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Orange Tablet Production Enhancement by Intervention of a Dry Method: A Central Composite Strategy for Experimental Design and Optimization

Hamed Homayounfar[1], and Reza Amiri Chayjan[1*]

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 1.13×10^{-3} 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.

Keywords: Tableting, Compressibility behavior, Tablet formation threshold.

INTRODUCTION

Citrus fruits are among the favorite fruit products regarding their pleasant flavor and nutritional value. The total world citrus production in 2020 was about 158 million tons, more than half of which were produced by oranges (FAO, 2020). About one-third of the annual world orange production is used for orange juice production (Pan *et al.*, 2019). Most of the horticultural output of oranges is used to prepare juice and concentrates, because they are a suitable replacement for different beverages such as coffee, tea, and carbonated soft drinks (Oduntan and Arueya, 2019). The juicing process usually produces a large amount of waste such as peel, pomace, cores, unripe, and damaged fruits (Bozkir *et al.*, 2021; Gomez and Martinez, 2018), leading to a serious environmental issue (Badaoui *et al.*, 2019). The residual solids contain around 50% of the whole fruit weight. Therefore, several thousand tons of pomace are produced as an agricultural by-product. Some post-harvest processing can convert these "waste" materials into high-value products (Pan *et al.*, 2019; Huang and Ma, 2016). One of these processes is making tablets from powders produced from food. In this study, unlike other studies, the powder is converted into tablets by a dry method (Naji-Tabasi *et al.*, 2021a). In addition to the

[1]Department of Biosystems Engineering, Faculty of Agriculture, Bu-Ali Sina University, Hamedan 65178-38695, Islamic Republic of Iran.
* Corresponding author; e-mail: amirireza@basu.ac.ir

fact that tableting by dry method is more applicable to industries, this method is cheaper, faster and easier. On the other hand, tableting is one of the most usual methods for preparation specific amount of food material, because their production and use are easy (Gaikwad and Kshirsagar, 2020).

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

The high moisture content of agricultural by-products lead to short self-life and high storage costs. By-products are perishable and more susceptible to rapid microorganism growth (Masud *et al.*, 2020). Therefore, providing appropriate processing methods for this product is highly important to reduce these costs and increase shelf life (Rashidi *et al.*, 2021). Drying by-product is one of the most widely used methods to process and preserve this agricultural waste (Wang *et al.*, 2021). The by-product in powder form has potential applications as food ingredients or even biomass in food waste management (Rashidi *et al.*, 2021).

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

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

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

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

Most of quality attributes of tablets manufacturing process such as appearance, content uniformity, hardness, thickness, friability, disintegration time and dissolution time are affected by tablet compression process. The weight effect of different parameters on the tableting process is different. Therefore, parameters optimization is necessary to find a balance between the effect of different parameter on the compression process (Jongwuttanaruk and Thavornwat, 2022; Garlapati and Roy, 2017). There are numerous methods to process optimization and Central Composite Design (*CCD*) is one of the most popular ones (Homayounfar *et al.*, 2023; Chen *et al.*, 2022). Some researchers tried to optimize tablet processing by *CCD* (Rashidi *et al.*, 2021; Ghasemi and Chayjan, 2018; Ghasemi *et al.*, 2018).

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

To our knowledge, no research has been conducted on evaluating and optimizing the orange pomace tableting process. Therefore, in this paper, the effect of pressure, diameter die, and the second compression on some of the physical properties of orange tablets (i.e., density, penetration resistance, tensile strength, and stability in water) were investigated and optimized by Central Composite Design (CCD).

