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Research Article

Polylactic Acid-Activated Coal Electrospun Mat Production and Characterization

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ABSTRACT

Polylactic acid (PLA) is used as a potential polymer for consumer products and biomedical applications. With the increasing environmental and sustainability concerns associated with traditional petrochemical-based polymers, PLA applications continue to increase day by day. Activated carbon (AC) is a substance obtained by carbonization of softwood parts such as linden and willow and is used as an antidote in industrial applications as it absorbs the toxin and prevents the absorption of toxins from the small intestine into the blood. Due to its adhesion to surfaces, it is toxin absorbent. The biggest problem for AC is the gradual filling of the adsorption surfaces of the AC by gaseous pollutants over time. In this study, PLA composite nanofibers reinforced with AC were obtained by electrospinning technique. The material properties of the produced composite membranes were determined by performing structural (FTIR, Fourier-Transform Infrared Spectroscopy), morphological (FEGSEM, Field Emission Gun Scanning Electron Microscopy), mechanical (Tensile Test) and biological (Cell Test) characterization studies. With the obtained biocomposites, it will be the ideal filtration material with a longer life and large surface area that can be used in health sector applications.

Keywords: *Polylactic acid, Activated carbon, Electrospinning, Clean air filtration*

Polilaktik Asit-Aktif Kömür Elektrospun Mat Üretimi ve Karakterizasyonu

ÖZ

Polilaktik asit (PLA), tüketici ürünleri ve biyomedikal uygulamalar için potansiyel bir polimer olarak kullanılır. Geleneksel petrokimya bazlı polimerlerle ilişkili artan çevresel ve sürdürülebilirlik endişeleri ile PLA uygulamaları her geçen gün artmaya devam ediyor. Aktif karbon (AK), ıhlamur ve söğüt gibi yumuşak ağaç kısımlarının karbonizasyonu ile elde edilen bir maddedir ve toksini emdiği ve toksinlerin ince bağırsaktan kana emilimini engellediği için endüstriyel uygulamalarda panzehir olarak kullanılır. Yüzeylere yapışması nedeniyle toksin emicidir. Klima için en büyük sorun, klimanın adsorpsiyon yüzeylerinin zamanla gaz kirleticiler tarafından kademeli olarak doldurulmasıdır. Bu çalışmada, elektroeğirme tekniği ile AK ile güçlendirilmiş PLA kompozit nanolifler elde edilmiştir. Üretilen kompozit membranların malzeme özellikleri, yapısal (FTIR, Fourier-Dönüşümlü Kızılötesi Spektroskopi), morfolojik (FEGSEM, Alan Emisyon Tabancası Taramalı Elektron

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Mikroskobu), mekanik (Çekme Testi) ve biyolojik (Hücre Testi) karakterizasyon çalışmaları yapılarak belirlendi. Elde edilen biyokompozitler ile sağlık sektörü uygulamalarında kullanılabilecek daha uzun ömürlü ve geniş yüzey alanına sahip ideal filtrasyon malzemesi olacaktır.

Anahtar Kelimeler: *Polilaktik asit, Aktif karbon, Elektroeğirme, Temiz hava filtrasyonu*

I. INTRODUCTION

The excessive consumption of fossil fuels and the accidents caused by consecutive oil spills seriously affect today's ecosystem. It has been determined that it causes a series of water and air pollution problems, especially oily wastewater. It is desirable to treat oily wastewater from functional materials with high adsorption capacity. Inorganic absorbents are divided into three as natural fiber absorbents and synthetic organic absorbents. A large number of adsorbents were widely used in the ecosystem and brought with them various disadvantages. There are some disadvantageous situations that still need to be overcome, such as secondary environmental pollution from complex production processes and post-use waste [1].

Filtration or filtration is defined as any of various mechanical, physical or biological processes that separate solids from liquids (liquids or gases) by adding a medium through which only liquid can pass. It is also called the filtrate of the passing liquid. In physical filters, solids in large liquids, particles in biological filters are retained, swallowed, metabolites are retained and removed. The filtration system takes place in nature and engineering systems; It has biological, geological and industrial forms. For example, in animals (including humans), renal physiology filtration removes waste from the blood, while in water purification and wastewater treatment, unwanted components are removed by absorbing them into a biological film grown on or on the filter media, as in slow sand. There are different filtration systems, cold and hot. Particulate removal processes are tedious and the filter pores fill up and cannot filter adequately [2]. Electrospinning system, which is a nanotechnological method, and new generation filter system studies with polymer additives for this problem were determined by literature research. In the electrospinning system, nanofiber membranes are obtained from polymer solutions with the help of electric field, and these membranes are a new generation, more efficient, inexpensive, effective and functional system that can easily reach filtration efficiency up to 99.999% [3]. When the filtration system studies were examined, it was determined that polylactic acid (PLA) based polymeric filter systems were also used. Today, activated carbon (AC) has a wide range of use in filter systems and in the health sector.

