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Thermal stability and flame resistance of cotton fabrics treated with whey proteins

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ABSTRACT

It is well described in the literature that whey proteins are able to form coatings, which exhibit high mechanical and oxygen barrier properties, notwithstanding a great water vapour adsorption. These peculiarities have been exploited for applying a novel protein-based finishing treatment to cotton and for assessing the protein effect on the thermal and thermo-oxidative stability and on the flame retardant properties of the cellulosic fabric. Indeed, the deposited whey protein coatings have turned out to significantly affect the thermal degradation of cotton in inert and oxidative atmosphere, and to somehow modify its combustion when a flame has been applied. Furthermore, the influence of the secondary and tertiary structure of these proteins on the morphology of the deposited coating, and thus on the thermal and flame retardant properties of the treated fabrics, has been evaluated by performing a denaturation thermal treatment before the protein application.

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1. Introduction

In the last few decades, the use of whey protein isolate (WPI) has received a remarkable interest for the production of films for food packaging. Indeed, whey protein components (α-lactalbumin and β-lactoglobulin) have been widely studied for their potential use as agents able to form edible and biodegradable films based on a waste stream from the cheese industry (Kokoszka, Debeaufort, Lenart, & Voilley, 2010). Usually, WPI products are obtained by ion exchange, followed by ultrafiltration or microfiltration and a subsequent ultrafiltration. This interest has been motivated by their high mechanical properties (Fairley, Monahan, German, & Krotcha, 1996; Khwaldia, Pereze, Banon, Desobry, & Hardy, 2004) and excellent oxygen barrier properties (Sothornvit, Olsen, McHugh, & Krochta, 2003, 2007). The film-forming properties of WPI have been applied for obtaining transparent, flexible, colourless, and odourless films. Sometimes, the incorporation of a minimal content of plasticizer (like glycerol and polyethylenoxide) within the protein mixture is necessary in order to favour the film formation and to reduce the film brittleness (Gounga, Xu, & Wang, 2007, 2010). Indeed, these plasticizers are capable to weaken intermolecular forces between adjacent protein chains: as a consequence, films having increased film extensibility and flexibility as well as decreased elasticity, mechanical resistance and oxygen/moisture barrier properties are obtained (McHugh, Avena-Bustillos, & Krochta, 1993; McHugh, Aujard, & Krochta, 1994; Hong & Krochta, 2003; Mate & Krochta, 1996).

In this context, the possibility to have remarkable oxygen barrier properties could represent a key issue when protein films (without plasticizer) are applied as coatings onto a polymeric substrate for two main reasons. First, these films could prevent, delay, or partially inhibit the thermal degradation of a polymer in inert and oxidative atmospheres. In addition, the water adsorption feature could be exploited for inhibiting the flammability of a thin polymeric substrate such as a fabric, since the absorbed water could partially dissipate the heat during the combustion and dilute the produced volatile species (Alongi & Malucelli, in press).

As far as plastics and textiles are concerned, the possibility of using green flame retardant systems for replacing the traditional additives is one of the driving forces that continue to stimulate the industrial and academic research toward novel and innovative solutions. In particular, the availability of a formaldehyde-free flame retardant system based on natural macromolecules such as proteins could be extremely interesting for a possible industrial application. Therefore, in the present work, the effect of the presence of a whey protein isolate coating (consisting of both folded and unfolded/denatured chains) on the thermal and thermo-oxidative stability and on the flame retardant properties of cotton fabrics has been thoroughly investigated by using thermogravimetry and flammability tests (in horizontal configuration), respectively. To the best of the authors' knowledge, this is the first example of

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Table 1
Amino acid profile of whey protein isolate.

Amino acid	Concentration [%]
L-cysteine	3.2
L-arginine	2.4
L-tyrosine	3.4
L-proline	3.6
Glycine	1.5
L-glutamic acid	14.2
L-aspartic acid	10.1
L-serine	3.6
L-threonine	4.3
L-valine	4.5
L-methionine	1.9
L-isoleucine	5.1
L-leucine	11.5
L-lysine	9.0
L-histidine	1.6
L-triptophan	1.9
L-alanine	4.6
L-phenylalanine	3.1

potential application of whey proteins to cotton fabrics for flame retardancy.

2. Experimental part

2.1. Materials

Cotton (COT, 220 g/m^2) was purchased from Fratelli Ballesio S.r.l. (Torino, Italy).

WPI powder (93.5 wt.% protein) was purchased from Anderson Research (Cervaro (FR), Italy); its overall composition also includes lipids (ca. 0.5 wt.%), carbohydrates (ca. 1 wt.%), ash (ca. 2.2 wt.%), and moisture (ca. 2.8 wt.%). Furthermore, the amino acid content is equal to 89.5 wt.% and its profile is listed in Table 1, as indicated in the data sheet provided by the supplier.

