert, and multiple responses
 215 were optimized (Table 1).

216 **Table 1.** Central composite design (CCD) matrix and coefficient of importance to optimize
 217 the responses.

Variable	Name	Level codes					Goal of optimization	Importance in optimization
		-2	-1	0	+ 1	+ 2		
Independent	Compression force (kN)	7	9	1 1	13	15	In range	-
	Die diameter (mm)	10	1 4	1 8	22	26	In range	-
	Relaxation force (kN)	0	1	2	3	4	In range	-
Response	Density (g mm ⁻³)	-	-	-	-	-	Maximum	***
	Penetration resistance (N)	-	-	-	-	-	Minimum	*
	Tensile strength (MPa)	-	-	-	-	-	Maximum	*
	Stability in water (g s dl ⁻¹)	-	-	-	-	-	Minimum	*****

218

219 **Table 2.** Central composite design matrix of independent factor for orange tablet optimization.

Run	Compression force (kN)	Die diameter (mm)	Relaxation force (kN)	Reptation
1	7	18	2	2
2	9	14	1	2
3	9	14	3	2
4	9	22	1	2
5	9	22	3	2
6	11	10	2	2
7	11	18	0	2
8	11	18	2	6
9	11	18	4	2
10	11	26	2	2
11	13	14	1	2
12	13	14	3	2
13	13	22	1	2
14	13	22	3	2
15	15	18	2	2

220

221 Figure 3 presents the flowchart of the experimental design in this research. According to
 222 Figure 3, there were four different stages in this study: The powder preparation, Chemical
 223 evaluation, the Tableting process, and the Optimization step for making orange pomace tablets
 224 from fresh orange.

