https://doi.org/10.30910/turkjans.1666602



TURKISH JOURNAL of AGRICULTURAL and NATURAL SCIENCES

www.dergipark.gov.tr/turkjans

Original Article

Comparison of Total Phenolic Contents and Antioxidant Activities of Ripe and Unripe Fig (*Ficus carica* L.) Extracts Using Different Solvents

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ABSTRACT

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TARIM ve DOĞA BİLİMLERİ

DERGISI

This study aimed to evaluate the effects of solvent polarity and fruit maturity on the antioxidant potential and total phenolic content of fig (Ficus carica L.) extracts. Unripe and ripe fig fruits collected from the Akseki district of Antalya, Türkiye, were extracted using three different solvents: ethanol, acetone, and ethyl acetate. The extraction yield, total phenolic content (TPC), total antioxidant capacity (TAC), and antioxidant activity (DPPH assay) were determined for each extract. In this study, extraction yield varied depending on both the type of solvent and the ripening stage of the fig. Ethanol was the most efficient solvent, particularly for ripe samples, yielding up to 80.3%. The highest TPC was observed in the ripe-acetone extract ($3976.2 \pm 526.5 \text{ mg GAE/kg DW}$), while the ripe-ethyl acetate extract exhibited the highest TAC (48.8 ± 0.87 mg AE/g DW). Antioxidant activity measured by the DPPH assay was greatest in the ripe-ethyl acetate extract (57.6 ± 10.37 mmol TE/kg DW). Among the tested extracts, the ripe-ethyl acetate extract exhibited the most favorable antioxidant properties, highlighting its potential as a promising source of bioactive compounds for functional applications. These results suggest that solvent polarity and fruit maturity interact to influence the extractability of phenolic compounds and antioxidant capacity. Semi-polar solvents such as acetone and ethyl acetate enhanced the recovery of bioactive compounds in both unripe and ripe samples, although the effectiveness varied depending on the specific antioxidant parameter evaluated. This study provides comparative data that may contribute to the optimization of extraction strategies for developing natural antioxidant sources from fig fruits.

Key words: Antioxidant activity, Ficus carica, Fig, Phenolic content, Ripening stage.

Farklı Çözücüler Kullanılarak Elde Edilen Ham ve Olgun İncir (*Ficus carica* L.) Ekstraktlarında Toplam Fenolik İçeriği ve Antioksidan Aktivitenin Karşılaştırılması

ÖZ

Bu çalışma, çözücü polaritesi ile meyve olgunluk düzeyinin incir (*Ficus carica* L.) ekstraktlarının toplam fenolik içeriği ve antioksidan potansiyeli üzerindeki etkilerini değerlendirmeyi amaçlamıştır. Antalya ili Akseki ilçesinden toplanan ham ve olgun incir meyveleri; etanol, aseton ve etil asetat olmak üzere üç farklı çözücü kullanılarak ekstrakte edilmiştir. Her bir ekstrakt için ekstraksiyon verimi, toplam fenolik madde içeriği (TPC), toplam antioksidan kapasite (TAC) ve antioksidan aktivite (DPPH yöntemi) analiz edilmiştir. Bu çalışmada, ekstraksiyon veriminin hem kullanılan çözücü türüne hem de incir meyvesinin olgunluk aşamasına bağlı olarak değişkenlik gösterdiği belirlenmiştir. Özellikle olgun meyve örneklerinde etanol, %80.3'e ulaşan verimle en yüksek ekstraksiyon etkinliğini sağlamıştır. En yüksek TPC, olgun–aseton ekstraktında (3976.2 ± 526.5 mg GAE/kg kuru madde) saptanırken; TAC açısından en yüksek değer, olgun–etil asetat ekstraktında ölçülmüştür (48.8 ± 0.87 mg AE/g kuru madde). Benzer şekilde, DPPH yöntemiyle belirlenen antioksidan aktivite de en yüksek düzeye olgun-etil asetat ekstraktında ulaşmıştır (57.6 ± 10.37 mmol TE/kg kuru madde). Elde edilen bulgular doğrultusunda, değerlendirilen ekstraktlar arasında olgun-etil asetat ekstraktı, güçlü antioksidan özellikleriyle öne çıkmakta olup, fonksiyonel uygulamalarda değerlendirilebilecek potansiyel bir biyoaktif bileşik kaynağı olarak dikkat çekmektedir. Elde edilen sonuçlar, çözücü polaritesi ile meyve olgunluğunun fenolik bileşiklerin ve antioksidan kapasitenin ekstrakte edilebilirliğini birlikte etkilediğini göstermektedir. Yarı-polar çözücüler olan aseton ve etil asetat, hem ham hem de olgun örneklerde biyoaktif bileşiklerin geri kazanımını artırmış; ancak bu etkinlik, değerlendirilen antioksidan parametreye göre değişiklik göstermiştir. Bu çalışma, incir meyvelerinden doğal antioksidan kaynaklarının geliştirilmesine yönelik ekstraksiyon stratejilerinin optimize edilmesine katkı sağlayabilecek karşılaştırmalı veriler sunmaktadır.