Polylactic acid (PLA) is an environmentally friendly thermoplastic polymer. Aliphatic polyesters such as PLA are a biocompatible polymer with mechanical properties, transparency and non-toxic properties. With these properties, it is used in consumer products such as packaging, automobile, furniture, filtration, food, textile and pharmaceutical sector [4]. The chemical formula of PLA ($C_3H_4O_2$) is shown as n. The chemical structure of PLA is shown in Figure 1.

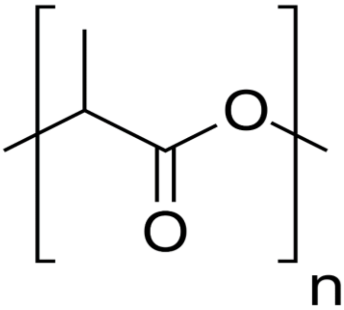


Figure 1. *Chemical structure of PLA [4]*

As a consumer product manufactured using PLA; clothes, utensils and food packages can be counted. PLA used in the medical sector; It can also be used in diapers, feminine hygiene products, medical sutures, stents and pharmaceutical applications. According to Ray et al. [5] studied a series of

PLA/organoclay based biocomposites and found that the PLA biocomposite was completely degraded and destroyed within two weeks. Kakroudi et al. [6] determined the gas permeability properties of PLA films using the microfibrillation process. According to Dasan et al. [7] improved the oxygen barrier properties of PLA/Poly (3-hydroxybutyric acid-co-3-hydroxyvaleric acid) (PHBV) polymer films prepared using nanocrystalline cellulose.

ACs can separate Volatile organic compounds (VOCs), odors and other gaseous pollutants from the air. They perform this cleaning differently than HEPA filters or other air cleaning filters that capture only particles from the air. Filters do this by trapping gas molecules in coal beds. They are the most commonly used filters to capture gases. They are generally used to filter volatile organic compounds released into the external environment. It is also preferred to remove odors in the air such as the smell of cigarette smoke. However, they cannot remove particles such as mold, dust and pollen from the air. Filters are carbons that have undergone some additional processing to better filter gas molecules. First, a lattice structure of small pores is created by the hot air supplied to the carbon, and then carbon dioxide (CO_2) or steam is injected to greatly increase the surface area. This provides more space for gas molecules to trap and makes the carbon more effective as a filter area. AC's were used in World War I to filter certain deadly gases used in gas masks against enemy troops, but this filter system was only effective against certain toxins. After World War II, the use of the AC increased, eventually contributing to the development of modern air and water filters. The adsorption process allows it to filter or capture organic chemicals (gases) in the air. The biggest problem with the air conditioner bed is that over time gaseous pollutants slowly fill the adsorption surfaces of the AC. When the bed is saturated, the filter can no longer hold the contaminants. AC the working system of the filters is given in Figure 2 [8, 9].

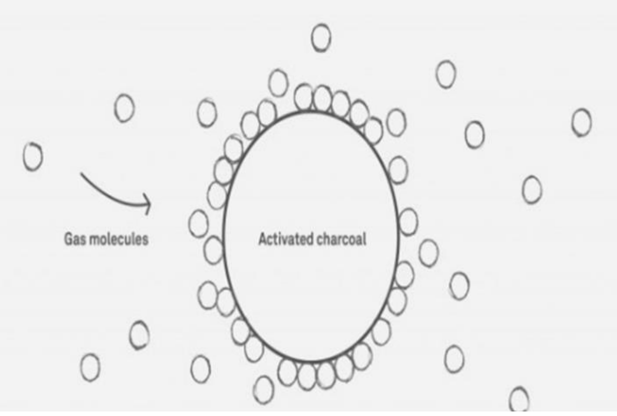


Figure 2. *AC filters working principle [8,9].*

Electrospinning system is a nanofiber production technique from polymer solutions with the help of electric field. It basically consists of syringe pump, high voltage power supply and collector system. In the electrospinning system, additives enlarge the surface area by wrapping around the polymer fibers, and fiber production consisting of tightly packed fibers is carried out. The non-existent material properties of the polymer can be enhanced by the use of additives and electrospinning systems. In addition to the fibers, the pore size between the fibers directly affects the material properties. As the pore ratio increases, the strength values of the material decrease [10]. There are many studies in the literature on clean air filtration from PLA and AC composite nanofiber membranes using electrospinning technique. The filtration efficiency was measured by producing composite membranes with fresh air filtration over clean air filtration [11-14]. Nanofiber membranes produced by electrospinning method have high specific surface area and porous structure. Along with good adsorption properties, their thin size makes it difficult to remove adsorbents after use. Membranes obtained by the use of electrospinning technique are recognized as a versatile and effective method for making absorbent materials with high surface to volume ratio and large porosity, which can significantly improve adsorption performance [15].

