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Production of polylactic acid-activated charcoal nanofiber membranes for COVID-19 pandemic by electrospinning technique and determination of filtration efficiency

Erdi Buluş[1,2*], Gülseren Sakarya Buluş[3], Fahrettin Yakuphanoglu[4]

[1]Polymer Science and Technology Program, Institute of Science and Technology, Kocaeli University, Kocaeli 41000, Turkey
[2]ArelPOTKAM (Polymer Technologies and Composite Applications and Research Center), Istanbul Arel University, Istanbul 34500, Turkey
[3]Silivri District Health Directorate, Istanbul 34570, Turkey
[4] Department of Physics, Faculty of Science, Firat University, Elazig, 23169, Turkey

It is the use of masks as protective equipment against corona virus, also known as COVID-19 pandemic, which is the current problem of today. The mask is a personal protective equipment that protects employees against dust and particles of physical, chemical and biological agents. It is classified as ffp1, ffp2, ffp3 in European standards. Dust masks, surgical masks, gas masks, medical masks and synthetic-based masks are used in health and industrial areas. The primary function of these masks is to be protected from harmful factors. Masks are insufficient in terms of duration and function. In this study, 1%, 5% and 8% activated charcoal (A.C.) reinforced polylactic acid (PLA) nanofiber membranes were produced by electrospinning technique. Structural (FTIR, Fourier Transform Infrared Spectroscopy), morphological (SEM, Scanning Electron Microscope), mechanical (Tensile) and filtration efficiency analyzes were performed on the produced membranes and the material properties were determined. The membranes obtained can be used as an ideal filtration membrane that can be used for long-term use for the use of the whole society, especially healthcare workers, as protective equipment, especially in the COVID-19 pandemic.

Key Words: Covid-19, activated charcoal, electrospinning, clean air filtration

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**Corresponding author: buluserdi@gmail.com (Erdi Buluş) Tel: +905346321017*

1. Introduction

Corona viruses are viruses that people encounter at a moment in their life. Human corona viruses usually cause mild to moderate upper respiratory tract diseases. There are four subgroups of corona viruses known as alpha, beta, gamma and delta. Corona viruses (CoV) can cause various diseases from the common cold to more serious diseases such as Middle East Respiratory Syndrome (MERS-CoV) and Severe Acute Respiratory Syndrome (SARS-CoV). Many people can die from bacteria and viruses, especially the corona virus, which is the current problem of today [1].

Protective masks are used to protect against bacteria and viruses. However, the pore size of the masks used cannot be effective against bacteria and viruses. Masks are classified as ffp1, ffp2, ffp3 in European standards. Dust masks, surgical masks, gas masks, medical masks and synthetic-based masks are used in health and industrial areas. The primary function of these masks is to be protected from harmful factors. An effective filtering feature cannot be created against carbon monoxide and harmful gases as well as other viruses and bacteria such as corona virus, the size of the corona virus is 100-160 nanometers, the N95 mask is 95% protected but can be used for 8 hours, it does not have functional features, does not create wide usage opportunity, high cost There are various problems such as not being able to breathe comfortably, sweating (the virus spreads faster in a humid environment). On the other hand, nanofiber membranes consisting of fibers with a large surface area have been developed with the nanotechnological electrospinning technique. While fiber structures smaller than the size of the corona virus do not allow the passage of viruses or bacteria, comfortable breathing capabilities can be brought to the membranes with optimization studies during the electrospinning technique. Electrospinning technique is a method of producing nanofiber from polymer solutions with the help of an electric field. In this way, membranes consisting of fibers with a wide surface area can be obtained, and it can be ensured that innovative products for the purpose are produced and developed in the health, filtration, food, agriculture, defense, textile sectors [2].

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, food, textile and pharmaceutical sector [3]. The chemical formula of PLA ($C_3H_4O_2$) is shown as n. The chemical structure of PLA is shown in Figure 1.

