

Swelling, erosion and physicochemical characteristics of plum powder tablets obtained by spray drying

Características fisicoquímicas, de hinchamiento y erosión de tabletas de polvo de ciruela obtenido mediante secado por aspersión

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Abstract

Mexican plum juice was spray dried to obtain plum powder to develop tablets as a source of vitamin C and pectins. Plum powder presented poor flow characteristics and low capacity to compaction so different binders were probed. Corn starch (CS), carboxymethyl cellulose (CMC) and sodium alginate (SA) were added in 15 and 30 % w/w and tablets were made at 30 and 40 kN of compression load. Water activity (a_w), moisture content (%X), color, hardness, disintegration time, % water uptake and erosion were evaluated. Tablets presented acceptable values of aw (0.364-0.513) and %X (1.14-2.79), color parameters indicated more presence of yellow-red coloration when maximum compression load was applied. Starch formulations presented shortest disintegration times (4.24-6.43 min) and acceptable hardness to be handled and transported without damage (3.441-4.8618 kg_f), CMC and alginate formulations took more time to disintegrate in water however were synergic in acidic medium allowing faster dissolution, compared to water, and high erosion (>80%).

Keywords: Mexican plum powder, spray drying, tablets, erosion, disintegration time.

Resumen

El jugo de ciruela mexicana se secó por aspersión para obtener polvo de ciruela para desarrollar tabletas como fuente de vitamina C y pectinas. El polvo de ciruela presentó escasas características de fluidez y baja capacidad de compactación por lo que se probaron diferentes aglutinantes. Se agregaron almidón de maíz (CS), carboximetilcelulosa (CMC) y alginato de sodio (SA) en 15 y 30 % p/p y se hicieron tabletas a 30 y 40 kN de carga de compresión. Se evaluó la actividad de agua (a_w) , contenido de humedad (%X), color, dureza, tiempo de desintegración, porcentajes de absorción de agua y erosión. Las tabletas presentaron valores aceptables de aw (0.364-0.513) y %X (1.14-2.79). Los parámetros de color indicaron mayor presencia de coloración amarillo-roja cuando se aplicó la máxima carga de compresión de 40 kN. Las formulaciones de almidón presentaron los tiempos de desintegración más cortos (4.24-6.43 min) y valores de dureza aceptables para ser manipuladas y transportadas sin daño (3.441-4.8618 kg_f). Las formulaciones de CMC y alginato tardaron más en desintegrarse en agua, sin embargo, fueron sinérgicas en medio ácido, lo que permitió una disolución más rápida en comparación con el agua y una alta erosión (>80%). *Palabras clave*: Polvo de ciruela mexicana, secado por aspersión, tabletas, erosión, tiempo de desintegración.

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1 Introduction

Tableting of fruit powders is an emergent field of study in food industry. Some researchers like Adiba *et al.* (2011), Saifulla *et al.* (2014), Sayyad and Sakhare (2018) and Sun *et al.* (2020) have worked with food powders compaction in order to obtain different tablet forms focusing on the development of new products incorporating novel ingredients with the absence of artificial colors (Osorio-Fierro *et al.*, 2017). It has been demonstrated that tablets can be consumed as health supplement and are easy to be identified and accepted by consumers. These tablets must have low moisture content as it facilitates stability and longer shelf life (Yusof *et al.*, 2011). Additionally tableting of powders facilitates storage, transportation, packaging.

In the last decade, the interest for developing healthier products has increased with the necessity of improving lifestyle and preventing chronic diseases in consumers alongside increases of life expectancy (Ozen *et al.*, 2012). However, interest of using benefits of natural ingredients is constantly implied. Fruits are an important source of nutrients as dietary fiber, vitamins, minerals and have potential applications as ingredients for functional foods or nutraceuticals (Aziz *et al.*, 2018).

In Mexico, there are several fruits with nutritional value such as plums. There are some recent studies related with physical, physiological, and chemical characteristics of Mexican plums (Muñoz-Lopez et al., 2018; Álvarez-Vargas et al., 2017; Villarreal-Fuentes et al., 2019) nevertheless there is scarce knowledge about obtaining and processing plum powders to develop functional products as tablets. Mexican plums (Spondias purpurea L.) have demonstrated to be a good source of antioxidants or functional elements such as vitamins, phenolic compounds, carotenoids, and antioxidants. Consumption of those compounds is important in the reduction of degenerative diseases, mainly of the cardiovascular type, diabetes, and some types of cancer (Álvarez-Vargas et al., 2017). However, plums present high moisture content also a short shelf life so becomes necessary to turn it into a dry product which has several advantages extending its stability during storage.

