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Rapid assessment of the effectiveness of antioxidant active packaging—Study with grape pomace and olive leaf extracts

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ABSTRACT

Natural antioxidants are mixtures of different components with their specific partition and diffusion coefficients and the exact description of the release kinetics into foods is, therefore, complex. For practical application however, the interest should be focused on the antioxidant effect rather than on the single compounds release, i.e., it is an approximation to describe the release of a mixture by treating it as single substance. The evaluation of such approach was subject of this study.

Film samples coated with Shellac (Shel) and cellulose nitrate (NC), containing olive leaf extract (OLE) and grape pomace extract (GPE) at different concentrations were put in contact with three food simulants, namely W (water), A (10% ethanol) and D1 (50% ethanol), and the antioxidant capacity was assessed by suitably modified ABTS test.

The equilibrium antioxidant capacity increased from W to A and D1 for GPE and OLE: the antioxidant levels attained films containing GPE were 0.272, 0.483 and 0.728 Trolox mEq/L at 5% concentration, and 0.705, 0.786 and 0.893 Trolox mEq/L at 10% concentration for W, A and D1 simulants, respectively. The method was also effective at comparing the lacquer retention performances, NC being more effective than the Shel, i.e., it showed a faster release of antioxidant compounds.

1. Introduction

Packaging materials with antimicrobial and antioxidant properties are an on-going topic of applied research (Colín-Chávez, Soto-Valdez, & Peralta, 2014; Hauser, Peñaloza, Rodríguez, Guarda, & Galotto, 2014; Hauser, Müller, Sauer, Augner, & Pischetsrieder, 2014; Marcos et al., 2014; Muppalla, Kanatt, Chawla, & Sharma, 2014). Trends in the food industry and food research are the application of natural ingredients, i.e., natural preservatives derived from natural sources, instead of synthetic substances and the reduction of additives in foods (Ali, Noh, & Mustafa, 2015; Shah, Bosco, & Mir, 2015; Wittenauer, Falk, Schweiggert-Weisz, & Carle, 2012). As compared to the direct addition of antioxidants in food, antioxidant packaging materials provide the release of active agents to the food at controlled rates, compensating the continuous using up of antioxidants during storage (Mastromatteo, Mastromatteo, Conte, & Del Nobile, 2010; Marcos et al., 2014). Furthermore, they are released to the surface of the food, this part being the most exposed to oxidation. The development of active packaging is set on various steps, starting with the evaluation of the in vitro antioxidant and/or antimicrobial efficacy of the applied compound, up to the final application in food packaging including shelf life studies of the food packed in the prototype materials. However, most studies do not consider the release kinetics of the active compounds. Indeed, the release of antioxidants and antimicrobials depends on the matrix in which they are included (Muppalla et al., 2014) and on the inclusion technique used, and also on the extractive capacity of the food which is meant to be packed in the active material. Therefore, the design of an active packaging should be addressed to a specific food or food category. Most publications focusing on antioxidant packaging have basically used one of the following approaches for the study of their effectiveness: (a) evaluation of the antioxidant capacity of the compound or mixture before inclusion in the film (Barbosa-Pereira, Aurrekoetxea, Angulo, Paseiro-Losada, & Cruz, 2014; Hauser, Peñaloza et al., 2014); (b) evaluation of the antioxidant capacity or recovery of active agents from an active film extract (Bentayeb, Vera, Rubio, & Nerin, 2009; Colín-Chávez, Soto-Valdez,

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Peralta, Lizardi-Mendoza, & Balandrán-Quintana, 2013; Li, Miao, Wu, Chen, & Zhang, 2014; Marcos et al., 2014; Colín-Chávez et al., 2014); (c) evaluation of the effects on a specific food product packed in the active material (Nerín et al., 2006; Pereira de Abreu, Losada, Maroto, & Cruz, 2010; Ünalan, Arcan, Korel, & Yemenicioğlu, 2013; Barbosa-Pereira et al., 2014; Contini et al., 2014). The inclusion technique and the affinity for the matrix could strongly influence the effectiveness of the releasing system, making the first approach insufficient to estimate the real performance of the active packaging. Moreover, active packaging is based on the principle of modulated release: the included compound should, therefore, be released during the storage time of the product, following a kinetic which will proceed until equilibrium between the film and the food matrix. For this reason, also the second approach seems less suitable to evaluate the real effectiveness of active films, as the quantitative extraction of the compound from the film leads to an overestimation of its antioxidant potential. The latter approach is effective since it evaluates the performances of the packaging system in the real conditions, but its application is limited to one specific food product.

Various procedures have been proposed for assessing the antioxidant activity of biological systems, based on producing and measuring radicals: these methods are generally used to measure the antioxidant activity of foods and natural extracts, and they could be exploited, if suitably adapted, for the assessment of the antioxidants released by the films (Gómez-Estaca, López-de-Dicastillo, Hernández-Muñoz, Catalá, & Gavara, 2014).

