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Investigation of Lignin as a Textile Fiber

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Abstract

Lignin is a substance that is in the state of waste in the forest industry and the paper industry, which is very much in nature after cellulose. It is mentioned in the literature that lignin can be used as raw material for carbon fiber, which is a technical fiber, briefly used as a raw material of carbon fiber. Although lignin is such an important area of use, unfortunately, it cannot be effectively and adequately assessed, it is destroyed by burning in the heat recovery units in factories. This is how lignin is used in the worst way, leading to environmental problems. This study aims to use lignin which is a waste for the paper industry as a renewable and sustainable resource for the textile industry. 11 lignin solutions and 4 Lignin-Polyvinyl Alcohol blends were formed. Blends were pulled by the electrospinning method, and fiber spinnability was investigated by a method that is similar to the melt spinning method solution. Obtained results are given in the table.

Keywords: Lignin, paper industry, forest Industry, recycling, textile fiber.

1. Introduction

With the developing technology and population growth, the need for energy and raw materials has increased. If these increasing needs are not met by renewable resources, it will be difficult to leave a livable world for future generations. Lignin is the most common natural polymer after cellulose in nature. Lignin, which constitutes 20-30% of woody biomass [1,2], is not used as a by-product of biofuel production and papermaking processes. There is not only one lignin chemical structure. It varies according to the type of plant. Lignin is removed during paper production. If the amount of lignin removed increases, the quality of the paper increases. Lignin is an impure substance in the paper, making paper poor quality. In addition to the disadvantage of poor quality of lignin paper, it is also a problem in animal husbandry [3]. Because of the disadvantages of the presence of lignin alongside cellulose, the lignin must be separated from the cellulose. This process delignification, that is called the removal of lignin. Although lignin is a poor-quality factor for paper, it is the opposite of wood. Lignin provides resistance to the climatic conditions of the tree against factors such as cold, heat, disease, and animal batting.

Due to the abundance of lignin in nature, low cost, and sustainability, lignin is being explored as raw material to produce higher-value chemicals. Lignin is an inexpensive precursor for carbon fibers [4]. Kadla et.al used lignin as a precursor for carbon fiber. They produced carbon fibers from hardwood lignin/synthetic polymer blend fibers. They used polyethylene and polypropylene with hardwood lignin [4]. Zhang and Ogela used an acetylated softwood kraft lignin. They used dry-spun into precursor fibers and successfully processed into carbon fibers [5]. Sudo et.al used lignin as a precursor for carbon fiber. They used lignin-based carbon fiber in 43.7% of yield based on a starting material [6]. Mainka et.al explained that lignin as an alternative precursor and reduction in the cost of carbon fiber-reinforced polymer and reduction of CO_2 emission during carbon fiber production [7]. Norberg used kraft lignin in his doctoral thesis as a carbon precursor. And also they used the melt spinning production method [8]. Hayashi et al. prepared activated carbons from lignin by chemical activation with $ZnCl_2$, H_3PO_4, and some alkali metal compounds [9]. Demir et.al presented a new fabrication method for the hetero atom-doped porous carbons derived from a renewable resource via HTC followed by KOH activation [10].

This study aims to use lignin which is a waste for the paper industry as a renewable and sustainable resource for the textile industry. This paper is organized as follows: a brief general purpose of the study in Section I. General structure of Lignin in Section II. Material and method in Section III. Experimental results are presented in Section IV. Results and discussions are in Section V. Finally the conclusion is in Section VI.

2. Lignin

The origin of the word lignin comes from the Latin word lignum (wood). One of the most important chemical components of plants [11]. From a chemical point of view, lignin is defined as a complex polymer chain of phenylpropane groups having thermoplastic properties. Therefore, the high lignin content ensures that the sprout of a plant can remain upright. One-year plants in the biomass (annual harvest cycle) have a much higher lignin content and therefore can withstand consistently high stresses despite a relatively low water content. After cellulose, it is the most natural polymer found in the plant world. Its main function in the cell wall is to keep the cellulose fibers together due to its adhesive properties. Most of the polymeric structure of lignin; three alcohols: sinapyl, p-kumaryl, and coniferyl [1], [12], [13](Figure1.).