Drying is described as a process applied to prevent the growth and reproduction of microorganisms and transforms the food into a new product and reduces packaging, storage and transportation (García-Valladares, *et al.*, 2022). Convective drying is considered by some authors (Morales-Tapia et al., 2022) an effective method also it is known by its simplicity, speed and low cost in comparison to other methods like freeze drying. Spray drying is considered an excellent option to reduce moisture content and water activity from fruits (Tontul and Topus, 2017) also is known because of the protection that provides to active compounds that are sensitive to free radical degradation, light and oxygen (Pudziuvelyte et al., 2019). This protection is owing to the fact that spray drying enables to apply high drying air temperatures and the fast evaporation keeps low droplet temperature affecting the minimum the product (Villegas-Santiago et al., 2020). Obtaining powders from fruits, like Mexican plum, may have several advantages, De Moura et al. (2015) mentioned some of them including physical stability, ease to be dosed and some important applications such as coloring and flavoring to foodstuffs or even a potential to be compressed to create solid doses. The produce of a suitable dosage form like tablets prepared by direct compression is an alternative for the oral administration of extracts for consumers and their acceptability is determined by the structure strength to be manipulated it easily and their ability to keep the form during transport, but also these solid forms should have acceptable disintegration properties for their intended purpose (Mitchel et al., 2017). Fruit powders usually have poor flowability and bad compressibility so some authors (Kurhajec et al., 2017), suggest using binders or fillers, like cellulose derivatives, to face these negative characteristics.

In this sense, the objective of this research was to assess swelling, erosion and physicochemical characteristics of tablets made by direct compression of plum powders obtained by spray drying of plum juice.

2 Materials and methods

2.1 Raw material

Mexican plum (*Spondias purpurea* L.) was obtained from a local market in Carrillo Puerto Veracruz, Mexico. The fruits were washed to remove the adhering dust. Seeds and peels were separated manually from pulp with the help of a strainer by squeezing the plums and collecting juice in a glass container. Maltodextrin D10 (7.12 % w/w) was added into the plum juice as a wall material and homogenized during 30 minutes at 2000 rpm on a magnetic stirrer.

2.2 Spray drying

Spray drying of plum juice was performed in a spray dryer (Mini spray dryer Büchi, Model B-290, Switzerland) which has an integrated two-fluid nozzle (0.7 mm diameter hole). Compressed air was used to disperse plum juice into fine droplets and subsequently dried in the cylinder. Air inlet and outlet temperatures were 179/90 °C respectively and the powder particles were separated from gas stream in a collection vessel and preserved in plastic bags for further analysis.

2.3 Powder properties

2.3.1 Moisture content (%X)

Drying efficiency can be measured by moisture content, and it should be < 5% to ensure the powder is microbiologically safe. Measurement of plum powders moisture was determined at 65°C by an infrared moisture balance (MA35 Halogen, Sartorius). Analysis was made using 1 g samples by triplicate.

2.3.2 Water activity (a_w)

Water activity values lower than 0.6 assure lack of deterioration by microorganisms and biochemical reaction on the spray dried plum juice powder (Martínez-Preciado *et al.*, 2021). This parameter was estimated by a water activity meter at 25 °C (Aqualab, Series 3 TE, Decagon, Washington).

2.3.3 Pectin yield (y_{pec})

Pectic extraction developed following was methodology reported by Muñoz-Lopez et al. (2018). 100 mL of distilled water were added to a 5 g of plum powder sample (P), then citric acid was added to adjust pH to 2.5 and solution was heated at 90°C during 60 min with continuous stirring. Cotton cloth was used to filter the resulting extract. Filtered solution was measured and an equal volume of ethanol (96%) was employed to coagulate it. After 2 hours coagulated pectin was filtered and dried in a vacuum oven (60°C) until achieve constant weight (B_i) . The following equation (1) was employed to calculate the pectin yield:

$$y_{pec}(\%) = 100 \left(\frac{P}{B_i}\right) \tag{1}$$

2.3.4 Ascorbic acid

Vitamin C (ascorbic acid) of plum powders was determined by iodometric titration according to Nweze *et al.* (2015). About 5 g (*m*) of plum powders were dissolved in metaphosphoric acid (10 % concentration) and filtered. The extracted solution was poured in a 100 mL flask and completed with distilled water. A 10 mL aliquot was taken and about 10 drops of starch solution (1%) were added. Titration was carried out with iodine 0.01 N until a blue colored solution was achieved. Ascorbic acid (Aa) reported as mg/100 g was calculated as indicates below in Equation (2):

$$Aa\left(\frac{mg}{100g}\right) = \frac{(i)(0.8806)(V_l)}{(V_f)(m)} \times 100$$
(2)

Were *i* is the volume of iodine used (mL) V_t is the volume of extracted solution (mL) and V_f is the volume of the aliquot (mL).