Anahtar kelimeler: Antioksidan aktivite, Ficus carica, Fenolik içerik, İncir, Meyve olgunluğu

INTRODUCTION

Fig (*Ficus carica* L.), a member of the Moraceae family, is one of the oldest cultivated fruit species and has long been appreciated for its nutritional and medicinal properties (Badgujar et al., 2014). In Türkiye, particularly in the Mediterranean region, fig cultivation holds both economic and cultural importance (Çalışkan and Dalkılıç, 2022). The fig fruit is a syconium, a compound structure with numerous small flowers lining the inside of a fleshy receptacle. Its outer peel, which may vary in color depending on the cultivar, contains significant levels of bioactive compounds such as pigments, tannins, and volatile substances that contribute to the overall antioxidant potential (Hajam and Saleem, 2022; Li et al., 2013). Figs can be consumed fresh or dried, and their composition—especially phenolic and antioxidant compounds—may vary depending on the maturity stage and extraction method used.

Figs are a rich source of bioactive compounds, particularly phenolics and flavonoids, which contribute significantly to their antioxidant potential (Oliveira et al., 2009; Harzallah et al., 2016). Several studies have identified phenolic constituents in fig extracts, such as quercetin derivatives, rutin, chlorogenic acid, and various organic acids, all of which play a role in scavenging free radicals and mitigating oxidative stress (Oliveira et al., 2009; Vallejo et al., 2012). These compounds contribute significantly to the fruit's antioxidant capacity and potential health benefits. Organic acids such as oxalic, citric, malic, quinic, shikimic, and fumaric acids have also been identified in fig extracts, which may play a role in flavor as well as bioactivity (Oliveira et al., 2009). Moreover, fig fruits contain pigments including carotenoids and α -tocopherol (vitamin E), both of which are known for their strong antioxidant properties (Solomon et al., 2006; Slavin, 2006). Oxidative stress, caused by excessive reactive oxygen species (ROS), contributes to cellular damage and the progression of various diseases. Phenolic compounds found in figs help mitigate this damage by supporting redox balance through multiple antioxidant mechanisms. These include acting as reducing agents, scavenging singlet oxygen, donating hydrogen atoms, and chelating metal ions (Ouchemoukh et al., 2012; Sirisha et al., 2010). These compounds help prevent oxidative stress-related conditions such as cancer, cardiovascular diseases, neurodegenerative disorders, and diabetes by inhibiting free radical formation and enhancing cellular defense systems (Sirisha et al., 2010). In this context, the fig fruit stands out as a significant source of natural antioxidants-including phenolics, flavonoids, carotenoids, and tocopherols—making it a valuable component of health-promoting diets. The effectiveness of these bioactive compounds is influenced not only by their concentration but also by factors such as ripeness stage and solvent polarity during extraction, which may alter their bioavailability and antioxidant potency (Harzallah et al., 2016; Hajam and Saleem, 2022; Li et al., 2013). The quantification of bioactive compounds in plant materials is significantly influenced by the choice of solvent used during extraction. The polarity of solvents such as ethanol, acetone, and ethyl acetate plays a pivotal role in the selective recovery of phenolic compounds and antioxidants from plant matrices (Hajam and Saleem, 2022; Li et al., 2013). Given that different phenolics exhibit varying solubility in polar and semi-polar solvents, optimizing solvent type is critical for maximizing the yield and antioxidant potential of extracts (Mujic et al., 2012). Moreover, the phytochemical composition of fig fruits can vary depending on their ripening stage, with several studies indicating that unripe and ripe fruits may differ in terms of their phenolic content and antioxidant activity (Pereira et al., 2017; Karantzi et al., 2021). These factors-solvent polarity and fruit maturity-must therefore be considered in evaluating the nutraceutical potential of fig-derived extracts.

