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Fabrication and characterization of tea polyphenols loaded pullulan-CMC electrospun nanofiber for fruit preservation

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ABSTRACT

Edible packaging films using polymer for food preservation have been developed for a long time. In this study, the effects of different concentrations (0.5%, 1%, 1.5%, w/v) of tea polyphenols incorporated into pullulan-Carboxymethylcellulose sodium (Pul-CMC) solutions on electrospun nanofiber films were evaluated. The fiber size distribution was characterized by scanning electron microscopy. The morphological features of nanofibers were modulated through adjusting process parameters (e.g. concentration of polymer solution, applied voltage and feeding rate). Increasing the applied voltage from 19 to 21 kV and the feed rate from 0.36 to 0.6 mL/h leads to a reduction in mean fiber diameter. Fruit packaging potential was evaluated using strawberry. The pullulan-CMC-TP nanofibers significantly decreased weight loss and maintained the firmness of the strawberries, and improved the quality of the fruit during storage. The findings demonstrate a facile packaging route to improve food sustainability and reduce waste.

1. Introduction

In the food industry, perishable food products increase the risk of food borne illness; and also easily spoiled causing waste and economic loss [1]; In order to slow down fresh fruits and vegetables metabolic processes and provide an effective preservation, many methods including controlled atmosphere storage [2], or coating edible film and irradiation [3] were tested to improve the physiological quality and prolong the shelf life of fresh fruits and vegetables.

Electrospinning is a broadly used technology for electrostatic fiber formation which utilizes electrical forces to produce polymer fibers with diameters ranging from 2 nm to several micrometers using polymer solutions of both natural and synthetic polymers. The technique has attracted great interests [4] because of many amazing properties such as large surface area-to-volume ratio [5] and high porosity with very small pore size [6]. The electrospun nanofibers have many applications include tissue engineering [7], filtration [8], protective clothing [9], wound dressing [10], catalysis reactions, and nano-sensor [11].

The food packaging materials need to contact with food products and are more acceptable by consumers when they are natural, efficient, non-toxic and do not produce off-flavor [12]. Currently, in the field of food packaging, it is of great interest in developing biodegradable active materials incorporate of active substances, such as antioxidants and antimicrobials [13]. Tea polyphenols (TPs) are bioactive catechins that have been reported to maintain the good quality of yellow croaker [14], pork meat patties [15], red drum [16], while incorporated into Pullulan or other polysaccharides films.

Pullulan is a water-soluble extracellular microbial polysaccharide produced by the fungus-like yeast, *Aureobasidium pullula*n [17]. The linear polymer mainly consists of maltotriose units interconnected to each other by an α-(1,6) glycosidic bond [18] and has several distinctive properties like high adhesion, sticking, lubrication, and film forming abilities [19]. Due to these excellent properties, Pullulan is utilized in low calorie foods, adhesive binders, thickeners, and encapsulating agents [20]. CMC is water-soluble and compatible with pullulan. Moreover, the formation of hydrogen bonds between the COO^{-1} groups of CMC and the —OH groups of pullulan may synergistically enhance the material properties of the resulting film.

While there are numerous studies reported on the material properties for the respective pullulan, and CMC films [21]. Few studies have been reported the effects of incorporation of TP in electrospun nanofibers for quality maintenance of fruits and vegetables. In this research the electrospinning technique was used to prepare nanofibrous for food preservation. For the electrospinning

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system, pullulan-CMC nanofibers were modulated through process parameters (e.g.active and polymer solution concentration, applied voltage and feed rate). Application of Pul-CMC-TP on fruit shelf-life was evaluated by using strawberry.

2. Materials and methods

2.1. Materials

Pullulan and CMC were purchased from Aladdin. Tea polyphenols were purchased from Pursue Biotechnology Co., Ltd. (Hangzhou, china). All chemicals and solvents were used without further purification. All chemicals were of analytical reagent grade unless specified and all the solutions were prepared with deionized water.

2.2. Preparation of solutions for electrospinning

3 g Pullulan was added into 20 mL of distilled water and stirred until it was dissolved completely, followed by the addition of CMC into the pullulan solution to make the final ratio of Pul:CMC in 90:10. The mixed polysaccharides solution was mechanically stirred in a flask for 1 h (300 rpm) at the ambient temperature (25 °C) to achieve a complete dissolution. Tea polyphenols (TP), at three different concentrations (0.5%,1%,1.5%,w/v) were added to Pul-CMC solution and mixed.