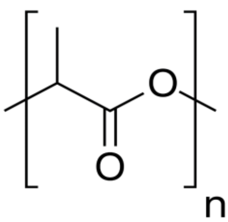

Figure 1. *Chemical structure of PLA* [3]

As a consumer product produced using PLA; clothes, utensils and food packages. PLA used in the medical sector, diapers, feminine hygiene products, medical sutures, stents and pharmaceutical applications can also be used. Ray et al. (2003) studied a series of PLA / organoclay-based biocomposites and observed that the biocomposite of PLA was completely degraded and destroyed in 2 weeks [4]. Kakroodi et al. (2017) developed the gas permeability properties of PLA films using microfibrillation process [5]. Dasan et al. (2017) improved the oxygen barrier properties in PLA / Poly (3-hydroxybutyric acid-co-3-hydroxvaleric acid) (PHBV) polymer films prepared using nanocrystalline cellulose [6].

Activated carbons are capable of separating volatile organic compounds (VOCs), odors and other gaseous pollutants from air. They perform this cleaning in a different way than HEPA filters or other types of air cleaner filters that capture only particles from the air. A.C. filters perform this process by trapping gas molecules in coal beds. A.C. filters are the most commonly used filters to capture gases. They are designed to filter gases in an A.C. bed and are often used to filter volatile organic compounds released into the external environment. In addition, A.C. filters are used to remove airborne odors, such as the smell of cigarette smoke. However, they cannot remove particles such as mold, dust or pollen from the air. A.C. filters are carbons that have undergone some additional processing to better filter gas molecules. First, the lattice structure of the small pores is formed by the hot air supplied to the carbon, and then carbon dioxide or steam is injected to greatly increase the surface area. This provides more space for the capture of gas molecules and makes carbon more effective as a filter area. Carbon was used in gas masks used to filter some of the deadly gases used against troops in World War I, but was only effective against certain toxins. A.C. production and use only II. It increased dramatically after World War II and eventually led to the development of modern air and water filters. The adsorption process allows it to filter or capture organic chemicals (gases) from the air for A.C. air filters. The most important problem in the A.C. bed is that over time, gaseous pollutants gradually fill the adsorption surfaces of the A.C.. When the bed becomes saturated, the filter is no longer able to retain contaminants [7].

In this study, 1%, 5% and 8% A.C. reinforced PLA nanofiber membranes were produced by electrospinning technique. By characterizing the produced membranes, it is aimed to be an ideal filtration material for long-term use of the entire society, especially healthcare workers, as protective equipment especially in the COVID-19 pandemic.

2. Experimental Studies

2.1 Materials

Apetech Automotive Distribution and Trade Inc. 10-15 kDa polylactic acid (PLA) measured by gel dispersibility chromatography (GPC) supplied by the company, dimethylformamide (DMF) organic solvent for the dissolution of the polymer with activated carbon (A.C.) and oil paper as the substrate in the electrospinning process were preferred.

2.2 Preparation of composite solutions

10 g of PLA granules were added to a 90 ml mixture of DMF solvent. PLA polymer solution was prepared according to desired temperature and mixing speed with the help of magnetic stirrer with heater. 10 ml of PLA solution was taken into a beaker and 1%, 5% and 8% A.C. four different compositions were obtained. The solutions were subjected to the values given in Table 1 and made into suitable form for nanofibre production by electrospinnig method. Table 1 shows the preparation values of composite solutions.

Table 1: Preparation values of composite solutions

Sample Name	Solution Mixing Time (min.)	Solution Mixing Temperature (°C)
10%PLA	45	30
10% PLA-1% A.C.	50	35
10% PLA-5% A.C.	55	40
10% PLA-8% A.C.	55	45

2.3 Composite production by electrospinning method

Prepared 10% PLA, 10% PLA-1% A.C., 10% PLA-5% A.C. and 10% PLA-8% A.C. nanofiber membranes were produced using the electrospinning parameters in Table 2 with the FYTRONIX FY 7000 ELECTROSPUN SYSTEM device. Device visual of FYTRONIX FY 7000 ELECTROSPUN SYSTEM is given in Figure 2. Figure 3 shows the composite membrane production stages with electrospinning technique. Electrospinning parameters applied for nanofiber membrane production are shown in Table 2.

