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Original article

Effect of Drying and Grinding on Biochemical and Functional Properties of Deglet Nour Date Fruit Powder

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Abstract

The objective of this study was to characterize a date powder from Deglet Nour. For this purpose, the effects of conventional drying and grinding processes were evaluated. Deglet Nour dates powders were obtained through a convective hot air drying (HAD), at a temperature of 70°C and air flow velocity value of 1.5m s-1. Color coordinates, water activity (aw), moisture, sugar, fat and ash contents, hygroscopicity, wettability, water solubility index (WSI), and total phenolic content of dates powder were determined. The obtained powder has great degree of hygroscopicity, and this is due to its low moisture <8 g g-1 H2O) and its low water activity (0.443) which increased the water concentration gradient between the product and surrounding air. Thus, it has higher capacity to absorb moisture from environment. On the other hand, both solubility index and sugar content were affected for date powder after drying and grinding processes. Our findings ascertained that intensification of both processes should be applied to overcome these negative impacts.

Keywords: Date Powder, Convective Drying, Grinding, Quality Attributes, Sugar Content.

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INTRODUCTION

Date palm fruits have high nutritional composition and present appreciable antioxidant properties and minerals components(Farahnaky, Mansoori, Majzoobi, & Badii, 2016). However, dates fruits may be highly perishable given their water content, which make them easily affected by micro-organisms mainly through fermentation reactions. In addition, in the dates industries sector, after sorting superior quality fruits intended to be sold fresh, the resulting sorting discrepancies are also fruits of valuable nutritional value and source of bioactive compounds that should be valued. These low-quality fruits may reach up to 10% of the dates which make it essential for the date sector to prevent these fruits from deterioration by using several available methods. Finding alternatives to reintegrate these fruits into the human consumption circuit may be challenging and of great interest for the date sector. Currently, low value dates are used to prepare syrup, jam, etc. However, developing date powder from sorting deviation dates, at the industry level, will allow these products range to be extended. Indeed, date powder may be used as a sweetener substitute for bakery, confectionary, and beverages products.

On the other hand, consumers’ awareness of the health benefits associated with eliminating or reducing sugar in their diets has led to an increase in the demand for natural sugar alternatives. Dates are high in sugar content and can therefore be used in many industries as a sugar substitute. For this purpose, dates must be stabilized in order to lengthen their shelf life but also should be presented in forms that facilitate their use in the agri-food sector. Drying, while reducing the product water content, may allow achieving these purposes. Indeed, Drying is the unit operation which removes moisture from the product for its safe storage, which will increase its shelf life. Moreover, convective hot air drying (HAD) is still considered as among the cheapest, easiest, and most common long term preservation technique, mainly for perishable products. Nowadays, dried products are having better good market benefits than the fresh because of their advantages (Karam, Petit, Zimmer, Baudelaire Djantou, & Scher, 2016; Sablani, Shrestha, & Bhandari, 2008).

Although several forms of dates such as fresh dates, date paste and date syrup are available in the market and used for different application, free flow date powder would be highly beneficial in improving shelf life, easiness in handling, and bendability with various foods prepared in domestic and industry level. In addition, using powders may lead to economic benefits due to their volume and weigh reduction reducing packaging, storage and transport fees.

This research aims to evaluate the effects of drying and grinding on the physicochemical composition of Tunisian Deglet Nour date. Besides, it aims to characterize the functional properties of the obtained date powder.

MATERIALS AND METHODS

Sample preparation

Dates (cv. *Deglet Nour*) were harvested at southern Tunisia, at 2020-2021 campaign. They were collected at the "Tamr stage" (full ripeness). They were brought from the VACPA BOUDJEBEL date drying process unit at Bni Khalled (Tunisia) where they were designated as fresh 'Deglet Nour' dry dates.

Fresh, dried dates samples and date powder were sorted into three batches for quality measurements. Only clear fruits were chosen and stored in a cold room at 4°C. Dates that were already physically damaged were rejected.

