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Micro/Nanoparticle Production to Improve the Performance of Industrial Adhesives

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In this study, sodium alginate micro/nanoparticle production containing our X-700 patented adhesive was carried out by electrohydrodynamic atomization technique (EHDA). Thus, the possibility of bonding industrial adhesives with the particles obtained will be increased. Morphological and mechanical properties of the obtained EHDA encapsulated products were determined by using field emission gun scanning electron microscopy (FEGSEM) and TENSILE test procedures. With these particles with a large surface area, the surface area to be bonded will expand and a more effective coating will be provided.

Keywords: *Industrial adhesive, X-700 patented adhesive, sodium alginate, micro/nanoparticle, EHDA*

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1. Introduction

The concept of adhesive is any material or substance that holds or attracts various materials to form a mechanical, chemical, adhesive whole by adhesive or cohesive force. Many products with chemical and herbal characteristics are currently used as adhesives. In its basic meaning, it is to connect two objects from their forehead surfaces. In this attachment, the long-term condition is essential. Some adhesives can be separated from their interfaces in a certain period of time when they are adhered to the forehead surfaces. For this reason, adhesives are a serious industrialbased issue of great importance.

In recent years, new types of polymeric experiments in industrial adhesives have attracted attention. In polymeric-based adhesives, good resistance, water repellency and sometimes flexibility and sometimes tightness can be demonstrated. Micro/nanoparticle production for industrial adhesives with the EHDA system was provided within the scope of our work. EHDA system basically consists of syringe pump, magnetic stirrer and high voltage power supply [1-12].

With this system, X-700 patented adhesive and SA encapsulated micro/nanoparticle production was achieved in order to improve the adhesion and bonding capacity of industrial adhesives.

2. Material and Method

2.1. Material

In this study, our X-700 patented industrial adhesive was used. Sodium alginate (SA) (Sigma-Aldrich, Germany) and pure water (distilled water) were preferred. Curcumin active ingredient is supplied ready-made isolated from

turmeric. SA crosslinker calcium chloride ($CaCl_2$) (Sigma-Aldrich, Germany) was used. Span 20, Tween 20 and Tween 80 (Sigma-Aldrich, Germany) were used as surfactants in the production of regular and uniform nanoparticles.

2.2. Method

***Preparation of feed solutions before electrospraying**

In our study, solutions were prepared according to the values in Table 2.1. before micro/nanoparticle production. The prepared solutions were filtered through whatman paper and the insoluble large particles were removed from the solution and made ready for micro/nanoparticle production [8-20]. The surfactant ratios to be added to the solutions to be prepared before electrospraying are shown in Table 2.1.

Table 2.1. *Surfactant ratios to be added to the solutions to be prepared before electrospraying*

Polymer Solution (ml)	**Polymer Solvent (ml)**	**Surfactant Ratio (v/v)**	**Mixture Time (min)**	**Mixture Temperature (ºC)**	**Mixture Speed (rpm)**
2% Sodium Alginate (SA)	100% Distile Water	-	60	45	600
2% Sodium Alginate (SA)	100% Distile Water	1% Tween 80-0.5 %Tween 20-0.5% Span 60	60	45	600
2% Sodium Alginate (SA)1% X-700 Patented Adhesive	100% Distile Water	-	60	45	600
2% Sodium Alginate (SA)1% X-700 Patented Adhesive	100% Distile Water	1% Tween 80-0.5 %Tween 20-0.5% Span 60	60	45	600

*** Production of encapsulated micro/nanoparticle with electrohydrodynamic atomization technique**

The solutions prepared according to the values in Table 2.1. were taken into beakers, respectively, and used in the electrospraying process. The polymer solutions to be used in the electrospraying process were drawn into a 10 ml syringe and placed in the syringe pump. Cross-linkers in Table 2.2. were dissolved in pure water and taken into a beaker. Particle production was achieved by applying the values in Table 1.2. A 20 gauge steel needle tip, where the anode part of the high voltage power supply will be located, is preferred. The cathode part is connected to a stainless steel ring that will provide conductivity. By providing a working distance of 12 cm between the syringe needle and the stainless ring, the working parameters in Table 2.2. were realized and micro/nanoparticle production was obtained. The operating parameters in Table 2.2. have been applied and the electrospraying process has been carried out in a heating magnetic stirrer system. The mixing time was 60-65 min, the speed was 600 rpm and the temperature was 45-50 ºC for all samples [13-23]. The operating parameters of the electrospraying process are given in Table 2.2.

