

Extraction, Identification, and Quantification of Bioactive Compounds from Globe Artichoke (*Cynara cardunculus* var. *scolymus*)

(Pengekstrakan, Pengenalpastian dan Pengkuantitian Sebatian Bioaktif daripada Sayur Articok (*Cynara cardunculus* var. *scolymus*))

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ABSTRACT

This study looked at the best conditions for extracting bioactive compounds from globe artichoke (*Cynara cardunculus* var. *scolymus*), such as total phenolics, flavonoids, ascorbic acid, and inulin. The obtained results showed that the optimum conditions for extraction of total phenolics and flavonoids from receptacle and bracts of artichoke, when applying maceration at room temperature, with 70% methanol for 4 h. Genstin was the major phenolic compound (57.86 and 25.6 mg/100 g DM) in artichoke receptacle and bracts, respectively. The highest content of ascorbic acid extracted from artichoke parts was obtained using 1% citric acid solution at 25 °C. In addition, the optimum conditions for extraction of inulin were autoclave at 120 °C for 15 min. The most abundant essential amino acids were aromatic amino acids (phenylalanine and tyrosine), followed by valine, lysine, and leucine but sulfur amino acids were the limited amino acids found in the artichoke parts. Our results also suggested that bioactive compounds from artichoke extracts might have a promising future in the management of oxidative stress on the gastrointestinal tract and recommended as an attractive ingredient for developing functional food.

Keywords: Artichoke receptacle; ascorbic acid; bracts; flavonoids; inulin; phenolics

ABSTRAK

Kajian ini melihat kepada keadaan yang terbaik untuk mengekstrak sebatian bioaktif daripada sayur articok (*Cynara cardunculus* var. *scolymus*), seperti jumlah fenol, flavonoid, asid askorbik dan inulin. Keputusan yang diperolehi mendedahkan keadaan optimum untuk pengekstrakan jumlah fenol dan flavonoid daripada penyangga dan braktea articok, apabila menggunakan proses maserasi pada suhu bilik, dengan 70% metanol selama 4 jam. Genstin ialah sebatian fenol utama (57.86 dan 25.6 mg/100 g DM) masing-masing dalam penyangga dan brakta articok. Kandungan tertinggi asid askorbik yang diekstrak daripada bahagian articok telah diperolehi menggunakan 1% larutan asid sitrik pada suhu 25 °C. Di samping itu, keadaan optimum untuk pengekstrakan inulin ialah dengan menggunakan autoklaf pada suhu 120 °C selama 15 min. Asid amino perlu yang paling banyak ialah asid amino aromatik (fenilalanina dan tirosina), diikuti oleh valina, lisina dan leusina tetapi asid amino sulfur adalah terhad yang terdapat dalam bahagian articok. Keputusan kami juga mencadangkan bahawa sebatian bioaktif daripada ekstrak articok mungkin mempunyai masa depan yang cerah dalam pengurusan tekanan oksidatif pada saluran gastrousus dan disyorkan sebagai bahan yang menarik untuk membangunkan fungsian.

Kata kunci: Asid askorbik; brakta; fenol; flavonoid; inulin; penyangga articok

INTRODUCTION

The globe artichoke is a vegetable that belongs to the Asteraceae family and is widely produced and consumed (Foury 1989; Sonnante et al. 2003). It has been used as

a meal and a treatment in folk medicine for generations (Marzi et al. 1975). Before the flowers open, there are the flower buds that are a suitable part as human food. On the base of the budding artichoke flower-head is a collection

of numerous budding small flowers (an inflorescence), as well as several tasty bracts. The structure transforms into a gruff, nearly edible form as the buds bloom (Rottenberg et al. 1996).

The globe artichoke is an important part of human nutrition, particularly nowadays (UNESCO 2010). According to several reports, artichokes carried from North Africa were produced in Sicily and Spain, and as a rarity in Florence and Venice, between 800 and 1500 AD, and breeding was done in European monasteries at the same time. Italian farmers consume artichokes as a curiosity, yet artichokes had been known in Europe since the fifteenth century, and they were brought to the United States of America by European emigrants in the eighteenth century. Artichokes are produced at a rate of 1,793,015 tonnes per year, with over 1,075,800 tonnes produced in Europe. Artichoke growing has gained in commercial importance in China in recent years (FAO 2013). In addition, artichokes contribute significantly to the Mediterranean agricultural economy, which accounts for almost 60% of global production. Italy is the largest producer (approximately 474,000 tonnes per year), with Spain (215,000 tonnes), France (55,000 tonnes), and Greece (25,000 tonnes) following closely behind (FAO 2007; Lattanzio et al. 2009). Artichokes have been increasingly popular in many parts of the world, including the United States, particularly in California, South America, particularly in Argentina, Chile, and Peru, and North Africa, particularly in Turkey, Iran, and China (Ierna & Mauromicale 2010; Pandino et al. 2011a, 2011b).

Artichoke flower heads are young composite inflorescences that include the edible component of the plant, which can be eaten fresh, canned, or frozen. It has anticarcinogenic, anti-HIV, antioxidative, and diuretic qualities, as well as antifungal and antibacterial activities. Leaves extract is commonly employed in herbal medicine as choleric and hepatoprotectors (Lattanzio et al. 2009). Artichokes' phenolic acids, particularly caffeic acid and its derivatives mono- and di-caffeoylquinic acids, are chiefly responsible for the artichoke's health advantages. These chemicals' bioavailability in the human diet has long been known (Azzini et al. 2007).

The non-edible by-products of artichoke, the outer bracts, stems and leaves, which account for nearly 60% of the plant could be used as raw materials for the extraction of useful substances such as polyphenols and flavonoids (Angelov et al. 2015; Georgieva et al. 2016). They found that the optimal condition to extract these substances were 50 to 70% ethanol, at 70 °C for 30 min

and solvent to material ratio (10:1). Many researches have looked at the antioxidant effects of artichoke, but few have looked at the polyphenolic chemicals found in the plant's head or during different phases of the growth (Lombardo et al. 2010). The particular polyphenolics and flavonoids contained in *Cynara scolymus* L. extract are responsible for the antioxidant and hepatoprotective properties. Its inhibition of hepatic cholesterol biosynthesis was attributed to the presence of luteolin (Aktay et al. 2000).

The health advantages of artichoke were attributed to its high amount of polyphenolics and inulin (Pandino et al. 2011a; Sonnante et al. 2003). In human nutrition, polyphenolics play a critical function where they are preventing the coronary disorders; osteoporosis, diabetes mellitus, cancer, and neurodegenerative diseases (Clifford & Brown 2006). Because artichoke is high in antioxidants, it could be exploited in the pharmaceutical industry (Ceccarelli et al. 2010). Caffeic acid, chlorogenic acid, cynarin, and luteolin are all natural bioactive compounds found in artichokes. *In vitro* biological research using these components reduce the generation of reactive oxygen species (ROS), lipid peroxidation, and low density lipoprotein oxidation (LDL) (Juzyszyn et al. 2008).

