

Effect of inulin concentration on physicochemical properties and antioxidant activity of date powders obtained by hot-air tray dryer

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Abstract

In the present study, it was aimed to produce free-flowing date powders using various levels of inulin as drying-aid agent (10, 20, 30, 40, and 50%) through hot-air drying at 60°C for 24 h. Effects of different inulin ratios on physicochemical properties of date powders were investigated. This is the first report which evaluated the suitability of this prebiotic carbohydrate as drying-aid agent to fabricate date powders. Inulin addition yielded date powders with high flowability. On the other hand, contents of bioactive compounds including total phenolics, flavonoids, and condensed tannins of date powders decreased significantly ($P < 0.05$) as the inulin concentration increased from 10% to 50%. Accordingly, DPPH-radical inhibition capacities reduced in date powders containing higher levels of inulin. Furthermore, significant correlations were detected between bioactives contents and antioxidant activity of date samples. The results showed that free-flowing date powders with improved prebiotic content may be produced by incorporating inulin up to ratio of 50% and used as sugar substitute in different food products.

Keywords: Condensed tannin, Date palm, Drying, Flavonoid, Inulin

Introduction

Date palm (*Phoenix dactylifera* L.) is one of the most economically important plants grown in arid and semi-arid regions of the world (Mohamed, Fageer, Eltayeb and Mohamed Ahmed, 2014). Egypt, Saudi Arabia, Islamic Republic of Iran, Algeria, Iraq, Pakistan, Sudan, Oman, United Arab Emirates, and Tunisia are top date-producing countries (Food and Agriculture Organization of the United Nations, 2018). The fruit is composed of flesh (85–90%), and seed or pit (6–12%) along with the skin which covers fleshy part of fruit (Al-Orf et al., 2012). Carbohydrates (mainly glucose, fructose, and sucrose) constitute almost 70% of date fruit, while lower amounts of dietary fibre, minerals (potassium, calcium, magnesium, sodium, and phosphorus), protein, vitamins (B1, B3, B5, B6, and C), and phenolic compounds

(chelicidonic acid, ferulic acid, dicaffeoyl shikimic acid) are also present (Al-Farsi, Alsalvar, Morris, Baron and Shahidi, 2005; Al-Shahib and Marshall, 2002; Aslam, Khan and Khan, 2013; Baliga, Baliga, Kandathil, Bhat and Vayalil, 2011; Khallouki, Ricarte, Breuer and Owen, 2018; Nadeem et al., 2019; Sola Agboola and Lateef Adejumo, 2013). The moisture contents of fresh date fruits at Tamr stage (fully ripened) vary over a wide range (6.8–39.25%) depending on differences in cultivar, climate, harvesting period, and drying conditions (Hasnaoui, Elhoumaizi, Hakkou, Wathélet, and Sindic, 2010; Saafi, Trigui, Thabet, Hammami and Achour, 2008).

Date palm fruits may be processed into syrup, juice, molasses, paste, stick, jam, vinegar, and liquor, whereas dried fruits of some date cultivars can be directly ground into powder and used as sugar substitute (Ahmadnia and Sahari, 2008; Al-

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Manhal, 2004; Alsenaien et al., 2015; Amin, Abdel Fattah, El kalyoubi and El-Sharabasy, 2020; Barimah, Laryea and Okine, 2015; Dhankhar, Vashistha and Sharma, 2019; Fahloul, Abdedaim and Trystram, 2010; A Hariri, Ouis and Bouhadi 2017; Hariri, Ouis, Bouhadi and Benatouche, 2018; Mohamed, Fawzy, Mohamed, Mostafa, and Zeinab, 2016; Nadeem et al., 2017; Nwanekezi, Ekwe, and Agbugba, 2015; Senthil Kumar and Yaashikaa 2019; Tang, Shi and Aleid, 2013). Moreover, the powder is incorporated in yoghurt and different bakery products including biscuits and pancakes for fortification of products in terms of dietary fibre and antioxidants (Elsharnouby, Al-Eid and Al-Otaibi, 2017; Jrad, Oussaief, Bouhemda, Khorchani and El-Hatmi, 2019; Messaoudi and Fahloul, 2018; Sakr and Hussien, 2017; Sulieman, Masaad and Ali, 2011). However, in general, production of powder from various date cultivars by different drying techniques is challenging due to high amounts of low molecular weight sugars including glucose, fructose, and sucrose found in the fruit (Sablani, Shrestha and Bhandari, 2008). The low glass transition temperatures (T_g) of these carbohydrates lead to stickiness that prevents the conversion of fruit into free-flowing powder form (Bhandari, Datta and Howes, 1997). The most common approach to overcome this problem is the addition of carrier(s) with high molecular weight as drying-aid agent to increase the T_g of the mixture (Bhandari and Roos, 2012). In this context, maltodextrin, liquid glucose, gum arabic, tapioca starch, cashew tree gum, apple pectin, whey protein concentrate, sodium caseinate, and soy protein isolate have been used as drying-aid agent for the production of free-flowing powders from sugar-rich foods (Bazaria and Kumar, 2016; de Oliveira et al., 2009; Jayasundera, Adhikari, Howes and Aldred, 2011; Moreira et al., 2009; Muzaffar and Kumar, 2016; Papadakis, Gardeli and Tzia, 2006; Phoungchandang and Sertwasana, 2010; Sarabandi, Peighambardoust, Sadeghi Mahoonak and Samaei, 2018; Tonon, Brabet, Pallet, Brat and Hubinger, 2009; Vardin and Yasar, 2012).

