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PROF. DR. AHMET NİYAZİ ÖZKER

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CHARACTERIZATION OF ZEIN-BASED NANOFIBERS CONTAINING METAL NANOPARTICLES FOR INTELLIGENT PACKAGING FUNCTIONS

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ABSTRACT

In this study, zein based nanofibers doped with tin dioxide and various amounts of silver and/or gold nanoparticles, were developed by uniaxial and coaxial electrospinning and characterized. For this purpose, zein (30% w/v) and tin dioxide (10% w/v) containing solutions were prepared with 70/30 ethanol/liquid gold nanoparticles, 70/20/10 ethanol/liquid gold nanoparticle/dimethlyfurfural, 70/15/15 ethanol/liquid gold/liquid silver nanoparticle. To increase the gold amount in nanofibers, solutions were also electrospinned by coaxial system with and without dried gold nanoparticles. Scanning electron microscopy (SEM) results showed the formation of ultrafine nanostructures and differences between nanofiber samples were evaluated. Elemental composition of metals identified and Energy Dispersive X-Ray (EDX) mapping results confirmed gold/tin dioxide presence in nanofibers. Since TMA concentration is evaluated as critical indicator to reveal the freshness of seafood products, TMA aqueous solution exposed to square cutted nanofibers in petri dishes. Diameter sizes of Sample 1 were increased after exposed to trimethlyamine solution and heavier fragile film structure structure observed. X-Ray Diffraction (XRD) analysis were performed to compare crystalline structure information after exposure. Results revealed that structural changes in nanofibers can be tested with XRD and SEM results and gas binding functions could be used for intelligent packaging. Therefore, nanofibers loaded with metal nanoparticles may be used as an indicator for sensing seafood product quality for intelligent packaging purposes in the future.

Keywords: Electrospinning, Intelligent Packaging, Metal Oxide, Nanoparticle, Trimethlyamine

INTRODUCTION

High quantities of trimethylamine oxide, a tiny, odorless, colorless amine oxide produced by gut microbial metabolism, can be found in the tissue of aquatic items. Trimethylamine oxide is an essential substrate for bacteria's anaerobic metabolism and the source of dimethylamine and trimethylamine in seafood. By using enzymes, certain spoiling organisms convert trimethylamine oxide to trimethylamine. Trimethylamine is one of the main metabolites that cause spoiling and is created when bacteria grow in fish muscle (Cetinkaya, 2022). Ammonia is also produced together with trimethylamine during the anaerobic reduction of trimethylamine oxide by *Shewanella putrefaciens* (Heising et al, 2012).

Conventional techniques are based on chemical methods can be complicated, slow, and expensive. Smart packaging systems can overcome some of these disadvantages (Chen et al, 2017; Gaudin, 2017). In a smart packaging system, a material is added to the packaging that interacts with the packaging environment and monitors the condition (storage time, temperature, shelf life, etc.) of the packaged food products. So, the consumer is informed about the spoilage condition of the food product (Forghani et al, 2021). A fish freshness indicator can be considered as part of smart packaging, that uses metabolites as "information" to monitor the status of fish freshness. These systems are portable, require small sample, selective response towards various analytes, are highly sensitive, and have good specificity over the traditional methods (Chen et al, 2017; Gaudin, 2017).

Electrospinning has been used as a unique way to produce intelligent packaging solutions in recent years. When fiber's diameter reaches nanometer size, surface area increases which lead nanofiber surface functionalization with nanoparticles. These properties make nanofibers potential candidates for intelligent packaging applications. Moroever, the high surface area may enhance the reactivity of the material and may speed up adsorption. So, the possibility to fill this nanomaterial with gas or volatile and biochemical species could lead to new approaches (Mercante et al, 2017). Metal nanoparticles such as gold and silver have also revolutionized the field of the sensor systems. Further research is necessary to determine whether functionalized metal nanoparticles can be used as an active probe for seafood products. Although the conventional techniques are suitable for the detection of some volatiles, expensive cost and long analysis time limit their wide scope application. Therefore, nanoparticles can be used to detection of target analytes with sensor functions. So, gas sensing performance of the zein based nanofibers incorporated with gold/ silver nanoparticles for intelligent packaging functions investigated in the current study.