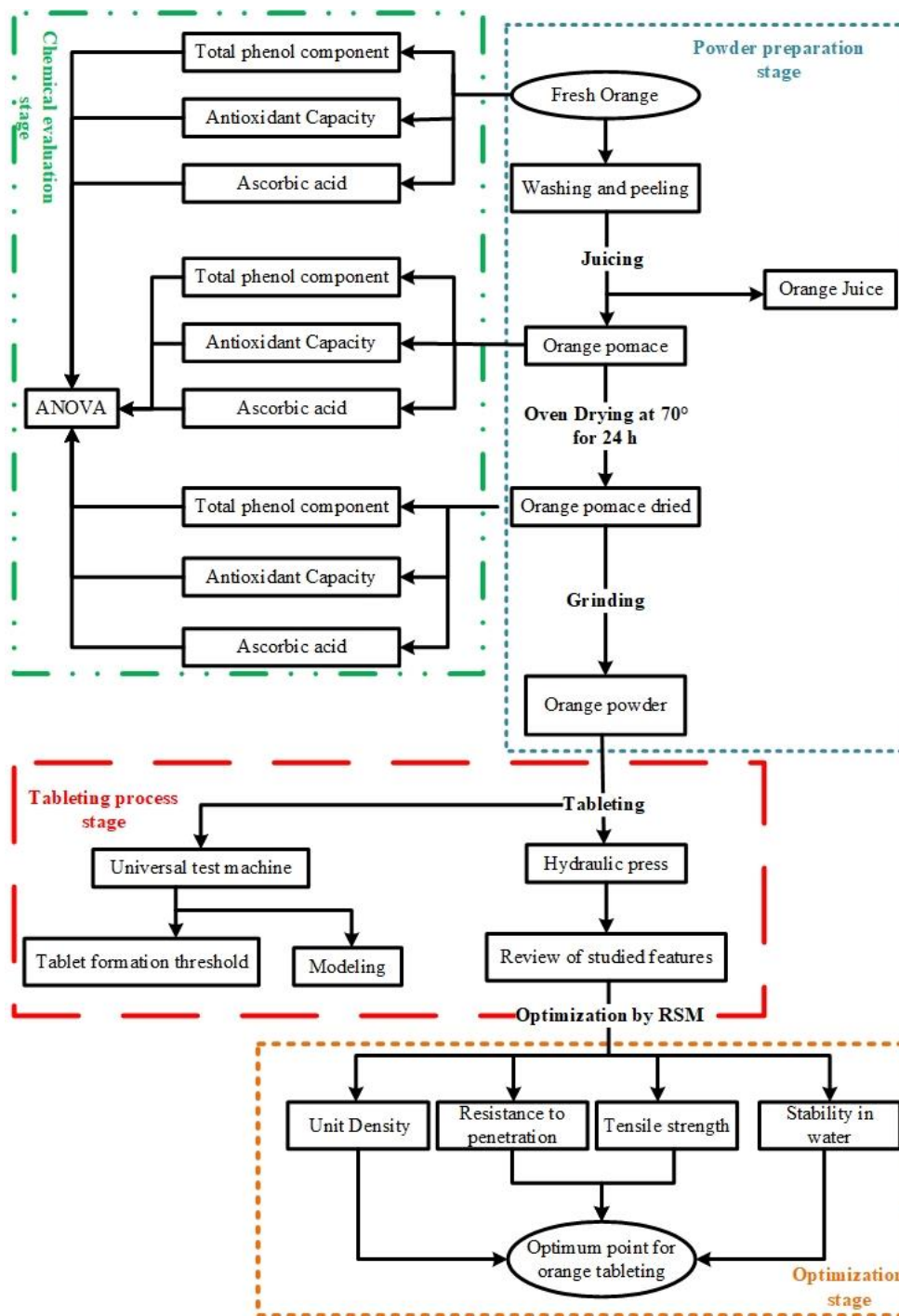


Figure 3. Flowchart of experiments and optimizing the process of tablet making from orange pomace.

RESULTS AND DISCUSSION

Table 3 presents ANOVA for the effect of *DD* on the minimum tension of deformation changes and the effect of the tableting process stage on the *AC*, *TPC*, and *AA* changes as some of the active ingredients indicate. According to Table 3, the effect of *DD* on the minimum tension of deformation changes was insignificant. Figure 4 shows the maximum curvature for making a tablet in 26 mm die and presents a regression between *DD* and tension with $R^2 = 0.93$.

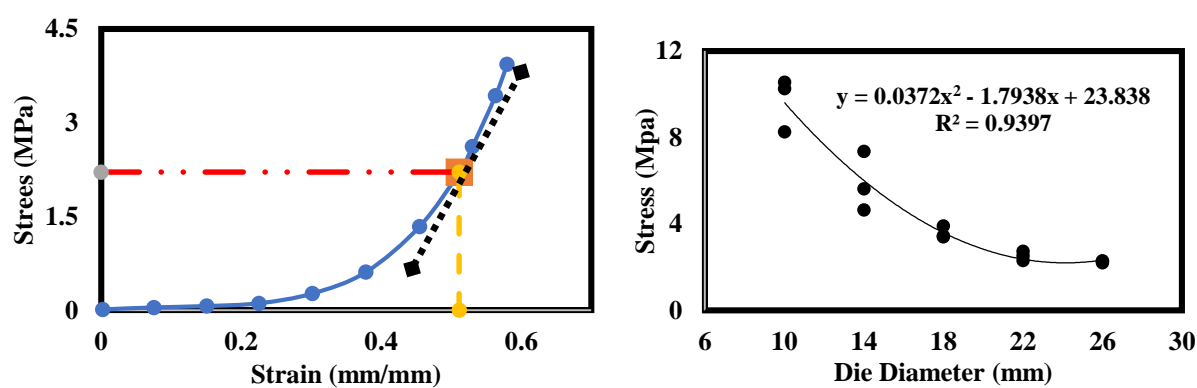
235 A insignificant change of minimum tension of deformation under the effect of *DD* changes
 236 means that the formation threshold of the tablet is dependent on the properties of the powder
 237 and independent of external factors. However, the results proved that some mechanical
 238 properties were affected by external factors.

239
 240 **Table 3.** Analysis of variance (ANOVA) results of the diameter dies and orange tableting
 241 process stage.

Source of Value	SS σ	SS AC	SS TPC	SS AA
Die diameter	225.4354 ^{ns}	-	-	-
Process stage	-	1102.4172**	3158.7785*	190.9783**

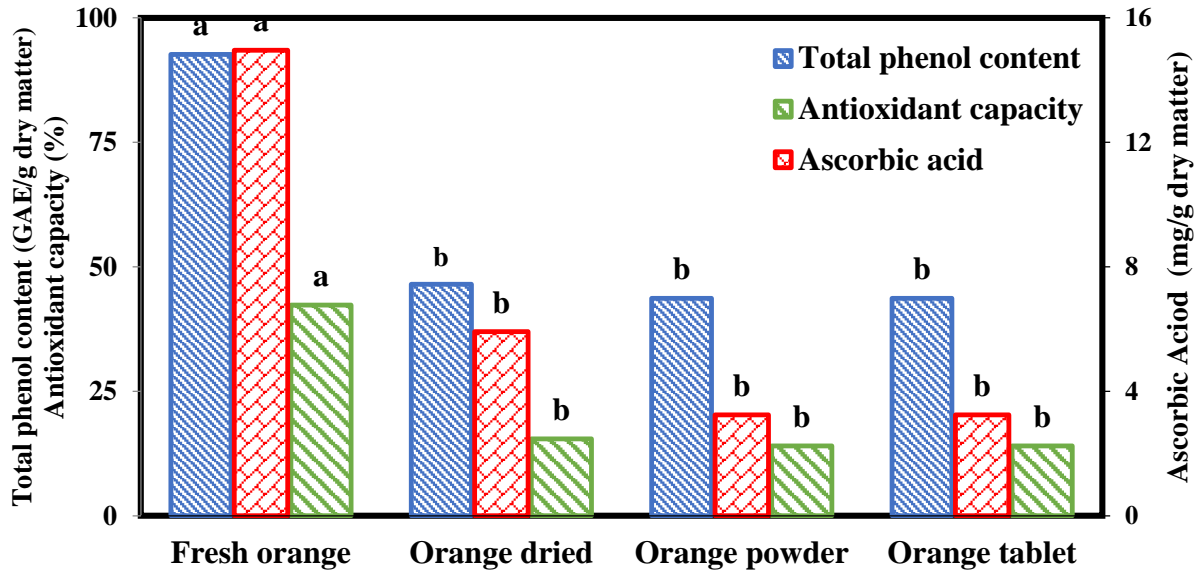
SS: Sum of Square, AC: Antioxidant Capacity, TPC: Total Phenol Content, AA: Ascorbic Acid, and σ : tension