The artichoke has high antioxidant capacity and polyphenol content, as well as a high inulin concentration. These substances are extremely beneficial to one's health. Artichokes are normally sold fresh, however the majority of their production might be used as a raw material in the food industry, particularly in the canning process (Ruiz-Cano et al. 2014). Only 15 - 40%, the heart and a few inner bracts, of artichoke are used in canning process, resulting in a large amount of raw materials as wastes (Ceccarelli et al. 2010). These wastes, which include significant levels of flammable valuable bioactive molecules, appear to have potential for use as natural substances in a variety of disciplines, especially as functional components in the food and pharmaceutical industries (Lattanzio et al. 2009).

Inulin extracted from artichoke is a soluble dietary fiber and due to its health-promoting qualities, such as digestibility, fat absorption, and the capacity to bind water and minerals, has a wide range of applications in the pharmaceutical business (Ruiz-Cano et al. 2014). Because humans lack the enzymes required for hydrolysis, inulin is neither digested or absorbed in the small intestine but fermented in the colon by beneficial bacteria such as bifidobacteria, making it a prebiotic (Cho & Samuel 2009; Frutos et al. 2008; Kelly 2008), so, it can be used as functional foods or dietary supplements (O'Sullivan

et al. 2010). Inulin's benefits include mineral binding, blood lipid absorption, and colon cancer prevention. Inulin is a low-calorie carbohydrate that can be used as a diet supplement, a fat-free snack and treat diabetes mellitus (Cho & Samuel 2009; Lattanzio et al. 2009; Pandino et al. 2011b). Inulin has found popularity as a fat substitute in table spreads, breads, dairy products, frozen sweets, and salad dressings. As a result, artichoke as an inulin source may open up new possibilities for unique and healthful food products in the food sector (López-Molina et al. 2005). The optimal conditions for extraction of inulin from a number of plant sources, such as chicory roots, Jerusalem artichoke tubers and globe artichoke and its by-product were temperature (60 - 80 °C) and time (20 - 60 min) (Redondo-Cuenca et al. 2021). While, the optimal condition for extraction of inulin using microwave was time (5-6 min) and microwave power (450-700W) at 95 °C (Abood 2020; Xiao et al. 2013). Elzeny (2020) stated that the optimal conditions for extraction of inulin from chicory leaves and roots by water bath method were (temperature 65 °C for 20 min), and using autoclave method were (temperature 121 °C for 6 min), while for microwave method were (microwave power 540 W for 5 min).

Because of the foregoing characteristics, it would be prudent to investigate the best conditions for extracting bioactive compounds such as phenolic compounds, flavonoids, inulin, and ascorbic acid from artichoke receptacles and bracts. In addition, phenolic compounds and amino acids were identified and quantified.

MATERIALS AND METHODS

MATERIALS

The artichoke fruits (*Cynara scolymus* L.), family *Asteraceae*, were collected from a farm at Kafr El-Dawar region, EL-Behera Governorate, Egypt (Season 2018). Bracts (outer parts of fruits) were manually separated from receptacle (edible part) and dried in a ventilated oven at 50±2 °C for two days. After that, they cut up into small pieces and ground in food grinder. Finally, the mills were sieved in a 50 mesh sieve and stored in polyethylene bags at 4 °C until needed.

METHODS

DETERMINATION OF TOTAL POLYPHENOLICS CONTENT OF ARTICHOKE BRACTS AND RECEPTACLE

Determination of total polyphenolics of artichoke parts was achieved in two steps: extraction then estimation of content.

Extraction of Total Polyphenolics from Artichoke Parts

Total phenolic compounds extraction was achieved accordance to the procedure outlined by Pereira et al. (2007). A 5 g sample was macerated in 50 mL of several solvents (99% methanol, 95% ethanol, 70% aqueous methanol, 70% aqueous ethanol and distilled water) at room temperature for different periods (1, 2, 3, 4, and 24 h). The extracts were filtered over a Büchner funnel. The filtrates were kept at 4 °C until determination of total phenolic compounds.

Estimation of Total Polyphenolics

Using the procedure provided by Thaipong (2006), the total polyphenolic compounds (TPC) content was estimated using Folin Ciocalteu reagent. A UV spectrophotometer (Varian, Melbourne, VIC, Australia) was used in measuring the content of polyphenolics. Gallic acid was used as a standard and the absorbance was read at 760 nm. The results were measured in mg of gallic acid equivalent/g dry matter (DM).

IDENTIFICATION OF PHENOLIC COMPOUNDS BY HPLC

The identification of phenolic compounds was carried out at the National Research Center in Cairo, Egypt, using the high-performance liquid chromatography (HPLC) technique as described by Hammouda et al. (1993).

TOTAL FLAVONOIDS DETERMINATION

Total flavonoids were determined using the technique described by Vuong et al. (2014). A UV spectrophotometer (Varian, Melbourne, VIC, Australia) was used to detect the absorbance at 510 nm. Using quercetin as a standard, total flavonoids content was calculated as mg of quercetin equivalent (QE)/g of dry sample.

DETERMINATION OF INULIN IN ARTICHOKE PARTS

The procedure recommended by Prosky and Hoebregs (1999) was used to determine inulin. Three distinct strategies were used to obtain the data. The first was heating for 30 min in a water bath at 60 °C, the second for 15 min in an autoclave at 121 °C, and the last for 5 min in a microwave. The ratio of sample to solvent was 1:10. The following equation (1) was used to calculate the inulin content:

1 mg inulin = 1.85 mL of 0.01 N potassium permanganate solution (1)

ASCORBIC ACID (VITAMIN C) DETERMINATION

The Folin-Ciocalteu Reagent (FCR) method was used to measure the ascorbic acid content of the artichoke receptacle and bracts as described by Dashman (1991), using 50 mL of different solvents (distilled water, 1% aqueous citric acid, and 2% aqueous citric acid) at various temperatures (4, 25, and 60 °C) for 30 min, exactly 5 g of fresh material was extracted. At 760 nm using a UV spectrophotometer (Varian, Melbourne, VIC, Australia), the absorbance was measured. The ascorbic acid content was measured in g/100 g of fresh sample.

DETERMINATION OF AMINO ACIDS COMPOSITION

Amino acids composition determination was performed in National Research Center, Cairo, Egypt according to the procedure of Duranti and Cerletti (1979) using amino acid analyzer (Beckman amino acid analyzer, Model 119CL).

