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OPTIMIZATION OF A GREEN ULTRASOUND-ASSISTED EXTRACTION OF PHENOLICS AND IN *VITRO* ANTIOXIDANT POTENTIAL OF DATE FRUIT (*PHOENIX DACTYLIFERA* L.)

OUARDA DJAOUDENE AND HAYETTE LOUAILECHE*

Laboratoire de Biochimie Appliquée, Faculté des Sciences de la Nature et de la Vie, Université de Bejaia, 06000, Bejaia, Algérie
* Corresponding author: halouailec@gmail.com

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The fruit of the date palm, *Phoenix dactylifera* L., is one of the richest fruit-based in biologically functional phytochemicals. The efficient extraction of phenolic compounds in this fruit by means of rapid, low cost, environment-free methods would be a desirable achievement. A deep eutectic solvent (DES) based on lactic acid and sucrose was considered as extraction solvent. DES are green solvents composed of natural compounds and characterized by their negligible volatility, high solubilization ability and tunable selectivity. In this line, the present study was based on a step-by-step optimization of the extraction, taking into consideration basic parameters, including solvent composition (lactic acid/sucrose ratio), solvent concentration (0–100%) sample/solvent ratio (100/15 to 300/15 mg/ml) and extraction time (3-40 min). The total phenolic compound content (TPC) was used to evaluate antioxidant content of the extracts. DPPH radical scavenging activity was used to evaluate antioxidant potential. Experimental results showed that all extraction parameters investigated had significant effects ($p < 0.05$) on TPC and antioxidant activity of the extracts. The best antioxidant activity (948.1mgAAE)/100 g) and the highest TPC (1393.5mgGAE)/100 g) were obtained with double extraction using lactic acid/sucrose mixture with ratio of 3:1 at a concentration of 100%, with a sample/solvent ratio of 100mg/15ml, for 40 min. These results suggested that the mixture lactic acid/sucrose may be an ideal candidate for use in eco-friendly extraction processes. As a function of investigated extraction parameters, phenolic contents were positively correlated with the antioxidant activity.

Keywords: Date fruit, green extraction, natural antioxidants, phenolics, natural deep eutectic solvent.

Introduction

Fruits are a good source of nutrients (vitamins, minerals, carbohydrates, etc.) and bioactive compounds that possess antioxidant, anti-inflammatory, anticancer, and anti-aging activities. These properties of the fruits are due to the presence of phenolic acids, flavonoids, tannins, etc. Most of these chemicals are used in nutraceutical preparations (Li *et al.*, 2013). Phenolics are secondary metabolites

widely available in fruits. Some of these compounds are particularly known for their preventive effects against reactive oxygen species and free radicals (Saini *et al*., 2012). It has been reported that fruit phenolics were used in treatment or prophylaxis of cardiovascular diseases, and digestive health (Kua *et al*., 2015). Among others, fruit of *Phoenix dactylifera* L. (*Arecaceae*), also known as date palm, plays an important role in social and economic perspective of the people living in the oasis of the Middle East by the virtue of its nutritional and pharmacological properties (Baliga *et al*., 2011). The fruit serves as an important source of nutrition in an arid region hostile to habitation of plants, its rich in certain nutrients and provide a good source of rapid energy, due to their high carbohydrate content (Sheikh *et al*., 2016). The date fruit has been utilized since ancient times as an important staple food and in ethnomedicine in different regions of the world (Nasir *et al*., 2015). The date palm fruits are consumed worldwide and are one of the most important commercial crops in the world (Mrabet *et al*., 2016). Date fruit cannot only provide antioxidant, antimutagenic, and immunomodulatory benefits to health but also has diverse medicinal properties, including gastroprotective, hepatoprotective, and nephroprotective properties (Mohamed *et al*., 2014).

