

## DEVELOPMENT AND OPTIMIZATION OF A SYSTEM FOR THE EXTRACTION, FILTRATION, AND CONCENTRATION OF DATE FRUIT SYRUP TO PRODUCE HIGH-QUALITY DIPS

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### ABSTRACT

A system for the extraction, filtration, and concentration of date fruit syrup was developed. The syrup was first extracted under 1000 mbar and under partial vacuums of  $-1.8$ ,  $-2.8$ , and  $-5.5$  mbar in an extractor developed by the authors, then filtered using a filtration ladder of 1.0, 0.25, 0.112, and 0.011-mm pore sizes and finally concentrated to the product known as a dip in the Arab world. The concentration of the syrup to dip was done in a rotary evaporator at  $50\text{ }^{\circ}\text{C}$  and 180 rpm for 9–11 hours and under direct sun rays at approximately  $48\text{ }^{\circ}\text{C}$ , placed in stainless steel trays ( $61 \times 46 \times 5$  cm) for 4 hours (12:30–4:30 p.m.). The quality of dip produced under sun evaporation was compared to that of a commercial one collected from the markets in Saudi Arabia and was found to be superior due to two decisive criteria, namely color (the appearance) and pH (the taste), and also in terms of other nutritional components.

**Key words:** date fruit, dips, extraction, filtration, sun concentration, syrup

### INTRODUCTION

The Kingdom of Saudi Arabia is one of the largest date palm growers in the world with about 31.2 million trees producing about 1.5 million tons of date fruits annually (NCPD 2018). Date fruits are a historic staple food in the country with high cultural and religious significance to the Saudi people, a fact that insures sustainability of production and consumption. The main component of date fruit is sugar, which amounts to 50–60% of its weight present in the form of glucose and fructose in approximately equal quantities (Al Khateeb & Dinar 2002; Ahmed et al. 2013). Dates are consumed as raw fruits or after processing into a product called dip or into a paste. Dips are very popular in Saudi Arabia; the product is mainly used as a sweetener in different food products. It is traditionally produced at household and farm levels and industrially. The industrially produced dip used in this study as extracted at  $80\text{ }^{\circ}\text{C}$  for 30 minutes and concentrated at  $80\text{ }^{\circ}\text{C}$  as described by the producer. Household and farm-level production is done by packing washed whole date fruits in perforated plastic sacks that are stacked in heaps of 5–10 sacks and arranged in about 10 rows in a room.

An appropriate piece of wood is placed on top of the rows and weighs (usually bricks) of up to one ton is placed on the wood to exert pressure. The concentrated syrup (dip) oozing out is collected in containers for up to 3 months, usually after the date of fruit harvest in the hot season of July to October (personal communication with producers). Reports of the Ministry of Environment, Water and Agriculture of Saudi Arabia (MEWA 2014) show that the amount of syrup produced by the industry in Saudi Arabia in 2011 was 700 tons. No data about the amounts of household and farm production is available, though this type of production is widely practiced in the country. The main drawback of such small-scale production is time-consuming production with low yield and unstable quality. The industrially produced syrup is dark in color and of relatively low organoleptic quality in consumers' opinion. The quality and yield of syrup depends mostly on the extraction method used. In this study, a new system for dips production is developed. It aims at producing high-quality dips at a high yield by using improved extraction and concentration methods.

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## MATERIALS AND METHODS

Date fruits used in this study were of the 'Reziz' cultivar. They were obtained from the local market in Al-Hasa, Saudi Arabia, in November 2017 and brought to the laboratories of the College of Agricultural and Food Sciences, King Faisal University, Al Ahsa, Saudi Arabia. The fruits were cleaned from debris, backed in plastic containers and kept in the refrigerator at 4 °C until use.

### Extraction

Extraction is performed under a partial vacuum. This will lead to a decreased temperature of extraction, which will result in a reduction of caramelization and reduction of volatile components loss of the extracted syrups (Sanches Silva et al. 2020). The system developed in this study is made of an extractor and a clarifier. The body of the extractor is fabricated from stainless steel with total and working volumes of 15 and 10 liters, respectively. It is equipped with a motor, impeller, blades, different outlets, and a stand. The partial vacuum is generated by connection to a vacuum pump and controlled by a vacuum gauge, temperature measurement, and control. Extraction starts with washing whole date fruits in running water, soaking (1 kg fruit in 4 liters of water) at about 75 °C for 30 minutes, blending in the extractor for 20 minutes at different partial vacuums, and filtration.

