Fabrication of Composite Nanofibre PEO/Lignin for Exhaust Gas Emissions by Electrospinning

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Abstract: Air pollution from the combustion of exhaust gas emissions increases with the rise in motorized vehicles use which affects human health. Therefore, it is important to control gas emissions from motorized vehicles. In this study, electrospinning was used to fabricate composite nanofibre lignin/PEO as a filter for gas emission. The nanofibers obtained were analyzed by using SEM before and after gas emission test. The morphological analysis showed that the presence of lignin in the PEO decreased the diameter of the nanofibre from 800 nm to 100 nm, hence improved the gas adsorption efficiency. After gas emission test, the filtration power was 61% for hydrocarbon gas, 81% for CO gas, and 33% for CO_2 gas.

1 INTRODUCTION

Dreadful air pollution is often accompanied by rapid industrialization. In the history, millions of premature deaths were related to extreme air pollution. World Health Organization (WHO) study reported that more than 80% of the world's population have been exposed to air pollution with levels that have surpassed 2018 WHO limits (World Health Organization, 2016). The presence of harmful pollutants, mixture of particles, toxic gases, and microorganisms have greatly endangered public health. In particular, fine particles in complex mixtures with diameters less than 2.5 µm have been identified as the main threat because they can easily penetrate into human lungs and bronchi, resulting in increased risks of asthma, lung cancer, stroke, and heart disease (Li *et al.*, 2019). There are 4 gas emissions produced by motorized vehicles, such as hydrocarbons (HC), carbon monoxide (CO), nitrogen oxides (NOx), and other particles that come out as exhaust gases. The main source of pollution comes from transportation of fuel oil, which produces 60% CO gas and 15% hydrocarbons (Ortega *et al.*, 2019).

Materials for air filtration are highly desired to protect human health from extreme particle emissions. They have been intensively studied for the past few years. The ideal air filtrations must be capable of efficiently trapping aerosol particles, while still allowing air to easily pass through at the same time. Various materials have been studied to achieve high efficiency air filtration, including foams, carbons, and fibers (Zhang *et al.*, 2019).

Lignin, a plant-based biopolymer, is available abundantly as the by-product of pulp/paper and cellulosic ethanol industries. It is a three-dimensional polyphenolic polymer and naturally available in the cell wall of plants (Poursorkhabi *et al.*, 2015). Different lignocellulosic materials have different lignin concentration, which can be about 15–30% of the materials. Cellulose processing industries must separate lignin from the raw materials. Given the high production and demand for both paper and cellulosic ethanol, there is a substantial amount of lignin produced annually. Current applications of lignin are limited to low-value products, such as dispersing agent, stabilizer, rheology control materials, and low-cost fuel to produce energy. However, lignin has the potential to be utilized as higher value products, hence improve the economics of the relevant industries

Electrospinning was commonly employed in the manufacturing of nanofiber sheets for applications in

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various fields such as nano sensors, ultrafiltration membranes and nanocomposites (Chen *et al.*, 2014; Ko *et al.*, 2015; Akgul *et al.*, 2018; Choi *et al.*, 2019; Zhang *et al.*, 2019). Pure lignin solution usually does not have enough viscoelasticity for spinning. Therefore, it is blended with another polymer, called as a binder. Blended solutions such as lignin and poly(ethylene oxide) (PEO), poly(vinyl alcohol) (PVA), or poly acrylonitrile (PAN) have been electrospun. Most of electrospun lignin fibers were produced from solutions prepared in N,N-dimethylformamide. Therefore, electrospinning of aqueous solutions is more preferred. Among water soluble polymers, electrospinning of aqueous solutions between lignin, PVA and cellulose nano-crystals has been reported. Schreiber *et al* reported electrospinning of aqueous solutions, such as sodium carbonate lignin and PEO, with maximum lignin to PEO ratio of 50/50. When PEO and lignin were mixed together in the presence of salts and water, the chain entanglements of PEO trapped lignin molecules. Eventually, the bridging of PEO chains created an association of induced complex.

Electrospinning has been the pioneer of nanostructure 1D fiber membrane production from a variety of materials, including polymers, composites, and ceramics. From the electrospinning fabrication process, the diversity of materials and their unique association with fibers can be used for various applications, such as biomedical, drug delivery, tissue engineering, wound dressing, filter, membrane, energy, and electronic applications (Bellan, 2008).

Ideal structures of carbon monoxide filters should be characterized from small fiber diameters and pore sizes, which are important to capture particles effectively. High porosity is responsible to allow air stream to pass through the gas easily. Small fiber diameters are beneficial to provide high filtration efficiency according to Kuwabara model. Moreover, small pores, by relying on sieving effect, can completely remove particles larger than they are. Therefore, EAFMs possessing ultrafine fibers and extra small pores are highly desired for efficient removal of fine particles (Li *et al.*, 2019).