METHODOLOGY

Material

Zein from maize, molecular weight (Mw) ≃22–24 kDa, was purchased from Sigma-Aldrich Inc. (Saint Louis, Missouri, USA). Dimethlyfurfural received from Merck. Technical ethanol (96%) was used for solution preperation. Gold (III) chloride hydrate ($HAuCl_4$) purchased from Sigma. Silver nitrate ($AgNO_3$) was purchased from Merck. Distilled water was used throughout all experiments. Trimethlyamine aquaous solution was purchased from Merck.

Method

Nanoparticle production

Firstly, silver/gold nanoparticles in liquid and dried gold nanoparticles were produced from silver nitrate $AgNO_3$ and gold salt ($HAuCl_4$) respectively. Fabrication methodologies were modified from studies reported by Cetinkaya (2022) and Rajar et al. (2021). Liquid nanoparticles were produced by using sodium borohydride and polyvinylpyrrolidone. Solutions were stirred until the yellow (silver nanoparticle) and pink (gold nanoparticle) color appear. To obtain dried gold nanoparticles, liquid nanoparticles were poured to glass petri

dishes, placed in a water bath, and heated until only powders left. Dried gold nanoparticles were collected by scraping glass petri dishes with spatula.

Ethanol-liquid gold nanoparticle (70/20 v/v) solution was used for dissolving zein at concentration of 30 g/100 ml and tin dioxide at concentration of 10 g/100 ml. 70/20/10 ethanol/liquid gold nanoparticle/dimethlyfurfural, 70/15/15 ethanol/liquid gold nanoparticle/liquid silver nanoparticle solutions were also prepared with same 3:1 zein: tin dioxide concentration. To increase the gold amount in nanofibers, solutions were also electrospinned by coaxial system. Furthermore 6.13% dried gold nanoparticles were added to the same solution for further electrospinning.

Electrospinning process

The electrospinning process was conducted by using Inovenso equipment with a pump unit (New Era Pump Systems, Inc., USA). Solutions were electrospinned at different feed rates with an applied voltage. The distance between the aluminum foil covered on collector and needle was 15 cm. Second pump used for the coaxial electrospinning.

Trimethlyamine exposion

Square cutted nanofibers exposed trimethlyamine solution to find the gas response. 2 mL trimethlyamine aqueous solution was poured into the petri dishes.

Scanning Electron Microscopy

Morphology of silver or gold nanoparticle loaded zein nanofibers was examined using Ultra-high-resolution Field Emission Scanning Electron Microscope (FE-SEM, Hitachi SU7000) under low vacuum at 2.5-10 KX magnification with a working distance of 6.4 mm. An accelerating voltage of 10 kV was determined for obtaining secondary electron images. Energy Dispersive Spectroscopy detector (Oxford Instruments) Octane Elect and APEX™ software used for elemental mapping.

X-RAY Diffraction

The crystal phase of the samples before and after exposed to trimethlyamine was obtained by Rigaku D/Max X-Ray Diffractometer -2200 PC equipment at 30 mA and 40 kV. The XRD pattern was scanned 10°-80° 2θ, for 2 C/min.

CONCLUSION AND DISCUSSION

SEM image of 3:1 zein: tin dioxide nanofibers produced from feed solution that contains: 70/30 ethanol/liquid gold nanoparticles presented in Figure 1. As can be seen clearly from image, there was not gold aggregates on the surface of fibers which indicates the well dispersion and distribution of the loaded gold nanoparticles.