242



243 **Figure 1.** The mathematical method for estimating the pre-compression stress from the
 244 simulated stress-strain curve of confined compression test for 26 mm die.
 245

246 Some of the active ingredients of orange material, from fresh to powder, were analyzed to
 247 determine the qualities of the raw materials used in this study. The results show a significant
 248 decrease in the active ingredients during the drying of the fresh orange. However, the change
 249 in the value of the active ingredients was insignificant when the dried orange was grind and
 250 powdered (Table 3). However, Hu *et al.* (2012) suggested that exposing some polyphenols to
 251 oxygen and heat might lead to degradation during grinding. Elsewhere, it was reported that ball
 252 milling did not affect the major structure of phenolics significantly (Ramachandraiah and Chin,
 253 2016). In contrast, some papers proved that sometimes the milling method had a significant
 254 effect on some of the active ingredients (Liu *et al.*, 2018); however, the minimum nutritional
 255 value of the orange tablets in this study was considered like the nutritional value of the orange
 256 powder. The explanation is that tableting was done with dried and homogeneous powder in this
 257 study. Figure 5 illustrates the values of active ingredients and Duncan's multiple range tests.



258

259 **Figure 2.** Duncan multiple ranges for antioxidant capacity, total phenol content, and ascorbic acid for orange during tablet processing (the same letters are insignificant).
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262 **Compaction model**

263 Table 4 provides the result of the Kawakita and Lüdde model for orange tablets. This model
 264 explains pressure-volume profile, it is often used to evaluate compression specifications. This
 265 model is based on the measurement of initial volume and deviations from this model are usually
 266 due to fluctuations in the measured values of V_0 . This model generally valid for low pressures
 267 and high porosities (Roslan *et al.*, 2021). The constant “a” represents the initial packing,
 268 compressing, and porosity related to particle size and consequently affected by the degree of
 269 grinding and sieving, and “b” denotes the resistance to compaction of the material. In other
 270 words, the higher constant “b”, the more volume reduction (Roslan *et al.*, 2021). Therefore, $1/b$
 271 was related to the cohesive forces of the powder particles. Based on Eq. (5), the $1/b$ parameter
 272 is directly related to the “a” parameter and represents the “a” degree of compression achieved
 273 by the applied pressure. These results were proven by another finding by Zea *et al.* (2013).
 274 According to Saifullah *et al.* (2016a), the constant ‘a’ was similar to the results of the Kawakita
 275 and Lüdde model for different fruits powder (Pitaya, Pineapple, Guava, and Mango), but the
 276 “b” constant was different because the tablets were made from orange pomace, and orange
 277 pomace has less sugar than the orange slice.

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Table 4. Kawakita and Lüdde model constants for the orange tablet.

Sample	<i>a</i>	<i>l/b</i> (kPa)	R ²
Orange tablet	0.521	0.6767	0.995

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283 **Analysis of variance**

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The ANOVA results for tableting from the orange powder are presented in Table 5. All the models suggested for responses were significant at 0.01, and their lack of fit (LoF) values were insignificant. Based on the experimental data, the density of the tablets was between $8.81 \times 10^{-4} \text{ g mm}^{-3}$ (for 11 kN of *CF*, 26 mm of *DD*, and 2 kN of *RF*) and $1.13 \times 10^{-3} \text{ g mm}^{-3}$ (for 11 kN of *CF*, 10 mm of *DD*, and 2 kN of *RF*). Penetration resistance of orange tablets was between 20.45 N (for 11 kN of *CF*, 26 mm of *DD*, and 2 kN of *RF*) and 128.95 N (for 11 kN of *CF*, 10 mm of *DD*, and 2 kN of *RF*), respectively. Minimum and maximum tensile strengths were 7.61×10^{-2} kPa (at 13 kN of *CF*, 14 mm of *DD*, 1 kN of *RF*) and 7.88×10^{-3} kPa (at 11 kN of *CF*, 26 mm of *DD*, and 2 kN of *RF*), respectively. The stability-in-water range was between 39 g.s dl⁻¹ (at 11 kN of *CF*, 26 mm of *DD*, and 2 kN of *RF*) and 176 g.s dl⁻¹ (at 15 kN of *CF*, 18 mm of *DD*, and 2 kN of *RF*), respectively. All the tablets made were complete and perfect in terms of appearance and kept their properties until the next tests.