2 EXPERIMENTAL

2.1 Materials

Lignin alkali (partially soluble 13.4 wt,% loss on heating at 316ºC, pH: 6.5 (25ºC), 5% aqueous solution, density: 1.3 g/mL at 25ºC), fully hydrolyzed PEO (Mw approx. 600000, with viscosity 20ºC, 4% water, ≥98.0% degree of hydrolysis) purchased from Sigma Aldrich, USA. The lignin and PEO were used as they were. The aqueous solutions were prepared by using distilled water. Triton X-100 as a surfactant from Fisons

2.2 Solution Preparation

Preliminary experiments on solubility of lignin alkali in aqueous sodium hydroxide solution with different pH showed that solution with 0.5 mol concentration (pH>13) was completely dissolved. This concentration was used to prepare the blend of lignin/PEO solution.

To prepare PEO/Lignin solution blend, lignin and PEO were first dissolved separately to ensure complete solubility of these materials. Alkaline water and distilled water were warmed to about 70ºC, before lignin and PEO were added. Stirring was necessary when PEO was added to the solvent to avoid agglomeration of particles. Next, each of the solution was stirred at 70ºC and 600 rpm for 2 h to completely dissolve the polymers. An equivalent volume of each solution was taken and mixed together for 15 min.

2.3 Electrospinning Process

The electrospinning process of PEO/lignin solution was prepared and carried out in a horizontal electrospinning machine (syringe SP20, high voltage power supply PS-35PV, and speed controller with drum collector ESD-30S, NLI, Malaysia) on a substrate material. The electrospinning machine had a horizontal configuration with distance between the needle and collector was 10 cm. Voltage applied and feed rate were kept constant at 20 kV and 0.4 mL h^{-1} respectively. After mixing, PEO/lignin mixtures were stored for at least 1 hour before spinning process to provide enough time to remove the effects of applied shear stress during stirring.

2.4 Characterization

2.4.1 Scanning Electron Microscopy

The surface morphology of electrospun nanofibers was observed by scanning electron microscopy (SEM) at an accelerating voltage EHT of 20.00 kV, probe = 101 Pa and signal A = SE1. The samples were placed on an adhesive-backed carbon tape and secured to the specimen. Next, the sample was sputter-coated by a thin layer of gold alloy (SC 500 emscope) to reduce charges during analysis.

2.4.2 Emission Analyzer Test

The electrospun PEO/Lignin nanofibers were tested by Automotive Emission Analyzer Machine from Nanhua Instrument. The nanofiber sheets were inserted into the engine analysis channel, and connected to the exhaust of motorcycles. The exhaust emission data was observed with and without the nanofibers.

3 RESULTS AND DISCUSSION

3.1 Morphology Fiber before Emission Gas Tested

Non-toxic PEO was chemically stable in acidic solutions and it has a molecular weight capable of forming electrical fibers (Nagapudi, 2014). Fibers, however, could not be formed from pure lignin solutions, hence the need to add a supporting polymer, PEO for instance. High resolution of SEM exhibited unique morphology as the function of PEO/Lignin ratio, and has an influence on conductivity and solution flow rate.

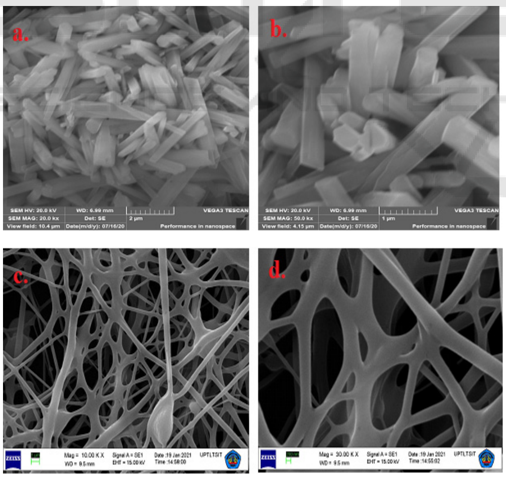

Figure 1. PEO/lignin fiber of various weight ratio surface morphology before emission gas test a) (6: 0) Mag 5 Kx; b) (6: 0) Mag 10 Kx; c) (6: 5) Mag 5 Kx; d) (6: 5) Mag 10 Kx

The morphology of PEO/lignin blend is shown in Figure 1 displays the surface morphology of PEO/Lignin fibers analyzed by SEM, showing the differences in the diameters of the fibers formed. The flow rate, viscosity, and conductivity of the solution had an impact on the morphology of fibers (Nagapudi, 2014).

Low viscosity of polymer solution would tend to form bead. In addition, higher concentration of solutions has been observed to have formed less beads (Harahap, 2018). In this study, PEO/Lignin with weight ratio of 6:0 produced ultrathin nanofibers with no presence of beads, as well as nanofibers in the addition of lignin (Poursorkhabi *et al.*, 2015).