STATISTICAL ANALYSIS

The data were statistically analyzed by T test analysis of variance (ANOVA) procedure by SPSS (Version 16.0) software.

RESULTS AND DISCUSSION

EFFECT OF EXTRACTION TIME AND SOLVENT TYPE ON TOTAL PHENOLICS CONTENT OF ARTICHOKE RECEPTACLE

The data in Table 1 shows that solvent concentration and

solvent type had a significant impact on total phenolics content. 70% methanol and 70% ethanol followed by 99% methanol and 95% ethanol were more effective than distilled water. The highest content of phenolics (43.22 mg GAE/g) was found using 70% methanol. The lowest content of phenolics (about 24 mg GAE/g) was found using distilled water and 95% ethanol. It could be said that 70% methanol was more effective than 70% ethanol.

The results of previous studies have observed that aqueous solution of ethanol is effective for polyphenols extraction from artichokes, and it keeps the activity of their antioxidant capacity (Georgieva et al. 2016; Zuurro et al. 2014). Georgieva et al. (2016) reported that the extraction of maximum of polyphenols from artichoke wastes is achieved with 50% and 70% ethanol. Chirinos et al. (2007) mentioned that solvents with a high polarity (water) and nonpolar ones (chloroform and hexane) are not appropriate for extracting a high content of polyphenols because they are mostly polar compounds. Furthermore, using water as the only solvent results in an extract that is high in contaminants (organic acids, sugars, and soluble proteins, for example) as well as phenolic compounds. Also, absolute alcoholic solvents reduce the amount of phenolic components in the extract. As a result, the mixing of water and organic solvents creates a moderately polar medium that ensures the best conditions for polyphenols extraction. The different surface tension of solvents might have slight influence due to change in surface wettability. The results in the same Table 1 show that total phenolics content increases gradually with the extraction time up to 4 h then the increase was not significant.

TABLE 1. Effect of extraction time and solvent type on total phenolics content of artichoke receptacle

Extraction time (h)	Solvent type				
	95% ethanol	70% ethanol	99% methanol	70% methanol	Water
	Total phenolics content mg GAE*/g DM				
1	^D 16.51± 0.16 ^d	^D 31.00± 0.46 ^b	^D 23.05± 0.26 ^c	^D 35.33± 0.62 ^a	^D 16.48± 0.16 ^d
2	^C 17.58± 0.19 ^d	^C 33.55± 0.38 ^b	^C 25.53± 0.28 ^c	^C 37.48± 0.73 ^a	^C 17.93± 0.17 ^d
3	^B 21.33± 0.26 ^d	^B 36.32± 0.47 ^b	^B 28.84± 0.19 ^c	^B 39.89± 0.76 ^a	^B 20.85± 0.26 ^d
4	^A 24.09± 0.21 ^d	^A 38.80± 0.66 ^b	^A 33.88± 0.36 ^c	^A 42.96± 0.82 ^a	^A 23.89± 0.22 ^d
24	^A 24.20± 0.24 ^d	^A 38.86± 0.53 ^b	^A 33.98± 0.46 ^c	^A 43.22± 0.77 ^a	^A 23.93± 0.25 ^d

Values in the row with the same small letter are not significantly different $P \leq 0.05$

Values in column with the same capital letter are not significantly different $P \leq 0.05$

*GAE means Gallic acid equivalent

The findings in Table 1 also show that total phenolics of edible part (receptacle) in artichoke extracted using 70% ethanol ranged from 31.00 to 38.86 mg GAE/g while in case of using 95% ethanol, the total phenolics content ranged from 16.51 to 24.20 mg GAE/g, with extraction time for 24 h yielding the maximum quantity. It could be observed that the amount of total phenolics extracted using 70% methanol ranged from 35.33 to 43.22 mg GAE/g indicating that 70% methanol was more effective than 70% ethanol. So, 70% methanol was the best solvent for 4 h followed by 70% ethanol for the same time in extracting the total phenolic compounds from artichoke receptacle. The results in Table 1 also show that the amount of total phenolics increased with increasing the extraction time but the variation between 4 h and 24 h was not significant. Anwar et al. (2013) extracted phenolic chemicals from cauliflower using absolute ethanol, 80% ethanol, absolute methanol, and 80% methanol. They found that each solvent system did vary significantly in their ability to extract phenolic compounds and the aqueous methanol was superior solvent. El Sohaimy (2013) discovered that cooking artichokes for 20 min in boiling water raised total phenolic content from 6.21 mg/100 g in fresh samples to 10.23 mg/100 g in cooked samples. He also reported that aqueous methanol was the superior solvent for extraction of polyphenolics

from artichoke receptacle and bracts comparing with other solvents, while water had the lowest values of polyphenolic compounds. El Sohaimy (2014) stated that the globe artichoke's total phenolic content was found to be 30.70 ± 1.87 mg GAE/g DM.

EFFECTS OF EXTRACTION TIME AND SOLVENT TYPE ON TOTAL PHENOLICS CONTENT OF ARTICHOKE BRACTS

The results in Table 2 show that the highest amount of total phenolic compounds of artichoke bracts was obtained using 70% methanol followed by 70% ethanol. The lowest content of phenolics was found using water and 95% ethanol. It could be observed that the content of phenolics from artichoke bracts increased gradually with time up to 4 h then the increasing was not significant. From the results in Tables 1 and 2, it could be observed that the total phenolic compounds extracted from artichoke receptacle was higher than that extracted from bracts. The obtained results agree with that published by Al-Subhi (2020), who found that the free total phenolic content of artichoke heads (14.16 mg/g DM) is higher than that of artichoke leaves (9.06 mg/g DM). However, bound phenolic compounds were found in lower concentrations in both the leaves and the head parts of the artichoke (5.35 and 4.20 mg/g DM, respectively).

