Effect of drying chamber for nano-particle embedded bio degradable polymeric films

Rakesh B.Gautam*, Satish Kumar, Mahesh Kumar and Savita Sharma

Department of Processing and Food Engineering
Punjab Agricultural University Ludhiana, Punjab, India
Email:- rksh_gautam@yahoo.co.in

Abstract

The aim of this work was to develop antimicrobial biodegradable polymeric films based on whey protein isolate and titanium dioxide nanopowder. Five levels of sonication 20%, 40%, 60%, 80% and 100% affect the color and strength of biopolymers films. Effect of locally made chamber on drying time during drying of biodegradable films after casting was also evaluated. Water losses during sonication at different levels of sonication were found 8.924, 8.900, 11.924, 12.924 and 13.607. Titanium dioxide nanopowder was used as antimicrobial agent and strengthens material.

Keywords: Sonication, whey protein isolate, drying chamber and TiO_2

Paper cited: Gautam, R.B., Kumar, S., Kumar, M. and Sharma, S. (2015). Effect of drying chamber for nano-particle embedded bio degradable polymeric films. *South Asian Journal of Food Technology and Environment*, 1(3&4): 225-232.

Received: 13/08/2015 Revised: 09/09/2015 Accepted 1/10/2015

Introduction

Development of materials from natural polymers for different applications has been a hot topic for several years due to increasing prices of petrochemicals and increasing environmental concerns (Farris *et al.*, 2009; Laine *et al.*, 2013). For more natural products, bio-based films or biopolymers, improving the quality of many products is important to satisfy the consumers demanding of more environmentally friendly packaging. This approach will continue to play an important role in the food industry (Cutter, 2006; Satyanarayana *et al.*, 2009). Membranes obtained by combining proteins (e.g. soy protein, collagen, and gelatin) with a natural polymer (e.g. cellulose, chitosan) have been developed in an attempt to supply the high demand for new materials for skin repair as wound dressings in the treatment of wounds caused by burns (Azad *et al.*, 2004; Jeschke *et al.*, 2005; Silva *et al.*, 2005). The presence of protein in a polysaccharide-protein blend may improve the cell adhesion response of the resultant material due the presence of more protein-binding sites (Silva *et al.*,

2005). However, mixtures of protein and polysaccharide are often unstable, which leads to separation into two phases (Bourriot *et al.*, 1999; Tolstoguzov, 2000). If the biopolymers are incompatible, i.e. they repel each other, thermodynamic phase separation occurs, also called segregation or depletion interaction.

Whey protein isolate (WPI), which is an abundant by-products in the cheese industry, is a biopolymer that has received much attention for use as a potential edible or biodegradable food packaging material because it has been shown to produce transparent films and coatings that can act as an excellent oxygen barrier and provide certain mechanical properties (Sothornvit and Krochta, 2000). The ability of WPI-based edible films to act as barriers to solute and gas and enhance food quality and shelf-life has been previously demonstrated (Krochta and De Mulder-Johnston, 1997; Pérez-Gago and Krochta, 2002). However, these films do not have good mechanical and water vapour barrier properties

because they are intrinsically hydrophilic (Krochta and De Mulder-Johnston, 1997). As a result of this property, they cannot be used for wide spread applications in the food industry

Titanium dioxide (TiO_2) can provide protection against food borne micro-organisms as well as odour, staining, deterioration, and allergens in the presence of relative low wavelength radiation near the ultraviolet region. However, TiO_2 particle easily aggregate and thus deteriorate the properties of the film. In addition, the direct contact between TiO_2 and the polymer matrix may lead to rapid degradation of polymer films themselves (Kadam *et al.*, 2013). Titanium dioxide (TiO_2) due to the advantages of high chemical stability, high photo catalytic activity, relative low cost and non-toxicity, is one of the most important photo active reagents which is widely used for degradation (Ali *et al.*, 2011). The addition of nano-reinforcements has been related to improvement in overall performance of biopolymers, enhancing their mechanical, thermal and barrier properties, usually even at very low contents. Thus nano-particles have an important role to improve feasibility of use of biopolymers for several applications including food packaging (Azeredo, 2009)

Materials and Methods

Materials

Two cardboard were procured from local market of Ludhiana, Two bulbs (100 watt

and 0 watt) along with 50 metres wires were also procured from local market. BiPro WPI (96.6 g protein/100 g total solids, fat 0.2%, coliform < 3 , standard plate count cfu/g, yeast and mold cfu/g $< 10^4$, and Salmonella - negative) was supplied by Davisco Foods International (Eden Prairie, MN, USA). Glycerol, which was used as a plasticizer, was purchased from Aldrich Co. (Milwaukee, WI, USA). Titanium dioxide ultrapure nanopowder (rutile, mw 79.87) supplied by Sisco Research Laboratories Pvt Ltd.