2.4 Mixing of powders

Powders obtained by spray drying were mixed with three different binders due to the lack of flowability and compressibility by themselves. Corn starch (CS), carboxymethyl cellulose (CMC) and sodium alginate (SA), were added to the plum powders and mixed by mean of a magnetic stirrer at 200 rpm to obtain 6 different samples. For tablet preparation 2 g of each sample were prepared and placed into plastic containers till tableting.

2.5 Densities

Powder and mix powder densities were calculated according to Yusof *et al.* (2011).

2.5.1 Bulk density

Bulk density expresses the mass of powder per volume (Ding *et al.*, 2020) and is also known as packing density. A graduated glass cylinder (previously tared) was used to pour 4 mL of plum powder, and the mass occupied by the sample was weighed. The bulk density was calculated by the Equation (3):

$$\rho_b = \frac{m}{v_b} \tag{3}$$

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Flow caracter of powders	Hausner Ratio (HR)	Carr Index (CI)
Excellent	1.00-1.11	<10
Goog	1.12-1.18	11-15
Fair	1.19-1.25	16-20
Passable	1.26-1.34	21-25
Poor	>1.35	>26

Table 1. Flow character of powder materials.

2.5.2 Tap density

Tap density is useful to determine the weight and amount of product that fits into a container and its storage conditions (Jimenez-Fernandez *et al.*, 2021). The tap density was measured from the previous sample. The glass cylinder with the plum powder was gently dropped 100 times from a 15 cm height and volume (v_t) was recorded. Tap density was calculated as follows:

$$\rho_t = \frac{m}{v_t} \tag{4}$$

2.6 Hausner ratio and Carr index

Flow of plum powder and mixes with binders was calculated by Carr Index and Hausner Ratio as reported by Saifullah *et al.* (2014). These parameters were calculated from bulk and tap densities according to the equations (5) and (6):

$$Carr Index (CI) = \frac{Tap density - Bulk density}{Tapped density}$$
(5)
Hausper Patie (HP) = Tap density (6)

Hausner Ratio (HR) =
$$\frac{H}{Bulk \text{ density}}$$
 (6)

Based on the limits reported by Kaleem *et al.* (2020) at Table 1, the flow character of powders was obtained.

2.7 Tablet formation

Tablets were made by direct compression using a pressing machine model TAB 5, Encamex, Mexico. Single circular punch and die (6 mm diameter) were used with a 0.7 mm depth. Low (L) and high (H) compression loads (30, 40 kN) were applied. Punch was filled with each mixture. Pressure was applied manually, and tablet was ejected by the machine.

2.7.1 Experimental design

A factorial design 3x2x2 was applied to evaluate different compression conditions.

Table 2.	Experimental	design	for	tablet	formul	ation	of
	plu	im pow	der	s.			

Run	Binder	Compression load (kN)	% Binder
1	CS	40	15
2	CS	40	30
3	CS	30	15
4	CS	30	30
5	CMC	40	15
6	CMC	40	30
7	CMC	30	15
8	CMC	30	30
9	SA	40	15
10	SA	40	30
11	SA	30	15
12	SA	30	30

Variables evaluated were: (1) type of binder (CS, CMC, SA); (2) compression load (30, 40 kN); (3) binder concentration (15, 30 %) as shown in Table 2.

2.8 *Physical properties of tablets*

2.8.1 Activity water, moisture content and color

Activity water and moisture content of tablets were evaluated as described previously. Tablets color was measured with a colorimeter (MiniScan XE plus, HunterLab, USA). *L*, *a* and *b* parameters were obtained from six tablets. Means were reported.

2.8.2 Tableting weight variation

Twenty tablets were precisely weighed using an analytical balance (model TE 214S, Sartorious, USA). Mean values were reported in mg and tablets were randomly selected from a batch.

2.8.3 Tableting thickness testing

The thickness of the obtained tablets was measured using a digital caliper (model HER 411, Steren, China) and mean values of all determinations were reported (mm).

