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Ultrasound extraction of phenolics and anthocyanins from jabuticaba peel

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ABSTRACT

Jabuticaba is a dark berry rich in phytochemicals such as polyphenols and anthocyanins. In the present study, ultrasound assisted extraction (UAE) of bioactive compounds from jabuticaba peels was evaluated. Ethanol was used as solvent because it is renewable (obtained from sugar cane) and GRAS. The effect of solvent concentration, pH and the extraction time on the target compounds (anthocyanins and phenolics) was evaluated by response surface methodology. The solvent to ratio was 1:20 and the extraction temperature was 30 °C. The anthocyanins and phenolics extracted were analyzed by LC–MS and HPLC and identified as cyanidin-3-*O*-glucoside (anthocyanin) and ellagic acid (phenol). The operating condition that has maximized the target compounds extraction required the sonicated of the peels for 10 min in a 46% (v/v) ethanol:water solution acidified at pH 1. The extraction yielded 4.8 mg/g dry peel of monomeric anthocyanin; 92.8 mg/g dry peel of gallic acid equivalent; 4.9 mg/g dry peel of cyanidin-3-*O*-glucoside and 7.8 mg/g dry peel of ellagic acid. The results showed that with an adequate operating condition it was possible to reach good yields using a simple extraction process.

1. Introduction

Jabuticaba tree (*Myrciaria* spp.) is an evergreen plant native to south central Brazil that has been cultivated for more than 400 years. The fruit of the jabuticaba tree measures 3–4 cm in diameter, contains up to four seeds and can be eaten fresh or as jams, juices and ice creams. The fruit is a dark berry with a sweet, white and soft juicy pulp with an acidic flavor (pH ~ 4.5). The pulp is rich in vitamin C and minerals, specifically potassium and calcium (Teixeira et al., 2011).

As other dark colored fruits, jabuticaba is a source of traditional nutrients, ingredients and phytochemicals (phenols and anthocyanins). These compounds have well-described biological properties including strong antioxidant and anti-inflammatory, anti-diabetic, and anti-obesity properties besides the potential to treat chronic obstructive pulmonary disease (COPD) (Wu et al., 2013b). The main anthocyanins found in jabuticaba are: cyanidin-3-glucoside; peonidin-3-glucoside and its aglycone (Einbond et al., 2004) and delphinidin-3-glucoside (Santos et al., 2010).

The anthocyanins contained in jabuticaba fruits are, in fact, found only in the fruit peel (the dark part), which is not directly edible but is used to produce jams and extracts designated to the beverage and ice cream industry. In addition, jabuticaba peel also contains phenolic compounds. Thus, the extraction of anthocyanins and other bioactive compounds, such as phenolics, from jabuticaba peels is of industrial interest. However, most of the published work on jabuticaba aimed to determine the fruit composition and its characterization. In these publications, extraction was carried out under an pure analytical point of view without worrying about the kind of solvent that was used, process optimization and scale up (Abe et al., 2012; Alezandro et al., 2013; Leite et al., 2011; Silva et al., 2014; Teixeira et al., 2011; Wu et al., 2013a). To be used as food, jabuticaba peel has to be processed and the extraction of the colored pigments, which are rich in anthocyanin, is needed. An efficient extraction process should maximize the recovery of target compounds with minimal degradation, resulting in an extract with high antioxidant activity using environmentally friendly technologies and low-cost raw materials (Santos et al., 2010).

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Table 1
Experimental planning and results obtained in the jabuticaba extracts.

Run	pH	ETOH (%)	Time (min)	Monomeric anthocyanin (mg/L)	Phenolics (μg/100 μL)	Cyanidin 3-*O*-glucoside (μg/mL)	Ellagic acid (μg/mL)
1	2.00	12.00	20.00	175.87 ± 21.80	269.12 ± 10.22	116.04 ± 5.80	187.10 ± 8.45
2	2.00	12.00	60.00	252.81 ± 50.20	411.63 ± 18.26	140.32 ± 4.02	239.05 ± 10.21
3	2.00	38.00	20.00	266.55 ± 20.06	447.81 ± 23.67	213.80 ± 11.89	328.13 ± 15.34
4	2.00	38.00	60.00	373.72 ± 76.51	579.49 ± 28.97	221.35 ± 8.07	331.05 ± 17.55
5	5.00	12.00	20.00	79.69 ± 36.18	124.14 ± 10.56	117.64 ± 4,88	220.37 ± 10.23
6	5.00	12.00	60.00	219.83 ± 38.27	250.59 ± 12.05	130.82 ± 6.54	279.38 ± 11.52
7	5.00	38.00	20.00	274.79 ± 38.53	389.22 ± 20.89	175.92 ± 7.98	311.88 ± 9.57
8	5.00	38.00	60.00	324.25 ± 50.17	464.13 ± 13.21	220.94 ± 9.05	342.11 ± 12.11
9	0.98	25.00	40.00	82.44 ± 166.87	424.59 ± 11.23	316.80 ± 12.84	319.50 ± 10.98
10	6.02	25.00	40.00	93.43 ± 89.56	132.96 ± 7.65	209.20 ± 8.46	273.94 ± 18.80
11	3.50	3.14	40.00	142.89 ± 16.98	250.59 ± 10.53	97.38 ± 3.56	183.12 ± 7.66
12	3.50	46.86	40.00	359.98 ± 83.59	579.99 ± 25.34	201.68 ± 8.06	327.87 ± 15.34
13	3.50	25.00	6.36	241.34 ± 31.59	341.44 ± 15.23	136.99 ± 5.98	222.70 ± 9.14
14	3.50	25.00	73.64	252.81 ± 9.15	425.90 ± 18.20	222.67 ± 8.93	313.42 ± 12.67
15	3.50	25.00	40.00	261.05 ± 28.50	394.00 ± 17.89	183.13 ± 7.12	264.73 ± 11.23
16	3.50	25.00	40.00	241.82 ± 10.52	399.00 ± 20.91	196.10 ± 8.81	263.73 ± 9.10
17	3.50	25.00	40.00	244.56 ± 16.29	391.19 ± 15.23	193.73 ± 7.79	281.72 ± 12.89

