Ultrasonics Sonochemistry 21 (2014) 1470–1476

Contents lists available at ScienceDirect

Ultrasonics Sonochemistry

journal homepage: www.elsevier.com/locate/ultson

Ultrasonic extraction of steroidal alkaloids from potato peel waste

Mohammad B. Hossain [a,*], Brijesh K. Tiwari [a], Nirupama Gangopadhyay [a], Colm P. O'Donnell [b], Nigel P. Brunton [c,1], Dilip K. Rai [a]

[a] Teagasc, Ashtown Food Research Centre, Ashtown, Dublin 15, Ireland
[b] School of Biosystems Engineering, University College Dublin, Dublin 4, Ireland
[c] School of Agriculture and Food Science, University College Dublin, Dublin 4, Ireland

ARTICLE INFO

Article history:
Received 2 September 2013
Received in revised form 8 January 2014
Accepted 25 January 2014
Available online 15 February 2014

Keywords:
Ultrasound
Extraction
Steroidal alkaloids
Response surface methodology

ABSTRACT

Potato processors produce large volumes of waste in the form of potato peel which is either discarded or sold at a low price. Potato peel waste is a potential source of steroidal alkaloids which are biologically active secondary metabolites which could serve as precursors to agents with apoptotic, chemopreventive and anti-inflammatory properties. The present study investigated the relative efficacy of ultrasound assisted extraction (UAE) and solid liquid extraction (SLE) both using methanol, to extract steroidal alkaloids from potato peel waste and identified optimal conditions for UAE of α-solanine, α-chaconine, solanidine and demissidine. Using response surface methodology optimal UAE conditions were identified as an amplitude of 61 μm and an extraction time of 17 min which resulted the recovery of 1102 μg steroidal alkaloids/g dried potato peel (DPP). In contrast, SLE yielded 710.51 glycoalkaloid μg/g DPP. Recoveries of individual glycoalkoids using UAE yielded 273, 542.7, 231 and 55.3 μg/g DPP for α-solanine, α-chaconine, solanidine and demissidine respectively. Whereas for SLE yields were 180.3, 337.6, 160.2 and 32.4 μg/g DPP for α-solanine, α-chaconine, solanidine and demissidine respectively. The predicted values from the developed second order quadratic polynomial equation were in close agreement with the experimental values with low average mean deviation ($E < 5\%$) values. Predicted models were highly significant ($p < 0.05$) for all parameters studied. This study indicates that UAE has strong potential as an extraction method for steroidal alkaloids from potato peel waste.

1. Introduction

Food processing industries particularly potato-crisps manufacturing industries generate a huge volume of potato peel as by-product. Industrial processing generates between 70 and 140 thousand tons of peels worldwide annually [1]. This by-product is usually discarded causing environmental concern or used as low value animal feed. This massive amount of waste offers significant economic potential for creative uses other than animal feeds or fertilizers. Moreover, the discarded potato peel represents a severe disposal problem to the potato industry as the wet peels are prone to microbial spoilage [2]. Utilization of potato peel for the extraction of steroidal alkaloids will reduce if not eliminate the disposal problem while paving the way for a new phyto-pharmaceutical industry.

Potato constitutes a very important part in human diet in many countries of the world. The tuber is a good source of carbohydrates, high quality proteins, antioxidative polyphenols [3], vitamins and minerals [2]. However, it also contains a group of toxic compounds known as steroidal alkaloids which are largely concentrated in the peels [4]. Steroidal alkaloids are secondary metabolites present mainly in the plants of Solanaceae family that have been associated with defence against bacterial [5], fungal [6] and insect attacks [7]. Symptoms associated with glycoalkaloid toxicity are colic pain in the abdomen and stomach, gastroenteritis, diarrhoea, vomiting, fever, rapid pulse, low blood pressure, and neurological disorders [8,9]. These adverse effects on human health could be attributed to the anticholinesterase activity and membrane disruption properties of steroidal alkaloids affecting mainly digestive and central nervous systems [10–12]. *In vitro* experiments showed that α-solanine and particularly α-chaconine are potent cytotoxins (IC50 = 4.1 μM), acting rapidly to induce cell lysis [13]. However in addition to their toxic effects, studies in the last decade have demonstrated that these compounds may possess beneficial properties such as anticancer and anti-inflammatory effects, depending

* Corresponding author.
E-mail addresses: mohammad.hossain@teagasc.ie (M.B. Hossain), nigel.brunton@ucd.ie (N.P. Brunton).
[1] Tel.: +353 7162807.

http://dx.doi.org/10.1016/j.ultsonch.2014.01.023
1350-4177/

on dose and conditions of use [14,15]. Therefore, maintaining or even enhancing these bioactivities but lowering the cytotoxicity to normal dividing cells by chemical modifications of the steroidal alkaloids could be a useful strategy to use the steroidal alkaloids for pharmaceutical applications. For this purpose efficient extraction techniques to recover sufficient quantities of starting material for chemical modification are required particularly for the alkamine solanidine (aglycone) as evidence indicates that many of the toxic effects of these compounds are related to the presence of glycosidic residues.

A number of techniques are available for the extraction of secondary metabolites from plants, including ultrasound-assisted extraction, supercritical fluid extraction, microwave-assisted extraction, and solvent extraction [16–18]. Among these, ultrasound-assisted extraction (UAE) offers an inexpensive, environmently friendly and time efficient alternative to conventional extraction techniques [19–21]. Similar to other secondary metabolites gylcoalkaloids are contained within cells and ultrasound induced cavitation can disrupt plant cell wall causing release of intracellular components thus enhancing extraction [22]. Ultrasound can also enhance extraction by reducing particle size and increasing the net hydrophobic character of the extraction medium (when the target molecule is apolar) [23].

