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The effects of extraction technique on phenolic compounds extracted from fig (*Ficuscarica*) pulp andskin

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Abstract

Recently, Subcritical Water Extraction (SWE) has been well known as a green technology for extraction of bioactive compounds from plants. In this study, Subcritical water extraction, ultrasound assisted extraction (UAE) and shaker solvent extraction (SSE) were compared for extraction of phenolic compounds from fig (Ficuscarica) pulp and skin. Antioxidant activity of the extracts was evaluated using DPPH radical scavenging, reducing power and rancimat tests. Subcritical waterhad the highest ability for extraction of total phenolic content (65.89±0.21 and 80.79±0.09 mg of gallic acid equivalents per gram of extract respectively) and flavonoid compounds (7.51±0.33 and 10.1±1.02 mg of quercetinequivalents per gram of extract, respectively)from both pulp and skin.The lowest IC50 in DPPH radical scavenging and reducing power tests were related to SWE of skin extract of fig. Furthermore, in extraction of total phenol and flavonoid compounds, subcritical water extraction showed to be a more suitable method than other solvent extraction methods, both in pulp and skin.

Keywords: Antioxidant activity; Fig; Extraction; Ultrasound; Subcritical water extraction

Introduction

The Common Fig (Ficuscarica L.) is a tree native to the Middle East and the Mediterranean region, which belongs to botanical family Moraceae. The Common (Flaishman *et al*., 2008; Oliveira *et al.*, 2009)fig is an excellent source of minerals, vitamins and dietary fiber; fat and cholesterol-free and contain a high number of amino acids (Caliskan, 2015; Viuda-Martos *et al*., 2015). In addition of several health benefits which were previously reported (Lansky *et al*., 2008; Viuda-Martos *et al*., 2015; Weli *et al.,* 2015), figs are an excellent source of phenolic compounds.

Polyphenols are natural antioxidants and the most abundant secondary metabolites of plants that possess interesting properties, such as free-radical scavenging and inhibition of various oxidative stress in the body(Dai and Mumper, 2010).Plant-derived antioxidants are molecules, which donate electrons or hydrogen atoms. These compounds are able to form less reactive antioxidant-derived radicals, which are efficiently quenched by other electrons or hydrogen sources to prevent cellular damage therefore, they may delay and inhibit lipid oxidation, protect human cells against oxidative damage, lead to a reduced risk of several oxidative-stress associated degenerative diseases, such as cancer, cardiovascular or neurodegenerative diseases (Scalbert *et al*., 2005).When added to foods, these antioxidant compounds tend to minimize rancidity, retard the formation of toxic oxidation products, help to maintain the nutritional quality and increase their shelf life (Fukumoto and Mazza, 2000).

Phenolic compounds from plants has been traditionally extracted using solvent extraction or steam distillation techniques. Traditional methods of extraction are cost and time consuming protocols, and require large volumes of solvents (Teixeira et al., 2006).Recently several new methods have been applied for plant phenolic extraction such as supercritical fluid extraction (SFE)(Herrero et al., 2010; McHugh and Krukonis, 2013; Pereira and Meireles, 2010), pulse electric

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field assisted extraction (PEFE)(Vorobiev et al., 2005), high pressure assisted extraction (HPE)(Corrales et al., 2008) and matrix solid-phase dispersion (MSPD) (Capriotti et al., 2010; Dawidowicz and Rado, 2010), which are less labor intensive and more environmentally friendly. Despite the use of this new extraction techniques solid–liquid extraction (SLE) is still commonly used.

Subcritical water extraction (SWE), also called pressurized polarity water extraction or superheated water extraction, is a technique using water as extraction solvent at temperatures between 100 and 374°C with a pressure high enough to maintain water in liquid state (Ma et al., 2015). Previous studies have proved that it is a promising technique because of its short extraction time, decrease the organic solvent consumption, less volatile oil loss and increase the quality of extracts(Jayawardena and Smith, 2010; Kumar et al., 2011; Rodríguez-Meizoso et al., 2010).

