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Research paper

Fennel oil and by-products seed characterization and their potential applications

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ABSTRACT

The implementation of renewable resources in the industrial production processes appears to be the most effective way to achieve sustainable development. However, in order to tackle the key issues of shifting to renewable resources, a full exploitation of biomass resources and efficient utilization of complex organic macromolecules and also other chemical constituents such as antioxidants in bio-refinery system will be crucial. In this regard, fennel (*Foeniculum vulgare*) seeds could be a promising bio-resource with significant interest as a rich source of both vegetable oil (VO) and essential oil (EO), in addition to rare phytochemicals. Thus, in the present paper, a trans-disciplinary assessment of a new bio-refinery process from fennel seeds was established: the development of an integrated valorization of fennel seeds, allowing the extraction of VO and EO and their exploitation in cosmetic applications as well as the valorization of residual by-products as a source of biologically active compounds, these processes constituted the basis of this bio-refinery concept. Laboratory obtained results and pilot-scale levels with fennel seeds reported extraction of high yield of both VO and EO (19.8% and 1.8%, respectively) with significant amounts of valuable components, petroselinic acid and trans-anethole (74.8% and 70.7%, respectively). Further, the valorization of these oils as functional ingredients in moisturizing cream formulas showed a positive impact on the overall emulsions structure and quality. Next to this, fennel oilseeds by-products exhibited a remarkable antioxidant potential with high phenols and flavonoids contents and exhibited good antimicrobial properties depending on the extract type. These promising findings are of great economic interest as they can lead to a wild range of novel, bio-based industrial applications from fennel seeds.

1. Introduction

Bio-refinery concept can be defined as the biomass conversion processes. It includes several conversion methods (biochemical, microbial, chemical and thermochemical) seeking for optimal use of biomass. Thus, added value chemicals, co-products and residues are obtained through strategic involvement of the chemical industry in the supply of final products to different domains such as petrochemical, pharmaceutical, building, cosmetic and others (Venskutonis and Jonušaitė, 2016). Before this step, biomass and their byproducts could be evaluated for these purposes.

Fennel (*Foeniculum vulgare* Mill.) is a commercially important Apiaceae species from the Mediterranean area and central of Europe and is among the most widespread medicinal plant worldwide, being extensively grown in arid and semi-arid regions as one of the oldest spice plants (Barros et al., 2010). It is recommended traditionally for gastrointestinal and neurological disorder, kidney stones, vomiting and diarrhea, it has also antispasmodic, antiseptic, carminative and anti-ulcer properties (Ghanem et al., 2012). Recently much attention has been focused on fennel due to the nutritional and health protective value of their seeds that are rich in vegetable and volatile oils (Matthäus and Musazcan Özcan, 2015). Fennel seeds are considered also as source of many health beneficial compounds including minerals, vitamins, and others which explain their applications for pharmaceutical, cosmetic, perfumery and food industries (Nassar et al., 2010).

In 2014, the European Commission authorised the use of coriander oilseed as a novel food ingredient under ‘Regulation (EC) No 258/97 of the European Parliament and of the Council’, this is due to its richness in the uncommon monounsaturated fatty acid, the petroselinic acid (C18:1n12). This fatty acid is a positional isomer of oleic acid used as

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valuable raw material to the synthesis of a series of bio-based compounds that could be of particular interest to chemical industries (Nguyen et al., 2015). In this context, fennel oilseed could present an attractive competitor as the next novel food ingredient owing to the presence of petroselinic acid, which constitutes over 80% of all fatty acids.

Plant seeds can be processed into high-quality vegetable or essential oils, the remaining portion may find various profitable applications due to their phytochemicals content and antioxidant activity. These by-products can thus be seen as economically promising raw materials for future applications in industrial products for pharmaceutics or cosmetics (Fekria et al., 2012).

Various vegetable oils can be applied for the moisturizing, protection and healing of problematic skins. Besides its nutritional benefits, fennel oil has several positive effects on the skin according to its richness in mono-unsaturated fatty acids (MUFAs) and especially petroselinic acid which can resolve some skin problems, such as dryness (Oyedeji and Okeke, 2010). With the recent trend towards environmentally friendly substances and more biodegradable options and in a bio-refining approach in term of valorization of fennel EO and VO in a non-food applications, the use of fennel oilseed in moisturizing cream formulations seem to be a promising option. Actually, oilseeds are easily biodegradable and skin lipid compatible and thus, their using in cream formulas could reduce the use of synthetic oil such as paraffin oil (Srivastava and Sahai, 2013).