Some articles have evaluated the release kinetics of active compounds from functional packaging (Zhong, & Li, 2011; Flores, Conte, Campos, Gerschenson, & Del Nobile, 2007; Licciardello, Muratore, Mercea, Tosa, & Nerín, 2013; Peng, Wu, & Li, 2013; Colín-Chávez et al., 2014; Marcos et al., 2014), this approach takes into account the effect of the matrix and the partition coefficients of the compounds between the matrix and the contact phase. However, in the case of antioxidant packaging, a simple method for the assessment of the effectiveness should focus on the effect produced (i.e., the total antioxidant capacity) rather than on the amounts of compound released, especially in the case of mixtures of compounds, such as natural extracts.

The aim of the present research was to develop a simple method and approximation for the assessment of the antioxidant capacity of active films containing olive leaf extract (OLE) and grape pomace extract (GPE); the method consists of one single step in which the extraction of active components (by simulant solutions) and their determination (by a common test for antioxidant activity) take place at the same time based on simultaneous extraction and reaction. The method was used to obtain information on the antioxidant release kinetics in different food systems: for this aim, three food simulants described in the EU Reg. 10/2011 (EU, 2011) were used, as to simulate the extractive capacity of non-fatty and fatty foods. The determination of the antioxidant capacity was performed by suitably modified ABTS test to compare the effectiveness of different bio based coatings and natural antioxidant substances.

2. Materials and methods

2.1. Materials

All solvents used in the experiments were HPLC grade and purchased from VWR (Darmstadt, Germany). Folin-Ciocalteu reagent, ABTS (2,2′-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid)), DPPH (2,2′-diphenyl-1-picrylhydrazyl), gallic acid and Trolox (6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid) were purchased from Sigma–Aldrich (Steinheim, Germany). All reagents were of analytical or HPLC grade.

The following coating lacquers were used: Superol–Barrier–Varnish, referred to as Shel (chemical basis: Shellac) and Cellax–Barrier–Varnish, referred to as CN (chemical basis: cellulose nitrate), both supplied by Landshuter Lackfabrick Eduard Leiss GmbH (Landshut, Germany).

Olive leaf extract (OLE) OleXelO-T was kindly supplied by N-Zyme BioTec GmbH (Germany).

2.2. Preparation of grape pomace extract (GPE)

Ground lyophilised grape pomace was extracted with a mixture of ethanol, water and acetic acid (50:49:1; v/v/v, 50 °C) for 60 min at a liquid/solid ratio of 1:20. After cross-flow-filtration, the extract was evaporated at 55 °C in a falling film evaporator. Aliquots of the concentrated extract were stored at −20 °C until analysis.

2.3. Preparation of coated films

Oriented polypropylene (OPP) (thickness 20 μm) was corona-treated (812 W) to obtain a final surface energy > 44 dyne/cm. The treated polyolefin film was cut into A4-sized sheets, which were coated with the lacquers described hereafter, by an automated coating machine (Coating-Unit CUF 5, Sumet-Messtechnik, Denklingen, Germany), set at 25 mm/s speed, dried at 50 °C for 1 min.

The lacquers were coated on corona-treated OPP with and without the addition of GPE and OLE. Shellac-based lacquer was incorporated with 5% and 10% (w/w) GPE, and 2.5% (w/w) OLE, CN was incorporated with 2.5% (w/w) OLE. The resulting thickness of the coating layer, as determined by the difference between the thickness of coated and uncoated films, measured by a micrometer, was averagely 5.27 ± 0.97 μm.

2.4. Total polyphenol content and antioxidant capacity of natural extracts

Total phenolics were determined by the Folin Ciocalteu method, according to Singleton and Rossi (1965) with slight modifications. Aliquots of 0.02 mL diluted extract, 1 mL water and 0.1 mL Folin reagent were mixed. After 3 min, 0.2 mL sodium carbonate solution (7.5%, w/v) and 0.68 mL water were added. Absorption was measured at 765 nm after 30 min using a Specord 210 plus spectrophotometer (Analytik Jena, Jena, Germany). Total polyphenols were quantified by calibration with standard gallic acid.

The total antioxidant capacity of the extracts was assessed by $ABTS^{+}$ (2,20-azino-di-[3-ethylbenzthiazoline sulphonate]) and $DPPH^{\bullet}$(2,2-diphenyl-1-picrylhydrazyl) radical scavenging methods as described by Re et al. (1999) and Brand-Williams, Cuvelier, & Berset (1995), respectively, and expressed as Trolox equivalents. ABTS stock solution was prepared dissolving 38.4 mg ABTS and 6.62 mg potassium persulfate in 10 mL water. Working solution was prepared diluting about 1.1 μL stock solution into 100 mL water, to reach an initial Abs value (734 nm) of 0.70 ± 0.02. The reaction was performed mixing 0.01 mL sample or standard solution with 9.99 mL working solution, and reading was performed at 734 nm after incubation for 15 min at 30 °C.

DPPH stock solution was prepared dissolving 23.6 mg DPPH in 50 mL ethanol. Working solution was prepared diluting stock solution 20 fold (final concentration 2.36 mg/100 mL). The reaction was performed mixing 0.01 mL sample or standard solution with 9.99 mL working solution and reading was performed at 517 nm after 60 min at 25 °C.

The antioxidant capacity was expressed as ABTS and DPPH% inhibition as: $I\% = (A_{blank} - A_{sample})/A_{blank} \times 100$. Percent inhibition was converted to Trolox mEq by comparison with a calibration curve obtained with appropriate dilutions of standard Trolox.

All determinations were performed in triplicate.