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Production of antimicrobial membranes loaded with potassium sorbate using a supercritical phase separation process

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ABSTRACT

The production of antimicrobial packaging is one of the most interesting challenges in the food industry; its scope is to prolong the shelf life of a food maintaining its safety and freshness. The major limitation of traditional techniques used to produce antimicrobial packaging, is the difficulty in controlling the release of the active agent from the device to the food surface.

In this work, a supercritical phase inversion process has been tested to produce potassium sorbate (PS) loaded cellulose acetate (CA) membranes, to be inserted in food packaging. The membranes have been obtained at different process conditions (pressures 150–250 bar, temperatures 35–55 °C) and at different polymer concentrations (10, 15 and 20% *w*/*w*). PS to CA weight ratio has been maintained constant at 5% *w*/*w* for all the formulations. The best process parameter combination to obtain the longest PS release time (about 325 min) was 250 bar and 35 °C.

Industrial relevance: The production of antimicrobial active packagings is one of the most attractive challenges in the food industry also catalyzed by consumers' demand for natural and safe foods and for environmental protection [1]. Antimicrobial food packaging reduces, inhibits or retards the growth of microorganisms that may be present in the food or packaging material itself [2], to extend the shelf life of the packed food. The most desired property is the controlled release of the antimicrobial agent to the food surface [3–6]. This characteristic is influenced by the technique used to produce the film. To overcome conventional processes limitations, it could be possible to use supercritical assisted processes, taking advantage of the properties of supercritical fluids, such as negligible surface tension, high diffusivity and low viscosity. In particular, a supercritical phase separation process has been successfully proposed to produce loaded polymeric membranes to be inserted in food packaging.

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1. Introduction

The production of antimicrobial active packaging is one of the most interesting challenges in the food industry. Its scope is to prolong the shelf life of a food and to maintain its safety and freshness. Traditionally, antimicrobial agents are directly mixed with the food formulation; but, it is difficult to control the agent activity and selectivity, due to the reactions with the food components (Appendini & Hotchkiss, 2002; Min & Krochta, 2005). An alternative to this process, can be the use of antimicrobial devices and antimicrobial films, inserted in the food packaging.

Some characteristics are required for the effectiveness of an active packaging; in particular, a suitable concentration and a proper release

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rate of the active agent to avoid burst effects or a too slow release (Appendini & Hotchkiss, 2002; Vermeiren, Devlieghere, Van Beest, De Kruijf, & Debevere, 1999).

Scientific literature reports several techniques to produce antimicrobial packaging (Appendini & Hotchkiss, 2002; Cha & Chinnan, 2004; Vermeiren et al., 1999): 1) sachets containing volatile antimicrobial agents; 2) incorporation of volatile and non-volatile antimicrobial agents directly into polymers; 3) coating or adsorption of antimicrobials onto polymer surfaces; 4) immobilization of antimicrobials on polymers by ion or covalent linkages; and 5) use of antimicrobial polymers.

The major limitation of all the proposed methods, is the difficulty in controlling the release of the active agent from the device to the food surface. Some authors tried to overcome this problem using multilayer films containing an active agent (Buonocore, Conte, Corbo, Sinigaglia, & Del Nobile, 2005; Mastromatteo, Barbuzzi, Conte, & Del Nobile, 2009; Mastromatteo, Mastromatteo, Conte, & Del Nobile, 2010; Uz & Altınkaya, 2011) or producing films loaded with the antimicrobial agent (Gemili, Yemenicioğlu, & Altınkaya, 2009; Tankhiwale & Bajpai, 2012). But, they had the problem of cross-linking the polymer to prolong the release of the active compound. In the case of membrane-like processes, an asymmetric porous structure was produced by evaporation of the organic solvent (Gemili et al., 2009). Using this process, it is difficult to control membrane morphology, since the operative parameters are temperature and humidity only.

Potassium sorbate (PS) is a potassium salt of the sorbic acid, well-known for its potential antifungal activity (Mehyar, Al-Qadiri, & Swanson, 2014; Sayanjali, Ghanbarzadeh, & Ghiassifar, 2011; Valencia-Chamorro, Palou, Del Rio, & Pérez-Gago, 2008). It has been used to inhibit the growth of molds and yeasts; but, it is active also against *Staphylococcus aureus*, *Clostridium botulinum*, *Salmonellae* and *Pseudomonads* (Zamora & Zaritzky, 1987). Its stability is not a problem; but, its release is problematic. Sayanjali et al. (2011) produced by solvent evaporation, carboxymethyl cellulose-based edible film containing PS as an antimicrobial agent. They demonstrated the efficacy of the antimicrobial film against *Aspergillus parasiticus* and *A. flavus*. Moreover, they coated pistachios with this film, obtaining mold growth inhibition (Sayanjali et al., 2011). Basch, Jagus, and Flores (2013) studied the antimicrobial effectiveness of nisin and PS incorporated in films produced by the solvent evaporation technique of tapioca starch and its mixtures with hydroxypropyl methylcellulose. They verified that the combination of antimicrobials was more effective against *Listeria innocua* and *Zygosaccharomyces bailii*, than their individual incorporation (Basch et al., 2013).

