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Production of pumice-containing nanofibers by electrospinning technique

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The scope of the study involves identifying the optimal means to effectively use the electrospinning technique to obtain pumice-containing nanofibers. Nanofiber containing pumice in a solution was electrospun to obtain smooth, cylindrical, bead-free, and ultrafine nanomaterials.

The study also analyzed the molecular [Fourier transform infrared spectroscopy (FTIR)], thermal [differential scanning calorimetry (DSC)], zeta potential, size, polydispersity index [dynamic light scattering (DLS)], and surface [scanning electron microscope (SEM)] parameters of

the pumice-containing nanofibers having JP6 (applied voltage: 6 kV) and JP12 (12 kV) properties. While the distance (10 cm), flow rate (0.8 mL/h), and other parameters of the electrospinning process were fixed, two different voltages were applied to obtain the pumice-containing nanofiber. The average diameter of the nanoencapsulated pumice produced at 6 kV was defined as 98.6 nm in gelatin nanomats with 31.8 nm. The average diameter of the nanocapsule pumice produced under a 12 kV voltage was found to be 85.8 nm, and the average diameter of the nanomats (non–nanoencapsulated) was 35.2 nm. The average zeta potential values of the pumice-containing nanofiber were also determined in the nanosize range. The JP6 and JP12 PDI values were determined as 0.165 and 0.566, respectively. Peaks characteristic of pumices as defined in the literature were observed in the FTIR results, while DSC analysis results revealed strong endo- and exothermic peaks.As a result of this study, it has been proved that pumice can be reduced to nanosize with the electrospinning technique and it is nanoencapsulated in nanofiber. When the obtained pumice-containing nanofiber was examined, it was determined that the surface area of the nanofiber was large and resistant to thermal heat.

Keywords: *nanotechnology, nanofiber, pumice, characterization, fabrication parameters, electrospinning*

1. Introduction

The use of different materials is able to offer the promise of enhancement of multifunctional materials, which can contribute to obtaining lighter, safer, and smarter vehicles, including vehicles important for commercial freight applications, such as aircraft and ships. In addition to the industries mentioned above, material science could play a role in the fabrication of food preservation and food packaging materials. Today, novel methods that provide alternative production techniques in the industry are gaining increasing importance. On the other hand, besides widely used materials in order to fabricate materials, novel raw materials could be integrated with the material science systems.

Pumice is an industrial raw material with high SiO_2 content geochemically, physically, abundantly porous, light, and high strength. Due to these properties, pumice has been used in many industrial applications; it is used widely in the construction, textile, agriculture, and chemical industries, and a great many other industries present a good opportunity for the involvement of pumice usage [1]. For example, pumice is used instead of sand in lightweight concrete production. Compared to normal concrete, pumice is preferred due to its lightness, ease of transportation, and thermal insulation. Since pumice saves energy in houses, it is of great importance, especially for foreigndependent countries, which use natural gas fuel for heating [2]. Also, pumice is defined as a cheap rock of volcanic origin, formed mostly by the combination of Al_2O_3 and SiO_2 at different ratios. Pumice can be effectively found in various regions of the world and is used as a raw material in a wide variety of fields, including as an adsorbent in environmental

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treatments [3, 4]. It has been observed that owing to its porous structure and the pores it contains, pumice can provide good sound and heat insulation. As an illustration, we may mention that for food packaging materials, the important issues are the barrier properties to gases, water vapor, light, odor, heat, and sealing with heat [5]. Moreover, thermal behavior or degradation of obtained material can be much more significant for material processing. In this respect, Ceylan et al. [6] reported that a reduction in mass (17%) of different materials such as liquid smoke, chitosan, and thymol was detected at temperatures below 147°C, which started clearly perceptible thermal decomposition for the mentioned materials. At this point, the content of materials such as polyethylene oxide, glutaraldehyde, and zeolitic imidazolate can play a key role in identifying the thermal behavior or degradation of the obtained materials. Additionally, the applied technology can enhance the properties of these materials.