TABLE 2. Effect of extraction time and solvent type on total phenolics content of artichoke bracts

Extraction time (h)	Solvent type				
	95% ethanol	70% ethanol	99% methanol	70% methanol	Water
	Total phenolics content mg GAE*/g DM				
1	^D 13.84± 0.12 ^d	^D 28.00± 0.26 ^b	^D 21.93± 0.12 ^c	^D 31.65± 0.32 ^a	^D 13.66± 0.12 ^d
2	^C 14.90± 0.15 ^d	^C 29.68± 0.19 ^b	^C 22.09± 0.16 ^c	^C 33.97± 0.24 ^a	^C 15.32± 0.35 ^d
3	^B 16.54± 0.18 ^d	^B 30.46± 0.36 ^b	^B 25.00± 0.22 ^c	^B 36.96± 0.36 ^a	^B 16.90± 0.32 ^d
4	^A 19.87± 0.17 ^d	^A 34.67± 0.33 ^b	^A 28.89± 0.19 ^c	^A 38.29± 0.42 ^a	^A 19.54± 0.22 ^d
24	^A 20.22± 0.12 ^d	^A 34.98± 0.06 ^b	^A 29.12± 0.12 ^c	^A 38.61± 0.12 ^a	^A 19.63± 0.12 ^d

Values with the different small letter in row are significant $P \leq 0.05$

Values with the different capital letter in column are significant $P \leq 0.05$

*GAE means gallic acid equivalent

It could be said that 70% aqueous methanol was the effective solvent for 4 h followed by 70% aqueous ethanol for the same time in extracting the total phenolic

compounds from artichoke receptacle and bracts. According to the European Medicines Agency, artichoke agro-industrial by-products such as leaves, outer bracts,

and stems, which account for around 80% of the plant, could be a promising and cost-effective source of polyphenolics (Lattanzio et al. 2009).

EFFECT OF THE EXTRACTION TIME AND SOLVENT TYPE ON TOTAL FLAVONOIDS CONTENT OF ARTICHOKE RECEPTACLE

The results in Table 3 show that total flavonoids content

of artichoke receptacle extracted using 95% ethanol ranged from 2.15 to 5.49 mg QE/g, and the highest amount was found with extraction time of 24 h. In case of using 70% ethanol, the content of total flavonoids ranged from 3.86 to 6.66 mg QE/g, and the highest amount was found with extraction time 24 h, while the lowest amount was found with extraction time of 1 h.

TABLE 3. Effect of the extraction time and solvent type on total flavonoids content of artichoke receptacle

Extraction time (h)	Solvent type				
	95% ethanol	70% ethanol	99% methanol	70% methanol	Water
	Total flavonoids content mg QE*/g DM				
1	^D 2.15± 0.06 ^d	^D 3.86± 0.16 ^b	^D 3.13± 0.18 ^c	^D 5.30± 0.26 ^a	^D 1.94± 0.07 ^d
2	^C 3.56± 0.14 ^d	^C 4.75± 0.21 ^b	^C 4.03± 0.26 ^c	^C 6.51± 0.28 ^a	^C 3.19± 0.11 ^d
3	^B 4.39± 0.16 ^d	^B 5.66± 0.26 ^b	^B 4.85± 0.17 ^c	^B 7.45± 0.33 ^a	^B 4.22± 0.16 ^d
4	^A 5.29± 0.26 ^d	^A 6.59± 0.25 ^b	^A 5.77± 0.24 ^c	^A 8.63± 0.26 ^a	^A 5.31± 0.15 ^d
24	^A 5.49± 0.19 ^d	^A 6.66± 0.17 ^b	^A 5.81± 0.26 ^c	^A 8.67± 0.31 ^a	^A 5.47± 0.26 ^d

Values with the different small letter in row are significant $P \leq 0.05$

Values with the different capital letter in each column are significant $P \leq 0.05$

*QE means quercetin equivalent

It could be observed that the amount of flavonoids extracted using 70% methanol ranged from 5.30 to 8.67 mg QE/g indicating that 70% methanol was more effective than 70% ethanol. So, 70% methanol was the best solvent for 4 h followed by 70% ethanol for 4 h in extracting the total flavonoid from artichoke receptacle. One can say that 4 h was the favorable period for extracting of the flavonoids from artichoke receptacle.

EFFECT OF THE EXTRACTION TIME AND SOLVENT TYPE ON TOTAL FLAVONOIDS CONTENT OF ARTICHOKE BRACTS

The results in Table 4 show that the highest amount of total flavonoids content of artichoke bracts was extracted using 70% methanol followed by 70% ethanol with extraction time of 24 h. The lowest amount of flavonoids was found using distilled water and 95% ethanol. The amount of total flavonoids extracted using 70% methanol ranged from 4.19 to 7.84 mg QE/g indicating that 70% methanol was more effective, in general, than 70%

ethanol. It could be observed that the content of flavonoids from artichoke bracts increased gradually with time up to 4 h then the increasing was not significant. From the results in Tables 3 and 4, it could be observed that the content of flavonoids extracted from artichoke receptacle was higher than that extracted from its bracts. Al-Subhi (2020) has also found that free and bound flavonoids contents in artichoke head extracts were significantly high (9.85 and 4.06 mg/g DM) when compared to the leaves of artichoke (5.91 and 2.17 mg/g DM).

POLYPHENOLICS COMPOSITION OF ARTICHOKE RECEPTACLE AND BRACTS

It is clear from the data in Table 5 that genstin was the most abundant phenolic compound in the artichoke receptacle while catechine dadzin was the second one and cinnamic was the third. Genstin was also the major phenolic compound of artichoke bracts and chrysin was the second one. According to Al-Subhi (2020), 5-o-Caffeoylquinic acid (Chlorogenic acid) was

discovered to be in high concentration (5.1021 mg/100 g dried sample) in the head, but in low concentration (0.7145 mg/100 g) in the leaves. Negro et al. (2012) stated that the most prevalent polyphenols discovered in artichoke plant tissues were chlorogenic acid and cynarin.

TABLE 4. Effect of the extraction time and solvent type on total flavonoids content of artichoke bracts

Extraction time (h)	Solvent type				
	95% ethanol	70% ethanol	99% methanol	70% methanol	Water
	Total flavonoids content mg QE*/g DM				
1	^D 2.07± 0.16 ^d	^D 3.82± 0.22 ^b	^D 2.98± 0.12 ^c	^D 4.19± 0.06 ^a	^D 2.21± 0.16 ^d
2	^C 3.08± 0.17 ^d	^C 4.81± 0.16 ^b	^C 3.66± 0.11 ^c	^C 5.31± 0.26 ^a	^C 3.16± 0.25 ^d
3	^B 3.87± 0.26 ^d	^B 5.65± 0.21 ^b	^B 4.57± 0.20 ^c	^B 6.58± 0.16 ^a	^B 3.75± 0.14 ^d
4	^A 4.60± 0.19 ^d	^A 6.29± 0.09 ^b	^A 5.48± 0.16 ^c	^A 7.71± 0.13 ^a	^A 4.58± 0.11 ^d
24	^A 4.63± 0.16 ^d	^A 6.32± 0.23 ^b	^A 5.47± 0.14 ^c	^A 7.84± 0.22 ^a	^A 4.66± 0.13 ^d

Values with the different small letter in row are significant $P \leq 0.05$

Values with the different capital letter in column are significant $P \leq 0.05$

*QE means quercetin equivalent

TABLE 5. Identification and quantification of phenolic compounds from artichoke receptacle and bracts extracts using HPLC (as caffeic acid derivatives)

