World Academy of Science, Engineering and Technology
International Journal of Nutrition and Food Engineering
Vol:8, No:10, 2014

Influence of the Compression Force and Powder Particle Size on Some Physical Properties of Date Fruit (*Phoenix dactylifera*) Tablets

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***Abstract*—**In recent years, the compression of date (*Phoenix dactylifera* L.) fruit powders (DP) to obtain date tablets (DT) has been suggested as a promising form of valorization of non commercial valuable date fruit (DF) varieties. To further improve and characterize DT, the present study aims to investigate the influence of the DP particle size and compression force on some physical properties of DT. The results show that independently of particle size, the hardness (y) of tablets increases with the increase of the compression force (x) following a logarithmic law ($y = a \ln (bx)$ where *a* and *b* are the constants of model). Further, a full factorial design (FFD) at two levels, applied to investigate the erosion %, reveals that the effects of time and particle size are the same in absolute value and they are beyond the effect of the compression. Regarding the disintegration time, the obtained results also by means of a FFD show that the effect of the compression force exceeds 4 times that of the DP particle size. As final stage, the color parameters in the CIELab system of DT immediately after their obtaining are differently influenced by the size of the initial powder.

***Keywords*—**Powder, valorization, tablets, date fruit (*Phoenix dactylifera* L.), hardness, erosion, disintegration time, color.

I. Introduction

In recent years, food processing is subject to enormous inputs in terms of innovation which involves health claim, convenience, method of obtaining and environmental awarness.

Many research studies were devoted to the fruit of various species of date palm (*Phoenix dactylifera* L.) in terms of physicochemical and biological characterization: Medicinal properties [1], chemical composition [2], physical properties [3], antioxidant activity [4] etc. Further investigations concern date fruit processing such as: obtaining juice and / or syrup [5], alcohol [6] yeast [7], vinegar [8], powder [9] etc.

Recently, date powder (DP) compression to obtain date tablets (DT) has been suggested as a possible form of valorization of date fruit (DF) [10]-[12]. This could be introduced as an innovation [13] involving significant changes in the conditions of use DF by the consumers.

In the present research work we assessed: 1) the influence of DP particle size on the hardness, disintegration time and color of the tablets obtained from the DF variety *Mech-Degla*; and 2) the influence of DP size, compression force and time on the erosion of the tablets by applying the FFD.

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II. Materials and Methods

A. Obtaining Tablets

DPs were obtained by drying DF at 85°C. The resulting powder was subjected to sieving for the sake to obtain powders with four different particle sizes: $250 < G_1 \leq 315$µm, $180 < G_2 \leq 250$µm, $80 < G_3 \leq 180$ µm and $G_4 \leq 80$ µm. As second step, the tablets were made from DP by direct compression [14]. Finally, five compression forces were applied: 0.5, 1, 1.5, 2, 2.5, 3 and 4 tons.

B. Hardness

The influence of particle size on the compression behavior of pharmaceutical mixtures has been studied [15]. In this section, the variation of the hardness versus compression force was analyzed for the four sizes chosen. The hardness measurement was carried out with a Shore Durometer. The hardness value was identified by the penetration of the Durometer indenter foot into the sample (tablets) under the action of a force. The hardness value was recorded after 3s penetration. The unit of measurement was "shore D". The measurement was repeated six times and the final result was expressed as mean ± standard deviation.

C. Erosion

According to the literature, the erosion of the tablets is usually assessed against time, in different dissolution media. It seems to us rational to analyze the phenomenon of erosion by applying a FFD. Three factors (independent variables) were considered including time of immersion in liquid media (distilled water), DP particle size and compression force.

% Erosion was exposed after DT drying at 80°C as follows:

$$\%\ erosion = 100\ \frac{W_0 - W_t}{W_0}$$

where W_0 and W_t = initial and at time (*t*) DT weight.

D. Disintegration Time

This test involves scoring the time required for complete disintegration of DT in a beaker containing 150 ml of distilled water at 37°C and using a stirrer (250TR / min). The FFD was also performed to study the variation of disintegration time in function of the compression force (X_1) and DP particle size (X_2).

E. Color

The color determination was performed on five DT prepared with different particle sizes of the initial DP. A reference tablet was prepared with a paste of crushed and lyophilized dates. The determination was made in the laboratory of paints (ENAP, Lakhdaria, Eastern Algiers, Algeria) by using of a colorimeter (Type CM-2500 MINOLTA).

