

## **Extracción de polifenoles de *Cynara scolymus* L., usando técnicas tradicionales y modernas. Una breve revisión**

Extraction of polyphenols from *Cynara scolymus* L. using traditional and modern techniques. A short review

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Recibido: 03/04/2018

Aceptado: 20/12/2018



## ABSTRACT

The nutritional value and health benefits of polyphenolic compounds presents in the artichoke, has emphasized the importance of extraction methods to obtain the highest recovery of this secondary metabolites. Polyphenols present in this herbaceous plant are represented by caffeoylquinic acid derivates and flavones apigenin, luteolin and their conjugates. This review highlights the theoretical aspects and recent developments of traditional techniques such as maceration and soxhlet extraction, and modern techniques like ultrasound-assisted, microwave-assisted and accelerated solvent extraction, which have been replacing traditional ones in the obtaining of these compounds in the last 10 years.

**Keywords:** artichoke, phenolic compounds, traditional techniques, modern techniques.

## INTRODUCTION

*Cynara scolymus* L., usually known as globe artichoke, is an ancient herbaceous perennial plant, originated from the Mediterranean areas of North Africa, and nowadays is widely grown around the world. The main global production of artichoke It's in Italy, followed by Spain, France and Greece (FAO, 2013). In South America the main producer country is Perú, followed by Chile and Argentina (FAO, 2014). In Colombia, the main producer departments are Cundinamarca and Boyacá (CCI, 2006).

The edible parts of the plant are the immature inflorescences, (capitula or heads), which are protected by fleshy leaves (bracts). The head of the artichoke contains levels of minerals, vitamins, carotenoids, and are source of dietary fiber, polyphenols and fatty acids (Lutz et al., 2011; Petropoulos et al., 2017).

The main compounds of artichoke are the polyphenols, such as luteolin and di-caffeoylquinic acids, in particular cynarin (1,3-Dicaffeoylquinic acid), chlorogenic acid (3-O-Caffeoylquinic acid) and apigenin-7-O-glucuronide, with antioxidant activity and a promising source of biopharmaceuticals (Alarcón-Flores et al., 2014; Lutz et al., 2011; Falé et al., 2013). These compounds have been associated with health-protective potential in hepatoprotective, anticarcinogenic, antibacterial, anti-HIV, hypocholesterolemic activities, and diuretic effects (de Falco et al., 2015).

Otálora M., Wilches A., & Cárdenas O., (2018). Extraction of polyphenols from *Cynara scolymus* L. using traditional and modern techniques. a short review. *Revista I3+*, 4(1), 29 - 38 p.p.

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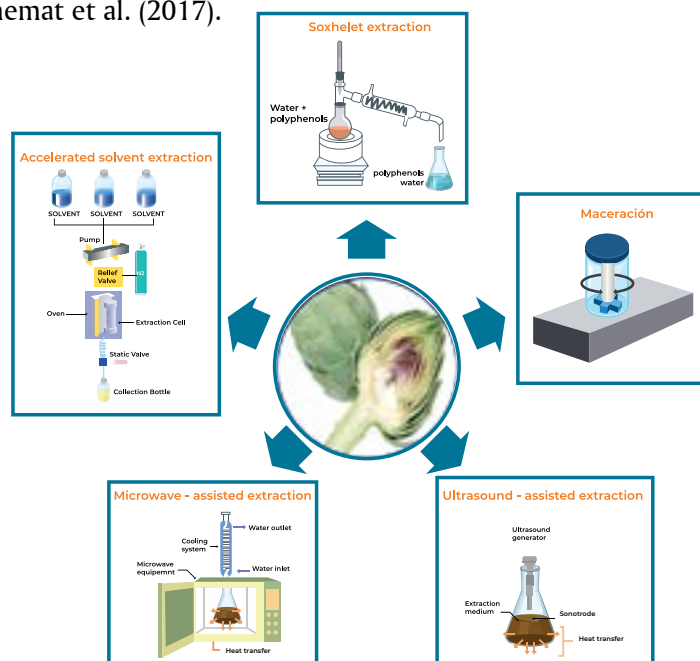
In polyphenols extraction, the quality of the extracts dependent on several factors, such as the extraction process, solvent used, quality and the origin of the raw material, storage conditions and its pretreatment (Domínguez-Rodríguez et al., 2017). In this context, the extraction techniques can be divided into traditional and modern ones (Fig 1). The traditional methods include maceration and Soxhlet extraction, which often involve long periods at high temperature and extended extraction times leading to the possibility of oxidation and hydrolysis of phenolic compounds (Panja, 2017). The modern methods include ultrasound-assisted extraction, microwave-assisted extraction and accelerated solvent extraction, which involve a reduced use of organic solvent, operational time, and better yield and quality of extract (Khoddami et al., 2013; Azmir et al., 2013); each technique has its own advantages and disadvantages.