Inulin is a kind of storage carbohydrate naturally occurring in chicory root, Jerusalem artichoke tubers, banana, asparagus, onion, and garlic (Huber and BeMiller, 2017). It is categorized as potent prebiotic along with fructooligosaccharides, human milk oligosaccharides, galactooligosaccharides, arabinoxylan oligosaccharides, isomaltooligosaccharides, xylooligosaccharides, pectin oligosaccharides, and pyrodextrins (Yang and Xu, 2018). Inulin cannot be hydrolysed by human digestive enzymes. Therefore, it is classified as “nondigestible oligosaccharide” (Flamm, Glinsmann, Kritchevsky, Prosky and Roberfroid, 2001). Furthermore, it reduces the digestion of digestible saccharides and decreases fasting blood sugar level (Roberfroid, 1993). Inulin has been used as drying-aid agent for powder production from *Araticum* (*Annona crassiflora*) pulp, blueberry waste extract, and coffee creamer (Botrel, Rodrigues, Souza and Fernandes, 2016; Hedayatnia and Mirhosseini, 2018; Waterhouse, Sun-Waterhouse, Su, Zhao and Zhao, 2017).

Until today, few attempts have been made to obtain date powder using maltodextrin (6, 10, 18, 19, and 20 dextrose equivalent), gum arabic, pectin, and whey protein concentrate through various drying techniques, mainly spray-drying (Dev,

Annamalai, Orsat, Raghavan and Ngadi, 2018; Farahnaky, Mansoori, Majzoubi and Badii, 2016; Manickavasagan et al., 2015; Moghbeli, Jafari, Maghsoudlou and Dehnad, 2019, 2020; Sablani et al., 2008; Seerangurayar, Manickavasagan, Al-Ismaili and Al-Mulla, 2017). However, to the best of our knowledge, there is no study on evaluation of inulin as drying-aid agent to obtain free-flowing date powder. Therefore, the aim of this study is to investigate the effects of different ratios of inulin (10, 20, 30, 40, and 50%) on some physicochemical properties (moisture, ash, and colour values, water activity, bulk and tapped density, flowability, solubility, contents of total phenolics, total flavonoids, total condensed tannins, and DPPH-radical scavenging activity) of date powders obtained through hot-air drying technique.

Materials and Methods

Materials and Chemicals

Date paste (Metro Chef) was bought from Metro Cash and Carry (İzmir, Turkey). Inulin and tricalcium phosphate were obtained from Smart Kimya Tic. ve Danışmanlık Ltd. Şti. (İzmir, Turkey). Aluminium (III) chloride, Folin-Ciocalteu's reagent, quercetin, potassium acetate, and methanol were purchased from Merck (Darmstadt, Germany). Distilled water was used to dilute date paste. 2,2-diphenyl-1-picrylhydrazyl (DPPH) reagent, sodium carbonate, and gallic acid were obtained from Fluka (Buchs, Switzerland). Vanillin, 6-hydroxy-2,5,7,8-tetramethylchromane-2-carboxylic acid (Trolox), and (+)-catechin hydrate were purchased from Sigma-Aldrich (St. Louis, MO, USA).

Methods

Drying by hot-air tray dryer

100 g of date paste was diluted with 100 mL distilled water and homogenized by using a hand-blender for 2 min (Tefal Activflow Power Soup 1000 Watt, İstanbul, Turkey). Then, certain amount of inulin (10, 20, 30, 40, or 50% of diluted date paste weight) and 7% of tricalcium phosphate were added to the mixture. Afterwards, the mixture was again homogenized for 3 min. Finally, the diluted date paste was spread on rectangular silicone baking mat and allowed to dry at hot-air tray dryer operating at 60°C for 24 h. Dried product was then ground into powder using a knife-mill (Grindomix GM-200, Retsch GmbH and Co, Haan, Germany).

Moisture and ash contents, water activity (a_w)

Moisture and ash contents of date powders were determined according to official methods of AOAC (AOAC, 2019). Water activity (a_w) was measured by using a digital a_w -meter (Rotronic Hygropalm, Bassersdorf, Swiss).

Bulk and tapped densities of powders

The bulk (ρ_B) and tapped (ρ_T) densities of date powders were determined according to the method of Chinta et al. (2009) which was then modified by Quispe-Condori, Saldaña, and Temelli (2011). Briefly, 3 g (m0) of date powders was weighed in a 25-mL graduated cylinder. Then, the powder residues sticking to the wall of cylinder was collected by hitting the cylinder gently. The volume (V0) was recorded and the bulk density was calculated using Eq. 1.

$$\rho_B(g/mL) = \frac{m_0}{V_0} \times 100 \quad (1)$$

For determination of tapped density, the powder was tapped until constant volume was achieved. The volume (V_n) was recorded and the tapped density was calculated using Eq.2.

$$\rho_T(g/mL) = \frac{m_0}{V_n} \times 100 \quad (2)$$

Powder flowability

The flowability of powders were evaluated using Carr's Index (CI) and Hausner Ratio (HR) (Quispe-Condori et al., 2011). The Eqs. 3-4 were used for determination of CI and HR values, respectively.

$$CI = \frac{\rho_T - \rho_B}{\rho_T} \times 100 \quad (3)$$

$$HR = \frac{\rho_T}{\rho_B} \quad (4)$$

Solubility of powders

Water solubility was determined according to the method of Cano-Chauca, Stringheta, Ramos and Cal-Vidal (2005). 1 g of date powders was homogenized with 100 mL of distilled water using a hand-blender for 5 min, followed by centrifugation at 3000 rpm for 5 min. Then, 25 mL of aliquot of supernatant was taken to pre-weighed petri plate and dried at 105°C till constant weight. Solubility was determined gravimetrically and expressed as “%”.

Colour of powders

Colour analysis was carried out with a bench-top colorimeter (Konica Minolta CR-5, Tokyo, Japan). CIE Lab scale was used, and the results were expressed as L^* (lightness), a^* (-a: greenness, +a: redness), and b^* (-b: blueness, +b: yellowness) values.

