CARPATHIAN JOURNAL OF FOOD SCIENCE AND TECHNOLOGY

journal homepage: **http://chimie-biologie.ubm.ro/carpathian_journal/index.html**

NEW TECHNOLOGICAL METHODS TO CONTROL HMF FORMATION IN DATE SYRUP DURING PROCESSING

Shahinaz A. Helmy[1]✉, Aya Y. Mostafa[2], Adel Z. M. Badee[1], Serag A. Farag, [2] and Mohamed E. Abdel-Aziz [1]

[1]Food Science Department, Faculty of Agriculture Cairo University, Giza, Egypt.
[2]Food Irradiation Research Department, Industrial Irradiation Division, National Center for Radiation Research and Technology (NCRRT), Egyptian Atomic Energy Authority (AEAE), Cairo, Egypt
✉shahinaz29@cu.edu.eg

https://doi.org/10.34302/crpjfst/2023.15.1.13

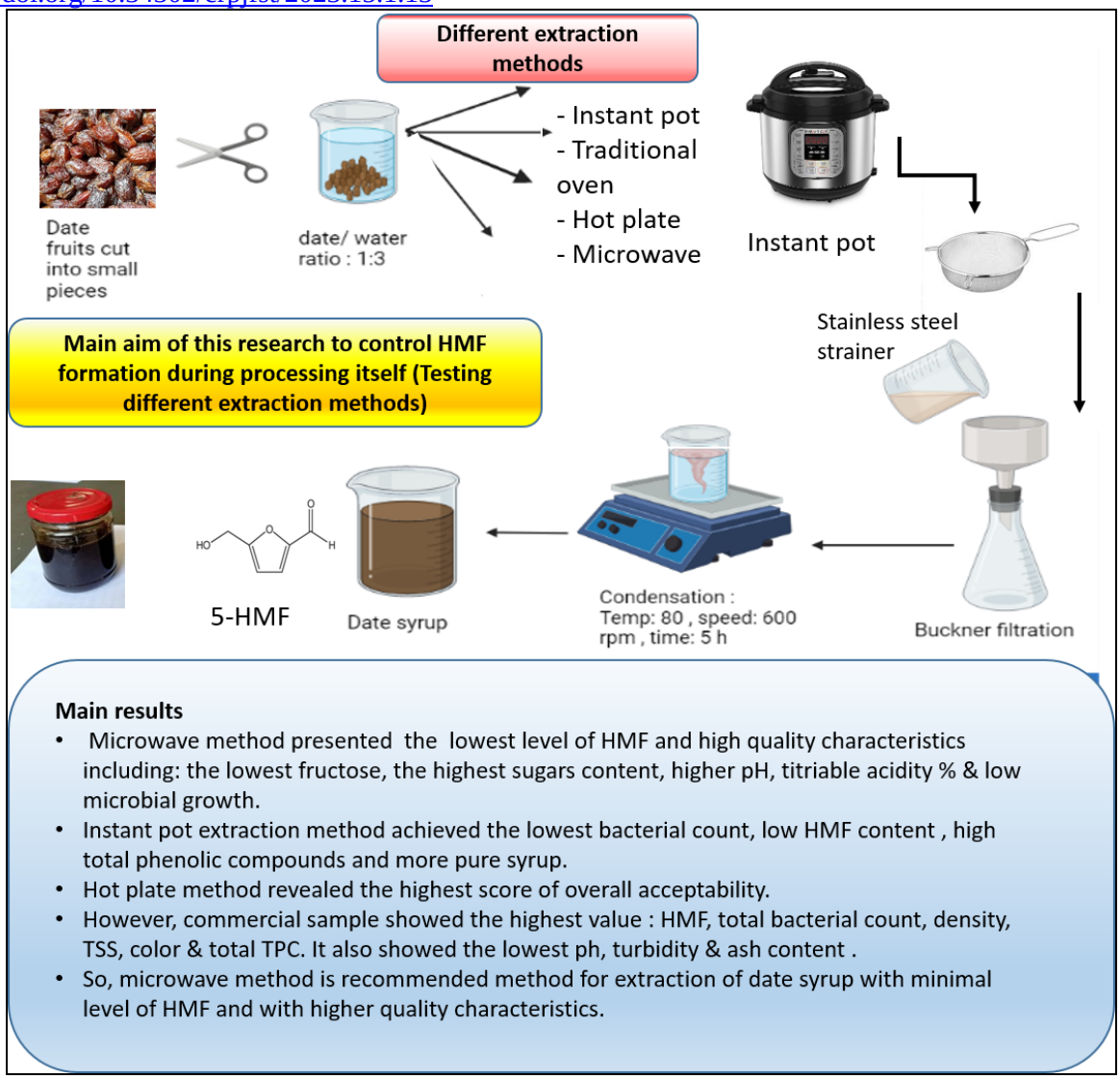


Figure 1. Graphical abstract clarify date syrup processing steps and the new proposed techniques (New extraction methods) that may reduce formation of HMF during manufacture processing itself.

Article history:
Received:
25 October 2022
Accepted:
25 December 2022

Keywords:
Reducing HMF;
Toxic compound;
Extraction methods;
Quality characteristics and date syrup.

ABSTRACT

Date occupies a position between many crops with its nutritional and medicinal values. The higher fruit loss induces the processing of these fruits to convert to useful products, such as date syrup and date paste. Date syrup is characterized by its high nutritional value. However, it contains a toxic-hazard compound (5-hydroxyl methyl furfural). This compound is usually formed as a result of the dehydration of sugars under higher temperatures during processing (Millard reaction). So, the present paper aimed to reduce HMF levels in date syrup during processing using different extraction methods. The obtained results emphasized that the extraction method has a great influence on HMF levels of the lab-produced date syrup compared with the commercial sample. Where instant pot, traditional oven, hot plate, microwave methods, and commercial sample recorded HMF levels ranged (from 503.63, 285.38, 1010.00, 240.13 & 1844.30 mg/ kg, respectively). In this regard, the microwave extraction method presented the best results concerning with safety of the product: the lowest level of toxic hazard (5- HMF) and high-quality characteristics including Fructose, pH, titriable acidity, and a high score of overall acceptability of Panel test. Meanwhile, the instant pot extraction method was characterized by a low level of HMF and the lowest microbial count. While the commercial sample featured elevated levels of (5-HMF), the highest bacterial count, and the darkest color. The commercial sample also showed the highest score in the following: TSS, density, specific weight, color, and total phenolic compounds. However, it showed the lowest score in turbidity, EC, total, and sulfated ash %. Also, sensory evaluation by Hedonic scale showed consumers' appreciation of date syrup especially extracted using microwave methods. So, the microwave method is recommended method for the extraction of date syrup with a minimal level of HMF and higher quality characteristics.

1. Introduction

Dates (*Phoenix dactylifera* L.) represent one of the most important fruit trees in many states worldwide, particularly in the middle east and Arabian countries as Hashem, *et al.* (2017) clarified. Nowadays, this fruit has also gained great importance in global trade. Within the recent period, world date production has doubled; this trend is expected to continue as per FAO projections according to Ghnimi *et al.* (2018). Egypt occupies the first position among the top five date-producing countries. Where Egypt's annual production of date surpasses 1.7 million tons (FAO, 2020). Dates (Semi-dry) resemble about 19.2% and the Siwi date is considered the most important semi-dry cultivar in Egypt as declared El-Samahy, *et al.* (2005). The rejected ratio of Siwi date reached about 15% (3937 tons) of the total date production of Baharia oasis, mainly used in animal feeding. So, it is very important to utilize these quantities in date dibs production The majority of date fruits are used up directly at different stages (Khalal, Rutab, and Tamr), with minimal or no processing. Nowadays, great attention is paid to the improvement of date processing. Various date products such as date syrup, liquid sugar, alcohol, and vinegar are successfully marketed (Yousif, *et al.*, 1987). One of the important popular products of dates is date syrup, which is principally produced from second-grade and non-marketable dates as Ramadan (1998) mentioned. Date syrup processing seems to be a suitable way for preserving the fruits and minimizing transportation and storage costs and could open new prospects for date fruits. A promising usage of date syrup recently emerges in food industries. It is used as a natural sweetener in many food products instead of harmful sugar such as beverages, jam, jelly, ice cream, yogurt, bakery products, date bars, date sheets, and confectionary (Abd El-Hady *et al.*, 2014; El-Samahy & Youssef, 2009; Hou et al.,

