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Composition, characteristics and antioxidant activities of fruit oils from *Idesia polycarpa* using homogenate-circulating ultrasound-assisted aqueous enzymatic extraction

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ABSTRACT

An efficient approach to isolate fruit oil from *Idesia polycarpa* using homogenate-circulating ultrasound-assisted aqueous enzymatic extraction (H-CUAEE) was developed. An enzyme cocktail comprising cellulase, hemicellulase, and pectinase proved to be the most effective in extracting oils. Nine potential variables that possibly affected the extraction yields – the liquid-solid ratio, incubation temperature, incubation time, amount of enzyme, homogenate time, ultrasound irradiation power, mark-space ratio of ultrasound irradiation, rate of agitation, and pH – were investigated using a Plackett–Burman design in order to screen out the significant variables. The liquid-solid ratio, incubation temperature, and incubation time were significant on a statistical basis and these three parameters were studied through a Box–Behnken design for the prediction of the best experimental conditions that maximize extraction yield of the fruit oil. A maximum fruit oil recovery of 79.36% with an error of 0.17% was achieved under the optimal extracting conditions: liquid-solid ratio of 7.13, the incubation time of 2.94 h, and the incubation temperature of 48.74 °C. The H-CUAEE process has expansive prospects for the preparation of oils from oil plants.

1. Introduction

Idesia polycarpa is a deciduous tree that belongs to the family Flacourtiaceae, and is indigenous to some Asian countries including China, Korea, Japan, and Russia's far-east (Kim et al., 2005; Wang et al., 2015; Gong et al., 2012). *I. polycarpa* is very tolerant and widely distributed in subtropical and warm temperate areas.

Idesia polycarpa grows rapidly, has a high fruit yield, and the fruits have a high oil content. The oil content of *I. polycarpa* fruit is more than 40% (Yang et al., 2009; Zhu et al., 2010), and this plant is an important potential resource for healthy food oil (Dai et al., 2016). The oil of *I. polycarpa* fruit contains unsaturated fatty acids, such as linoleic acid (Ye et al., 2014; Wang et al., 2011a,b). *I. polycarpa* fruit oil is nontoxic and has been used to produce cooking oil in Asia for many years (Yang et al., 2009). This oil is also suitable for the preparation of conjugated linoleic acids (Wang et al., 2013). Besides the cooking oil and the preparation of conjugated linoleic acids, *I. polycarpa* fruit oil is used more broadly in industry. For example, owing to its high oil content, it has been reported to be used for biodiesel production and the fuel properties of obtained oil are similar to light diesel fuel (Yang et al., 2009). Besides, *I. polycarpa* fruit oil is also a resource of biodegradable lubricant. Oil composition of pericarp and seed has been investigated (Li et al., 2016) and found that *I. polycarpa* fruit was unique with oil present in both seed and pericarp. At harvest time, pericarp was richer in oil than seed. These two parts showed difference in percentage of individual fatty acids. For instance, linoleic acid was the major components in seed and pericarp, which accounted for 83.92% and 62.08%. The relative abundance of palmitic acid and palmitoleic acid was higher in pericarp than in seed. From the industrial point of view, the use of whole fruit including pericarp and seed is more favorable.

Extract edible oils from plants generally use organic solvent (primarily hexane) extraction or squeeze (Li et al., 2015). These approaches make the cost of initial industrial processing very high. Furthermore, hexane can cause some pollution (Nader et al., 2016; Dickey et al., 2008). The defatted oilcake contains nutrients useful for humans but is normally discarded as waste after oil extraction. The oilcake of *I. polycarpa* fruit contains a variety of compounds such as (−)-idesolide and (−)-idescarpin. The structures of (−)-idesolide and (−)-idescarpin

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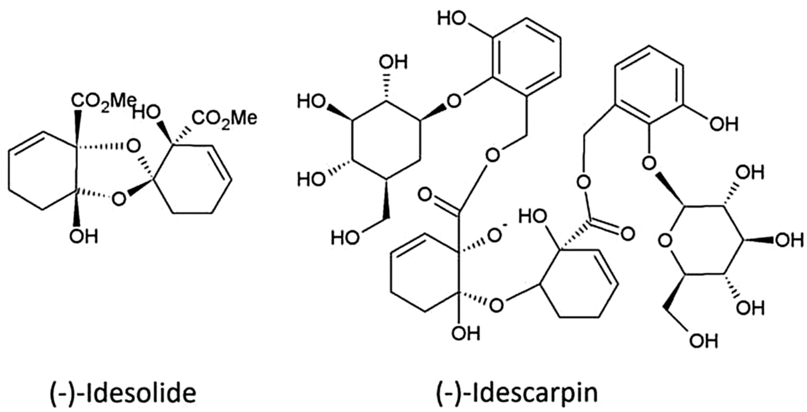

Fig. 1. The chemical structures of (−)-idesolide and (−)-idescarpin.