Water content Determination

AOAC official method 934.04 at 105 °C for 24 h (AOAC, 2012) was used to determine the water content of fresh, dried, and dried powder samples of "Deglet Nour" dates.

Hot air drying (HAD)

Drying experiments were performed on a convective dryer with an automatic weighing system, at a temperature of 75°C and air velocity of 1.5ms^{-1}. This was a fully instrumented and controlled open-loop dryer with air temperature, relative humidity, and velocity as the adjustable parameters, as well as computerized data acquisition. Slices were dried until reaching approximately 8 g 100 g^{-1} db (dry basis) of water content.

Fresh dates and powder Analysis

Water activity

Water activity was measured by a pre-calibrated rotronic water activity meter (Hygro-Lab C1, Decagon Devices, Inc.) at 20°C ± 0.6.

Proximate analysis

Crude Fat, crude proteins, and ash content were determined by standard methods (AOAC, 2012). Ash was obtained after mineralization of the samples in a muffle furnace at 550 °C for 6 h. The percentage of crude protein was estimated with Kjeldahl Method by multiplying the total nitrogen content by a factor of 6.25. Fat content was measured by using a Soxhlet extraction apparatus with petroleum ether as a solvent for 6 h. All contents were expressed as percent of wet basis (g 100g^{-1} wb).

Sugar content determination

The concentration of total and reducing sugar content (RSC) was analyzed by Fehling's standard procedure (Kamal et al., 2023).

Non reducing sugar content (NRSC) was estimated following Eq. 2:

$$NRSC = (TS - RSC) * 0.96 \quad \textbf{Eq. 1.}$$

Total phenolic content (TPC)

Procurement of standard

Gallic acid was procured from Sigma-Aldrich. Standards were prepared in distilled water (to establish a calibration curve)(Hajji, Bellagha, & Allaf, 2020).

Extraction and estimation of TPC

The phenolic content of fresh and dried dates extracts was determined by the Folin-Ciocalteu reagent using gallic acid as the reference compound. 125 µl of the prepared extract was mixed with 500 µl of distilled water in a test tube and 125 µl of Folin-Ciocalteu reagent was added. The samples were mixed well and left to stand for 6 min before adding 125 µl of a 7% aqueous sodium carbonate solution. Water was added to adjust the final volume to 3000 µl. The resulting mixture was vortex-mixed and stored for 90 min in the dark at room temperature(Ben Haj Said, Najjaa, Neffati, & Bellagha, 2013; Hajji et al., 2020).

Absorbance was measured for both types of extracts at 765 nm with a JENWAY 6715UV/V spectrophotometer. Finally, absorbance values were compared with those of similarly prepared standards with known concentrations of Gallic acid. The total polyphenol content of DIC dehydrofrozen quince and strawberry samples was expressed in grams of Gallic acid equivalents per 100 g dry basis (g GAE/100 g db) for three replications, using the calibration curve with Gallic acid in the range 0-500 µg ml^{-1} (Roy, Takenaka, Isobe, & Tsushida, 2007).

Physicochemical properties of date powders

Color Evaluation

The color of fresh date fruit and date powder was determined using a colorimeter (Konica Minolta CR-410, Tokyo, Japan), with CIELAB (L*, a* and b*; with 2° observer at D65 illuminant). The instrument was calibrated with black and white standard tiles before each experiment. The results were expressed as Hunter color values of "*L*," "*a*," and "*b*," where the "*L*" value is used to denote lightness (+) and darkness (−), the "*a*" valueis used to denote redness (+) and greenness (−), and the "*b*" value is used to denote yellowness (+) and blueness (−). Each coordinate value was the mean of six measurements.

Whiteness Index, Whiteness indices (WI) are widely measured to yield numbers correlating closely with consumers' preferences for white colors. It mathematically combines lightness and yellow–blue into a single term. The WI represents the overall whiteness of food products that may indicate the extent of discoloration during the drying process, it is estimated as follows (Pathare, Opara, & Al-Said, 2013).

$$WI = 100 - \sqrt{(100 - L)^2 + a^2 + b^2} \qquad \textbf{Eq.2.}$$

Bulk density

The bulk density of powders was determined as mass per unit of volume(Shenoy et al., 2015) and was expressed as g per cm^3.