Total phenolic content (TPC)

Samples (2.5 g) were extracted with 50 mL of methanol/water (1:1, v/v), followed by vortexing for 1 min. After shaking of mixture using a shaking incubator (IKA Ks4000i, Staufen, Germany) at 20°C for 1 h, it was centrifuged at 4000 rpm for 20 min to obtain methanolic extract. TPCs of date paste and date powders were determined spectrophotometrically according to the method described by Singleton and Rossi (1965) and modified by Li et al. (2006). A gallic acid calibration curve ($R^2 = 0.9984$) was used and the results were expressed as mg gallic acid equivalent (GAE) per 100 g sample.

Total flavonoid content (TFC)

Methanolic extracts previously prepared were used for TFC determination. TFCs of date paste and date powders were estimated spectrophotometrically according to the method developed by Olumese and Oboh (2016) and modified by Aksoylyu Özbek, Çelik, Günç Ergönül, and Hepçimen (2020). A quercetin calibration curve ($R^2 = 0.9996$) was used and the

results were expressed as mg quercetin equivalent (QE) per 100 g sample.

Total condensed tannin content (CTC)

Methanolic extracts previously prepared were used for CTC estimation. CTCs of date paste and date powders were determined spectrophotometrically by using the vanillin/HCl method suggested by Broadhurst and Jones (1978). A (+)-catechin calibration curve ($R^2 = 0.9996$) was used and the results were expressed as mg catechin equivalent (CE) per 100 g sample.

DPPH-radical scavenging activity

Total antioxidant activities of date paste and date powders were determined using the DPPH-radical scavenging assay developed by Brand-Williams, Cuvelier, and Berset (1995) and modified by Singh, Chidambara Murthy, and Jayaprakasha (2002). A Trolox calibration curve was used ($R^2 = 0.9987$) and the results were expressed as millimoles Trolox equivalent (TE) per 100 g sample.

%inhibition was calculated using Eq.5.

$$\%Inhibition = \left[\frac{Abs_{control} - Abs_{sample}}{Abs_{control}} \right] \times 100 \quad (5)$$

Statistical analysis

A completely randomized design was used in this study. Inulin ratio was the only factor. All analyses were carried out in triplicate. The results were subjected to one-way analysis of variance (ANOVA) by using General Linear Model (PROC GLM) procedure in SAS (Version 8.2, Sas Institute, Inc., Cary, NC, USA). Differences were compared by Fisher's LSD test. All statistical analyses were conducted at a significance level (alpha) of 0.05. PROC CORR procedure of SAS were used to calculate Pearson correlation coefficients.

Results and Discussion

Influence of inulin amount on physicochemical characteristics of date powders

The physicochemical properties of date powders containing different ratios of inulin were presented in Table 1. Inulin concentration had statistically significant influence on moisture contents. They varied from 1.48% to 3.65% which were in accordance with the recommended maximum moisture levels (3–4%) for food powders (Klinkesorn, Sophanodora, Chinachoti, Decker, and McClements, 2006). Moisture contents increased as the ratio of inulin increased in the formulation. This increase may be explained by the hindrance of water diffusion to the drying air due to high affinity of this carbohydrate for water (Botrel et al., 2016). Additionally, a rapid crust formation caused by inulin may also restrict the water diffusion and evaporation from date paste (Fernandes, Borges, and Botrel, 2014). A similar relationship between inulin concentration and moisture content of beetroot juice powder was previously detected by Carmo et al. (2019). Our results are comparable with the findings of Moghbeli et al. (2019) who reported moisture contents ranging from 1.48% to 3.45% for spray-dried date powders including pectin-whey protein complexes. Similar moisture contents were also

reported by Raza et al. (2019) for maltodextrin/gum arabic mixture-containing date powders obtained through oven-drying at 60°C (1.55–2.59%) and spray-drying (1.47–2.55%). On the other side, our findings are lower than those of Botrel

et al. (2016) (5.06–6.78%) who investigated the utilization of different ratios of inulin (20% or 30%) to produce araticum (*Annona crassiflora*) pulp powder using cabinet convective air dryer at 70, 80 or 90°C.

Table 1. Physicochemical properties of date palm fruit powders containing different ratios of inulin

| Characteristic | 10% IN | 20% IN | 30% IN | 40% IN | 50% IN |
|------------------------|---------------------------|---------------------------|---------------------------|---------------------------|---------------------------|
| Moisture, g/100g | 1.48 ± 0.11 ^e | 3.23 ± 0.02 ^d | 3.40 ± 0.01 ^c | 3.50 ± 0.05 ^b | 3.65 ± 0.01 ^a |
| Ash, g/100g (dw) | 13.99 ± 0.04 ^a | 11.80 ± 0.01 ^b | 10.25 ± 0.03 ^c | 8.89 ± 0.08 ^d | 8.09 ± 0.02 ^e |
| Water activity | 0.16 ± 0.01 ^d | 0.21 ± 0.001 ^c | 0.24 ± 0.01 ^b | 0.25 ± 0.01 ^b | 0.27 ± 0.01 ^a |
| Bulk density, g/mL | 0.76 ± 0.01 ^a | 0.77 ± 0.01 ^a | 0.79 ± 0.01 ^a | 0.77 ± 0.01 ^a | 0.73 ± 0.03 ^a |
| Tapped density, g/mL | 0.87 ± 0.01 ^a | 0.89 ± 0.01 ^a | 0.91 ± 0.01 ^a | 0.88 ± 0.01 ^a | 0.83 ± 0.04 ^a |
| CI | 12.50 ± 0.01 ^a | 12.50 ± 0.01 ^a | 12.50 ± 0.01 ^a | 12.50 ± 0.01 ^a | 11.81 ± 0.69 ^a |
| HR | 1.14 ± 0.01 ^a | 1.14 ± 0.01 ^a | 1.14 ± 0.01 ^a | 1.14 ± 0.01 ^a | 1.14 ± 0.01 ^a |
| Solubility in water, % | 12.53 ± 0.20 ^a | 11.68 ± 1.07 ^a | 11.29 ± 0.47 ^a | 11.65 ± 0.39 ^a | 12.62 ± 0.15 ^a |
| <i>L</i> * | 57 ± 1.05 ^d | 61.04 ± 0.65 ^c | 63.49 ± 0.86 ^b | 67.44 ± 0.49 ^a | 67.55 ± 0.26 ^a |
| <i>a</i> * | 9.23 ± 0.40 ^a | 8.15 ± 0.16 ^b | 7.34 ± 0.14 ^c | 6.32 ± 0.12 ^d | 6.19 ± 0.04 ^d |
| <i>b</i> * | 22.25 ± 0.44 ^a | 21.91 ± 0.23 ^a | 21.42 ± 0.15 ^b | 20.30 ± 0.09 ^c | 19.28 ± 0.06 ^d |