Figure 2. *Device image of FYTRONIX FY 7000 ELECTROSPUN SYSTEM*

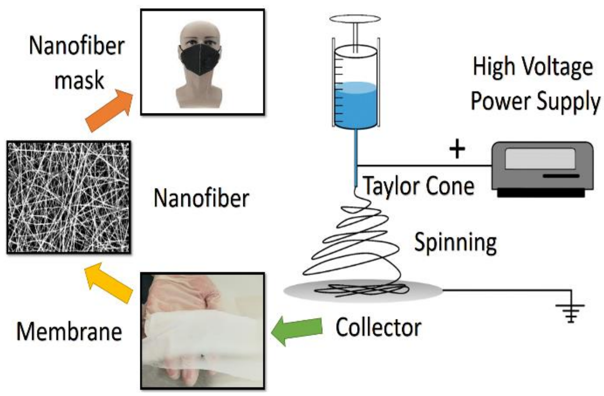

Figure 3. *Process steps of the production of composites with electrospinning technique*

Table 2: Electrospinning parameters applied for nanofiber membrane production

Sample Name	Flow Rate (ml/hr.)	High Voltage (kV)	Working Distance (cm)
10% PLA	2.5	25.8	15
10% PLA-1% A.C.	2.5	25.8	15
10% PLA-5% A.C.	3.0	30	15
10% PLA-8% A.C.	3.0	30	15

2.4 Characterization of composites

Structural (FTIR) analyzes of the membranes were performed on the Jasco 6600 model analyzer at wavelength ranges of 400 to 4400 cm^{-1}. The bonds in the structures of the samples were determined depending on the permeability percentage (% T) determined in the wavelength range of 400-4400 cm^{-1}. Tissues placed in the holders were examined and photographed with a ZEISS EVO SEM microscope. During the examination of the diameter and dimensions of the produced composite nanofibers, images magnified at x6000 times were examined for SEM analysis at 7 kV potential. The average diameter thickness of the nanofibers was measured on high resolution SEM photographs using Image j (National Health Organization) software. The mechanical (tensile) test was carried out by cutting the electrospun mats 1x5 cm in length according to the ASTM standard and repeating them 3 times under 500 Newton load. Filtration efficiency of the produced membranes is determined according to the conditions in the European standard EN 14683.

3. Result and Discussion

3.1 Structural (FTIR) analysis

The peak of the PLA observed at 1452 cm^{-1} was strong (CH_3, CH_2, CH) of saturated CH groups, and the strong (C = O) of the peak carbonyl group (C = O) at 1751 cm^{-1} was found at 1065-1182 cm^{-1}. ester groups of ether bonds strong (C-O-C), 2946-2994 cm^{-1} wave number of the C-H group weak, 3417 cm^{-1} peak O-H group was determined [8]. The contribution of the electrospun mat formed in the FTIR spectrum of the PLA when the peak of the initial PLA was found to be gradually reduced, but the contribution rate is very low due to the apparent A.C. peak was not found. PLA-A.C. FTIR spectra of composites are given in the Figure 4.

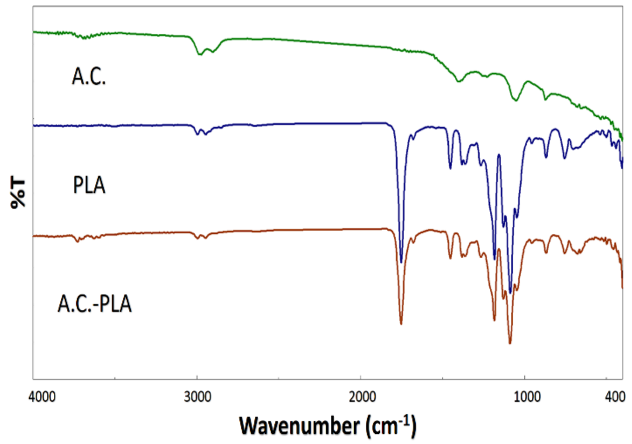

Figure 4. *PLA-A.C. FTIR spectra of composites*

3.2 Morphological (SEM) analysis

The nanofibre distribution and diameters of the electrospun mats were performed by SEM analysis. 40 nanofiber measurements were measured on the image at x6000 magnification. SEM images of composites are shown in the Figure. Table 3 shows the mean values of nanofibers measured on SEM images. 10% PLA, 10% PLA-1% A.C., 10% PLA-5% A.C. and 10% PLA-8% A.C. SEM images of composites are shown in Figure 5, Figure 6, Figure 7 and Figure 8. 10% PLA-8% A.C. When SEM image of composite was examined, it was determined that nanofibers were directed. Tensile test was applied from this nanofiber directed region and the values obtained were higher than other composites. The most important contribution here is A.C. homogeneously distributed oriented fiber structures were obtained by increasing the electrical conductivity resistance of PLA nanofibers and increasing the nanofibre formation [9,10].