In recent years, ultrasound-assisted extraction method has become an effective method for edible oils and fats from natural product extraction UAE is an inexpensive, simple and efficient alternative to conventional extraction techniques (Wang and Weller, 2006). The mechanism of UAE is attributed to mechanical and cavitation efficacies which can result in disruption of cell wall, particle size reduction, and enhanced mass transfer across cell membrane(Wang *et al*., 2013). The objective of this study is to compare antioxidant activity of the phenols extracted from the pulp and skin of fig by ultrasound-assisted aqueous extraction, Subcritical water extraction and solvent extraction

Materials and methods

Material

Fig fruit (*F. carica L.*) from Siyah variety collected from Gorgan city on September 2014. Canola oil was purchased from Alia Golestan company (Kordkooy,iran) All other chemicals used in this study were of analytical grade and were purchased from chemical suppliers.

Preparation of extracts

The figs (Siyah variety) were weighed and immediately peeled. The pulp was cut and made into flat sheets. Thereafter, the pulp and skin of fruit were shade-dried for 5 days followed by drying at 60°C in an oven for 24 hours to ensure complete drying (Memmert 100-800, Germany). The samples were then milled and sieved through No. 67. Samples obtained were kept in polyethylene bags

Solvent extraction

Ten grams of each sample was mixed with water-ethanol (70%) in a ratio of 1 to 10 and gently stirred. For a better extraction, the mixture was shaken (120 rpm) at dark at 25 °C for 24 hours on a shaker, then the supernatant was filtered by Buchner funnel and Whatman filter paper No. 1. The extracts containing solvent was poured in a glass plate and placed in oven at 40°C for 24 hours. After evaporation of the solvent, the extracts were placed in desiccator until constant weight and kept at -18°C for further analyses (Esmaeilzadeh Kenari *et al.,* 2014).

Ultrasound assistant extraction

Dried powders of sample (10 g) were mixed with ethanol (1:10). The mixture was sonicated for 20 min at 40 °C in an ultrasonic bath (Elma s 30 H model, total power consumption: 280 W, heating power: 200 W, operating at 37 KHz frequency and internal dimensions: 198 × 106 × 50 cm). The temperature was controlled and maintained at 40 °C by circulating water. The extract was filtered and subsequently evaporated using a rotary evaporator. The concentrated extracts were stored at -18°C until further analyses (Esmaeilzadeh Kenari *et al*., 2014).

Subcritical water extraction

Subcritical water extraction was carried out by a system consists of a distilled water tank, a pump (Comet type:MTP AX 2/70 m) providing pressure up to 170±5 bar, an extraction cell with 140 ml capacity, a heating coil, a pressure gauge and a temperature

control device. The powdered sample was loaded into the cell. Extraction was carried out at temperature of 160ºC for 30 minutes (Hassas-Roudsari et al., 2009). The impurities of liquid (extract) was removed using filter paper (Whatman paper No. 4) under vacuum condition. The filtered extracts were cooled and stored at -18ºC in dark polypropylene bags until used in the analysis(Shaddel *et al*., 2014; Sharifi *et al.,* 2013).

Determination of total phenolic content

Total phenolic content of each extract was determined by the Folin–Ciocalteu micro-method according to revised methods of Javanmardiet al. (2003). Briefly, in a 50 ml volumetric flask, 1 ml of a standard solution of gallic acid, 6 ml of methanol, 2.5 ml of the Folin-Ciocalteau reagent, and 5 ml of 7.5% Na2CO3 were added.The final volume was achieved by addition of distilled water. The solutions were stored overnight and the spectrophotometric analysis was performed at λ=765 nm. (PG-instrument, USA). The total phenol content of samples and canola oil were expressed as gallic acid equivalents per g of extract using the following linear equation based on the calibration curve:

$y= 0.0008x+0.029 \quad R^2= 0.094$

Where y is absorbance at 765 nm and x is concentrations of gallic acid equivalents (mg/g)(Capannesi *et al.,* 2000; Javanmardi *et al*., 2003).