2022). Where debis is a very rich source of natural sugar, polyphenol, antioxidant activity, vitamins (Niacin), and minerals (Fe, Ca, Na, Mg, Zn). So, date syrup is a great source of essential nutrients and various health benefits as declared by Shahein et al. (2022). However, dates processing face a health hazard concern. Where date fruits exposes to elevated temperature during the processing of date syrup which can influence the quality, nutritional value, and product safety. Higher heat stimulates the formation Millard reaction products like furan and 5-hyroxy methyl furfural A higher concentration of HMF causes cytotoxic, oxidative stress and genotoxic effects on human health (Hou et al., 2022). 5-HMF is one of the most important heat-induced food toxicant (food contaminants) formed in various foods including honey, jams, fruit juices, milk, spirits, coffee, cereals, bakery products, and vinegar (Farag et al., 2020). HMF is a heterocyclic product (Choudhary et al., 2021). It is yellow, low-melting solid and highly water-soluble. The molecule composed of furan is a ring, having aldehyde and alcohol functional groups. HMF formed by the caramelization of sugars as well. HMF is a quality parameter to monitor heating (mode & rate) or storage (period & conditions) in many foods (Rahimzadeh, et al., 2014).The toxicity of 5-HMF has been experimented by many researchers. They reported that 5-HMF is a potential toxin, mutagen, and carcinogen for humans (Eshete & Eshete, 2019; Farag et al., 2020; Michail et al., 2007). Where HMF is metabolized by human body (catalyzed by sulfotransferase) to genotoxic and carcinogenic compound called 5-sulfoxymethylfurfural (SMF) (Capuano & Fogliano, 2011; Severin et al., 2010; Shapla et al., 2018). SMF is also considered nephrotoxic (Bakhiya, Monien, Frank, Seidel, & Glatt, 2009). Unfortunately, SMF is considered a non-excretable compound and may accumulate in human body. Therefore, HMF contaminant take over a great attention from many researchers (Shapla et al., 2018).Different carcinogenic effects of HMF were reported on various living things and organs (Yang, *et al.*, 2019): highly toxic to honey bees (Le Blanc et al., 2009). 5-HMF raise the occurrence of deviant crypt foci in rat colon, skin papillomas in mice, lipomatous tumors in rat kidney (Capuano & Fogliano, 2011), small intestine adenomas in mice (Svendsen, *et al.*, 2009), hepatocellular adenomas in female mice, and has mutagenic effects on *S. typhimurium* (Lee, *et al.*, 1995). Where 5-SMF also induces small intestine adenomas potential in mice (Svendsen et al., 2009) and regeneration and atypical hyperplasia of tubules and hepatotoxic effects and sororities of peritoneal tissues (Bauer-Marinovic, *et al.*, 2012). Humans revealed higher sensitivity to 5-HMF because the existence of sulfotransferase, which can convert 5-HMF into SMF, is appeared in extra hepatic tissues at higher levels than rodents (Teubner, *et al.*, 2007).

Safety margin of HMF is also called (preclinical level), in which no toxic effects have been perceived. The estimated daily intake of 5-HMF for humans is approximately 2.5 mg/kg body (Capuano & Fogliano, 2011). HMF consumption beyond the safety limit is also cytotoxic to humans and causes irritation to the mucous membranes of the upper respiratory tract, skin and eyes etc. as declared by Choudhary *et al.* (2021); (Pastoriza de la Cueva et al., 2017; Shapla et al., 2018). Doses differ 2–30 mg / person/day according to human consumption (Abraham et al., 2011; Pastoriza de la Cueva et al., 2017).This range was found to be safe and does not cause health risks to humans (Janzowski, *et al.*, 2000).CODEX (Codex Alimentarius Commission) established a maximum limit for HMF in the honey of 40 - 80 mg/kg in tropical honey (Abraham et al., 2011). HMF levels should not surpass 80 mg/kg in tropical zones as mentioned by Commission. (Amended in 2019); (Eshete & Eshete, 2019)

In another study, Shapla et al. (2018) stated tolerable daily intake (TDI); at daily doses ranging from 80 to 100 mg/kg body weight. In fact, it is important to get ways to reduce 5-HMF levels in processed food so that

the total intake of 5-HMF can be reduced for humans.

Higher concentrations of HMF were found in date syrup and its fresh products ranged between 1000 to 2675 mg⁄kg (Jafarnia, *et al.*, 2016). This higher amount of HMF was produced in date syrup when the high temperatures (110℃) processing method was followed (Naknean, *et al.*, 2009). The same findings were reported for other foods such as bakery products (Ramírez-Jiménez, *et al.*, 2000) and the formation of HMF is dependent on the time of process in boiling juice. A highly-variable amount of HMF, changing from 12.8 to 3500 µg/kg, was found in boiled juices (Jafarnia et al., 2016). The challenge that concerns the present study is the formation of HMF with higher levels in date syrup and how to interfere and control HMF formation during the processing steps. So, the present study aimed to investigate the possibility to reduce such toxic compounds (5-HMF) during processing (using different extraction methods) and the influence of different extraction methods on the physicochemical and microbial characteristics of date syrup.

2. Materials and methods

2.1. Materials

Raw Materials & Chemicals: Mature and fresh date fruits (semi- dry, Siwi cv.) were purchased from local markets, Giza, without any impact and physical damage. The fruits were immediately packed in poly ethylene bags after sorting, washing with tap water and storing until processing. Commercial date syrup samples were purchased from the local markets, Giza. Standard of 5-HMF (97%) was purchased from Sigma Aldrich Company, (St. Louis, Missouri, USA). All chemicals used in this investigation were of analytical grade.

2.2. Methods

2.2.1. Preparation of date juice and syrup

First, date juice samples were prepared with four different extraction methods. The treatments were carried out in comparison with a commercial date syrup sample from local market (Giza governorate). In all previous extraction methods, date juice (date extract) was prepared according to constant ratio (1:3 for date: water, respectively) according to Hashem et al. (2017). Date syrup preparation including steps of clarification were illustrated in Fig.1.

2.2.2. Extraction methods

a. Instant pot: In this novel method, Electrical pressure cooker (PALSON instant pot: made in china, model: CR-34J, Capacity: 8 liters, power: 1200- 1400 W, frequency: 220- 240 V, 50/ 60 Hz) was used for extraction of date juice. TSS $_{initial}$ of date extract or date juice = 20 %. Work conditions: Pressure, 15 psi (pounds/ square inch), temperature: 130 ^{o}C, time: 20 min (Fig.1).

b. Traditional oven: date juice was extracted using traditional oven (100 ± 10 ^{o}C for 2 hours). TSS initial of date extract or date juice = 15 %.

c. Hotplate: date juice was extracted using hot plate (JOANLAB hotplate Stirrer) with the same pervious ratio at 100±5° C. This mix was heated on a high temperature (100 ± 5 ^{o}C) for 30 min. to produce date extract (date juice). TSS $_{initial}$ of date extract = 17 %.

d. Microwave: about 250 g date fruits /750 ml tap water. This mixture was put into the microwave oven (JAC microwave, Mode: NGM-2002, Capacity 20 liters, Power: 1200 W, frequency: 230V- 50 Hz) using medium temperatures level (ranging: 70 -80 ^{o}C) for 20 min. TSS $_{initial}$ of date extract = 15 %.

***2.2.3. Date juice filtration*:** different extracted samples are filtered and concentrated to produce date syrup (TSS= 68 %) as follows:

Coarse filtration: This procedure was done using Muslin cloth (4 layers) and coarse stainless steel strainer to get rid of date fruit parts.

Fine filtration: This procedure was applied using Muslin cloth, followed by filtration with

filter paper (pore size: 102 um) using Buckner funnel (Fig.1).

2.2.4. Date extract concentration: Date syrup was prepared by concentration of date juice to (TSS= 68 %) using hot plate: Concentration conditions: Temp: 80 ± 5 °C, Speed: 600 rpm and time: 5 hours as mentioned in Fig.1.

***2.2.5.* Physicochemical characteristics of date syrup**

2.2.5.1. Determination of HMF:

5-HMF content of lab-produced date syrup was determined after processing comparing with the commercial date syrup sample. HMF stock solution and serial dilutions were prepared (7 points of standard solution) and standard curve was plotted (Calculating R2 predicted: it should be ≥ 85 %).

Extraction of HMF: 5-HMF completely dissolves in water, so it is extracted easily in distilled water. The extraction ratio is 2 ml of date syrup / 25 ml of distilled water.

Purification procedures were applied using Carrez I solution (15 g of potassium hexacyano ferrate dissolved in 100 ml of distilled water) and Carrez II (30 g of zinc acetate dissolved in 100 ml of distilled water). About 0.5 ml of each carrez I and CarrezII were added to the HMF extract. The mixture was vortexed for 2 min. allowing the samples to precipitate. Remark the filtrate (containing HMF), then it was filtered using filter paper (qualitative filter paper 102 moderate (110 mm)) excluding the precipitate. After that, extracted samples were filtered using syringe membrane filter (0.45 um). Finally, filtrated samples were kept (in dark small vials) frozen until to be injected in HPLC- UV instrument. Suitable injection volume in HPLC instrument: 20 ul. Sample preparation with carried out according to Baltaci and Akşit (2016) with some modification.

Device Specification: Waters 2690 Alliance HPLC system equipped with a Waters 996 photodiode array detector. About10 µl was taken of standard vial & unknown sample and injected to HPLC. Also, about 10 µl of un-known sample was injected. HPLC analysis conditions: Column C18 Inertsil: 4.6x250 mm, 5µm, Mobile phase: Water: Methanol. (85%: 15%), Mode of elution: Isocratic, Flow rate: 1ml/min, and ambient temperature at wavelength 285 nm.

2.2.5.2. Total soluble solids (TSS %):

Total soluble solids (TSS%) expressed as °Brix or percentage, were measured at 20±0.5°C by a Hand Refractometer (model (ATAGO, Japan)as described by Ranganna (1977).

2.2.5.3. pH values:

pH of date extract and syrup were measured using a digital pH-meter (Hanna, HI 902 meter, Germany).

2.2.5.4. Density & specific weight (SP):

Density (g/ cm^3) of syrup was measured by using a pycnometer. The results were calculated according to method mentioned in (Lullah-Deh, Khan, & Eneji, 2018). Determination of specific weight (Ratio): The specific weight of date syrup was calculated as the following: Specific weight = Weight of sample in pycnometer / weight of the same volume of water (SP = W of 25ml of date syrup / W of 25 ml of water). This determination was done according to Lullah-Deh et al. (2018).

2.2.5.5. Titratable acidity (TA):

It was titrated with 0.1N sodium hydroxide to the endpoint in the presence of phenolphthalein indicator and the results were expressed as gram malic acid per 100 gram sample (A.O.A.C, 2000).