Table 2.2. *Electrospray process operating parameters*

Polymer Solution (ml)	**Polymer Solvent (ml)**	**Surfactant Ratio (v/v)**	**Crosslinker Ratio (v/v)**	**Crosslinker Syringe Pump Feed Rate (ml/hour)**	**High Voltage Rating (kV)**	**Distance Between Syringe Needle and Stainless Ring (cm)**
2% Sodium Alginate (SA)	100 % Distile Water	-	1% $CaCl_2$	3.0	25.0	12
2% Sodium Alginate (SA)	100 % Distile Water	1% Tween 800.5 %Tween 20-0.5% Span 60	1% $CaCl_2$	3.0	25.0	12
2% Sodium Alginate (SA) -1% X700 Patented Adhesive	100 % Distile Water	-	1% $CaCl_2$	3.0	25.0	12
2% Sodium Alginate (SA) -1% X700 Patented Adhesive	100 % Distile Water	1% Tween 800.5 %Tween 20-0.5% Span 60	1% $CaCl_2$	3.0	25.0	12

The products produced as a result of the electrospraying process are taken into centrifuge tubes and stored at 5,000 rpm for 30 minutes. time centrifuged. At the end of the process, half of the centrifuge tube was emptied into a container, pure water was added and centrifuged at 5,000 rpm for 30 minutes. This procedure was applied three times. After the last process, half of the tubes were discharged into a container, the caps of the centrifuge tubes were removed and closed with parafilm, and small holes were opened on the parafilm. After the procedure, it was kept in a refrigerator at -80 ºC for two days. After the waiting period, it was taken to the lyophilizer device and the drying process was carried out. Micro/nano particles were obtained as a result of the lyophilizer process. The polymer/crosslinker ratio was set to 6/1. Thus, it was desired to produce monodisperse particles of equal size. Micro/Nanoparticles were stored at -18 °C until use [10-24]. In Figure 2.1., the micro/nanoparticle production stages of our SA-X-700 patented product are shown.

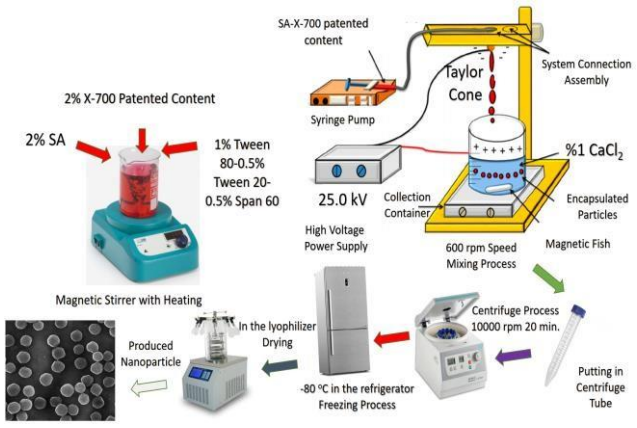

Figure 2.1. *SA-X-700 Micro/nanoparticle production steps of our patented product*

***Characterization studies**

Before the morphological FEGSEM analysis, the samples were coated with gold-palladium. In this way, the FEGSEM image was taken. Particle spacing was determined by taking the arithmetic averages of 40 selected particles.

The tensile test process was performed by applying the mechanical properties of the wooden bow tie on the sample with a tensile test device in accordance with the ASTM standard. The mechanical properties were determined with the arithmetic mean by repeating the samples three times.

