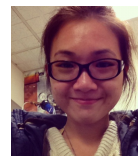


Encapsulation of gallic acid in zein electrospun sub-micron fibers

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INTRODUCTION AND OBJECTIVE

Electrospinning has come forward as an efficient and cost effective technique for producing fibers with ultra-fine diameters. The high surface area to volume ratio yields ultrafine fibers with enhanced properties. Zein as a hydrophobic protein extracted from corn maize is known for its high thermal resistance and great oxygen barrier properties which find plentiful of applications in packaging sector (Shukla and Cheryan, 2001). Gallic acid is a phenolic acid that is widely present in the plant kingdom. Studies have shown that gallic acid and its derivatives are antioxidants that exhibit anti-inflammatory, antimicrobial and also free radicals scavenging abilities (Lu, Nie et al., 2006; Chusinsuan, Chimnoi et al., 2009).

In this study, gallic acid was used as the model functional ingredient to study the physicochemical changes of gallic acid loaded zein fibers using electrospinning as an encapsulation practice. The objective of the present study was to better understand the use of electrospinning as a one-step preparation technique to obtain gallic acid loaded sub-micron structured zein fibers.

MATERIALS AND METHODS

Zein electrospun fibers containing 10 % of gallic acid in the solid fibers was prepared by first dissolving gallic acid in the 80% ethanol aqueous solutions, followed by dissolving zein powder to obtain 25 wt.-% zein solutions. The prepared solution was electrospun at 16 kV with a distance between needle tip and collector of 13 cm and feedrate of 0.8 mL hr⁻¹. The prepared gallic acid-zein fibers were evaluated for various physicochemical characterizations including morphologies, physical states and thermal properties of the electrospun fibers before and after the incorporation of gallic acid using scanning electron microscopy (SEM), X-ray diffraction (XRD) and differential scanning calorimetry (DSC). The radical scavenging properties of gallic acid after electrospinning were determined using 1,1'-diphenyl-2-picrylhydrazyl (DPPH) assay according to method suggested by Paneva et al. (2011).

RESULTS AND DISCUSSION

Figure 1 shows the SEM image of a 10% gallic acid loaded zein electrospun fibers. Distribution of fiber diameters calculated from SEM images indicated an evenly distributed diameter with an average of 343 nm after the incorporation of gallic acid (Figure 2). It was

found that the average fiber diameter increased from 290 to 343 nm as compared to the neat zein fibers. The obtained fibers were smooth and had no evidence of particles separating out from the fiber matrix as observed from the SEM image.

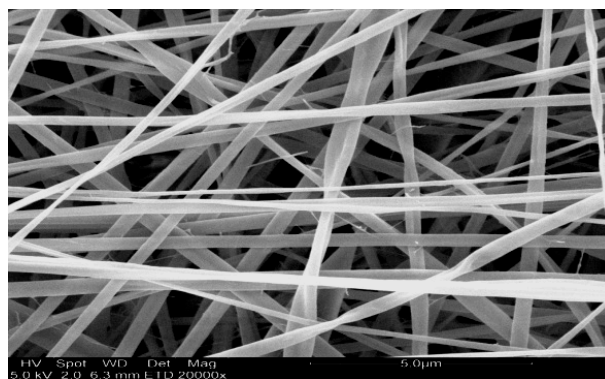


Figure 1 : SEM image of 10% gallic acid loaded zein electrospun fibers

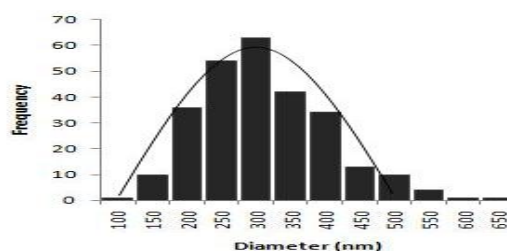


Figure 2 : The fiber diameter distribution of 10% gallic acid loaded zein electrospun fibers

Both DSC and XRD were used to provide information about the physical status of gallic acid in the zein sub-micron fibers. Glass transition temperature (T_g) of the zein fibers was at 156°C. The gallic acid-loaded zein fibers had demonstrated a T_g value lower than that of neat zein fibers at 137°C. The reduction of T_g can be due to the plasticizing effect of the incorporated gallic acid that increased the mobility of zein molecular chains (Xie et al., 2010).

There was a loss of the signature diffraction peaks of gallic acid in the XRD pattern of gallic acid loaded zein electrospun fibers (Figure 3). This indicated that the physical status of the gallic acid had been modified in the electrospun fibers.