Wettability time

The wettability time was measured according to Raza et al. (2019) For this purpose,10 g sample was weighed in a petri plate and 250 ml water was taken in a beaker. A glass plate was placed on the beaker where sample was placed. The whole sample was introduced to water surface in 2.5 s. The duration was noted from the time that powder touched the water surface till all the particles became wet and denoted as wettability time.

Solubility index

One gram of sample from date powder was added to 100 ml ofdistilled water. Mixing was done on a magnetic stirrer, and after a compate mixing, it was allowed to stand at 37°C for 30 min. The solution (dispersed date powder) was subjected to centrifugation (3000 tr for 5 min). A 25 mL aliquot from the supernatant was dried at 105°C untilconstant weight is reheated in a laboratory drying oven UFE 400 (Memmert, Schwabach Germany). The solubility index (%) was calculated by taking the weight difference between the samples before and after oven drying (Caparino et al., 2012).

Hygroscopicity

One gram date powder was takenand spread on petri plate evenly to ensure high surface area. Petri plate was placed in a desiccator with higher relative humidity, that is, about72±2%. The weight gain was noted every 2 h in 24 h till a constantweight. This parameter showed powder capacity to carry highest amount of the moisture from the atmosphere. Weight gain was expressed as gram of water/gram of powder (Manickavasagan et al., 2015).

Statistical Analysis

All analytical determinations were performed at least in triplicate. Values were expressed as the mean ± standard deviation (± SD)

Statistical analyses were carried out using a statistical software program Statgraphics Plus software for Windows (1994, version4.1, Levallois-Perret, France). Data were subjected to analysis of variance using the general linear model option (Duncan's test) to determine significant differences between samples ($P < 0.05$).

RESULTS AND DISCUSSION

Physicochemical attributes

The determination of physicochemical and nutritional compositions of Deglet Nour dates is important for storage, processing, and commercialization purposes.

Water activity

The water activity is one of the important parameters to determine shelf life of food products. High water activity leads to shorter shelf life due to high free-water for biochemical and microbial degradations (Ahmed, Ramaswamy, & Khan, 2005). In this study, water activity of fresh dates, dried ones and the produced date powders was 0.7 ± 0001, 0.446±001 and 0.443±0.002, respectively. These values proved that dried products (dried dates and powder) might be considered microbiologically safe owing to the fact that dried food matrixes with water activity lower than 0.6 are in general stable, microbiologically and chemically (Ubairah et al., 2014).

Proximate analysis

In the present study, as presented in **Table 1**, protein and fat occur in small amounts in Deglet Nour date fruit variety. Fresh dates contained 2.05g per 100g (wb) and 0.56g per100g (wb) of protein and fat crudes, respectively. Ali, Waly, Essa, and Devarajan (2012) reported similar protein values i.e. 2.03-2.7 % for different date fruit varieties. Although dates are not a rich source of protein, they have been reported to contain high quantities of some of the essential amino acids compared to other fruits.

On the other hand, fat content of Deglet Nour date fruit variety was in close agreement to the range reported by Walid Al-Shahib and Richard J Marshall (2003)and Walid Al-shahib and Richard J. Marshall (2003)

In the current study, the ash content of the fruit pulp was at 1.54 g per 100 g (wb). These findings are compatible with other results on Deglet Nour fruit reported by Al-Farsi* and Lee (2008). Compared to the mineral content of some common fruits like grapes, apples, plums and oranges which have been reported to range from 1.89 to 3.13% (dry basis), Deglet Nour date fruits are equally good sources of various minerals as these more common fruits. The slight decrease amount of protein and fat after drying are mainly due to moisture loss.