Wang et al. [1] investigated that a large amount of oily wastewater is generated from various sources with the rapid industrial and urbanization development and that these wastewaters affect human health mutagenic and carcinogenic and seriously damage the ecosystem. PLA/ Hybrid nanoparticles (ZIF-8@C600) nanofibers were prepared by electrospinning technique for oil adsorption. They determined that the addition of ZIF-8@C600 could adjust the diameter, porosity, pore size and hydrophobicity of the composite electrospun nanofibers. PLA/ZIF-8@C600 porous membranes were found to have good oil absorption properties in oil-water mixture and emulsion. They found that PLA/ZIF-8@C600 fibers are good candidates as oil adsorbents. The adsorption kinetics of the membranes on paraffin liquid were found to be compatible with pseudo-second-order kinetics, and the addition of hybrid ZIF-8@C600 increased the hydrophobic properties and oil adsorption capacity of PLA/ZIF-8@C600 porous membranes. They suggested that composite membranes based on PLA could prevent secondary pollution, since the PLA they used in their study is an environmentally friendly polymer. According to Nugraha et al. [16] investigated the change of wettability behavior of electrospun PLA fiber by electrospinning technique by adding tungsten oxide (WO_3)/amino-functionalized carbon quantum dots (N-CQD) to the polymer matrix. The modified PLA/WO_3/N-CQDs fibers showed significantly improved hydrophobicity of the fibers while maintaining the surface super-oleophilicity. These behaviors were obtained by varying the surface contact angle and surface morphology of the composite PLA fiber using Tungsten trioxide (WO_3). After obtaining a composite membrane by using WO_3 /N-CQDs as filler, PLA/WO_3 /N-CQDs EDA fiber has the highest water contact angle of 132.37º for n-hexane and an oil absorption capacity of 35,752 g/g. It exhibits oil separation performance at 8,326,048 L m^{-2} h^{-1}. They provided decolorization of methylene blue (MB) at 11,961,364 L m^{-2} h^{-1} and 91.80% for n-hexane and n-heptane. PLA/WO_3/N-CQDs electrospun fibers provided excellent separation performance and durability after ten cyclic separation performance tests in their studies. With their work, they contribute to a potential application in the field of oily wastewater treatment. According to Piloni et al. [17] produced Spirulina biochar from rapid pyrolysis and obtained it as an alternative to commercial activated carbon for lactic acid (LA) purification from fermentation broth. They tested activated Spirulina biochars by rapid pyrolysis to thermally treat the biochar in an nitrogen (N_2) atmosphere at 350 and 400 °C for 4 hours and chemically impregnate the algae material with potassium hydroxide (KOH) solution to obtain the biochar. Two purification methodologies were evaluated: filtration and mixing. The mixing method was found to be simpler, faster and more disintegrating with excellent purification results. All evaluated biochars performed comparable to activated carbon in the mixing methodology. Spirulina biochars and KOH activated biochar had the best results, with LA recovery of 92% and 82% and protein removal efficiency of 82% and 90%, respectively. He proposes biochars from Spirulina as an alternative material for purification of lactic acid from the fermentation broth. According to Sattar et al. [18] highlighted that there is great interest in the preparation of porous materials using biodegradable materials and environmental pollution control. A non-toxic, user-friendly and inexpensive adsorbent bead was prepared using a phase inversion technique from PLA and AC. The porous structure of PLA/AC. They were characterized by bead scanning electron microscopy and nitrogen adsorption-desorption methods. A kinetic adsorption study at 60 at 1°C at pH 4 found that Rhodamine B molecules diffuse very rapidly into interconnected pores of 5% by weight PLA/AC. They are very well compatible with the beads and intraparticle diffusion model. The negative value of Gibb's free energy revealed a spontaneous adsorption process and the enthalpy change (23.92 kJ mol^{-1}) revealed an endothermic adsorption process. Desorption studies of PLA-AC containing composites observed that the percentage of Rhodamine B solution desorbed increased with decreasing pH. The beads will provide a new alternative granular adsorbent for removing contaminating cationic dyes from water.

