



## Preparation and characterization of bionanocomposite films filled with nanorod-rich zinc oxide



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### ABSTRACT

The effects of zinc oxide nanorods (ZnO-nr) incorporation on the physical, mechanical, heat sealability, barrier, thermal and antibacterial properties of sago starch and bovine gelatin bionanocomposites films were investigated. The ZnO was incorporated into the films at different concentrations (1–5%, w/w total solid). All films were plasticized with 40% (w/w of total solid) of a combination of sorbitol/glycerol at 3:1 ratio. Incorporation of 5% of ZnO-nr to starch and gelatin films decreased the permeability to oxygen by 40% and 55%, respectively. Moisture content and water absorption capacity of the films were decreased by increasing the ZnO-nr contents. Mechanical and heat seal properties of the films were increased more than 20%. The films exhibited UV absorption and displayed an excellent antimicrobial activity against the *Escherichia coli*. These properties suggest that bionanocomposites based on ZnO-nr have the potential as an active packaging material for food and pharmaceutical industries.

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### 1. Introduction

Over the past decade, the combination of nanoscale science and other technologies have been explored to develop various novel materials and applications (Narayanamurti, 2006). For example, incorporation of nanoparticles to composite materials to produce nanocomposite has generated a great deal of attention due to enhancement of polymer properties such as thermal, mechanical and gas barrier (Kurian, Dasgupta, Galvin, Ziegler, & Beyer, 2006).

Metal or metal oxides, particularly ZnO, TiO<sub>2</sub>, MgO, and CaO, have attracted a great deal of attention (Fu et al., 2005) due to both stability under harsh condition processes and safe material for application in animals and human (Lin, Akil, & Mahmud, 2009).

Bionanocomposites as a new generation of nanocomposites represent the combination of a biopolymer and an inorganic material that shows at least one nanometer scale dimension (Darder, Aranda, & Ruiz-Hitzky, 2007). Bionanocomposites have been shown to exhibit enhanced thermal, mechanical, and functional properties due to nanoparticle fillers (Jia et al., 2006). Furthermore, biopolymer-based nanocomposites can be regarded as green technology and show the biocompatibility and biodegradability

in various applications such as agriculture, packaging, and pharmaceutical technology (Ma, Chang, Yang, & Yu, 2009; Nafchi, Moradpour, Saeidi, & Alias, 2013; Shameli, Ahmad, & Yunus, 2010).

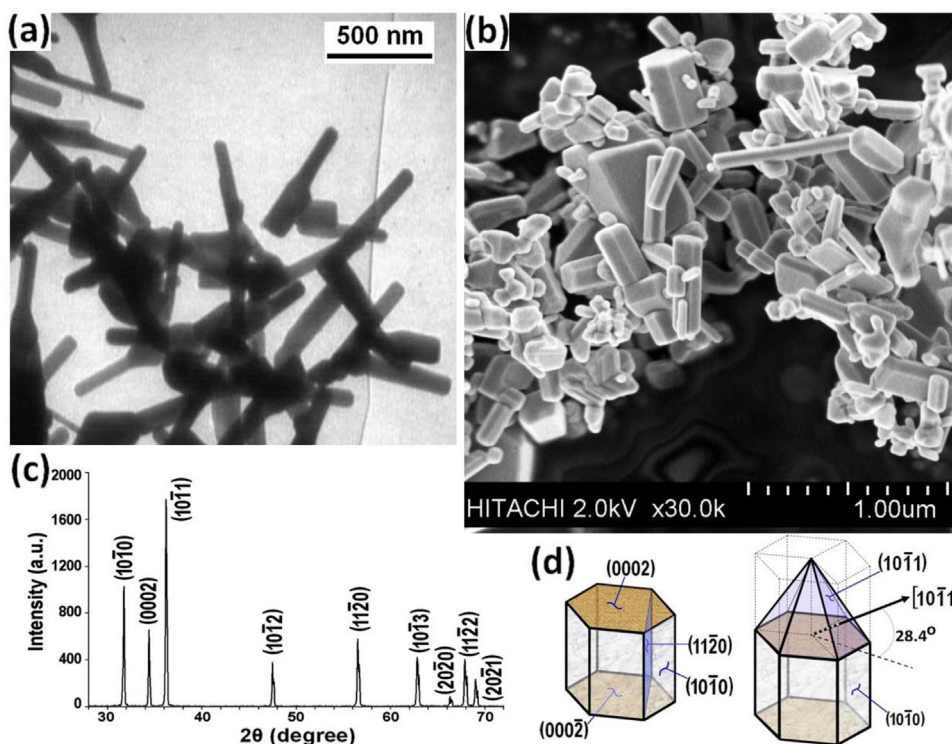
Development of biopolymer films is another emerging research area; studies using starch and gelatin have been widely reported. Starch is widely available for various food and non-food uses while gelatin is known as good film former especially in pharmaceutical industry. Sago starch, among other commercial starches, is relatively unknown and come from uncommon source (*Metroxylon sagu* palm tree) (Karim, Tie, Manan, & Zaidul, 2008).

The nanofillers have high surface energy and large specific surface area, giving rise to a strong interfacial interactions between polymer and the nanofillers and bring about a significant enhancement in the polymer properties (Kovačević, Vrsaljko, Lučić Blagojević, & Leskovic, 2008).

