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Gas assisted mechanical expression (GAME) as a promising technology for oil and phenolic compound recovery from tiger nuts

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ABSTRACT

The aim of this work was to investigate the potential of gas assisted mechanical expression (GAME) process as an innovative technology for oil extraction and polyphenol recovery from tiger nuts. GAME process was first studied by varying the supercritical carbon dioxide (SC-CO_2) and the mechanical expression (ME) pressures (10–30 MPa), then was compared to separate processes applied alone, using either SC-CO_2 or ME. The results showed that the better conditions for GAME were found using 20 MPa for SC-CO_2 and 30 MPa for ME. Under these conditions, 50% of oil was released from tiger nuts after 10 min extraction, compared to only 10% and 20% when using SC-CO_2 and ME separately at 20 and 30 MPa pressures, respectively. In addition to the faster extraction of oil using GAME process, the obtained results showed that this oil contains higher amount of polyphenols than that obtained using either SC-CO_2 or ME. These molecules were extracted and identified using ultra performance liquid chromatography–high resolution mass spectrometry (UPLC–HRMS). Polyphenol profiles showed that GAME process led to the maximum polyphenol's recovery with 57 compounds, followed by SC-CO_2 with 48 compounds, and ME process with only 27 compounds, concurring with the trends observed for total phenolic compounds. Although non-significant differences were observed in oil recovery after applying GAME or SC-CO_2 alone, scanning electron microscopy (SEM) images revealed that GAME had a higher impact on cell structure, which can facilitate the release of important valuable compounds. All of these results pave the way towards the industrialization of GAME process as an innovative and alternative technology for enhanced oil extraction, thus replacing the conventional processes.
Industrial relevance: The increasing need in application of more efficient extraction processes in full correspondence with green extraction concept has led to deeper interest in new non-conventional methods that can reduce the extraction time, process temperature and consumption of toxic solvents (i.e. hexane). For instance, gas assisted mechanical expression (GAME) technology may constitute a useful tool for food industry as it allows to improve oil recovery and the amount of bioactive compounds (polyphenols) from tiger nuts, compared to supercritical fluid extraction and conventional methodologies (mechanical expression and hexane extraction) thus avoiding the use of toxic solvents.

1. Introduction

Tiger nuts (*Cyperus esculentus* L.), also called “chufa” in Spanish, are tubers widely used for animal (feed) and human consumption. In Spain, these tubers are mainly used to make a milk-like beverage called “horchata de chufa”. This beverage is a non-alcoholic refreshing drink of dairy appearance and is usually consumed in summertime (Mosquera, Sims, Bates, & O'Keefe, 1996; Sanchez-Zapata, Fernandez-Lopez, & Angel Perez-Alvarez, 2012). Several beneficial properties have been associated with tiger nuts intake including the prevention of heart diseases and thrombosis as well as the activation of blood circulation (Chukwuma, Obioma, & Christopher, 2010) and reducing the risk of colon cancer appearing (Adejuyitan, Otunola, Akande, Bolarinwa, & Oladokun, 2009). These beneficial properties have been mainly attributed to their high content in minerals (mainly phosphorus and potassium), and vitamins E and C (Belewu & Belewu, 2007). Moreover, they are rich in energy content (starch, fat, sugar, and proteins). Therefore tiger nuts can constitute an important source of high-added value compounds which can be used as food additives and/or for the development of nutraceuticals.

Abbreviations: GAME, gas assisted mechanical expression; SC-CO_2, supercritical carbon dioxide; ME, mechanical expression; TPC, total phenolic compound; UPLC–HRMS, Ultra performance liquid chromatography–high resolution mass spectrometry.

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For instance, tiger nut oil has several beneficial properties and has been used from ancient years for different applications including alimentation, nutritional purposes as well as in the pharmaceutical industry, mainly for its high content in oleic acid, polyunsaturated fatty acids (linoleic and linolenic acids), vitamin E, especially α-tocopherol, and also for its high oxidative stability compared to other oils, mainly attributed to its content of polyunsaturated fatty acids and γ-tocopherol (Ezebor, Igwe, Owolabi, & Okoh, 2006; Okladnikov, Vorkel', Trubachev, Vlasova, & Kalacheva, 1977). Moreover, tiger nut oil has also been used in textile industry in order to waterproof textile fibers (Bamishaiye & Bamishaiye, 2011).

Traditionally, the application of mechanical expression for batch (small amounts of material operated upon for a time period) or continuous operations has been used for the recovery of oil from different plant food materials including palm. Cold pressing combined with *n*-hexane is the most used methodology to recover oil from tiger nuts, since cold pressing alone does not remove all the oil from the plant matrix (Ali Rehab & El Anany, 2012; Yeboah, Mitei, Ngila, Wessjohann, & Schmidt, 2012). However, the use of *n*-hexane has been questioned due to environmental and safety issues (Lasekan & Abdulkarim, 2012).

The need for obtaining greener, sustainable and viable processes has led both food industries and food scientists to develop alternative processes in full correspondence with green extraction concept (Koubaa, Roselló-Soto, Šic Žlabur, Režek Jambrak, Brnčić, Grimi, et al., 2015). This concept assumes to make use of renewable plant resources and alternative green solvents, reduction of energy consumption and unit operations, production of extracts with high quality and purity (non-denatured and biodegradable) and generation of co-products instead of wastes (Chemat, Vian, & Cravotto, 2012).