2.8.4 Tablets hardness

A texturometer, CT3TM Texture Analyzer, model CT3-4500, EUA, was used to determinate hardness of tablets. A single tablet was set on a base table and a probe moved down slowly at pretest speed (2 mm/s)

until a threshold value (the trigger, 1 g) was reached. The tablet was compressed until breakdown. Test was run by triplicate. Hardness was reported as kg_f .

2.8.5 Disintegration test

Disintegration test of tablets was carried out following methodology reported by Sriamornsak *et al.*, (2007). In 800 mL of distilled water or HCl 0.1 N at 37 \pm 0.5 °C. Experiments were run in triplicate. The disintegration time was measured by a stopwatch until observing full disintegration of the plum tablets.

2.8.6 Swelling and erosion studies

Measurements of swelling and erosion of plum tablets were performed following the reported by Sriamornsak et al., (2007) with non-significative modifications. Weighed tablets (W_0) were placed in a vessel containing tri-distilled water or HCl 0.1 N at 37 \pm 0.5 °C. To avoid floating, tablets were set in a plastic container. Every 2, 5, 10, 20, 60 and 120 min, each container was taken out from the test medium and the tablet was blotted to remove excess water and then weighed (W_1) . Wet samples were eventually taken to an oven and dried at 80 °C for 24 hours. After drying the tablets were cooled into a desiccator (ambient temperature) until constant weight was registered (W_2) . Experiments were run by triplicate. A new tablet was used for each time interval. Water uptake was reported as the percentage of weight change according to the Equation 7:

% Weight change =
$$\frac{W_1 - W_0}{W_0} \times 100$$
 (7)

Following equation was used to calculate the percentage of remaining tablets after erosion (ES):

$$\% Remaining = 100 - ES \tag{8}$$

Where erosion was estimated by the equation bellow:

$$ES = \frac{W_0 - W_2}{W_0} \times 100$$
 (9)

2.9 Statistical analysis

The statistical analysis was conducted based on the designed described before, developing an analysis of variance (ANOVA) with the software Minitab® 18.1.0. The significant differences were determined by Tukey's test at the 95 % confidence level.

3 Results and discussion

3.1 Physicochemical properties of plum powders

Powders presented water activity values lower than 0.6 indicating that plum powders obtained were microbiologically stable and have an acceptable shelflife. Moisture content also presented acceptable values between 3.05 and 3.57 %. Pectin and ascorbic acid presented values of 6.29 % and 82.87 mg/100 g, respectively. Both parameters were found to be higher compared to those observed in the fresh fruit (1.226%, 34.733 mg/100 g). Some authors (Zea et al., 2013) attribute it to the concentration of ingredients in the dried form and due to the much higher solid content in powder than in the fresh fruit. As described previously, plum powders obtained by spray drying were characterized. Short life of food products and the presence of biochemical reactions is determined by high water activity and more free water available (Todisco et al., 2013). Several authors as Queck et al., (2007) have reported that food with water activity under 0.6 is microbiologically stable and if any possible spoilage occurred it will be caused by chemical reactions instead of microorganisms.

3.2 Mix powder properties

Table 3 contains properties of plum powders and mixes. Based on limits mentioned before in Table 1 of Carr Index and Hausner Ratio plum powders obtained by spray drying presented poor flow properties, which may difficult compaction behavior. Plum powders conditioned with binders provided fair and passable flow. Javanbakht and Shaabani (2019) reported CMC useful for various drugs as metoprolol, losartan and ibuprofen because of its good characteristics as hydrophilicity, non-toxicity, ability to gel-forming and bio adhesivity. Corn starch has shown less flowability in comparison to other materials as microcrystalline cellulose (Zhang et al., 2003) however has presented binding and fragmentation mechanisms and according to Jelkmann et al. (2019) seem to be promising excipient for mucosal drug delivery. Also controlled release tablets have been prepared with starch as excipient with diprophylline (treatment of respiratory disorders) contributing to the development of novel advanced drug delivery systems (Elgaied-Lamouchi et al., 2021).

	Bulk density g/cm ³	Tapped density g/cm ³	Hausner Ratio	Carr Index
Plum	0.2775 ± 0.0019	0.4625 ± 0.0031	1.6667	40
P+CS 15%	0.2839 ± 0.0019	0.3786 ± 0.0026	1.3333	25
P+CMC 15%	0.2990 ± 0.0022	0.3737 ± 0.0027	1.25	20
P+SA 15%	0.3373 ± 0.0024	0.4497 ± 0.0032	1.3333	25
P+CS 30%	0.2815 ± 0.0018	0.3519 ± 0.0022	1.25	20
P+CMC 30%	0.2900 ± 0.0031	0.3867 ± 0.0041	1.3333	25
P+SA 30%	0.3190 ± 0.0018	0.3987 ± 0.0022	1.25	20

Abbreviations:P+CS, Plum+Corn starch; P+CMC, Plum+Carboxymethyl cellulose; P+SA, Plum+Sodium Alginate.