Ultrasound also offers a mechanical effect allowing greater penetration of solvent into the sample matrix, increasing the contact surface area between the solid and liquid phase, and as a result, the solute quickly diffuses from the solid phase to the solvent [24,25]. A number of parameters can be manipulated to optimise extraction of target compounds using ultrasound including extraction time and temperature, ultrasound amplitude and extracting solvent. Investigations involving the manipulation of all these parameters to optimise extraction can be time consuming and expensive. However, response surface methodology (RSM) is a statistical technique which allows the user to identify optimal conditions for a selected response while minimising the number of experiments required. Therefore in the present study a response surface methodology (RSM) approach was undertaken to optimise the ultrasound assisted extraction parameters such as amplitude level and extraction time by employing a central composite design to maximise extraction of steroidal alkaloids from potato peel slurry provided a major potato processor in Ireland. The optimal conditions were benchmarked against a standard solid liquid extraction approach. The study for the first time has reported the effect of ultrasound on the extraction of steroidal alkaloids from potato peel.

2. Material and methods

2.1. Samples and reagents

The steroidal alkaloids α-solanine, α-chaconine, solanidine and demissidine were purchased from Extrasynthese (Genay Cedex, France). High performance liquid chromatography (HPLC) grade solvents such as methanol, water, formic acid and acetonitrile were purchased from Sigma–Aldrich (Wicklow, Ireland).

2.2. Drying of the peel slurry

Potato peel slurry was provided by Largo Foods Limited (Meath, Ireland). Prior to freezing the slurry was pressed by hand to remove excessive water. Then the peels were spread on aluminium trays and cooled to −40 °C. Freeze-drying was carried out on the frozen peel in A 6/14 freeze-drier (Frozen in Time Limited York, UK) at a temperature of −54 °C and a pressure of 0.064 mbar for 72 h. Freeze dried samples were immediately powdered, vacuum packed and kept in −20 °C for extraction within two weeks. The film (75 micron thickness) of the vacuum pack pouches (Allfo vakuumverpackungen Hans Bresele KG, Germany) were composed of a mixture of polyamide (PA) and polyethylene (PE) with an oxygen and carbon dioxide permeability rate of 60 cm^3/m^2/24 h/atom and 180 cm^3/m^2/24 h/atom respectively (23 °C, 75% RH). The water vapour permeability of the film was 2.7 g/m^2/24 h at 23 °C and 85% RH.

2.3. Sonication treatment

Dried and powdered potato peel (7 g) were placed in a 100 mL jacketed vessel through which water was circulated at 15 ± 0.5, 25 ± 0.5 and 35 ± 0.5 °C with a flow rate of 0.5 L/min. Extraction solvent (70 mL methanol) was added to the sample and the ultrasound probe was submerged to a depth of 25 mm in the solvent. A 1500 W ultrasonic processor (VC 1500, Sonics and Materials Inc., Newtown, USA) with a 19 mm diameter probe was used for sonication (Fig. 1). Samples were processed at a constant frequency of 20 kHz. The energy input was controlled by setting the amplitude of the sonicator probe. Extrinsic parameters of amplitude (24.40, 30.5, 42.70, 54.9 and 61.0 μm) and processing time (3, 5, 10, 15 and 17 min) were varied with pulse durations of 5 s on and 5 s off. Ultrasound intensities of the mentioned amplitudes were calculated as 9.24, 10.16, 13.28, 17.17 and 22.79 W/cm^2 respectively. The ultrasonic power (P) was determined as described by Tiwari et al. [26] using Eq. (1) where dT/dt is the change in temperature over time (°C s^{-1}), C_p is the specifc heat of water (4.18 kJ kg^{-1} °C^{-1}), and m is the mass (kg).

$$P = mC_p(dT/dt)_{t=0} \quad (1)$$

Ultrasonic intensity (UI) dissipated from an ultrasonic probe tip with diameter D is given by Eq. (2)

$$UI = \frac{4P}{\pi D^2} \quad (2)$$

All treatments were carried out in two batches which included three replications in each sample.

2.4. Conventional solid/liquid extraction

In order to benchmark optimal UAE condition against a conventional extraction technique, solid/liquid extractions were carried out according to the method of Shan et al. [27] with slight modifications. Briefly, dried and ground samples (7 g) were mixed with 70 mL methanol and shaken for 17 min at room temperature (~23 °C). The sample suspension was then immediately filtered through Buchner funnel (pore size: 1.0 μm). The extracts were kept at −20 °C until subsequent analysis. Extraction was performed on two batches which included three replications in each sample.

2.5. Identification and quantification of steroidal alkaloids in potato peel by ultra-performance liquid chromatography coupled with tandem mass spectrometry

Steroidal alkaloids were analysed using Waters Acquity (Waters Corporation, MA, USA) ultra-performance liquid chromatography coupled with tandem mass spectrometry (UPLC-MS/MS). The compounds were separated on a Waters Acquity BEH C18 column (50 × 2.1 mm, particle size 1.7 μm) using 0.5% formic acid in water (Solvent A) and a mixture of acetonitrile, 2-propanol and formic acid in the ratio of 94.5:5:0.5 (solvent B). The following stepwise gradient program was carried out: 0–1 min 10% B, 2–6 min 20.5% B, 7–9 min 30% B, 9.5 min 90% B and 10–11 min 10% B at a flow rate of 0.5 mL/min. The injection volume for all the samples was 5 μL. All the standards in the concentration range from 0.1 to 1 μg/mL