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Review

Separation of functional macromolecules and micromolecules: From ultrafiltration to the border of nanofiltration

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The recovery of functional compounds from underutilized bioresources is today accomplished in five distinct stages, whereas ultrafiltration has been utilized for the separation and the clarification of macromolecules from smaller molecules or the opposite. The current article highlights the outcomes of an integral study including research articles, which cover the separation mechanisms dominating during UF (from 100 to 1 kDa) of different feed solutions and extracts, under similar processing conditions. Target macromolecules concern dietary fibers (i.e. pectin, β-glucan), proteins and polymeric anthocyanins, while assayed micromolecules were sugars, cations, monomeric anthocyanins and different phenolic classes such as o-diphenols, hyxrodycinnamic acid derivatives and flavonols.

Introduction

As it is well known, functional compounds and the so-called "nutraceuticals" are today used as additives in foodstuff due to their ability to provide advanced technological properties and health claims, respectively, to the final product (Galanakis, 2013; Galanakis, Markouli, & Gekas, 2013b; Ramaa, Shirode, Mundada, & Kadam, 2006). Indeed, epidemiological studies have shown that health benefits (i.e. reduced risk of coronary heart disease and stroke, diabetes, obesity and cancer) may be attributed to the consumption of both macro- and micro-nutrients (Elleuch *et al*., 2011; Schieber, Stintzing, & Carle, 2001). For instance, macromolecules like soluble dietary fiber is known for its ability to lower blood lipid level and at the same time possesses advanced gelling properties that can replace fat in foods, stabilize emulsions and improve the shelf-life of the product (Elleuch *et al*., 2011; Galanakis, 2011; Galanakis, Tornberg, & Gekas, 2010c; Rodríguez, Jiménez, Fernández-Bolaños, Guillén, & Heredia, 2006). Proteins have also been used as fat replacement in meat and milk products, flavor enhancers in confectionary, food and beverage stabilizers (Kristinsson & Rasco, 2000; Pogaku, Seng, Boonbeng, & Kallu, 2007; Prakash, 1996). On the other hand, natural antioxidants have been connected to both nutritional (reduction of oxidative stress, prevention of cancer, arteriosclerosis, ageing processes) and functional (preservative of vegetable oils and emulsions) properties. Antioxidants include typically smaller compounds like polyphenols, carotenoids, tocopherols and ascorbic acid (Boskou, 2006; Kiokias & Oreopoulou, 2006; Moure *et al*., 2001).

Undervalued bioresources and natural products are considered as target substrates for the recovery of the above compounds. The later process follows typically the principles of analytical chemistry, whereas it has recently been proposed to accomplish with the so-called "5-Stages Universal Recovery Processing" including: (i) macroscopic pre-treatment, (ii) separation of macro- and micromolecules, (iii) extraction, (iv) isolation-purification and finally (v) product formation or encapsulation (Galanakis, 2012, 2014). Ultrafiltration (UF), nanofiltration (NF) and other membrane technologies are among the key physicochemical and non-destructive techniques applied in the second, third and fourth step of the above downstream processing. Particularly, researchers target to concentrate macromolecules and release smaller molecules in the permeate stream, respectively.

This procedure seems to be simple in theory since membranes are able to separate compounds via sieving mechanism, based on their molecular weight (MW). However,

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this is not the case in practice, as the asymmetric manufacture of membrane pores does not always reflect a narrow range of molecular weight cut-off (MWCO). The effect of the latest parameter attenuates when the solubility of the solutes and the hydrophobicity of the membrane surface are incorporated (Gekas, Trägårdh, & Hallström, 1993; Pinelo, Jonsson, & Meyer, 2009). Thereby, MWCO is not an absolute barrier for the separation of macro- and micro-molecules. Besides, larger and smaller functional molecules exist in clusters inside bioresource matrixes, i.e. phenols bind either dietary fibers of plant materials (Bravo, Abia, & Saura-Calixto, 1994) or dietary proteins (non-covalently) (Rawel, Meidtner, & Kroll, 2005). Subsequently, smaller molecules (i.e. antioxidants) recaptured in the concentrate stream due to the structural characteristics of the macromolecules or the reverse. This is the main reason why natural extracts containing compounds that are not so antioxidant (i.e. oligosaccharides or proteins), appear to have advanced antiradical and reducing properties. The above aspect could be desirable depending on the product that the food technologist is willing to develop. However, in terms of food separation, the simultaneous recovery of macro- and micro-molecules in one stream is a problem leading to additional purification stages, yield loss and finally increased cost.