Borojeni et al. [19] within the scope of their studies, they emphasized that air filtration provides a significant increase in the global market due to the COVID-19 epidemic and that nanofiber non-woven mats can reach certain efficiencies with low pressure drop. It has a very high surface area to volume ratio, will be able to filter out submicron particles and customize the fiber material to better suit its purpose. Although nonwoven mats produced by the electrospinning technique have been very well studied and tested and reported, it has been determined that there are not many studies combining them. In this review, they refer to various ways of producing nonwoven fibers for use as air filters, the mechanisms by which the fibrous nonwoven air filter stops particles from passing through, and the methods by which nonwoven mats can be modified by morphology, with an emphasis on electrospinning. Besides the structure and material parameters, metallic, ceramic and organic nanoparticle coatings as well as electrospinning solutions with the same materials and the properties of the fibers and the effects of air filtration were investigated. Buluş et al. [20] activated carbon added PLA-AC composite membranes were produced by electrospinning technique. They examined the filtration efficiency by characterizing the filter system they developed for the COVID 19 pandemic. They found that the filtration efficiency increased with the additive and the efficiency increased as the amount increased. They determined that they provide 99.999% particle filtration. They revealed that the nanofiber membranes they obtained have potential to be used as a protective material in the COVID 19 pandemic.

In the light of literature research, in studies on oil spillage, water pollution and oil removal in general, AC and PLA is more preferred. AC reinforced PLA nanofiber biocomposites were produced, characterization studies of nanofiber biocomposites were provided and it was aimed to be used as an alternative clean air filtration material in the health sector, especially in the COVID 19 pandemic.

II. EXPERIMENTAL SECTION

A. EXPERIMENTAL

A.1. Materials

10-15 kDa polylactic acid (PLA) (Resinex-BMY Joint Stock Company) and 20-40 μm particle size activated carbon (AC) measured by Gel Permeability Chromatography were obtained from Kocaeli University, Faculty of Engineering, Department of Chemical Engineering. Waxed paper was used as support material in the electrospinning step and dimethylformamide (DMF) (Sigma/Aldrich, England) was used to dissolve the polymer.

A.2. Method

A.2.1. Preparation of Biocomposite Solutions

10 g of PLA granules were added to a 100 ml mixture of DMF solvent. PLA polymer solution was prepared according to the desired temperature and mixing speed with the help of a heated magnetic stirrer. 10 ml of PLA solution was taken into a beaker and 1%, 5% and 8% of AC four different compositions were obtained. The solutions were subjected to the values given in Table 1. and made suitable for nanofiber production by electrospinning method. Table 1 shows the preparation values of the biocomposite solutions.

Table 1. *Preparation values of biocomposite solutions*

Sample name	Solution mixing time (minutes)	Solution mixing temperature (°C)
10% PLA	60	80
10% PLA-1% AC	65	80
10% PLA-5% AC	70	85
10% PLA-8% AC	70	85

A.2.2. Biocomposite Production

Prepared 10% PLA, 10% PLA-1% AC, 10% PLA-5% AC and 10% PLA-8% AC nanofiber membrane production from biocomposite solutions was produced by Inovenso brand NS24XPpro model electrospinning method based on the values in Table 2. Production stages of biocomposites with electrospinning technique are shown in the Figure 3.

Table 2. *Electrospinning process parameters*

Sample Name	**Flow rate (ml/hour)**	**High voltage (kV)**	**Working distance (cm)**	**Collector rotation speed (rpm)**
10% PLA	3.5	24.0	16	200
10% PLA-1% AC	3.5	24.0	16	200
10% PLA-5% AC	4.0	35	16	200
10% PLA-8% AC	4.0	35	16	200