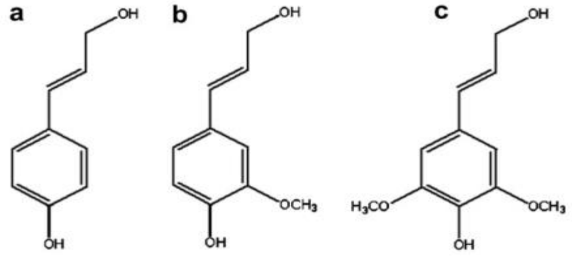

Figure 1. (a) p- Kumaryl alcohol, (b) Coniferyl alcohol (c) sinapyl alcohol [12], [14].

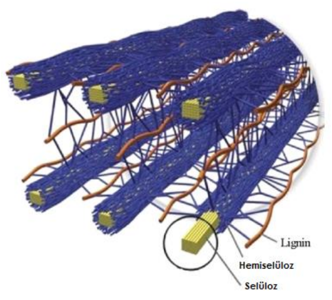

Figure 2. Structure of Cellulose with Hemicellulose and Lignin [15].

2.1. Alternative Uses of Lignin

Lignin is a renewable, non-toxic, a commercially available and low-cost natural resource that has a real potential of being used as a basic raw material for the polymer industry. Recent developments in the lignin - synthetic polymer and lignin -modified cellulose systems have shown an increased awareness of both the inherent limitations and the potential of the reactivity of lignin, and of the vast prospects of its application in thermoplastics, thermosets, adhesives, sealants; molding powders, rubbers, foams, films and many others [16]. The alternative uses of the lignin have been classified as follows.

1- As an energy source in the energy production field,
2- As binders in paper, sheets, etc production,
3- Disintegration and chemical conversion products: Vanillin, Dimethylsulfite and dimethylsulfoxide, Phenols and catechols, Benzene derivatives, Synthetic gas, etc.
4- Uses in macromolecules (solution systems): Dispersants, Emulsion stabilizers. Complex substances, coagulants, precipitators, etc. as.
5- Use of macromolecular or particulate materials in polymeric material systems: Thermoset resins, Rubber additives, Polyblends, Stabilizer applications (e.g. antioxidants) in bakelite production and production of packaging foams,
6- As matrix or other applications: Adsorption, Desorption, etc. As an ion exchanger,
7- Carbonization and pyrolysis applications: Activated carbon, Carbon or graphite fibers and foams in production processes,
8- Soil and fertilizer applications [17] It is available in a wide range of applications such as agricultural purposes.

Although lignin has so many alternative uses, it is unfortunately not effective and properly used. In factories, heat is recovered by burning in recovery units. This is the way lignin is used in the worst possible way and causing environmental problems. As a result of the literature searches, it is possible to draw lignin as carbon fiber raw material which is a technical fiber in textile fibers [4,7,19,26].

2.2. Preparation of Lignin as Textile Fiber

Lignin is drawn as a textile fiber by melt spinning method [26] and electrospinning method [18] in literature. Uraki et al used softwood lignin and hardwood lignin and they compared them. They used acetic acid in their experiments. In their study softwood acetic acid lignin free from a high-molecular-mass fraction could be spun at 220 ºC by a spinning machine equipped with an extruder [27]. Baker et al. schematized the attraction of lignin as carbon fiber in

Figure 3 [26]. It is seen in figure 3 that energy consumption increases step by step.

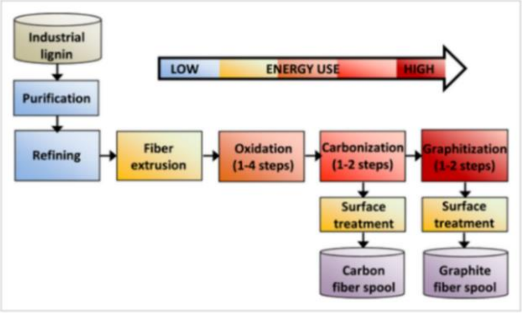

Figure 3. Lignin Carbon Fiber Extrusion [26].