Table 1. Means ± SD for proximate composition (% wb) of fresh Deglet Nour dates fruits and date powder

Date sample	Moisture content	Crude protein	Crude fat	Ash content	Total Sugar content	Reducing sugar content
RM*	30.20^{a}±0.007	2.05^{a}±0.06	0.56^{a}±0.02	1.54^{a}±0.005	69.77^{a}±1.44	31.04^{a}±0.72
Dried Slices	7.35^{b}±0.029	1.94^{a}±0.05	0.57^{a}±0.05	1.53^{b}±0.00	69.86^{a}±0.30	33.61^{b}±0.63
Powder	7.07^{b}±0.003	1.95^{a}±0.04	0.56^{a}±0.01	1.54^{b}±0.003	69.30^{a}±0.75	33.44^{b}±0.40

*RM: Raw Material

Sugar content

Sugars are the most important constituents of dates, making them a rich source of energy for the human system. Dates carbohydrates are present mainly as reducing sugars in the form of glucose, fructose, mannose and maltose and non-reducing sugars (primarily sucrose), as well as small amounts of polysaccharides such as cellulose and starch (Walid Al-shahib & Richard J. Marshall, 2003). The Tunisian Deglet Nour dates, known as “fingers of light” contain 69.77% of total sugars, which remains between 67 and 76% as reported by Mennouche, Boubekri, Chouicha, Bouchekima, and Bouguettaia (2017). The reducing sugar content in the samples was at 31.04%. This value is higher than the reported reducing sugar content which ranges between 19 and 23% of the dry Deglet Nour varieties (Kadri, Ben Mimoun, & Ben Salah, 2008; Zaid & Arias-Jimenez, 1999). Nevertheless, Baliga, Baliga, Kandathil, Bhat, and Vayalil (2011) found that the reducing sugar composition of dates ranged for glucose from 17.6 - 41.4%, and for fructose from 13.6-36.8%. The sucrose rate for these dates was found to be 36.79%, which is also within the accepted range 36.1% suggested by Reynes (1995). These results are in good agreement with the usual values of sugar contents measured on samples of hard dates characterized by a high amount of sucrose.

Determinations of total sugar content did not reveal any significant variations after hot air drying. Indeed, the total sugar content was relatively well conserved, suggesting an absence of sugar loss by oxidization. Furthermore, the distribution of sugars between sucrose and reducing sugars (glucose and fructose) was found to be closer to that found by Boubekri, Benmoussa, Courtois, and Bonazzi (2010) for dried dates and Ismail and Hassine (2021) for fresh dates.

Nevertheless, as it is shown in Table 1, it could be seen that a significant increase of reducing sugars has been depicted. This may be explained by a minor conversion of sucrose into reducing sugars (glucose and fructose) occurred during drying. Indeed, this increase in reducing sugars during drying might be due to hydrolysis of polysaccharides and inversion of non-reducing sugars to reducing sugars. The results are in accordance with those obtained by Naikwadi, Chavan, Pawar, and Amarowicz (2010) in dehydrated fig and El kadri, Ben Mimoun, and Ben Salah (2006) in dried dates.

Interestingly, these results showed that this richness in reducing sugars could contribute to the reduction of crystallization phenomenon and provide a good source of rapid energy(Abbès et al., 2011). In addition, reducing sugars and proteins interaction, during the Maillard reaction (a well-known non-enzymatic browning reaction), could produce colored or colorless reaction products, which can play an important role in the oxidation prevention (Yilmaz & Toledo, 2005). Thus, they could be incorporated in functional foods.

Total polyphenol content TPC

Interest in phenols has greatly increased in recent years because of their antioxidant ability, which is intimately associated with potential benefits for human health, especially for dates fruits. Indeed, phenolic contents of date fruits are symbol of its nutraceutical properties owing to the antioxidant potential (Hammouda et al., 2013).

The initial phenolic content was 0.62g AGE per 100g (db) for fresh dates. Levels of Deglet Nour variety dates phenols for the unprocessed fruit were in the range of those reported by other researchers (572 to 665 mg of GAE per 100 g dry basis)(Alam, Alhebsi, Ghnimi, & Kamal-Eldin, 2021; AlFaris et al., 2021; Shahdadi, Mirzaei, & Daraei Garmakhany, 2015).