In this line, one previous study has evaluated the potential of supercritical carbon dioxide (SC-CO_2) extraction to recover oil from tiger nuts, obtaining good results (Lasekan & Abdulkarim, 2012). In fact, supercritical fluid extraction is a promising technology which has been used during last decades in different food and agricultural applications as well as fuel industries, followed by analysis/chromatography, pharmaceuticals, environmental contaminants, metal-ion extractions and pesticides. In the last two decades, it has acquired a special relevancy for the extraction of nutritionally valuable compounds obtained from different sources (Barba, Grimi, & Vorobiev, 2014). This technique highly reduces (often to zero) the use of toxic organic solvents. Moreover, the elimination of the main supercritical fluid (CO_2) is achieved without residues, yielding a solvent-free extract, and the operation can be controlled to recycle it. In addition, this technology allows direct coupling with analytical chromatographic techniques (Ibañez, Herrero, Mendiola, & Castro-Puyana, 2012). However, depending on the compound, a long extraction time is required compared to other non-conventional techniques. Possible use of modifiers (also called co-solvents), to improve the extraction and the selectivity, can be used (Ezeh, Gordon, & Niranjan, 2014; Roselló-Soto et al., 2015). According to the specific requirements, the design of a supercritical fluid extraction system can be relatively simple or highly complex, increasing thus the cost.

For instance, in order to avoid long extraction times, applying supercritical fluid extraction along with mechanical pressing is an interesting alternative to improve the recovery of seed oils and antioxidant valuable compounds. This process called gas assisted mechanical expression (GAME) has been previously applied to extract oil from plant matrices (Müller & Eggers, 2014; Venter, Willems, Kuipers, & de Haan, 2006; Venter, 2006; Willems & de Haan, 2011; Willems, Kuipers, & de Haan, 2008; Willems, 2007).

To the best of our knowledge, there are no reports evaluating the combined effect of supercritical carbon dioxide and mechanical expression to recover oil and phenolic compounds from tiger nuts. Therefore, the main objectives of the present work were: 1) to evaluate the promising technology; GAME to recover oil and bioactive compounds from tiger nuts; 2) to find the better GAME processing conditions (influence of mechanical and CO_2 pressures), for oil and phenolic compound recovery from tiger nuts; 3) to compare GAME process with either ME or SC-CO_2 extraction, tested separately; 4) to identify and quantify the polyphenols of the different extracted oils from tiger nuts; and finally 5) to analyze the microstructure of resulting tiger nut matrices after ME, SC-CO_2 and GAME processes.

2. Materials and methods

2.1. Chemicals and reagents

Methanol, acetonitrile, Folin–Ciocalteu reagent and sodium bicarbonate were obtained from Fisher Scientific (Illkirch, France). DPPH (2,2-diphenyl-1-picryl-hydrazyl), Tween-20, and formic acid were purchased from Sigma-Aldrich (Saint-Quentin Fallavier, France). All chemicals were used as received.

2.2. Samples and pre-treatment

Tiger nuts (*C. esculentus*) were purchased from a local market (Valencia, Spain). According to the retailer, the nuts were grown in Valencia (Spain). The tiger nuts were grinded to get an average size of 479 μm, determined using a "Malvern Mastersizer 2000" granulometer; allowing the measurements of the particle size under vacuum according to their volume, then used to extract oil using GAME, ME, and SC-CO_2.

2.3. Oil extraction from tiger nuts

Oil from previously grinded tiger nuts was extracted using supercritical fluid (SC-CO_2), gas assisted mechanical expression (GAME), and mechanical expression sole (ME). Polyphenols were then extracted from seed oils, quantified, and identified using UPLC–HRMS. Fig. 1 shows the adopted methodology during this work.

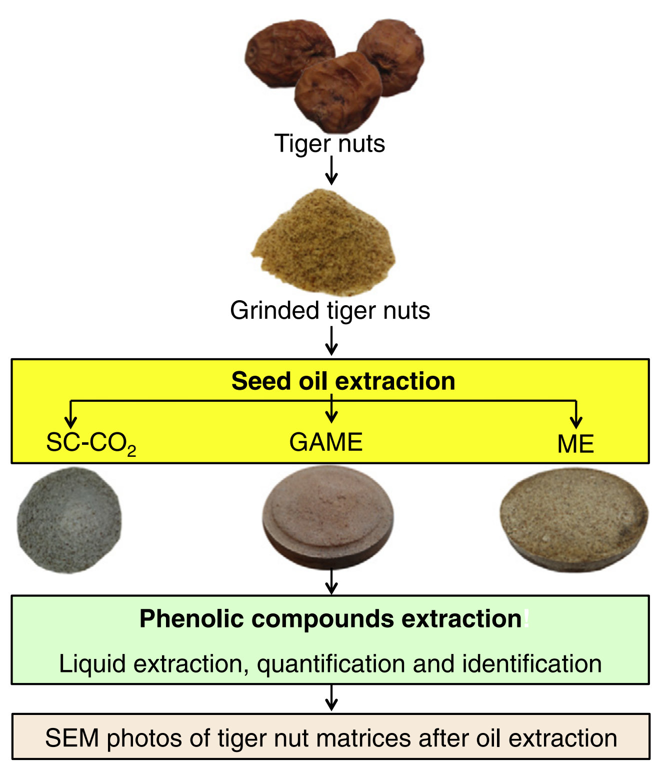

Fig. 1. Methodology followed during this work. SC-CO_2; supercritical CO_2, GAME; gas assisted mechanical expression, ME; mechanical expression, SEM; scanning electron microscopy.