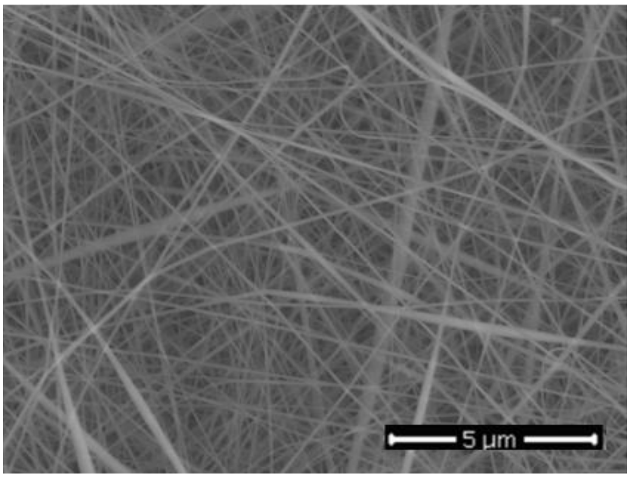

Figure 5. *Image of 10% PLA x6000 SEM*

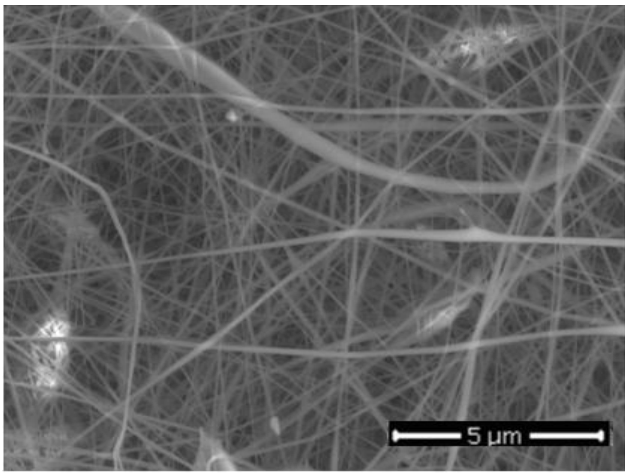

Figure 6. *Image of 10% PLA-1% A.C. x6000 SEM*

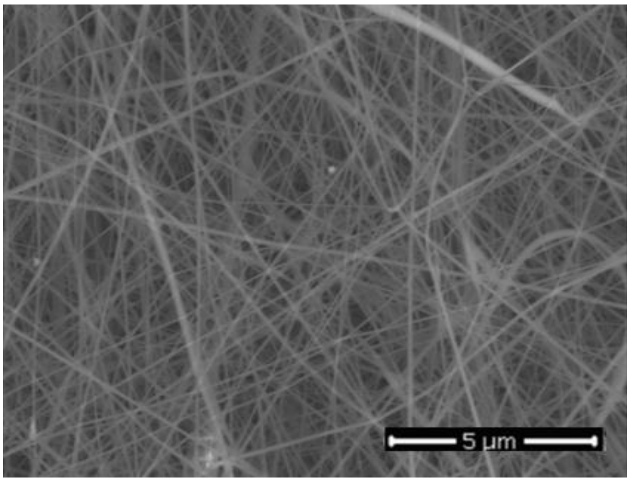

Figure 7. *Image of 10% PLA-5% A.C. x6000 SEM*

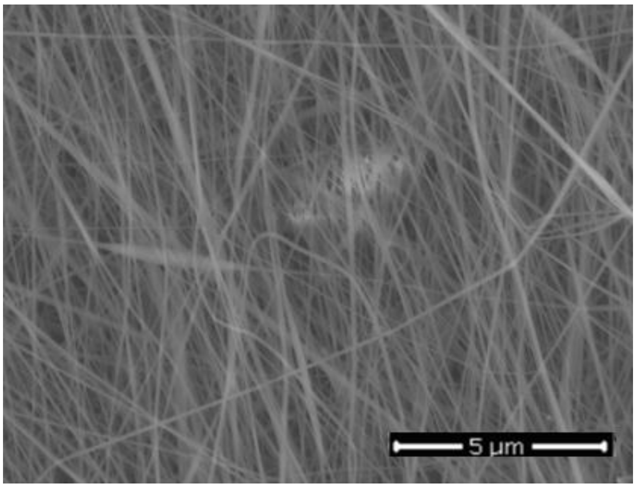

Figure 8. *Image of 10% PLA-8% A.C. x6000 SEM*

Table 3: Nanofiber average diameter ranges of electrospun mats

Sample Name	Average Diameter Ranges of Nanofibers (nm)
10% PLA	140-300
10% PLA-1% A.C.	155-550
10% PLA-5% A.C.	90-320
10% PLA-8% A.C.	80-240