Determination of total flavonoid content

Colorimetric aluminum chloride method was used for flavonoid determination. Briefly, 0.5 ml solution of each plant extracts in methanol were separately mixed with 1.5 ml of methanol, 0.1 ml of 10% aluminum chloride, 0.1 ml of 1 M potassium acetate, and 2.8 ml of distilled water, and left at room temperature for 30 minutes. The absorbance of the reaction mixture was measured at 415 nm with a double beam Perkin Elmer UV/Visible spectrophotometer (PG-instrument, USA). The calibration curve was prepared by preparing pure quercetin solutions at concentrations 12.5 to 100 mg/ ml in methanol.Total flavonoid content was calculated as quercetinper g of extract using the following linear equation based on the calibration curve:

$y= 0.0064x+0.0124 \quad R^2= 0.9982$

Where y is absorbance at 415 nm and x is concentrations of flavonoids compounds (mg quercetin /g extract)(Nabaviet al., 2012).

DPPH Radical-Scavenging Activity

Stable 2, 2'-diphenyl-1-picryl hydrazyl radical (DPPH) was used for determination of free radical-scavenging activity of the extracts. Different concentrations of each extracts were added, at an equal volume, to methanol solution of DPPH (100 μM). The samples were kept at room temperature in darkness and after 15 min the absorbance of each sample was measured at 517 nm and the percentage of scavenging activity was calculated from the equation 1. The experiment was repeated for three times. IC50 values denote the concentration of sample, which is required to scavenge 50% of DPPH free radicals (Aksoy *et al*., 2013).

$$DPPH\ scavenging\ activity\ (\%) = \frac{Absorbance\ of\ control - Absorbance\ of\ sample}{Absorbance\ of\ control} \quad (1)$$

Reducing power

Ability of extracts to reduce iron (III) was evaluated using the method of Yildirim *et al*.(2001). Samples (2.5 ml) were mixed with 2.5 ml of phosphate buffer (0.2 M, pH 6.6) and 2.5 ml of potassium ferricyanide (K3Fe (CN) 6; 10 g/L) and incubated for 30 min at 50°C. Then 2.5 ml of trichloroacetic acid (100 /L) were added to the solution and centrifuged for 10 min. Finally, 2.5 ml of supernatant was combined with 2.5 ml of distilled water and 0.5 ml $FeCl_3$ (1 g/L). The absorbance of samples was measured at 700 nm. Higher absorbance means higher reducing power (Yildirim *et al.,* 2001).

Determination of the thermal oxidative stability index (Rancimat test)

One mg/ml of each sample was mixed with 100 ml pure canola oil (without antioxidant) and then oxidative stability measured by Rancimat (Metrohm, 743, Switzerland) test based on the AOCS method (2007). The air flow rate and the temperature was set at 20 m3/h and at 110 °C, respectively.

Statistical analysis

Each experiment was carried out at least in duplicate and measurement performed at least in triplicate. Statistical analysis of data was performed using Microsoft Excel. Analysis of variance was calculated using the SPSS program with a confidence level of 0.05, to find any significant difference between treatments.Duncan multiple range test (MRT) was used for mean separation at P< 0.05 where treatment effect was significant

Result and discussion

The effect of extraction on phenolic and flavonoid compounds

Analysis of variance (ANOVA) showed that the techniques used for extraction of phenolic compounds in skin and pulp of the fig were significant different (P<0.05). Subcritical water method in both skin and pulp samples showed the highest amount of total phenolic (80.79±0.09 and 65.89±0.21 mg gallic acid/g of extract, respectively) and flavonoid (10.1±1.02 and7.51±0.33 mg quercetin/g of extract, respectively) contents whereas there was no difference between UAE and SSE (Table 1).