2.2. Application of whey protein coatings to cotton fabrics

The suspension of folded proteins (10 wt.%) was prepared by slowly dissolving WPI in distilled water under magnetic stirring (300 rpm) at room temperature for 10 min. The suspension of unfolded/denatured proteins was obtained from the folded protein suspension: first, pH was corrected to 7.0 with NaOH (0.1 M), then the suspension was heated up to 90 °C in a thermostatic bath under magnetic stirring (300 rpm) for 2 min, and finally cooled down to room temperature. Cotton fabrics were impregnated with the (folded or unfolded) protein suspensions for 1 min in a climatic chamber (30 °C and 30%R.H.); the excess of the suspension was then removed with a rotary drum and the impregnated fabrics were dried to constant weight in climatic chamber.

The treated samples were coded as COT_WP and COT_DWP, which refer to cotton fabrics treated with folded (WP) and unfolded/denatured proteins (DWP), respectively.

The total dry solids add-on on cotton samples (A, wt.%) was determined by weighting each sample before (W_i) and after the impregnation with the solution and the subsequent thermal treatment (W_f), using a Gibertini balance ($\pm 10^{-4}$ g). The uptake of COT_WP (ca. 25%) and COT_DWP (ca. 20%) was calculated according to the following equation:

$$A = \frac{W_f - W_i}{W_i} \times 100$$

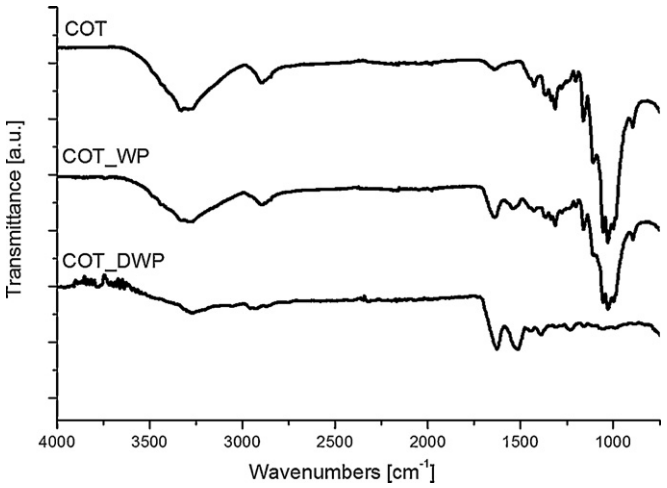

Fig. 1. ATR spectra of COT, COT_WP and COT_DWP.

2.3. Characterization techniques

The surface morphology of the treated samples was studied using a LEO-1450VP Scanning Electron Microscope (beam voltage: 5 kV); an X-ray probe (INCA Energy Oxford, Cu-Kα X-ray source, k = 1.540562 Å) was used to perform elemental analysis. Fabric pieces (5 mm × 5 mm) were cut and fixed to conductive adhesive tapes and gold-metallized.

The thermal and thermo-oxidative stability of the fabrics was evaluated by thermogravimetric (TG) analyses in nitrogen and in air, respectively, from 50 to 800 °C with a heating rate of 10 °C/min. A TAQ500 analyzer was used, placing the samples (ca. 10 mg) in open alumina pans, in inert or oxidative atmosphere (gas flow: 60 ml/min).

Flammability tests in horizontal configuration were carried out by applying a methane flame for 5 s on the short side of the specimen (50 mm × 100 mm). These tests were repeated 3 times for each formulation. Total burning time and rate after the flame applications as well as the final residue were measured.

The chemical structure of the prepared samples and residues left after TG analyses was evaluated by Attenuated Total Reflectance (ATR) spectroscopy. ATR spectra were recorded at room temperature in the range 4000–600 cm^{-1} (32 scans and 4 cm^{-1} resolution), using a Frontier FT-IR/FIR spectrophotometer, equipped with a diamond crystal.

In order to determine the moisture content of the pure and treated cotton fabrics, a Karl-Fisher titrator (Mettler Toledo, model V20) was used: small pieces (ca. 0.1 g) were heated up to 150 °C and the moisture content was determined by using a mixture of methanol, potassium metabisulphite and I_2 as the titration system. The error was 0.5%.

3. Results and discussion

3.1. ATR spectroscopy

The effectiveness of the deposition of the whey protein isolate coatings on the cotton fabrics has been assessed through ATR spectroscopy. Fig. 1 shows the ATR spectra of COT, COT_WP and COT_DWP. First of all, the typical vibration modes of cellulose are well detectable in the pure cotton (namely, ν(OH) at ca. 3300, $\nu(CH_2)$ at 2900, δ(OH) at 1640, $\delta(CH_2)$ at 1425, δ(CH) at 1370, δ(OH) at 1310, ν(C—C) at 1020, and δ(OH) at 894 cm^{-1}). In the ATR spectra of COT_WP, two other peaks located at 1646 and 1533 cm^{-1} and substantially attributable to amide I and amide II vibrational

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