In this sense, nanotechnology applications have invited a great deal of attention. Nanoscale materials such as nanofibers [7], nanomats [8], nanoparticles [9], nanotubes [10], and nanoclays [11] could be produced and integrated into a material as value additions. The use of nanomaterials could provide a larger surface-area-to-volume ratio of the material as compared to the micro-sized materials. In this respect, pumice in the nanoscale application has been preferred because of the potential development of packaging materials for further studies based on nanoscale pumice.

The main aim of this study is to produce pumice-containing nanomaterial using the electrospinning technique. Additionally, to catalog the properties of electrospun nanoencapsulated pumice synthesis, a study was undertaken into its molecular characterization, morphological properties, thermal degradation, zeta size (ZS), and polydispersity indexes (PDI). The present study analyses and practically demonstrates the potential use of nanoencapsulated pumice as a novel material for the industry.

2. Materials and Methods

2.1. Materials

Gelatin powder EMPROVE® exp (Catalog No. 1.04078.1000) was received from Merck KGaA (Darmstadt, Germany). Acetic acid was purchased from EMBOY Chemicals & Trade Co. Ltd (Istanbul, Turkey). Distilled water was used throughout all experiments. Whatman™ filter paper (in the 8-μm particle size range) was used for filtering the pumice. Raw pumice samples were obtained from Van, Turkey. For obtaining nanofibers, a Fytronix ESP-900 electrospinning system (Elazığ, Turkey), consisting of a high voltage unit, syringe pump unit, and a flat collector, was used.

2.2. Methods

2.2.1. Pretreatment and electrospinning process

Ten grams of pumice and 14.4 mL of acetic acid were stirred for 2 h and then filtered. Two milliliters of the filtrate was taken, and 0.8 g of gelatin and 2 mL of distilled water were added. Then, it was solved at 40°C at 500 rpm. During the production, 50% acetic acid filtrate, 20% gelatin, and 50% pure water were used. The solution was transferred to a 5-mL syringe and was subjected to the electrospinning process, the variations of which are given below. The electrospinning parameters were defined for a flow rate of 0.72 mL/h, a distance between the collector and Taylor cone of 10 cm, applied voltages (6 kV and 12 kV), and a 17G needle.

2.2.2. Morphological characterization

The nanostructure morphologies of electrospun JP (gelatin and pumice-based nanomaterial) samples were determined by scanning electron microscopy (SEM) with different magnifications (50,000×; 100,000×; 200,000×). Electrospun JP (gelatin and pumice-based) nanostructures were examined under a low vacuum in a field emission scanning electron microscope (FEI, Quanta Feg 250, USA) with a working distance of 8 mm. An accelerating voltage of 5 kV was determined to be ideal for obtaining secondary electron images. The average diameters of nanofibers and nanocap-

sules were obtained using different measurements for each nanostructure, separately ($n = 50$).

2.2.3. Molecular characterization by attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR) spectroscopy

The chemical structures of JP nanomaterials were revealed using (ATR-FTIR). A Bruker Tensor 27 spectrometer (Bremen, Germany) equipped with a DLaTGS detector and a KBr beam splitter was utilized to save the spectrum. A diamond ATR cell was utilized in an ATR accessory. The designed experiment was controlled and saved using OPUS software (version 7.2) for Windows. The ATR-FTIR spectra of JP6 and JP12 nanomaterials were saved with a resolution of 2 cm^{-1}, accumulating 16 scans per spectra. The spectra of JP6 and JP12 were recorded from 4,000 to 600 wavenumber/cm. All spectra were subtracted against the background air spectrum. After every scan, a new reference related to the air background spectrum was obtained.

2.2.4. ZS and PDI analysis

PDI and ZS values of JP6 and JP12 nanosamples were obtained using Nano ZSP (Malvern Instruments Corp., Worcestershire, UK). The measurements were applied in phosphate buffer solutions by a disposable folded capillary cell containing electrodes. The cell was placed in a temperature-controlled holder at 25°C. The PDI and ZS values of nanosamples were measured three times as described by Ceylan [12].

