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Functionalization of PVC by chitosan addition: Compound stability and tensile properties

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ABSTRACT

Chitosan is a biomaterial characterized by biodegradability, biocompatibility with human tissues, antimicrobial and antifungal activities, and for these reasons has been proposed in many different fields. The present work highlights the potentiality of chitosan as additive for active PVC-based composites, prepared by termo-mechanical process, with good mechanical-physical properties and antimicrobial activity. A specific mechanical characterization shows as elastic modulus and tensile strength increase with the chitosan concentration, as well as dynamical storage and loss moduli. These findings evidence that a significant interaction between chitosan and PVC occurs. The thermal stability and preliminary antimicrobial tests are encouraging.

1. Introduction

PVC is one of the most commonly used synthetic polymers in biomedical applications, especially as a catheter material for the circulatory system [1]. However, microbial colonization of medical device surfaces and the subsequent biofilm formation represents one of the most serious issues in healthcare-associated infection [2]. For this reason, during the last decade, there was an increasing interest in developing PVC-based materials with self-sterilizing and antibacterial properties. One possible approach is to incorporating antibacterial agents into the PVC matrix such as metal (Ag, Au, Cu, etc.) [2] and metal oxide (ZnO, TiO_2, CuO, CaO, MgO, etc.) nanoparticles [3–9]. However, processability of nanopowders is lowered by their tendency to agglomerate and several chemical and/or physical treatment strategies has to be used to avoid formation of clusters or agglomerates and achieve a uniform particle distribution in the polymer matrix.

A great effort was thus turned to find natural antibacterial molecules or materials that could be easily incorporated in the polymeric matrix by the common techniques. Among other possibilities, natural polymer such as chitosan offers real potential due to its physico-chemical properties, short time biodegradability, biocompatibility with human tissues, antimicrobial and antifungal activities, and non-toxicity. Chitosan is a linear polysaccharide prepared by alkaline deacetylation of chitin, commonly found in shells of marine crustaceans and cell wall of fungi [10,11]. Chitosan exhibits high antimicrobial activity against pathogenic and spoilage micro-organisms, including fungi, and both Gram-positive and Gram-negative bacteria [11,12] and, for these properties, silver chitosan-based nanoparticles, gold chitosan-based film and magnetic chitosan-based nanocomposites has been synthesized and proposed in many different fields, including medicine, food and chemical engineering, pharmaceuticals, nutrition, and agriculture [13–15].

Different techniques have been used to produce chitosan-based materials, such as solvent casting to produce β-chitosan-kudzu starch composite films [16], electrospinning process for pure chitosan nanofibers [17]; ultrathin films have been prepared by spin-coating of chitosan solution [18], and chitosan microsphere by spray-drying [19]. However chitosan, as powder or flakes, can be directly blended by extrusion or internal mixing with a molten thermoplastic polymer, such as polyethylene (PE) [20–22] or biodegradable aliphatic polyesters (poly-ε-caprolactone, PCL, poly(butylene succinate, PBS, poly(lactic acid), PLA, poly(butylene terephthalate adipate, PBTA, and poly(butylene succinate adipate), PBSA) [23]. To the authors' knowledge, there is not literature that studied the use and the effect of chitosan on the final properties of polyvyncloride (PVC) composites prepared by thermo-mechanical mixing. Some work reported the possibility to blend the chitosan with PVC polymer in solution: Dwivedi et al. [1], i.e. studied the blending of PVC with chitosan through simultaneous casting of their separate solutions in suitable solvents.

Then, since sustainable chemistry and engineering require the developments of materials and technologies based on natural polymers, this research investigates the role played by chitosan as additive for

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Table 1
Composition of the PVC/CS composites and Tg values as determined by DMA analysis.

Composite code	PVC (wt%)	Chitosan (wt%)	Tg (°C)
PVC	100	0	49
PVC_15	85	15	54
PVC_25	75	25	54
PVC_30	70	30	52
PVC_40	60	40	54