CI: Carr's Index, HR: Hausner Ratio, IN: inulin

Mean values within a row with different superscripts are significantly different (P<0.05).

Inulin-containing date powders had ash contents of between 8.09% and 13.09% (Table 1). Comparatively low ash contents in date powders (4.73–5.76%) with maltodextrin/gum arabic mixture were declared by Raza et al. (2019). This remarkable difference may be the result of various drying-aid agent concentrations used in these studies and different accumulation of minerals in fruits due to cultivars, climate, growing location, and cultivation practices (organic/conventional) (Boussaa et al., 2020; Kapoulas, Milenković and Mirecki, 2013; Marzouk and Kassem 2011). The amount of inulin increased, the ash contents of date powders significantly decreased in our study. As pure inulin used in our study did not include components comprising ash content, this finding is not surprising. A similar observation was previously reported by Caliskan and Dirim (2013) and Caliskan and Dirim (2016) who examined the effects of different ratios of maltodextrin on the properties of sumac extract powder obtained through spray-drying or freeze-drying.

As shown in Table 1, a_w was significantly influenced by inulin concentration. The a_w values of date powders increased from 0.16 to 0.27 as the ratio of inulin increased from 10% to 50%. In general, a_w values between 0.2 and 0.3 correspond to a suitable moisture content region which ensures maximum shelf-life in dehydrated foods (Labuza and Altunakar, 2007). Similar a_w values were reported for spray-dried date (0.23–0.28), watermelon (0.20–0.29), açai (0.19–0.25), and pitaya powders (0.30–0.35) accompanied with maltodextrin with different dextrose equivalents, gum arabic or tapioca starch as

drying-aid agent (Manickavasagan et al., 2015; Quek, Chok and Swedlund, 2007; Tonon et al., 2009; Tze et al., 2012).

The bulk and tapped density values of the date powders ranged between 0.73–0.79 g/mL and 0.83–0.91 g/mL, respectively (Table 1). The inulin concentration did not affect nor bulk density neither tapped density values significantly. Bulk density of the powders with inulin were higher than foam-mat freeze dried date powders containing different ratios of maltodextrin (0.60–0.68 g/cm³) and gum arabic (0.56–0.70 g/cm³) and spray-dried instant date powders with maltodextrin (0.41–0.51 g/cm³) (Nortuy, Suthapakti and Utama-ang, 2018; Seerangurayar et al., 2017). Comparable bulk density values (0.66–0.79 g/cm³) were reported for oven dried maltodextrin-containing date powders (Sablani et al., 2008). The density values of the powders considerably increased (13.70–15.58%) after tapping. This increase may lead to poor flowability (Onwulata, Konstance and Holsinger, 1996). Inulin-containing date powders had higher tapped density values than spray-dried instant date powders (0.54–0.71 g/cm³), while were comparable to foam-mat freeze dried date powders (0.80–0.90 g/cm³ for maltodextrin, and 0.75–0.87 g/cm³ for gum arabic-added powders) (Nortuy et al., 2018; Seerangurayar et al., 2017).

The flowability of any food powder is an essential parameter for various processes including storage, transportation, formulation and mixing, compression and packaging (Teunou, Fitzpatrick and Synnott, 1999). The flowability and cohesiveness of the powders were evaluated through CI and

HR values, respectively. Incorporation level of inulin did not have statistically significant effect on CI and HR. As shown in Table 1, except date powder including 50% inulin (11.81), all powders had a CI value of 12.50. On the other hand, all powders had the same HR value (1.14). Based on CI and HR values, the flowability of powders can be classified as excellent (CI≤10, HR: 1.00–1.11), good (CI: 11–15, HR: 1.12–1.18), fair (CI: 16–20, HR: 1.19–1.25), passable (CI: 21–25, HR: 1.26–1.34), poor (CI: 26–31, HR: 1.35–1.45), very poor (CI: 32–37, HR: 1.46–1.59), and awful (CI>38, HR: >1.60) (Quispe-Condori et al., 2011). Our results showed that inulin-containing date powders had good flowing properties. Inulin as a drying-aid agent yielded free-flowing date powders by increasing Tg and preventing bridging (Juliano and Barbosa-Cánovas, 2010). However, instant date powders containing maltodextrin exhibited poor flowability (CI:20.37–36.06, HR: 1.26–1.57) (Nortuy et al., 2018), while Seerangurayar et al. (2017) reported good flowability for 50% gum arabic containing date powders and fair flowability for freeze dried date powders with 40% or 50% maltodextrin, or 40% gum arabic and intermediate cohesiveness for all samples. In conclusion, inulin enables to produce fruit powders with improved flowability and therefore, it may be suggested as a promising alternative to common drying aids such as maltodextrin and gum arabic.