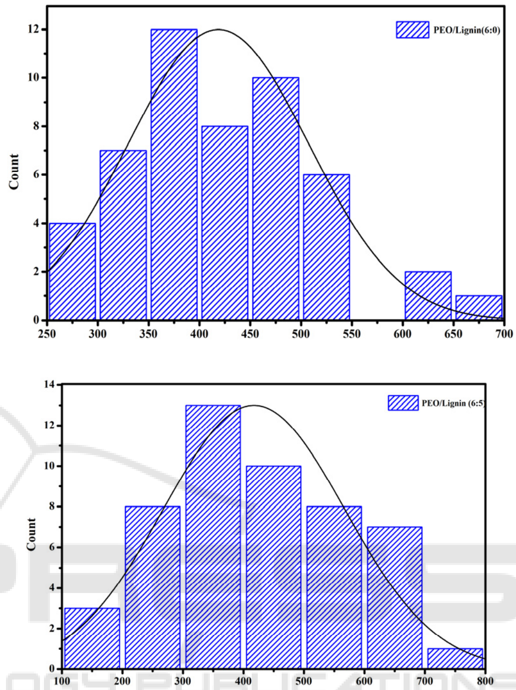

Figure 2. Diameter distribution of PEO/Lignin nanofiber a) (6:0); b) (6:5)

Figure 2 presents that the analysis of diameter distribution in PEO/Lignin nanofibers. The diameter data of fibers was analyzed randomly by taking 50 fiber spots as analysis points. There were significant differences with each lignin additional variation. The fiber of pure PEO nanofibre was in the range of 250-700 nm, with most fibers at 350-500 nm diameter sizes. Meanwhile, PEO/Lignin nanofibre were at the range of 100-800 nm, with most fibers at 300 nm diameter (Widianto *et al.*, 2018).

3.2 Scanning Electron Microscopy Morphology

Surface morphology of PEO/Lignin nanofibers before and after CO emission test is shown in **Figure 3**. Gas emission test from the motorcycle without treatment was treated as control. Pure PEO nanofibre was a fiber sample used as the control parameter. The data shows the ability of each fiber to filter gas emission. The analysis of nanofiber before and after the emission gas test had significant differences. After the emission test, the morphology of nanofibers had rough surfaces with many beads, indicating that the nanofibers had maximally adsorbed the gas.

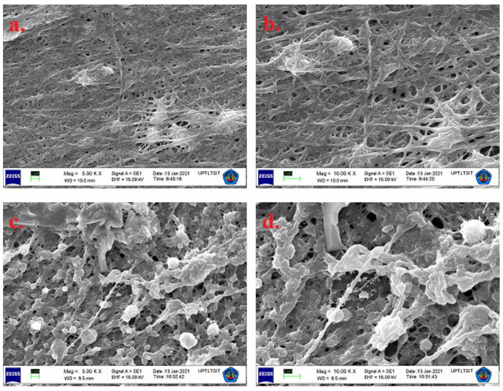

Figure 3. Surface morphology of PEO nanofibre with magnification of (a) 5 Kx, (b) 10 Kx, and PEO/Lignin nanofibre with magnification of (a) 5 Kx, (b) 10 Kx.

Figure 3 shows the results of the SEM morphological analysis, that the ability of lignin nanofibres to absorb gas emissions.

Numerical figures from the analysis of emission gas test are presented in the Table 1.

Table 1. Numeric results of gas emission

No	Sample	HC (ppm)	NO (ppm)	CO (%)	CO^2 (%)
1	Control	3.50	0.08	2.43	1.80
2	6:0	1.44	0.16	1.06	1.49
3	6:5	1.35	0.69	0.44	1.20

Reduction of gas may occur because the composition of the adsorbent mass of lignin used is greater than the others, so that the capacity to absorb gas emissions was higher. When the mass of the adsorbent was higher, the absorption of gas would be higher too. This was indicated by the results of exhaust emission measured. The contact region between the adsorbent and emission gas was the adsorption zone (adsorbate). If the area of the adsorption zone was larger, the more gas had been absorbed. This has caused the concentration of gas emissions released to be reduced (Rina *et al.*, 2018).

4 CONCLUSION

Lignin/PEO nanofibers fabricated by electrospinning method can be used as a filter for exhaust gas emissions. The analysis of SEM morphological prove that the electrospinning fibers have a shape of nanostructures with 1D dimensions and 100-800 nm fiber diameters.

The absorption characteristic of lignin nanofibers is due to porous fibers produced which can absorb emission of gases. The successful fabrication of nanofibers also supports by gas emission test, in which the reduction of HC, CO, and CO_2 gas is up to 61%, 81%, and 33% respectively.

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