### Filtration

The clarifier is a filtration system made of four filters connected in a descending ladder of pore sizes to remove impurities of different particle sizes. The extracted syrup moves directly from the extractor system to the clarifier with the help of a vacuum pump.

### Concentration

The syrup was concentrated in a rotary evaporator and under direct sun rays. Rotary evaporator (EYELA 1200B, Tokyo, Japan) at 50 °C and 180 rpm, time for one liter of syrup is 9–11 hours to obtain syrup at approximately 71 Brix as a minimum required by the Saudi Standard. Sun evaporation was carried out on the 23, 24, 25, and 26th of July 2017 at approximately 48 °C. The syrup (2.5 liters) was placed into stainless steel trays (61 × 46 × 5 cm) for 4 hours (12:30–4:30 p.m.). The weather conditions at the beginning and end of evaporation were: temperature 47.9 °C and 48.1 °C, wind speed 0.9 and 1.8 m·s<sup>-1</sup>, solar radiation 0.925 and 0.531 K wat·m<sup>-2</sup>, and the average atmospheric pressure and relative humidity were 0.99 bar and 6%, respectively.

### Chemical analysis

Moisture, protein, and acidity were determined according to standard methods (AOAC 2000). The soluble solid content was evaluated using an Abbe refractometer (Milton Roy, USA) as described by Entezari et al. (2004). Potassium and sodium content was measured using Flame Photometer Model PFP7/C (Jenway, UK), and calcium, magnesium, chromium, iron, zinc, copper, and manganese were estimated using Atomic Absorption AA-7000 Shimadzu (Kyoto, Japan). Total sugars were determined using the p-hydroxybenzoic acid hydrazide procedure according to Blakeney and Mutton (1980). The color was evaluated using Hunter lab (Chroma meter CR-410 from Konica Minolta Japan). Acidity was measured according to AOAC (1984), and ash content according to the AOAC (1995) method No. 923.03. Total phenolic compounds were determined using the Folin–Ciocalteu method (Shui & Leong 2006; Biglari et al. 2008).

### Statistical analysis

A one factorial design was used. Experimental factors including fruit to water ratio and blender mixing speed were kept at fixed levels depending on findings from former work done in our lab (unpublished data), only vacuum was varied at four levels. Statistical analysis was done using SAS (2007) for practical experiments in completely randomized three-replicated (ANOVA) in addition to experimental error effect. Comparison between the averages of different treatments performed using a least significant difference (LSD) method.

## RESULTS AND DISCUSSION

### Effect of extraction pressure on the quality of the filtered date fruit syrup

Date fruit syrup was extracted under four pressures and then filtered. The results are presented in Tables 1 and 2. 72–73% of the initial volume was filtered as a syrup containing approximately 15% total soluble solids and 27–28% of the initial volume remained as residue. This residue contained approximately 83% moisture, meaning that about 23% of the extracted sugars were lost in this residue. The filtration system needs improvement. No significant differences between the four pressures were observed in syrup yields, total soluble solids (TSS) content, amount of residue, or moisture content of this residue (Table 1).

The pH values of the syrups extracted under the pressures of 1000, -1.8, and -2.8 mbar showed no significant differences, while the pH of the syrup extracted at the pressure of -5.5 mbar had a significantly higher pH value than the syrups extracted under the other pressures (Table 2). On the other hand, the color of the syrup was significantly affected by the pressure of extraction.  $\Delta E$  of the syrups increased steadily with decreased pressure (Table 2). This can be attributed to the fact that reduced pressure had a milder effect on sugar caramelization, hence the brighter colors.

