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Economic and Technical Feasibility of Betanin and Pectin Extraction from *Opuntia ficus-indica* Peel via Microwave-Assisted Hydrodiffusion

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ABSTRACT: Investigating the feasibility of betanin and pectin extraction from *Opuntia ficus-indica* peel via microwave-assisted hydrodiffusion and gravity, this study identifies selected important economic and technical aspects associated with this innovative production route starting from prickly pear fruit discards. Which benefits would be derived from this process? Would production be limited to *Opuntia*-growing countries or, likewise to what happens with dried lemon peel chiefly imported from Argentina? Would production take place abroad also? Can distributed manufacturing based on clean extraction technology compete with centralized production using conventional chemical processes?

1. INTRODUCTION

We have lately discovered that microwave-assisted hydrodiffusion and gravity (MHG) applied to the peels of red and green fresh *Opuntia ficus-indica* (OFI) fruits harvested in Sicily affords under solvent-free and mild extraction conditions (1 h extraction at 70 °C) a red natural extract mostly containing betalains, pectin, and polyphenols.[1]

After storage for 4 months at room temperature, the extract fully retains its original red color, pointing to the lack of betanin molecular degradation. The lyophilized pectic polymer obtained after ready separation via dialysis has a high degree of crystallinity and 53% degree of esterification (DE).[1]

Closing the materials cycle and offering a low energy route to valorisation of a biological resource so far mostly discarded as an agro-food industry waste, the method establishes a circular bioeconomy method to obtain two valued bioproducts from biowaste available in significant amounts.

Widely approved as a food additive (label E162 in the EU), betanin is a valued violet-red betacyanin stable at pH between 3.8 and 6.8 and particularly well suited for use as a natural colorant in beverage, confectionary, bakery, dairy, and frozen products.[2]

Widely used as a food colorant in desserts, bacon burgers, ice-cream, jams, jellies, liquorice, meat soup, sauces, and sweets, the dye is currently obtained from red beetroot (*Beta vulgaris* L var. *ruba*) after 2 years of cultivation.[2] Shifting its production to *Opuntia and* giving its fruits several times a year would be highly desirable.

Similarly, pectin is the most sought-after natural hydrocolloid in the food industry.[3] Sourcing pectin from the peel of OFI fruits would provide an alternative to current production routes based on dried lemon and orange peel or apple pomace as raw materials. Well characterized in 2004,[4] the structural features of *Opuntia*'s pectins provide them with rheological and functional behavior ideally suited as thickening and gelling hydrocolloids in food, personal care products, and pharmaceutical applications.[5,6]

Pioneered by Chemat and Visinoni,[7] the MHG extraction technology, combining microwave heating and earth gravity at atmospheric pressure, has rapidly emerged as one of the most

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attractive techniques to extract and to separate value-added bioproducts from biological matrices.

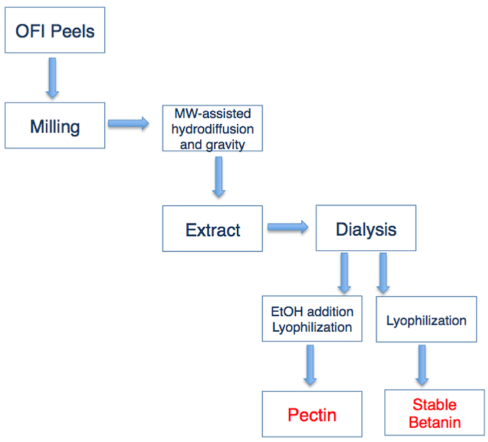
Its advantages include high reproducibility, less energy consumption, shorter procedures, higher purity of the final product, elimination of the organic solvent, and consequent elimination of waste effluents.[8]

Investigating the feasibility of betanin and pectin extraction from OFI peel via MHG, this study identifies selected important economic and technical aspects associated with this innovative production route, starting from prickly pear epidermis.

2. RESULTS AND DISCUSSION

Scheme 1 shows the solvent-free conditions process[1] lately developed on the laboratory scale for the extraction of pectin

Scheme 1. Extraction of Pectin and Betanin from OFI Peels via MHG



and betanin involves milling the fresh peel with the aid of a knife and irradiation with microwaves for 1 h so as to heat the mixture at 70 °C.