Both XRD and DSC results had demonstrated interactions between gallic acid and zein and the formation of a new sub-micron structured component with the existence of gallic acid.

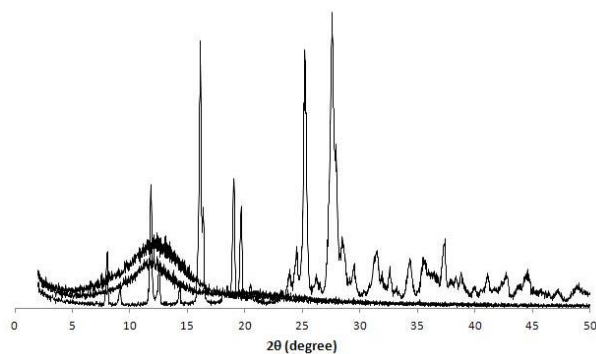


Figure 3 : XRD patterns of neat zein fibers (25 wt.-%); 10% gallic acid loaded zein electrospun fibers and gallic acid powder

Figure 4 illustrates the volume of sample solutions needed to reduce the intensity of DPPH absorption by 50%. It has been found that the gallic acid-loaded zein electrospun fibers required a smaller volume of solutions in order to reduce the intensity of DPPH by 50% as compared to the neat zein fiber solution. The neat zein fiber solution did not exhibit significant DPPH scavenging abilities as shown in Figure 4a.

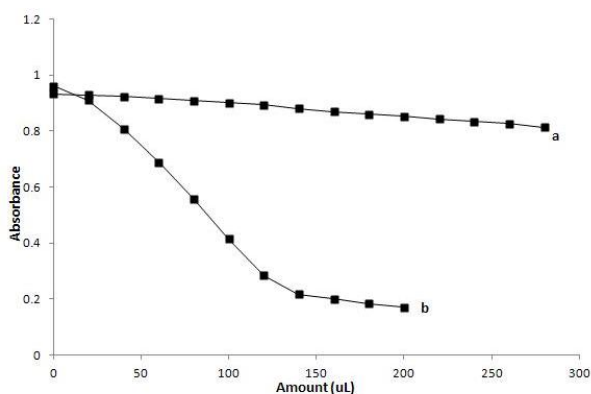


Figure 4 :The reduction of DPPH absorbance as a function of volume required from the electrospun fibers in ethanol. (a) neat zein fibers (25 wt.-%); (b) 10% gallic acid loaded zein electrospun fibers, respectively.

The results implied that gallic acid loaded zein electrospun fibers had exhibited antioxidant activities higher than the neat zein fibers. The loaded gallic acid retained its antioxidant activities even after the electrospinning process.

CONCLUSIONS

In this present study, gallic acid was successfully incorporated into zein sub-micron fibers through electrospinning for the first time. As a conclusion, electrospinning can be used as an effective tool for the production of polymeric system containing functional components. The fabricated sub-micron structured complex provides insight for novel encapsulation

technique which have great potential in food industry especially for their utilization as packaging or highly functional materials.

REFERENCES

- Chuysinuan, P., Chimnoi, N., Techasakul, S., & Supaphol, P. (2009). *Gallic Acid-Loaded Electrospun Poly(L-Lactic Acid) Fiber Mats and their Release Characteristic*. *Macromolecular Chemistry and Physics* 210(10): 814-822.
- Lu, Z., Nie, G., Belton, P. S., Tang, H., & Zhao, B. (2006). *Structure-activity relationship analysis of antioxidant ability and neuroprotective effect of gallic acid derivatives*. *Neurochemistry International* 48(4): 263-274.
- Shukla, R. and M. Cheryan (2001). *Zein: the industrial protein from corn*. *Industrial Crops and Products* 13(3): 171-192.
- Paneva, D., Manolova, N., Argirova, M., & Rashkov, I. (2011). *Antibacterial electrospun poly(ϵ -caprolactone)/ascorbyl palmitate nanofibrous materials*. *International Journal of Pharmaceutics*, 416(1), 346-355.
- Xie, C., Li, X., Luo, X., Yang, Y., Cui, W., Zou, J., & Zhou, S. (2010). *Release modulation and cytotoxicity of hydroxycamptothecin-loaded electrospun fibers with 2-hydroxypropyl- β -cyclodextrin inoculations*. *International Journal of Pharmaceutics*, 391(1-2), 55-64.

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