MATERIALS AND METHOD

Powder Preparation

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

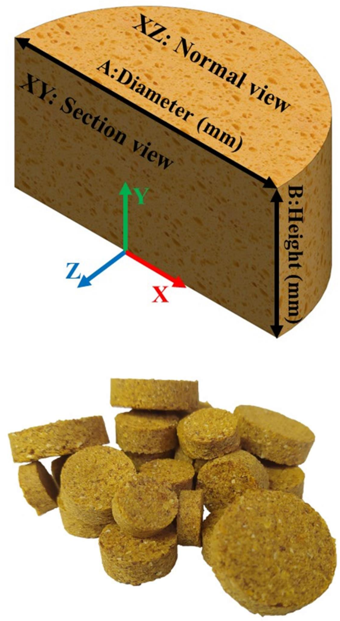
Chemical Properties

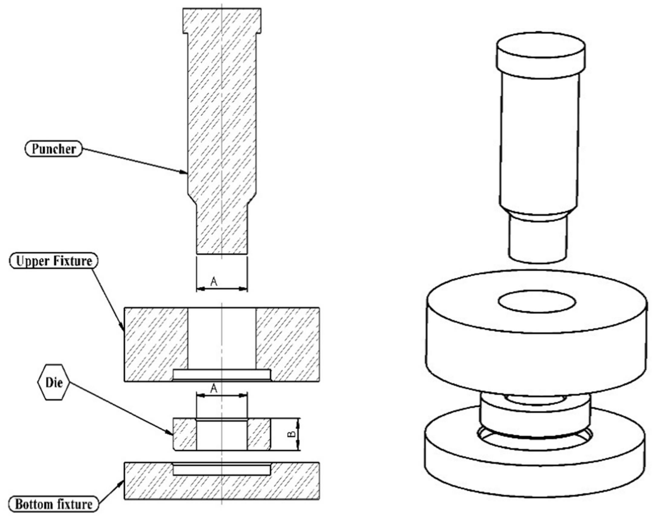
Some of the chemical properties were measured by converting the fresh orange into a tablet. For this purpose, Total Phenol Content (TPC), Antioxidant Capacity (AC), and ascorbic acid (AA) were measured for fresh orange, dried orange, and orange powder.

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

Tableting Process

The prepared powder was compressed into a closed-end die by a hydraulic press (Ghasemi and Chayjan, 2018), and the orange powder formed into a flat round tablet (Yohannes and Abebe, 2021). Figure 1B shows the schematic diagram of the tablet dies. Based on the Die Diameter (DD), it was classified into different ranges of 10 to 26 mm. A hydraulic press equipped with pressure control, flow control, and jack position control systems pushed the puncher. Then, the material was compressed into the die, formed as the tablet (Figure 1-I), and the tablet was extruded. For studying the effect of second compression on mechanical characteristics, the second compression was applied when the compressive force was relaxed around a specific value (Relaxation Force or RF). For example, when the





Parameter	Value (mm)				
A	10	14	18	22	26
B	10.25	10.9	13.85	15.03	12.9

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

compressive force relaxed to 1, 2, 3 or 4 kN, compressive force was applied again and 0 relaxation force means second compression was not applied.

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

Unit Density

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

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

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

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

Food Texture

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

Puncture Test

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

Uniaxial Tensile Strength (UTS)

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

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

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

Stability in Water

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

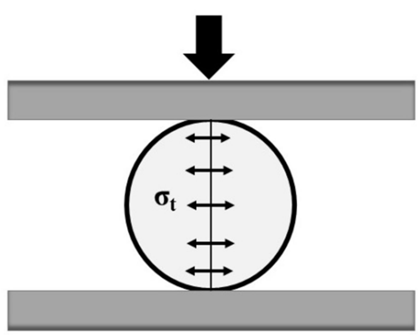


Figure 2. Schematic illustration of force acting on a cylindrical tablet during the diametral compression test.

Tablet Formation Threshold

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

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

Where, ε_{j+1} and ε_j are strain (mm mm^{-1}) of two consecutive points and σ_{j+1} and σ_j are corresponding stress (kPa). The difference between the slop of every pair of consecutive points was equal to K (Equation 4):

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

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

Compaction Model

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

$$\frac{P}{C} = \frac{1}{ab} + \frac{P}{a}$$

$$C = \frac{V_0 - V}{V_0}$$

Where, P is Pressure (Pa) and C is the degree of volume reduction that depends on the density. Also, V_0 and V are the initial Volume (mm^3) and Volume of the powder bed under pressure or tablet volume (mm^3).

Optimization of Tableting

In this study, CCD was used to evaluate the effect of three independent variables, CF (kN), DD (mm), and RF (kN), on four responses of density (g mm^{-3}), penetration resistance (N), break force (N), and stability in water (g s dL^{-1}). To this end, 34 experiments were implemented based on CCD (Tabel 2). Finally, the results were analyzed by Design-Expert, and multiple responses were optimized (Table 1).