Although several studies have reported the presence of phenolic compounds and antioxidant activities in fig, most have focused on either a single extraction solvent or a specific fruit maturity stage. There remains a lack of comprehensive data that simultaneously compare multiple solvents and the maturity-dependent variations in bioactive compound content. One of the most critical factors influencing the extraction efficiency of phenolics and their associated antioxidant activity is the type of solvent used during extraction (Ngo et al., 2017). Polar and semi-polar solvents are commonly employed for recovering phenolic compounds from plant materials due to

their ability to solubilize a wide range of phytochemicals (Do et al., 2014). Ethanol, acetone, and ethyl acetate were selected in this study to represent a range of polarities and extraction capabilities. Ethanol is widely used in food and nutraceutical applications and has been reported to be effective in extracting hydrophilic phenolic compounds (Burdock et al., 2004; Do et al., 2014). Acetone has been found suitable for extracting a broad spectrum of phenolics, including higher molecular weight flavanols (Do et al., 2014), while ethyl acetate, a medium-polar solvent, is effective for extracting moderately polar compounds such as flavonoid aglycones and certain phenolic acids [Kaewseejan et al., 2015). Since the extraction performance of solvents is also influenced by the plant matrix and tissue type, no universal solvent system can be recommended for all plant materials (Wijekoon et al., 2011). Comparative analyses involving these three solvents, particularly in both unripe and ripe fig samples, are still limited in the current literature. Addressing this gap could provide valuable insights into how extraction conditions and fruit developmental stages influence the phenolic composition and antioxidant capacity of fig extracts.

In this study, total phenolic contents and antioxidant capacities of unripe and ripe fig fruits were investigated using three different extraction solvents: ethanol, acetone, and ethyl acetate. The aim was to evaluate how solvent polarity and ripening stage affect the phytochemical profile and antioxidant activity of fig extracts. By conducting a comparative analysis, this study seeks to provide useful data for optimizing extraction strategies in the development of natural antioxidant sources from fig fruits.

MATERIALS AND METHODS

Chemicals

Analytical grade reagents including acetone (Sigma-Aldrich, 90872), ethyl acetate (Sigma-Aldrich, 58958), ethanol (Sigma-Aldrich, 493511), gallic acid (Sigma-Aldrich, G7384), ascorbic acid (Sigma-Aldrich, PHR1008), Trolox (Sigma-Aldrich, 238813), Folin–Ciocalteu reagent (Sigma-Aldrich, F9252), sulfuric acid (Sigma-Aldrich, 339741), sodium phosphate (Sigma-Aldrich, 342483), ammonium molybdate (Sigma-Aldrich, 277908), sodium carbonate (Sigma-Aldrich, S7795), and 2,2'-diphenyl-1-picrylhydrazyl (DPPH; Sigma-Aldrich, D9132) were used without further purification.