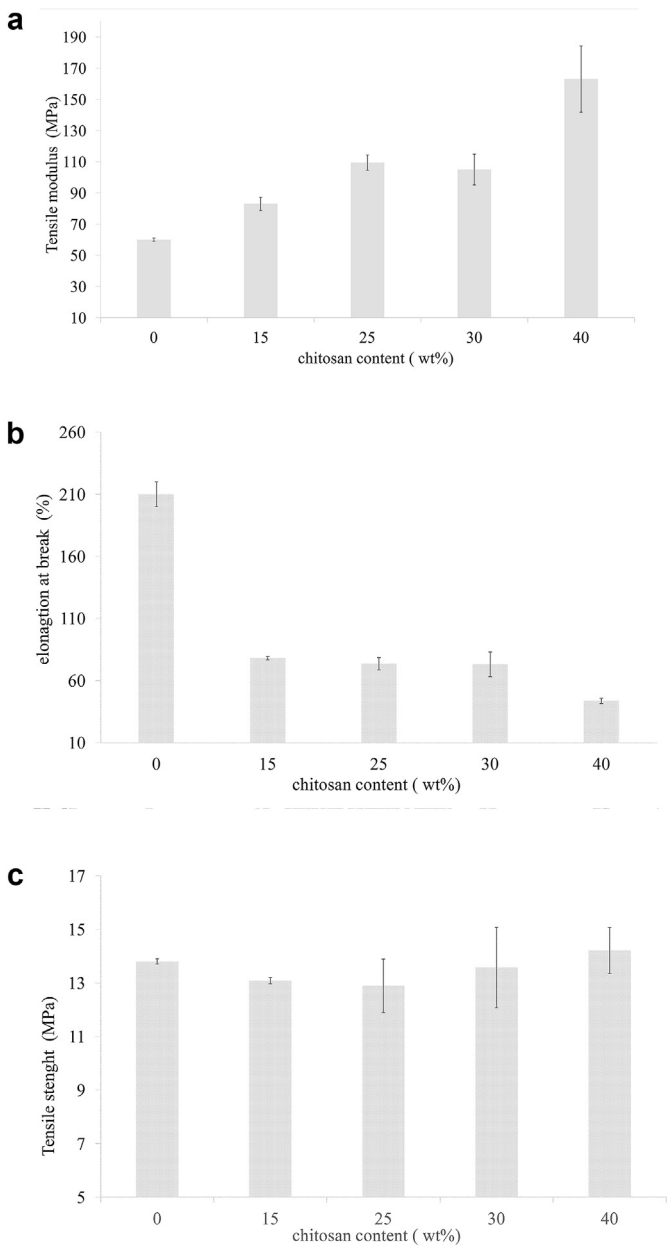

Fig. 1. Tensile properties of PVC/chitosan composites: tensile modulus (a), tensile strength (b), and elongation at break (c).

active PVC-based composites obtained by thermo-mechanical process. Subsequently, mechanical, thermal, structural and antibacterial properties were examined.

2. Materials

Chitosan flakes (CS) from crab shells (Practical grade > 85% deacetylated; Brookfield viscosity > 400 cps) were purchased from Sigma–Aldrich Chemical Co. Chitosan flakes were pulverized into a powder (average diameter below 250 μm) before compounding. The polymer matrix, PVC, was used as supplied (Scor SAS, Casaloldo, Italy, extrusion grade).

2.1. PVC/chitosan composite manufacturing

The polymer and chitosan were weighed at the desired addition level and premixed in a glass beaker. The mix was then melt-compounded in an internal mixer (HAAKE RHEOMIX, PolyLab System) at 150 °C for 10 min, at a mixing rate of 100 rpm. The composite formulations and the used codes are reported in Table 1. The mixed samples were compression-molded using a Carver hydraulic press of 12 tons (USA) at 150 °C for 10 min under a 250 bar pressure, and then cooled to ambient temperature under the same pressure.

2.2. Characterization

In order to evaluate the effect of CS on the mechanical properties of the matrix, standard tensile tests were performed with an Instron 3365 machine (USA). The samples were dog bones type V described in the standard procedure ASTM D638, with a gage length of 20 mm and cross section of $4 \times 2\ mm^2$; the tests were carried out at ambient temperature at the constant elongation rate of 1 mm/min. The average of at least five samples was reported for each formulation.

Dynamic mechanical analysis (DMA) was carried out on a TA Instruments DMA Q800 (New Castle, DE, USA) analyzer using a dual cantilever fixture, to determine the storage modulus, loss modulus, and tanδ of the samples. The test was performed in the temperature sweep mode from −30 °C to 100 °C at a frequency of 1 Hz and a heating rate of 3 °C/min. The sample dimensions were in this case $7 \times 4 \times 2\ mm^3$. At least three samples for each batch were tested.

The chemical compatibility between PVC and CS was studied by FT-IR analysis, recorded on a Avatar 330 FTIR ThermoNicolet. 32 scans between 4000 cm^{-1} and 700 cm^{-1} were averaged for each spectrum at intervals of 1 cm^{-1}. FT-IR spectra were generated by pressing 500 mg of dried sample on KBr crystal.

The microstructure of the samples was also observed on fracture surfaces using a scanning electron microscopy NovaNanoSem 450 (FEI, USA). An accelerating voltage of 10 kV was used. The samples were gold-coated in a sputter coater before collecting the images.

A thermogravimetric (TGA) analyzer (TGA 4000 System, PerkinElmer, USA) was used to investigate the thermal stability of the samples. Measurements were carried out under flowing nitrogen (50 ml/min) at a heating rate of 25 °C/min over a temperature range of 35–900 °C, with an initial sample weight of approximately 10 mg.

Finally, the antibacterial activity of the materials against *Escherichia coli* and *Staphylococcus aureus* was determined by Japanese Industrial Standard (JIS) Z2801:2000 method [24], slightly modified for our purposes. Briefly an appropriate inoculum (about 10^5 colony forming units, CFU) of an overnight cultures (37 °C) of *Staphylococcus aureus* ATCC 6538 and *E. coli* ATCC 25922 (indicator strains) in Tryptic Soy broth (TSB, Oxoid, Milan, Italy) were spread on the surface of the test pieces and covered with a polyethylene film. Each test piece was placed in a sterilized petri dish and incubated for 24 h at 35 °C temperature and at a relative humidity higher than 90% to prevent drying. Untreated "control" pieces were subjected to the same procedure. In order to recover the viable cells, the covering film and the test pieces were placed in a sterile plastic containers with 10 mL of saline solution. The microbial suspensions, opportunely diluted, were sown on Tryptic Soy agar (TSA; Oxoid) plates. After 24 h of incubation at 37 °C, the colonies were visually counted, and the number of CFU of the survival indicators were recorded and compared with those of untreated samples. Experiments were carried out in triplicate.

The antibacterial activity was quantified according to the following

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