However, in these studies, the authors produced dense films. Therefore, the active agent release and its action were reduced by the large mass transfer resistance in the polymer matrix. Moreover, solvent evaporation technique can produce the deposition of the active agent on the bottom surface of the film due to the slowness of the process, changing film performance in dependence of which surface is in contact with food.

Table 1
Weight percentages of each component used for the sample preparation.

CA + acetone (95% *w*/*w*)		PS + H_2O (5% *w*/*w*)	
CA	Acetone	PS	H_2O
10% *w*/*w*	90% *w*/*w*	10% *w*/*w*	90% *w*/*w*
15% *w*/*w*	85% *w*/*w*	15% *w*/*w*	85% *w*/*w*
20% *w*/*w*	80% *w*/*w*	20% *w*/*w*	80% *w*/*w*

To overcome conventional process limitation, it could be possible to use supercritical assisted processes, taking advantage of the properties of supercritical fluids, such as negligible surface tension, high diffusivity and low viscosity. These characteristics have been already successfully used to produce micro- and nanoparticles, in pharmaceutical, food and biomedical fields (Campardelli, Baldino, & Reverchon, 2015; Della Porta, Falco, Giordano, & Reverchon, 2013; Garofalo et al., 2014; Liparoti, Adami, & Reverchon, 2015; Prosapio, Reverchon, & De Marco, 2014; Reverchon & Adami, 2013; Espirito Santo et al., 2014), in the separation and/or extraction of natural compounds (De Melo, Silvestre, & Silva, 2014).

A supercritical phase separation process has been proposed to produce loaded polymeric membranes; the literature shows that this process is more versatile with respect to the traditional ones, since changing operative conditions (i.e., pressure and temperature) makes it possible to influence the kinetics and thermodynamics of the process

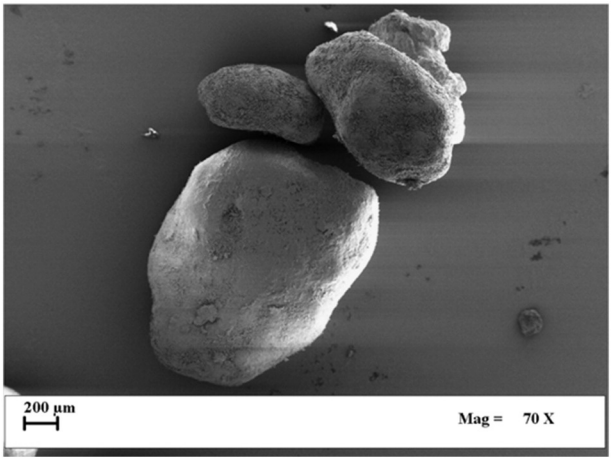

Fig. 1. Untreated potassium sorbate powder.

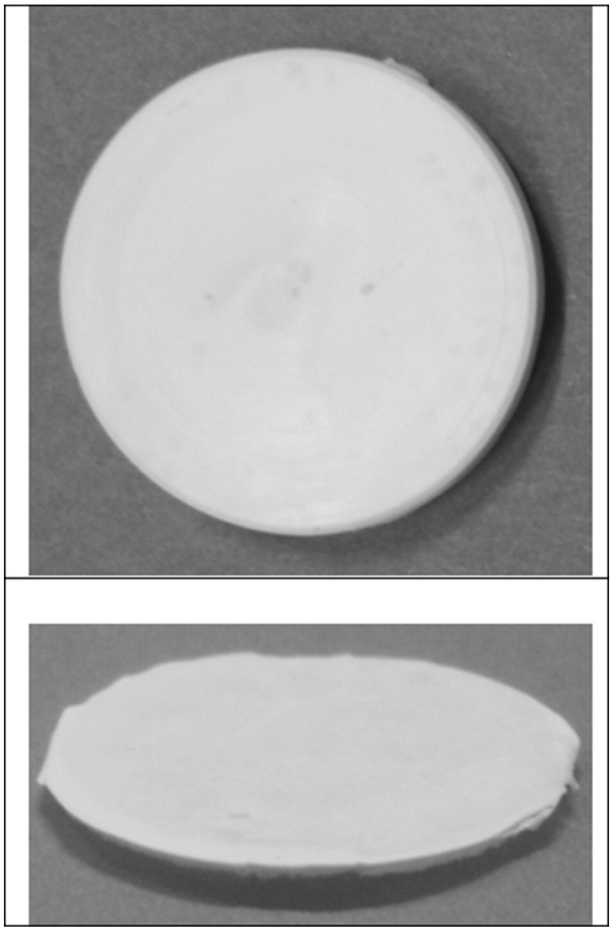

Fig. 2. Pictures of an antimicrobial CA membrane processed by supercritical phase separation at 200 bar and 45 °C.