In fact, these differences connected with fruits total polyphenols content were closely related to various factors such as variety, growing condition, maturity, season, geographic origin, fertilizers, soil type, amount of sunlight received and experimental conditions (storage, extraction) among others (Nuñez-Mancilla, Pérez-Won, Uribe, Vega-Gálvez, & Di Scala, 2013).

As described in **Figure 1**, the total phenolics yields of Deglet Nour fruits decrease significantly ($p = 0.02 < 0.05$) after convective hot air drying. Indeed, once these health-promoting compounds are exposed to drying conditions, TPC, which are usually stable within the original fruit matrix, are highly vulnerable to deterioration, due to their thermolabile characteristic (Oladzad, Fallah, Mahboubi, Afsham, & Taherzadeh, 2021; Vidinamo, Fawzia, & Karim, 2020).

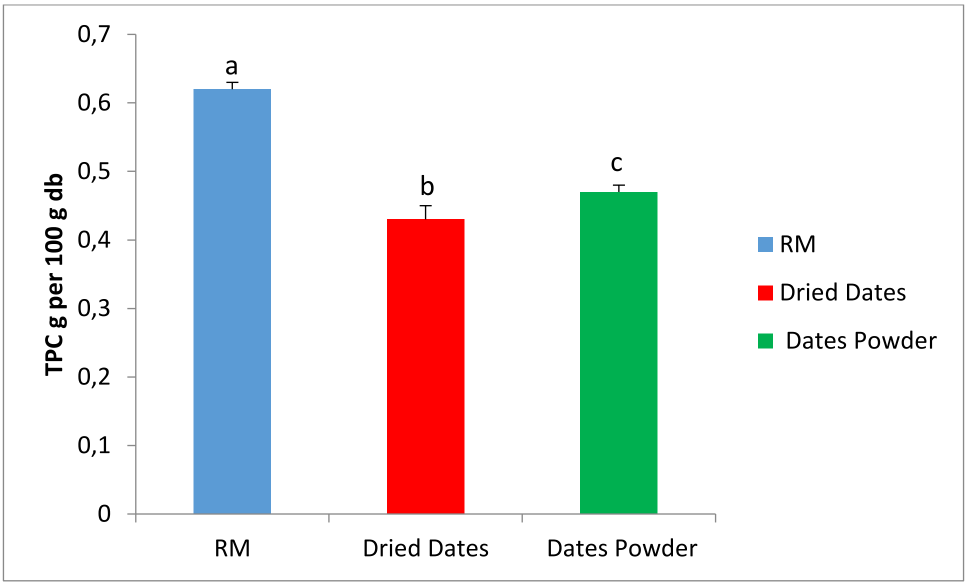

Figure 1. Impact of convective hot air drying on Deglet Nour TPC content samples

Interestingly, as shown in Figure 1, total polyphenol content increased in powder dates compared to dried samples. This may be explained by the mechanic action of grinding dates samples. Indeed, several studies have proved that grinding process improved antioxidant activity and scavenging capacity in powder, which was concomitant with the high total phenolics, flavonoids, carotenoids, and ascorbic acid contents, as observed for prickly pear seeds, wheat barn and green tea powders(Becker et al., 2017; Karam et al., 2016; Tian, Sun, Chen, Yang, & Wang, 2019).

Color change

Color is one of the most important quality attributes of foods as it is critical in the acceptance of food products by consumers.

In order to study the impact of drying process, color experiments were carried out on dates samples before and after drying procedure, in term of L, a, and b coordinates (**Fig.2**.).