#### Effect of concentration methods on dip quality

Dips were concentrated using the two methods of evaporation shown in Table 3, namely, sun and rotary evaporation of syrups extracted under the four pressures (1000, -1.8, -2.8, -5.5 mbar). The obtained dips were analyzed for TSS, pH, and color values. The syrups were concentrated to approximately 88% TSS in both the sun and the rotary evaporation, with no significant differences resulting from the four pressures (Table 3). The pH of dips produced under sun evaporation was slightly higher than that of dips produced under rotary evaporation,

but both had pH values lower than the pH values of the syrups from which they were produced (Tables 2 & 3). Evaporation resulted in a relative increase in the concentration of the acids of the syrup from which dips were produced. In both methods of evaporation, the pH values of the resultant dips remained constant at all pressures with a small and insignificant increase at -5.5 mbar (Table 3). The pressure of syrup extraction did not significantly affect the color of dips produced by both methods of concentration, though  $\Delta E$  increased slightly with decreased pressure of extraction for dips concentrated under the sun (Table 3).  $\Delta E$  of dips concentrated under the sun was significantly higher than that of dips concentrated under rotary evaporation as indicated by the values of the paired t-test, which means that the sun concentrated dips were clearer and brighter in color (Table 3). The highest difference was for dips produced from the syrup extracted under the atmospheric pressure (1000 mbar). The difference decreased with the decreasing of extraction pressure (Table 3). It can therefore be said that dips concentrated under the sun were of brighter color, which is more acceptable to the consumer.

Table 1. Extraction and filtration of date syrup using a ladder system of 1.0, 0.25, 0.112, and 0.011-mm pore sizes at different pressures

Pressure (mbar)	Initial volume (ml)	Filtrate volume (ml)	Syrup yield (%)	TSS (Brix)	Residue volume (ml)	Moisture of residue (%)
1000	4523	3271	72	14.9 <sup>a</sup> ±0.04	1252	83
-1.8	4487	3226	72	14.6 <sup>a</sup> ±0.06	1261	83
-2.8	4560	3324	73	14.7 <sup>a</sup> ±0.03	1236	83
-5.5	4439	3236	73	14.9 <sup>a</sup> ±0.10	1203	84

No significant differences between means in a column having same superscript letters ( $p < 0.05$ ). All values are averages of three replicates ± SD

Table 2. Color and pH of the syrups produced

Pressure (mbar)	pH	$\Delta E^*$
1000	5.4 <sup>b</sup> ±0.01	42.8 <sup>d</sup> ±0.1
-1.8	5.4 <sup>b</sup> ±0.01	43.3 <sup>c</sup> ±0.1
-2.8	5.4 <sup>b</sup> ±0.01	46.4 <sup>b</sup> ±0.04
-5.5	6.0 <sup>a</sup> ±0.06	47.5 <sup>a</sup> ±0.03

\*The higher  $\Delta E$ , the brighter the color. No significant differences between means in a column having same superscript letters ( $p < 0.05$ ). All values are averages of three replicates ± SD

Table 3. TSS, pH and the color of concentrated date syrups (dips)

Pressure (mbar)	Sun concentration			Rotary concentration			T-test	P-value
	TSS	pH	$\Delta E$	TSS	pH	$\Delta E$		
1000	87.4 <sup>a</sup> ±0.60	4.9 <sup>a</sup> ±0.01	84.5 <sup>a</sup> ±0.02	88.3 <sup>a</sup> ±0.03	4.2 <sup>a</sup> ±0.01	81.5 <sup>a</sup> ±0.03	312.01	0.0001
-1.8	87.7 <sup>a</sup> ±0.02	4.9 <sup>a</sup> ±0.01	85.2 <sup>a</sup> ±0.05	87.8 <sup>a</sup> ±0.02	4.2 <sup>a</sup> ±0.02	82.7 ±0.04 <sup>a</sup>	42.66	0.0001
-2.8	87.9 <sup>a</sup> ±0.03	4.9 <sup>a</sup> ±0.06	85.2 <sup>a</sup> ±0.05	88.3 <sup>a</sup> ±0.02	4.2 <sup>a</sup> ±0.01	82.4 <sup>a</sup> ±0.04	38.23	0.0001
-5.5	88.0 <sup>a</sup> ±0.03	5.0 <sup>a</sup> ±0.08	85.6 <sup>a</sup> ±0.02	87.8 <sup>a</sup> ±0.07	4.3 <sup>a</sup> ±0.03	82.0 <sup>a</sup> ±0.08	33.01	0.0001

The higher  $\Delta E$ , the clearer the dips color. No significant differences between means in a column having same superscript letters ( $p < 0.05$ ). All values are averages of three replicates  $\pm$  SD

### Chemical analysis of sun concentrated dips

Dips concentrated under the sun exhibit characteristics that make it superior in quality to dips concentrated by rotary evaporation. It had lighter color and higher pH, both known to positively affect its consumers' acceptability, the first affects its general appearance and the second its taste. Therefore, dips concentrated under the sun were chosen for further investigations. It was chemically analyzed for the contents of acidity, ash, moisture, sugars, and protein and compared to commercial dips obtained from the Saudi Market (Table 4).