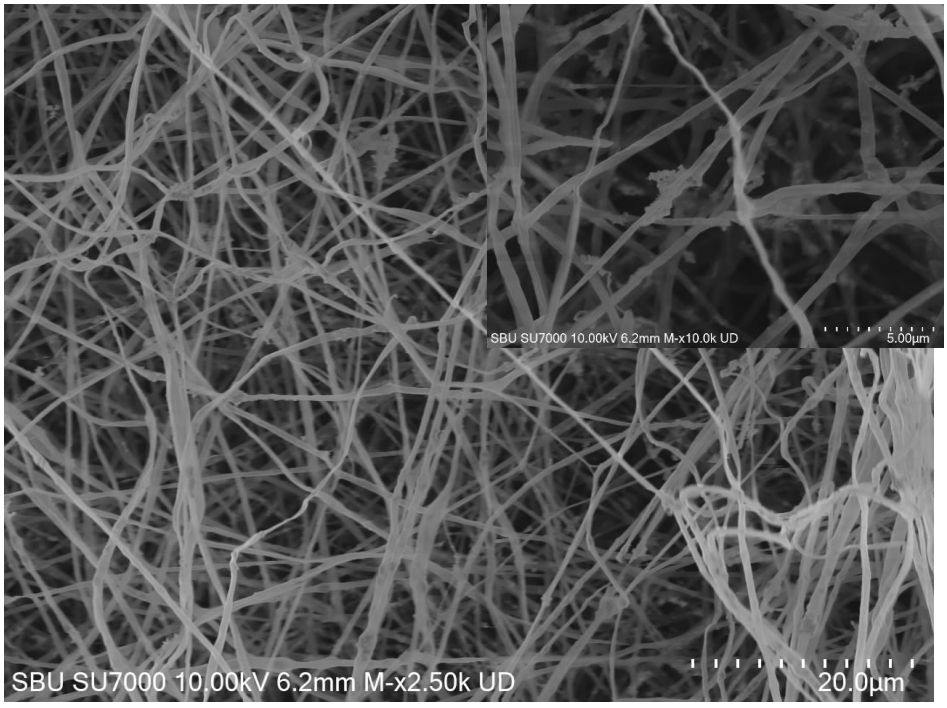


Figure 1. Electrospun 3:1 zein:tin dioxide based nanofibers. Upper right inset : Higher magnification.

Morphological changes were observed after the trimethlyamine exposion for two minutes. As shown in the Figure 2 nanofibers formed thicker network structure and their sizes increased becasuse of the absorption of trimethlyamine. Furthermore it was obserbed that material had harder structure after TMA exposure.

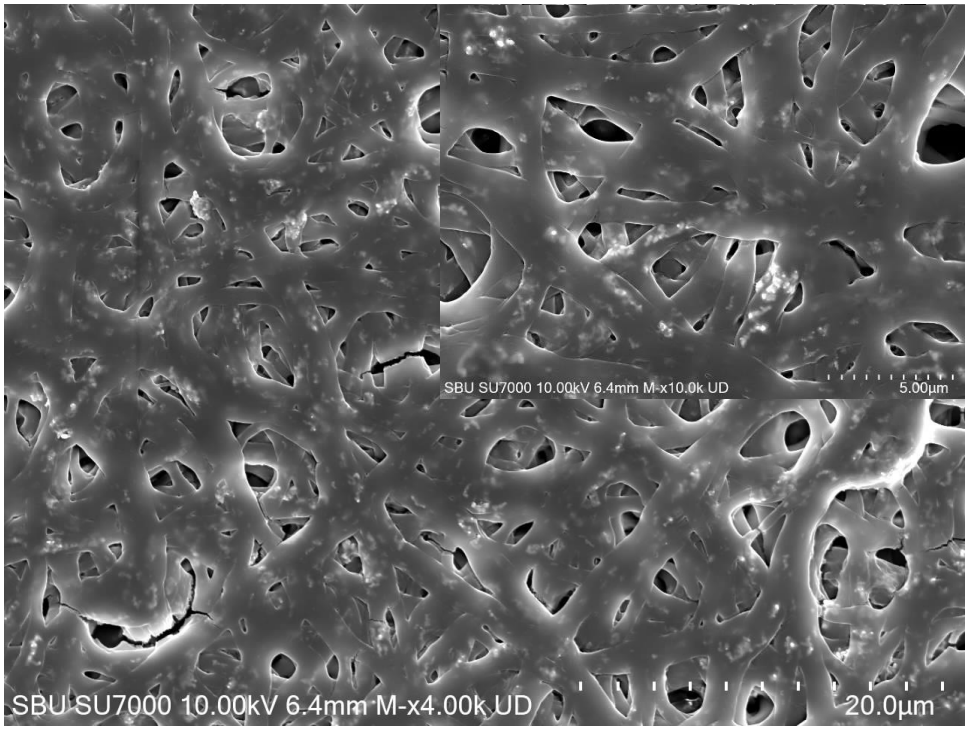


Figure 2. Electrospun 3:1 zein:tin dioxide based nanofibers after exposed to trimethlyamine. Upper right inset : Higher magnification.