Table 5. Estimated coefficients in terms of coded factors of the fitted second-order polynomial regression.

Source of Variations	Density	Penetration resistance	Tensile strength	Stability in water
Model (Sum of Squares)	$8.989 \times 10^{9**}$	432.24**	4.59**	0.0024**
Transformation	-1.88	0.86	0.37	-0.32
<i>CF</i>	$1.03 \times 10^9*$	176.65 ^{ns}	3.74**	0.0069**
<i>DD</i>	$5.13 \times 10^{10**}$	2829.07**	9.54**	0.0047**
<i>RF</i>	5.46×10^{8ns}	323.13*	1.33**	0.0003 ^{ns}
<i>CF</i> × <i>DD</i>	2.37×10^{8ns}	0.24 ^{ns}		0.0016**
<i>CF</i> × <i>RF</i>	8.09×10^{8ns}	9.88 ^{ns}		0.0002 ^{ns}
<i>DD</i> × <i>RF</i>	1.95×10^{7ns}	0.32 ^{ns}		1.8×10^{-5ns}
<i>CF</i> ²		368.29*		0.0019**
<i>DD</i> ²		66.36 ^{ns}		0.001*
<i>RF</i> ²		19.35 ^{ns}		0.0009*
Residual	1.903×10^8	54.72	0.0484	0.0001
Lack of Fit (Sum of Squares)	1.980×10^{8ns}	103.75 ^{ns}	0.0728 ^{ns}	0.0002 ^{ns}
R ²	0.95	0.80	0.91	0.90
Adeq Precision	27.75	9.20	31.61	17.03

CF: Compression force, *DD*: Die Diameter, *RF*: Relaxation Force

** : Significant at the level of 0.01, * : Significant at the level of 0.05, *ns*: Insignificant.

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The model proposed for tablet density was significant at 0.01 with $R^2 = 0.95$ (Table 4). There is an inverse relationship between die diameter and density based on Eq. (1). Accordingly, *DD* was the most effective factor on the density at the level of 0.01. Tablet density decreased by about 79% as *DD* raised from 10 to 26 mm (Figures. 6a and 6c). On the other hand, as Figure 6 shows, stress and strain increased simultaneously, and more strain led to a decrease in the

303 volume. Therefore, with CF decreasing from 15 to 7 kN, CF was significant at 0.05 and density
304 decreased by about 95%. By applying a high-pressure compression, most particles of orange
305 powder deform plastically or even fracture. Thus, the reduced free space between particles and
306 permanent shape changes of particles led to bulk density changes. At low-pressure levels, bulk
307 density changes mainly occur due to the rearrangements accompanied by the reduction of the
308 free spaces between particles or clusters because the particles are just slightly deformed but not
309 plastic (Cabiscol *et al.*, 2020).

310 The tablet properties are compared based on the relative final density or porosity. For example,
311 a relative density of 0.9 is commonly used to compare the strength of tablets in pharmaceuticals
312 (Sun, 2005). Paul and Sun (2017) reported that porosity decreased with increasing compaction
313 pressure, leading to a higher density (Basim *et al.*, 2019). Hence, in this study, density raised
314 with increasing the CF . However, based on Eq. (1), at constant CF , the pressure decreases by
315 25% by doubling the diameter (Figures. 6a and 6c). This result explains the significant effect
316 of diameter changes at the level of 0.01 and compressing force at 0.05. Zea *et al.* (2013) and
317 Saifullah *et al.* (2016b) reported a similar result for mixed fruit tablets.

318 The orange tablets must have sufficient strength and resistance to breakage and cracking to
319 tolerate the effects of pressure and stress during production, packaging, and transportation
320 (Naji-Tabasi *et al.*, 2021b). The model of penetration resistance was significant at 0.01 with
321 $R^2=0.80$ (Table 3). Effects of DD at the level of 0.01 and RF were significant at 0.05 on the
322 penetration resistance. The DD was the most effective parameter on the penetration resistance
323 changes. In this respect, by reducing DD from 26 to 10 mm and increasing RF from 0 to 4 kN,
324 penetration resistance increased by 3.8 and 1.4 times, respectively. However, the significant
325 effect of the RF on the penetration resistance indicates that the second compression caused the
326 tablet shell to become harder. According to Figures. 6d-6f, penetration resistance increases by
327 increasing the compression factors (e.g., CF increase and DD reduction) or even secondary
328 compression. Our results are consistent with those reported by Basim *et al.* (2019). **Figure 6**
329 **show force-displacement curve of puncture test for the tablets which were made at CF 7, 11**
330 **and 15 kN with DD 18 mm and RF 2.**