Table 1- Total phenol and flavonoids of pulp and skin of fig extract with different extraction methods

Extraction technique	Total Phenol mg gallic acid/g of extract		Total Flavonoid mg quercetin/g of extract	
	pulp	skin	pulp	skin
SSE	58.35±0.05^{b}	68.59±1.02^{b}	2.04±0.13^{b}	4.29±0.12^{b}
UAE	58.84±0.13^{b}	70.09±0.32^{b}	2.06±0.23^{b}	4.32±0.12^{b}
SWE	65.89±0.21^{a}	80.79±0.09^{a}	7.51±0.33^{a}	10.1±1.02^{a}

Means with different letters within column indicate significance difference at $P < 0.05$.
SSE: Shaker Solvent Extraction; UAE: Ultrasonic Assisted- Extraction; SWE: Subcritical Water Extraction

Flavonoids are strong inhibitors of hydroxyl and peroxide radicals. These compounds can influence on free radicals even when they form complexes with metal ions (Koda *et al*., 2008). Flavonoid compounds have pharmaceutical, antibacterial and anti-oxidation properties. Quercetin is a flavonol which is one of the most powerful natural antioxidants and a major component known in figs (Solomon *et al.*, 2006). Antioxidant properties of flavonoids, especially quercetin, are rendered through metal ion chelating, radical scavenging, and stimulating the expression of protective enzymes (Baghel *et al*., 2012). Therefore, measurement of these compounds in figs deems very important.

Higher efficiency of SWE in the extraction of flavonoids also reported by earlier studies (Ko *et al*., 2011; Liang and Fan, 2013; Rangsriwong *et al*., 2009). Extraction of a wide range of flavonoids by common extraction methods is limited due to poor solubility in water. By application of subcritical water, the polarity, viscosity, surface tension, and disassociation constant of subcritical water are significantly lowered compared to water at ambient temperature and pressure, with more similar chemical properties to those of organic solvents. The temperature and pressure of subcritical water extraction provide higher penetration rate that improves its efficiency (Rangsriwong *et al*., 2009). Higher efficiency of SWE also could possibly be related to the effect of hydrolysis reaction caused by the increase in the ionization constant (Kw) of water at subcritical conditions.

Extraction time considerably decreased in ultrasonic process (from 24 h in SSE to 20 min in UAE) due to the positive effect of cavitation

although no significant difference was detected between the methods in terms of amount of phenolic and flavonoid compounds. Solid–liquid extraction is assisted by ultrasounds leading to formation of cavitation bubbles. The collapse of these bubbles near the cell walls is expected to cause cell disruption along with good penetration of the solvent into the cells through the ultrasonic jet action. This process leads to an intensification of mass transferand improved solvent penetration into the plant tissue (Da Porto *et al.,* 2013). Nevertheless, extraction conditions (e.g. time, temperature, solvent) greatly influence on the efficiency of extraction of phenolic compounds and flavonoids in plant tissue. Similar results which compared ultrasound and solvent methods, were in consistent with our results (Chemat *et al.,* 2004; Da Porto *et al.,* 2013; Kimbaris *et al*., 2006; Milić *et al.,* 2013; Vinatoru *et al*., 1997).

The effect of extraction on DPPH Radical-Scavenging Activity

DPPH scavenging activity assay is widely used to evaluate the ability of compounds to scavenge free radicals or donate hydrogen, and determine the antioxidant activity in foods (Bidchol *et al*., 2011). Subcritical water extraction of fig pulp at concentration of 1 mg/ml showed the highest radical scavenging activity (65.66 ± 1.54%) while a sharp decline in antioxidant activity was observed with higher concentrations (e.g. 1.5 and 2 mg/ml) (Figure 1). This extracts showed the highest inhibition at lower concentrations. While the other extracts with increasing concentrations, increased antioxidant activity. Subcritical water extraction of fig skin at concentrations of 0.5, 1, 1.5 and 2 mg/ ml and UAE and SSE extract at concentrations of 2.5 and 3 mg/ml had the highest percentage of inhibition. Radical scavenging activity in the UAE- and SSE-extracts from both skin and pulp of fig was dependent on concentration where a higher radical scavenging was achieved with higher concentrations of phenolic and flavonoid compounds; this effect might be owing to higher amount of hydroxyl groups and consequently, increased probability of hydrogen donation to free radicals (Sanchez-Moreno *et al*., 1999). However, radical scavenging activity of subcritical water extracts decreased with higher extract concentrations. A logic behind this observation is the reaction between released chemicals in solvent leading to formation of new compounds and interference in identification of target compounds in higher concentration (Plaza *et al*., 2010).