Plant Material

Unripe and ripe fig fruits were collected in August 2016 from a local genotype cultivated in the Akseki district of Antalya, Türkiye (coordinates: 37° 02′ 23″ N, 31° 46′ 48″ E; elevation: 1110 meters). Both unripe and ripe fruits were harvested from the same trees during the same period. Unripe fruits were identified by their firm texture, green outer skin, and bland taste, whereas ripe fruits exhibited a softer texture, purplish skin, and a distinctly sweeter flavor. The taxonomic identification of the plant material was confirmed according to the *Flora of Turkey and the East Aegean Islands* (Davis, P.H., 1965–1988). Immediately after collection, all fruit samples were frozen at –80 °C to preserve their phytochemical integrity until further processing.

Extractions of fig

Freshly harvested fig fruits were first freeze-dried using a lyophilizer (Scanvac CoolSafe Freeze Dryer, LaboGene, Denmark), then finely ground and stored at -20 °C until use. For the extraction process, 1 g of fig powder was combined with 80 mL of ethanol, ethyl acetate, or acetone (solid-to-solvent ratio 1:80, w/v), and homogenized at 10,000 rpm for 2 minute using a remote-controlled homogenizer (WiseTis, Witeg, Germany) (Tram et al., 2015). The homogenized mixtures were centrifuged at 4000 × g for 10 minutes at 4 °C, and the resulting supernatants were filtered through Whatman No. 1 filter paper. The pooled extracts were then evaporated under reduced pressure at 40 °C using a rotary evaporator (Scilogex RE100-Pro, USA) to remove the solvents. The dried fig extracts were stored at -20 °C until analysis. A total of six different fig extracts were prepared: unripe-ethanol, ripe-ethanol, unripe-ethyl acetate, ripe-ethyl acetate, unripe-acetone, and ripe-acetone. The extraction yield (%) was calculated using the following equation:

Extraction yield (%) =
$$\frac{\text{Total weight of extract obtained (g)}}{\text{Total weight of powered plant material (g)}} \times 100$$

Extraction yield was calculated from a single experiment per sample due to material limitations. Therefore, no standard deviation is provided for yield values. This represents a limitation of the study and should be considered when interpreting extraction efficiency results.

Determination of Total Phenolic Content (TPC)

The total phenolic content (TPC) of the fig extracts was determined using the Folin–Ciocalteu colorimetric method, based on the procedure originally described by Slinkard and Singleton (1977). Briefly, 0.5 mL of the extract was mixed with 2.5 mL of 0.2 N Folin–Ciocalteu reagent, followed by the addition of 2.0 mL sodium carbonate solution (75 g/L). The reaction mixture was incubated in the dark at room temperature for 2 hours. Absorbance was measured at 765 nm using a Multiskan-90 spectrophotometer (Thermo Scientific, USA). TPC values were calculated from a standard curve prepared with gallic acid (concentration range: 5 –100 ppm, $R^2 = 0.9966$) and expressed as milligrams of gallic acid equivalents (GAE) per 100 grams of dry extract weight (DW).

Total Antioxidant Capacity (TAC)

The total antioxidant capacity (TAC) of the extracts was evaluated via the phosphomolybdenum assay, as described by Prieto et al. (1999). For each sample, 0.1 mL of extract was added to 1 mL of reagent mixture containing 0.6 M sulfuric acid, 28 mM sodium phosphate, and 4 mM ammonium molybdate. The resulting solution was incubated at 95 °C for 90 minutes using a temperature-controlled heat block. After cooling the samples to room temperature, absorbance was recorded at 695 nm. The TAC was quantified using an ascorbic acid calibration curve (concentration range: 0.03125-0.5 mg/mL, R² = 0.9987) and results were presented as milligrams of ascorbic acid equivalents (AE) per gram of dry weight (DW).