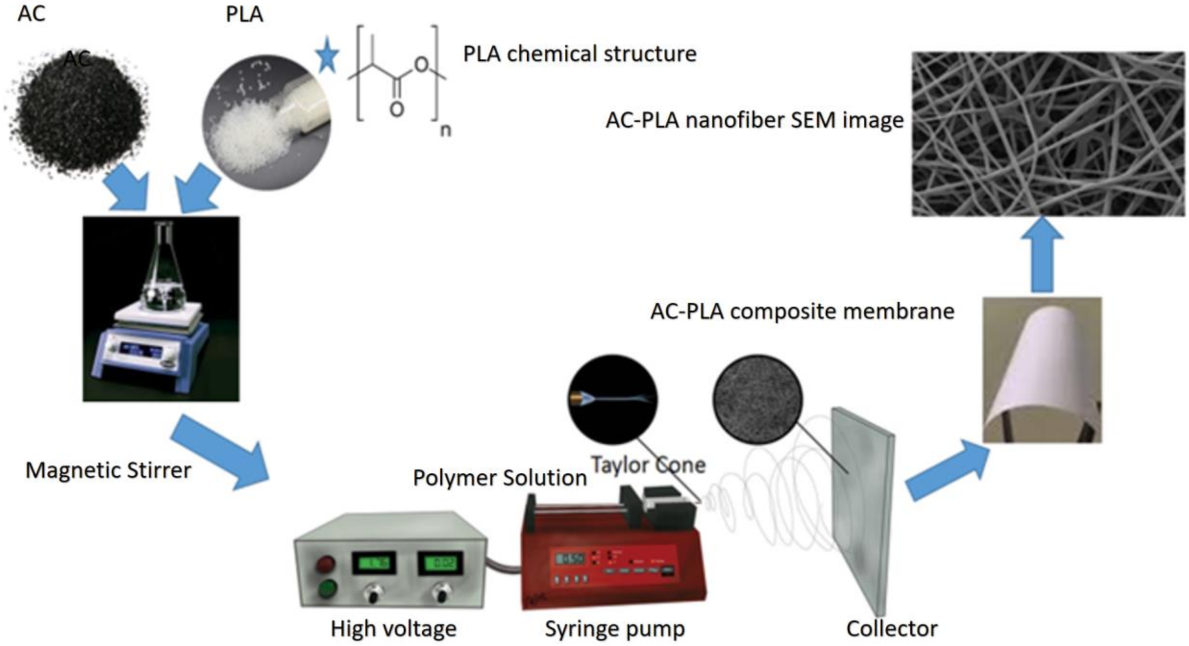


Figure 3. *Process steps of the production of biocomposites with electrospinning technique*

A.2.3. Characterization Method

Structural (FTIR) analyzes of biocomposites were performed on a Jasco 6600 model FTIR device in the wavelength range of 400 to 4000 cm^{-1}. Depending on the percent transmittance (T%) determined in the wavelength range of 400-4000 cm^{-1}, the functional bonds in the structures of the samples were determined. Tissues placed in holders were examined and visualized with the FEI FEG QUANTA 450 SEM microscope. During the examination of the diameter and dimensions of the produced composite nanofibers, images were examined at 5 kV potential at three different magnifications such as 3000x, 6000x and 12000x for FEGSEM analysis. The surface morphology of the biocomposite nanofibers was determined by the measurements made on the FEI brand FEG QUANTA 450 model Scanning Electron Microscope (SEM). The arithmetic mean fiber distribution range was determined by providing the mean diameter thickness of nanofibers and 40 different fiber diameter measurements taken from SEM images. The mechanical (tensile) test was carried out by cutting the membranes produced by electrospinning technique in 1x5 cm length according to ASTM standard and repeated 3 times under 500 Newton load. Biological (Cell culture) analysis; 3T3 cells cultured in DMEM-F12 containing 10% FBS, 1% pen strep were removed with a cell scraper, then centrifuged and cell counted. Cells incubated in a 37^{o}C incubator with 5% CO_2 were cultured for 72 hours. Counting Cells and Determining Vitality: Counting was performed after coverslip placement on a sample of Thoma slides on a 0.1 mm^3 volume count (0.1 mm high liquid). Each frame has 16 large squares and 400 small squares. Cells were counted by this counting method. The number of cells was calculated from the formula ml = 16 x 104 x number of cells x dilution factor. It is used continuously to convert the count result in a volume of 104 = 0.1 mm^3 to the number in 1 ml. Total number of cells = number of cells in ml x calculated from the formula for total volume. After centrifugation, 4% trypan blue solution was mixed with suspended cells at a ratio of 1:1. After waiting 5 minutes, Thoma was counted on the slide. Since the membrane integrity and permeability of living cells are not impaired, they cannot take the dye into the cell. Based on this feature, the percentage of stained and unstained cells and viability (%) were calculated.

III. RESULTS AND DISCUSSION

A.3. The Results of The Analysis

A.3.1. Structural (FTIR) Analysis

The PLA peak observed at 1452 cm^{-1} is stronger than the saturated CH groups (CH_3, CH_2, CH) and the carbonyl group (C=O) at 1751 cm^{-1}. Strong ester groups (C-O-C) of ether bonds at 1065-1182 cm^{-1}, weak wave number of C-H group 2946-2994 cm^{-1} and peak O-H group 3327-3727 cm^{-1} were determined [21]. When the PLA membrane was examined, the AC-PLA peak intensities decreased. AC the peaks coincided with the PLA peaks. Peak formations overlapped. The peak intensity softening in PLA showed that composite membrane fabrication was successful [22]. PLA-AC FTIR spectra of biocomposites are given in Figure 4.