2.2.5.6. Turbidity:

Turbidity was measured using pen turbidity meter (Model: Milwaukee MI 415-Romania). The results were expressed in NTU (Nephelometeric Turbidity Units).

2.2.5.7. Electrical conductivity (EC):

Date syrup samples is very viscose, so it is better to be diluted, so, 1 ml of date syrup was diluted with 25 ml of d.w., the mixture was

vortexed for 5 min. and was measured using Electrical conductivity meter (digital electrical Cond. & TDS: mode: AZ8361., China), according to AOAC (1999) with some modification. The results were recorded in mS/ cm.

2.2.5.8. Total ash & sulphated ash:

Total ash (%) was calculated based on EC value as described by Sancho, Muniategui, Sánchez, Huidobro, and Simal (1991) using EC equation: Total Ash % = 0.083 EC - 0.092. However, sulphated ash % also calculated based on EC value according the following equation: Sulphated Ash (%) = 0.121 EC - 0.097.

2.2.5.9. Non-Enzymatic browning (420 nm):

Non-enzymatic browning was determined as a color indication at wave length (420 nm) using spectrophotometer (Model Labomed, Inc, New York, USA) according to Fathi, *et al*.(2013) with some modifications.

2.2.5.10. Non-reducing and reducing sugars:

Non- reducing and reducing sugars were determined using HPLC according to Al-Farsi (2003) with slight modification based on the injection of external standards: Sucrose, glucose and fructose as external standard solutions, which were prepared as described (Sesta, 2006; Zhang, Aldosari, Vidyasagar, Shukla, & Nair, 2015). The results were expressed as g / 100 ml date syrup. Measurements were done using Waters 2690 Alliance HPLC system equipped with a Waters RI detector (Refractive index). 20 µl of each standard and unknown sample were injected. HPLC analysis conditions: Column BP 100 Ca: 300 x7.8 mm, mobile phase was acetonitrile /Water (75 / 25 v/v). Mode of elution: Isocratic, flow rate: 0.4ml/min. temperature was 65 ^{o}C.

2.2.5.11. Total phenolic compounds (T.P.C):

Preparation of phenolic extract :phenolic extract preparation was carried out according to Rasheed, Cobham, Zeighmami, and Ong (2012) with some modification.

Preparation of Gallic acid standard curve: (Serial dilution of gallic acid solutions): Dissolve about 30 mg of Gallic acid in 50 ml solvent (methanol 80 %) according to Mistrello et al.(2014).

Determination of total phenolic content (T.C.P):determination of T.C.P were done according to the Folin-Ciocalteu method (Farhadi et al., 2016). Add 750 ul Folin solution (diluted 10 %) to 200 ul of phenolic extract and vortex. Let the mixture for 10 minutes. Then about 750 ul of aqueous sodium carbonate (2 %) was added. The samples were vortexed and kept at room temperature for 60 mint in the dark. Spectrophotometer measurements: The absorbance was recorded at wave length 765 nm (model: UVKON 860). Using the gallic acid calibration curve, calculate the total phenol concentration (using graph and linear equation. The results were presented as mg of gallic acid equivalent (mg GAE / 100 g sample) according to Farhadi et al. (2016).

2.2.6. Determination of antioxidant activity: (DPPH methods):

Radical scavenge activity was determined using DPPH (1.1-diphenyl-2- picrylhydrazyl). DPPH scavenging analysis was carried out following the (Brand-Williams et al., 1995). Antioxidant activity (AO %) was expressed as a percentage of DPPH scavenging relative to control (DPPH only). DPPH scavenging activity = [(Absorbance of control – Absorbance of sample)/ Absorbance of control] x 100 (Shahdadi, Mirzaei, & Daraei Garmakhany, 2015).IC $_{50}$ of DPPH: The IC_{50} is the concentration of phenolic extract that achieve inhibition for 50 % of DPPH radicals. This value was calculated from standard curve (Scavenging activity versus phenolic concentration of sample) as Farahnaky, Mardani, Mesbahi, Majzoobi, and Golmakani (2016)clarified.

2.3. Microbial examination:

The total plate counts as well as total yeasts and molds of different-extracted date syrup were determined using pour plate technique on

Nutrient agar media and Potato Dextrose Agar, respectively (Sulieman & Elkashif, 2009). Date syrup samples were serially diluted using sterile saline (0.85% NaCl) and the appropriate dilution was then plated. The plates for total count were incubated for 48 h at 35°C, however, the plates for yeasts and molds were incubated for 5 days at 28 °C. Colonies were then counted. Each test was performed in duplicate and the results were expressed as colony forming units (CFU) per milliliter.

2.4. Sensory evaluation:

Date syrup samples were given three-digit codes and organoleptically examined by ten panelists from Food Radiation Lab., Food irradiation research dept., National Center for Radiation Research and Technology (NCRRT), Industrial Irradiation Division, Egyptian Atomic Energy Authority (EAEA). Panelists were asked to evaluate each attribute (1-5).All samples were evaluated for color, purity, flowing rate (viscosity), sweetness, odor, taste, consistency and sugar crystal. The panelists were also asked to tell the final decision about the overall acceptability of date syrup using 9.point hedonic scale (Hyvönen & TÖRMÄ, 1983). Where samples Scores ranging from like extremely (9) to dislike extremely (1).Water and neutral wafers were also served for cleaning palate between samples.

2.5. Statistical analysis:

A complete randomized design was followed with one factor (4 different extraction methods were tested comparing with commercial date syrup sample). Each treatment was performed in 3 replications the number of treatment (n=15).

The obtained results (mean values of three separate experiments, unless otherwise stated, were statically analyzed using Minitab (18) software (Minitab 18, 2021). One way Analysis of Variance (ANOVA) and standard deviation (SD) followed by comparison test (Tukey test) were calculated. Pearson method (R^2) test were done to test correlation between some variables. All tests were performed at ($p < 0.05$).

3. Results & discussion

The present research aimed to reduce HMF formation in palm syrup during processing *via* different extraction methods and determine the effects of these methods on physico-chemical, microbial properties and organoleptic characteristics as well.

3.1. Effect of different extraction methods on HMF level in date syrup

The obtained results (Table 1 & Figure 2) emphasized that the extraction methods of date juice and heating levels play an important role in formation of HMF compound in processed date syrup. Where the commercial sample recorded the highest score followed by heat extraction sample (1844.30 & 1010.00 mg/ kg, respectively). Meanwhile, date syrup sample extracted by microwave methods showed the lowest HMF content followed by syrup extracted by traditional oven (240.13 & 285.38 mg/ kg). These findings were in a similar trend with pervious study (El-Nagga & Abd El–Tawab, 2012). Where they found that Microwave method showed the lowest concentration of HMF when compared with rotary extraction and water bath method. So, microwave extraction method is considered the safest method for date juice extraction (Containing low levels of such toxic compound, HMF).

Table 1. Effect of using different extraction methods on 5-HMF, Non-reducing & reducing sugars concentration of processed date syrup

Extraction methods	HMF content (ug/ mg) or (mg/ kg)	Non-reducing sugars(g/ 100 ml)	Reducing sugars (g/ 100 ml)		Total reducing sugars
		Sucrose	Glucose	Fructose	
Instant pot	503.63	35.69	30.38	13.14	43.52
Traditional oven	285.38	38.50	32.56	11.87	44.43
Hot plate	1010.00	41.92	34.41	12.31	46.72
Microwave	240.13	48.92	39.71	11.68	51.39
Commercial sample	1844.30	37.91	32.61	12.77	45.38
Pearson correlation between HMF & different sugars	—	**-0.34**	**-0.29**	**0.502**	

Instant pot: Electrical pressure cooker, Data were statically analyzed using Minitab (18) program.

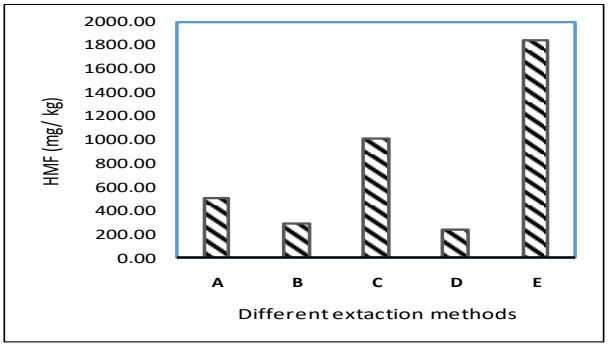


Figure 2. Effect of different extraction methods on HMF content (mg/ kg) of date syrup
Where, A: instant pot (Electrical pressure cooker), B: Traditional oven, C: hotplate method, D: Microwave method, and E: commercial date syrup sample.