IC_{50} is defined as a concentration of the extract required to scavenge 50% of DPPH radicals. Subcritical water extract of skin revealed the lowest IC50 (0.45±0.02 mg/ml); in other word, it is the best extract to scavenge free radicals. On the contrary, subcritical water extract of pulp had significantly higher IC50(0.65±0.09 mg/ml) valuesthan other treatments (figure 2).

The effect of extraction on reducing power

Reducing property is generally defined as the ability ofdonating a hydrogen atom and thereby breaking aradical chain.Furthermore, reluctant react with peroxideprecursors and prevent the formation of peroxides [45]. Thus, samples with higher reducing powers aremore able to donate electrons. The reducing powers ofdifferent samples are shown in Fig. 3 (a,b). In all concentrations, SWE of fig pulp and skin had the highest reducing power, UAE and SSE had no significant difference. The results showed that the compounds in fig skin extracts were good electron or hydrogen donors and could successfully terminate radical chain reactions. So, it can be considered a good alternative to synthetic antioxidants in the diet. These results were consistent with Gou *et al*. (2003) who evaluated the antioxidant activity of the skin and the pulp and kernel of twenty-eight common fruit in China via reducing power analysis and reported that fruit peels and kernels had higher antioxidant activity than pulps (Guo *et al*., 2003).The results also showed the advantages of SWE because with higher temperature, the dielectric constant of water reduces leading to lower polarity of water. Therefore, compounds with differentpolarities including a variety of

flavonoids can be extracted via SWE, which increases the reducing power of extracts. Increasing concentrations lead to increase in the reducing power of extracts due to the increased amount of phenolic compounds present in the extract. These data largely confirm the DPPH test. The only difference is that a better distinction of antioxidant activities of extracts with varying concnetrations is achieved through reducing power assay. Since this method is usually used to measure the antioxidant capacity of hydrophilic compounds (Pérez-Jiménez et al., 2008)and also because of the nature of hydrophilic compounds in the figs extracts (due to the presence of anthocyanins), this method seems more accurate to measure the antioxidant activity fig extract.