2.2.5. Differential scanning calorimetry (DSC)

The thermal behavior of pumice-containing nanomaterials named JP6 and JP12 was assessed by the use of a DSC (Q100, TA Instruments, Inc., New Castle, DE, USA). At a rate of 10°C/min, 10 mg samples were sealed in a hermetic aluminum pan and then heated to 500°C. At a flow rate of 20 mL/min, nitrogen was used as the transfer gas, and also, an empty aluminum pan was used as a reference.

2.2.6. Statistical analysis

The obtained data were subjected to analysis of variance (ANOVA) to determine the average diameters of nanoencapsulated pumice and nanomats. Minitab software was applied to reveal significant differences between the two groups by ANOVA. While a significant ($p < 0.05$) main effect was obtained, the average values of the samples were further analyzed by Duncan's multiple range comparison tests.

3. Results and Discussion

3.1. Electrospinning for Pumice

The electrospinning technique is a process utilized to obtain ultrathin materials with different diameters [13]. To achieve this aim, different kinds of materials have been used in recently published studies. Besides the use of zein, bioactive compounds, poly(vinyl alcohol), probiotic bacteria, and poly(caprolactone) in electrospinning techniques [7, 9, 14–17], different kinds of elements such as silver and copper are used to fabricate nanostructures [17, 18]. In the industry, the obtaining of novel materials is gaining importance, and in this respect, alternative production techniques with new materials, providing a larger contact area on the surface of the materials, have become the object of immense attention. In the present study, preprocesses supported with the electrospinning technique provided the means for obtaining a nanoscale material. Therefore, the results indicated below reflect that the electrospinning techniques, together with potential preprocessing conditions, could be evaluated as a promising technique for other potential geological-based materials.

3.2. Morphological Characterization

SEM images of the electrospun nanomats are given in Figure 1 (SEM images of JP6 nanomaterials), Figure 2 (SEM images of JP12 nanomaterials), Figure 3 (SEM images of pumice before electrospinning), and Figure 4 (EDX-SEM of electrospun pumice nanomaterials).

According to the images, the average diameters of the nanomaterials indicate the efficiency of the electrospinning process. In the present study, the average diameter of nanoencapsulated pumice fabricated at 6 kV (JP6) was defined as 98.6 ± 92 nm