Zinc oxide nanoparticles as functional filler have been widely used as UV-absorbers in cosmetics, coating materials, pigments, and barrier enhancer in a number of industrial products (Kumar & Singh, 2008; Li et al., 2009; Yu, Cai, & Liu, 2004). In addition, zinc oxide nanoparticles have been reported to exhibit antimicrobial effects (Li et al., 2009; Li, Xing, Li, Jiang, & Ding, 2010; Zhang, Ding, Povey, & York, 2008). Zinc oxide is one of the five zinc compounds that are currently listed as generally recognized as safe (GRAS) by the U.S. Food and Drug Administration (21CFR182.8991). Also, Zinc oxide nanoparticles, which are nontoxic and biocompatible, have been utilized as drug carriers and medical filling materials (Lin et al., 2009b; Mirhosseini & Firouzabadi, 2012).

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**Fig. 1.** (a) TEM micrograph of a cluster of ZnO nanorods (b) FESEM micrograph of nanorod-rich ZnO, (c) XRD spectrum of the ZnO, and (d) ZnO crystal simulation.

Generally the intrinsic properties of nanoparticles depend on the size, crystallinity, composition, morphology, and the shapes of the particles (Shahrom & Abdullah, 2006; Yamamoto, 2001). Nanorod shapes of Zinc oxide have been reported as the optimum shapes for UV-absorption activity (Lin et al., 2009b). Although some reports are available (Ma et al., 2009; Yu, Yang, Liu, & Ma, 2009) on the effect of ZnO nanoparticles incorporation on the starch film properties, to our knowledge ZnO nanorod-reinforced biopolymer films have not been investigated. It is hypothesized that by application of low concentration nanorod zinc oxide into the film, biopolymeric film with a UV shielding property can be produced for the edible and non-edible packaging industries.

In this paper, ZnO nanorods was used as a filler to prepare sago starch/ZnO-nr or gelatin/ZnO-nr bionanocomposites. The films were characterized for their antibacterial, physicochemical, mechanical, heat sealability, barrier, and thermal properties.

## 2. Materials and methods

### 2.1. Materials

Sago starch (31% amylose and 69% amylopectin) was purchased from SIM Company Sdn. Bhd (Penang, Malaysia) and bovine gelatin (Type B) was purchased from Sigma Chemical Co (St. Louis, MO, USA). Liquid sorbitol and glycerol were purchased from Liang Traco (Penang, Malaysia).

Zinc oxide nanorod (ZnO-nr) was synthesized from a method known as catalyst-free combust-oxidized mesh process. The properties and synthesis of ZnO-nr were fully described by Shahrom and Abdullah (2007). The structure of ZnO-nr powder is shown in the TEM and FESEM micrographs (Fig. 1(a) and (b)) that show ZnO-nr with 40–100 nm diameter and 200–700 nm length. Some micro-particles are also present but in smaller amount. The XRD spectrum in Fig. 1(c) shows four major crystal indices,  $(10\bar{1}0)$ ,

$(0002)$ ,  $(10\bar{1}1)$  and  $(11\bar{2}0)$ , that form the ZnO rod crystal facets shown in the crystal simulations of Fig. 1(d).

*Escherichia coli* O157:H7 culture for antimicrobial assay was obtained from the culture collection center (School of Industrial Technology, Universiti Sains Malaysia, Malaysia). The bacterial culture was grown on nutrient agar slants and kept at 4 °C.

### 2.2. Preparation of nanobiocomposite films

ZnO-nr was dispersed in water at different concentrations (1, 2, 3, and 5%, w/w of total solid) and heated to 60 °C with continuous stirring for 1 h, then sonicated in an ultrasonic bath (Marconi model, Unique USC 45 kHz, Piracicaba, Brazil) for 30 min. The solution was cooled to room temperature and was used to prepare the aqueous starch dispersion 4% (w/w) and gelatin 8% (w/w). A mixture of sorbitol and glycerol (3:1) at 40% (w/w) of total solid was added as plasticizers. The choice of plasticizers and the concentration was based on Abdorreza, Cheng, and Karim (2011). The starch nanocomposites were heated to  $90 \pm 5$  °C and held at this temperature for 45 min to complete the gelatinization whereas gelatin nanocomposites were heated to  $58 \pm 2$  °C and held for 1 h. Upon completion of starch gelatinization, the solution was cooled to room temperature. The gelatin nanocomposite solution was cooled to 45 °C and the bubbles were removed by a laboratory scale vacuum pump.

A portion (90 g starch or 45 g gelatin) of the dispersion was cast on Perspex plates fitted with rims around the edge to yield a  $16 \times 16$  cm<sup>2</sup> film-forming area. The films were dried under controlled conditions in a climatic chamber (25 °C and 50% RH). The control films were prepared with the same plasticizers but without addition of the nanoparticles. Dried films were peeled and stored at  $23 \pm 2$  °C and  $50 \pm 5\%$  relative humidity (RH) until tested. The thickness of each film was measured at five different locations and to the nearest 0.01 mm with a hand-held micrometer (Mitutoyo, Tokyo, Japan). All films (including control) were prepared in triplicate.

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