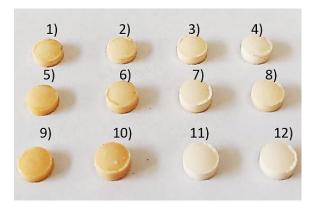


Figure 1. Plum powder tablets obtained at different conditions.

On the other hand, sodium alginate has fulfilled several roles such as tablet binder in acetyl salicylic acid tablets (Holte et al., 2003), masker of the bitter taste of amiprilose hydrochloride (Kaneko et al., 1997) or controlled-release matrix tablets like capsules containing pseudoephedrine hydrochloride and different grades of sodium alginate (Veski et al., 1994).

3.3 *Physicochemical* characteristics of plum tablets

Figure 1 shows tablets obtained by direct compression for each formulation. Color evaluation indicated luminosity values (L) between 47.65 and 59.84 (Table 3). ANOVA showed a significant effect of compression load. Luminosity (L) of tablets compressed at 30 kN was higher and there were no significant differences of binder or concentration added. Compression load and type of binder had significant effect on parameter a, that represents the red color in all samples. Tablets compressed at 40 kN and starch as binder presented

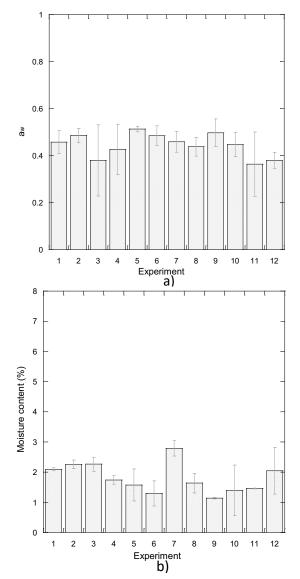


Figure 2. a) Water activity and b) moisture content of plum tablets.

Table 4. Color parameters of plum tablets.						
	P+CS		P+CMC		P+SA	
	15%	30%	15%	30%	15%	30%
Parameter		30 kN				
L	53.7	58.48	50	52.305	59.84	57.19
a	2.37	1.99	3.205	2.78	1.23	1
b	14.255	14.99	13.97	15.88	15.7	16
40 kN						
L	48.29	47.955	48.605	47.65	48.65	48.14
а	4.795	4.17	4.005	3.81	3.13	3.685
b	16.83	17.315	18.885	19.575	17.6	18.74

Table 5. Physical properties of plum tablets.

Run	Weight (mg)	Thickness (mm)	Hardness	Density	Disintegratio	on time (min)
	n= 20	n = 10	(kg_f)	mg/mm ³	H ₂ O	HCl 0.1N
1	81.80 ± 1.158	2.47 ± 0.06	3.4410 ± 0.408	116.19 ± 3.009	5.12 ± 0.283	5.00 ± 0.165
2	82.48 ± 0.370	2.20 ± 0.00	4.8618 ± 0.659	132.25 ±0.491	4.24 ± 0.460	6.43 ± 0.306
3	91.46 ±1.753	2.73 ± 0.06	3.5848 ± 0.523	117.15 ± 0.332	5.27 ± 0.024	6.41 ± 0.295
4	88.64 ± 0.780	2.73 ± 0.06	4.2855 ± 0.718	114.15 ± 1.496	4.64 ± 0.413	5.43 ± 0.589
5	87.08 ± 1.314	2.40 ± 0.00	4.4590 ± 0.644	127.08 ± 1.003	36.56 ± 0.413	11.03 ± 0.743
6	97.44 ± 0.404	2.50 ± 0.00	5.1908 ± 0.106	137.56 ± 0.536	61.20 ± 6.388	9.06 ± 1.120
7	82.46 ± 0.830	2.80 ± 0.00	2.8833 ± 0.251	103.58 ± 0.505	32.24 ± 0.766	8.73 ± 0.283
8	87.82 ± 0.432	2.80 ± 0.00	3.9320 ± 0.102	110.57 ± 0.318	61.02 ± 1.485	9.10 ± 1.391
9	90.02 ± 0.661	2.57 ± 0.06	3.2570 ± 0.221	123.60 ± 3.006	30.38 ± 0.118	10.41 ± 0.342
10	95.54 ± 0.720	2.53 ± 0.06	4.4935 ± 0.410	132.90 ± 2.323	63.18 ± 1.827	20.76 ± 0.931
11	80.88 ± 0.687	2.70 ± 0.00	2.9223 ± 0.303	105.32 ± 0.347	22.03 ± 4.702	10.20 ± 0.236
12	78.22 ± 0.217	2.70 ± 0.00	3.9300 ± 0.242	102.30 ± 0.00	51.48 ± 3.701	15.55 ± 1.273