Antioxidant Activity (DPPH)

The antioxidant activity of the fig extracts was determined using the DPPH (2,2'-diphenyl-1-picrylhydrazyl) free radical scavenging method, following the approach outlined by Brand-Williams et al. (1995), with slight modifications. In this assay, 0.1 mL of fig extract was added to 3.9 mL of a methanolic DPPH solution (6×10^{-5} M). The mixture was allowed to stand in the dark at approximately 25 °C for 30 minutes. After incubation, the absorbance of the solution was measured at 515 nm. The antioxidant activity was expressed as millimoles of Trolox equivalents (TE) per kilogram of dry weight (DW), based on a standard curve prepared with Trolox solutions (concentration range: 0.03–0.5 mM; R² = 0.9970).

Statistical Analysis

All experimental data were obtained from at least three independent replicates. The results are expressed as mean \pm standard deviation (SD). Statistical analysis was performed using GraphPad Prism (version 5.0; GraphPad Software, San Diego, CA, USA). Unpaired two-tailed t-tests were used to compare ripe and unripe fig samples within each solvent group (P1). One-way ANOVA followed by Tukey's post hoc test was conducted to evaluate differences among extraction solvents (P2). A p-value of less than 0.05 was considered statistically significant (*p < 0.05, **p < 0.01, ***p < 0.001).

RESULTS AND DISCUSSION

Extraction Yield

The extraction yields obtained in this study demonstrated a clear dependence on both the type of solvent and the ripening stage of the fig fruits. Ethanol was the most efficient solvent, particularly in ripe samples, yielding 80.3%, while acetone and ethyl acetate resulted in considerably lower yields (17.3% and 7.1%, respectively). These values are substantially higher than those reported in previous studies on *Ficus carica*, where yields typically ranged between 9.95% and 11.47% using pure methanol or mixed solvents (Soni et al., 2023; Kebal et al., 2024). This difference may be attributed to the use of fruit tissue rather than leaves, the polarity of ethanol, and the homogenization-assisted extraction method employed in the present study. It is well established that extraction yield is strongly influenced by plant matrix, solvent polarity, and extraction parameters (Soni et al., 2023).

Interestingly, the ripe–ethanol extract yielded significantly more than its unripe counterpart (26.5%), suggesting that ripening enhances extractability. This effect may result from structural changes in the cell wall or increased moisture content during ripening, which facilitate solvent penetration (Li et al., 2010). In contrast, ethyl acetate yielded less extract from ripe fruits. This reduction may be due to the degradation or transformation of semi-polar phenolics during ripening, such as aglycones or certain esters, which are more efficiently extracted by ethyl acetate. Ripening is also known to shift the metabolite profile toward more hydrophilic compounds, including sugars and organic acids, which are poorly soluble in ethyl acetate (Ydjedd et al., 2017). As a result, the reduced abundance of semi-polar extractable components may explain the lower yield obtained with ethyl acetate in ripe samples.

It is also important to consider that extraction yield reflects not only phenolic content but also the presence of other soluble compounds. As previously reported, substances such as proteins, carbohydrates, and sugars can contribute to higher extraction yields when extracted with polar solvents like ethanol or water (Zieliński and Kozłowska, 2000). This may partly account for the significantly higher yield observed in the ethanol–ripe group, where both phenolics and other co-extracted components may have contributed to the total yield. Therefore, the combined influence of solvent polarity, fruit developmental stage, and co-extractable non-phenolic compounds should be considered when evaluating extraction efficiency.



Figure 1. Extraction yields (%) of unripe and ripe fig extracts obtained using different solvents.

Total Phenolic Content (TPC)

The total phenolic contents of fig extracts varied depending on both the ripening stage and the solvent used for extraction. Phenolic contents were expressed as gallic acid equivalents (GAE), based on a standard curve generated from gallic acid solutions (Figure 2A). In unripe fig samples, the highest phenolic concentration was observed in the acetone extract (3680.25 ± 186.75 mg GAE/kg DW), followed by ethanol (1333.2 ± 49.4 mg GAE/kg DW) and ethyl acetate (595.7 ± 334.6 mg GAE/kg DW). For ripe fig samples, phenolic content was highest in the acetone extract (3976.2 ± 526.5 mg GAE/kg DW), followed by ethyl acetate (1220.45 ± 50.95 mg GAE/kg DW) and ethanol (153.65 ± 52.15 mg GAE/kg DW) (Figure 2B and Table 1). A statistically significant difference was observed between unripe and ripe ethanol extracts (p = 0.0387), indicating a marked decrease in phenolic content upon ripening, while no significant differences were found for acetone (p = 0.6869) or ethyl acetate (p = 0.3161) extracts. Overall, phenolic content was significantly influenced by solvent type (p = 0.0002), highlighting the importance of extraction conditions and suggesting that ethanol extraction is more sensitive to maturity-related changes in phenolic composition compared to acetone and ethyl acetate.