The aqueous extract undergoes dialysis to separate the pectic polymer from betanin and biophenols. Pectin precipitated with ethanol at −18 °C is isolated in the form of highly pure crystals via lyophilization. The same treatment is used to isolate stable betanin powder enriched with biophenols (two phenolic acids, and 10 flavonoids including isorhamnetin, quercetin, and kaempferol derivatives) present in the peel of OFI red and white fruits.[9]

The decentralized extraction of pectin and betanin from OFI peel on a semi-industrial scale might be carried out, for example, using a MAC-75 (Milestone, Italy), namely, a digitally controlled and integrated extractor for the industrial production of extracts on small scale (75 L rotating drum).[10]

Energy consumption for one processing cycle would amount to 8.7 kW h (Table 1).

The extraction of pectin and betanin from the OFI fruit peel via MHG transforms a cost item into a revenue item. Currently, for instance, a fruit processing company based in Sicily (Italy) using the OFI fruits to extract juice either pays waste processing companies to dispose waste OFI peel or gives away for free the fresh peels which are used for cattle feeding.

Table 1. Energy Consumption per Extraction Cycle

step	power (W)	time (min)	energy (kW h)
heating	9000	20	3.0
maintaining	4000	40	2.7
chilling	3000	60	3.0

Besides the initial capital investment required to purchase the extractors (one MAC-75 sells at 160−170 k€), the two major costs faced by a company willing to extract betanin and pectin from OFI fruit peels would be: (i) labor and (ii) electricity needed to power the extractor and the lyophilizer.

Driven by the megatrend global demand for "naturals", pectin has become the preferred natural hydrocolloid at food and beverage companies. The current 60 000 tonnes, \$1.2 billion market,[11] is forecasted to grow at >7% annual rate until 2025.[12]

The price of extracted pectin (E440) has been steadily increasing in the course of the last decade[11] to reach current values at around \$8.00/lb for high methoxyl (HM) and around \$9.00/lb for low methoxyl (LM) pectin.[13] We remind that commercially pectins are classified according to their methoxyl content: pectin commercial grades with DE lower and higher than 50% are classified as LM and HM pectins, respectively. When designing pectin gels for specific applications, it is important to consider simultaneously the DE, the monosaccharide content (homogalacturonan, HG), and the spatial disposition of the cross-linking blocks (RG).

Similarly, the price of extracted betanin (beetroot red, E162) is high and its demand is generally increasing.[2]

For example, by early 2019, powdered beetroot red extract ready for use as a natural food colorant was sold online by an Italy's food colorant manufacturer at €130/kg.[14] European specifications for beetroot red E162 define that not less than 0.4% of the commercial material must be betanin pigment, with the remaining 99.6% being composed of sugars, salts, and proteins naturally occurring in red beets and a small amount of other betalains.[15]

Globally, already in 2009, 10 per cent of the global demand of food colorants (45 000−50 000 tonnes) was met by beetroot red.[16] The beetroot powder market (90 000 tonnes, \$15 billion) in 2016 is forecasted to expand at a compound annual growth rate of 5% until 2027.[17]

A number of unique technical advantages can be expected from extracting pectin and betanin from the peels of OFI fruits via MHG technology.

First, the quality of pectin, obtained under acid-free and solvent-free conditions, is particularly high. The process results in pectin having larger percentages of "hairy" rhamnogalacturonan I (RG-I, the side chains of mainly α-L-arabinofuranose and α-D-galactopyranose) regions that promote the formation of more entangled structures, which plays a gel-stabilizing role.[18]

On the other hand, the relative content in galacturonic acid [HG, regions: partially 6-methylated and 2- and/or 3-acetylated poly-α(1−4)-D-galacturonic acid residues] "smooth" regions estimated for pectin from OFI peel is low in comparison to those of citrus or grapefruit pectin and for most commercial ones.[19]

The relative proportions of interconnected HG and RG-I regions determine the flexibility and rheological properties of the polymer in solution: the HG regions enhance molecular interactions, whereas the branched RG regions promote the formation of entangled structures.[20]

Extracting pectin via the conventional hydrolytic process in hot acidic water, Rodríguez-Hernández and co-workers have already shown that pectin extracted from the peel of *Opuntia*

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albicarpa Scheinvar "Reyna" fruits has enhanced ability to form soft and elastic gels when compared to lemon-derived pectin.[21]