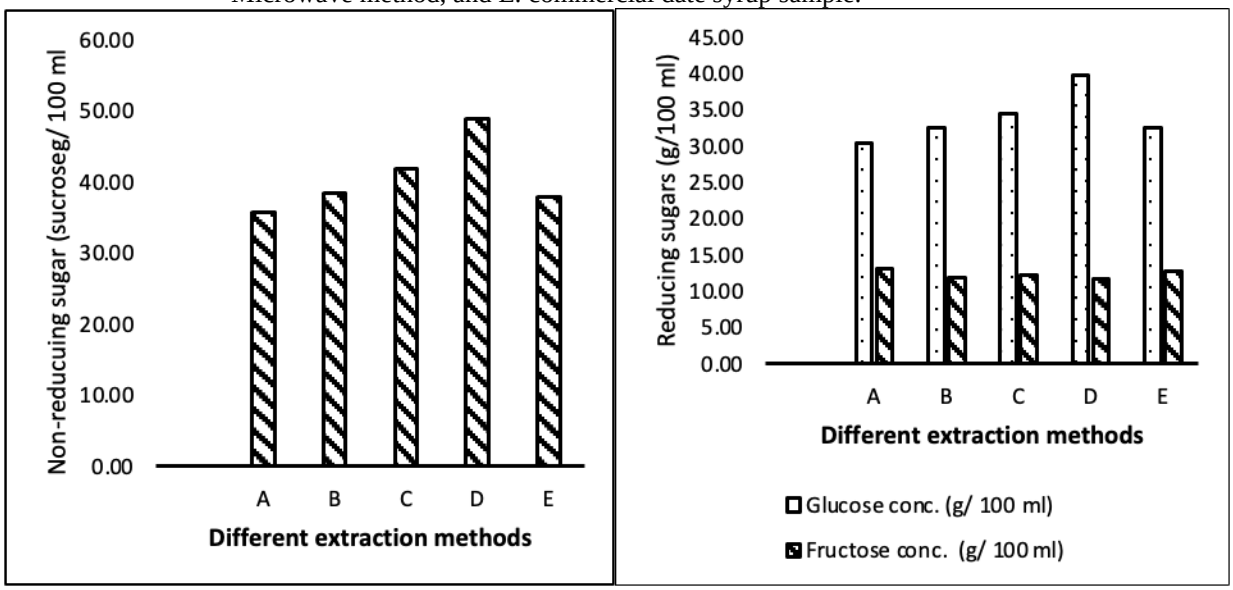


Figure 3. Effect of different extraction methods on reducing and non-reducing sugars of date syrup
Where, A: instant pot (Electrical pressure cooker), B: Traditional oven, C: hotplate method, D: Microwave method, and E: commercial date syrup sample.

3.2. Effect of different extraction methods on physico-chemical characteristics

3.2.1.Reducing and non-reducing sugars **:**it is noteworthy that reducing sugars, being the main components in date fruits varied 52.1–62.8 g/100 g fruit weight with smaller variation between cultivars (6.2%) as reported by Ghnimi et al. (2018).The obtained results in Table 1& Figure 3. demonstrate that extraction methods that followed higher & direct temperatures led to increase in fructose content thus increase in HMF content. So, sample extracted using microwave method (lower temperature) recorded the lowest fructose content followed by traditional oven methods (11.68 & 11.87 g/ 100 ml respectively).The explanation for that the enolization incident of glucose molecules to fructose under higher temperature causes increase formation rate of HMF subsequently. In this context, ***there were two ways of formation HMF*****: direct way:** Dehydration of fructose under higher temperature and converted to HMF

Indirect way: Conversion of glucose molecule to fructose (Enolization) under elevated heat conditions as Ståhlberg, Sørensen, and Riisager (2010) clarified.

Statistic for Table (1) emphasized that there were a proportional relationship between HMF & fructose content. (Pearson correlation = 0.502 at p-value = 0.139). Thus, increasing of fructose content raise the formation of HMF. Concerning with sucrose & glucose content, it was noticed a weak reversible relationship between HMF and sucrose & glucose content (Table1).

3.2.2. TSS %: there were no significant difference between different extraction methods, but the three significant difference between extraction methods and commercial that recorded the highest TSS % (73 .00 %) as shown in Table 2.

3.2.3. pH: hot plate method showed the highest pH value followed by microwave method (4.60 & 4.58 respectively). Meanwhile, commercial sample recorded the lowest pH value (4.40) as present in Table 2 and Figure 4. Correlation analysis (Pearson method) was performed between TSS% &pH. It was illustrated that there were a reversible relationship between TSS% &pH. (Pearson correlation = -0.837 at p-value = 0.077).

This relation was emphasized by Farahnaky et al. (2016). There were a reversible relationship between Brix &pH. Higher concentrations of date syrup caused greater pH reduction, which is due to date syrup when become diluted (the more moisture content %) this able to reduce organic acids in sample.

Table 2. Effect of using different extraction methods on TSS%, pH &TA % of date syrup

Extraction methods	TSS % (Brix)	pH	Titratable acidity %
Instant pot	67.00 ^{b} ± 1.00	4.49 ^{ab} ± 0.07	0.367 ^{ab} ± 0.058
Traditional Oven	67.33 ^{b} ± 0.58	4.54 ^{ab} ± 0.00	0.500 ^{ab} ± 0.183
Hot plate	67.00 ^{b} ± 0.82	4.60 ^{a} ± 0.07	0.225 ^{b} ± 0.050
Microwave	67.50 ^{b} ± 0.50	4.58 ^{a} ± 0.07	0.675 ^{a} ± 0.275
Commercial sample	73 .00 ^{a} ± 1.00	4.40 ^{b} ± 0.00	0.500 ^{ab} ± 0.100

*Means (±SD) followed by different superscripts within each column are significantly different (p≤0.05). Data were statistically analyzed using Minitab (18) program

Instant pot: Electrical pressure cooker

3.2.4. Titeriable acidity %: data in Table 2 and Figure 4. demonstrate that date syrup extracted using microwave methods recorded the highest acidity (0.675 %), while sample extracted using hot plate recorded the lowest (0.225 %) as shown in Table.2). The proposed explanation for that direct & higher heat may contribute to degradation of mallic acid (the dominant acid in date syrup).

3.2.5. Density (g/ cm^3): The importance of measuring density summarized in its relation to moisture content. When density of date syrup is less than particular value, the syrup is could be cheated. As shown in Table 3 and Figure 4 , the commercial sample recorded the highest density value followed by syrup extracted using instant pot (1.64 & 1.62 g/ cm3 respectively), while sample extracted using traditional oven recorded the lowest (1.59 g/ cm3).

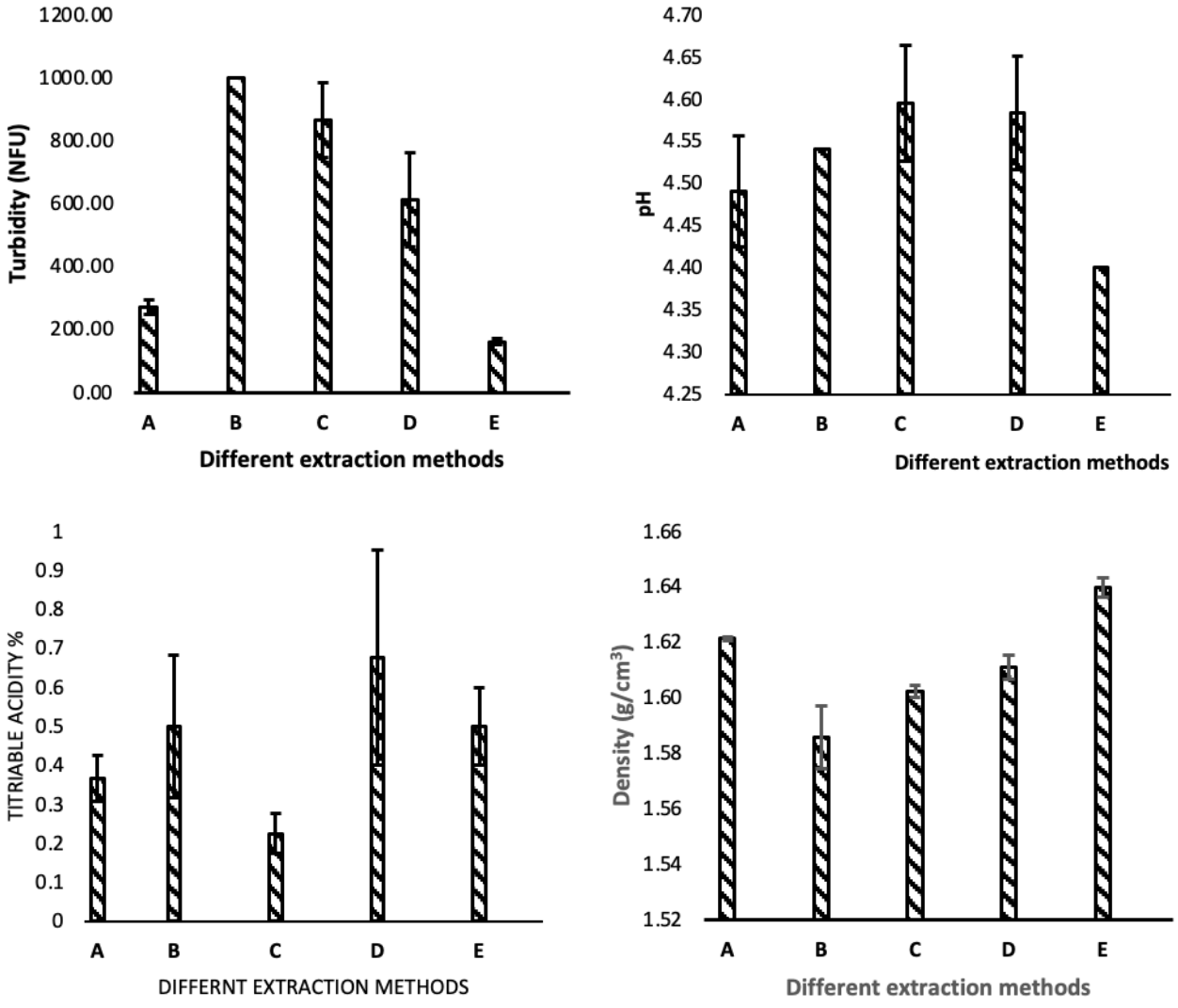


Figure 4. Effect of different extraction methods on pH, turbidity, TA % & density of date syrup
Where, A: instant pot (Electrical pressure cooker), B: Traditional oven, C: hotplate method, D: Microwave method, and E: commercial date syrup sample.