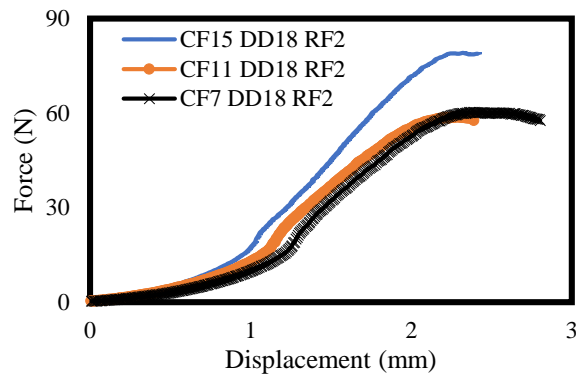


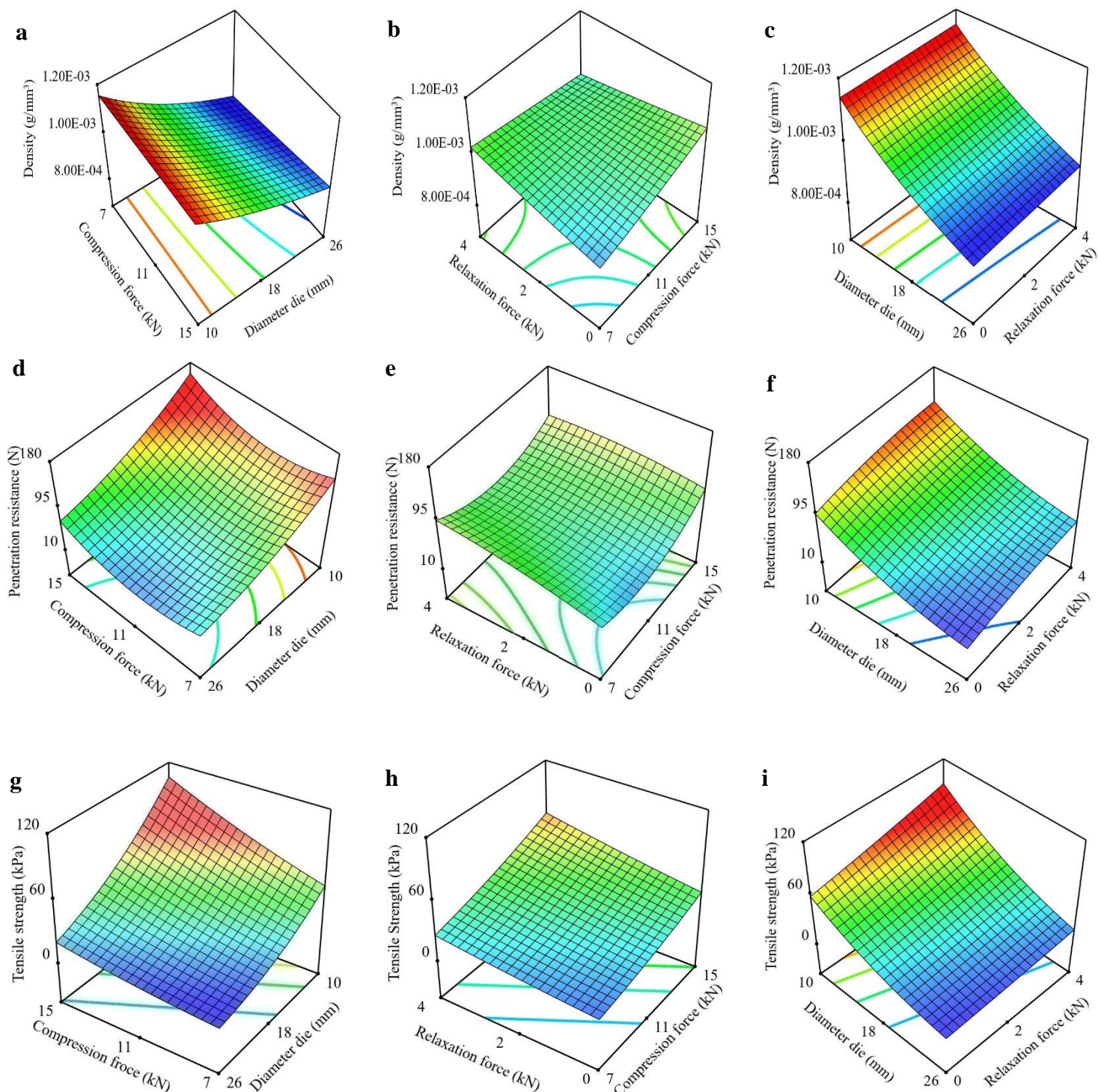
Figure 6. Force-displacement curve of puncture test for orange pomace tablet. *CF*=Compression Force (kN), *DD* = Die Diameter (mm), and *RF*=Relaxation Force (kN).