Ultrasound-assisted extraction (UAE) is an interesting process to obtain high valuable compounds. Ultrasound is usually applied in either solid/fluid media, being the fluid gas or liquid. Solid/liquid systems are the most common application. The enhancement of the mass transfer brought about by acoustic-induced cavitation in a liquid medium is one of the beneficial effects. When mechanical waves are transmitted through a fluid, the average distance within molecules is modified, oscillating around their equilibrium position. During the compression cycle, the intermolecular distance shortens and lengthens again in the rarefaction cycle. When the pressure decrease in the rarefaction cycle is enough to exceed the critical distance between molecules, cavitation can appear in the bulk liquid. Those incipient bubbles keep on growing until the system reaches its minimal pressure and the subsequent compression cycle starts (Esclapez et al., 2011). One of the main effects of these compression cycles is the disruption of the cell tissue with consequent increase of the solvent access to the target compounds improving the extraction rate (Rodrigues et al., 2008). As anthocyanins are vacuolar pigments, which accumulates in the plant cell central vacuole (Pourcel et al., 2010), cavitation and cell disruption caused by ultrasound waves may enhance the mass transfer from the solid matrix to the solvent improving the extraction of anthocyanins.

In the present study, ultrasound assisted extraction (UAE) of phenolics and anthocyanins from jabuticaba peels was optimized using response surface methodology. Ethanol was used as solvent because it is affordable, it is obtained from a renewable source (sugar cane) and it is classified as GRAS (generally recognized as safe) solvent, altogether fitting to a green chemistry approach. The effect of the pH of the solvent on the extraction was also taken into account. The results obtained herein were superior to the reported elsewhere, even applying ultrasound assisted extraction protocols.

2. Material and methods

2.1. Plant material

Fresh mature jabuticaba (*Myrciaria cauliflora*) fruits were obtained from the Municipal Market at São Paulo (São Paulo, SP, Brazil). The peels were removed by hand squeezing the fruit and were air dried at 60 °C in a conventional air forced oven for 24 h. After drying, the peels were triturated in a domestic blender and

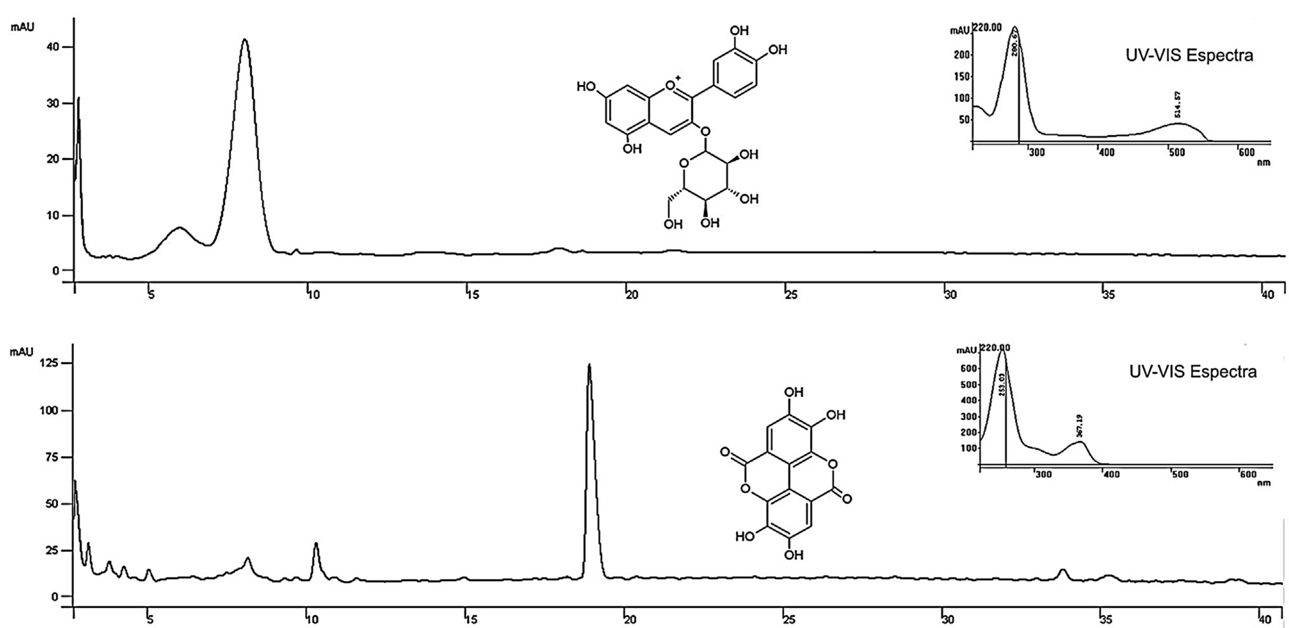

Fig. 1. HPLC chromatogram of jabuticaba extract at 530 nm (a) and 375 nm (b), corresponding to cyanidin 3-*O*-glucoside and ellagic acid, respectively.