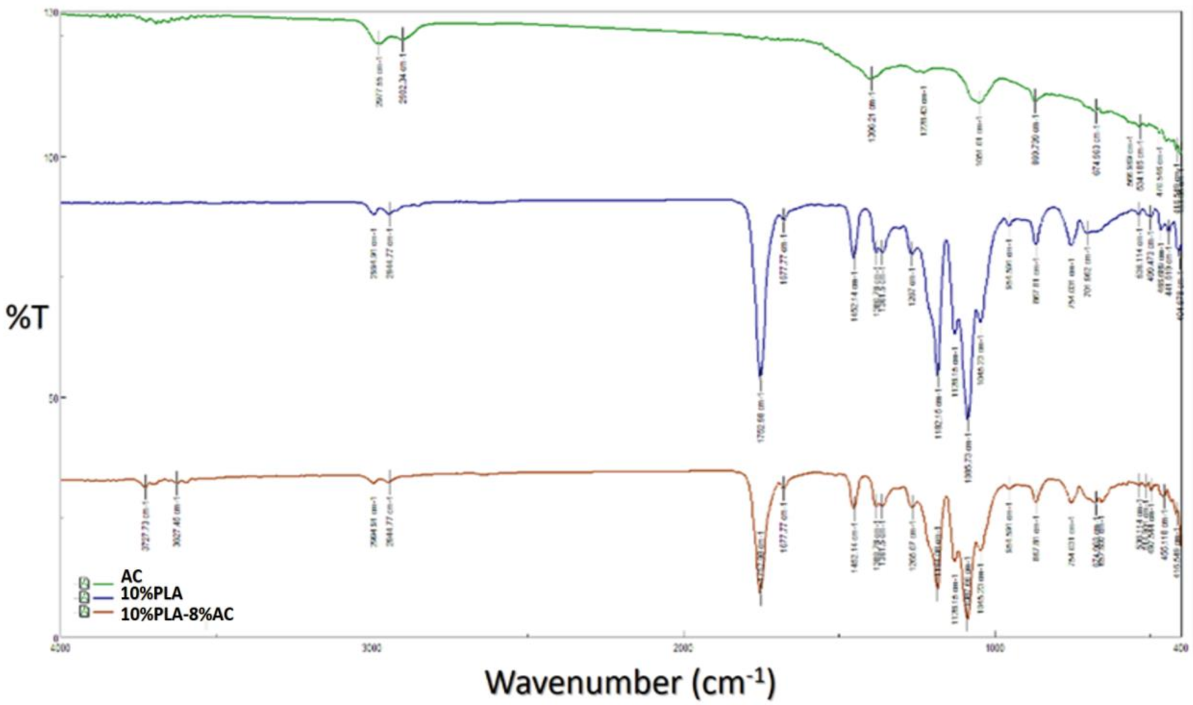
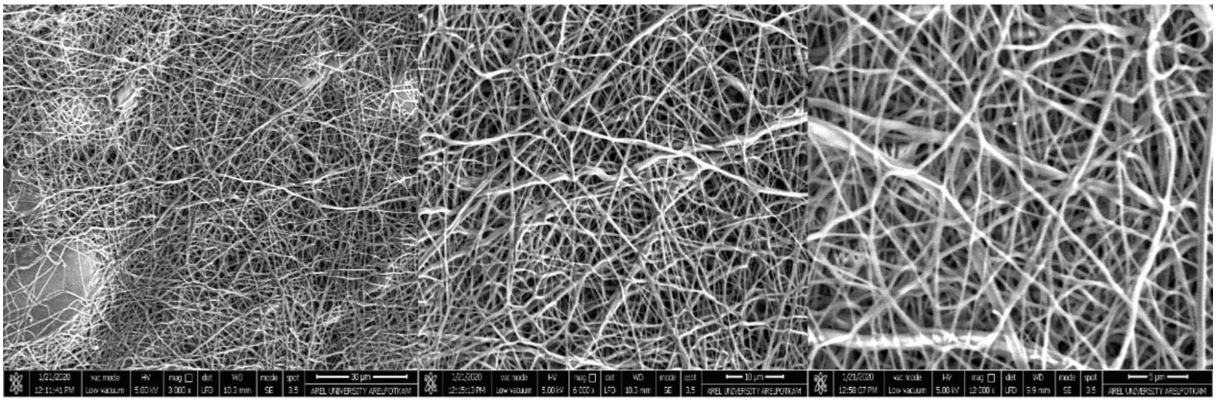