The TPC values obtained in this study fall within a wide range of previously reported data but are generally higher than many earlier findings. For instance, Bayrak et al. (2023) reported TPC values ranging between 35.98 and 47.30 mg GAE/100 g fresh weight (FW) for methanolic extracts of green to dark purple fig varieties from Türkiye's Black Sea region. Converting these values to dry weight basis (assuming ~80% moisture), the estimated range would be approximately 180–236 mg GAE/kg DW, which is considerably lower than our findings. Similarly, Ayoub et al. (2019) reported a TPC of 96.46 μ g GAE/mg extract (i.e., 96.46 mg GAE/g = 96,460 mg GAE/kg DW), which is significantly higher than our results, likely due to differences in plant part (leaves vs. fruit), extraction solvent, or sample origin.

Kebal et al. (2022) also reported variable TPC levels among fig varieties, with light peel types having lower values (~730.88 mg GAE/100 g FW) and darker types showing higher values (~951.06 mg GAE/100 g FW). Again, when these are adjusted to dry weight, they reflect much higher values, supporting the idea that fig phenolic content is strongly influenced by variety and peel pigmentation. Furthermore, previous studies have reported extreme variation in phenolic concentrations depending on fig variety and peel color, with total phenolic contents ranging from as low as 19.4 mg GAE/100 g FW to as high as 6147 mg GAE/100 g FW in fresh fig fruits (Caliskan and Polat, 2011; Harzallah et al., 2016; Konak et al., 2017). The relatively high TPC values observed in our study, particularly in acetone– unripe extracts, may be attributed to both the intermediate polarity of acetone and the greater abundance of extractable phenolics in unripe fig tissue. Additionally, differences in geographical origin, genotype, solvent system, and extraction methodology can all contribute to the variability

seen across studies. These findings underscore the importance of standardizing sample preparation and solvent selection when comparing phenolic contents across fig varieties.



Figure 2. A) Gallic acid standard graph. **B)** Total phenolic contents (mg GAE/kg) of unripe and ripe fig extracts obtained using different solvents (ethanol, acetone, and ethyl acetate). The error bars represent the standard deviation (SD) of three independent measurements (*p < 0.05, **p < 0.01, ***p < 0.001).

Total Antioxidant Capacity (TAC)

The total antioxidant capacity (TAC) of fig extracts varied according to both the solvent type and the ripening stage of the fruit. Total antioxidant capacity was expressed as ascorbic acid equivalents (AE), calculated based on a standard curve prepared with ascorbic acid solutions (Figure 3A). In unripe fig samples, total antioxidant capacity (TAC) was measured as 22.9 ± 0.51 mg AE/g for ethanol, 27.3 ± 0.82 mg AE/g for acetone, and 15.2 ± 0.11 mg AE/g for ethyl acetate. In ripe samples, TAC values significantly increased in acetone (41.9 ± 0.76 mg AE/g, p = 0.0484) and ethyl acetate (48.8 ± 0.87 mg AE/g, p = 0.0166) extracts, while a significant decrease was observed in the ethanol extract (8.6 ± 0.06 mg AE/g, p = 0.0227) (Figure 3B and Table 1). Among all tested samples, the highest TAC was recorded in ripe figs extracted with ethyl acetate. Statistical analysis revealed that the extraction solvent had a significant effect on TAC values (p < 0.0001), emphasizing the impact of solvent polarity. These findings indicate that both the choice of solvent and the maturity stage of the fruit substantially influence the antioxidant capacity of fig extracts, with ethyl acetate demonstrating superior extraction efficiency in ripe samples.