Nowadays, researchers focus more and more on membrane applications and separation of proteins, dietary fiber, polyphenols, anthocyanins, tannins, flavonoids, saccharides and sugars in fruit juices, solutions, agricultural wastewaters and beverages (Cassano, Conidi, Giorno, & Drioli, 2013; Díaz-Reinoso, Moure, Domínguez, & Parajó, 2009; García-Martín *et al*., 2010; He, Girgih, Malomo, Ju, &

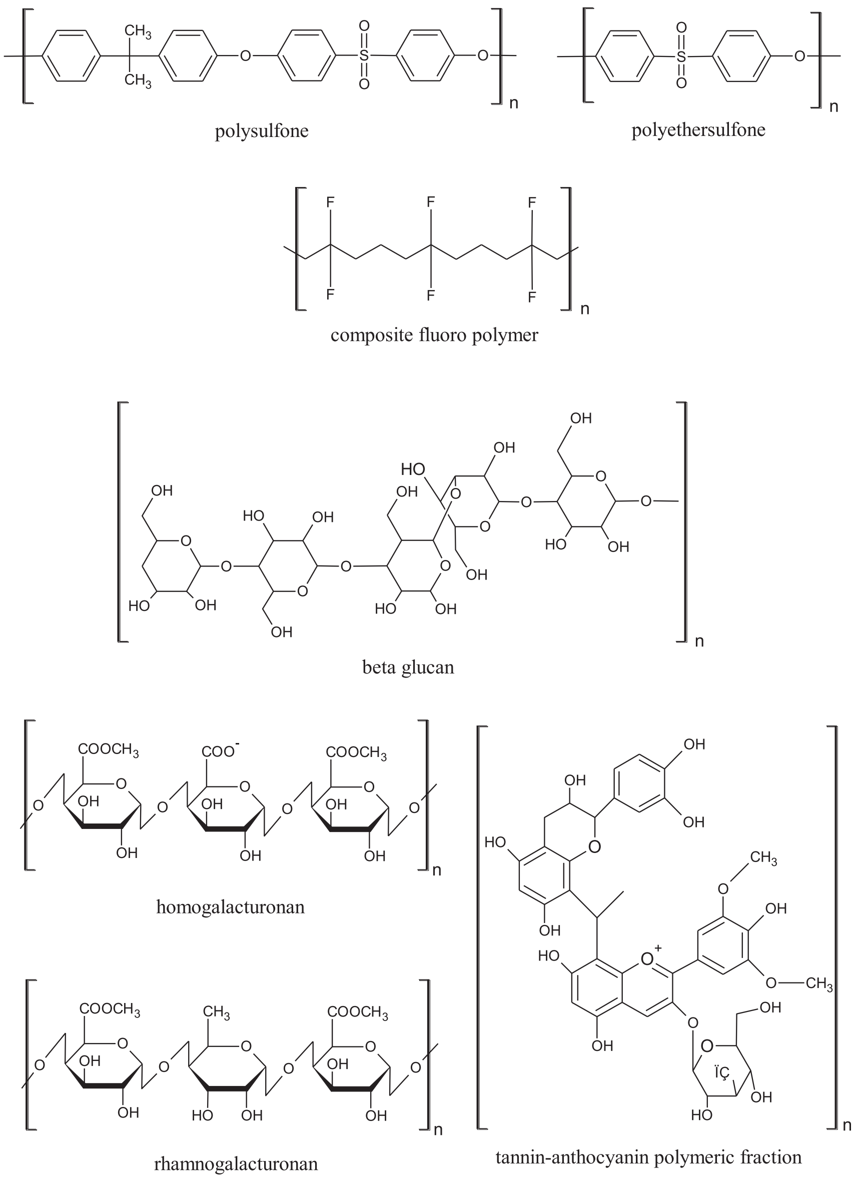

Fig. 1. Chemical structural units of polymeric membrane materials and polymeric functional compounds.

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