3.2.6. Specific weight: this parameter related to density. Commercial sample recorded the highest specific weight followed by syrup extracted using instant pot (1.36 & 1.35 respectively), while sample extracted using traditional oven recorded the lowest (1.32) as presented in Table 3.

3.2.7. Turbidity (NFU): Turbidity is important parameter that indicates the purity of date syrup, statistical analysis for Table (3) proved that there were significant differences between different extraction methods. Where the oven-extracted sample revealed the highest score followed by heat-extracted sample (1000.00 & 863.33 NFU respectively). While the commercial showed the lowest followed by instant pot (161.00 & 270.33 NFU, respectively.) as shown in Figure 4. Regardless the effect of extraction methods on turbidity and purity of date extract, elevated heat induced coagulation for proteins, causing easier separation, leaving a more clear extract, the higher absorbance and lower turbid (El-Nagga & Abd El–Tawab, 2012). That explains why commercial sample and instant pot-extracted sample recoded lower turbidity values.

Table 3. Effect of using different extraction methods on density, specific weight (Sp) & turbidity of date syrup

Extraction methods	Density (g/cm^3)	Specific weight	Turbidity (NFU)
Instant pot	1.62 ^{ab} ± 0.00	1.35 ^{ab} ± 0.00	270.33 ^{c} ± 24.09
Traditional Oven	1.59 ^{c} ± 0.01	1.32 ^{c} ± 0.01	1000.00 ^{a} ± 00.00
Hot plate	1.60 ^{bc} ± 0.00	1.33 ^{bc} ± 0.00	863.33 ^{a} ± 118.51
Microwave	1.61 ^{b} ± 0.00	1.34 ^{b} ± 0.00	611.67 ^{b} ± 149.25
Commercial sample	1.64 ^{a} ± 0.00	1.36 ^{a} ± 0.00	161.00 ^{c} ± 8.72

*Means (±SD) followed by different superscripts within each column are significantly different ($p \leq 0.05$). Data were statistically analysis using Minitab (18) program. Instant pot: Electrical pressure cooker

3.2.8. Electrical conductivity: it gains an especial importance it indicate the minerals content in liquid food sample. Data tabulated in Table 4 and drawn in Figure 6 proved that different extraction method influenced the EC of different samples. The highest value was for oven-extracted followed by heat-extracted (35.44 & 35.25 mS/cm respectively). Meanwhile, commercial sample showed the lowest followed by microwave extracted sample (26.73 & 30.47 mS/cm respectively). It was supposed that higher heat in commercial sample and microwave radiation in microwave method may causes reduction in minerals content.

Moreover, Pearson correlation was carried out for the relation between two variables (EC & turbidity). It was observed that there were a positive relationship between EC & turbidity (Pearson correlation = 0.692 at p-value = 0.195).

3.2.9. Total ash %: As a result for difference incident in EC between different methods of extraction. Subsequently, there was a significant difference between date syrup samples extracted by different methods. Oven-extracted sample recorded the highest minerals content (ash) followed by heat extracted sample (2.85 & 2.83 % respectively). While commercial sample showed the lowest followed by microwave- extracted sample (2.13

& 2.44 % respectively) as shown in Table 4 and Figure 6. Calculated total ash % in this study was in agreement with Farahnaky et al. (2016). Where they determined Total Ash % in date syrup in similar value (2.18 ± 0.01). For explanation for the different ash content of different extracted samples. It could be notice that minerals content greatly affected by tannin content. Where tannin compounds reduces the bioavailability of minerals (Marin, Siqueira, & Arruda, 2009) lead to decreasing ash. It was appeared that elevated heat (in commercial sample) and microwave may increase tannin content within possible chemical polymerization, thus mineral and ash content decreased. On the other side, tannin content may didn't change in oven-extracted sample. So, it recorded the highest minerals content (ash). In this context, Hassan, Osman, Rushdi, Eltayeb, and Diab (2009) found out duel reversed responses to gamma irradiation. Where irradiation increased tannin content in sorghum (That is in agreement with our findings). While gamma irradiation significantly reduced tannin content of maize cultivar. It worth to mention that the change in tannin content affected by heating temperature, irradiation and food material. In previous study (Duodu, *et al.,* 1999), it is reported that cooking and gamma irradiation caused significant reduction in phytic acid level of sorghum. Similarly, treatment of soybean seeds with gamma irradiation, alone or in combination with soaking reduced the level of phytate compared to untreated seeds (Sattar & Akhtar, 1990).

3.2.10. Sulphated ash %: It tends to have done so total ash% (Table 4 & Figure 6) the relationship between EC and sulphated ash summarized in following equation:

Sulphated Ash % = 0.121 EC - 0.097 (1)
(Sancho et al., 1991).

Table 4. Effect of using different extraction methods on EC, total ash & sulphated ash of date syrup

Extraction methods	Electrical conductivity (EC)	Total Ash (%)	Sulphated Ash (%)
Instant pot	34.33 ^{ab} ± 0.37	2.76 ^{ab} ± 0.03	4.06 ^{ab} ± 0.04
Traditional Oven	35.44 ^{a} ± 1.06	2.85 ^{a} ± 0.09	4.19 ^{a}± 0.13
Hot plate	35.25 ^{ab}± 3.17	2.83 ^{ab} ± 0.26	4.17 ^{ab}± 0.38
Microwave	30.47 ^{bc} ± 2.25	2.44 ^{bc} ± 0.19	3.59^{bc}± 0.27
Commercial sample	26.73 ^{c} ± 0.05	2.13 ^{c} ± 0.00	3.14 ^{c}± 0.01

EC should be expressed in mS / cm or mS / ml. EC should be measured at 20 ^{o} C. If temp > 20, we should use correction factor. Total Ash % = 0.083 EC - 0.092, Sulphated Ash % = 0.121 EC - 0.097.
Data were statistically analyzed using Minitab (18) program.
*Means (±SD) followed by different superscripts within each column are significantly different ($p \leq 0.05$).

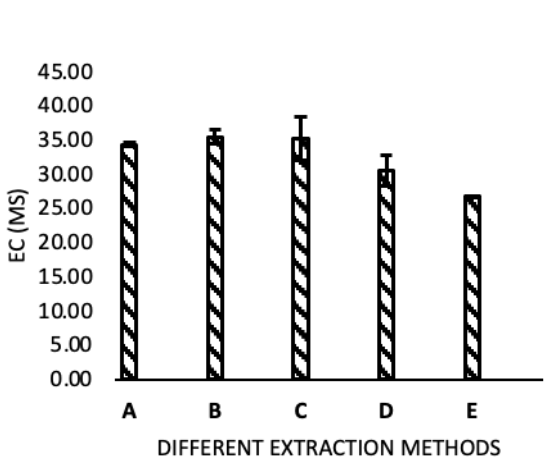
Table 5. Effect of using different extraction methods on color, total phenol & antioxidant activity of processed date syrup

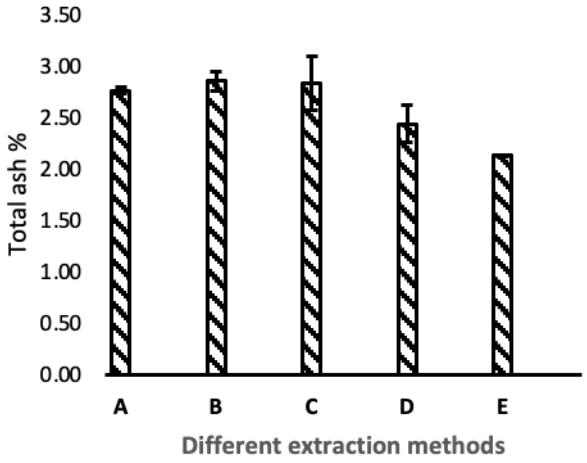
Extraction methods	Color (non-enzymatic browning)	Total phenol (TPC) (mg GAE/ 100 g)	Antioxidant Activity %(AO)
Instant pot	0.614 ^{a} ± 0.002	395.43 ^{b} ± 12.12	43.78 ^{a} ± 8.51
Traditional Oven	0.561 ^{b} ± 0.004	249.60 ^{c} ± 13.52	38.10 ^{a} ± 6.67
Hot plate	0.511 ^{c} ± 0.022	291.14 ^{c} ± 1.09	44.53 ^{a} ± 6.48
Microwave	0.551 ^{bc} ± 0.011	288.62 ^{c} ± 28.28	40.56 ^{a} ± 4.74
Commercial sample	0.656 ^{a} ± 0.006	493.34 ^{a} ± 13.52	35.64 ^{a} ± 1.84

TCP= c * V / m, c : Gallic acid concentration obtained from std. curve (mg/ L), V : Volume of extract in ml (or DF : dilution factor = 20 ml), m: mass of extraction in g. For conversion of (mg / L) to (mg / 100 g): TPC=(c * V)* 100 / 1000.

*Means (±SD) followed by different superscripts within each column are significantly different ($p \leq 0.05$). Data were statistically analyzed using Minitab (18) program.