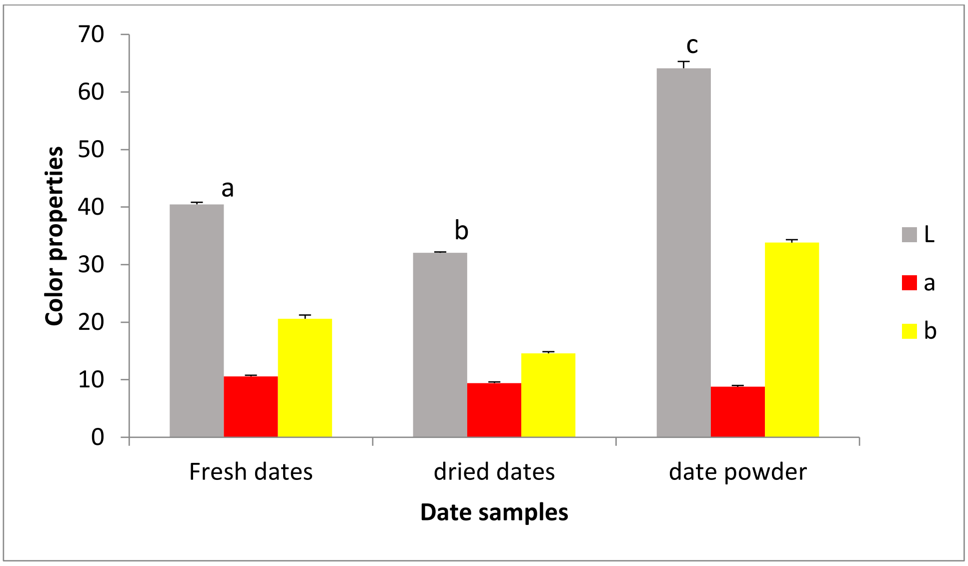

Figure 2. Color attributes for fresh, dried samples and powder of Tunisian Deglet Nour date

As illustrated in **Figure 2**. , the color attributes L, a, b, of fresh Deglet Nour dates were 40±0.35,10.60 ± 0.18, and 20.60± 0.65, respectively. After drying, a significant decrease trends in color attributes ($p=0.013<0.05$) has been detected. The loss of L (lightness) and b (yellowness) values gives the darker appearance of the dried dates. In fact, yellow color of dates is attributed to its concentration in carotenoids color pigment. During the drying process, date fruit color changes, caused by thermal treatment, may be strongly related to pigment degradation, especially the degradation of carotenoids and the formation of brown pigments by non-enzymatic reactions (Maillard reaction) and enzymatic reactions. Indeed, several researches on fruits and vegetables such as green pepper and pumpkins (Yildiz & İzli, 2019), dates (El-Sharnouby, Al-wesali, & Al-Shathri, 2007), quince(Hajji et al., 2020), and some other fruits and aromatic herbs (Calín-Sánchez et al., 2020) stated that the color changes were possibly due to the presence of heat sensitive reactions during processing. This involves degradation of sensitive pigment, browning of ascorbic acid and non-enzymatic Maillard reactions, non-enzymatic browning that was more prevalent at higher temperatures and generally due to Maillard reactions involving reducing sugars.

The increasing trend of color attribute of L value could be attributed to the decrease in moisture content such that date samples became more opaque and the reflection of light became stronger. Color change is a complex phenomenon and its kinetics depends on the moisture content of the product(Özkan, Kirca, & Cemeroğlu, 2003). This aspect has been well proved with Whiteness index values (WI) which increase from 36.08 for fresh dates to 49.87 for date powder. In fact, colors of powders are related to the fineness of the powder and depends much on the variety, growing conditions, dehydration and storage conditions, as well as on powder coarseness.

Bulk density

Bulk density is an indicator of the ease of reconstitution, packaging, transportation and storage of powdered food. As illustrated in Table 2. the bulk density of the obtained Deglet Nour date powders was of 0.689g per cm^3. This value is in the range of 0.66-0.79 g per cm^3 (Sablani et al., 2008), and higher than values recorded byFarahnaky et al. (2016) (0.58-0.66 g per cm^3), and by Manickavasagan et al. (2015) (0.39-0.50 g per cm3). Bulk density variation is possibly due to the difference in raw materials (date cultivars), and drying methods (Seerangurayar, Manickavasagan, Al-Ismaili, & Al-Mulla, 2017). Indeed, spray dried and drum dried dates powder showed lower density than oven dried ones as observed by Farahnaky et al. (2016), Manickavasagan et al. (2015) and Sablani et al. (2008), and this work respectively.