In this study, the first step is making a test to lignin if it can be spinnable or not. There is some plasticizer to make a spinnable solution with lignin. Ethylene glycol was used as a plasticizer. The schematic drawing figure of a preparing blend is in figure 4. Blends are prepared in certain proportions and the solution is heated to 205 ⁰C under the air atmosphere. The fine-tipped object is dipped into the solution. If the solution shows thin elongation when withdrawn the fine-tipped as in figure 5, means that the solution is suitable for melt spinning.

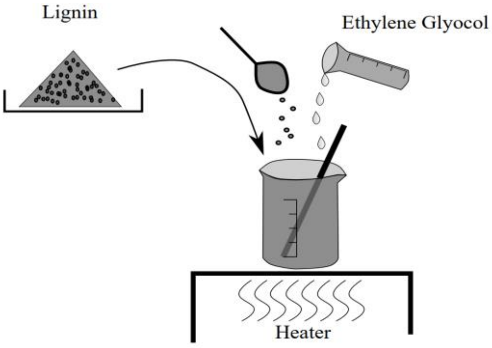

Figure 4. Schematic drawing figure of a preparing lignin-ethylene glycol blend.

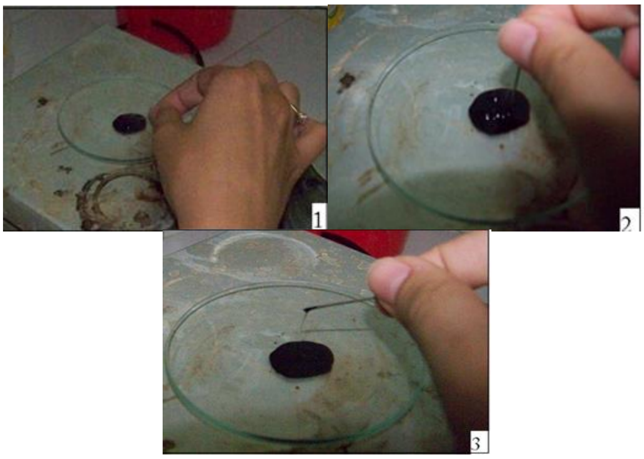

Figure 5. Spinnability test for lignin solutions

3. Materials and Methods

3.1. Materials

In the study, the main material was lignin, lignin solvents, and plasticizers were used to prepare the solution with lignin. Polyethylene glycol, sodium hydroxide, ethanol, methanol, acetic acid, acetone, and ZAG brand Polyvinyl alcohol polymer are used. The properties of the lignin used are given below.

- Lignin brand: INDULIN® AT kraft lignin.
- Physical property: Brown powder
- Volume : in free 26 lb/ft3, in package 32 lb/ft3
- Burning point: 198 ˚C
- Flash point: 176 ˚C
- Specific gravity: 1,3
- Surface tension (1% aqueous) : 43 mN/m
- Weight loss as a result of heating, in 149˚C : 3.3 %
- In 204 ˚C : 5.7%
- In 260 ˚C : 8.5%
- In 316 ˚C : 13.4%

Prepared solutions and blends are in Table 1. and Table 2.

Table 1. Solutions and Mixing Ratios.

Solution no	Lignin (gr)	Solvent	Solvent Amount
LÇ$_1$	1	Ethyl alcohol	10 ml
LÇ$_2$	2	Ethyl alcohol	10 ml
LÇ$_3$	2	Methanol	8gr
LÇ$_4$	2	Acetic acid	10 ml
LÇ$_5$	2	Acetic acid	5 ml
LÇ$_6$	2	Acetic acid	10 gr
LÇ$_7$	2	Acetone	8 gr
LÇ$_8$	2	Ethyl alcohol	7 gr
LÇ$_9$	1	Ethylene glycol	2.5 gr
LÇ$_{10}$	1.5	Ethylene glycol	3 ml
LÇ$_{11}$	1.5	20% NaOH	3 ml

Table 2. Blends and Mixing Ratios.