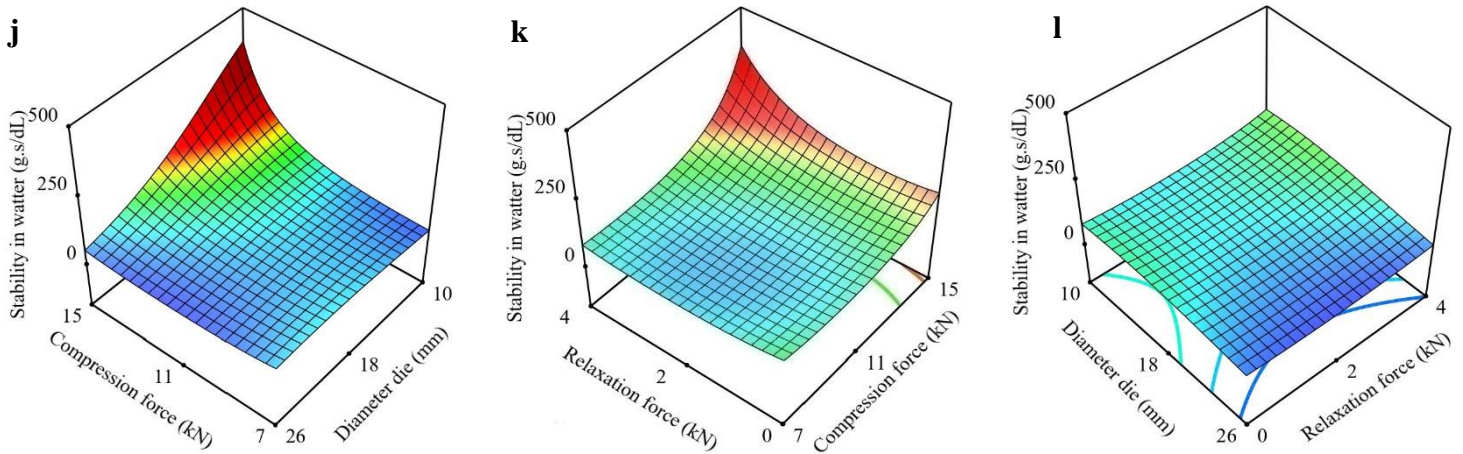
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335 Tabletability is defined as the relationship between the *CF* of the tablet and tensile strength.
336 It is generally reported as a graphical plot of tablet tensile strength versus the *CF* (Khorsheed
337 *et al.*, 2019). The prediction model of tensile strength was significant at 0.01, and the effect of
338 all the independent variables was significant at 0.01 (Table 5). Secondary adhesive contacts,
339 which appear during the deformation of bulk powder, may also affect the resultant tensile
340 strength of the tablets (Horabik *et al.*, 2019). In this regard, Dudhat *et al.* (2017) reported that
341 tensile strength increased with increasing compression pressure. Some researchers have
342 reported similar results (Khorsheed *et al.*, 2019; Shah *et al.*, 2019; Pawar *et al.*, 2016). The
343 present study shows that tensile strength, likewise the penetration resistance, rose with any
344 factor leading to pressure increase, such as the *DD* reduction (by about 8.4 times) or the *CF*
345 increase (by about three times) (Figures. 7g-7i). The noteworthy point about the *RF* is that
346 second compression just had a significant effect on the mechanical properties and its effect on
347 the density and stability in water were insignificant. As a result, second compression increased
348 the tablet's resistance to cracking, breaking, and crushing during production, packaging, and
349 transportation.

350 The quadratic model suggested for stability in water of orange tablets was significant with
351 $R^2=0.90$. The interaction effect of *CF* and *DD* was significant at the level of 0.01. Consequently,
352 the stability in water of orange tablets increased by about 13.8 times from 26 mm of *DD* and 7
353 kN of *CF* to 10 mm of *DD* and 15 kN of *CF* (Table 4 and Figure 7j). The disintegration rate is
354 influenced by the rate of water influx into the tablets, which also depends on the porosity of the
355 tablets (Naji-Tabasi *et al.*, 2021b). Typically, people prefer to use fruit powder tablets in juice
356 form after dissolving them in water or as candy. Hence, fruit powder tablets that dissolve fast
357 will be more acceptable to consumers (Saifullah *et al.*, 2016b). It is of note that the orange
358 pomace is not dissolved and is only disintegrated inside the water. Increasing the *CF* while

359 decreasing the DD led to density raised. Mitchell *et al.* (2017) reported that the dissolution of
 360 tablets depends strongly on the water temperature and material molecular weight. Although the
 361 influence of CF is less clear, tablets compacted at higher pressures take more time to dissolve
 362 properly due to the decreased porosity. Thus, CF affected the type of dissolution regime, and a
 363 stronger bond formed between particles resisting disintegration and dissolution. In other words,
 364 more density means lower porosity, and lower porosity causes more stability in water and, in
 365 some case, lower dissolution.