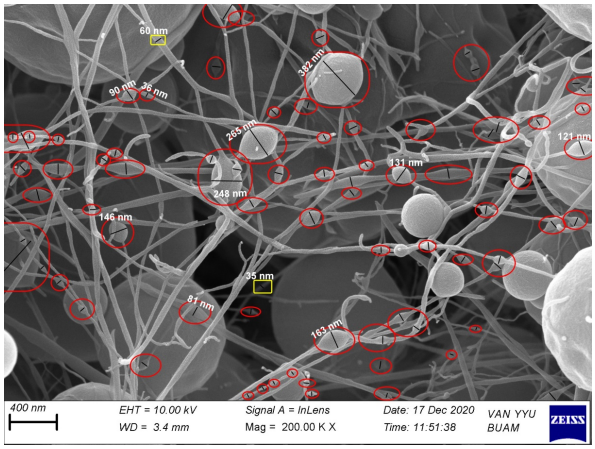


Fig. 1. SEM image of JP6 nanomaterials. SEM, scanning electron microscope

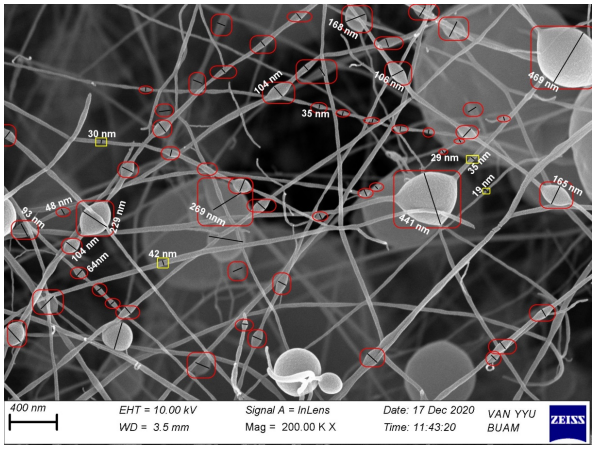


Fig. 2. SEM image of JP12 nanomaterials. SEM, scanning electron microscope

within gelatin nanomats (GN6) having 31.8 ± 9 nm ($p < 0.05$). In this nanoprocess, the minimum and maximum values of NP6 were found to be 25.4 nm and 482.5 nm, while those of GN6 were determined as 9.0 nm and 50.6 nm, respectively. Among nanotechnology studies available from the literature, Ceylan et al. [7] defined that the average diameter of probiotic bacteria-loaded nanostructures was 381.1 ± 131 nm. On the other hand, the average diameter of non–nanoencapsulated materials was lower in the same study. As can be seen from the previous studies forming part of the literature, the use of different materials in nanoscience can affect the morphological structures. However,

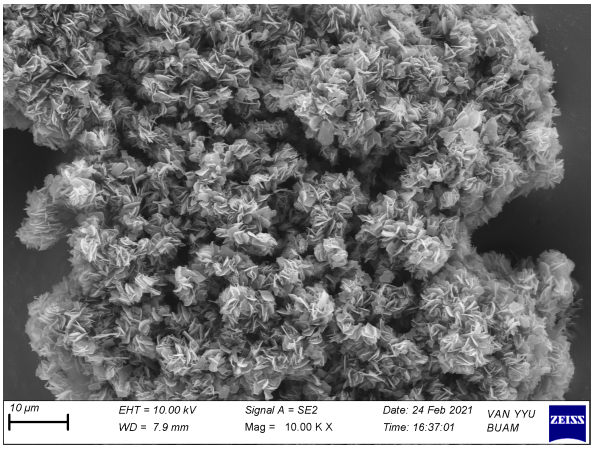


Fig. 3. SEM image of pumice before electrospinning. SEM, scanning electron microscope

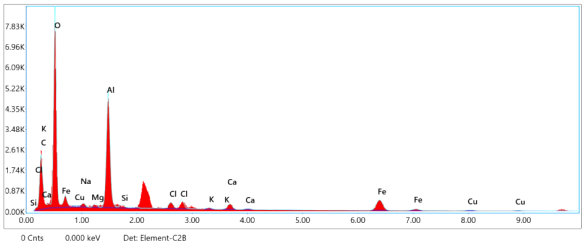


Fig. 4. *Scanning Electron Microscopy* (*SEM*) with Energy Dispersive X-Ray Analysis (*EDX*) of electrospun pumice nanomaterials

loading of different materials to the nanostructures increased the average diameters of the obtained nanostructures. Ceylan et al. [19] reported that *Lactobacillus rhamnosus*-loaded poly (vinyl) alcohol and sodium alginate-based nanofibers (LR) and also poly(vinyl) alcohol and sodium alginate-based ranging within 60.1 nm $<$ diameter of nanofibers $<$ 522.1 nm could be produced. Meral et al. [8] revealed that nisin and curcumin-loaded nanomats having 172 nm average diameters were obtained. In the present study, electrospun nanomaterials obtained at 6 kV had 31.8 nm $\leq x \leq$ 98.6 nm as average diameters. These obtained nanomaterials including pumice might provide a larger contact area on the surface of the potentially used materials.

Electrospinning parameters may directly affect the properties of the nanomaterials. For example, the average diameters of the nanoencapsulated

pumice (JP12) were 85.8, while the average diameter of the nanomats (non–nanoencapsulated: GN12) was found to be 35.2 nm ($p < 0.05$). Also, when the supplied voltage was increased (12 kV), the average diameter of nanoencapsulated materials decreased (from 98.6 nm to 85.8 nm), while that of nanomats increased (from 31.8 nm to 35.2 nm). Minimum and maximum values of the nanoencapsulated materials obtained from pumice were decreased as well. As could be seen from the values, these changes in NP12 were in the range of 2.68% and 13.21%. On the other hand, the changes in minimum and maximum values excluding pumice (GN12) were 75.96% and 36.30%, respectively. It is revealed that nanoencapsulated materials, especially, possessed a higher average diameter stability as compared to the nanomats obtained using the electrospinning technique. In the production of nanoencapsulated pumice, the electrospinning parameters and the applied voltage can affect the diameter of electrospun nanomaterials. As stated by Ojha et al. [20], the electrospinning parameters, including distance, voltage, and flow rate, can play a role in obtaining electrospun nanomaterials. The nanomats, including nanoencapsulated pumice, which could be used in the production of various materials, were successfully developed by electrospinning technique using two different applied voltages.