The potential nutritional and functional properties of agrowastes are studied previously such as polyphenols in hemp, flax and canola seed cakes (Teh et al., 2014), proteins, fibers and other nutrients in *Arachis hypogaea* seed cakes (Fekria et al., 2012) and antioxidant activity of extracts of *Guizotia abyssinica* (Wettasinghe and Shahidi, 1999) and *Rosa damascena trigintipetala* Dieck (Abdel-Hameed et al., 2012) byproducts. Regarding *F. vulgare* seeds, several quantitative estimation of protein and fiber contents and total phenols and flavonoids contents, as well as their antibacterial and radical scavenging properties have been done (Christova-bagdassarian et al., 2014; Shah et al., 2015). Nevertheless, all of these studies were limited on a single aspect of investigation including oil composition or biological activity, but none have addressed all of them together. Moreover, remaining residues after oil extraction have not gotten much interest. Therefore, a new bio-refining approach was established in this study in order to fully exploit fennel seeds for a wide range of industrial applications (Fig. 1). The aim of this paper is to evaluate the chemical composition of fennel oilseeds and their potential addition to moisturizing cream formulas, we aim also to analyze the potential usefulness of byproducts fennel seeds by assessing the total phenolic and flavonoid contents, as well as the antioxidant and antibacterial properties of their extracts.

2. Material and methods

2.1. Oil extraction and analysis

2.1.1. Essential oil extraction and analysis

Two hundred grams of sweet fennel (*Foeniculum vulgare* Mill. var. dulce) grinded seeds were immersed in 2L of distilled water contained in a 6L round-bottomed flask. Distillation was carried out using a Clevenger apparatus for 180 min after boiling. The extracted essential oils were recovered and stored in a refrigerator at 4 °C.

Essential oil samples were analyzed on a Hewlett Packard 5890 series Π Gas Chromatograph coupled with a 5970 mass spectrometer and equipped with fused-silica capillary columns HP-5 MS (0.25 µm × 0.25 mm × 30 m) and Carbowax (0.25 µm × 0.25 mm × 30 m). GC–MS parameters with a HP-5 MS column: carrier gas: helium (flow rate: 0.6 ml/min), oven temperature programming: rising from 60 °C to 220 °C at 3 °C/min and then held at 220 °C for 12 min. Injector temperature: 250 °C, ion source temperature: 280 °C. Electron ionization: 70 eV; mass spectra range: 30–300 amu and 2.77 scan/s; split ratio: 1/100; injection volume: 1 µl, pentane solution. GC–MS parameters with a carbowax column: carrier gas: helium (flow rate: 0.6 ml/min), oven temperature programming: at 70 °C for 2 min, rising to 220 °C at 5 °C/min and then held at 220 °C for 8 min. Injector temperature: 250 °C, ion source temperature: 280 °C. Electron ionization: 70 eV; mass spectra range: 30–300 amu and 2.77 scan/s; split ratio: 1/100; injection volume: 1 µl, pentane solution. Identification of individual components in the essential oils or volatile extracts was based on the comparison of their retention indices calculated with reference to a series of *n*-alkanes, with those found in the literature (Adams, 2007). Further identification was made by comparing their mass spectra with those in the mass spectra library of data process software (NBS75 K database, Wiley 7th NIST 98 EPA/NIH Mass Spectral Library, Mass finder 3/Hochmuth and FFNSC2/Mondello, 2nd Edition, 2011 Nov.), and also those found in published data. The relative percentage of each component in the essential oil was given according to the normalization results of peaks in GC chromatograms.

2.1.2. Vegetable oil extraction and fatty acid analysis

Vegetable oil was extracted using Soxhlet apparatus. A sample of 25 g of grounded seeds from fennel was extracted using cyclohexane as solvent, for 5 h. After extraction, the solvent was removed by rotary evaporator; the oil was kept at 4 °C in a dark bottle.

After Soxhlet extraction, VO was methylated and converted into Fatty Acids Methyl Esters (FAMEs). One milliliter of MTBE (methyl-*tert*-butyl-ether) was added to 20 mg of VO, then; 100 µl of this solution was transferred to an insert and 50 µl of trimethylsulfonium hydroxide (TMSH) was added, the solution was gently stirred. FAMEs were analyzed using GC/FID. The analysis was performed using a capillary

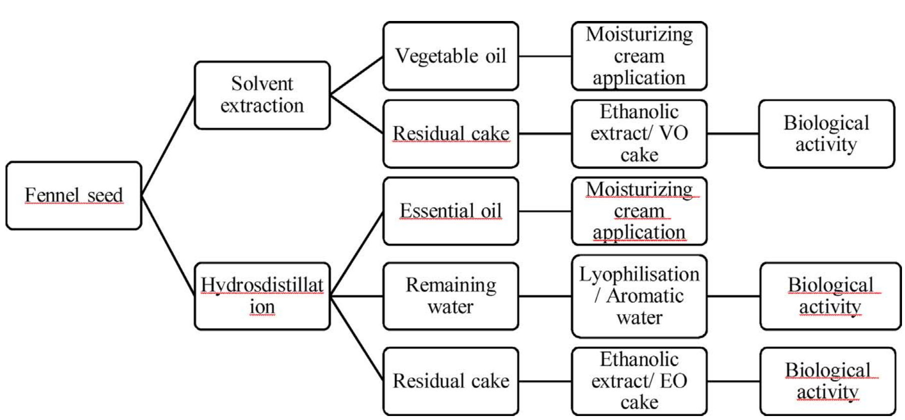

Fig. 1. Outline of bio-refining process of F. vulgare seeds applied in the present study.

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