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

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. An insignificant change of minimum tension of

Table 1. Central Composite Design (CCD) matrix and coefficient of importance to optimize the responses.

Variable	Name	Level codes					Goal of optimization	Importance in optimization
		-2	-1	0	+1	+2		
Independent	Compression force (kN)	7	9	11	13	15	In range	-
	Die diameter (mm)	10	14	18	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	*****

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

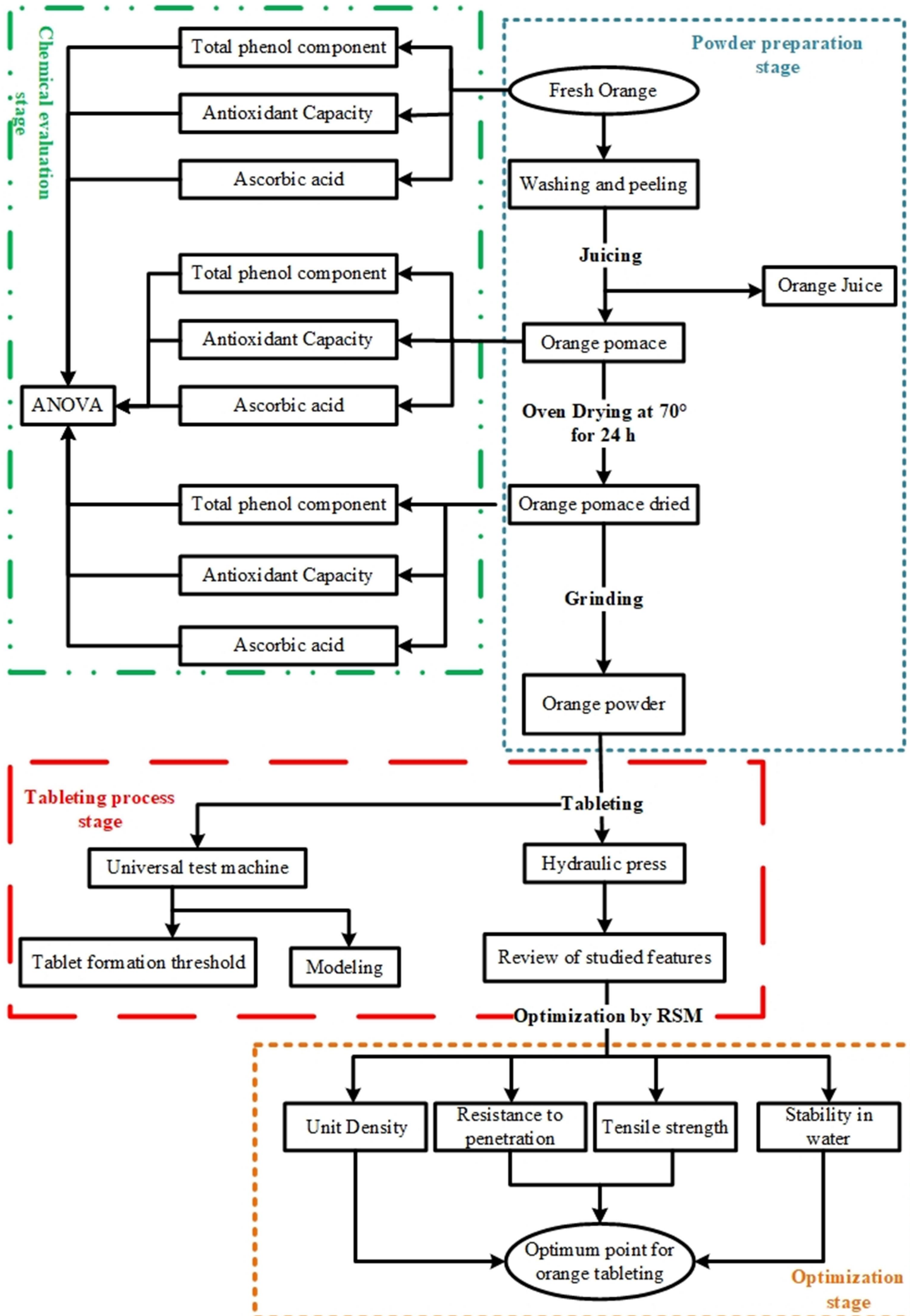


Figure 3. Flowchart of the experiments and optimizing the process of tablet-making from orange pomace.