Equipments used

Electronic Weight Balance: FX400 FCOSET electronic balance (Fig.1) was used for weighing the samples. 5 grams of TiO_2 , 5 grams of whey protein isolate and 1.5% glycerol were weighed. Before using the electronic balance, it should be tare to get zero on the screen then it will ready to give accurate weight. It can measured maximum 410 g weight and $d=0.001\text{g}$. It may be directly connect to the printer to get printed values of weight.

Teflon coated sheet: sheet is used. Teflon coated sheet ($24 \times 30 \text{ cm}^2$) was procured from "Corrosion Care Specialities" Chennai. Teflon coated sheet (Fig. 2) fixed on a plain wooden board or glass. It has a fantastic property that no material is fixed on the surface of the sheet. That's why nano-particle embedded film is easy to peel from it. This was observed that without Teflon coated sheet 50% film gone waste at the time of peeling.

Fig.1: Electronic balance Fig 2: Teflon coated sheet on plain glass

The colour meter: Hunter colorcolorimeter (Color Reader Model CR-10) was used for the measurement of colour of the developed packaging film (Cho *et al.*, 2010). The color was measured by using CIELAB scale at 10° at D_{65} illuminant. It works on the principle of focussing the light and measured energy reflected from the sample across the entire visible spectrum. The three dimensional scale $L^* a^* b^*$ was used. L^* is the lightness coefficient, ranging from 0 (black) to 100 (white), a^* represents greenness and redness (+100 for red and -80 for green). While b^* represents yellowness and blueness (+70 for yellow and -80 for blue), C represents Chroma while H represents Hue angle.

The instrument (Fig. 3) was standardised before placing the sample by placing black tile and white tile provided with the instruments. Once the instruments standardised, it was ready to measure the color. It can also be cross checked by placing the white tile

Fig 3 Colormeter

Fig 4 pH meter

Fig 5: Homogenizer

Homogenizer: A homogenizer (model TH-50, made in India) was used for proper mixing the solution (Fig. 5). There is a system to adjust the height of rotating tool, which can be adjusted according to the size of beaker and height of solution in to the beaker. Each sample was homogenized for 10 min at 1000 rpm for adequate mixing of all ingredients present in the solution.

The drying chamber: A locally available cardboard was used for making drying chamber. 100 watts bulb was fitted and checked the rising the temperature of the chamber by putting the thermometer inside the

which was provided by the $L^* a^*$ and b^* values. The sample was placed in the sample cup. The deviation of the color of the sample to standard was also observed and recorded in the computer interface. The measurement replicated thrice. The color was measured of nanoparticle embedded biopolymer solution as well as end product films.

pH meter: pH meter (Model no pH-600, Milwaukee, made in Portugal) was used for measurement of pH. Its range is to measure is 0.0 to 14. According the design expert 13 levels of pH was measured as 8.5, 7.0, 10.0, 7.0, 8.5, 8.5, 8.5, 8.5, 10.0, 6.2, 10.0, 6.38 and 8.50 for different levels of sonication. For calibration of pH meter, distilled water was taken in a beaker and dip the nob of pH meter into the distilled water and adjust the pH at 7.00 by a screw driver which is given in the same box, then nob dip into solution of biopolymer to measure the pH of solution.

chamber. This chamber was found adequate for winter season when temperature of environment in north India ranges from $5-20^\circ\text{C}$. In case of second chamber in which zero watt (15 watt) bulb was fitted to see the effect of increasing temperature. It was found the few degree temperature was increased. Drying chamber was kept in plane area in which Teflon coated sheet is kept for drying. The specifications of drying chambers are as follows: First Chamber's dimensions (Length = 54cm, Width = 52cm, Breadth = 30 cm and Area = 2808 cm^2) and Second chamber's dimensions'

(Length = 43.5 cm, Width = 32 cm, Breadth =

24.5 cm and Area = 1392 cm²).

Fig. 6: Chamber with 100 W bulb Fig. 7: Chamber with 0 W (15 W) bulb

Development of protein based nano-particle embedded polymeric packaging films

It is reported that the films based on soy protein isolate and whey protein isolate by casting were flexible and hydrophobic and thus depending upon the application desired the processing method can be chosen (Garrido *et al.*, 2013). Hence, for the present study, the films were developed by casting the SPI and WPI films solutions.