III. Results and Discussion

A. Obtained Powders and Tablets

Fig. 1 shows the four samples of powders obtained according to different DP particle sizes. As it is displayed, the external appearance of the powders is influenced by the particle sizes each: a small particle size might give a brighter product. DT is obtained by applying different five compression forces on these DP (Fig. 2).

Fig. 1 Different types of date powder (DP) according to the particle size

Once again, it can be visually noted that the external appearance of the DT depends on the DP particle size as well as the compression force applied during the tableting process.

TABLE I
Parameters of the Logarithmic Model Related to the Hardness

Particle size	a	b	R^2
$250 \prec \mathbf{G_1} \leq 315\mu m$	12,08	18,08	0,993
$180 \prec \mathbf{G_2} \leq 250\mu m$	12,36	19,92	0,986
$80 \prec \mathbf{G_3} \leq 180\mu m$	12,72	21,63	0,980
$\mathbf{G_4} \leq 80\mu m$	12,22	25,61	0,994

y= a ln (b) where *y* = hardness value.

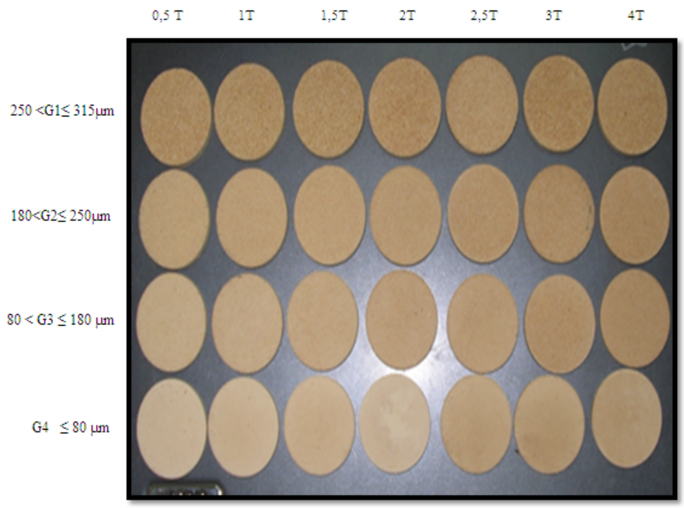

Fig. 2 Different DT manufactured with different DP particle sizes and compression forces (in tons, T) G: DP particle size.

B. Hardness

It is commonly known that the hardness is among the important physical properties of the tablets [16]. Fig. 3 shows that the logarithmic profile of the curve is identical for all applied sizes. The shape of the curves is similar to that reported by different authors about different kinds of pharmaceutical tablets [17]-[19]. Additionally, from the Fig. 3 it is clear that the DP particle size influences the hardness of DT. The model parameters given in Table I confirm this observation since the coefficient b can be sketches in the following order: G4> G3> G2> G1.

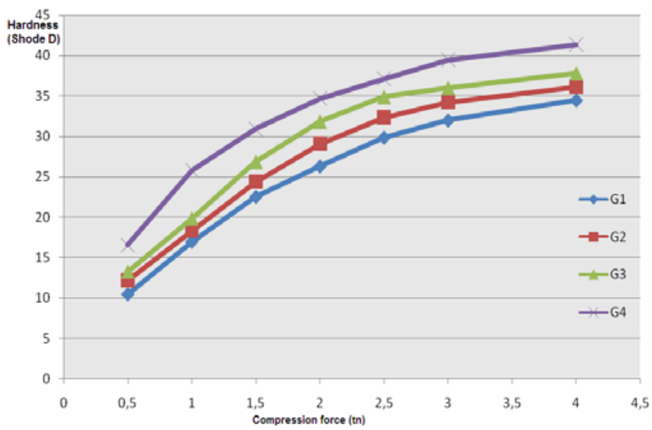

Fig. 3 Hardness versus compression force, according to the diffrent DP particle size (G)

C. Erosion

Table II shows the matrix of experience that summarizes the experimental conditions and the responses in terms of erosion.

The calculator (Gc = 0.3965) and tabulator (Gt = 0.6798) Cochrane criteria indicated homogeneity of the variances (Gc < Gt) and the experiences are replicated.