XRD results indicated the state of polymer matrix and crystalline materials. Chemical bond interactions with trimethlyamine decreased the intensity of the peaks, reducing the crystallinity of nanofiber. Furthermore as can be seen from graph some of the tin dioxide peaks were dissappeared (Figure 3).

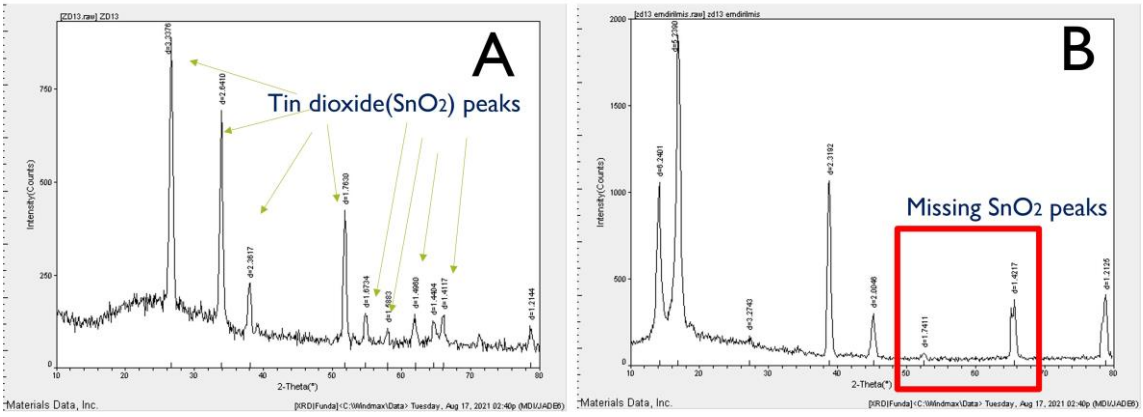


Figure 3. Electrospun 3:1 zein:tin dioxide based nanofibers before A) and after B) exposed to trimethlyamine.

SEM image of 3:1 zein:tin dioxide nanofibers produced from feed solution that contains: 70/20/10 ethanol/liquid gold nanoparticles/dimethlyfurfural presented in Figure 4. With a closer look to inset image small spots on nanofibers were observed. These spots could be attirubuted to gold nanoparticles. Furthermore, inhomogeneous agglomerates and clusterings were observed. These structures could be the reason of tin dioxide integration on nanofibers.