Many studies have been conducted on phenolic compounds and antioxidant capacity in date fruit. However, in the literature available at present, the authors used toxic solvent such as ethyl acetate and methanol, etc. for extraction in depth study on extraction procedure is required so that the species can suitably be harnessed for its beneficial effects. The extraction of antioxidants is an important process, since these bioactive substances are often used in functional foods, food additives, nutraceuticals, pharmaceuticals and cosmetic industries. However, various factors such as extraction temperature, time, along with solvent concentrations, pH, solid liquid ratio, etc. are known to influence the extraction process of phenolics (Ng *et al*., 2012). Conventional solvents are often highly flammable and toxic. Thus, numerous investigations are currently focusing on the replacement of hazardous solvents with more environment-friendly alternatives. Therefore, the objective of the current investigation was to optimize extraction conditions using non-toxic solvents (mixtures lactic acid/sucrose).

Deep eutectic solvents (DES) are mixtures of organic compounds and are made up of different components, such as choline, urea, and sugars. These solvents contain components that are abundant in food, being cheap, sustainable, and safe. Interestingly, some DES show a very high solubilization ability of both non-polar and polar compounds, even macromolecules. This predicts a great potential for DES as solvents in the extraction of valuable secondary metabolites for their application in the food or pharmaceutical industry (Dai *et al*., 2013a).

Recent reports support the idea that lactic acid/glucose aqueous mixtures may be very effective in extracting phenolic compounds, yet the information provided is rather limited to fully assess its potential with regard to a process destined for efficient phenolic recovery from plant material (Paradiso *et al*., 2016).

Lactic acid/sucrose mixture is a natural liquid, unexamined for extraction processes of phenolic compounds, although it possesses interesting characteristics, such as lack of toxicity, lack of flammability, as well as low cost.

On the basis of this concept, the study presented herein was undertaken with the view to optimizing phenolic extraction from date fruits. The extractions performed were assisted by ultrasounds and the optimization included factors crucial to the process, such as ratio of lactic acid/sucrose aqueous solutions, solvent concentration, sample to solvent ratio and extraction time.

Materials and methods

The extraction process is an important step for isolating and identifying phenolics. To optimize the parameters of the extraction of bioactive compounds from date palm fruit, a one factor one-time approach was deployed, also known as single experiment, in which only one factor is variable at one time while all others are kept constant. The screening carried out was designed to evaluate the effect of four selected variables, that is, the molar ratios of aqueous mixture of lactic acid/sucrose (DES), solvent concentration, sample/liquid ratio and extraction time.

Chemical reagents

Folin–Ciocalteu reagent, lactic acid, sodium carbonate and sucrose (D-(+)-sucrose) were from Biochem, Chemopharma (Georgia, USA; ≥ 99.0% purity); gallic acid from Prolabo (Montreuil, France) and diphenyl-picrylhydrazyl (DPPH) from Sigma Chemical (Sigma-Aldrich GmbH, Germany).

Plant material

Two ripe cultivars of date palm fruits locally known, as Ourrous (OUR) and Ouksaba (OUK) were harvested in November 2016. Fruits were collected from Ghardaïa (Algeria). They were selected identically in terms of size, ripening stage, without infection and were stored in paper bags at 4°C until use.

Deep Eutectic Solvent (DES) preparation

Mixtures of lactic acid and sucrose with molar ratio ranged from 3:0 to 3:2 in distilled water were prepared. The DES evaluated in this study was obtained by heating with stirring at 50°C for a period of time ranging from 30 minutes to 2h due to the addition of water until obtaining a clear solution.

Extraction procedure

A quantity of 0.2 g of crushed and pitted date fruits was homogenized in 15 ml of extracting solvent (DES) based on lactic acid and sucrose; the mixture was shaken for 15 minutes using a magnetic stirrer (VELP Scientifica, Italy) and then subjected to ultrasound-assisted extraction (Sonics Vibra Cell, VCX130PB, USA). Solvent ratio and concentration, sample/solvent ratio and extraction time were set according to the single factor experiment. The extracts were then centrifuged at 5000g (Nüve NF 200, Ankara, Turkey) for 15 min and paper filtered.

Extraction conditions

Solvent molar ratio

By fixing solid/solvent ratio (200 mg/15 ml) and extraction time (shaken 15 min and sonicated for 3min) samples were extracted with water and different ratio of DES (3:0, 3:0.5, 3:1, 3:1.5 and 3:2).

Solvent concentration

Using the best extraction molar ratio of DES selected in the previous step, samples were extracted with solvent 0%, 25%, 50%, 75% and 100% (v/v) by fixing the solid/solvent ratio (200 mg/15 ml) and extraction time.