Phenolic compounds	Receptacle (mg/g)	%	Bracts (mg/g)	%
Phenol	0.49	2.28	0.98	5.25
P-coumaric	0.90	4.19	0.81	4.34
Salicylic	1.33	6.19	0.70	3.75
Feruleic	1.23	5.72	0.61	3.27
Cinnamic	1.77	8.24	0.78	4.18
Quercetin	0.66	3.07	0.74	3.97
Euganol	0.52	2.43	0.53	2.84
Chrysin	0.71	3.30	1.91	10.25
Galangin b	1.23	5.72	1.82	9.75
3-5-dimethoxy	0.42	1.95	0.47	2.52
Cynarin	0.53	2.47	1.52	8.28
Benzyl	0.72	3.35	0.96	5.15
Catechine Dadzin	1.91	8.89	1.28	6.86
Genstin	6.78	31.54	3.76	20.15
Dadazien	1.45	6.75	0.95	5.09
Genstein	0.84	3.91	0.81	4.34
Total phenolic	21.49	100	18.66	100

Shen et al. (2010) noted that artichoke contains three active compounds: 5- chlorogenic acid, cynarin and 1,5-di-ocaffeoylquinic acid, which are likely to be responsible for the antiatherogenic activity. Catechine dadzin was the most abundant phenolic compound in artichoke receptacles followed by cynarin, while catechine dadzin was the most abundant phenolic compound in artichoke bracts (27.60 mg/100 g). The major phenolic acid of artichoke receptacle was salicylic acid, represented about 12.71 mg/100 g followed by caffeic acid (3.96 mg/100 g), whereas the major phenolic acids of artichoke bracts were chlorogenic, caffeic and cinnamic acids which valued 10.65, 8.82, and 6.02 mg/100 g, respectively (Gomaa 2010). The most prevalent polyphenol in the artichoke extract is diaffeoylquinic acid, which accounts for over half of the total phenolic components (Nearly 46%). It is possible that this chemical is a cynarin isomer like 1,5 di-O-caffeoylquinic acid (Ruiz-Cano et al. 2014). Dicafeoylquinic acid and chlorogenic acid account for over 80% of the phenolic chemicals found. However, the flavone was found at far lower concentrations (around 5%). In Italian artichoke

varieties, apigenin-7-glucuronide was the most abundant flavonoid (Pandino et al. 2015). The differences between our results and these authors may be related to the status of the raw materials, the origin and conditions of agriculture.

ASCORBIC ACID CONTENT OF ARTICHOKE RECEPTACLE AT DIFFERENT TEMPERATURES AND SOLVENTS

The results in Figure 1 show that, in case of using the water as a solvent, the amount of ascorbic acid extracted from artichoke receptacle increased with increasing the temperature but using of citric acid with high temperature has negative effect on the amount of ascorbic acid, especially with the higher concentration of citric acid (2%). This result may be due to the decomposition of ascorbic acid at high temperature and acidity. It could be observed that the highest amount of ascorbic acid (0.87 ± 0.03) from artichoke receptacle was obtained using 1% citric acid, as a solvent, at 25 °C but the lowest amount (0.68 ± 0.07) was obtained using 2% citric acid at 60 °C.

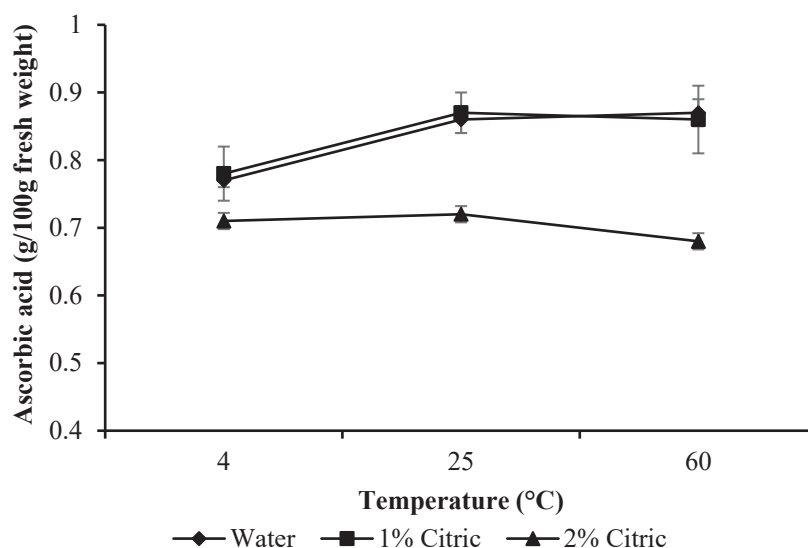


FIGURE 1. Ascorbic acid (g/100 g fresh weight) content of artichoke receptacle at different temperatures and solvents

ASCORBIC ACID CONTENT OF ARTICHOKE BRACETS AT DIFFERENT TEMPERATURES AND SOLVENTS

The results in Figure 2 indicated that the yield of ascorbic acid extracted from bracts of artichoke increased from 0.23% to 0.28% of fresh weight with increasing the temperature from 4 °C to 60 °C in case of using the water as a solvent.

It was noticed that the using of citric acid as a solvent for extraction of ascorbic acid from artichoke, bracts improved its yield especially at low temperatures (4 °C and 25 °C) and lower citric acid concentrate (1%) but at higher temperature (60 °C) and higher concentrate of citric acid (2%) the amount of ascorbic

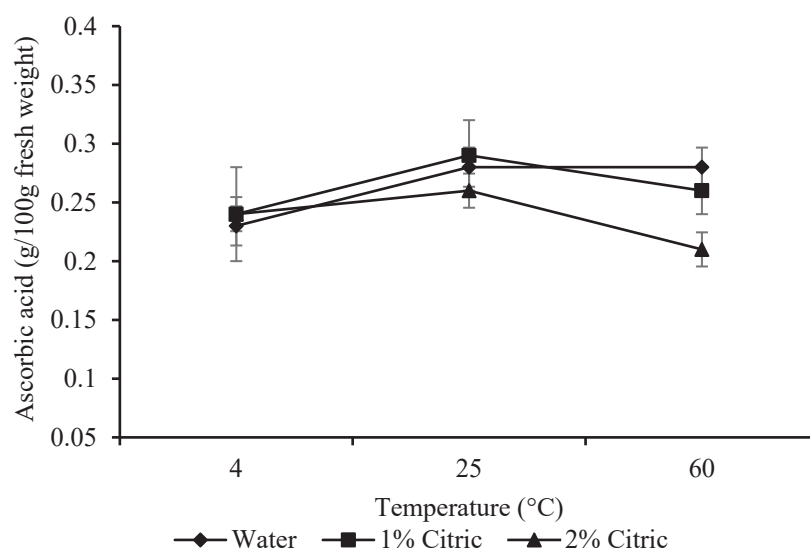


FIGURE 2. Ascorbic acid (g/100 g fresh weight) content of artichoke bracts at different temperatures and solvents

acid decreased. The high temperature and acidity may cause decomposition of ascorbic acid. From the results in Figures 1 and 2, it could be observed that the amount of ascorbic acid extracted from artichoke receptacle is higher than that extracted from artichoke bracts. In addition, no significant differences were recorded between the two solvents of extraction, water and citric acid, so we recommended to use water instead of citric acid because it is not expensive.

