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Biorefinery concept for discarded potatoes: Recovery of starch and bioactive compounds

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ABSTRACT

The integral valorisation of discarding potatoes from three local varieties using processes of low environmental impact to recover the starch contained in the flesh as well as the bioactive compounds present in the skin or in the processing wastewaters was studied. The remaining flesh after starch extraction was also recovered to further processing. The extraction of starch and active extracts using environmentally friendly technologies, the physicochemical and phytochemical characterisation of the extracts, the formulation and mechanical characterisation of the corresponding functional hydrogels, have allowed proposing potential food and non-food applications. Results indicated that subcritical water extraction (220 °C) was an efficient technology to recover antioxidants from the potato skin. Processing wastewaters exhibited high protein content. The extracted starch featured comparable physicochemical properties to that available commercially and the corresponding hydrogels exhibited enhanced mechanical properties with absence of syneresis. It should be remarked that Agria and Neiker varieties provided the highest total starch and bioactive content in terms of phenolics, TEAC value and DPPH inhibition percentage, respectively.

1. Introduction

Potato is the world's fourth largest food crop with an estimated production of 388 million tonnes in 2017, led by Asia with over 50.4% of the world total and followed by Europe (31.5%), America (11.4%), Africa (6.4%) and Oceania (0.6%) (FAOSTAT, 2017). The major species grown worldwide is *Solanum tuberosum*, and modern varieties of this species are the most widely consumed (Bzducha-Wróbel et al., 2015). During the harvest and storage of this edible perishable tuber a high degree of discarding (up to 30%) is generated, since they do not meet the size, quality, colour or suffer from plagues, and currently has a low added value being used primarily for animal feed (Priedniece et al., 2017). Another massive wastes from the potato processing industry are the peels and outer flesh layers as well as flesh or wastewaters remaining from the starch extraction, where important amounts of gelling or bioactive fractions are contained and could be easily recovered (Gientka et al., 2019). Losses produced from potatoes peeling can represent up to 40% depending on the procedure used (Pathak et al., 2018); whereas those from the flesh after the starch processing in industry can account for 75% of initial product mass (Ahmed et al., 2018) and the corresponding wastewaters represented about 10.8 million m^3 in 2017 (Dupuis and Liu, 2019). Consequently, the valorisation of these wastes containing high valuable compounds (starch, protein, antioxidants or fibre, among others) is a real need that could be of relevance to the potato industry helping to improve the economical balance of the industrial process and to commercialize new products (Priedniece et al., 2017; Nazarian-Firouzabadi and Visser, 2017).

The functional properties of the potato recovered components highly depend on the extraction procedure. In the case of mild extraction procedures, the obtained potato proteins are highly soluble and exhibit remarkable foaming and emulsifying characteristics. These methods also allow to recovery fibers that can lead to development of a three-dimensional network in the end-product which impressively improve the texture and stability of the matrix (Priedniece et al., 2017). An alternative with great potential is the recovery of biopolymer compounds with gelling properties as the starch required to alleviate the growing market of biopolymer-based hydrogels with food and non-food applications (Torres et al., 2018). As well as, bioactive extracts with antioxidant, anti-inflammatory, antimicrobial or antitumor properties (Friedman et al., 2017), present in potatoes, using extraction techniques

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based on so-called clean technologies as subcritical water extraction, following the current trends in green chemistry, using water as the only extraction agent (González et al., 2017). Briefly, subcritical water extraction is a pressurized hot water extraction, which is also called high-temperature water extraction or autohydrolysis. As it is well-known, this technique consist in applying high temperature and pressure in the subcritical water region, between the boiling temperature at normal conditions (100 °C) and the critical point (374 °C). In this reaction medium, the biopolymers can react with high conversion rates and in very short residence times (Burnner, 2009). Some recent studies can be found in the literature on the valorisation of the potato disposals (see as e.g. Sepelev and Galoburda, 2015; Nazarian-Firouzabadi and Visser, 2017; Pathak et al., 2018), but a comprehensive research on the integral valorisation as proposed here has not been reported yet.

Hydrogels from starch are the most available and economic materials. The incorporation of functional components in formulations with high starch content and bioactive compounds is a growing trend for food and non-food applications (Witczak et al., 2015). An intense search for new renewable sources to produce natural biopolymers for edible coatings or biodegradable films is observed as they offer lower environmental costs (Larotonda et al., 2016). In addition, other sectors, such as biomedical, pharmaceutical and cosmetic, increasingly use compounds with high bioactivity extracted from natural sources (Liu et al., 2015). The future of biopolymer materials is to show that vegetable materials from natural, under-utilized and renewable sources can overcome their traditional counterparts based on oil. These biopolymers recently gained attention for emerging manufacturing technologies such as 3D printing, since it is much easier to work with them during processing, they are compatible for use in food, pharmacy or cosmetics, non-toxic and smell-free (Jonathan and Karim, 2016). The knowledge of the thermomechanical behaviour during the formulation of starchy-based gels provides relevant information to select the optimum processing conditions to achieve well-defined and tailor-made final products, especially for hydrogels from extracts from disposal sources (Torres et al., 2018). This developing market has enormous potential to improve the welfare of the population.