Solid-to-solvent ratio

Samples were extracted using the best solvent type and the best solvent concentration. The extraction was repeated by varying the solid-to-solvent ratio (100/15, 150/15, 200/15, 250/15, and 300 mg/15 mL), while fixing the extraction time at 3 min.

Extraction time

Samples were extracted by using the best solvent concentration and sample/solvent ratio. The extracts were prepared by varying the extraction time (3, 9, 15, 21, 27, 33 and 40 min).

Total phenolic compound contents (TPC)

Total phenolics were determined using Folin-Ciocalteu reagent (Al-Farsi *et al*, 2005). Folin-Ciocalteu reagent (750 µL) and 750 µL of sodium carbonate [6% (w/v)] were added to 100 µL of extract. After 60 min, the absorbance was measured at 760 nm (UVline 9400 UV-visible Secomam, France). The TPC was expressed as mg of gallic acid equivalent (GAE) per 100 g of fresh matter (FM) using a calibration curve.

2,2-Diphenyl-1-picryl-hydrazyl (DPPH) radical-scavenging activity (RSA)

The free radical-scavenging activity of the extracts was measured as described by Brand-Williams *et al.* (1995). An aliquot (100 µL) of the extract was added to 1mL of a DPPH (60 µM). The absorbance was measured at 517 nm after 30 min of reaction. Ascorbic acid was used as a standard and the scavenging activity of the date extract was expressed as mg ascorbic acid equivalents per 100 g fresh weight (mg AAE/100g FW).

Statistical analysis

Results were analyzed using the Statistica software 5.5. All values are expressed as mean ± standard deviation (SD) of triplicate extractions and replicate assays. One-way analysis of variance (ANOVA) with the LSD (least significant difference) test was used to determine significant differences ($p<0.05$) among the means.

Results and discussion

Solvent molar ratio

The choice of extraction solvents is critical as it usually determines the type and amount of phenolic compounds being extracted. Solvents with different polarities can have large effects on phenolic extraction efficiency. The hydroxyl group,

length of hydrocarbon and molecular size of a phenolic compound determine its solubility in solvent (Dai and Mumper, 2010). In the current study, we have investigated the effects of lactic acid and sucrose mixtures solvent system on phenolic extraction of date palm fruit compared to water. Besides, the use of mixtures as a solvent has tremendous benefits as a green extraction solvent because lactic acid and sucrose are not only inexpensive and environmentally benign; but it is also non-flammable, nontoxic, providing opportunities for clean processing and pollution prevention.

All these compounds are of natural origin with no reports pertaining to toxic or adverse effects.

To assess the effect that might be mixing exerted by lactic acid and sucrose, a series of solutions with variable molar ratio was tested and the extraction efficiency was evaluated by determining two representative indices, TPC and RSA. The range of molar ratio was chosen on the basis of already tested in a wider range prior to optimization.

The selected optimum conditions for extracting phenolics from investigated date fruit cultivars (200 mg/15ml as sample to solvent ratio, solvent concentration 100% and 3min extraction time), were used to extract phenolics with five different ratios of lactic acid/sucrose mixtures (Figure 1). It was observed that Solvent ratio had a significant influence ($p < 0.05$) on TPC and antioxidant activity (RSA) of extracts. The highest TPC (Figure 1A) and the best antioxidant activity (Figure 1B) for both OUR and OUK varieties were observed at lactic acid/sucrose ratio of 3:1.

Garcia *et al*, (2016) have investigated the extraction efficiency of phenolics from virgin olive oil using the choline chloride: lactic acid (1:2) and choline chloride: sucrose (1:1 and 4:1) mixtures.

Solvent concentration

After the determination of the best lactic acid/sucrose ratio for antioxidant extraction (3:1), the optimal water proportion in DES was determined. Aqueous lactic acid/sucrose mixtures molar ratio 3:1 at different concentrations (0%, 25%, 50%, 75% and 100%) was employed as the extracting solvent in the current study.