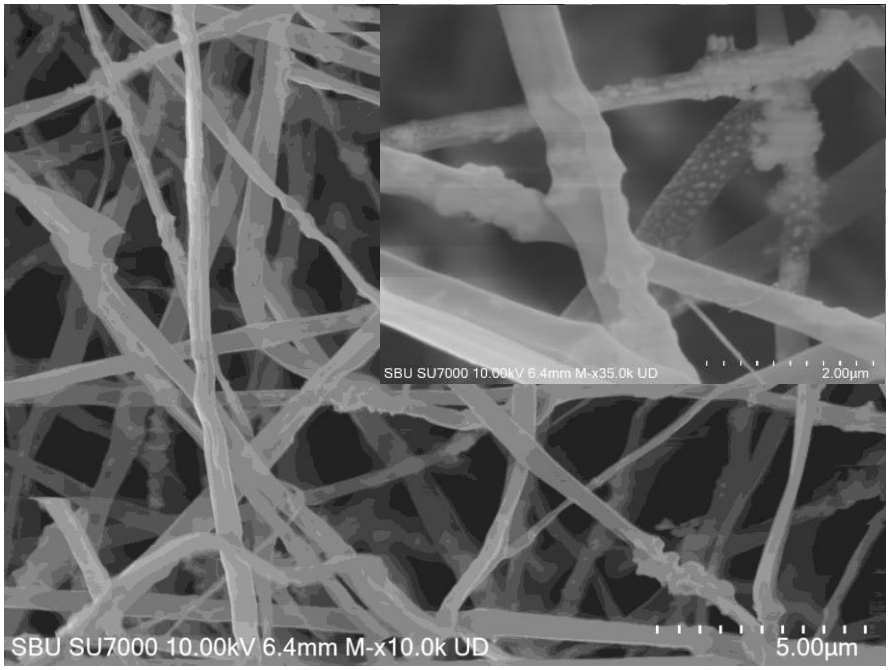


Figure 4. Electrospun 3:1 zein:tin dioxide based nanofibers prepared with 70/20/10 ethanol/dimethyfurfural solution. Upper right inset : Higher magnification.

Similar structure was observed when gold and silver nanoparticles combined at the ratio of 1:1. (Figure 5).

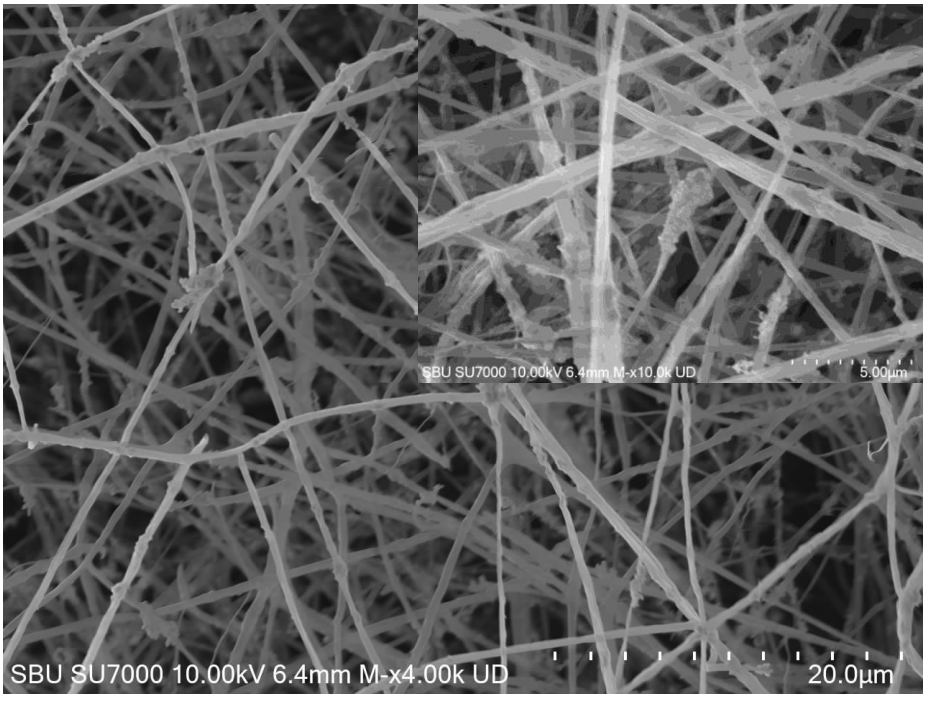


Figure 5. Electrospun 3:1 zein:tin dioxide based nanofibers prepared with 70/15/15 ethanol/liquid gold /liquid silver nanoparticle solution. Upper right inset: Higher magnification.

To investigate the morphological difference between uniaxial and coaxial electrospinning, a second pump was used. Therefore, in coaixal electrospinning process, there are two

concentric capillaries to eject solutions. The feed rate was set as 1.2ml/h for the zein solution, and feed rate was set to 0.8 ml/h for liquid gold (Figure 6).