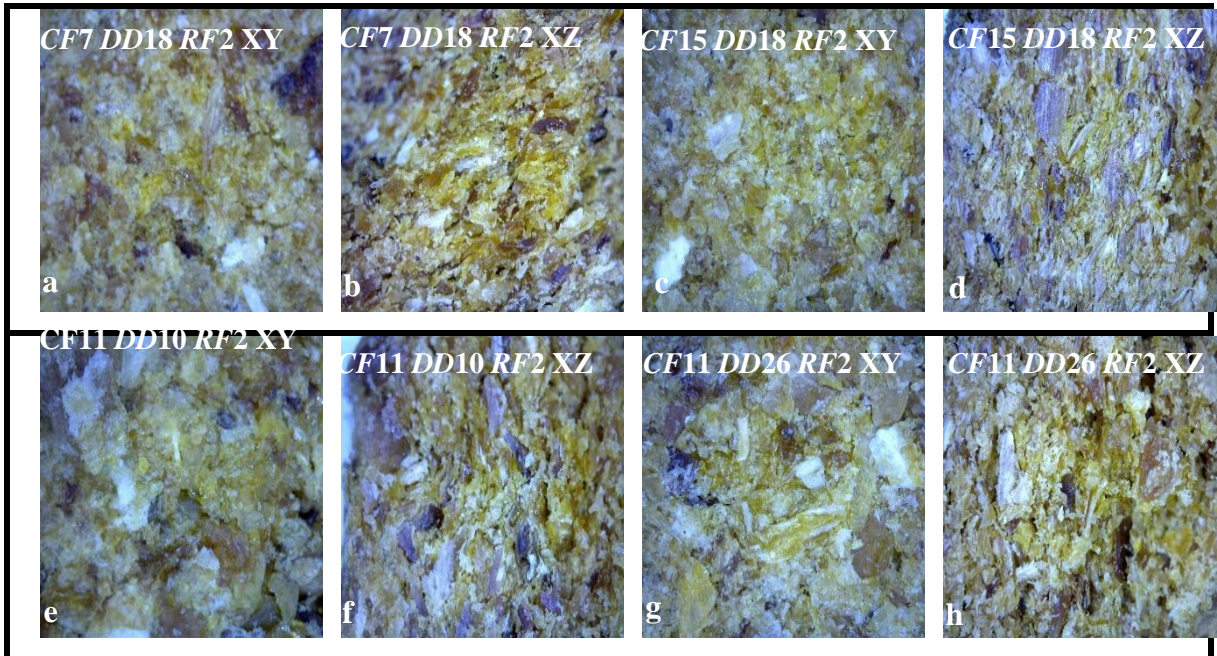


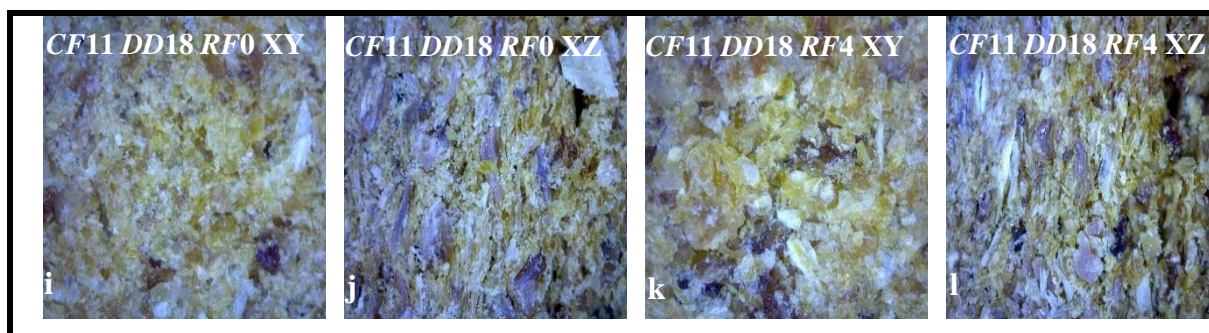
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Figure 7. The effect of compression force, die diameter, and relaxation force on the density, penetration resistance, tensile strength, and stability in the water as the response factor for the optimization of tableting.

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Figure 8 shows a microscopic image taken from the tablets. Figures. 8a to 8d show the effect of *CF* that powder granulation increases with increasing *CF* from 7 to 15 kN, Figures. 8e-8h show the effect of *DD* which tablet compression decreased with increasing die diameter from 10 to 26 mm., and Figures. 8i-8l represents the effect of *RF*, the application of *RF* (second compaction) leads to more compression of the formed layers. Here, pictures ‘XY’ were taken from the normal view, and ‘XZ’ was taken from the section view.