Table 3. Analysis Of Variance (ANOVA) results of the diameter dies and orange tableting process stage.[a]

Source of Value	SS σ	SS AC	SS TPC	SS AA
Die diameter	225.4354[ns]	-	-	-
Process stage	-	1102.4172**	3158.7785*	190.9783**

[a] SS: Sum of Square, AC: Antioxidant Capacity, TPC: Total Phenol Content, AA: Ascorbic Acid, and σ: Tension.

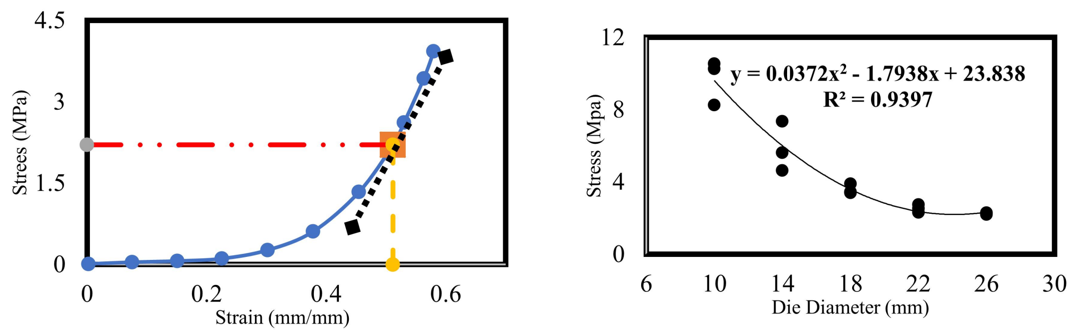


Figure 4. The mathematical method for estimating the pre-compression stress from the simulated stress-strain curve of the confined compression test for 26 mm die.

deformation under the effect of DD changes means that the formation threshold of the tablet is dependent on the properties of the powder and independent of the external factors. However, the results proved that some mechanical properties were affected by the external factors.

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

Compaction Model

Table 4 provides the result of the Kawakita and Lüdde model for orange tablets. This model explains pressure-volume profile, which is often used to evaluate compression specifications. This model is based on the measurement of initial volume and deviations from this model are usually due to fluctuations in the measured values of V_0. This model is generally valid for low pressures and high porosities (Roslan *et al.*, 2021). The constant "a" represents the initial packing, compressing, and porosity related to particle size and, consequently, affected by the degree of grinding and sieving, and "b" denotes the resistance to compaction of the material. In other words, the higher constant "b", the

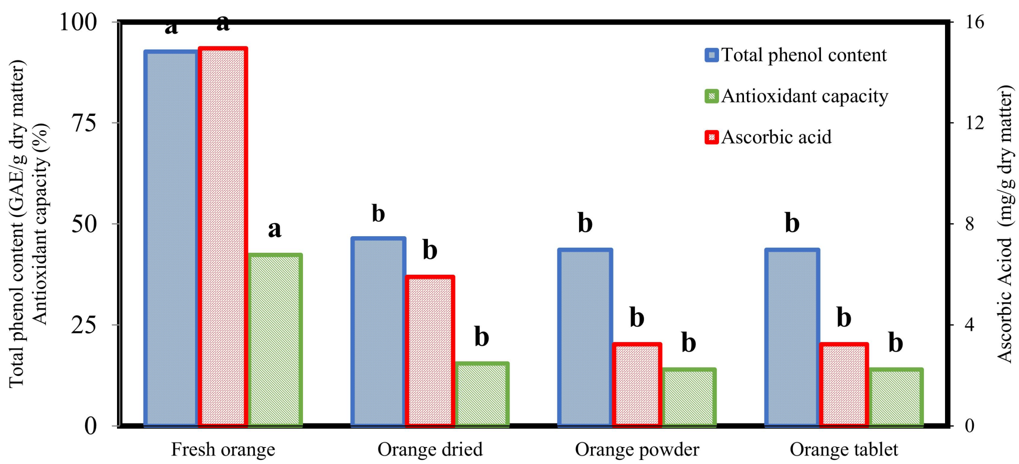


Figure 5. Duncan multiple ranges for antioxidant capacity, total phenol content, and ascorbic acid for orange during tablet processing (the same letters are insignificant).