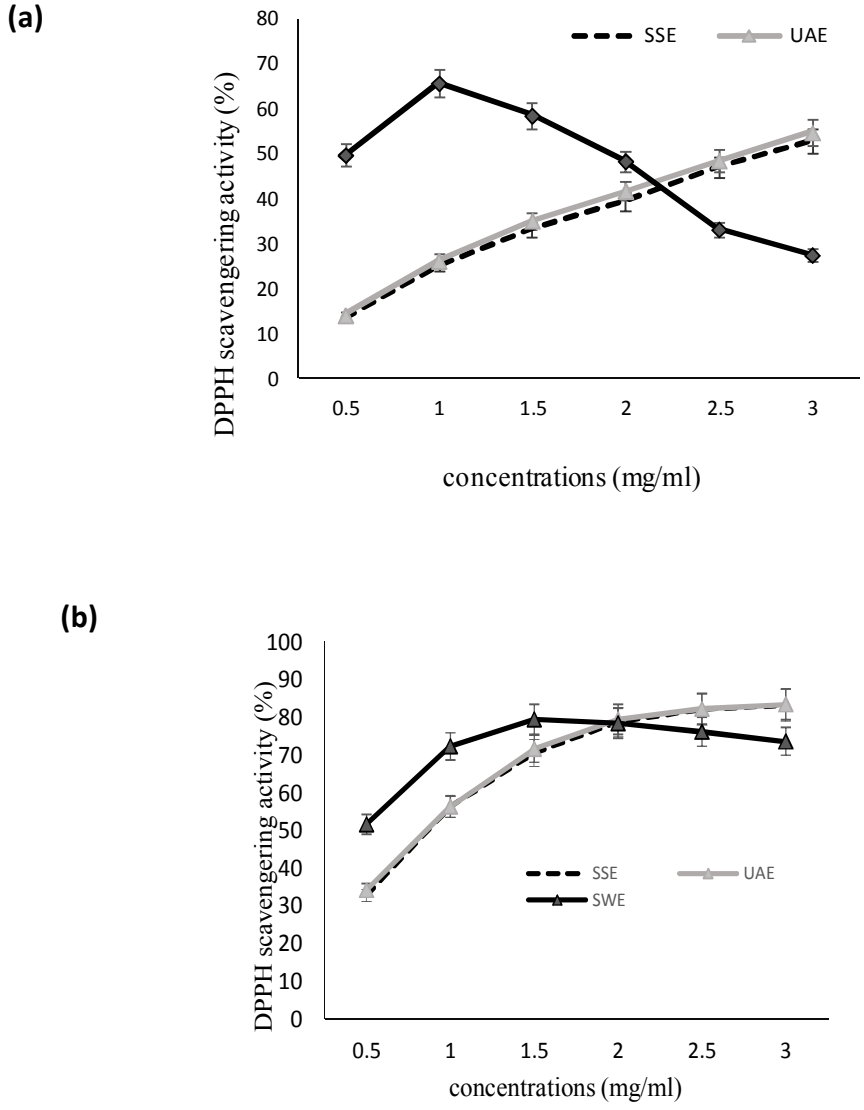

Fig. 1. DPPH scavenging activity of fig extract with different extraction methods: (a) fig pulp and (b) fig skin (SSE: Shaker Solvent Extraction; UAE: Ultrasonic Assisted Extraction; SWE: Subcritical Water Extraction)

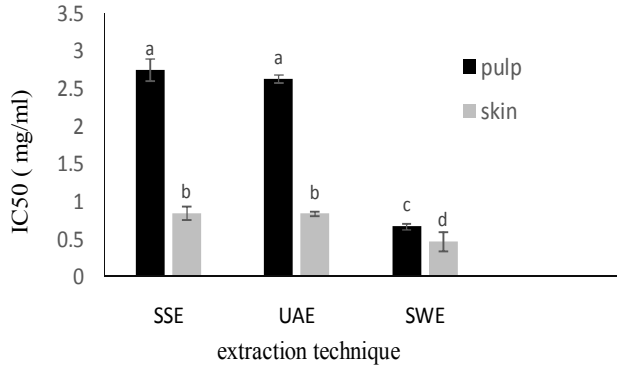

Fig. 2. IC_{50} of extracts in DPPH assay with different extraction methods (SSE: Shaker Solvent Extraction; UAE: Ultrasonic Assisted Extraction; SWE: Subcritical Water Extraction)