TABLE II
EXPERIENCE MATRIX RELATED TO THE DT EROSION

N	X_1	X_2	X_3	Y_1	Y_2	Mean	SD
1	-	-	-	0.88	0.85	0.865	$0.045.10^{-2}$
2	+	-	-	0.59	0.54	0.565	$0.125.10^{-2}$
3	-	+	-	16.29	16.1	16.22	$0.98.10^{-2}$
4	+	+	-	8.70	8.55	8.755	$4.5.\ 10^{-2}$
5	-	-	+	23.34	23.1	23.26	$1.28.10^{-2}$
6	+	-	+	3.65	3.50	3.575	$1.13.10^{-2}$
7	-	+	+	68.10	67.8	67.98	$2.88.10^{-2}$
8	+	+	+	24.42	24.2	24.37	$0.41.10^{-2}$

X_1: Compression force, X_2: time, X_3: particle size, Y_1 and Y_2: responses (erosion), SD: standard deviation.

Pursuing this further, the significance of regression coefficients has been demonstrated through the Student test (Table III).

TABLE III
RESULTS OF STUDENT TEST: CASE OF HARDNESS

Coefficient designation	Student t value	Values of model coefficients
a_0	610.924	18.20
a_1	298.077	8.88
a_2	373.704	11.133
a_3	389.313	11.598
a_{12}	130.408	3.885
a_{13}	232.957	6.940
a_{23}	176.127	5.247
a_{123}	70.290	2.094

As therefore, the equation of the mathematical model is as follow:

$$Y = 18.20 - 8.88F + 11.133T - 11.598G - 3.885FT$$
$$- 6.940FG + 5.247TG - 2.094FTG$$

In the light of this equation, it is clear that the time (T) and the size (G) are the most influent factors, compared to the compression force. The latter is in any case known as the factor, which influences the quality of the tablets [20]. Further, the effects of time (T) and the size (G) are identical in absolute value but they are of opposite signs. Needless to say it has become also from the model that the time effect is higher than that of the DP particle size. Negative interactions between F/T and T/G indicate antagonism between the corresponding factors. Still, a synergistic effect is observed between T and G.

D. Disintegration Time

The variation of disintegration time in function of compression force for the three sizes (G1, G2 and G3) is illustrated in Figs. 4 (a)-(c). The curve corresponding to the size G1 (Fig. 4 (a)) is of "S" shape in the form of "S", unlike particle size G2 (Fig. 4 (b)) and G3 (Fig. 4 (c)) which are straight (R2 > 0.98). The "S" shape has been revealed by [21] the study of the dissolution of an active ingredient versus time in the case of tablets based on alginate-gelatin mixture.