Figure 5. The appearance (color) of different – extracted date syrup (1: Instant pot, 2: Traditional oven, 3: Hot plate (100± 5 C), 4: microwave)





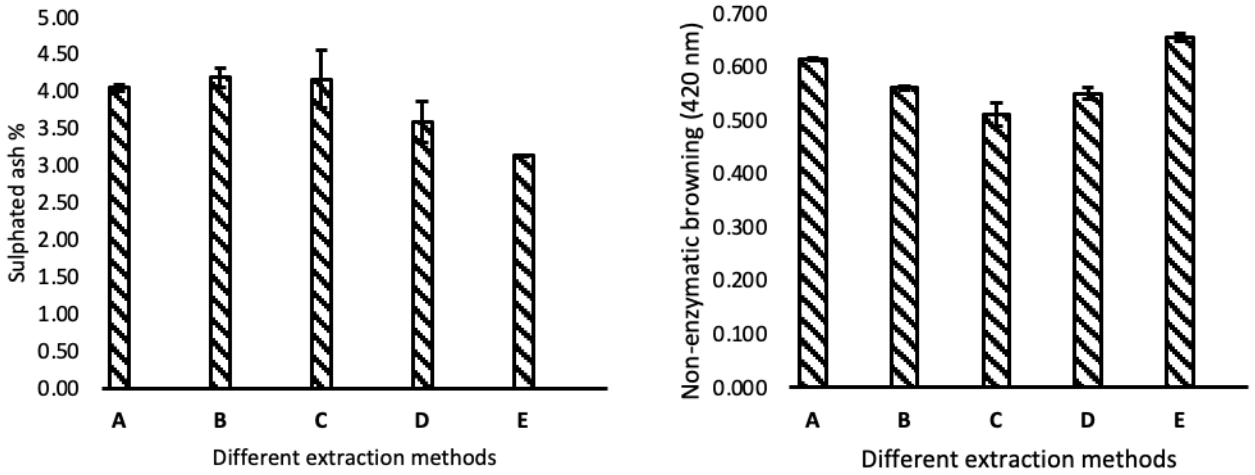


Figure 6. Effect of different extraction methods on EC, total ash %, sulphated ash %, and browning of date syrup. Where, A: instant pot (Electrical pressure cooker), B: Traditional oven, C: hotplate method, D: Microwave method, and E: commercial date syrup sample.

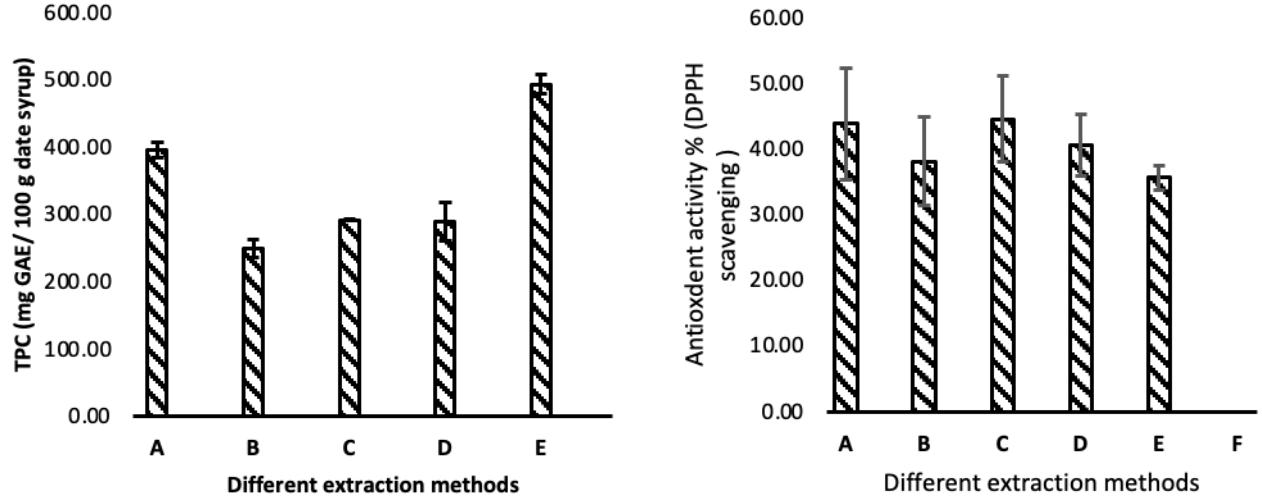


Figure 7. Effect of different extraction methods on total phenolic compounds (T.P.C) & Antioxidant activity % (AO %) of date syrup. Where, A: instant pot (Electrical pressure cooker), B: Traditional oven, C: hotplate method, D: Microwave method, and E: commercial date syrup sample.

3.2.11. Color (non-enzymatic browning): Data shown in Table 5 and Figure 5 & 6demonstrated that different extraction methods showed significantly different degrees of color (non- enzymatic browning). The highest value for commercial followed by instant pot; 0.656 and 0.614, respectively. In agreement with what Hashem et al. (2017) have reported, commercial sample recorded the highest value of color (browning). Where the lowest value were for sample extracted using heater (100± 5 °C) followed by microwave and traditional oven; 0.511, 0.551 & 0.561 respectively. That could be explained that extraction methods that following higher temperature may increase the extraction of pigments and polyphenols of processed fruits unlike the treatments that do not use higher temperature like microwave and traditional oven. In this context, Millard reaction products

increases by increase of extraction temperature thus may increase non-enzymatic browning.

3.2.12.Total phenol content (TPC): data presented in Table 5 and Figure 7 revealed that the commercial date syrup sample has the highest T.P.C. (mg GAE/100 g) followed by sample extracted with instant pot and extracted by heat (100 ± 5 °C): 493.34, 395.43 & 291.14 respectively. While date syrup extracted using traditional oven recorded the lowest T.P.C content followed by sample extracted by microwave: 249.60 & 288.62 respectively. In a previous study carried out by Farahnaky et al. (2016), T.P.C content of date syrup determined in similar values (T.P.C. of date syrup: 453.04 (mg GAE/100g sample). Statistical analysis revealed that there were a strong relationship between Color (non- enzymatic browning and T.P.C. (Pearson correlation = 0.89 at p-value = 0.043).

3.2.13. Antioxidant activity % (AO %): as presented in Table 5, the statistical analysis proved that there were no significant difference ($p \geq 0.05$) between different extraction methods and commercial sample in antioxidant activity (AO %). Thus the extraction methods did not significantly affect the antioxidant activity (Fig.6). IC_{50} of phenolic extract of date syrup was generally calculated (IC_{50}= 540 ug/ml). A lower IC_{50}indicates better radical scavenging as of date syrup as Farahnaky et al. (2016) clarified.

3.3. Effect of different extraction methods on microbial properties of date syrup

Microbial analysis of date syrup was examined and tabulated in Table 6. Regardless total bacterial count, the commercial date syrup sample showed the highest total bacterial count, followed by sample extracted using traditional oven (2.08 x 10^3 &1.62 x 10^3cfu/ ml respectively). While date syrup extracted using instant pot contained the lowest bacterial count (1.16 x 10^3cfu / ml) as shown in Figure.8. That refers to the pressure and higher temperature conditions of instant pot method that reduces the microbial growth in date syrup sample to minimum level. It can be concluded that instant pot methods produced the most sanitized date syrup sample with minimum level of bacterial count.

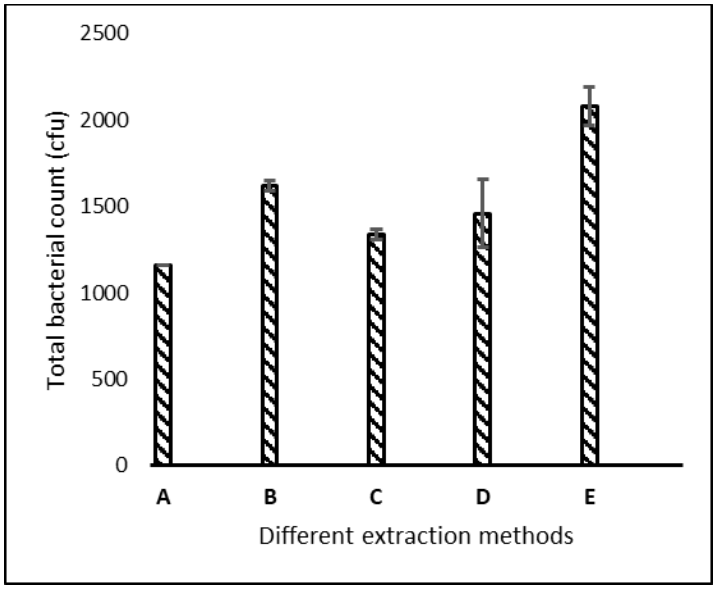


Figure 8. Effect of different extraction methods on total bacterial count (CFU) of date syrup
Where, A: instant pot (Electrical pressure cooker), B: Traditional oven, C: hotplate method, D: Microwave method, and E: commercial date syrup sample.

Table 6. Effect of different extraction methods on total microbial count of processed date syrup compared with commercial sample

Extraction methods	Total bacterial count in date syrup (CFU x10^3/ ml)	Total Fungal count (CFU/ ml)
Instant pot	1.16	Nil
Traditional Oven	1.62	5x10
Hot plate	1.34	Nil
Microwave	1.46	Nil
Commercial sample	2.08	Nil

CFU: Colony forming unit (cell/ml) of Siwi dibs.
Data were statistically analyzed using Minitab (18) program.
Instant pot: Electrical pressure cooker.

Total mold & yeast counts: Generally the elevated of sugar concentration in date syrup (68 - 75 %) is considered a preservative agent and inhibit the yeast & mold growth.

So the different extracted date syrup sample did not contain fungal growth.

Except for the samples extracted using traditional oven (Table 6). This may be due to the sample contained air bubbles (where aeration conditions increase fungal growth).

3.4. Effect of different extraction methods on sensory evaluation of date syrup

Sensory evaluation of different samples of date dibs were evaluated and the obtained results were tabulated in Table 7.