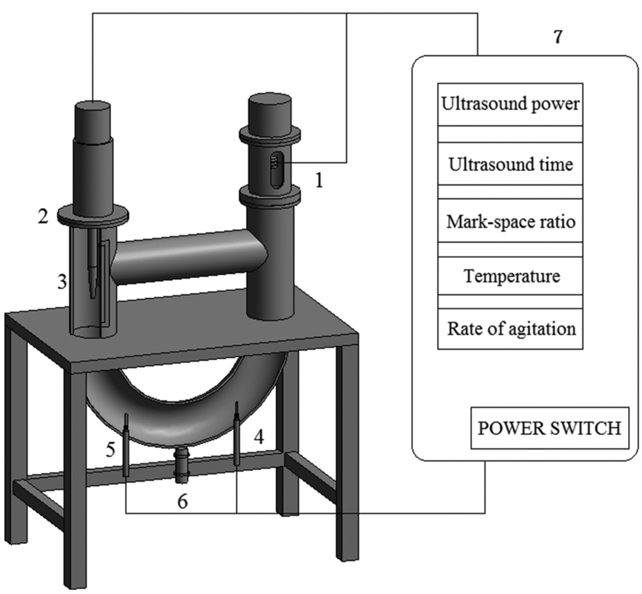

Fig. 2. Schematic of the ultrasonic circulating extraction equipment: (1) agitator, (2) heater, (3) temperature sensor, (4) inlet, (5) outlet, (6) ultrasonic generator, (7) control panel.

were shown in Fig. 1. (−)-Idesolide has been reported that its structure is a spiro-type compound embedded with a tetrahydrobenzodioxole ring unsymmetrical dimer of its monomeric form (Nagasawa et al., 2010), and the (−)-idescarpin was confirmed as a phenolic glycoside (Kim et al., 2014). (−)-Idesolide may be useful in combating obesity (Kim et al., 2005; Hwang et al., 2012) and may delay or prevent skeletal muscle atrophy (Jung et al., 2010). (−)-Idescarpin has potential to improve metabolic syndromes, especially obesity and diabetes mellitus (Lee et al., 2013), and may be used as a skin-whitening agent by inhibiting melanin biosynthesis (Baek et al., 2006). An extract of *I. polycarpa* oilcake showed anti-skin-aging activities and may be developed as cosmetics for the treatment of age-related conditions. However, residual organic solvent from traditional oil extraction precludes the use of *I. polycarpa* oilcake for these applications. An environmentally friendly and economical oil extraction is needed to replace traditional methods.

Aqueous enzymatic processing (AEP) is an environmentally cleaner alternative oil extraction technology that has been widely applied for a variety of oil-bearing seeds and fruits (Teixeira et al., 2013; Najafian et al., 2009). AEP is a distinctive principle compared with conventional solvent extraction methods that are based on the solubility of the oil in organic solvents (Zhang et al., 2012a; Anwar et al., 2013). The variation in the ultrastructure and chemical composition of the cell wall and membranes of oleosome — namely, variable proportions of cellulose, hemicelluloses, pectin, and other substances — is a substantial challenge for the application AEP (Mehanni et al., 2017; Ribeiro et al., 2016). Aqueous processing generally has lower oil extraction efficiency compared with conventional methods (Tzompa-Sosa et al., 2014; Baby and Ranganathan, 2016). The degradation of the cell wall by enzymes increases the permeability of oil through the fruit tissue. The yields of oil can be improved if microwave (Zhang et al., 2012a; Anwar et al., 2013), ultrasound (Shah et al., 2005; Sharma and Gupta, 2006), or ohmic heating (Pare et al., 2014) assisted treatment is applied during AEP. Combining these techniques could be more effective because of synergistic action demolishing the cell walls.

The ultrasound method disrupts the cell wall structure, giving rise to partial structural collapse and cellular expansion, and in this particular case the mass transfer barrier between the plant cell wall and the enzymes in solution can be lowered. This accelerates the rate of lipid release, improves oil yield, and shortens processing time. However, ultrasonic assisted extraction is often conducted in an ultrasound cleaning bath which is a static extraction and the chemical constituents from the samples are saturated by the surrounding extracting solution. The ultrasonic field in the typical ultrasonic-assisted extraction is focused in a finite space (Shah et al., 2005; Sharma and Gupta, 2006), so the samples being extracted are immobilized. As a result, the interaction between samples and ultrasonic waves is limited. Hence, a dynamic ultrasonic-assisted extraction needs to be investigated to avoid the saturation of chemical constituents in surrounding solution. A reflux-circulating controller (as can be seen in Fig. 2) was applied to facilitate relative motion between samples and the ultrasonic field in the ultrasonic circulating extraction (UCE). Under the influence of agitation, there is a strong mobility of biomaterial and the ultrasonic field fully contacts with biomaterial. The two arms of the device were connected together as one device with the horizontal pipe between them. The agitation part in the left arm contained a screw propeller as cycle stirring devices that diffused the propulsive flow and mix circulation of suspension of two arms. Hence, the working efficiency can be greatly enhanced. UCE was highly efficient for the extraction of polysaccharides from *Ganoderma lucidum* (Chen et al., 2014) and *Asparagus officinalis* (Zhao et al., 2011), purification of biflavanones and coumarin from *Stellera chamaejasme* (Zhang et al., 2015b), and in the extraction of alkaloids from *Uncaria tomentos*a (Zhang et al., 2015a). To our best knowledge, the extraction of oil by a circulating ultrasound-assisted aqueous enzymatic process has not yet been reported.

Mechanical pulverization is a traditional way in the preparation of