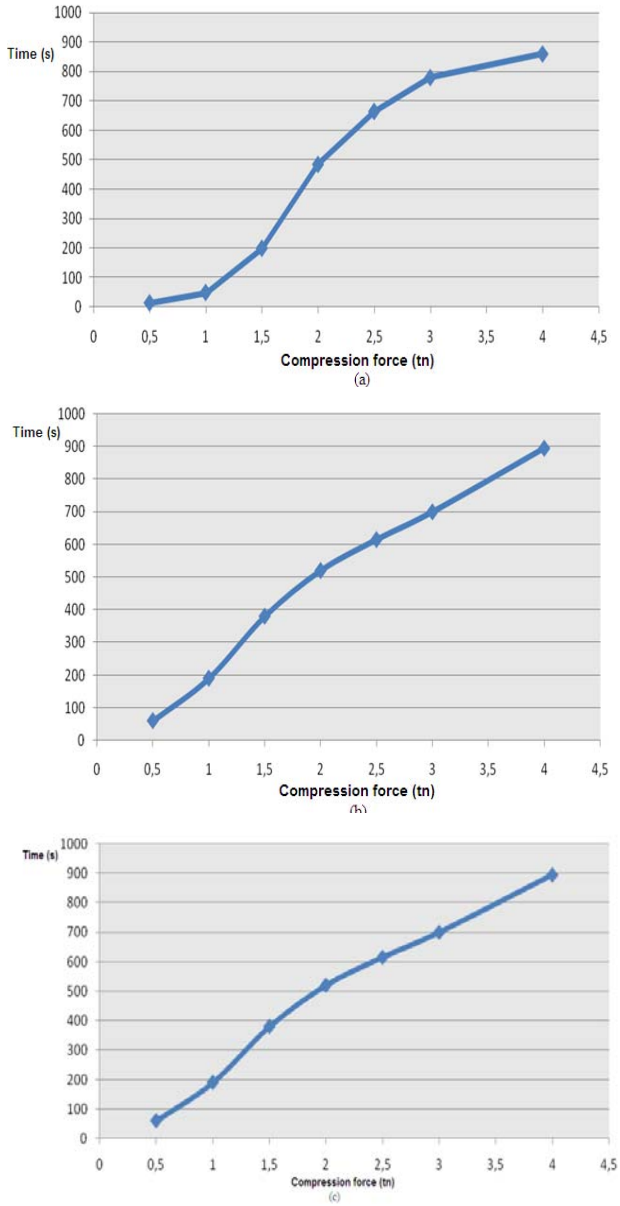

Fig. 4 Disintegration time versus compression force, according to the DP particle size: G1 (a), G2 (b) and G3 (c)

The experience matrix (Table IV) summarizes the results obtained results of FFD; the independent variables being the force (X1) and particle size (X2).

TABLE IV
EXPERIENCE MATRIX RELATED TO THE DT DISINTEGRATION TIME

N^O	X_1	X_2	Y_1	Y_2	Mean
1	-	-	275	285	280
2	+	-	960	970	965
3	-	+	12	18	15
4	+	+	855	865	860

X_1: Compression force, X_2: particle size and Y: response (disintegration time).

The calculated (Gc = 0.2976) and tabulated (Gt = 0.9065) Cochrane criteria indicate that variances are homogeneous

(Gc < Gt) and the experience is reproducible. More exactly, the significance of regression coefficients has been demonstrated through the Student test as Table V.

TABLE V
RESULTS OF STUDENT TEST: CASE OF DISINTEGRATION TIME

Coefficient designation	Student t value	Values of model coefficients
a_0	100.95	530
a_1	72.86	382.5
a_2	17.62	92.5
a_{12}	7.62	40

The corresponding mathematical model is of the following form:

$$Y = 530 + 382.5F - 92.5\,G + 40FG$$

The effect of the compression force is higher and exceeds 4 times the effect of particle size. It is widely argued that the compression force is defined as a factor, which increases the disintegration time of the tablets.

E. Color

The histogram of Fig. 5 shows that the color parameters of the tablets are differently influenced by the particle size of the initial DP. Fluctuations in the values is observed for the parameters, except for whiteness (L) which appears to move in a uniform manner with the particle size what confirms the visual observation previously mentioned above (see section III *A* and Fig. 1). All in all, the other color parameters are close to those of the control.

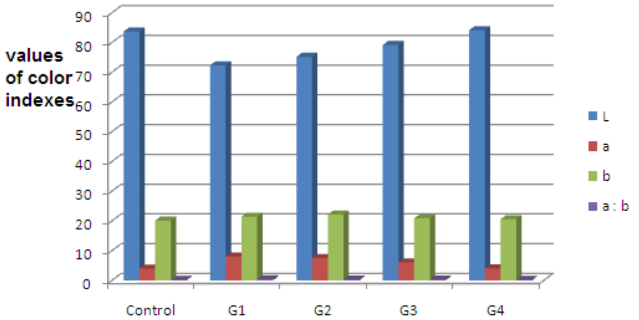

Fig. 5 Color parameters of DT versus DP particle size

IV. CONCLUSION

Thus, our results show that the DT behaves globally like those of pharmaceuticals; thereby they can be characterized by the same tests. The DT hardness varies with the pressing force, which variation is properly described by a logarithmic equation. Further, the applied FFD to analyze the % of erosion indicate that the effects of time and particle size are the same in absolute value and beyond the effect of the compression.

Regarding the disintegration time, the obtained results regarding FFD show that the effect of the compression force exceeds 4 times the one of DP particle size.

Finally, the color parameters in CIELab system, obtained on DTs immediately after their processing, seem to be differently influenced by the size of the initial DP.

The current study demonstrates the feasibility of producing DT from DF which opens new options for the use of DF in food processing as well as for consumers' consumption.

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