higher a value (4.17, 4.79). There were significant differences in parameter b (represents the yellow of the sample). According to the statistical analysis 40 kN load and CMC presented the higher values of parameter b (18.885-19.575). According to the results obtained, high compression loads and P+CS/P+CMC formulations created, more red-yellow tablets. Color surely has an influence on flavor appreciation by consumers and, in fact, may outweigh flavor even when the flavors are pleasant, and the food is a popular one (Clydesdale, 1993).

3.4 Water activity and moisture content of tablets

Tablets presented activity water values under 0.6 and low moisture content (Figure 2a). Statistical analysis demonstrated a significant effect of compression load. Activity water increased with the increasing of compression load; it can be related to the particle breakdown and the subsequent release of water contained in the matrix when a high load was applied. With respect to moisture content there were no significant differences in measurements for each experiment. Moisture content presented values under 5%, a similar range was reported by Sun et al. (2020) in dispersible fruit tablets (mango, Chlorella, and cactus powder) from 2.46-3.62%). They suggested that moisture content affects powder flowability and a low moisture content in preferable for the preparation of tablets.

Physical properties of plum powder 3.5 tablets

Weight, thickness, density, hardness and disintegration time corresponding to the tablets created following the experimental design are summarized in Table 5. Weight and thickness of the formulations ranged from 78.22 to 97.44 mg and 2.20 to 2.80 mm respectively. Weight variation among different formulations was attributed to the binders and the quantity added.

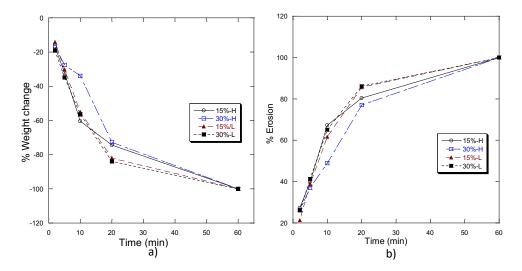


Figure 3. a) Percentage of weight change and b) erosion versus immersion time of different starch tablet formulations in H₂O (H=High compression load, L=Low compression load).

Volume increased when compression load was low, allowing more space between powder particles. Density values were affected significatively by binder percentage and load. Tablets created at high levels of the design (30%, 40 kN) presented a mayor density.

Hardness has a significant influence on the resistance of the tablets to breakage under storage, transport, and transfer conditions before usage (Divya *et al.*, 2011). Hardness of plum tablets (2.8833-5.1908 kg_f) was influenced by binder amount and compression. As expected, the increasing of both factors, created harder tablets. Values of hardness are similar to barberry effervescent tablets, 3.98-4.59 kg_f (Naji-Tabasi *et al.*, 2021) and isolated mucilage tablets, 3.1-4.2 kg_f (Sayyad and Sakhare, 2018) which indicated an excellent ability and strength to resist physical and mechanical stress conditions when tablets are manipulated.

Binders and concentration had a significant effect on disintegration. High presence of binders increased disintegration time; those materials acted as a glue, adding more cohesiveness between plum powder particles (Al-Achi, 2019). Tablets containing starch as binder dissolved faster than CMC or alginate formulations. Manudhane et al. (1969) found that starch acts as a disintegrating agent in lower concentrations (18.5%) and dissolution of the tablets in this study took from 5-15 min. Water as a disintegration medium increased the disintegration time, except for starch formulations that diffused quickly into the media. CMC and alginate formulations showed a faster disintegration in HCl when compared to H₂O. Some authors found a similar behavior and suggest that rapid dissolution on HCl is due to the pH of the fruit powders (pH=3.45). Zea *et al.* (2013) studied guava and pitaya powders (pH \approx 4) and the biological compounds of the fruit tablets were synergistic in the pH 4 buffer solution. The authors explained that when the pH of the fruit juice is less than 4, the fruit powder tablet dissolution study can be easily carried out with HCl or a buffer solvent at low pH.