3.3. Mechanical (Tensile) analysis

Tensile tests of composite 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. According to the tensile test values of the membranes, 10% PLA-8% A.C. its composite has the highest value in the study. The most important reason for this is that it has the thinnest nanofiber structure and the orientation of the fibers [9-10]. The membrane tensile test chart is shown in Figure 9. Table 4 shows the tensile strength values of composite mats.

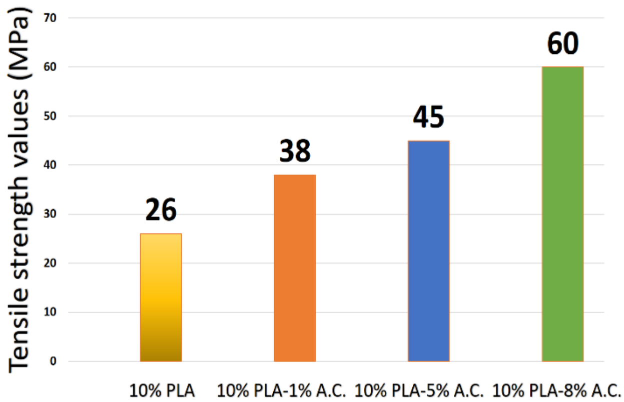

Figure 9. *Membranes tensile test graphic*

Table 4: Tensile strength values of composite mats

Sample Name	Mat Thickness (mm)	Tensile Strength (MPa)
10% PLA	0.27	26
10% PLA-1% A.C.	0.28	38
10% PLA-5% A.C.	0.30	45
10% PLA-8% A.C.	0.33	60

3.4 Filtration efficiency analysis

Bacterial filtration efficiency (BFE) (%), differential pressure (delta p, mm H2O / cm2, submicron particle filtration efficiency (%), resistance to penetration (Synthetic Blood) (mm Hg) and flame spread processes within the scope of filtration efficiency analysis of membranes Its protective properties against COVID-19 pandemic were determined by applying the filtration efficiency values of the membranes are shown in Table 5. When the filtration efficiencies of the membranes were examined, it was determined by the measurements that four different samples had high filtration efficiency [9-12].

Table 5: Filtration efficiency values of membranes

Assessment / Testing	High Level Barrier
Bacterial Filtration Efficiency (BFE) (%)	≥ 98
Differential Pressure (Delta P, Mm H2O / Cm2	< 5.0
Submicron Particle Filtration Efficiency (%)	≥ 98
Penetration Resistance (Synthetic Blood) (Mm Hg)	160
Flame Spread	Class I

4. Conclusion

With electrospinning technique 10% PLA, 10% PLA-1% A.C., 10% PLA-5% A.C. and 10% PLA-8% A.C. composites have been successfully obtained. When the structural (FTIR) analysis results of the produced materials are examined, it is seen that A.C. has peaked, but depending on the increase in the amount, A.C. and the amount of PLA peaks appears to be reduced. Morphological (SEM) analysis shows that as the additive amount of A.C. increases, the nanofibers thinning occurs. When the arithmetic mean of the 40 nanofiber measurements made on SEM images was taken, the average nanofiber diameter range was found to be 80-240 nm. The best fibers in the study are 10% PLA-8% A.C. obtained in the composite. This thinning A.C. the rate of contribution has increased. By adding and increasing the additive, the electrical resistance of the solution was increased and finer nanofibers were obtained. Mechanical (tensile) analyzes were made according to ASTM standard and the highest strength value was 10% PLA-8% A.C. 60 MPa was measured in the composite. It has been determined that it is above ≥98 in terms of filtration efficiency. When the results of the study are evaluated, PLA-A.C. composites have ideal material properties that can be preferred in protective equipment production and filtration applications, especially in the COVID-19 pandemic process.

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