Second, the betanin extract of endless stability was obtained, thanks to the concomitant presence of high amounts of antioxidant polyphenols present in the OFI fruit's peel is of unique technical relevance. Stable at acidic pH, betanin extracts, in general, suffer from poor stability against oxidative degradation.[22] This single aspect so far has limited the use of betalain natural pigments as functional ingredients (and health promoters) in nutraceutical, pharmaceutical, and cosmetic products, requiring the development of costly optimal processing conditions to maximize the stability of betalains and their extraction yields.[22]

Third, the absence of acid and added solvent including water to directly obtain the aqueous mixture of valued bioproducts in the fruit peel cell water itself affords an entirely green process of higher yield (no production losses: all extractable pectin and betanin are extracted), producing no effluents and thus requiring no treatment.[8] Along with the cost of the dried citrus peels, the cost of the treatment of diluted acidic waters obtained in conventional pectin plants is the highest among those faced by pectin manufacturing companies.[11]

Fourth, no drying of the peels is required. The very same polyphenols abundantly present in the peel have a potent antibacterial, antioxidant, and fungicide activity.[9] This prevents the rapid microbial degradation of the peel as it happens with the peel of squeezed citrus (lemon and orange) fruits requiring rapid and costly drying prior to pectin extraction.

Fifth, widely and increasingly harvested OFI affords its fruits several times per year, offering an excellent potential alternative to both citrus and red beetroot as a natural source of pectin and betanin.

Sixth, the extraction process is intrinsically safe: the lack of flame and noise reflects into safer, healthier, and more comfortable operation conditions.[8] Besides lowering insurance costs, the latter are key advantages for manufacturing bioproducts which are sold to food, nutraceutical, cosmetic, and pharmaceutical companies.

Seventh, with complete photovoltaic (PV) systems now routinely installed in Italy at approximately \$1/W for systems of nominal power >50 kW,[23] the cost of electricity of the extraction company would be reduced by self-producing the electricity through a PV array whose cost, because of the solar energy revolution that occurred in the last decade,[24] has now reached unprecedented low levels.

3. CONCLUSIONS

The concomitant extraction of pectin and betanin in the stabilized form from OFI peels using MHG under solvent-free conditions is technically feasible.

Microwave-based hydrodiffusion and gravity extraction processes in reactors optimized to ensure good homogeneity of heating and good product transfer with capacities up to many hundred kilograms per hour indeed are already commercialized.[8,10,25]

Can the concomitant MHG extraction of pectin and betanin from *Opuntia*'s agro-food industry fruit waste be also made economically viable in comparison to pectin multistep chemical extraction in large plants, and beetroot red hydro-alcoholic in smaller, dedicated plants?

A thorough techno-economic analysis will quantify the economic benefits, the return on investment, and the payback times to answer this question in detail. Of relevance to the present bioeconomy study addressing the use of a prominent green chemistry technology to produce two bioproducts in high and increasing demand are the key differences with the conventional approach to scaling up chemical processes.

Rather than aiming to scale up the process in batch, first on a pilot scale and then, following optimization, in large industrial plants, the MHG technology applied to the present circular economy process directly targets production using a series of batch reactors in parallel or one continuous flow reactor. The setup time is brief, essentially consisting of the commissioning time.

Rather than aiming at economies of scale of manufacturing in large production units typical of chemical productions, the process aims to produce pectin and betanin at the rate of customer demand, with quick, clean, and flexible production capable to meet the variable customer demand, with no stock throughout the process and with the ideal batch size being one.[26] Similar to Ohno's production system, which was focused on the flow of the work, this production mode tolerates higher unit costs, as it would not be dependent on low costs per unit.[26]

Remarkably, with the emergence of new manufacturing technology, a similar trend from centralized to decentralized and distributed manufacturing, allowing products to be manufactured and distributed close to customers, is being observed for many productions.[27] The same technology shift is clearly emerging also for natural products extraction based on new technology.[28]

Finally, the energy (electricity) required to carry out the process under mild conditions will be mostly self-produced from sunlight, thanks to today's low cost and digitally managed PV technology equipped with energy storage in Li-ion batteries and solar hydrogen, so as to maximize energy self-consumption and enable natural product production in developing regions and nations where solar irradiation is plentiful.[29]

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Notes

The authors declare the following competing financial interest(s): Milestone manufactures extractors for microwave-assisted extraction of natural products.

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