3. Result and Discussion

Morphological (FEGSEM) analysis results

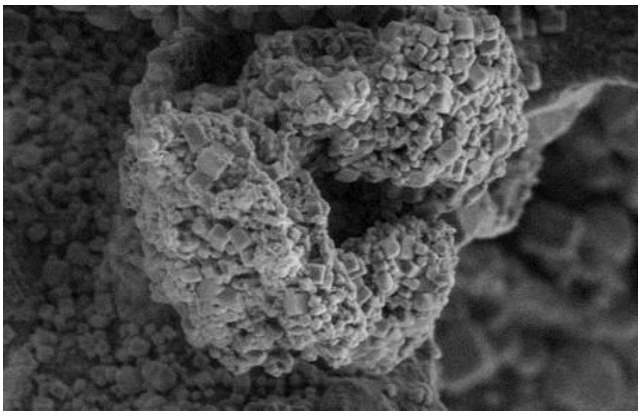

Figure 3.1. *SA micro/nanoparticle FEGSEM image*

SA micro/nanoparticle FEGSEM image is shown in Figure 3.1. Morphological image of the micro/nanoparticle FEGSEM produced by the EHDA system cross-linked with 1% $CaCl_2$ of the solution obtained as a result of the mixture of 2% Sodium Alginate (SA)-100% Pure Water.

SA particles were produced with the EHDA system and production was achieved by optimizing two different parameters. Considering the surfactant and electric field strength value, improvements were made in nanoparticle sizes and morphologies. While there was a particle range of 900-1800 micrometers, a nanoparticle diameter distribution of 400-780 nm was obtained as a result of parameter optimization [18-26].

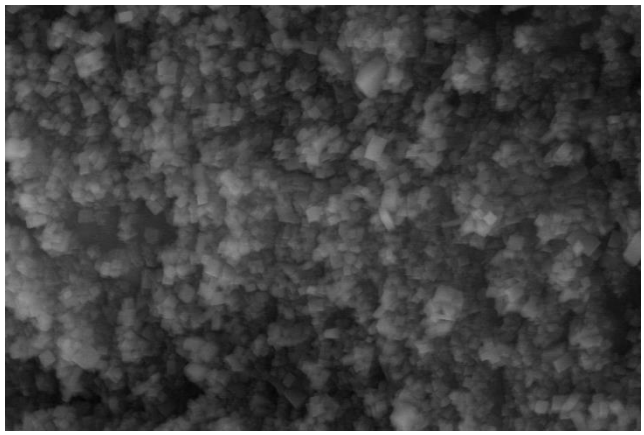

Figure 3.2. *SA-X700 Patented adhesive micro/nanoparticle FEGSEM image*

2% Sodium Alginate (SA)-1% X700 Patented adhesive-100 Distile Water-1% Tween 80-0.5% Tween 20-0.5% Span 60 mixture of the solution obtained with 1% $CaCl_2$ cross-linked micro/nanoparticle FEGSEM produced by EHDA system its morphological image is given in Figure 3.2. It was determined by FEGSEM analysis that there were particles with a size in the range of 300-500 nm [8-17].

Mechanical (TENSILE) analysis results

Before the tensile test process, micro/nanoparticle-free adhesive was applied to one wood surface and micro/nanoparticle adhesive containing our X-700 patented adhesive was applied to the other wood and allowed to dry. The expected bowtie specimens were drawn three-center to the ASTM standard and mechanical properties were determined. While the X-700 patented adhesiveencapsulated wood sample showed 65 MPa mechanical properties, the X-700 patented adhesive-encapsulated wood sample showed 125 MPa mechanical properties. The reason for this situation is that it shows a homogeneous dispersion with the increase of the surface area.

4. Conclusion

Based on the results of the study, micro/nanoparticle production was successfully achieved with the EHDA technique. Compared to SA particles, the particle size of SAs containing X-700 patented adhesive is smaller. In addition, the impact performance increased as the surface area increased. This was supported by the results of the tensile test we conducted. The tensile test process was carried out in accordance with ASTM standards, and significant differences were revealed in the mechanical properties of EHDA unencapsulated and unencapsulated

adhesives. The main reason for this situation is that the EHDA encapsulated particles increase the surface area and create a synergistic effect due to the material interplay. The adhesion performance of industrial adhesives will be increased with the new generation adhesives containing particles.

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