Film solution preparation

WPI or WPI/SPI films (Fig. 8) were prepared with fixed level of TiO₂ and fixed amount of glycerol contents, i.e. 5% of titanium dioxide nanopowder and 1.5% glycerol. The control WPI film solution was prepared by dissolving 5 g of WPI in 100 ml distilled water with 1.5 g of glycerol (Kadam *et al.*, 2013). In the preparation of WPI/SPI film solutions, a precisely weighed TiO₂ was first dispersed into distilled water (100 ml) and stirred using a magnetic stirrer overnight to reach complete hydration/swelling. Then 5 g of WPI was added to obtain an aqueous solution, followed by the addition of 1.5 g of glycerol. All the film solutions were heated to 90°C for 15 min in a water bath, cooled to room temperature and degassed using a bath-type ultrasound sonicator (Sonica, ultrasonic cleaner, 2200 MH, SPINCOTECH PVT LTD). The film

forming solutions were then cast onto levelled Teflon-coated glass plates (24 x 30 cm) framed of four sides. The cast films were dried at an ambient temperature (20–25°C, 50±5%RH) for 2 days and then peeled off from the glass plates. The flow chart of nano film preparation is given below:

Fig. 8: Flow chart of nano-film preparation

Results and Discussion

Color of film: Polymer materials, such as films, may be subjected to various kinds of

stress during use. The determination of the mechanical properties involves not only scientific but also technological and practical aspects. The remarkable increase is in the tensile strength at different levels of sonication. From Table 1, it is clearly indicated the L^* values were decreased with increasing the levels of sonication. At 20 % sonication level, the L^* value is 93.83 and at last 100% sonication level it is 85.36, therefore it shows that more sonication darker the color of films but still it is white. Values of a^* ranges from -3.0 to 5.6 and b^* ranges from 2.9 to 4.0. It shows that films were developed are in light color.

Thickness of film: Five measurements of thickness by micrometer were taken on each test specimen. All film samples were conditioned for at least 48 h in a constant-temperature humidity chamber at 25 °C and 50% RH before testing. Triplicate measurements were used to determine each film property. The film was kept average as value 0.031 mm for all samples to measure the different mechanical, rheological properties-

likes' tensile strength, elongation %, WVTR etc. Thin films give better results compared to thick film. When film will be thick its brittleness does increased (Table 1)

Drying chambers: As shown in Table 1, is clearly defined that when temperature is increasing in drying chamber the time of drying reducing respectively. Two chambers were studied for development nano-particle embedded film and found that 60 watts chamber takes 6.583 to 4.233 hrs and 0 watt chamber takes 24.00 to 21.50 hrs for the same ingredient films.

Weight loss due to sonication: According to Table 2, it shows that the level of sonication 20% to 100 % increased the weight loss of solution. When sonication used 20% then 8.924g of water evaporate from the main sample, similarly for 40%, 60%, 80% and 100% of sonication levels then weight loss was found 8.900, 11.924, 12.924 and 13.607 respectively. This water loss takes place because the temperature increased from room temperature to up to 60°C.

Table 1: Effect of sonication on the physical properties of film

Name of film	Sonication level (%)	Average thickness, cm	Color of film	Time of drying, hrs	
				60W, Chamber	0W, Chamber
WPI(7.0 pH)	20	0.030	$L=93.83$ $a = -3.6$ $b = 2.9$	6.583	24.00
WPI(7.0 pH)	40	0.031	$L=93.00$ $a = -3.0$ $b = 3.5$	6.250	23.50
WPI(7.0 pH)	60	0.032	$L=90.52$ $a = -3.2$ $b = 3.8$	5.800	22.50
WPI(7.0 pH)	80	0.302	$L=88.53$ $a = -3.6$ $b = 4.0$	5.250	22.50
WPI(7.0 pH)	100	0.031	$L=85.36$ $a = -5.6$ $b = 3.5$	4.233	21.50

Table 2: Effect of sonication on weight loss of solution and time of drying

Name of film	Sonication level (%)	Initial sample size (g)	Time of drying,hrs		Weight loss of solution after sonication (g)
			60W, Chamber	0W, Chamber	
WPI (7.0 pH)	20	111.50	6.583	24.00	8.924
WPI (7.0 pH)	40	111.50	6.250	23.50	8.900
WPI (7.0 pH)	60	111.50	5.800	22.50	11.924
WPI (7.0 pH)	80	111.50	5.250	22.50	12.924
WPI (7.0 pH)	100	111.50	4.233	21.50	13.607

Fig.8: Comparison of two drying chambers with level of sonication

From Fig. 8, it is tremendously dropped in time taken in case of 60 watt chamber compare with 0 watt chamber. Actually 0 watt chamber shows their effect as room temperature conditions. When sonication levels were increased then drying time is decreased. In the food sector, one of the most important problems is the time-consuming and laborious process of food quality-control analysis. Innovative devices and techniques are being developed that can facilitate the preparation of food samples and their precise and in-

expensive analysis. From this point of view, the development of nano-sensors to detect micro-organisms and contaminants is a particularly promising application of food nanotechnology.