The content of vitamin C of vegetables cooked in boiling water was found to greatly destroy. In artichoke, vitamin C is destroyed by percentages between 23.9 and 94% during the boiling process. This could be owing to its high-temperature instability and water-soluble nature, which causes it to be removed

from the boiling water (Bach 2012). Also, Agbemaflle et al. (2012) found the cooking at long time reduced the concentration of vitamin C. As a result, it is recommended that vegetables be cooked for the shortest time possible in order to preserve the majority of this essential vitamin.

EXTRACTABLE INULIN% FROM RECEPTACLE AND BRACTS OF ARTICHOKE USING DIFFERENT METHODS

The results in Figure 3 showed that artichoke receptacle had higher amount of inulin than that of artichoke bracts using all extraction methods. The most inulin was extracted using an autoclave at 120 °C for 15 min. The lowest amount of inulin was obtained using microwave for 5 min.

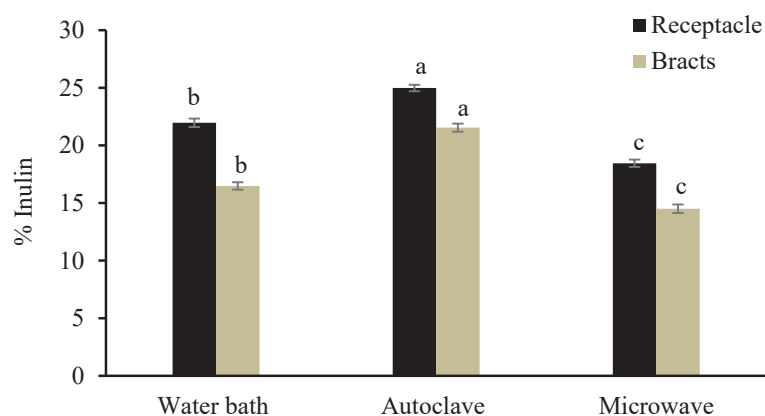


FIGURE 3. Extractable inulin% from receptacle and bracts of artichoke using different methods. Where water bath was at 60 °C for 30 min, autoclave was at 120 °C for 15 min, and microwave was for 5 min, values with the different letters are significant at $P \leq 0.05$

As shown in Figure 3, the amount of extractable inulin increased with rising the temperature and time. Gaafar (2010) found that extracting the largest quantity of inulin from artichoke was achieved using a sample to solvent ratio of 1:5 w/v at 80 °C for 90 min. As reported by Leroy et al. (2010), the effect of storage temperature and preservation method on inulin content in artichoke is great.

AMINO ACID COMPOSITION OF ARTICHOKE RECEPTACLE AND BRACTS

Amino acid content of artichoke receptacle and bracts proteins are shown in Table 6 along with the provisional pattern recommended by the FAO/WHO/UNU (1985). Results in Table 6 show that all essential amino acids of artichoke receptacle were higher than these of artichoke bracts.

TABLE 6. Amino acids composition of artichoke receptacle and bracts (% of protein)

Amino acids	Artichoke receptacle	Artichoke bracts	FAO/WHO/UNU (1985) pattern
Essential amino acids (EAA)			
Histidine	2.74	2.16	1.90
Isoleucine	4.21	4.00	2.80
Leucine	6.40	5.95	6.60
Lysine	6.70	5.99	5.80
Methionine + Cystine	0.32	1.01	2.50
Phenylalanine+ Tyrosine	9.75	8.93	6.30
Threonine	3.95	3.87	3.40
Valine	6.97	6.82	3.50
Tryptophan	ND*	ND	1.10
Total essential amino acids	41.04	38.73	33.90
Non-essential amino acids (NEAA)			
Alanine	4.67	5.14	
Arginine	9.24	8.12	
Aspartic acid	21.61	22.44	
Glutamic acid	10.05	10.15	
Glycine	3.60	5.35	
Proline	6.24	6.55	
Serine	3.65	3.52	
Total non-essential amino acids	58.96	61.27	
EAA/NEAA	0.70	0.63	

* ND not determined.

It could be observed that all essential amino acids except methionine + cystine amino acids of artichoke receptacle and bracts were higher than those of provisional pattern recommended by the FAO/WHO/UNU (1985). Table 6 shows that aromatic amino acids (phenylalanine and tyrosine) were the most abundant essential amino acids in the receptacle and bracts artichoke, followed by valine, lysine, and leucine. The results show that sulfur amino acids (methionine and cysteine) were the first limited amino acids and histidine was the second one for receptacle and bracts artichoke protein.

As for non-essential amino acids, it could be observed that aspartic and glutamic acids are the most abundant amino acids of receptacle and bracts artichoke proteins. That is to say, the acidic amino acids dominate amino acid composition and account near from a third (31.66 and 32.59%) for receptacle and bracts of the total amino acids. For artichoke receptacle protein, aspartic accounted 21.61% while, glutamic acid showed a value of 10.05%. Similarly, bracts protein showed to have high content of aspartic acid (22.44%) and glutamic acid (10.15%). Abu-Salem and Ibrahim (1989) reported that protein concentration from artichoke bracts was characterized by high levels of leucine and glutamic acid followed by aspartic acid. Moharram et al. (1981) found that, all amino acids except tryptophan, cysteine, lysine, and methionine were found in lower levels while the other essential amino acids were present in high levels in the protein of artichoke as compared with the FAO provisional pattern.

The amino acids composition of artichoke bracts protein had lower total concentration of essential amino acids (39.73%) than that of the artichoke receptacle protein (41.02%). In this concern, the two tested proteins showed to have higher total amounts of essential amino acids than the FAO/WHO/UNU (1985) recommended pattern. These results are similar to findings of Hussein et al. (1999) and Moharam et al. (1981).