3.3. ZS and PDI

The maximum ZS of JP6 and JP12 were found as 237.2 nm and 1,038.6 nm, respectively (Figure 5). The small size obtained in the present study can provide resistance to gravitational separation, flocculation, and coalescence. Also, the PDI values of the JP6 and JP12 nanomaterials were determined to be 0.165 and 0.566. In case of PDI, the obtained values were found to be higher than 0.2, which defined a higher heterogeneity as compared to the samples with lower diameters. The obtained result supported this knowledge because of the fact that the maximum size was increased when the applied voltage was increased. In this respect, as could be seen from the results of the present study, depending on the increase of applied voltage and PDI values, the ZS of nanostructures obtained from the electrospinning technique was increased. According to the results of the study as described by Meral et al. [8], a relationship between ZS and PDI values of the nanomaterials was detected. The present study reveals that nanomats with lower diameters produced from pumice were affected by the applied voltage. Ceylan [12] reported that the ZS of chitosan nanoparticles integrated in poly(vinyl alcohol) nanofibers were <600 nm. Also, a previous study noted that homogenization could decrease droplet size, using a sonicator equipped with a probe [21]. Moreover, the initial material properties such as diameter and the type of the used material in nanoscience, combination processes, homogenization, and electrospinning could affect the fabricated nanomaterials' ZS and PDI in the present study.

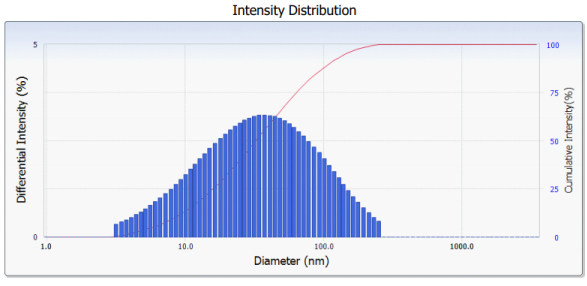


Fig. 5. Diameter distribution of JP nanomaterials

3.4. Molecular Characterization

FTIR can be used to monitor changes in the chemical structure of electrospun nanomaterials. In addition, it is defined as a useful tool to efficiently and quickly characterize the encapsulated molecules [6]. In this respect, Figure 6 indicates the infrared spectra of JPN. In the nanomaterial obtained from pumice, the peak values related to Si–O and Al–O were represented between 600 cm^{-1} and 720 cm^{-1}. The strong peak at wavenumbers between 3,280 cm^{-1} and 3,320 cm^{-1} reflected hydroxyl groups that could be defined based on their moisture content. In the present FTIR spectra, peaks about the 1,450 cm^{-1} band presented O–C–O anti-asymmetric stretching vibration related to sodium and carbonate forms, while 1,040–

860 cm^{-1} belonged to the symmetric stretching vibration of Si–O–Si as well. Al–OH stretching was determined at around 3,600 cm^{-1}, as asymmetric and symmetric stretching vibrations of C–H bonds at 2,920 cm^{-1} were revealed. In the literature, according to Ersoy et al. [22], the peaks at 400 cm^{-1} and 800 cm^{-1} of the pumice samples might have been associated with the Si–O, while the strong peak at around 1,100 cm^{-1} was related to Si–O stretching vibrations. Also, as stated by Prajaputra et al. [23], the peaks at ~720, 1,000, 1,650, and 3,475 can be described as the bending vibration of the Si–O–Si bond. Similar peaks [$(SiO_4)^{-2}$ group: Si–O–Si, H–O, H–O–H, etc.) related to pumice in the present study were also revealed in the literature [3, 24]. The present study results revealed that electrospun pumice synthesis was successfully obtained in the nanomaterials, as shown in Figure 6 using SEM images and zeta values. There was no significant difference between FTIR JP6 and JP12. Hence, the results of JP12 are presented here.