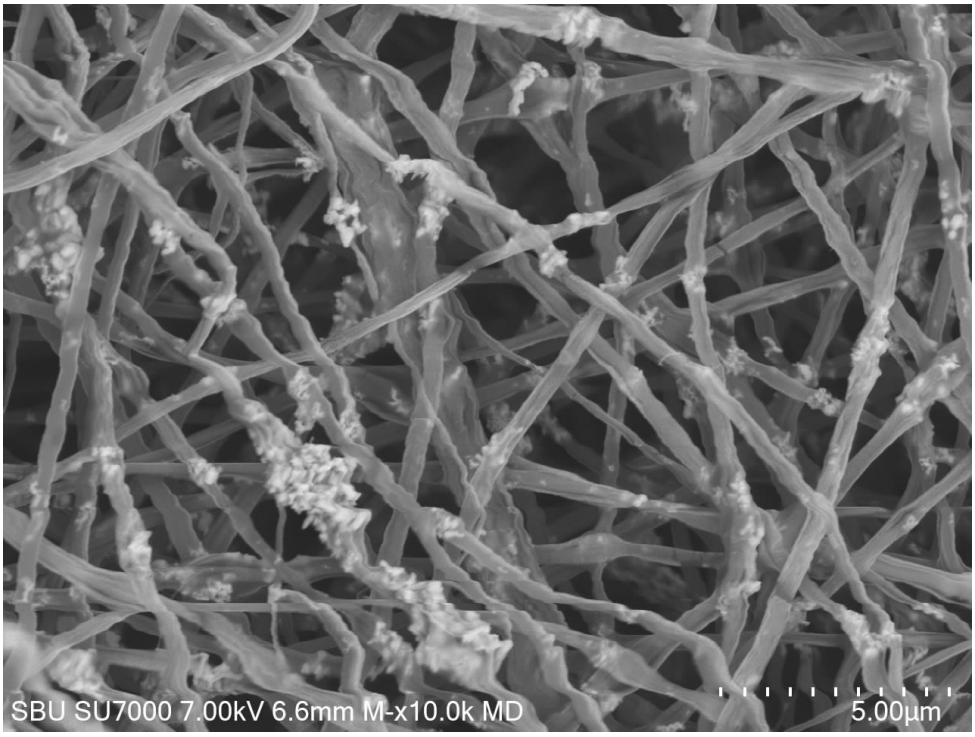


Figure 6. Coaxial electrospun 3:1 zein:tin dioxide nanofiber prepared with 70/30 ethanol/liquid gold nanoparticle solution (1.2 ml/h) and liquid gold nanoparticle (0.8 ml/h).

EDX elemental analysis was performed to obtain information about the metals in nanofiber sample. According to results nanofiber sample consists of three main elements: carbon oxygen, and tin. Carbon is the major component since zein has a carbon–carbon linkages. Results indicated that important constituents of gold and tin dioxide attached to the nanofiber. Location of C, O, Sn atoms determined by EDX color mapping to confirm their attachment.