CONCLUSION

The obtained results in this investigation show that optimum conditions for extraction of phenolics, flavonoids from receptacle and bracts of artichoke were maceration at room temperature in 70% methanol for 4 h. Genistin was the major phenolic compound (57.86 and 25.6 mg/100 g DM) in receptacle and bracts of artichoke, respectively. The highest content of ascorbic acid extracted from artichoke receptacle and bracts was obtained using 1% citric acid solution at 25 °C. The optimum conditions for extraction of inulin were using

autoclave at 120 °C for 15 min. Aromatic amino acids followed by valine, lysine, and leucine were the most abundant essential amino acids but sulfur amino acids were the limited amino acids in receptacle and bracts of artichoke. Receptacles of artichoke contain higher contents of phenolics, flavonoids, vitamin C, inulin, and essential amino acids compared with bracts of artichoke.

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REFERENCES

- Abood, A. 2020. Microwave-assisted extraction of inulin from Jerusalem artichoke and partial acid hydrolysis. *Iraqi Journal of Agricultural Sciences* 51(1): 401-410.
- Agbemaflle, R., Obodai, E.A., Adukpoo, G.E. & Amprako, D.N. 2012. Effects of boiling time on the concentrations of vitamin c and beta-carotene in five selected green vegetables consumed in Ghana. *Advances in Applied Science Research* 3(5): 2815-2820.
- Aktay, G., Deliorman, D., Ergun, E., Ergun, F., Yeşilada, E. & Cevik, C. 2000. Hepatoprotective effects of Turkish folk remedies on experimental liver injury. *Journal of Ethnopharmacology* 73(1-2): 121-129.
- Al-Subhi, F.M.M. 2020. Artichoke as a tool to natural antioxidants for lowering diabetics and hypolipidemia parameters. *Alexandria Science Exchange Journal* 11: 46-54.
- Angelov, G., Georgieva, S., Boyadzhieva, S. & Boyadzhiev, L. 2015. Optimizing the extraction of globe artichoke wastes. *Comptes rendus de l'Académie bulgare des Sciences* 68(10).
- Anwar, F., Kalsoom, U., Sultana, B., Mushtaq, M., Mehmood, T. & Arshad, H. 2013. Effect of drying method and extraction solvent on the total phenolics and antioxidant activity of cauliflower (*Brassica oleracea* L.) extracts. *International Food Research Journal* 20(2): 653.
- Azzini, E., Bugianesi, R., Romano, F., Di Venere, D., Miccadei, S., Durazzo, A., Foddai, M., Catasta, G., Linsalata, V. & Maiani, G. 2007. Absorption and metabolism of bioactive molecules after oral consumption of cooked edible heads of *Cynara scolymus* L. (cultivar Violetto di Provenza) in human subjects: A pilot study. *British Journal of Nutrition* 97(5): 963-969.
- Bach, V. 2012. Sensory quality and chemical composition of culinary preparations of root crops. PhD Thesis. Department of Food Science, Aarhus University (Unpublished).
- Ceccarelli, N., Curadi, M., Picciarelli, P., Martelloni, L., Sbrana, C. & Giovannetti, M. 2010. Globe artichoke as a functional food. *Mediterranean Journal of Nutrition and Metabolism* 3(3): 197-201.

- Chirinos, R., Rogez, H., Campos, D., Pedreschi, R. & Larondelle, Y. 2007. Optimization of extraction conditions of antioxidant phenolic compounds from mashua (*Tropaeolum tuberosum* Ruiz & Pavón) tubers. *Separation and Purification Technology* 55(2): 217-225.
- Cho, S.S. & Samuel, P. 2009. *Fiber Ingredients: Food Applications and Health Benefits*. Boca Raton: CRC Press. pp. 1-516.
- Clifford, M. & Brown, J. 2006. Dietary flavonoids and health-broadening the perspective. In *Flavonoids: Chemistry, Biochemistry and Applications*, edited by Anderson, O.M. & Markham, K.R. Florida: CRC Press. pp. 319-370.
- Dashman, T., Blocker, D.E. & Baker, N. 1991. *Laboratory Manual for Human Nutrition*. Reading: Harwood Academic Publishers. pp. 1-237.
- Duranti, M. & Cerletti, P. 1979. Amino acid composition of seed proteins of *Lupinus albus*. *Journal of Agricultural and Food Chemistry* 27(5): 977-978.
- El Sohaimy, S. 2013. The effect of cooking on the chemical composition of artichoke (*Cynara scolymus* L.). *African Journal of Food Science and Technology* 4(8): 182-187.
- El Sohaimy, S.A. 2014. Chemical composition, antioxidant and antimicrobial potential of artichoke. *The Open Nutraceuticals Journal* 7(1): 15-20.
- Elzeny, T.R.S. 2020. Chemical and biological studies on chicory (*Cichorium intybus* L.). Faculty of Agriculture, Kafrelsheikh University.
- FAO. 2007. *Statistical Database*. <http://faostat.fao.org>. Accessed on 11 November 2011.
- FAO/WHO/UNU. 1985. *Expert Consultation. Energy and Protein Requirements. Technical Report Series 724*. Food and Agriculture Organization/World Health Organization/United Nations University (FAO/WHO/UNU).
- Foury, C. 1989. Ressources génétiques et diversification de l'artichaut (*Cynara scolymus* L.). *Acta Horticulturae* 242: 155-166.
- Frutos, M., Guilabert-Antón, L., Tomás-Bellido, A. & Hernández-Herrero, J. 2008. Effect of artichoke (*Cynara scolymus* L.) fiber on textural and sensory qualities of wheat bread. *Food Science and Technology International* 14(5_suppl): 49-55.
- Gaafar, A., El-Din, M.S., Boudy, E. & El-Gazar, H. 2010. Extraction conditions of inulin from Jerusalem artichoke tubers and its effects on blood glucose and lipid profile in diabetic rats. *Journal of American Science* 6(5): 36-43.
- Georgieva, S.S., Boyadzhieva, S.S. & Angelov, G. 2016. Intensification of extraction of bioactive substances from artichoke wastes. *Bulgarian Chemical Communications* 48(Special Issue E): 451-455.
- Gomaa, M.A.H. 2010. Chemical and technological studies on some foods chemical, technological and biological studies on artichoke (*Cynara scolymus* L.). Kafrelsheikh University. Ph.D. Thesis (Unpublished).
- Hammouda, F., Seif El-Nasr, M., Ismail, S. & Shahat, A. 1993. Quantitative determination of the active constituents in Egyptian cultivated *Cynara scolymus*. *International Journal of Pharmacognosy* 31(4): 299-304.
- Hussein, L., El-Fouly, M., El-Baz, F. & Ghanem, S. 1999. Nutritional quality and the presence of anti-nutritional factors in leaf protein concentrates (LPC). *International Journal of Food Sciences and Nutrition* 50(5): 333-343.
- Ierna, A. & Mauromicale, G. 2010. *Cynara cardunculus* L. genotypes as a crop for energy purposes in a Mediterranean environment. *Biomass and Bioenergy* 34(5): 754-760.
- Juzyszyn, Z., Czerny, B., Pawlik, A. & Drożdżik, M. 2008. The effect of artichoke (*Cynara scolymus* L.) extract on ROS generation in HUVEC cells. *Phytotherapy Research* 22(9): 1159-1161.
- Kelly, G. 2008. Inulin-type prebiotics--a review: Part 1. *Alternative Medicine Review* 13(4): 315-329.
- Lattanzio, V., Kroon, P.A., Linsalata, V. & Cardinali, A. 2009. Globe artichoke: A functional food and source of nutraceutical ingredients. *Journal of Functional Foods* 1(2): 131-144.
- Leroy, G., Grongnet, J.F., Mabeau, S., Corre, D.L. & Baty-Julien, C. 2010. Changes in inulin and soluble sugar concentration in artichokes (*Cynara scolymus* L.) during storage. *Journal of the Science of Food and Agriculture* 90(7): 1203-1209.
- Lombardo, S., Pandino, G., Mauromicale, G., Knödler, M., Carle, R. & Schieber, A. 2010. Influence of genotype, harvest time and plant part on polyphenolic composition of globe artichoke [*Cynara cardunculus* L. var. *scolymus* (L.) Fiori]. *Food Chemistry* 119(3): 1175-1181.
- López-Molina, D., Navarro-Martínez, M.D., Rojas-Melgarejo, F., Hiner, A.N., Chazarra, S. & Rodríguez-López, J.N. 2005. Molecular properties and prebiotic effect of inulin obtained from artichoke (*Cynara scolymus* L.). *Phytochemistry* 66(12): 1476-1484.
- Marzi, V., Lattanzio, V. & Vanadia, S. 1975. Il carciofo pianta medicinale.
- Moharram, Y., Khalil, M. & Mostafa, M. 1981. Artichoke bracts (*Cynara scolymus*) as a source of protein. *Monoufeia Journal of Agricultural Research* 4: 273-283.
- Negro, D., Montesano, V., Grieco, S., Crupi, P., Sarli, G., De Lisi, A. & Sonnante, G. 2012. Polyphenol compounds in artichoke plant tissues and varieties. *Journal of Food Science* 77(2): C244-C252.
- O'Sullivan, L., Murphy, B., McLoughlin, P., Duggan, P., Lawlor, P.G., Hughes, H. & Gardiner, G.E. 2010. Prebiotics from marine macroalgae for human and animal health applications. *Marine Drugs* 8(7): 2038-2064.
- Pandino, G., Lombardo, S., Mauromicale, G. & Williamson, G. 2011a. Phenolic acids and flavonoids in leaf and floral stem of cultivated and wild *Cynara cardunculus* L. genotypes. *Food Chemistry* 126(2): 417-422.
- Pandino, G., Lombardo, S., Mauromicale, G. & Williamson, G. 2011b. Profile of polyphenols and phenolic acids in bracts and receptacles of globe artichoke (*Cynara cardunculus* var. *scolymus*) germplasm. *Journal of Food Composition and Analysis* 24(2): 148-153.
- Pandino, G., Lombardo, S., Moglia, A., Portis, E., Lanteri, S. & Mauromicale, G. 2015. Leaf polyphenol profile and SSR-based fingerprinting of new segregant *Cynara cardunculus* genotypes. *Frontiers in Plant Science* 5: 800.