Solubility values of date powders ranged from 11.29% to 12.62% (Table 1) and inulin ratio did not significantly affected this characteristic. In other studies, highly soluble (66–88.60%) date powders including maltodextrin or gum arabic and maltodextrin/gum arabic mixture were produced through spray-drying or oven-drying techniques (Manickavasagan et al., 2015; Raza et al., 2019). Date powders obtained in the current study had considerably lower water solubility than spray-dried guava (85.87–95.43%), cast-tape- or spray-dried mango powders (77.22–79.17%) including maltodextrin as drying-aid agent (Patil, Chauhan and Singh, 2014; Zotarelli, Durigon, da Silva, Hubinger and Laurindo, 2020). Poor solubility of date powders may be attributed to drying technique (hot-air tray drying) used in our study which leads to a dense structure. This structure reduces the access of water through powder (Caliskan and Dirim, 2016). Furthermore, the poorly water soluble nature of drying-aid agent (inulin) at room temperature and high amounts of insoluble dietary fibres and non-starch polysaccharides as well as other lipophilic substances such as carotenoids found in date palm fruit may also contribute to the limited water solubility of date powders (Elleuch et al., 2008; Kha, Nguyen and Roach, 2010; Kim, Faqih and Wang, 2001; Ozdikiclerler, Dirim and Pazir, 2014; Vinita and Punia, 2016).

The colour results summarized in Table 1 show that inulin addition led to increase in L* and decrease in a* and b* values of date powders. These changes were found to be statistically significant. White colour of powdered inulin increased the lightness (L*) of date powders. On the other side, inulin concentration-dependent decreases observed in a* and b* values may be attributed to the dilution of date palm fruit and underestimated perception of date palm fruit pigments, mainly carotenoids as previously stated by Chong and Wong (2017). Similarly, (Michalska-Ciechanowska, Majerska,

Brzezowska, Wojdyło and Figiel, 2020) reported increases in L* and decreases in a* and b* values of freeze- or spray-dried cranberry powders depending on increasing inulin levels (15, 25, and 35%). Except a* values, our results (L* and b*) were in accordance with the findings of (Sablani et al., 2008) who recorded higher a* values in oven-dried date powders as the proportion of maltodextrin increased in the formulation.

Influence of inulin amount on bioactives contents of date powders

Total phenolic content

Chelidonic acid, t-ferulic acid, dicaffeoyl shikimic acid derivatives (1, 2, and 3), taxifolin-3-O-rhamnoside, gallic acid, p-coumaric acid, caffeic acid, sinapic acid, syringic acid, ellagic acid, catechin, quercetrin, rutin, isoquercetrin, and quercetin were identified as the main phenolic compounds of date palm fruits (Benmeddour, Mehinagic, Meurlay and Louaileche, 2013; Nizar Chaira et al., 2009; Hamad, 2014; Khallouki et al., 2018; Mansouri, Embarek, Kokkalou and Kefalas, 2005). TPCs of fresh date paste and date powders containing different amounts of inulin were illustrated in Fig.1. The fresh date paste had a TPC of 550.85 mg GAE/100g. Mansouri et al. (2005), Biglari, AlKarkhi and Easa (2008), Chaira, Mrabet and Ferchichi (2009), Saafi, El Arem, Issaoui, Hammami and Achour (2009), Nadeem, Salim-ur-Rehman, Anjum and Bhatti (2011), Al Juhaimi, Ghafoor and Özcan (2014), Louaileche, Hammiche and Hamoudi (2015), Ali Haimoud, Allem and Merouane (2016), Matloob and Balakit (2016) and Nadeem et al. (2019) reported lower TPCs (2.49–8.36 mg GAE/100g, 2.89–4.82 mg GAE/100g (dw), 3.88–9.71 mg GAE/100g, 209.42–447.73 mg GAE/100g, 140.67–296.67 mg GAE/100g, 94–198 mg GAE/100g, 127.97–334.58 mg GAE/100g, 2.06–6.53 mg GAE/100g (dw), 147.60–475.50 mg GAE/100g, and 142.52–298.02 mg GAE/100g, respectively) in ripe and fresh date palm fruits grown in Algeria, Iran, Pakistan, Saudi Arabia, Iraq, and Tunisia. On the other side, comparable TPCs were reported for the fresh fruits of Bahraini (276–342 mg GAE/100g in Tamr stage), Mauritanian (405.50–661.10 mg GAE/100g (dw)), Iranian (298–845 mg GAE/100g (dw)), Moroccan (331.86–537.07 mg GAE/100g (dw)) date varieties (Allaith, 2008; Bouhlali et al., 2017; Mohamed Lemine et al., 2014; Sadeghi, Valizadeh and Shermeh, 2015). Previously, Al-Turki, Shahba and Stushnoff (2010) showed the effects of growing location and variety on TPC of date palm fruits. In addition to these, very different TPCs of date fruits may be explained by climate conditions, and pre/post-harvest factors that greatly influence accumulation of these secondary metabolites in plant and the polarity of solvent used for extraction of phenolic compounds (Kozłowska, Gruczyńska, Ścibisz and Rudzińska, 2016; Mnari, Harzallah, Amri, Dhaou Aguir and Hammami, 2016). A comprehensive research demonstrated that date fruit had the highest TPC (585.52 mg GAE/100g) among 62 different fruits including sweetsop (405.41 mg GAE/100g), guava (194.11 mg GAE/100g), pomegranate (146.94 mg GAE/100g), cherry (114.56 mg GAE/100g), persimmon (112.09 mg GAE/100g), plum (102.43 mg GAE/100g), and pineapple (94.04 mg GAE/100g) (Fu et al., 2011).