The best solvent concentration for TPC recovery and antioxidant activity of OUR and OUK date varieties was 100% (Figure 2). The statistical analysis of the results revealed that mixture concentration had significant effects ($p < 0.05$) on total phenolic contents and antioxidant activity of date fruit extracts. Phenolic content and the antioxidant potential increased with the increasing proportion of mixtures lactic acid/sucrose in the water up to 100% with values of 1056.5 mg GAE/100 g for OUR variety, and 410.3 mg GAE/100 g for OUK variety, and antioxidant activities of 55.8 and 108.4mg AAE/100 g, respectively.

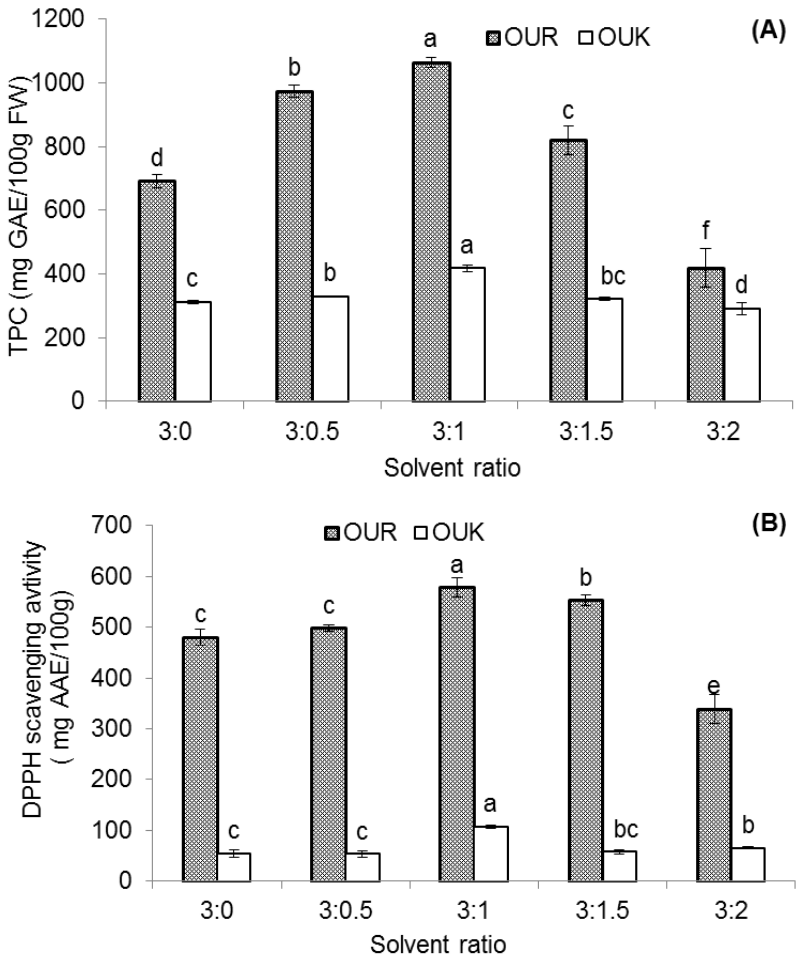

Figure 1. Influence of lactic acid/sucrose ratio on the total phenolic content(A) and antioxidant activity (B) of date fruit extracts.

Values with different letters are significantly different ($p<0.05$).

Extraction with water (0%) showed low TPC recovery (575.63 mg GAE/100 g for OUR variety and 304.12 mg GAE/100 g for OUK variety), and antioxidant activities of 400 and 67.2 mg AAE/100 g, respectively, due to low solubility of these antioxidants in water. The use of pure water for extraction presents some problems, such as dissolution of undesired proteins and polysaccharides, particularly at high temperatures. Water dissolves many nutrients, like sugar and protein. Al-Farsi and Lee (2008) reported that water extraction shows low ability to extract phenolics in date seeds. The obtained results showed that DES dilution with water decreased its efficiency, performing much better with no addition of water. Phenolic recovery from plant materials is influenced by solubility in the solvent used for the extraction process. Furthermore, the solvent polarity plays a key role in increasing the phenolic solubility (Naczk and Shahidi, 2006).