- Pereira, A.P., Ferreira, I.C., Marcelino, F., Valentão, P., Andrade, P.B., Seabra, R., Estevinho, L., Bento, A. & Pereira, J.A. 2007. Phenolic compounds and antimicrobial activity of olive (*Olea europaea* L. Cv. Cobrançosa) leaves. *Molecules* 12(5): 1153-1162.
- Prosky, L. & Hoebregs, H. 1999. Methods to determine food inulin and oligofructose. *The Journal of Nutrition* 129(7): 1418S-1423S.
- Redondo-Cuenca, A., Herrera-Vázquez, S.E., Condezo-Hoyos, L., Gómez-Ordóñez, E. & Rupérez, P. 2021. Inulin extraction from common inulin-containing plant sources. *Industrial Crops and Products* 170: 113726.
- Rottenberg, A., Zohary, D. & Nevo, E. 1996. Isozyme relationships between cultivated artichoke and the wild relatives. *Genetic Resources and Crop Evolution* 43(1): 59-62.
- Ruiz-Cano, D., Pérez-Llamas, F., Frutos, M.J., Arnao, M.B., Espinosa, C., López-Jiménez, J.Á., Castillo, J. & Zamora, S. 2014. Chemical and functional properties of the different by-products of artichoke (*Cynara scolymus* L.) from industrial canning processing. *Food Chemistry* 160: 134-140.
- Shen, Q., Dai, Z. & Lu, Y. 2010. Rapid determination of caffeoylquinic acid derivatives in *Cynara scolymus* L. by ultra-fast liquid chromatography/tandem mass spectrometry based on a fused core C18 column. *Journal of Separation Science* 33(20): 3152-3158.
- Sonnante, G., De Paolis, A. & Pignone, D. 2003. Relationships among artichoke cultivars and some related wild taxa based on AFLP markers. *Plant Genetic Resources* 1(2-3): 125-133.
- Thaipong, K., Boonprakob, U., Crosby, K., Cisneros-Zevallos, L. & Byrne, D.H. 2006. Comparison of ABTS, DPPH, FRAP, and ORAC assays for estimating antioxidant activity from guava fruit extracts. *Journal of Food Composition and Analysis* 19(6-7): 669-675.
- UNESCO. 2010. *UNESCO Intangible Heritage Lists*. United Nations Educational Scientific and Cultural Organization (UNESCO).
- Vuong, Q.V., Hirun, S., Chuen, T.L., Goldsmith, C.D., Bowyer, M.C., Chalmers, A.C., Phillips, P.A. & Scarlett, C.J. 2014. Physicochemical composition, antioxidant and anti-proliferative capacity of a lilly pilly (*Syzygium paniculatum*) extract. *Journal of Herbal Medicine* 4(3): 134-140.
- Xiao, Z.J., Zhu, D.H., Wang, X.H. & Zhang, M.D. 2013. Study on extraction process of inulin from *Helianthus tuberosus*. *Modern Food Science and Technology* 29: 315-318.
- Zuorro, A., Maffei, G. & Lavecchia, R. 2014. Effect of solvent type and extraction conditions on the recovery of phenolic compounds from artichoke waste. *Chemical Engineering* 39: 463-468.

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