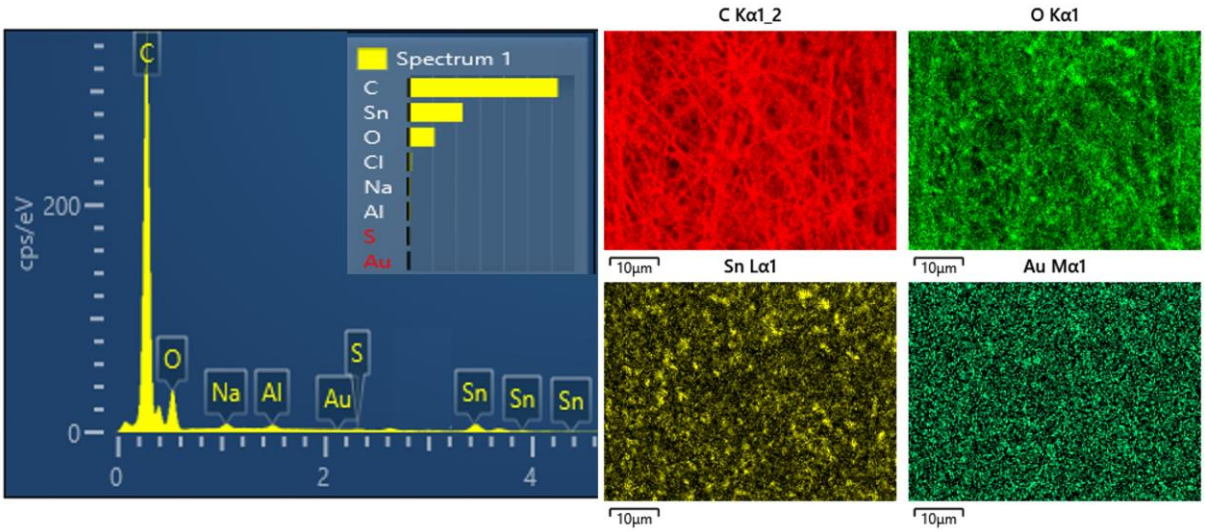


Figure 7. EDX mapping of coaxial electrospun 3:1 zein:tin dioxide based nanofiber prepared with 70/30 ethanol/liquid gold nanoparticle solution (1.2 ml/h) and liquid gold nanoparticle (0.8 ml/h).

Coaxial electrospun dried gold nanoparticle containing zein based nanofibers prepared with 70/30 ethanol/liquid gold nanoparticle solution (1.2 ml/h) and liquid gold nanoparticle (0.3 ml/h). Zein, tindioxide and dried gold nanoparticle concentrations were 30%, 10%, and 6.1% respectively. According to SEM images (Figure 8), many small dots observed with the addition of dried gold nanoparticles and there were more agglomerates when the voltage increased to 14 kV.

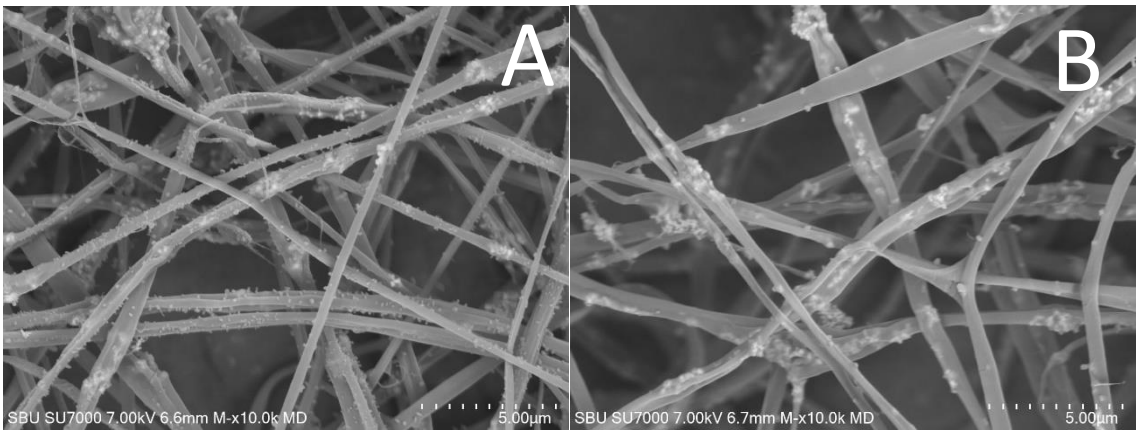


Figure 8. Coaxial electrospun dried gold nanoparticle containing zein based nanofibers at A) 13 kV voltage B) 14 kV voltage.

In this study, novel nanofibers for smart packaging functions produced at various concentrations to obtain optimal one. Structure and integrity of the nanofibers were affected after tin dioxide and dried gold nanoparticle integration. SEM images indicated that tin dioxide and dried gold nanoparticles were deposited on the surface. After exposion to trimethlyamine probably because of the interfacial hydrogen bonding, structure and crystallinity was changed. Addition of dried gold nanoparticle probably decreased the conductivity, which could produce thicker diameter fibers owing to decreasing electrostatic force and jet elongation. The results of the present work suggest that zein based nanofibers incorporated with tin dioxide and gold/ silver nanoparticles can be utilized for smart food packaging systems.

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