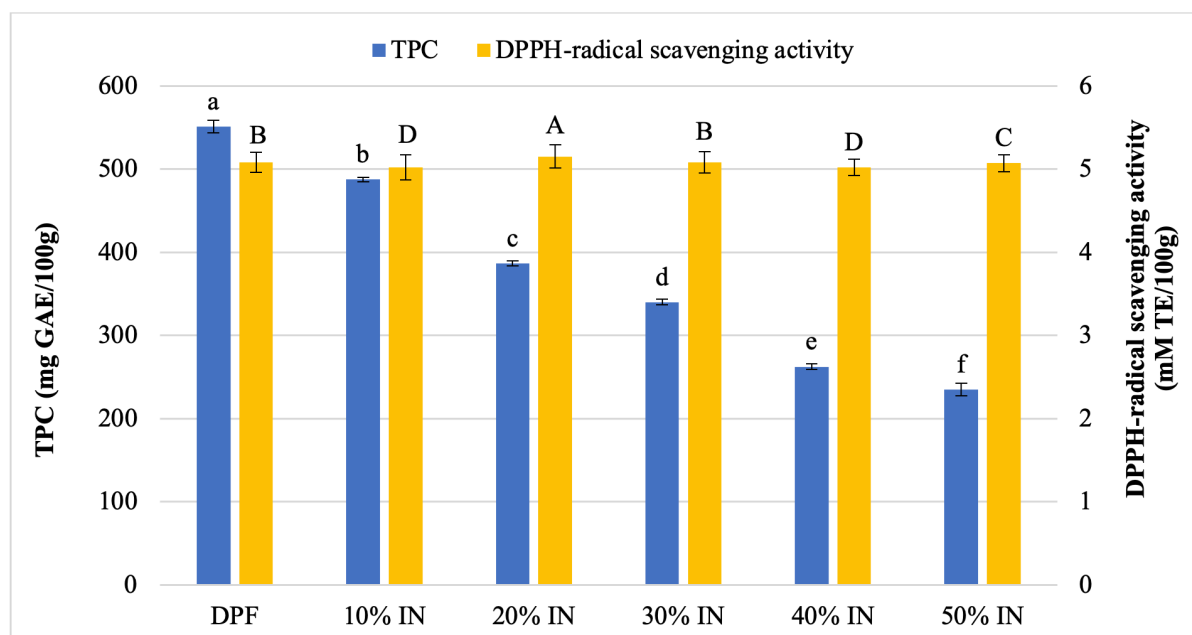


Figure 1. Total phenolics contents and DPPH-radical scavenging activities of fresh date palm paste and date powders containing different ratios of inulin. Different lowercase letters indicate statistically ($P < 0.05$) significant differences between TPCs of the samples. Different uppercase letters indicate statistically ($P < 0.05$) significant differences between DPPH-radical scavenging activities of the samples. DPF: date palm fruit, GAE: gallic acid equivalent, IN: inulin, TE: Trolox equivalent, TPC: total phenolic content

Increasing amount of inulin significantly reduced TPCs of date powders. TPC of date powder including 10% inulin was 487.42 mg GAE/100g, while that of date powder with 50% inulin decreased to 234.75 mg GAE/100g. Seerangurayar et al. (2017) reported that TPCs of foam-mat freeze-dried date powders were in the range of 84–173 mg GAE/100g and 85–197 mg GAE/100g including 40%–50% maltodextrin or gum arabic, respectively. Another study showed that TPCs of spray-dried date powders with whey protein-pectin complexes varied from 345 mg ascorbic acid equivalent (AAE)/100g to 701.25 mg AAE/100g. However, it should be kept in mind that different drying techniques cause changes in TPC of fruit powders in different ways. For instance, Raza et al. (2019) stated that oven-drying at 60°C for 48h yielded maltodextrin/gum arabic mixture-containing date powder with higher TPC (77.24–438.94 mg GAE/100g) than spray-drying technique (76.92–411.77 mg GAE/100g). The sensitivity of date palm fruit phenolics to high drying temperatures was also proved by Shahdadi, Mirzaei, Daraei Garmakhany, Mirzaei and Ghafari Khosroshahi (2013), Manickavasagan et al. (2015), Shahdadi, Mirzaei and Daraei Garmakhany (2015), İzli (2016) and Moghbeli et al. (2020). In accordance with our findings, Seerangurayar et al. (2017) detected significant decreases in TPCs of date powders as the concentrations of drying-aid agents increased. Higher levels of drying-aid agents lead to the dilution of raw material (date paste) in the formulation which decreases the amounts of phenolics to be extracted as previously reported by Caliskan and Dirim (2013) and Mishra, Mishra, and Mahanta (2014).

Total flavonoid content

Quercetin, luteolin, apigenin, isoquercetrin, rutin, catechin,

isorhamnetin, and chrysoeriol are the flavonoids which have been identified in fresh date fruit extracts up to now by some researchers (Amira et al., 2012; Farag, Mohsen, Heinke and Wessjohann, 2014; Hamad et al., 2015). As shown in Table 2, TFC of fresh date paste was 37.59 mg QE/100g. Wide ranges of TFCs for ripe date fruits grown in Tunisia (6.28–54.46 mg QE/100g; 300–2609 mg QE/100g (dw)), Algeria (15.22–299.74 mg QE/100g (dw); 15.89–40.78 mg QE/100g), and Mauritania (39.50–112.50 mg QE/100g (dw)) were reported by several researchers (Benmeddour et al., 2013; Chaira et al., 2009; Lekbir et al., 2015; Masmoudi-Allouche et al., 2016; Mohamed Lemine et al., 2014). Our finding partially agrees with these results. Geographical, climatic and genetic factors which control accumulation of secondary metabolites as well as use of different solvents to extract bioactive compounds may lead to considerable variations in TFCs of plant materials (Kozłowska et al., 2016; Mnari et al., 2016). TFC of fresh palm fruit is higher than mango (5.30–28.10 mg QE/100g), plum (5.40–20.43 mg QE/100g), sweet (14.80 mg QE/100g) and acid (10.30 mg QE/100g) starfruits, passion fruit (12.10 mg QE/100g), pink (11 mg QE/100g) and white (20.90 mg QE/100g) guavas, pineapple (2–15.90 mg QE/100g), banana (5.60–9.90 mg QE/100g), avocado (2.10 mg QE/100g), and papaya (1.50–1.70 mg QE/100g), whereas lower than mangosteen (257.10 mg QE/100g), strawberry (207.60 mg QE/100g), red Chinese guava (71.20 mg QE/100g) and litchi (53.30 mg QE/100g) (Barcelo et al., 2016; Cosmulescu, Trandafir, Nour and Botu, 2015; Luximon-Ramma, Bahorun and Crozier, 2003; Septembre-Malaterre, Stanislas, Douraguia and Gonthier, 2016).