Although the phosphomolybdenum assay has been less frequently applied in fig-related studies, contextual comparisons can still be made using alternative antioxidant assays such as DPPH, ABTS, and FRAP. For instance, Konak et al. (2017) reported DPPH radical scavenging activities of dried fig extracts ranging from 387.26 to 462.16 µmol TE/100 g DM, while Kamiloglu and Capanoglu (2015) measured ABTS values between 222 and 255 mg TE/100 g DM in dried and fresh Turkish figs. In comparison, the highest DPPH activity observed in our study (ripe-ethyl acetate extract) reached 57.6 ± 10.37 mmol TE/kg DW (5760 µmol TE/100 g DW), indicating markedly stronger antioxidant potential. Moreover, FRAP values reported by the same authors (96 ± 25 mg TE/100 g DM) were also considerably lower than the TAC values measured in our ethyl acetate extracts using the phosphomolybdenum method (Kamiloglu and Capanoglu, 2015). Therefore, direct numerical comparisons with previous reports could not be made. Nonetheless, it is widely recognized that antioxidant capacity in figs is strongly influenced by phenolic composition, solvent polarity, and the maturity stage of the fruit (Çalışkan and Polat, 2012; Harzallah et al., 2016; Buyuktuncel et al., 2014). In this study, the high TAC values obtained from ripe fig samples extracted with ethyl acetate and acetone may be linked to factors such as changes in fruit composition during ripening and the polarity of the solvents.



Figure 3. A) Ascorbic acid standard graph. **B)** Total antioxidant capacity (mg AE/g) of unripe and ripe fig extracts obtained using different solvents (ethanol, acetone, and ethyl acetate). The error bars represent the standard deviation (SD) of three independent measurements (*p < 0.05, **p < 0.01, ***p < 0.001).

Antioxidant Activity (DPPH Assay)

The antioxidant activity of fig extracts, as determined by the DPPH radical scavenging method, varied depending on both the extraction solvent and the ripening stage of the fruit. Antioxidant activities were expressed as Trolox equivalents (TE), based on a standard curve generated from Trolox solutions (Figure 4A). In unripe fig samples, antioxidant activity was measured as 19.96 ± 3.32 mmol TE/kg DW for ethanol, 53.9 ± 6.17 mmol TE/kg DW for acetone, and 45.6 ± 0.05 mmol TE/kg DW for ethyl acetate. In ripe fig samples, the values were 6.0 ± 0.59 mmol TE/kg DW (ethanol), 54.5 ± 3.08 mmol TE/kg DW (acetone), and 57.6 ± 10.37 mmol TE/kg DW (ethyl acetate) (Figure 4B and Table 1). Although unripe–ethanol extracts showed significantly higher activity than ripe–ethanol ones (p = 0.1513), no statistically significant differences were detected between unripe and ripe samples in the acetone (p = 0.9559) or ethyl acetate (p = 0.4543) groups. According to statistical evaluation, antioxidant activity was significantly affected by solvent type (p = 0.0019), suggesting that both solvent polarity and fruit ripening stage play a role in modulating the radical scavenging capacity of fig extracts.

A previous study reported DPPH activities of aqueous fig extracts ranging from 1657.04 to 2241.20 mg Trolox/100 g DW (equivalent to 16.57–22.41 mmol TE/kg DW) (Nakilcioğlu and Hışıl, 2013). The values obtained in this study for acetone and ethyl acetate extracts—particularly in ripe samples—exceeded this range, supporting the efficiency of semi-polar solvents in recovering antioxidant compounds (Stalikas, 2007). Notably, ethanol-extracted unripe figs showed higher activity than their ripe counterparts, despite having a lighter color (Lopez-Martinez et al., 2012). This contrasts with findings by Harzallah et al. (2016), who reported higher DPPH activity in dark-colored fig peels, suggesting that pigmentation contributes to antioxidant capacity. Our results, however, imply that color intensity alone does not predict antioxidant potential (Rodríguez et al., 2013). Instead, factors such as phenolic concentration, solvent affinity, tissue integrity, and cultivar-specific traits likely interact to determine extract activity (Slatnar et al., 2011; Li et al., 2013; Pereira et al., 2017).