The soxhlet extraction is described as the universal chemical extraction process. However, this methodology requires large extraction times and the use of large amount of organic solvents, resulting in low extraction efficiency (Azmir et al. 2013).

The maceration is the traditional solid-liquid extraction techniques mostly used for obtaining of polyphenols from plant material. The variables of the method are the type of solvent (methanol, ethanol, acetone and water), time and temperature extraction, sample/solvent ratio, stirring rate and final separation method (Vieitez et al., 2018).

**Keywords:** gallic acid, total polyphenols, Folin-Ciocalteu reagent.

Fig 1. Traditional and modern extraction techniques. Taken and modified from Passos et al. (2014); Kettle. (2013); Chemat et al. (2017).



In order to address these drawbacks, there is a need for alternative methods. In recent years, the ultrasound-assisted extraction (UAE), simplest and inexpensive technique, that improves the efficiency of the extraction of polyphenols, acceleration mass transfer, decrease in time and temperature of extraction, reduction in organic solvents use and environmental impact, increase in security and safety (Chemat y Khan., 2011; Chemat et al., 2017) and does not have restrictions on the polarity of the compound of interest nor to the moisture of the matrix; it has showed high reproducibility and potential for scale-up (Ghitescu et al., 2015) have been used. In other words, UAE avoiding thermal deterioration to the extracts and preserving the structural and molecular properties of bioactive compounds (Tian et al., 2013). However, previous study reported that using UAE at high temperature and at high ultrasonic power and intensity degrades the phenolic compounds (Guo et al., 2014).

The microwave-assisted extraction (MAE), is one of advanced methods currently used to obtaining polyphenols. Its use has increased significantly as a result of its inherent advantages: quick heating, shorter extraction time (minutes) by controlling power, reduction in solvent volume, higher extraction yield, high purity of final products, reproducibility and lower energy consumption due to the efficient heating process of microwave radiation (Panja, 2017; Leone et al., 2015; Espinosa et al., 2017). However, the use of higher temperatures decreases the extraction yield breaking down the molecular structure of bioactive compounds (Veggi et al., 2013).

Accelerated solvent extraction (ASE) is the oldest technique used for the polyphenols extraction. The solvent extraction technique has the ability to perform the extractions on a liquid or solid sample with the minimal effort. Solvent extraction using hexane, petroleum ether, chloroform and ethyl acetate obtaining different polarities is a widely used method. The residues of these xenobiotic compounds tend to accumulate into the extract and consequently distribute in body, affecting the health of the consumer, due to its highly toxicity (e.g. hexane exposure causes central nervous system and neurotoxic effects) (Li et al., 2016). Because of these health problems, the replacement of toxic solvents for phenolic compounds extraction of high quality is imperative. The extraction of polar polyphenols from vegetal materials, involves the use of organic solvents which must be compatible with the further uses of these bioactives in pharmaceutical, cosmetic and food industry. Most methods employ acetone, ethanol, and methanol (Soural et al., 2015). In a previous study related with the effect of organic solvents on the health, the ethanol/water mixture is preferred solvent, compared to hexane and acetone (Rufino et al., 2010) for its relatively low evaporation temperature, which facilitates the sustainable recovery of polyphenols. In relation with recovery of phenolic compounds from artichoke bracts and stems, methanol/water mixture is the most effective solvent.

## POLYPHENOLS EXTRACTION METHODS

### Traditional techniques

#### Soxhlet extraction

This is a conventional technique providing the highest recovery of polyphenolic compounds using solvents at boiling temperature and ambient pressures. Saleh et al. (2016) recorded the highest yield of chlorogenic acid from artichoke leaves using 80% methanol for 6 h. In another experiment, chlorogenic acid from artichoke leaves was obtained using petroleum ether for 8 h by soxhlet extraction (Özbilgin et al., 2015).