Blend	Lignin (gr)	Solvent	Solvent amount	%10PVA : Lignin
LB$_1$	1	Ethylene glycol	2 ml	2
LB$_2$	1.5	10% NaOH	3 ml	2
LB$_3$	1.5	Ethyl alcohol + Ethylene glycol	1ml + 1 ml	2
LB$_4$	1	Ethylene glycol	3 ml	2

3.2. Method

There are two methods which are used for lignin fiber production in this paper. One is electrospinning and the second method is a new experimental melt spinning method which is shown in figure 6 and figure 7.

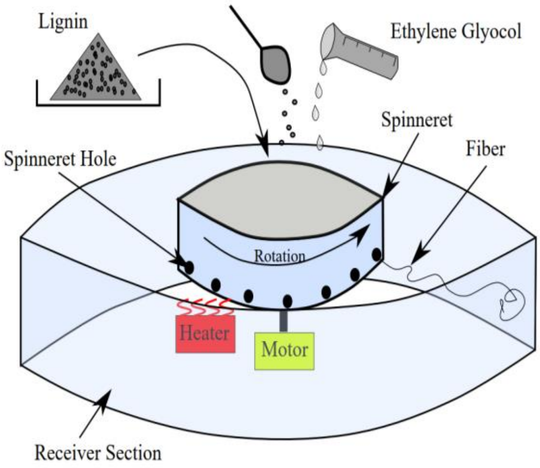

Figure. 6 Schematic Drawing of Experimental Melt Spinning Method.

In the new system, which was designed by modifying the fiber extraction method from the melt, the lignin was dissolved by dropping the solution, and as a result of the work of the experimental apparatus, the fiber was drawn by the rotation of the perforated chamber in the middle and the lignin in the middle coming out of the holes by the effect of centrifugal force. The solution was heated to 205ºC under air atmosphere with heater and Direct current(DC) motor has 12 Volt and 0.5 Ampere features and it runs 1000 revolution per minute (rpm).

Figure. 7 New Device Designed in the Scope of the Modification of Melt Fiber Extraction Method.

The apparatus in the new device, which was designed in the scope of the study by modifying the melt spinning method, consists of 4 parts;

1. Heater section
2. Perforated rotating hopper serving as a spinneret
3. Receiver section
4. The section that moves the system.

Figure 8 shows the exit of the lignin from the perforated chamber, which acts as a spinneret from the melt with the aid of a plasticizer.

Figure.8 The output of Lignin Fibers from the Perforated Chamber with Nozzle Function

In the electrospinning method, the solution is taken into the syringe and the fiber is drawn by DC generator and pump. Initially, attempts were made to form a viscous solution ready to be drawn in the electrospinning of the lignin polymer. In below Figure 9 shows the electrospinning fiber production device.

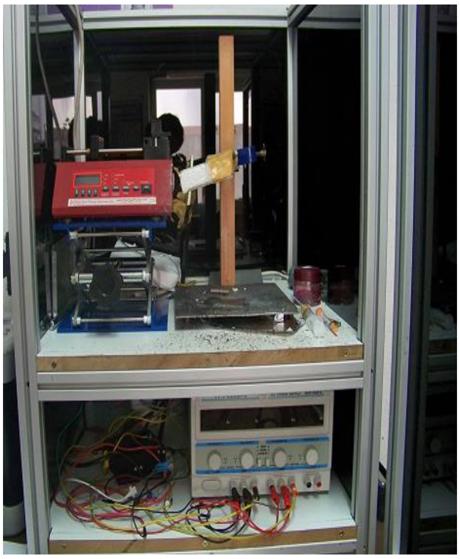

Figure. 9 Electrospinning Fiber Production Device.

Lignin blends were placed in a 5 mL syringe with a stainless-steel needle. The needle was connected to the DC high-voltage power supply, which generated DC voltages in the range of 0–30 kV. The electrospun lignin blend fibers were collected on to an aluminum foil connected to the ground. The feeding rate of the syringe was 9μL/h and applied voltage of 22 kV and tip-to-collector distance of 10 cm.