377 **Figure 8.** micro picture from the orange tablets under different conditions. *CF*=Compression
 378 Force (kN), *DD* = Die Diameter (mm), and *RF*=Relaxation Force (kN). ‘XY’ was taken in the
 379 micro picture in the compressed direction. ‘XZ’ was taken as the micro picture perpendicular
 380 to the compact direction.
 381

382 **Optimization of tableting**

383 The optimum point for orange tableting based on the goals of the responses was suggested at
 384 8.6 kN for *CF*, 10 mm for *DD*, and 1.14 kN for *RF*. In this regard, the optimum density,
 385 penetration resistance, tensile strength, and stability value in water were determined to be
 386 $1.13 \times 10^{-3} \text{ g mm}^{-3}$, 111 N, 52.4 kPa, and 51 g.s dl⁻¹, respectively.

387 As mentioned in subsection 2.3, the relationship between density and stability in water was
 388 positive. In other words, higher density led to higher stability in water. Also, stability in the
 389 water took 5-star importance because of customer-friendliness importance (Saifullah *et al.*,
 390 2016b), while density after stability in the water took the second place of importance. However,
 391 based on Table 3, their goals were opposite to each other. Hence, making a balance between
 392 them (i.e., stability in water and density) is very serious. Table 6 shows that the balance was
 393 established at 8.6 kN for *CF* and 10 mm for *DD*. It is noteworthy that the effect of *RF* on the
 394 density and stability in water was insignificant.

395 A high-quality tablet should resist breaking and cracking during production, packaging, and
 396 transportation. These factors depend on the responses of penetration resistance and tensile stress
 397 (Naji-Tabasi *et al.*, 2021b). While the optimization goals of these two responses were the
 398 opposite, the optimum point was suggested at 8.6 kN for *CF*, 10 mm for *DD*, and 1.14 kN for
 399 *RF*. Notably, the effect of *RF* on the penetration resistance and tensile stress was significant.
 400 Table 5 shows that applying the second compaction may lead to a suitable tablet at lower *CF*.

401 **Based on the optimum point prediction, a test (9 kN for compression force, 10 mm for**
 402 **diameter die and 1 kN for relaxation force) was done and $1.15 \times 10^{-3} \text{ g mm}^{-3}$ for density, 110 N**
 403 **for penetration resistance, 54 MPa for tensile strength and 55 g.s dL⁻¹ for stability in water was**
 404 **recoded.**

405 The nutritious value of the tablet, which formed at the optimum point, probably would be 9%
 406 for AC, 28.18 mg GAE $\text{g}_{\text{dry matter}}^{-1}$ for TPC, and 2.09 mg $\text{g}_{\text{dry matter}}^{-1}$ for AA. Also, the tablet
 407 formation threshold would be 9.1 MPa.

408 **Table 6.** Optimum point suggested for tableting from orange pomace.

Independent variable			Responses				Desirability
Compression force (kN)	Die diameter (mm)	Relaxation force (kN)	Density (g mm^{-3})	Penetration resistance (N)	Tensile strength (MPa)	Stability in water (g.s dl^{-1})	
8.6	10	1.14	1.13×10^{-3}	111	52.4	51	71.4

409

410 CONCLUSIONS

411 This study demonstrated the insignificant effect of die diameter (*DD*) on the minimum tension
 412 of deformation changes, suggesting that the formation threshold of the tablet depends on the
 413 powder's properties. Also, the constant 'a' of the Kawakita and Lüdde model was similar to the
 414 results of different fruits powder. The results show significant changes in chemical properties
 415 (i.e., total phenol content, antioxidant capacity, and ascorbic acid) while converting the dried
 416 orange pomace to an orange pomace tablet. In addition, the results show that the *DD* was the
 417 most effective factor in the density at the level of 0.01. Tablet density increased by about 1.26%
 418 and 1.1%, with *DD* reducing from 26 to 10 mm and *CF* increasing from 7 to 15 kN, respectively.
 419 By reducing the *DD* from 26 to 10 mm, penetration resistance increased by 3.8 times, and
 420 tensile strength increased by about 8.4 times. Besides, the significant effect of relaxation force
 421 was only noticeable on mechanical properties because the tablet shell became harder under the
 422 effect of stress relaxation and second compression. The interaction effect of *CF* and *DD* on the
 423 stability in water was significant at 0.01. As a result, the stability in water of orange tablets
 424 increased about 13.8 times from *DD* of 26 mm and *CF* of 7 kN to *DD* of 10 mm and *CF* of 15
 425 kN. The results showed that the optimum point for the tablet making from the orange pomace
 426 with the highest density and tensile strength and the lowest penetration resistance and stability
 427 in water was 8.6 kN for *RF*, 10 mm for *DD*, and 1.14 kN for *RF*. Finally, the result of this study
 428 showed that tableting form orange pomace is a new opportunity to maximum usage from food
 429 sources. For this purpose, study about enriching tablets with additives, appley new tableting
 430 technology to make orange tablet, or even simulate tableting process by finite element method
 431 to find a deeper understanding about this process are suggested to other researchers.

432

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