__Figure 4.__ PLA-AC FTIR spectra of biocomposites

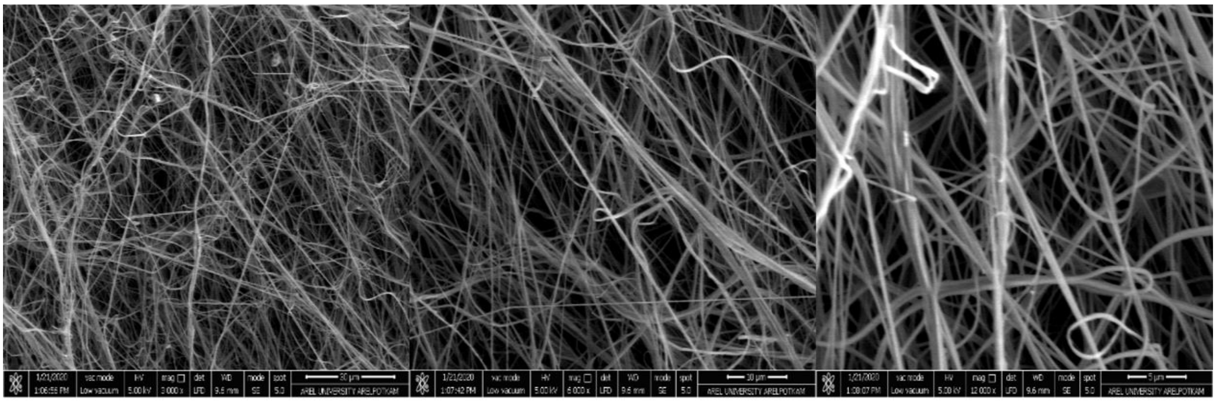
A.3.2. Morphological (FEGSEM) Analysis

The nanofiber distribution and fiber diameters of the membranes produced by the electrospinning technique were determined by FEGSEM analysis. By taking FEGSEM images at 3000x, 6000x and 12000x magnifications, fiber diameter distribution ranges were determined based on 40 nanofiber measurements on the image. The fiber diameter distribution was determined by taking the arithmetic average of these measurements. Table 3 shows the average values of nanofibers measured in FEGSEM images. 10% PLA, 10% PLA-1% AC, 10% PLA-5% AC and 10% PLA-8% AC FEGSEM biocomposite images are shown in Figure 5, Figure 6, Figure 7 and Figure 8. In the study, fiber orientations were 10% PLA- 8% AC composite was determined by FEGSEM image examinations. Tensile test was applied from the region where these nanofibers were oriented and the obtained values were higher than other biocomposites [23]. The main reason for this is AC It is the case that the material wraps the PLA fibers homogeneously.