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

Sample	a	1/b (kPa)	R^2
Orange tablet	0.521	0.6767	0.995

more volume reduction (Roslan *et al.*, 2021). Therefore, 1/b was related to the cohesive forces of the powder particles. Based on Equation (5), the 1/b parameter is directly related to the "a" parameter and represents the "a" degree of compression achieved by the applied pressure. These results were proven by another finding by Zea *et al.* (2013). According to Saifullah *et al.* (2016a), the constant 'a' was similar to the results of the Kawakita and Lüdde model for different fruits powder (Pitaya, Pineapple, Guava, and Mango), but the "b" constant was different because the tablets were made from orange pomace, which has less sugar than the orange slice.

Analysis of Variance

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 8.81×10^{-4} g mm^{-3} (for 11 kN of CF, 26 mm of DD, and 2 kN of RF) and 1.13×10^{-3} 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 CM, 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.

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 Equation (1). Accordingly, DD was the most effective factor on the density at the level of 0.01.

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)	8.989×10^{9}**	432.24**	4.59**	0.0024**
Transformation[a]	-1.88	0.86	0.37	-0.32
CF	1.03×10^{9}*	176.65^{ns}	3.74**	0.0069**
DD	5.13×10^{10}**	2829.07**	9.54**	0.0047**
RF	5.46×10^{8ns}	323.13*	1.33**	0.0003^{ns}
CF×DD	2.37×10^{8ns}	0.24^{ns}		0.0016 **
CF×RF	8.09×10^{8ns}	9.88^{ns}		0.0002^{ns}
DD× RF	1.95×10^{7ns}	0.32^{ns}		1.8×10^{-5ns}
CF^2		368.29*		0.0019**
DD^2		66.36^{ns}		0.001*
RF^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

[a] 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.

Tablet density decreased by about 79% as DD raised from 10 to 26 mm (Figures 6-a and -c). On the other hand, as Figure 6 shows, stress and strain increased simultaneously, and more strain led to a decrease in the volume. Therefore, with CF decreasing from 15 to 7 kN, CF was significant at 0.05 and density decreased by about 95%. By applying a high-pressure compression, most particles of the orange powder deform plastically or even fracture. Thus, the reduced free space between particles and permanent shape changes of particles led to bulk density changes. At low-pressure levels, bulk density changes mainly occur due to the rearrangements accompanied by the reduction of the free spaces between particles or clusters, because the particles are just slightly deformed but not plastic (Cabiscol *et al*., 2020).

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

The orange tablets must have sufficient strength and resistance to breakage and cracking to tolerate the effects of pressure and stress during production, packaging, and transportation (Naji-Tabasi *et al.*, 2021b).

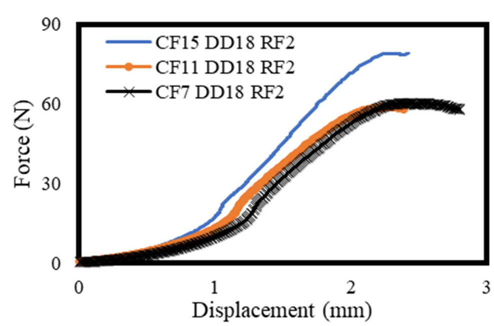


Figure 6. Force-displacement cure of punture test for orange pomace tablet. CF= Compression Force (kN), DD= Die Diameter (mm), and RF= Relaxation Force (kN).

The model of penetration resistance was significant at 0.01 with R^2= 0.80 (Table 3). Effects of DD at the level of 0.01 and RF were significant at 0.05 on the penetration resistance. The DD was the most effective parameter on the penetration resistance changes. In this respect, by reducing DD from 26 to 10 mm and increasing RF from 0 to 4 kN, penetration resistance increased by 3.8 and 1.4 times, respectively. However, the significant effect of the RF on the penetration resistance indicates that the second compression caused the tablet shell to become harder. According to Figures 6d-6f, penetration resistance increases by increasing the compression factors (e.g., CF increase and DD reduction) or even secondary compression. Our results are consistent with those reported by Basim et al. (2019). Figure 6 shows the force-displacement curve of the puncture test for the tablets that were made at CF 7, 11 and 15 kN with DD 18 mm and RF 2.