The sugar content of dips produced in this study was significantly higher than that of the commercial products (Table 4). The sugar content of dips produced from the syrup extracted at the atmospheric pressure (1000 mbar) was approximately 720 g·kg<sup>-1</sup>. The sugar content increased insignificantly to approximately 728 g·kg<sup>-1</sup> in dips produced from the syrup extracted at -1.8 mbar and then significantly to 759 and 761 g·kg<sup>-1</sup> in dips produced from syrups extracted at -2.8 and -5.5 mbar, respectively. Again, the contents of protein, ash, and acidity of dips produced from syrups extracted under reduced pressures were significantly higher than their contents in dips produced from the syrup extracted under atmospheric pressure and in the commercial dips (Table 4). On the other hand, sun evaporation performed in this study produced dips, which was more concentrated than the commercial one, with moisture contents of approximately 14% compared to the content of more than 19% in the commercial dips (Table 4).

### Phenolic compounds in dips and their effect on its color

The content of the total phenolic compounds in the dips under study was determined and compared to their content in commercial products (Table 5). The content of these compounds in the dips produced in this study was significantly higher than the content in commercial ones. The content increased with decreased pressure of extraction of the syrups from which dips were produced, which indicates that reduced pressures increased the extraction rate of these compounds. The difference in content between dips produced from syrups extracted at atmospheric pressure (1000 mbar) and dips produced from syrups extracted at lower pressure was significant. Although the content increased further with decreasing pressure of syrup extraction, the differences were not significant (Table 5). As a general observation, it is interesting to notice that the higher the content of phenolic compounds in dips, the brighter is its color as indicated by an increased  $\Delta E$  with increasing content of these phenolic compounds in dips (Table 5). Although the correlation between phenolic compounds and the color of dips was not examined in this study, it seems that the bright color of the phenolic compounds was reflected in the bright color of dips produced in this study, in contrast to the much darker color of the commercial dips and its relatively low content of these antioxidants. Reports in the literature (Cömert et al. 2020) link color hues in foods to their content of antioxidant compounds.

### The mineral content of dips

The content of five minerals, namely, Fe, Cu, Zn, Ca, and Na was significantly higher in dips produced in this work than in the commercial dip samples (Table 6). Regarding the other four elements – Cr, Mg, K, and Mn – the difference in content was not significant,

but still, the content in dips produced in this work was slightly higher. As shown above (Tables 4 & 5), these results confirm that the extraction of syrups from dates under a partial vacuum is more efficient in the extraction of all nutrients in the fruit including the minerals, sugars, proteins, and phenolic compounds.

Table 4. Chemical parameters of commercial and sun evaporated dips

Pressure	Sugar (g per kg)	Protein (%)	Ash (%)	Acidity (%)	Moisture (%)
Commercial	628.8 <sup>c</sup> ±0.64	2.8 <sup>b</sup> ±0.03	1.7 <sup>b</sup> ±0.03	0.49 <sup>b</sup> ±0.01	19.5 <sup>a</sup> ±0.01
1000	719.7 <sup>b</sup> ±1.15	3.0 <sup>b</sup> ±0.1	1.8 <sup>ab</sup> ±0.03	0.53 <sup>b</sup> ±0.02	13.8 <sup>b</sup> ±0.05
-1.8	727.8 <sup>b</sup> ±2.25	4.0 <sup>a</sup> ±2.14	2.1 <sup>a</sup> ±0.01	0.64 <sup>a</sup> ±0.01	13.5 <sup>b</sup> ±0.01
-2.8	759.1 <sup>a</sup> ±2.25	3.8 <sup>a</sup> ±0.07	2.0 <sup>a</sup> ±0.02	0.66 <sup>a</sup> ±0.01	14.0 <sup>b</sup> ±0.03
-5.5	761.0 <sup>a</sup> ±3.46	3.9 <sup>a</sup> ±0.01	2.2 <sup>a</sup> ±0.04	0.68 <sup>a</sup> ±0.06	13.6 <sup>b</sup> ±0.02