Figure 4. A) Trolox standard graph. **B)** Antioxidant activity (mmol TE/kg) of unripe and ripe fig extracts using different solvents (ethanol, acetone, and ethyl acetate) determined by the DPPH assay. The error bars represent the standard deviation (SD) of three independent measurements (*p < 0.05, **p < 0.01, ***p < 0.001).

Table 1. Total phenolic content (TPC), total antioxidant capacity (TAC), and antioxidant activity (DPPH assay) of unripe and ripe fig (Ficus carica L.) extracts obtained using different solvents.

Solvents	TPC (mg GAE/kg DW)		- P1	TAC (mg AE/g DW)		P1	Antioxidant activity (mmol TE/kg DW)		- P1
	Unripe Fig	Ripe Fig	• •	Unripe Fig	Ripe Fig	• -	Unripe Fig	Ripe Fig	• •
Ethanol	1333±49.4 ^b	154±52.2 ^b	0.0387	22.9±0.51 ^d	8.60±0.06 ^f	0.0227	19.9±3.32°	6.00±0.59 ^d	0.1513
Acetone	3680±186ª	3976±527ª	0.6869	27.3±0.82 ^c	41.9±0.76 ^b	0.0484	53.9±6.17ª	54.5±3.08ª	0.9559
Ethyl Acetate	595±335 ^b	1220±50.9 ^b	0.3161	15.2±0.11 ^e	48.8±0.87ª	0.0166	45.6±0.05 ^b	57.6±10.4ª	0.4543
P2	0.0002			< 0.0	0001	0.0019			

Note: Values are expressed as mean \pm standard deviation (n = 3). P1: Statistical comparison between unripe and ripe fig extracts for each solvent, calculated using unpaired t-test with Welch's correction. P2: Overall comparison among all groups using one-way ANOVA followed by Tukey's multiple comparison test. Superscript letters (e.g., ^a, ^b, ^c...) indicate statistically significant differences among all extract groups based on Tukey's test. Groups sharing the same letter are not significantly different (p > 0.05).

CONCLUSION

This study demonstrated that both solvent polarity and fruit ripening stage significantly affect the extraction yield, phenolic content, and antioxidant potential of fig extracts. Among the tested solvents, ethanol yielded the highest extraction efficiency, particularly in ripe samples. The highest phenolic content was obtained from ripe figs extracted with acetone, while the greatest antioxidant capacity (TAC) was observed in ripe figs extracted with ethyl acetate. Antioxidant activity measured by the DPPH method was highest in ripe–ethyl acetate extracts. However, the lack of experimental replication in extraction and the use of a single antioxidant assay (DPPH) limit the generalizability of the results. Future studies should incorporate a broader range of antioxidant tests such as ABTS and FRAP, repeated extractions, and explore different fig varieties and plant parts (e.g., peel, leaf) to validate and expand these findings. Overall, these results highlight the importance of optimizing extraction parameters to enhance the recovery of bioactive constituents and support the potential of fig fruits as a valuable natural source of antioxidants.

Declaration of interests

The authors declare that they have no conflict of interest.

Author Contributions

Zeynep DOĞRU: Conceptualization; data curation; formal analysis; investigation; methodology; project administration; software; writing— original draft; writing—review and editing.

Mehmet AKBULUT: Data curation; funding acquisition; investigation; writing-review and editing.

Hüsamettin VATANSEV: Conceptualization; formal analysis; funding acquisition; project administration; writing—review and editing.

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Article History	
Submission received:	27.03.2025
Revised:	26.05.2025
Accepted:	29.05.2025

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