2.3. Electrospinning of pul-CMC-TP blended nanofibers

Pul-CMC-TP solution with different tea polyphenols (0.5%,1%,1.5%,w/v) was loaded into 2.5 mL plastic syringe, individually. The inner and outer diameter of needle is 0.6 and 0.9 mm, respectively. Different feed rates (between 0.36 and 0.6 mL/h) and electrical voltage (between 19 and 21 kV) was applied. A distance of 15 cm between the tip of needle and collector was set. Aluminum foil was used as the fiber collector substrate. Nanofibers electrospun by Pul-CMC solution with no tea polyphenols was used as control group. Relative humidity and temperature were fixed respectively at 45% ± 2% and 20 °C ± 2 °C.

2.4. Nanofibers morphology assessment

The morphology of the electrospun Pul/CMC/TP blended nanofibers was characterized by scanning electron microscope (VEGA-3-SBH, TESCAN, Czech). Samples were cut from an electrospun mat on the aluminum foil and mounted on aluminum stubs that were coated by an ultrathin layer of gold using a sputter coating device (Hummle VII, Anatech, Istanbul, Turkey) for better conductivity during imaging. Fiber diameters were also determined by using Image-J. For each electrospun mat, at least 100 fibers were considered from three different images to calculate the average diameter.

2.5. Fourier transform infrared spectroscopy

Fourier Transform Infrared (FTIR) spectroscopy was used to determine the presence of materials, chemical interactions and chemical stabilities of electrospun Pul/CMC/TP fibers. For electrospun fibrous film, ATR was used for FT-IR measurement. A total of 128 scans were accumulated for the signal-averaging of each IR spectral measurement to ensure a high signal-to-noise ratio with a 10 cm^{-1} resolution. The spectra of the samples were recorded over a wavenumber range of 600–4000 cm^{-1}.

2.6. Thermal analysis

Differential scanning calorimetric analyses (DSC) were carried out using a differential scanning calorimeter (Q20, TA Instruments, USA). Approximately 9 mg of Pul, CMC, nanofibers sample were equilibrated at 25 °C, and then heated to 250 °C at a scanning rate of 5 °C/min.

2.7. Assessment of strawberry quality

2.7.1. Sample treatment

The electrospun films were peeled off from the aluminum foil and then used to cover fruit surface for film application studies. To demonstrate the preservative function of fibrous Pul/CMC/TP films, four treatment groups (six portions of fruits in each group) were stored at a temperature of 4 °C and humidity of 55 ± 2%. The four treatments include the fruits packaged without film (control), fruits packaged with different nanofibers: nanofibers A (electrospun by Pul + CMC), nanofibers B (electrospun by 0.5%TP + Pul + CMC),nanofibers C (electrospun by 1%TP + Pul + CMC),nanofibers D(electrospun by 1.5%TP + Pul + CMC).

2.7.2. Weight loss

Analysis of weight loss was performed as described by Fan and co-workers [22]. The percentage of weight loss was calculated by weighing the samples every 2 days.

2.7.3. Titratable acidity (TA) analysis

The determination of titratable acidity was performed as a previous described method [23]. Briefly, four fruits were cut into pieces and 10 g of the mixed pieces was homogenized in distilled water, using a homogenizer (M-100L, Microfluidics, USA) at a speed of 70/min. The homogenate was subsequently filtered and centrifuged. Briefly, 5 mL of the strawberry juice was titrated by 0.1 mol/L sodium hydroxide to an end point of pH 8.1; the results were expressed as grams of citric acid per liter.

2.7.4. Firmness measurements

The firmness of the strawberry was evaluated using TA-TX plus texture analyzer (TA-TX plus texture analyzer, Stable Micro System Ltd. UK), as described in Yun's paper [24]. Puncture test was conducted by using a 3.5 mm cylinder probe, the penetration depth was 10 mm at a speed of 10 mm/s. Five of fruits were randomly selected from each treatment, and firmness was measured on the equatorial zone on two sides of each fruit. Results were expressed in $\times 10^5$ Pa.

2.8. Data analysis

Statistical analyses were performed using Origin (ver. 6.0, IBM software, Chicago, USA). One-way analysis of variance (ANOVA) was used to evaluate the significance of differences between sample groups at a level of $P \leq 0.05$.

3. Results and discussions

3.1. Effect of applied voltage and feed rate

By setting an applied voltage range during stable jetting (19–21 kV), the effect of voltage on fiber morphology was assessed. In the electrospinning process a crucial element is the applied voltage. The relationship between applied voltage and mean fiber diameter can be seen in Fig. 1a. Increasing the applied voltage from 19 to 21 kV leads to a reduction in mean fiber diameter from 0.187 to 0.092 μm. However, at a higher voltage there is also greater probability of beads formation. An increase in the applied voltage (i.e.,

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