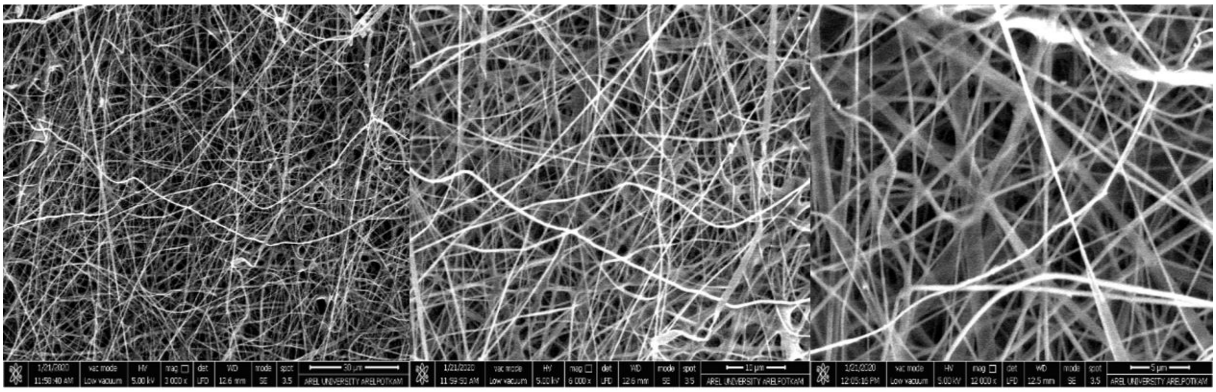
3000x **6000x** **12000x**

__Figure 5.__ Image of 10% PLA 3000x, 6000x and 12000x FEGSEM



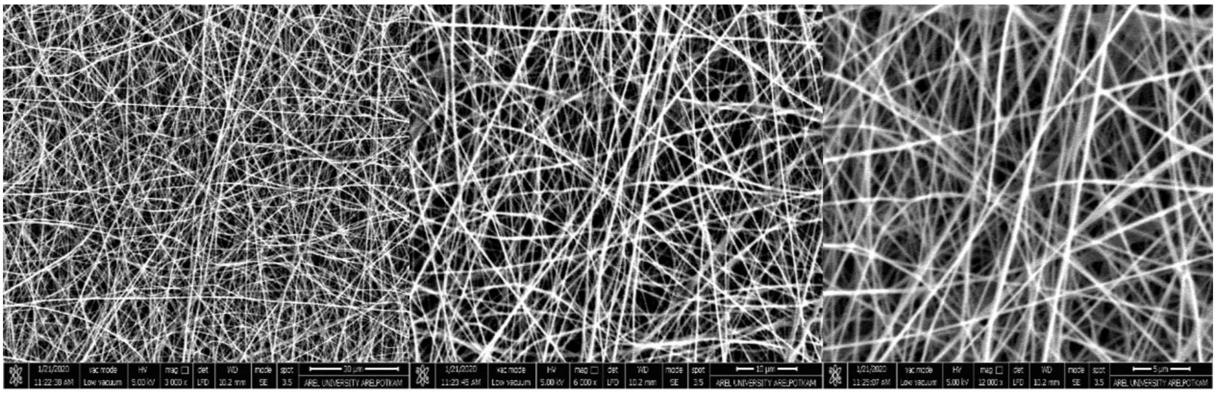
3000x 6000x 12000x

Figure 6. Image of 10% PLA-1% AC 3000x, 6000x and 12000x FEGSEM



3000x 6000x 12000x

Figure 7. Image of 10% PLA-5% AC 3000x, 6000x and 12000x FEGSEM



3000x 6000x 12000x

Figure 8. Image of 10% PLA-8% AC 3000x, 6000x and 12000x FEGSEM

Table 3. *Nanofiber average diameter ranges of electrospun mats*

Sample name	**Average diameter ranges of nanofibers (nm)**
10% PLA	160-400
10% PLA-1% AC	120-600
10% PLA-5% AC	95-270
10% PLA-8% AC	60-200

A.3.3. Mechanical (Tensile) Analysis

Tensile tests of biocomposite mats were prepared according to ASTM standard and analysis was performed. Thickness measurements of electrospun mats were provided with micrometer and these values were entered to tensile tester and required values were entered for analysis [24]. The strength value of PLA membrane is 29.2 MPa. PLA strength increased as it joined AC, and as the amount of AC increased in the composite structure in which it was produced with PLA, it provided a direct increase in strength. The source of this situation is related to the FEGSEM morphological analysis results. Pure PLA fiber diameters are 160-400 nm. However, AC As it is added, it has increased the surface area by wrapping the PLA fibers homogeneously. With this increase, tightly packed material structures have emerged. Thus, the strength values increased linearly. In this study, the highest tensile strength value was 10% PLA-8% AC with a value of 69.6 MPa. It was determined that it belonged to the composite membrane. Table 4 shows the tensile strength values of biocomposite mats.

Table 4. *Viable / dead cell amounts after 72 hours after cell cultivation of electrospun mats*

Sample name	**Mat thickness (mm)**	**Tensile strength (MPa)**
10% PLA	0.22	29.2
10% PLA-1% AC	0.30	40.9
10% PLA-5% AC	0.35	46.1
10% PLA-8% AC	0.35	69.6

A.3.4. Biological (Cell Culture) Analysis

3T3 fibroblast cell cultivation was performed on electrospun mats and cell counting was performed after 72 hours [25]. When the cell culture live/dead status was examined, 75% live and 25% dead cell status was observed on the PLA membrane in 3T3 cells. AC as the amount increases, cell viability increases in parallel with 3T3 cells. AC it was determined that the substance was good for 3T3 cells and the cells remained alive. In the study, the highest cell viability was 10% PLA-8% AC found in the composite membrane. Table 5 shows the amount of viable / dead cells after 72 hours of electrospun mats.

Table 5. *Viable / dead cell amounts after 72 hours after cell cultivation of electrospun mats*

Sample name	**Live cell (%)**	**Dead cell (%)**
10% PLA	75	25
10% PLA-1% AC	88	12
10% PLA-5% AC	95	5
10% PLA-8% AC	97	3

IV. CONCLUSION

Biocomposite membranes have been successfully produced by the electrospinning technique. When the structural (FTIR) analysis results of the produced materials are examined, AC peaks, AC and PLA peaks decreased due to the increase in their amounts. Morphological (FEGSEM) analysis shows that nanofibers become thinner as the amount of additive AC increases. When the arithmetic average of 40 nanofiber measurements made on FEGSEM images was taken, the average nanofiber diameter range was found to be 60-200 nm. The finest fibers in the study were 10% PLA-8% AC biocomposite. Mechanical (Tensile) analyzes of the membranes were made according to the ASTM standard and 10% PLA-8 AC with the highest tensile strength value of 69.6 MPa. in the biocomposite. When biological (cell culture) studies were examined, cell live/dead counts were made as a result of culturing 3T3 fibroblast cells. When the number of live/dead cells was examined, 97% viability and 3% mortality rate were determined. When the results of the study are evaluated, PLA-AC composite membrane will be an ideal material candidate in clean air filtration systems. In addition, considering the industrial use properties of the substances in the study, they will have sectoral uses as innovative and value-added products that can appeal to different sectors such as health, cosmetics, food, textile. It is aimed to be used as an alternative clean air filtration material in the health sector, especially in the COVID 19 pandemic.

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