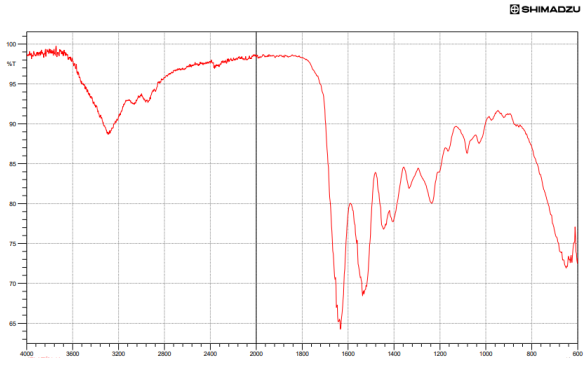


Fig. 6. FTIR of JP nanomaterial. FTIR, Fourier transform infrared spectroscopy

3.5. Thermal Characterization

DSC thermograms of nanomaterial (JP) are given in Figure 7. There was no remarkable difference between the JPN samples depending on the applied voltages to obtain nanomaterials from pumice. Also, DSC analysis was applied to reveal the alterations in the physical properties of both JP6 and JP12. Endothermic processes, such as the evaporation levels of both JP samples, were determined at around 100°C. The melting point in JP was obtained between 200°C and 300°C. This result may be observed due to the interfacial interaction between the gelatin chair and pumice filler nanostructures. A study related to pumice particles in the thermal, electrical, and mechanical properties of poly(vinyl alcohol)/poly(vinyl pyrrolidone) composites reported that glass transition temperature was observed at around 104°C and 100°C. Also, when the amount of pumice was increased in the given combination, rapid changes were observed from exothermic to endothermic [25]. On the other hand, adding pumice powder did not significantly affect melting temperature as stated by Sahin et al. [26]. In the present nanoformulation, significant differences in DSC results were not detected in JP6 and JP12 nanosamples depending on the increase in applied voltage.

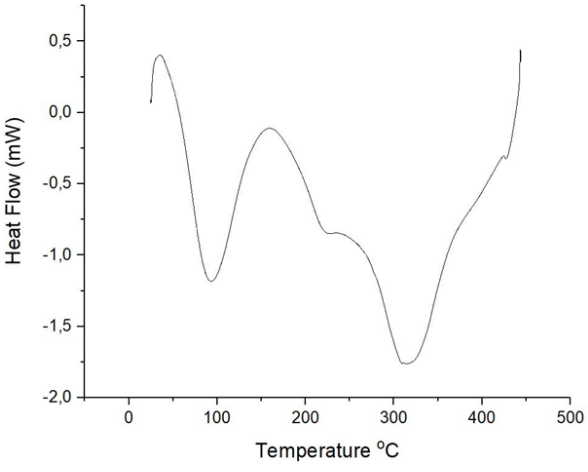


Fig. 7. Thermal behavior (DSC) of JNP. DSC, differential scanning calorimetry

4. Conclusion

Pumice in nanoformulation could be successfully obtained using the electrospinning technique. The use of two different high voltages played a key role in obtaining pumice-containing nanomats. A voltage of 6 kV during the electrospinning process was more effective for obtaining homogenous nanomaterials. SEM images and other characterization analyses resulted in the most successful production. Both applied voltages presented <100 nm

diameter nanostructure. Also, melting point in JP was obtained between 200°C and 300°C.

The results of the study demonstrate that pumice can be reduced to nanosize with the electrospinning technique, and that it can be nanoencapsulated in nanofiber. When the obtained pumice-containing nanofiber was examined, it was determined that the surface area of the nanofiber was large and resistant to thermal heat, and it was seen that the pumice preserved the properties that it had at the beginning. When all these properties are evaluated, the results revealed that pumice-containing nanofiber can be effectively used in the present nanoformulation in various industrial applications ranging from packaging material to various special usages.

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