In this context, the main aim of this work is the integral valorisation of discarded potatoes from different local varieties using sustainable approaches based on the biorefinery concept for potential food and non-food applications. For this purpose, the extraction of biopolymer and bioactive compounds using environmentally friendly technologies is proposed. The physicochemical and phytochemical characterisation of the extracts and the mechanical properties of the corresponding functional hydrogels are studied.

2. Materials and methods

2.1. Raw materials

Low-sized or irregular shape discarded potatoes from three varieties (*i.e.* Agria, Kennebec and Neiker) harvested in Galicia (Spain), were kindly provided by the *Instituto Ourensán de Desenvolvemento Económico* (INORDE). Above varieties were selected based on their potential high content of starch and bioactive compounds, when compared with other local varieties. Commercial potato starch (CAS: 9005-84-9, Scharlau, Spain) was also used as raw material with comparative purposes.

2.2. Extraction of high valuable compounds

2.2.1. Starch

The starch extraction conditions (*i.e.* water ratios, samples sizes, mixing times, sieve size, among others) were optimised for the potato samples using only water as extraction reagent to increase the starch yield and purity. The employed procedure was based on the conventional extraction process used for the industrial potato starch extraction (Wu, 2016). Concisely, the proposed method to extract starch from discarding potatoes consisted of several sequential steps: (i) manual peeling of potatoes; (ii) cutting and milling the flesh; (iii) adding water (solid:liquid ratio, 1: 2), (iv) mixing the milled flesh with water (60 min, 200 rpm) and (v) filtering with a sieve (80 μm), separating the flesh from the liquid filtrate. After this, (vi) washing the flesh to drag the remaining starch; (vii) leave overnight the filtrate and washing water in the fridge 24 h to promote the starch decantation and (viii) separate the starch from filtering and washing waters. Then, (ix) drying the starch in an air drying convective oven at 40 °C for 48 h and (x) homogenizing the moisture content of the starch placing the samples in petri dishes in a desiccator containing $MgCl_2$ for about one week until constant weight. Finally, (xi) storing of the starch in closed plastic boats at room temperature and storing of processing wastewaters and remaining flesh after starch extraction in jars in the freezer (−20 °C) until further analysis.

2.2.2. Bioactive fractions

In order to recover the bioactive fractions, the coarse milled potato skin (0.25–2 mm) was subjected to subcritical water extraction. Initially, a temperature sweep between 120 and 220 °C was performed with the most bioactive potato variety to select the autohydrolysis conditions. Then, the milled skin was contacted with water (solid:liquid ratio, 1:15), the aqueous suspension was stirred and heated up to selected temperature (220 °C) in a pressurized reactor (Parr Instruments series 4848, Illinois, USA). The liquid fraction was separated by filtration and was stored in a fridge (4 °C) until further phytochemical analysis. In all cases, analyses were performed before a week.

2.3. Physicochemical characterisation

2.3.1. Composition

Moisture content was determined according to the standard gravimetric method, 925.10 (AOAC, 2000). Ash amount was obtained after calcination in a muffle for 6 h at 575 °C. Total starch and amylose content were calculated using two enzymatic kits (Megazyme, Co., Wicklow, Ireland) following standard procedures (AACC, 2010). Protein content was quantified by Kjeldahl method using a Flash EA 1112 Elemental Analyser (Thermo Fisher Scientific, MA, USA) with the nitrogen conversion factor of 6.25 (Torres et al., 2019). Na^+ and K^+ content was determined by atomic emission spectrophotometry; whereas Ca^{2+}, Mg^{2+} and Fe^{2+} content was obtained by atomic absorption spectrophotometry. In both cases, minerals experiments were conducted on a 220 Fast Sequential Spectrophotometer (Varian, NY, USA). Previously, a pre-treatment of the samples consisting of an acid digestion with HNO_3 in a microwave (Marsxpress-CEM Co., NY, USA) operating at 1.6 KW for 15 min followed by 200 °C for 10 min was necessary in this work, following the conditions detailed by Flórez-Fernández et al. (2019). Carbohydrates determination was carried out by High Performance Liquid Chromatography, using a 1200 series Hewlett-Packard chromatograph with a refractive index (RI) detector, as reported elsewhere (e.g. Flórez-Fernández et al., 2019). In order to determine the fraction of carbohydrates, aliquots of liquors obtained at different temperatures during the autohydrolysis treatment were analysed by HPLC-RI (Aminex HPX-87H, BioRad, Hercules, USA). Note here that a previous filtration through 0.45 μm cellulose acetate membranes was made. Saccharide fractions were determined by means of a 300 × 7.8 mm Aminex HPX-87H column (BioRad, Hercules, USA). The operating conditions were 5 N H_2SO_4, 0.6 mL/min and 50 °C. The samples were also subjected to posthydrolysis with 4% sulphuric acid in autoclave at 121 °C for 20 min and then were cooled down at room temperature. Posthydrolysis was made to hydrolyse the oligomers and to evaluate the content of oligomers in liquors by difference between the content of monomers in the posthydrolysis liquor and in the autohydrolysis liquor.

2.3.2. Size measurements

The average size/weight of the used potatoes (25 samples per variety) was determined using a calibre and an analytical balance,