Table 2. Contents of total flavonoids, condensed tannins and DPPH-radical inhibition values of fresh date pulp and date powders containing different ratios of inulin.

| | DPF | 10% IN | 20% IN | 30% IN | 40% IN | 50%IN |
|----------------------------|----------------------------|---------------------------|---------------------------|---------------------------|---------------------------|---------------------------|
| TFC, mg QE/100g | 37.59 ± 0.86 ^a | 29.91 ± 0.01 ^b | 25.85 ± 0.60 ^c | 20.49 ± 0.25 ^d | 17.82 ± 0.82 ^e | 14.21 ± 0.01 ^f |
| CTC, mg CE/100g | 101.62 ± 0.41 ^a | 81.18 ± 1.20 ^b | 76.15 ± 1.88 ^c | 62.06 ± 1.58 ^d | 47.51 ± 0.71 ^e | 36.23 ± 0.83 ^f |
| DPPH-radical inhibition, % | 92.03 ± 1.69 ^a | 89.55 ± 3.11 ^a | 89.55 ± 0.26 ^a | 81.58 ± 0.13 ^b | 69.75 ± 0.01 ^c | 61.14 ± 1.30 ^d |

CE: Catechin equivalent, CTC: condensed tannin content, DPF: date palm fruit, IN: inulin, QE: quercetin equivalent, TFC: total flavonoid content. Mean values within a row with different superscripts are significantly different (P<0.05).

TFCs of inulin-containing date powders ranged from 14.21 mg QE/100g to 29.91 mg QE/100g. Due to the reasons explained for TPCs of date powders above, the TFCs also decreased significantly as the ratio of inulin increased in the formulation. Therefore, less flavonoids were extracted from the date powders containing higher levels of inulin as a result of dilution effect. However, this result contrasts with the findings of Alcantara Marte, Alcantara Marte, Tejada and Ros Berruezo (2018) who reported higher TFCs for spray-dried lemon juice powders including increased levels of pulverized mesocarp of *Citrus paradisi* Macf. as a drying aid due to its ability to protect flavonoids from degradative reactions. Similarly, improved betalains and anthocyanin retention in spray-dried beetroot juice concentrate powder and Jamun pulp powder were obtained by increasing the ratio of drying-aid agents due to preservation of these bioactive compounds effectively against oxidation and/or degradation (Bazaria and Kumar, 2016; Singh, Paswan and Rai, 2019). Our finding may suggest that inulin did not protect the date flavonoids against undesirable reactions during hot air-drying process at 60°C.

Condensed tannin content

Immature date fruit is rich in soluble tannins that give astringency to the fruit. These compounds are converted to their insoluble forms which are known as “condensed tannins (procyanidin polymers)” as the fruit ripens. Therefore, mature date palm fruits are less astringent and more palatable than immature ones (Amira et al., 2012; Hussain, Farooq and Syed, 2020; Myhara, Al-Alawi, Karkalas and Taylor, 2000). Condensed tannins constitute almost 80% of TPC of date fruits at commercial maturity (Tamr stage) (Hammouda, Chérif, Trabelsi-Ayadi, Baron and Guyot, 2013). CTC of fresh date paste was 101.62 mg CE/100g in our study (Table 2). Our finding partially agrees with the CTCs of Algerian (74.22–394.27 mg CE/100g), Tunisian (41.85–102.37 mg CE/100g), and Moroccan (57.56–92.14 mg CE/100g (dw)) date fruits at Tamr stage (Arem et al., 2013; Benmeddour et al., 2013; Bouhlali et al., 2016; Souli et al., 2018). Total amount of condensed tannins in date palm fruit is lower than hawthorn (920–4110 mg CE/100g) and higher than strawberry (2.93–31.55 mg CE/100g), rosehip (2.02–2.34 mg CE/100g), blackberry (14.28–16.59 mg CE/100g), and blackthorn (9.53–10.50 mg CE/100g) (Dou, Leng, Li, Zeng and Sun, 2015; Turker, Kizilkaya, Cevik and Gonuz, 2012).

In our study, the lowest CTC (36.23 mg CE/100g) was

obtained from date powder including 50% inulin, whereas 10% inulin addition yielded the date powder with the highest CTC (81.18 mg CE/100g). Increased inulin concentration significantly reduced CTCs of date powders. As explained in TPCs and TFCs of date powders, lower CTCs were determined in samples with more inulin due to dilution of raw material (date paste). Similar observations were reported for lycopene and carotenoid contents of spray or freeze-dried watermelon powders as the drying-aid agent concentration increased in the formulation (Oberoi and Sogi, 2015).

DPPH-radical scavenging activity

DPPH-radical scavenging activities of fresh date paste and date powders were illustrated in Fig.1. DPPH-radical scavenging activity of fresh date paste was 5.08 mM TE/100g, while those of hot air-dried date powders ranged between 5.02 mM TE/100g and 5.15 mM TE/100g. Unlike our study, comparatively low DPPH-radical scavenging activities were determined in fresh date palm fruits grown in U.S.A., Saudi Arabia, Iran and Mauritania (Al-Jasass, Siddiq and Sogi, 2015; Al-Turki, 2008; Al-Turki et al., 2010; Hemmateenejad, Karimi, Javidnia, Parish and Khademi, 2015; Mohamed Lemine et al., 2014). Similarly, İzli (2016) reported lower DPPH-radical scavenging activity (0.14 mM TE/100g (dw)) for methanol/water extracts of fresh date fruits sold in Turkey.