The data ascertained that the highest color scores were recorded for commercial sample followed by instant (5.00 & 4.00 respectively), while the lowest score for oven extracted (2.33) as shown in Figure 9.

For purity, the most pure sample was instant pot sample followed by commercial (4.67 & 4.33 respectively).

While the lowest purity was recorded by oven- extracted sample (Table7 & Fig.9).

Regarding with viscosity, commercial sample showed the highest viscosity followed by microwave method, however instant pot showed the lowest (Figure 9).

Concerning with sweetness, the commercial sample pertained the highest sweet taste, but the other samples were not significantly different (Table 7). As for consistency: Hot plat sample showed the highest consistency taste (Figure 9).

Although extraction method affected the odor intensity of date syrup slightly, these difference were non-significant ($p \geq 0.05$), commercial sample characterized with burned sugar odor (Table 7).

Finally, the panelists were asked to assess the overall acceptability of date syrup using (9.point hedonic scale), where hot plate sample achieved the highest score (7± 1) followed by oven (7 ± 0.58).

While both of microwave and instant pot methods recorded the same score (6.00± 0.58). However, commercial sample showed the lowest (5± 1.15) as clarified in Table7.

Table 7. Effect of different extraction methods on sensory properties of date syrup

Extraction methods	Appearance			Taste			Odor			Overall acceptability
	Color (1-5)	Purity (1-5)	Flowing rate (1-5)	Sweetness (1-5)	Consistency (Softness; 1-3)	Sugar crystal	Good smell	Burned sugar smell	Stran -ge smell	
Instant pot	4.00 ^{a} ± 0.00	4.67 ^{a} ± 0.58	1.00 ^{c} ± 0.00	3.00 ^{b} ± 0.00	2.00 ^{b} ± 0.00	No	3.67 ^{a} ± 1.15	No	No	6.00 ^{ab} ± 0.58
Oven	2.33 ^{b} ± 0.58	2.33 ^{b} ± 0.58	2.33 ^{bc} ± 1.15	3.00 ^{b} ± 0.00	2.00 ^{b} ± 0.00	No	3.33 ^{a} ± 0.58	No	No	7.00 ^{ab} ± 0.58
Hot plate	2.67 ^{b} ± 0.58	3.33 ^{ab} ± 1.15	2.33 ^{bc} ± 1.15	3.00 ^{b} ± 0.00	3.00 ^{a} ± 0.00	No	3.00 ^{a} ± 1.00	No	No	7.00 ^{a} ± 1.00
Microwave	2.67 ^{b} ± 0.58	3.67 ^{ab} ± 0.58	3.67 ^{ab} ± 1.15	3.00 ^{b} ± 0.00	2.00 ^{b} ± 0.00	No	2.00 ^{a} ± 1.00	No	No	6.00^{ab} ± 0.58
Commercial	5.00 ^{a} ± 0.00	4.33 ^{a} ± 0.58	5.00 ^{a} ± 0.00	5.00 ^{a} ± 0.00	1.00 ^{c} ± 0.00	No	3.67 ^{a} ± 1.53	Yes	No	5.00 ^{b} ± 1.15

Means (±SD) followed by different superscripts within each column are significantly different (p≤0.05). Data were statistically analyzed using Minitab (18) program

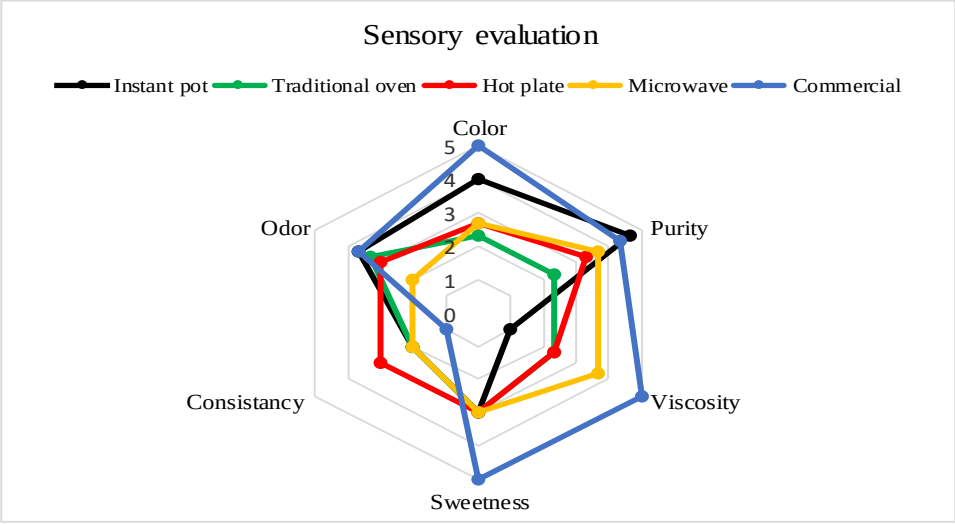


Figure 9. Spider web clarifying sensory attributes of date syrup extracted by different methods

4. Conclusions

From the obtained results, it can be concluded that among lab-processed date syrup, microwave extraction method presented the best results concerning with safety of the product: the lowest level of toxic hazard (5-HMF) and high quality characteristics including: the lowest level of fructose, the highest reducing sugar content, pH and titriable acidity %. Hot plate method has shown the highest score of overall acceptability of Panel test. Meanwhile, instant pot extraction methods revealed moderate level of HMF, density, specific weight, turbidity, color and total phenolic compounds.

Furthermore, instant pot extraction method achieved the lowest bacterial count due to temperature & pressure conditions (Most sanitized date syrup sample).

On the other hand, traditional oven extraction method showed good results regarding with low HMF level, the highest EC, Total ash %, sulphated ash %. However, this method showed the highest fungal count. Regarding with other quality characteristics, it revealed the highest score of TSS %, density, specific weight, color and total phenolic compounds. It also recorded the lowest score in turbidity, EC, total ash %, sulphated ash %. So, microwave extraction method is recommended method for extraction of date syrup with minimal level of HMF and with higher quality characteristics.

5. References

AOAC. (1999). Official Method of Analysis of Association of Official Analysis Chemists. . *15 th ed. Arlington U.S.A. AOAC*, Pp 1-50.

AOAC (2000). Official method 942.15 acidity (Titriable) of fruits product read with A.O.A.C official method 920 .149 preparation of test sample *17. the edn*.

Abd El-Hady A. El-Sayed , Y., Khaled M., Shatta, Adel A. and, & El-Samahy, S. K. (2014). Physico-Chemical , Rheological and Sensory Properties of Date P*hoenix dactylifera* var. Shamia Sheets. *Agriculture and Veterinary Sciences Journal, 7*(1), 59-68. doi:DOI:10.12816/0009462

Abraham, K., Gürtler, R., Berg, K., Heinemeyer, G., Lampen, A., & Appel, K. E. (2011). Toxicology and risk assessment of 5-Hydroxymethylfurfural in food. *Molecular Nutrition & Food Research, 55*(5), 667-678.

Al-Farsi, M. A. (2003). Clarification of date juice. *International Journal of Food Science & Technology, 38*(3), 241-245.

Bakhiya, N., Monien, B., Frank, H., Seidel, A., & Glatt, H. (2009). Renal organic anion transporters OAT1 and OAT3 mediate the cellular accumulation of 5-sulfooxymethylfurfural, a reactive, nephrotoxic metabolite of the Maillard product 5-hydroxymethylfurfural. *Biochemical Pharmacology, 78*(4), 414-419.

Baltaci, C., & Akşit, Z. (2016). Validation of HPLC Method for the Determination of 5-hydroxymethylfurfural in Pestil, Köme, Jam, Marmalade and Pekmez. *Hittite Journal of Science and Engineering, 3*(2), 91-97.

Bauer-Marinovic, M., Taugner, F., Florian, S., & Glatt, H. (2012). Toxicity studies with 5-hydroxymethylfurfural and its metabolite 5-sulphooxymethylfurfural in wild-type mice and transgenic mice expressing human sulphotransferases 1A1 and 1A2. *Archives of Toxicology, 86*(5), 701-711.

Capuano, E., & Fogliano, V. (2011). Acrylamide and 5-hydroxymethylfurfural (HMF): A review on metabolism, toxicity, occurrence in food and mitigation strategies. *LWT-Food Science and Technology, 44*(4), 793-810.

Choudhary, A., Kumar, V., Kumar, S., Majid, I., Aggarwal, P., & Suri, S. (2021). 5-Hydroxymethylfurfural (HMF) formation, occurrence and potential health concerns: Recent developments. *Toxin Reviews, 40*(4), 545-561.

Commission, C. A. (Amended in 2019). Draft Amended Standard for Standard for Honey. *CXS 12–1981*, 1–9.

Duodu, K. G. Minnaar, A., & Taylor, J. R. N. (1999). Effect of cooking and irradiation on the labile vitamins and antinutrient content of a traditional African Sorghum porridge and Spinach relish. *Food Chemistry, 66*(1): 21-27. *doi:10.1016/S0308-8146(98)00070-3*

El-Nagga, E., & Abd El–Tawab, Y. (2012). Compositional characteristics of date syrup extracted by different methods in some fermented dairy products. *Annals of Agricultural Sciences, 57*(1), 29-36.

El-Samahy, S., & Youssef, K. (2009). Physico-chemical and rheological properties of date fruits extract during concentration. *Alex. J. Fd. Sci. & Technol, 6*, 85-93.

El-Samahy, S. K., Adel, A. S., & Khaled, M. Y. (2005). *Production of date pickles*. Paper presented at the Conference: The 6th Arabian Conference For Horticulture, Ismailia, Egypt.