Conclusion

In the scientific literature, an increasing number of publications have reported the production and application of biopolymers with natural and/or biodegradable plasticizers, such as citrate, polyols, triacetate, oligomeric ester-amides and fatty acid derivatives. Nevertheless, a deeper understanding of their interactions and fundamental physicochemical and

biochemical properties is still needed in order to enable the design and production of desirable and competitive materials using compatible plasticizers. Sonication and drying chamber for nano-films have a significant importance.

References

1. Ali, B., Mujeeb, M., Aeri, V., Mir, S.R., Faiyazuddin, M. and Sakeel, F. (2011) Anti-inflammatory and anti-oxidant activity of four *Ficus* species leaves. *Natural Product Research*, 1:1-6.
2. Azad, A.K., Sermsintham, N., Chandkrachang, S. and Stevens, W.F. (2004). Chitosan membrane as a wound-healing dressing characterization and clinical application. *Journal of Biomedical Materials Research Part B-Applied Biomaterials*, 69B (2): 216–222.
3. Azeredo, H.M.C., Mattoso, L.H.C., Wood, D., Williams, T.G., Avena-Bustillos, R.J. and McHugh, T.H. (2009). Nanocomposite edible films from mango puree reinforced with cellulose nanofibers. *Journal of Food Science*, 74(5):31-35.
4. Bourriot, S., Garnier, C. and Doublier, J.L. (1999). Micellar-casein-carrageenan mixtures. I. Phase separation and ultrastructure. *Carbohydrate Polymers*, 40: 145–157.
5. Cho, Y.S., Lee, Y. S. and Rhee, C. (2010). Edible oxygen barrier bilayer film pouches from corn zein and soy protein isolate for olive oil packaging. *LWT – Food Science and Technology*, 43: 1234–1239.
6. Cutter, C.N. (2006). Opportunities for bio-based packaging technologies to improve the quality and safety of fresh and further processed muscle foods. *Meat science*, 74(1):131–142.
7. Farris, S., Schaich, K.M., Liu, L.S., Piergiovanni, L., and Yam, K.L. (2009). Development of polyion-complex hydrogels as an alternative approach for the production of bio-based polymers for food packaging applications: a review. *Trends in Food Science & Technology*, 20(8):316–332.
8. Garrido, J.A., Ros, E. and De Angelo, E. (2013). Spike timing regulation on the millisecond scale by distributed synaptic plasticity at cerebellum input stage: a simulation study. *Computational Neuroscience*, 7:64.
9. Jeschke, M.G., Sandmann, G., Schubert, T., and Klein, D. (2005). Effect of oxidized regenerated cellulose/collagen matrix on dermal and epidermal healing and growth factors in an acute wound. *Wound repair regulation*, 13: 324–331.
10. Kadam, D.M., Thunga, M., Wang, S., Kessler, M.R., Grewell, D., Lamsal, B. and Yu, C. (2013) Preparation and characterization of whey protein isolate films reinforced with porous silica coated titania nanoparticles. *Journal of Food Engineering*, 117(1): 133–140.
11. Krochta, J.M. and De Mulder-Johnston, C. (1997). Edible and biodegradable polymer films challenges and opportunities. *Food Technology*, 51 (12): 61–74.
12. Laine, C., Harlin, A., Hartman, J., Hyvärinen, S., Kammiovirta, K., Krogerus, B., Pajari, H., Rautkoski, H., Setälä, H., Sievänen, J., Uotila, J. and Vähä-Nissi, M. (2013). Hydroxyalkylated xylans – Their synthesis and application in coatings for packaging and paper. *Industrial Crops and Products*, 44: 692–704.
13. Pérez-Gago, M.B. and Krochta, J.M. (2002). Formation and properties of whey protein films and coatings. In Gennadios, A. (Ed.). Protein based films and coatings. (pp. 159–180). Boca Raton, FL: CRC Press LLC.
14. Satyanarayana, K.G., Arizaga, G.G.C. and Wypych, F. (2009). Biodegradable composites based on lignocellulosic fibers—An overview. *Progress in Polymer Science*, 34 (9):982–1021.
15. Silva, S.S., Santos, M.I., Coutinho, O.P., Mano, J.F. and Reis, R.L. (2005). Physical properties and biocompatibility of chitosan/soy blended membranes. *Journal of*

- Materials Science-Materials in Medicine*, 16(6):575–579.
16. Sothornvit, R. and Krochta, J.M. (2000). Plasticizer effect on oxygen permeability of b-lactoglobulin films. *Journal of Agricultural and Food Chemistry*, 48:6298-6302
 17. Tolstoguzov, V. (2000). Phase behaviour of macromolecular components in biological and food systems. *Nahrung-Food*, 44(5): 299–308.