Note: See Table 1

Table 5. Phenolic content and color in commercial and sun evaporated dips

Pressure (mbar)	Phenolics (mg GAE per 100 g)	ΔE
Commercial	4.8 <sup>c</sup> ± 0.02	80.0 <sup>b</sup> ± 0.02
1000	5.2 <sup>b</sup> ± 0.10	84.5 <sup>a</sup> ± 0.01
-1.4	5.7 <sup>a</sup> ± 0.03	85.2 <sup>a</sup> ± 0.02
-2.8	5.8 <sup>a</sup> ± 0.02	85.2 <sup>a</sup> ± 0.02
-5.5	5.9 <sup>a</sup> ± 0.04	85.6 <sup>a</sup> ± 0.02

GAE – gallic acid equivalent. No significant differences between means in a column having same superscript letters ( $p < 0.05$ ). All values are averages of three replicates ± SD

Table 6. Mineral content (ppm) in commercial and sun evaporated dips

Pressure	Cr	Fe	Cu	Zn	Mg	Ca	Na	K	Mn
Commercial	29.7 <sup>a</sup> ±0.02	32.6 <sup>b</sup> ±0.02	15.8 <sup>b</sup> ±0.06	6.4 <sup>c</sup> ±0.01	268.3 <sup>a</sup> ±0.40	159.4 <sup>c</sup> ±0.10	45.5 <sup>b</sup> ±0.20	1166.0 <sup>a</sup> ±0.41	16.5 <sup>a</sup> ±0.12
1000 mbar	30.0 <sup>a</sup> ±0.02	32.9 <sup>b</sup> ±0.06	18.9 <sup>a</sup> ±0.06	19.0 <sup>b</sup> ±0.04	267.0 <sup>a</sup> ±0.20	197.3 <sup>b</sup> ±0.20	56.1 <sup>a</sup> ±0.20	1168.6 <sup>a</sup> ±0.36	16.8 <sup>a</sup> ±0.30
-1.8 mbar	31.4 <sup>a</sup> ±0.08	45.6 <sup>a</sup> ±0.03	18.6 <sup>a</sup> ±0.20	19.8 <sup>b</sup> ±0.06	271.9 <sup>a</sup> ±0.40	261.6 <sup>a</sup> ±0.01	55.5 <sup>a</sup> ±0.20	1170.7 <sup>a</sup> ±0.90	17.2 <sup>a</sup> ±0.30
-2.8 mbar	32.0 <sup>a</sup> ±0.01	46.2 <sup>a</sup> ±0.06	19.1 <sup>a</sup> ±0.20	22.3 <sup>b</sup> ±0.06	270.6 <sup>a</sup> ±0.02	256.3 <sup>a</sup> ±0.02	58.3 <sup>a</sup> ±0.20	1171.0 <sup>a</sup> ±0.80	17.3 <sup>a</sup> ±0.20
-5.5 mbar	32.5 <sup>a</sup> ±0.01	45.5 <sup>a</sup> ±0.02	19.2 <sup>a</sup> ±0.20	24.6 <sup>a</sup> ±0.02	274.6 <sup>a</sup> ±0.10	262.7 <sup>a</sup> ±0.02	57.0 <sup>a</sup> ±0.30	1170.8 <sup>a</sup> ±0.90	17.9 <sup>a</sup> ±0.30

Note: See Table 1

## CONCLUSION

A system for the extraction, filtration, and concentration of date fruit syrup was successfully developed. Extraction under partial vacuum increased the yield of nutrients including sugars, proteins, minerals, and phenolic compounds. The concentration of the syrups under direct sunrays and in a rotary evaporator produced dips with superior quality in color, pH value, and the content of nutrients compared to dips produced commercially. Concentration under the sun resulted in higher quality compared to dips concentrated in the rotary evaporator. With all other parameters of the obtained dips showing no significant differences, extraction at  $-2.8$  and  $-5.5$  mbar gave the highest sugar content with no significant difference between the lower pressures. Hence, extraction at  $-2.8$  mbar is recommended for cost consideration.

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### Disclosure statement

The authors declare no conflict of interest.

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