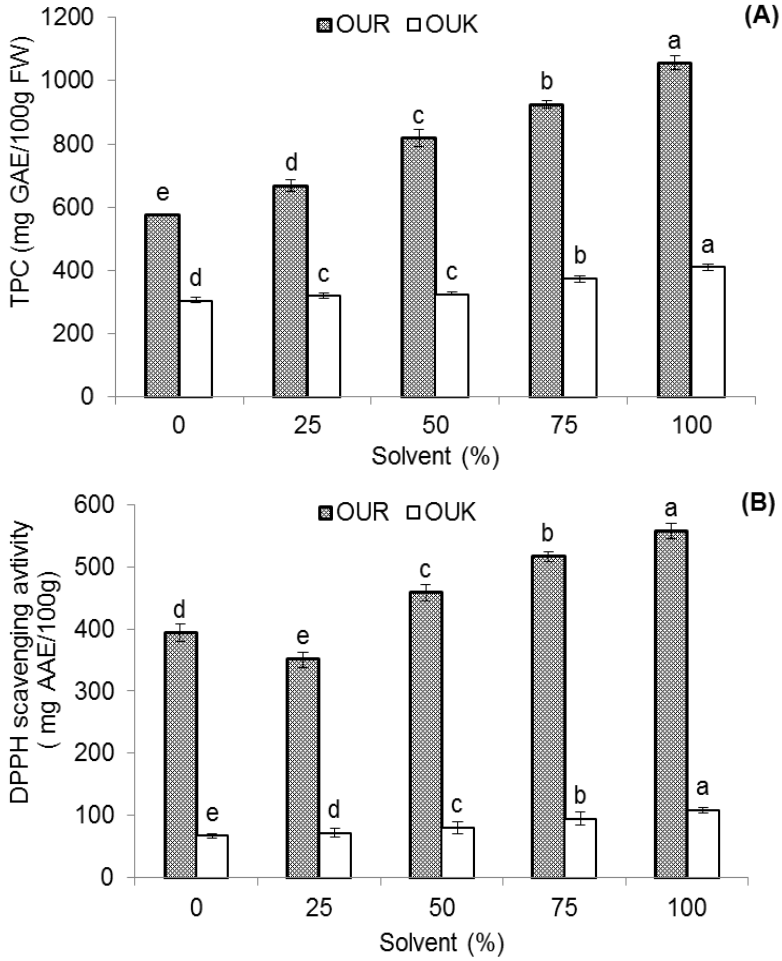

Figure 2. Influence of the solvent concentration on the extraction efficiency of TPC (A) and antioxidant activities (B) of date fruit extracts.

Values with different letters are significantly different ($p<0.05$).

Similar results for *Carthamus tinctorius* L. were reported by Dai et al., (2013 b), who found that the highest extraction yield of phenolic metabolites was achieved with no addition of water in DES based on lactic acid and glucose.

DES high solubilizing capacity is related to their supramolecular structure and broad polarity range.

Solid-to-solvent ratio

The effect of solid/solvent ratio on the recovery of phenolic compounds and the antioxidant activity is shown in Figure 3. Samples were extracted using the best solvent ratio and the best solvent concentration. The extraction was repeated by varying the sample/solvent ratio 100/15, 150/ 15, 200/15, 250/15 and 300/15 mg/ml, while fixing the extraction time at 15 minutes using a magnetic stirrer and 3

minutes in an ultrasound-assisted extraction. Extraction of antioxidants reached a maximum with a ratio of 100mg/15 ml (1141.8 mg GAE/100 g for OUR variety, and 448.4 mg GAE/100 g for OUK variety) (Figure 3A). The antioxidant activity decreased with changing ratio from 100mg/15ml to 300mg/15ml (Figure 3B). The further increase in the solid/solvent ratio decreased the extraction. This is probably because the diffusion, transfer and salvation capacity of the solvent was decreased (Prakash, *et al*., 2015). The results of the one-way analysis of variance showed that there were significant differences among the studied ratios, with the highest values of extracted phenolic compound for 100mg/15 ml in the case of OUR sample. However, for the OUK variety the figure 3A showed insignificant difference for sample/solvent ratio from 100 to 200mg/15ml. This is consistent with mass transfer principles, which outline that the concentration gradient (the driving force) is higher when there is more solvent present, leading to higher diffusion rates (Tan *et al*., 2011). Determination of the appropriate solid to liquid ratio is necessary. A low ratio causes oxidation of phenolics, and more liquids may provide more dissolved oxygen, which increases oxidation with a long extraction time, particularly at high temperatures. A high solid to liquid ratio produces incomplete extraction and the solvent becomes saturated before substrate exhaustion (Shi *et al*., 2003). According to Pinelo *et al.* (2005), the highest phenolic compound concentration and the best antiradical capacity were obtained when a low sample-to-solvent ratio is used. The reduction in extraction efficiency is explained by a poor solid-to-solvent interaction, possibly due to a caking of sample, which decreases the solubility of phenolics in extracting solvent (Luthria2012).