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

The quadratic model suggested for stability in water of orange tablets was significant with R^2= 0.90. The interaction effect of CF and DD was significant at the level of 0.01. Consequently, the stability in water of orange tablets increased by about 13.8 times from 26 mm of DD and 7 kN of CF to 10 mm of DD and 15 kN of CF (Table 4 and Figure 7j). The disintegration rate is influenced by the rate of water influx into the tablets, which also depends on the porosity of the tablets (Naji-Tabasi *et al.*, 2021b). Typically, people prefer to use fruit powder tablets in juice form after dissolving them in water or as candy. Hence, fruit powder tablets that dissolve fast will be more acceptable to consumers (Saifullah *et al.*, 2016b). It is of note that the orange pomace is not dissolved and is only disintegrated inside the water. Increasing the CF while decreasing the DD led to higher density. Mitchell *et al.* (2017) reported that the dissolution of tablets depends strongly on the water temperature and the material molecular weight. Although the influence of CF is less clear, tablets compacted at higher pressures take more time to dissolve properly due to the decreased porosity. Thus, CF affected the type of dissolution regime, and a stronger bond formed between particles resisting disintegration and dissolution. In other words, more density means lower porosity, and lower porosity causes more stability in water and, in some case, lower dissolution.

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, Figure 8 (e-h) shows the effect of DD, when tablet compression decreased with increasing die diameter from 10 to 26 mm, and Figure 8 (i-l) 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