In addition to DPPH-radical scavenging capacities of the date samples in terms of Trolox equivalent, DPPH-radical inhibition ability values were also determined. As shown in Table 2, while the fresh date paste had the highest DPPH-radical inhibition ability (92.03%), date powders with lower antioxidant capacities depending on increasing levels of inulin were obtained in our study. Inulin addition more than 20% led to significant reductions in antioxidant activities of date powders compared to fresh date paste. The methanol/water (50/50, v/v) extract of fresh date sample used in the present study has higher DPPH-radical inhibition capacity than those of methanol/water (80/20, v/v) extracts of date palm fruits (69.50–72.70% in Tamr stage, and 62.50–65% in Rutab stage) grown in Sultanate of Oman, methanol/water (50/50, v/v) and acetone/water (70/30, v/v) extracts of Tunisian date palm fruits (57.54–90.12%, 83.57–88.24% in Tamr stage, respectively (Kchaou, Abbès, Blecker, Attia and Besbes, 2013; Singh, Guizani, Essa, Hakkim and Rahman, 2012). The differences observed for antioxidant activities of date palm fruits in various studies may arise from various factors such

as growing location and variety which influence accumulation of secondary metabolites that are responsible for antioxidant activity as well as the composition and polarity of extraction solvents (Al-Turki et al., 2010; Kchaou et al., 2013).

Correlations between bioactives contents and DPPH-radical inhibition ability of date samples

Table 3. Pearson correlation coefficients between total phenolics, total flavonoids, total condensed tannins, and %DPPH-radical inhibition values of date palm samples.

| | TPC | TFC | CTC | %Inhibition |
|-------------|-------|------------------|------------------|------------------|
| TPC | 1.000 | 0.9840 <.0001 | 0.9763 <.0001 | 0.8958 <.0001 |
| TFC | | 1.000 | 0.9867 <.0001 | 0.8846 0.0001 |
| CTC | | | 1.000 | 0.9405 <.0001 |
| %Inhibition | | | | 1.000 |

TPC: Total phenolic content, TFC: total flavonoid content, CTC: condensed tannin content

Extremely high positive correlation coefficients suggest that phenolics, flavonoids and condensed tannins contributed to the DPPH-radical inhibition capacity of date samples to a great extent. The positive relationships between catechin, rutin (dominant flavonoids of date palm fruits) and oxidation inhibition capacity of aqueous extracts of date palm fruits were also approved previously (Saleh, Tawfik and Abu-Tarboush, 2011). Therefore, the very high positive correlation coefficient determined for TFC/DPPH-radical inhibition capacity ($r = 0.8846$) in our study is not surprising. In accordance with our findings, Odeh et al. (2014) and Hemmateenejad et al. (2015) detected very strong positive correlations for TFC/antioxidant activity ($r = 0.935-0.961$) and TPC/antioxidant activity ($r = 0.97$) of Palestinian and Iranian date palm fruits, respectively. However, our findings contrast with the results of Lekbir et al. (2015) who reported very weak correlations between TFC/DPPH-radical scavenging capacity ($r = 0.24$) and TFC/TPC ($r = 0.342$) of Algerian date palm fruits. Additionally, positive relationships between TPCs, TFCs, and CTCs of date samples were also confirmed by high correlation coefficients. The great contribution of TFC to TPC of date palm fruits ($r = 0.99$) was also declared by Abbas, Foroogh, Liong and Azhar, (2008) and Biglari et al. (2008).

Conclusion

The results obtained in this study showed that inulin can be effectively used as a drying-aid agent to produce free-flowing date powders. Stickiness problem occurring during hot air drying of date fruit as a result of its high simple sugar content was completely eliminated by means of inulin addition. This prebiotic carbohydrate may serve as a convenient alternative to common carbohydrate-based drying-aid agents like maltodextrin, gum arabic, and pectin which have been used for fabrication of free-flowing date powders up to now. Inulin concentration (10–50%) considerably affected physicochemical properties including moisture and ash contents, water activity, and colour of date powders. On the other hand, bulk and tapped density values, solubility in water as well as flowability

A correlation analysis was carried out to determine the relationships between bioactives contents including total phenolics, flavonoids and condensed tannins and DPPH-radical inhibition ability of fresh date pulp and date powders (Table 3).

were not influenced by different ratios of inulin. As a general trend, bioactives contents (TPC, TFC, and CTC) of date powders decreased as the inulin amount in the formulation increased. Therefore, date powders with lower DPPH-radical scavenging activities depending on increasing ratios of inulin were obtained in our study. However, date powders including inulin still had quite high DPPH-radical inhibition capacities (61.14–89.95%). In conclusion, free-flowing date powders can be successfully produced by incorporation of this prebiotic carbohydrate (inulin) up to 50% ratio into formulation. Date powders containing inulin may be used as sugar substitute in different food products. Our further study will focus on the determination of glass transition temperatures of these powders to determine the suitable storage temperatures in order to ensure powder stability.

Compliance with Ethical Standards

Conflict of interest

The authors declared that for this research article, they have no actual, potential or perceived conflict of interest.

Author contribution

Zeynep Aksoylyu Özbek carried out the data curation, investigation, formal analysis, writing-original draft. Kıvılcım Çelik contributed the investigation, formal analysis, visualization. Pelin Günç Ergönül contributed the supervision, writing-review and editing.

All the authors read and approved the final manuscript. All the authors verify that the Text, Figures, and Tables are original and that they have not been published before.

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Data availability

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