Extraction time

To date, the development of a single standard method for efficient extraction of phenolics from plant matrices has remained a challenge due to the inherent limitations of various conventional extraction techniques. The exploitation of phenolics as bioactive compounds has motivated scientists to explore more eco-friendly, efficient, and cost-effective extraction methods, based on a green extraction approach.

In order to seek more environmental friendly methods, decrease the solvent consumption, shorten the extraction time, increase the extraction yield, and enhance the quality of extracts, various novel extraction techniques have been developed for the extraction of nutraceuticals from plants (Wang and Weller, 2006). Among these, ultrasound-assisted extraction has emerged as a promising technique that fulfills the required criteria as an inexpensive green extraction technique. Notable ultrasound-assisted extraction features include versatility, simplicity, safety, rapidity, eco-friendliness, and cost-effectiveness, due to the reduced consumption of time, energy, expensive solvents volume and better extraction efficiency, which is in contrast to traditional extraction techniques (Ameer et al., 2017). The enhancement of extraction obtained by using ultrasound is mainly attributed to the effects of acoustic cavitations produced in the solvent by the passage of an ultrasonic wave. Ultrasounds also exert a mechanical effect, allowing greater penetration of solvent into the sample matrix, increasing the

contact surface area between solid and liquid phase; as a result, the solute quickly diffuses from solid phase to the solvent and lead to shortening of the extraction time (Wang *et al*., 2008*;* Prokhorov *et al.,* 2017). In addition, there is no chemical involvement in the ultrasound-assisted extraction, which could prevent possible chemical degradation of targeted compounds (Muñiz-Márquez *et al*., 2013).