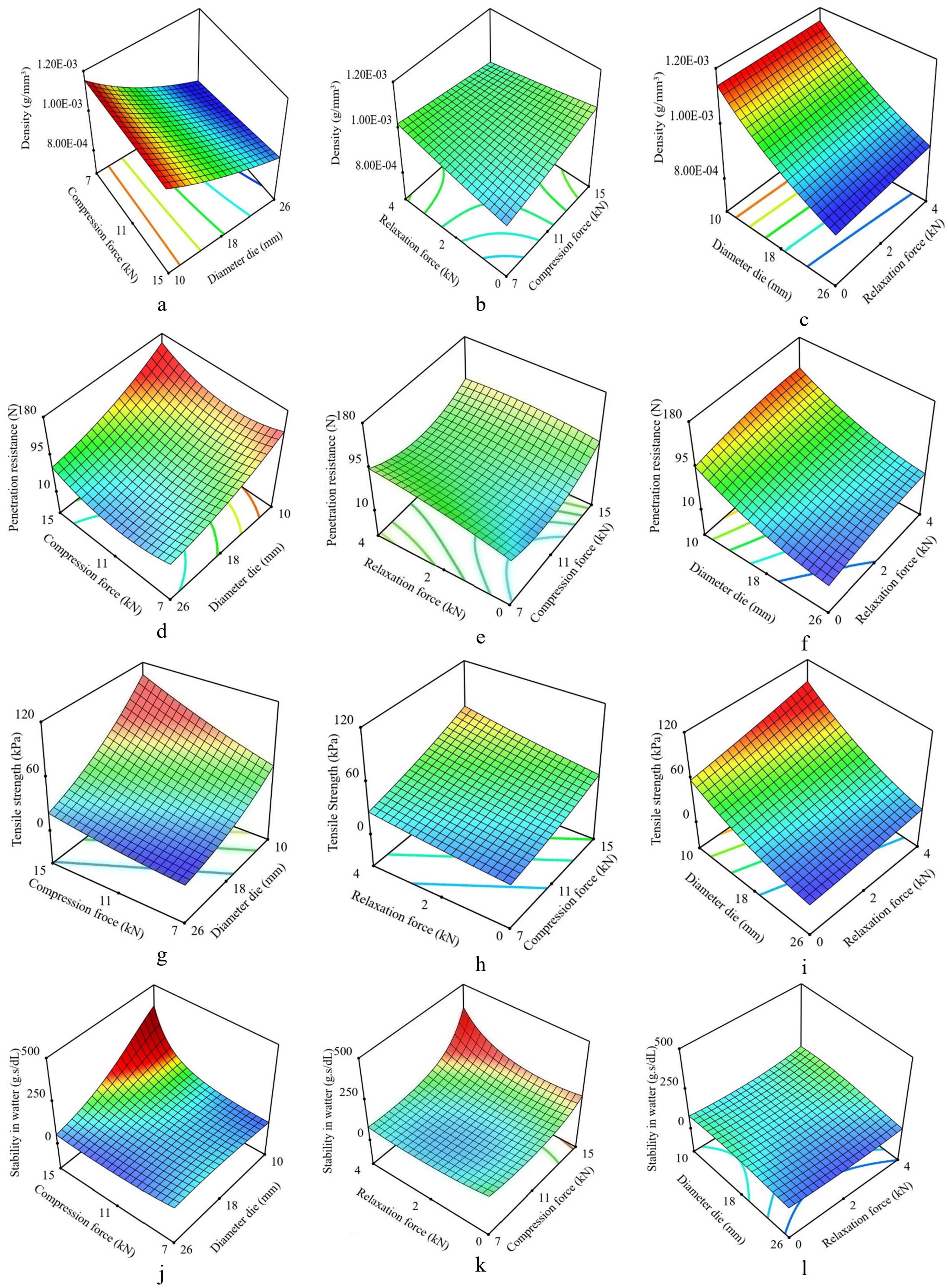


Figure 7. The effect of compression force, die diameter, and relaxation force on the density, penetration resistance, tensile strength, and stability in the water as the response factor for the optimization of tableting.