Wettability time

The wettability has a direct relationship with the dissolution of powder components. Wettability time recorded for Deglet Nour powder is 64.80s. In fact, powder with increased solubility takes less time to wet their surface, with consequently lower wetting time or wettability. Wettability of powders was affected by the ingredients; smaller molecules with higher water affinity took less time for wetting (Seth, Mishra, & Deka, 2017).

Hygroscopicity

Sablani et al. (2008) and Manickavasagan et al. (2015) found range of hygroscopicity in date powders from 0.03 to 0.08 g per gram. Hygroscopicity can vary from powder to powder depending upon their constituents (Shishir, Taip, Aziz, & Talib, 2014).

This parameter indicates powder's strong capacity to attract water molecules when in contact with the surrounding air. Mujumdar (2006) explained that drying of sugar-rich fruits requires high temperature and usually a dry to very low moisture thin sheet product. These drying conditions usually cause the product to be very hygroscopic. In addition, Dates with low molecular weight amorphous sugars are very hygroscopic in nature which may induce stickiness and caking phenomena. In food transformation sectors requiring free flowing powders, these phenomena may represent obstacles to the use of these powders. Solubility index

As illustrated in table 2, water solubility index of Deglet Nour powder, obtained through convective drying, was 81.60%. Researchers found the solubility index of date powders in the range of 67%-88% depending upon carrier agents, conditions of preparations, and type of raw material(Manickavasagan et al., 2015; Sablani et al., 2008). Oven dried date paste from the Shahani variety showed a solubility index of 91% (Muñoz-Tebar, Viuda-Martos, Lorenzo, Fernandez-Lopez, & Perez-Alvarez, 2023; Rafiee Darsangi, Badii, & Salehifar, 2020). The lower value observed in this work may be related to the low molecular weight molecules composition difference between Deglet Nour and Shahani varieties, among other. Water solubility with water content and bulk density are among the critical criteria for determining the reconstitution quality of the powder. Hence, solubility is the most reliable criterion to evaluate the behavior of powder in aqueous solution(Vivek et al., 2021).

Table 2. Functional properties of Deglet Nour powder date

	Bulk density (g per cm$^{3)}$	Wettability time (s)	Hygroscopicity%	**Solubility index %**
Date Powder	**0.689±0.004**	**64.80±1.483**	**8.758±0.036**	**81.608±0.720**

Conclusion

The Deglet Nour date powder had low water activity and moisture content and thus may be stored at for a long period. Furthermore, biochemical composition in terms of proteins, fat, ash, and total sugar content were well preserved at these air dryer conditions (70°C and 1.5m s^{-1}). Moreover, the solubility of date powder was higher than 80% making it an ideal ingredient to blend with any hydrophilic food. This higher solubility creates opportunities to use date powder as a potential substitute for added sugar at industry level. Thus, date powder production using HAD approach shows a great potential to add value to dates. Nevertheless, it is worth noting that one of the challenges of producing fruit powders in general and dates powder in particular, is to reduce stickiness during drying and safe handling and storage of the powder. The stickiness in powders is mainly due to the presence of low molecular weight sugars, such as fructose, glucose, sucrose and some organic acids in the fruit. The date is also comprised of these sugars. These sugars and organic acids are very hygroscopic in their amorphous state and have low glass transition temperature. Hence, to reduce stickiness powder further studies on the applied drying technology are needed to better monitor and optimize dates powder attributes.

Finally, it is well known that hot air drying is an energy-intensive process accounting for 10 to 25% of the total energy used in the food manufacturing process worldwide. In addition, it is equally important that the drying process be time-efficient. It is therefore not surprising that the quality indicators and process performance such as the energy efficiency have been identified as the key drivers of research and development in drying technology, and the main reasons for the growing interest in drying process intensification towards better process performance and greater quality attributes.

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