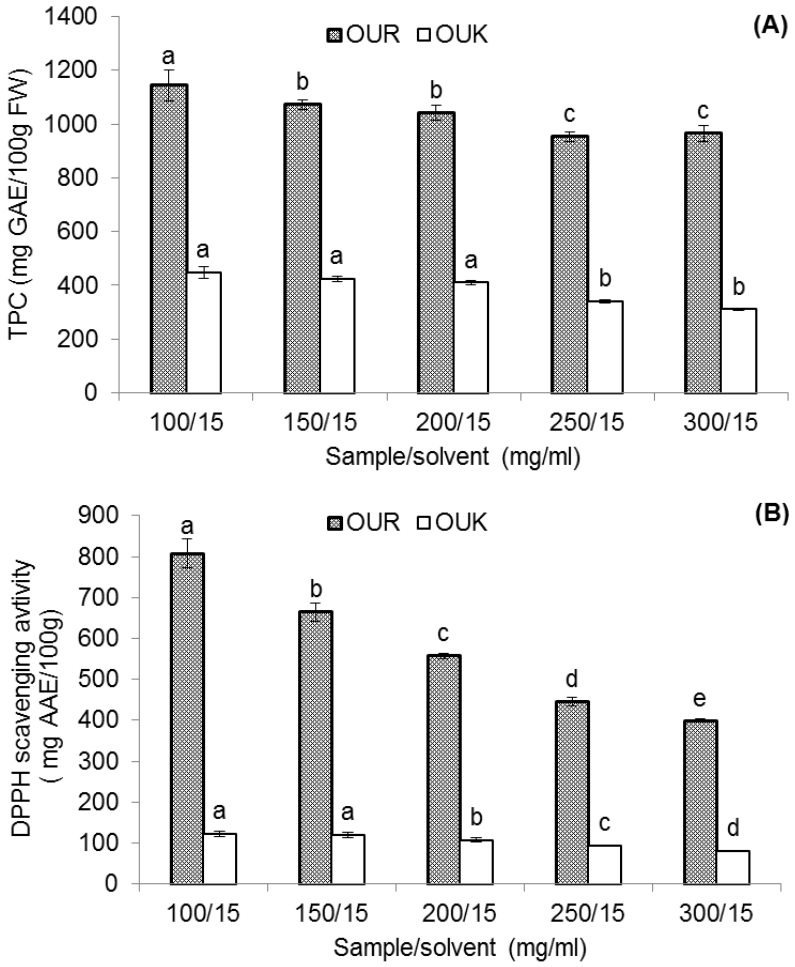

Figure 3. Influence of the sample/solvent ratio on the extraction of TPC (A) and antioxidant activity (B) of date fruit

Values with different letters are significantly different ($p<0.05$)

On such a conceptual background, this study was undertaken to examine whether aqueous lactic acid/sucrose mixtures can be used as a non-expensive, non-toxic and efficient mean of recovering phenolic antioxidants from date palm fruit, assisted by application of ultrasonication.

The extraction duration is another crucial parameter influencing phenolic extraction. There are a large number of published studies describing the effect of extraction duration on phenolic extraction from plant materials. In our study, the range of extraction time was premeditated on practical and economical aspects. The effect of sonication time on the phenolic compound recovery was examined for 3, 9, 12, 15, 21, 27, 33 and 40 minutes in the process of the ultrasound-assisted extraction. The phenolic compound recovery, in parallel with antioxidant activity significantly increased with extraction time increasing from 3 to 33min (Figure 4). This duration allowed extraction of 1393.5 and 528.4 mg GAE/100 of TPC from OUR and OUK date variety, respectively, which corresponded to respective antioxidant activities of 948.1 and 170.4 mg AAE/100g.

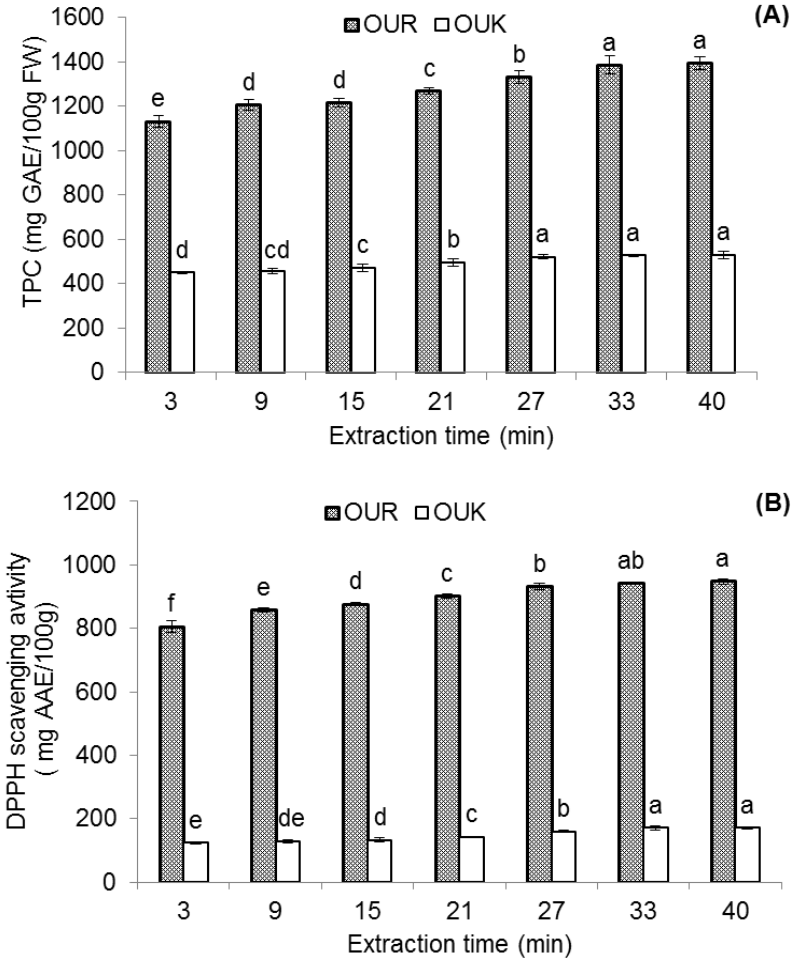

Figure 4. Influence of the extraction time on the extraction of TPC (A) and antioxidant activity (B) of date fruit extracts.