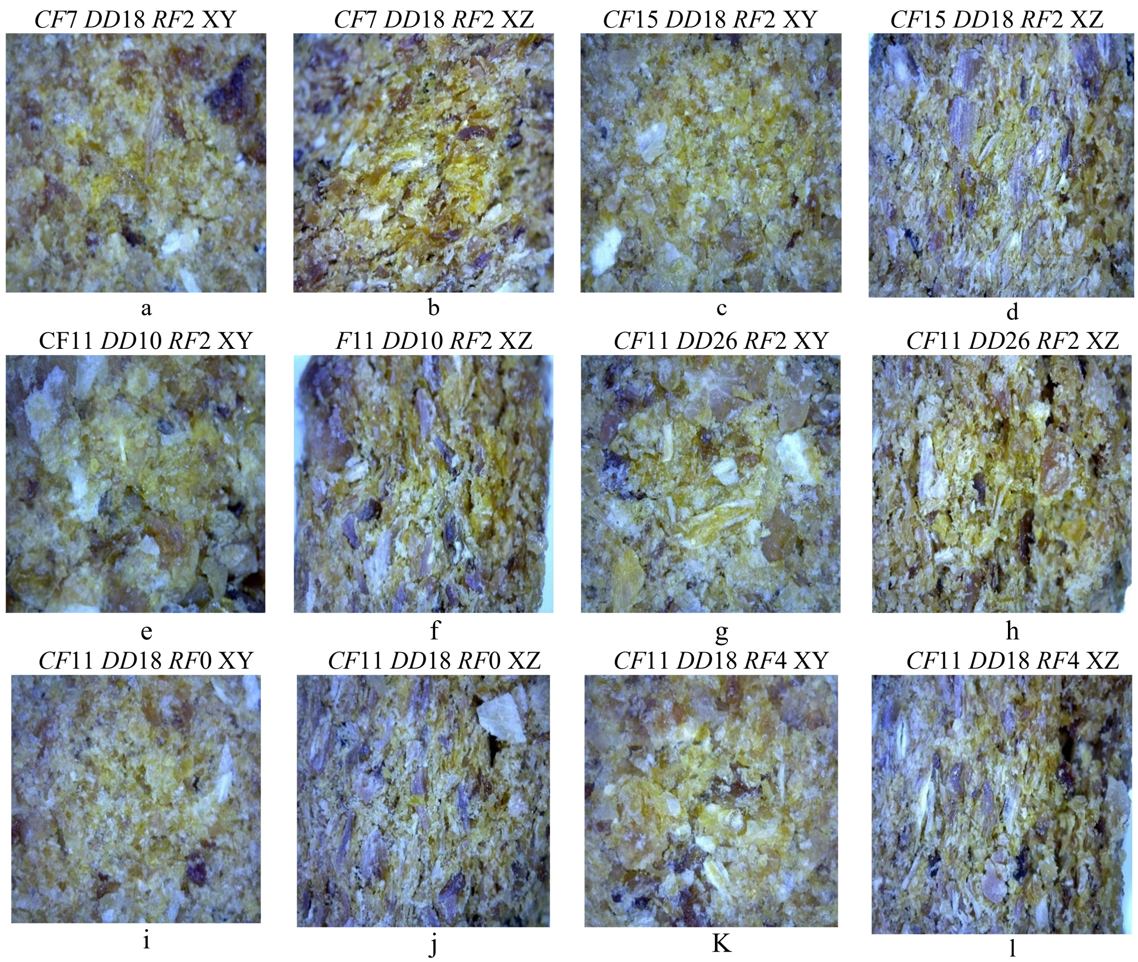


Figure 8. Micro picture from the orange tablets under different conditions. CF= Compression Force (kN), DD= Die Diameter (mm), and RF= Relaxation Force (kN). 'XY' was taken in the micro picture in the compressed direction. 'XZ' was taken as the micro picture perpendicular to the compact direction.

the normal view, and 'XZ' was taken from the section view.

Optimization of Tableting

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

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

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

Independent variable			Responses				
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})	Desirability
8.6	10	1.14	1.13×10^{-3}	111	52.4	51	71.4

effect of *RF* on the density and stability in water was insignificant.

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

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

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

CONCLUSIONS

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

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)

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افزایش تولید قرص پرتقال با مداخله روش خشک: یک استراتژی ترکیبی مرکزی برای طراحی و بهینه‌سازی تجربی

حامد همایونفر، و رضا امیری چایجان

چکیده

مطالعه حاضر با هدف تولید قرص تفاله پرتقال به عنوان یک افزودنی با ارزش غذایی انجام شد. پودر تفاله پرتقال به روش خشک کردن تحت تأثیر نیروی فشاری، قطر قالب و تغییر نیروی رهایش به قرص تبدیل شد. برخی از خواص فیزیکوشیمیایی (به عنوان مثال، چگالی، مقاومت در برابر نفوذ، استحکام کششی، پایداری در آب، محتوای فنل کل، ظرفیت آنتی اکسیدانی و اسید اسکوربیک) مورد بررسی قرار گرفت. فرآیند قرص سازی با طراحی مرکب مرکزی بهینه شد. مدل Kawakita و Lü dde با $R2 = 0.995$ برای توصیف رفتار تراکم پذیری پودر تفاله پرتقال استفاده شد. اثر قالب قطر بر حداقل کشش تغییرات تغییر شکل ناچیز بود. نتایج نشان داد که تغییرات در ارزش مواد موثره زمانی که پرتقال خشک شده، پودر شده و به قرص تبدیل می‌شود، ناچیز است. همچنین تأثیر تغییرات قطری بر روی همه پاسخ‌ها در ۰/۰۱ معنی‌دار بود، در حالی که تأثیر نیروی فشاری فقط برای مقاومت در برابر نفوذ ناچیز بود. اثر نیروی رهایش بر خواص مکانیکی (در سطح ۰.۵ برای مقاومت در برابر نفوذ و ۰.۰۱ برای مقاومت کششی) و بر چگالی و پایداری در آب ناچیز بود. در نهایت، نقطه بهینه برای قرص سازی از تفاله نارنجی در نیروی فشرده سازی ۸.۶ کیلو نیوتن، قالب قطری ۱۰ میلی متر و نیروی رها شدن 1.14 کیلونیوتن برای تراکم ۱.۱۳×۱۰−۳ گرم میلی متر در ۳، مقاومت در برابر نفوذ ۱۱۱ نیوتن، استحکام ۵۲.۴ مگاپاسکال، قدرت ۱−۵۲ مگاپاسکال در تنش ۵۱ گرم در آب پیشنهاد شد.