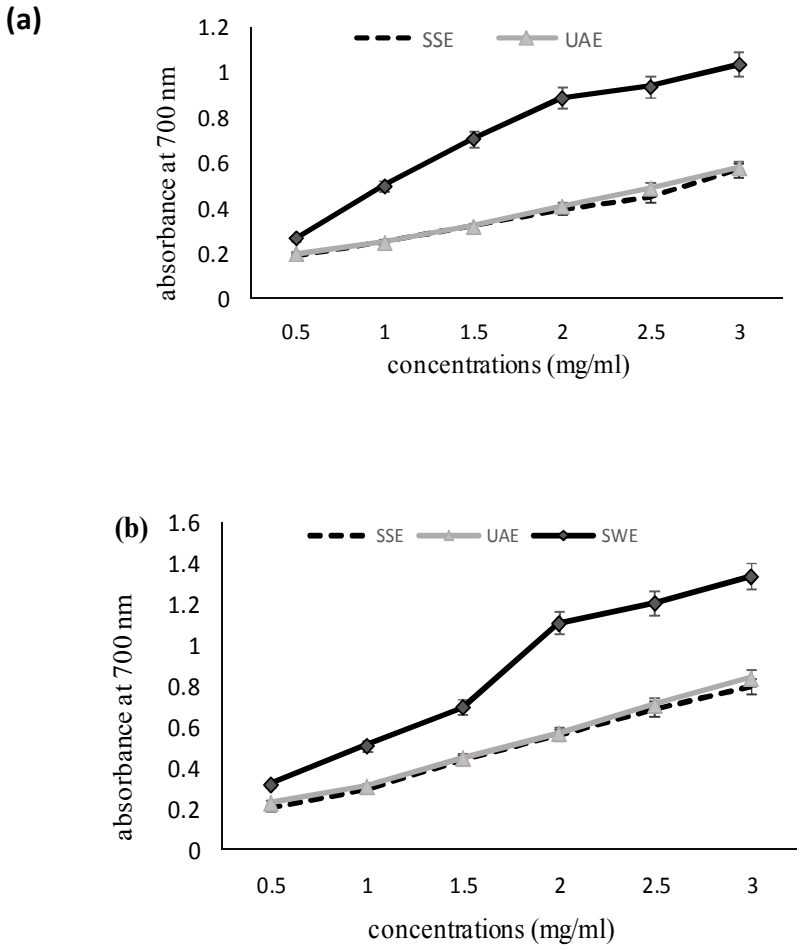

Fig. 3.Reducing power of fig extract with different extraction methods: (a) pulp and (b) skin (SSE: Shaker Solvent Extraction; UAE: Ultrasonic Assisted Extraction; SWE: Subcritical Water Extraction)

Figure 4 shows IC_{50} in reducing power assay (mg/ml). In the case of reducing power, the IC_{50} value presents the concentration at which the absorbance is 0.5. Similar to DPPH assay, Subcritical water extracts in both skin and pulp samples showed significantly the lowest IC_{50} (0.90±0.1 and 0.96±0.05 mg/ml) and the highest reducing power.

Antioxidant activity of the extracts in edible oil was measured based on the electrical conductivity of water in addition to the accumulation of volatile compounds from oxidation, especially carboxylic acids under accelerated oxidation. Oxidative stability index (OSI) is defined as oil stability time at a given temperature. The results has shown that the effect of the extraction method was significant on oxidative stability and thus antioxidant activity ($P<0.05$). In this study, UAE showed higher stability among other extracts (Figure 5). This can be due to differences in methods of extraction and the resulting difference is in the type of phenolic compounds in the extracts. These factors are important in solubility of the extracts in oil and hence in the oxidative stability of the oil.

Conclusion

The result presented in this study show that the subcritical water extraction method is an alternative for extraction of several phenolic and flavonoid compounds from skin and pulp of fig, and more efficient than both solvent extraction method and ultrasound. Subcritical water extracts of skin and pulp of fig showed higher antioxidant activities in the DPPH radical scavenging and reducing power assays, while in rancimat test ultrasound method was found to yield more antioxidant extracts. This difference can be justified by varying nature of the methods of measuring antioxidant activity. Additional research and development studies are necessary to thoroughly compare subcritical extraction with other recent methods.

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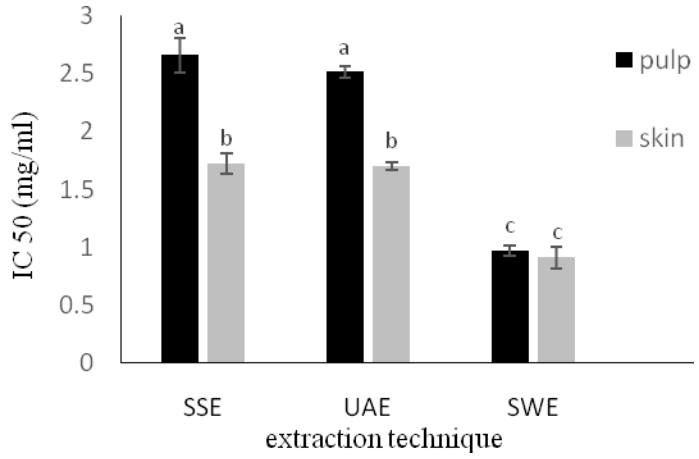