3.6 Swelling and erosion of plum powder tablets in H₂O

Figure 3 shows weight change and erosion of P+CS tablets immersed in water (37°C). As can be observed all formulations presented negative weight variation (Figure 3a) during the essay due to the rapid disintegration of the tablets. It can be observed that plum tablets followed a similar behavior except 30%-H formulation due to the high presence of binder and more compacted ingredients (40 kN). Erosion was completed at 60 min so that the assessment was interrupted due to the absence of tablets to be weighted. Figure 3b shows formulations compressed at high loads eroded slower than low ones. The water uptake (Figure 4a) was more significant on carboxymethyl cellulose tablet owing to the extremely hydrophilic nature of CMC with increasing its content in the matrix the swelling is increased (Javanbakht and Shaabani, 2019). Also, Wan and Prasad (1989) found that increased of percentage of weight at a high concentration of CMC is caused by the slow hydration of the binder on the surface and the outer layers of

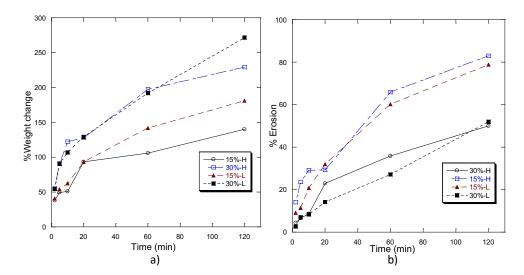


Figure 4. a) Percentage of weight change and b) erosion versus immersion time of different CMC tablet formulations in H₂O (H=High compression load, L=Low compression load).

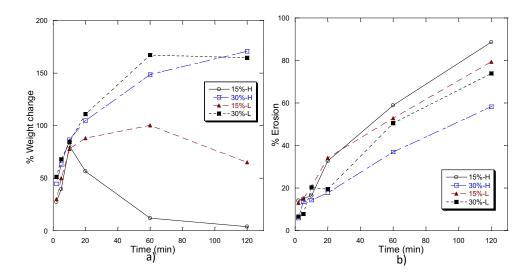


Figure 5. a) Percentage of weight change and b) erosion versus immersion time of different sodium alginate tablet formulations in H₂O (H=High compression load, L=Low compression load).

the tablet. As long as this takes place, the penetration of water into the tablet is blocked by the swollen binder. As a consequence of this behavior, erosion was slower at high concentrations of CMC (Figure 4b). With respect to water uptake essays of alginate-based tablets (Figure 5a), was observed a similar behavior among formulations until 10 minutes, when tablets with 15% of binder concentration and compressed at high load started to erode and percentage of weight change was reduced. Graphic of weight change shows that each formulation reached a maximum water uptake and subsequently started to erode, depending on the quantity of binder and compression load. First eroded tablets were those containing 15% of alginate. Followed by 30% and low compression load (30kN) at 60 min. Tablets with the highest binder content and compression load continued swelling until 150 minutes and started to decreased the powder content, nevertheless, the study was stopped at 120 min due to the disintegration of the rest of the tablets and difficulty to be weighted. Efentakis and Buckton (2002) reported a similar behavior of theophylline



Figure 6. Formed viscous gel layer at the plum+alginate tablets surface.

tablets containing alginate; matrices exhibited a maximum water uptake and swelling in water between 2 and 4 hours and then decreased when erosion started, which was completed between 3 and 5 h. It was also observed a highly viscous gel at the compact surface when tablet encountered water as shown in Figure 6 with plum alginate (30%) tablets.

3.7 Swelling and erosion of plum powder tablets in HCl 0.1 N

Figure 7 represents weight change and erosion of plum+CS tablets immersed in HCl 0.1 N. As it was observed in water experiments, tablets immersed in HCl registered a negative weight change because of the instant erosion when tablets were in contact with a liquid media. 15%-L and 30 %L tablets decreased its content faster and described a quick and almost a complete erosion during the first 20 minutes. Tablets created at high compression loads, achieved complete erosion before 120 minutes but more time was required to achieve at least 50% of erosion when compared to 15%-L and 30%-L tablets. When the tablets exhibited lower bonding strength, the liquid media penetrated easily and in consequence facilitated faster tablet breakdown (Ong *et al.*, 2014).