Values with different letters are significantly different ($p<0.05$).

No further significant increase in the extraction of date fruit phenolics was observed after 33 min for OUR and 27 min for OUK. This observation was supported by Fick's second law of diffusion, which states that "final equilibrium will be achieved between the solute concentrations in the solid matrix (plant matrix) and in the bulk solution (solvent) after a certain time", hence, a longer extraction time was not useful to extract more phenolic antioxidants (Silva*et al*.,2007). Furthermore, extended extraction process might lead to oxidation of phenolics owing to prolonged light or oxygen exposure.

Chaalal *et al*. (2012) revealed that after 90 min, increase in process duration did not significantly ($p < 0.05$) improve the recovery of phenolics and antioxidant activity of prickly pear seeds when using 75% acetone as solvent.

Each plant species like vegetables, fruits, and medicinal plants has different chemical profile; with their different compounds contributing to varying antioxidant activity (Babbar*et al*., 2011). Thus, it is important to determine which group of compounds is the major contributor to the antioxidant properties for further isolation. Phenolic compounds have been shown to have a direct correlation with their antioxidant properties (Sulaiman *et al*., 2011). In order to appreciate more the relationships between antioxidant capacities and phenolic content of OUR and OUK date variety extracts, correlations between assays under different extracting conditions were analyzed and the results were shown in Table 1. Under different extraction parameters, correlations between TPC and antioxidant activity were positively high ($0.71 < r < 0.99$, $P<0.001$). This relationship suggested that the hydrogen electron donating ability of the extracts was directly proportional to the total phenolic content. Thus, phenolic compounds might be the major contributors to the antioxidant capacities of theses date fruit extracts.

Our findings are in agreement with results reported by Benchikh and Louaileche (2014) who reported high correlation between TPC and antioxidant activities (DPPH, and FRP) in carob pulp with r values ranging from 0.84 to 0.9.

Table 1. Correlation between TPC and antioxidant activity of date extracts under influence of extraction conditions.

	Correlation coefficient (r)		Equation	
	OUR	*OUK*	*OUR*	*OUK*
Solvent molar ratio	0.90^{***}	0.71^{ns}	y = 2.41x -387.39	y = 1.13x +264.82
Solvent concentration	0.94^{***}	0.99^{**}	y = 2.13x - 158.56	y = 2.58x +127.56
Solid-to-solvent ratio	0.98^{***}	0.98^{***}	y = 0.46x + 768.42	y = 3.27x + 43.60
Extraction time	0.98^{***}	0.98^{***}	y = 1.86x - 391.70	y = 1.64x + 251.47

ns not significant, **significant at $p < 0.01$ and ***significant at $p < 0.001$.

Conclusions

The results of the present investigation provided for the first time that a combination of lactic acid and sucrose, two non-toxic substances, might be a very effective co-solvent system regarding the extraction of antioxidant phenolics from two date palm fruits varieties, with the assistance of ultrasonication. Optimization of sample preparation is essential for an accurate quantitative determination of phenolic compounds. The experimental design based on a single factor experiments approach was used to determine the optimization of the extraction. The results from the present work showed that all experimental parameters explored had statistically significant ($p < 0.05$) effects. It can be concluded that the optimum conditions for recovery of antioxidants from the OUR and OUK varieties of date were determined: a double extraction with 100% mixture solvent molar ratio (lactic acid: sucrose) of 3:1 using a 100 to 200mg/15ml solid to solvent ratio and contact time from 33 min. These conditions allowed recovery of 1393.5 (OUR date) and 528.4 mg/100 g (OUK date), and produced DPPH radical scavenging activities of 948.1 and 170.4 of AAE mg/100 g, respectively.

On the ground of these data, suggests that the solvent system examined may be used for the development of eco-friendly processes. On the other hand, the incorporation of lactic acid /sucrose extracts in food, cosmetics, pharmaceutical and nutraceutical formulations might be simpler and straight forward, since solvent compounds are extensively used as a constituent in such products.

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