Fig. 4. IC_{50} of extracts in reducing power assay with different extraction methods (SSE: Shaker Solvent Extraction; UAE: Ultrasonic Assisted Extraction; SWE: Subcritical Water Extraction)

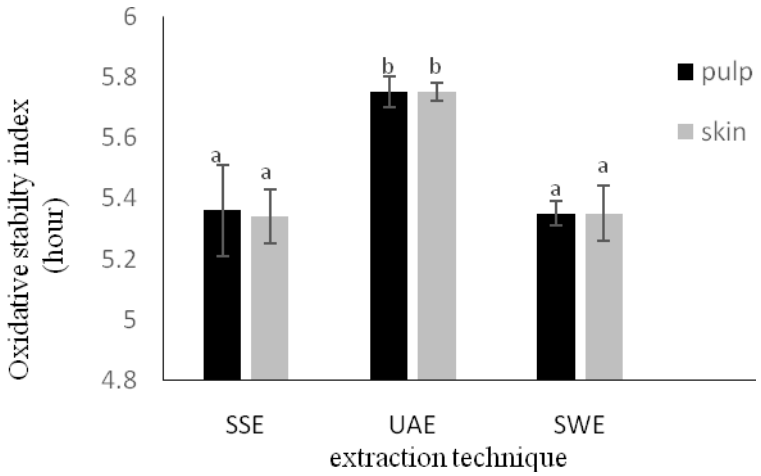

Fig 5- Oxidative stability index of pulp and skin extract with different extraction (SSE: Shaker Solvent Extraction; UAE: Ultrasonic Assisted Extraction; SWE: Subcritical Water Extraction)

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اثر روش‌های استخراج بر ترکیبات فنولی استخراج‌شده از پوست و پالپ انجیر

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چکیده

اخیرا استخراج آب زیر بحرانی (SWE) به عنوان یک تکنولوژی سبز برای استخراج ترکیبات زیست فعال گیاهان شناخته شده است. در این مطالعه، استخراج آب زیر بحرانی، استخراج به کمک اولتراسوند (UAE) و استخراج حلالی همزنی (SSE) جهت استخراج ترکیبات فنولی پوست و پالپ انجیر مقایسه شدند. فعالیت آنتی‌اکسیدانی عصاره‌ها با آزمون‌های مهار رادیکال DPPH، قدرت احیاکنندگی و رنسیمت ارزیابی شد. آب زیر بحرانی نشان داد که بالاترین توانایی را در استخراج ترکیبات فنولی کل پالپ و پوست (به ترتیب، ۰/۲۱±۶۵/۸۹ و ۰/۰۹±۸۰/۷۹ معادل میلی‌گرم گالیک اسید در گرم عصاره) و ترکیبات فلاونوئیدی (به ترتیب، ۰/۳۳±۷/۵۱ و ۱/۰۲±۱۰/۱ معادل میلی‌گرم کوئرستین در گرم عصاره) دارد. همچنین، کمترین IC50 در آزمون‌های مهار رادیکال DPPH و قدرت احیاکنندگی به عصاره‌ی آب زیر بحرانی پوست انجیر مربوط بود. بنابراین، آب زیر بحرانی روش مناسب‌تری برای استخراج ترکیبات فنولی و فلاونوئیدی کل پوست و پالپ انجیر می‌باشد.

واژه‌های کلیدی: فعالیت آنتی‌اکسیدانی، انجیر، استخراج، اولتراسوند، استخراج آب زیر بحرانی

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