#### Maceration

Different authors hypothesized that maceration was superior to other methods, probably due to the non-requirement of a heating system during extraction, which avoids the thermal degradation of bioactive compounds, and minimizes the contact time between solvent and raw material. Using 95% ethanol as solvent, a sample/solvent ratio of 1:10 g/mL, and room temperature during seven days, was obtained the best recovery of phenolic compounds (94.7% yield) from powdered residue parts of globe artichoke (Dabbou et al. 2015). In a comparative study, the highest yield of the phenolic compounds-rich extract from powdered *Cynara scolymus* L., was obtained using 75% ethanol as solvent (solvent/sample, 5:1 at 25 °C for 48 h), compared with hexane, butanol, ethyl acetate, 75% ethanol/water, and water (Salem et al., 2017). In another report, the highest yield in total phenols content from dried leaves of *Cynara scolymus* L was obtained using 75 % methanol solvent for 24 h at 150 rpm and 20 °C, compared to the extraction by percolation using 75% ethanol for 2 h and 75 °C (Vamanu et al., 2011). In other studies, the highest yield in chlorogenic acid extraction from artichoke was obtained using methanol, as compared to ethanol and water (Claus et al., 2015).

### Modern techniques

#### Ultrasound-assisted extraction (UAE)

An effective technique of polyphenols extraction in *Cynara scolymus* L. is the ultrasound-assisted. The intensity, temperature, time and density (sample to solvent ratio) are factors required to obtain an efficient extraction of this secondary metabolites. Using UAE, a significant increase in yield of chlorogenic acid extraction (up to a 50%) from powdered artichoke was achieved using 80% methanol as solvent, temperature of  $25 \pm 5$  °C, ultrasonic power 300 W, sample/solvent ratio of 1:20 g/mL and du-

ration of 15 min (Saleh et al., 2016). In this study, the use of 50% ethanol as solvent, a sample/solvent ratio of 1:10 g/mL, ultrasonic power 240 W, an extraction temperature of  $25 \pm 1$  °C and extraction time of 60 min were used to obtain the highest yield of chlorogenic acid (95%) from the artichoke (Rabelo et al., 2016).

### **Microwave-assisted extraction (MAE)**

This is one of the advanced methods currently used for recovering polyphenols. MAE is a simple, rapid and economic method for polyphenols extraction, requiring a very short extraction time and low amount of solvents. Zhang et al. (2008) optimized the extraction of polyphenol from dried leaves of artichoke by MAE, using pH 7, solid to liquid ratio of 1:8 (w/v) and 700 W power for 90 s. The extraction yield of polyphenol increased 26.1% compared to the one that uses heat process during 1h. Caffeoylquinic acids, liable to oxidation by heated, were not affected by the microwave treatment. Also, Alupului et al. (2012) optimized the MAE for phenolic acids from *Cynara scolymus* L. leaves using 50% ethanol in solid to liquid ratio of 1:8 (w/v), and 400 W power (24 kJ equivalents) for 5 min. Alupului y Lavric (2012) compared the efficiency of MAE and ultrasound with classic thermal method to extract the polyphenolic acids from dried leaves of artichoke, authors concluded that effects of the main factors (temperature and duration of exposure) effect on the performance of extraction of secondary metabolites.

### **Accelerated solvent extraction (ASE)**

This technique involves extraction at constant high pressure, facilitating cellular permeability, intermolecular physical interactions and the extracting solvents penetration, improving the mass transfer of phenolic compounds. ASE at 25 °C, using methanol/water (80:20, v/v), 150 rpm and 1 h of extraction time, were optimum conditions to extract phenolic compounds of globe artichoke (Petropoulos et al., 2017). Using these ASE conditions, high recovery of phenolic compounds was obtained in a short extraction time (30 min) from lyophilized artichoke (Abu-Reidah et al., 2013). 25 °C, 1 h and 50:50 (v/v) methanol/water as extraction solvent, were optimal conditions for the recovery of polyphenol from fresh artichoke head (Garbetta et al., 2014). Zuorro et al. (2014) assayed several solvents including acetone, ethanol, hexane, ethyl lactate, water and a 50:50 ethanol/water mixture, for predict the extraction efficiency of polyphenols in ASE. The results of this study indicate that the maximum extraction efficiency of phenolic compounds from the outer bracts and the stems as artichoke waste was achieved using aqueous ethanol at 40–60 °C and 30 – 90 min (Zuorro et al., 2016).

## **CONCLUSIONS**

Due to high content of polyphenolic compounds in the artichoke, biomolecules with beneficial effects in prevention of degenerative diseases, is very important the impact of technique used to extraction and functional recovery of these secondary metabolites. Efficiencies of traditional and modern extraction

techniques depend, mostly, from temperature, pressure, solvent concentration, solid–solvent ratio, sample particle size, extraction time, microwave power (MAE), ultrasonic power, frequency (UAE) and solvent type (ASE). However, relation efficiency - cost, environmental safety and reproducibility of these techniques need to be evaluated in order to select the best methodological and technological option that allows efficient extraction of interest natural compounds.

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