Eshete, Y., & Eshete, T. (2019). A review on the effect of processing temperature and time duration on commercial honey quality. *Madridge Journal of Food Technology, 4*(1), 158-162.

FAO. (2020). *Food and Agriculture organization. Egypt date production report. https://www.fao.org/countryshowcase/selected-product-detail/en/c/1287948/*. Retrieved from https://www.fao.org/countryshowcase/selected-product-detail/en/c/1287948/

Farag, M. R., Alagawany, M., Bin-Jumah, M., Othman, S. I., Khafaga, A. F., Shaheen, H. M., Samak, D., Shehata, A.M., Allam, A.A.,& Abd El-Hack, M. E. (2020). The toxicological aspects of the heat-borne toxicant 5-hydroxymethylfurfural in animals: A review. *Molecules, 25*(8), 1941.

Farahnaky, A., Mardani, M., Mesbahi, G., Majzoobi, M., & Golmakani, M.-T. (2016). Some physicochemical properties of date syrup, concentrate, and liquid sugar in comparison with sucrose solutions. *18*(3), 657-668.

Farhadi, K., Esmaeilzadeh, F., Hatami, M., Forough, M., & Molaie, R. (2016). Determination of phenolic compounds content and antioxidant activity in skin, pulp, seed, cane and leaf of five native grape cultivars in West Azerbaijan province, Iran. *Food Chemistry, 199*, 847-855.

Fathi, G., Rezaei, K., Emam-Djomeh, Z., & Hamed, M. (2013). Decolorization of Iranian Date Syrup by Ultrafiltration. J. Agric. Sci. Techol., 15: 1361-1371.

Ghnimi, S., Al-Shibli, M., Al-Yammahi, H. R., Al-Dhaheri, A., Al-Jaberi, F., Jobe, B., & Kamal-Eldin, A. (2018). Reducing sugars, organic acids, size, color, and texture of 21 Emirati date fruit varieties (*Phoenix dactylifera*, L.). *NFS Journal, 12*, 1-10.

Hashem, H. A., Abd El-Daym, H. H., El-Sharnouby, G., Farghal, S., & Badr, H. A. (2017). The Effect of Extraction Method, Bleaching and Clarification Processes on Quality Second Grade Siwi Date Dibs. *Ind. Eng, 1*, 17-23. doi:DOI:10.11648/j.ie.20170101.12

Hou, Y., Zhang, X., Liu, X., Wu, Q., Hou, J., Su, P., & Guo, Q. (2022). Comparison of the Effects of 5-Hydroxymethylfurfural in Milk Powder Matrix and Standard Water on Oxidative Stress System of Zebrafish. *Foods, 11*(12), 1814.

Hyvönen, L., & TÖRMÄ, R. (1983). Examination of sugars, sugar alcohols, and artificial sweeteners as substitutes for sucrose in strawberry jam. Product development. *Journal of Food Science, 48*(1), 183-185.

Jafarnia, A., Soodi, M., & Shekarchi, M. (2016). Determination and comparision of hydroxy methyl furfural in industrial and traditional date syrup products. *Iranian Journal of Toxicology, 10*(5), 11-16.

Janzowski, C., Glaab, V., Samimi, E., Schlatter, J., & Eisenbrand, G. (2000). 5-Hydroxymethylfurfural: assessment of mutagenicity, DNA-damaging potential and reactivity towards cellular glutathione. *Food and Chemical Toxicology, 38*(9), 801-809.

Lee, Y.C., Shlyankevich, M., Jeong, H.K., Douglas, J. S., & Surh, Y.J. (1995). Bioactivation of 5-hydroxymethyl-2-furaldehyde to an electrophilic and mutagenic allylic sulfuric acid ester. *Biochemical and Biophysical Research Communications, 209*(3), 996-1002.

Lullah-Deh, J. A., Khan, M. E., & Eneji, I. S. (2018). Physicochemical characteristics of honey samples from Mambilla Plateau, Nigeria. *Journal of Biomaterials, 2*(1), 7.

Michail, K., Matzi, V., Maier, A., Herwig, R., Greilberger, J., Juan, H., Cunert, O., & Wintersteiger, R. (2007). Hydroxymethylfurfural: An enemy or a friendly xenobiotic? A bioanalytical approach. *Analytical and Bioanalytical Chemistry, 387*(8), 2801-2814.

Minitab 18, s. s. (2021). [Computer software]. State collage, PA: minitab, Inc. (www.minitab.com).

Mistrello, J., Sirisena, S. D., Ghavami, A., Marshall, R. J., & Krishnamoorthy, S. (2014). Determination of the antioxidant capacity, total phenolic and flavonoid contents of seeds from three commercial varieties of culinary dates. *International Journal of Food Studies*, *3*(1), 34-44.

Naknean, P., Meenune, M., & Roudaut, G. (2009). Changes in physical and chemical properties during the production of palm sugar syrup by open pan and vacuum evaporator. *Asian Journal of Food and Agro-Industry*, 2(4), 448-456.

Pastoriza de la Cueva, S., Álvarez, J., Végvári, Á., Montilla-Gómez, J., Cruz-López, O., Delgado-Andrade, C., & Rufián-Henares, J. A. (2017). Relationship between HMF intake and SMF formation *in vivo*: An animal and human study. *Molecular nutrition & food research, 61*(3), 1600773.

Rahimzadeh, N., Alizadeh, M., & Ghaemmaghami, H. S. J. (2014). Estimated bioaccessibility to 5-hydroxymethylfurfural from frequently consumed dried fruits in Iran. *Journal of Chemical Health Risks (JCHR)* 4 (3), 15- 23.

Ramadan, B. (1998). *Preparation and evaluation of Egyptian date syrup*. Paper presented at the First international conference on date palm. Al-Ain. UAE.

Ramírez-Jiménez, A., García-Villanova, B., & Guerra-Hernández, E. (2000). Hydroxymethylfurfural and methylfurfural content of selected bakery products. *Food Research International, 33*(10), 833-838.

Ranganna, S. (1977). Manual of analysis of fruit and vegetable products. Tata McGraw-Hill.P 634.

Rasheed, A., Cobham, E., Zeighmami, M., & Ong, S. (2012). *Extraction of phenolic compounds from pineapple fruit*. Paper presented at the Conference: The 2nd International Symposium on Processing & Drying of Foods, Vegetables and Fruits (ISPDFVF), University of Nottingham, Malaysia Campus, Malaysia.

Sancho, M., Muniategui, S., Sánchez, M., Huidobro, J., & Simal, J. (1991). Relationships between electrical conductivity and total and sulphated ash contents in Basque honeys. *Apidologie*, *22*(5), 487-494.

Sesta, G. (2006). Determination of sugars in royal jelly by HPLC. *Apidologie, 37*(1), 84-90.

Severin, I., Dumont, C., Jondeau-Cabaton, A., Graillot, V., & Chagnon, M. C. (2010). Genotoxic activities of the food contaminant 5-hydroxymethylfurfural using different in vitro bioassays. *Toxicology Letters, 192*(2), 189-194.

Shahdadi, F., Mirzaei, H., & Daraei Garmakhany, A. (2015). Study of phenolic compound and antioxidant activity of date fruit as a function of ripening stages and drying process. *Journal of Food Science and Technology, 52*(3), 1814-1819.

Shahein, M. R., Atwaa, E. S. H., Elkot, W. F., Hijazy, H. H. A., Kassab, R. B., Alblihed, M. A., & Elmahallawy, E. K. (2022). The impact of date syrup on the physicochemical, microbiological, and sensory properties, and antioxidant activity

of bio-fermented camel milk. *Fermentation, 8*(5), 192.

Shapla, U. M., Solayman, M., Alam, N., Khalil, M., & Gan, S. H. (2018). 5-Hydroxymethylfurfural (HMF) levels in honey and other food products: effects on bees and human health. *Chemistry Central Journal, 12*(1), 1-18.

Ståhlberg, T., Sørensen, M. G., & Riisager, A. (2010). Direct conversion of glucose to 5-(hydroxymethyl) furfural in ionic liquids with lanthanide catalysts. *Green Chemistry, 12*(2), 321-325.

Sulieman, A. M. E., & Elkashif, M. E. (2009). Quality and evaluation of syrup from local date (Phoenix dactylifera L.) cultivars. *Gezira Journal of Agricultural Science, 7*(2).

Svendsen, C., Husøy, T., Glatt, H., Paulsen, J. E., & Alexander, J. (2009). 5-Hydroxymethylfurfural and 5-sulfooxymethylfurfural increase adenoma and flat ACF number in the intestine of Min/+ mice. *Anticancer Research, 29*(6), 1921-1926.

Teubner, W., Meinl, W., Florian, S., Kretzschmar, M., & Glatt, H. (2007). Identification and localization of soluble sulfotransferases in the human gastrointestinal tract. *Biochemical Journal*, 404(2), 207-215.

Yang, W., Zhang, C., Li, C., Huang, Z. Y., & Miao, X. (2019). Pathway of 5-hydroxymethyl-2-furaldehyde formation in honey. *Journal of Food Science and Technology*, *56*(5), 2417-2425.

Yousif, A., Al-Shaawan, A., Mininah, M., & El-Taisan, S. (1987). Processing of date-preserve, date-jelly and date-kutter. *Date Palm Journal (FAO/NENADATES), 5*(1), 73-86.

Zhang, C.-R., Aldosari, S. A., Vidyasagar, P., Shukla, P., & Nair, M. G. (2015). Determination of the variability of sugars in date fruit varieties. *Journal of Plantation Crops, 43*(1), 53-61.