Figure 8a describes swelling behavior of plum+CMC tablets. A marked difference was observed between the formulations. 15%-H tablets registered an increase of weight during the first 2 minutes and eventually a dispersion of its components in the media. A similar performance exhibited 15%-L tablets. 30%-H tablets, presented a higher capacity to swell, reaching a maximum at 5 min and immediately started to diminish the tablet form and mass. 30%-L swelled until 20 min before starting to disintegrate. Tablets presented high levels of erosion >80% (Figure

8b) at the end of the assessment. Varshosaz *et al.* (2006) found similar results with Metoprolol+CMC tablets. Reported that increment of the CMC ratio decreased the water uptake and erosion of the tablets. During first 10 minutes, erosion reported values between 25-35% very close to plum+CMC tablets with ranges between 24.3-26.7 % of erosion.

Figure 9a shows plum+SA tablets weight change during immersion in HCl 0.1N. 30%-L tablets increased considerably its weight until 10 minutes, subsequently, values at 20, 60 and 120 min were very close (52.89, 53.07 and 63.71 %) indicating an imminent disintegration and reduction of weight as seen on the rest of formulations. 30%-H tablets allowed to observe increasing and reduction of tablet content registering a negative weight change after minute 20. 15% alginate tablets described a similar behavior nevertheless positive weight change was registered until minute 5. At that point tablets started to dissolve and losing weight. The 15%-L tablets reached a complete disintegration at minute 120, it is possible that they hydrated slower, forming a gelatinous barrier in a longer time, therefore, a more rapid erosion or dissolution of the alginate particles took place. Alginate content was determinant to extend time to swell and erode, high concentration of alginate extended time owing to hydration of an alginate matrix leads to the formation of a characteristic gel layer which usually acts as a diffusion barrier (Liew, et al., 2006).

Conclusions

Spray drying of Mexican plum juice produced powders with potential to be compressed in tablet forms and provide to consumers a practical form to consume plums and its beneficent compounds as ascorbic acid and pectin. Binders, in small amounts 15-30% provided to the plum powders the necessary features to be compressed and flowable. Plum juice powder tablets with a diameter of 6 mm and thickness 2.2-2.8 mm were obtained. Tablets of plum + corn starch (30%) and compressed at 40 kN were selected as the best samples due to its short times to disintegrate, hardness necessary to prevent damages $(>45 \text{ N or } 4.59 \text{ kg}_f)$ and good color characteristics, however erosion and disintegration studies were important to determine the possible uses of plum tablets.

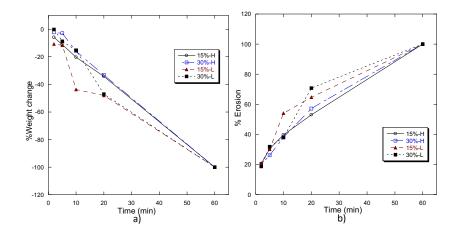


Figure 7. a) Percentage of weight change and b) erosion versus immersion time of different starch tablet formulations in HCl 0.1 N (H=High compression load, L=Low compression load).

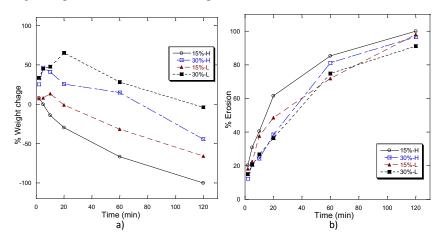


Figure 8. a) Percentage of weight change and b) erosion versus immersion time of different CMC tablet formulations in HCl 0.1 N (H=High compression load, L=Low compression load).

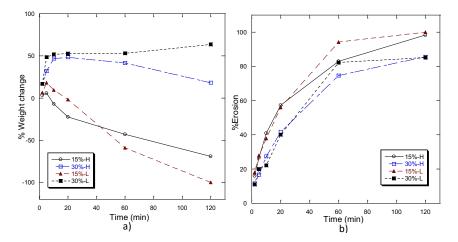


Figure 9. a) Percentage of weight change and b) erosion of different sodium alginate tablet formulations in HCl (H=High compression load, L=Low compression load).

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Those containing CMC and alginate had the potential to be used by consumers with some difficulties to chew or swallow and have the preference to eat tablets as a candy, also plum tablets are able to be part of delivery systems in combination with other drugs.

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Nomenclature

CS	Corn starch
CMC	Carboxymethyl cellulose
SA	Sodium alginate
a_w	Water activity
Урес	Pectin yield [%]
Aa	Ascorbic acid
$ ho_b$	Bulk density
ρ_t	Tap density
L	Luminosity
а	Red-green color
b	Blue-yellow color
HR	Hausner Ratio
CI	Carr index
ES	Erosion
Н	High compression load
L	Low compression load
P+CS	Plum+Corn starch
P+CMC	Plum+Carboxymethyl cellulose
P+SA	Plum+Sodium Alginate

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