4. Experimental Results

The solutions and blends are prepared in the scope of the study are given below:

- LÇ$_1$: The solution was not suitable for electrospinning, coffee consistency. Since no viscous solution was formed for electrospinning, drop formation was observed in electrospinning.
- LÇ$_2$: The solution was not suitable for electrospinning, coffee consistency. Since no viscous solution was formed for electrospinning, drop formation was observed in electrospinning.
- LÇ$_3$: It was observed that the solution was formed in some form in solution and that no suitable solution was formed for electrospinning.
- LÇ$_4$: The solution was formed but it was observed that the viscosity is not suitable for electrospinning.
- LÇ$_5$: A viscous solution was formed, but the fibers could not be drawn in the electrospinning.
- LÇ$_6$: A viscous solution was formed, but the fibers could not be drawn in the electrospinning.
- LÇ$_7$: A coffee-like solution was formed. It did not occur suitable for electrospinning.
- LÇ$_8$: A coffee-like solution was formed. It did not occur suitable for electrospinning.
- LÇ$_9$: A viscous solution was formed, but the fibers could not be drawn in the electrospinning.
- LÇ$_{10}$: A viscous solution suitable for electrospinning occurs.
- LÇ$_{11}$: Dense solution was formed for electrospinning.
- LB$_1$: Electrospinning occurred that could be drawn with a nice blend.
- LB$_2$: Electrospinning occurred that could be drawn with a nice blend.
- LB$_3$: Electrospinning occurred that could be drawn with a nice blend.
- LB$_4$: Electrospinning occurred that could be drawn with a nice blend.

4.1. Scanning Electron Microscopy (SEM) Images of fibers formed after electrospinning

The purpose of the study is to investigate the lignin as a textile fiber. All solutions did not form a fiber. Only LÇ $_{11}$ solution and LB$_1$, LB$_2$, LB$_3$, and LB$_4$ formed a fiber with the electrospinning process. SEM images of the solutions and blend that showed fiber formation during the fiber spinning process are as follows.

LÇ$_{11}$: The solution was included lignin and 20% NaOH. Proportions of the solution were already given in Table 1. The solution was dense for electrospinning. Fiber formation with a diameter of 2.52 μm was observed after electrospinning. SEM image of it is seen in figure 10.

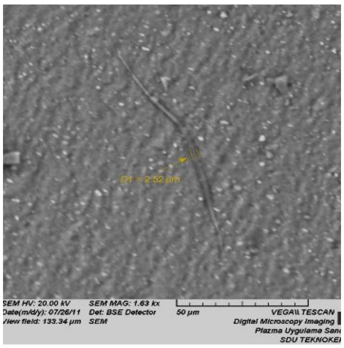

Figure 10. SEM image of LÇ$_{11}$ Electrospinning Fiber Formation.

LB$_1$: Blend was included 10% PVA, lignin, and ethylene glycol as a plasticizer for lignin. Electrospinning occurred. Fiber formation was observed in SEM images after electrospinning from this blend. Obtained fiber diameters were measured as 0.52μm, 0.78μm, 0.82μm, 1.14μm. SEM images of the formed fibers can be seen in figure 11.

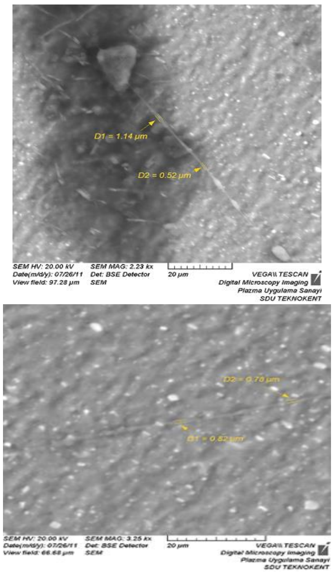

Figure 11. After electrospinning LB1 SEM Image.

LB$_2$: Blend was included PVA, lignin, and 10% NaOH as a plasticizer for lignin. Electrospinning occurred. Fiber formation was observed in SEM images after electrospinning from this blend. It is seen in Figure 12. Fiber diameters were 1.95 μm, 2.09 μm, and 2.22 μm.

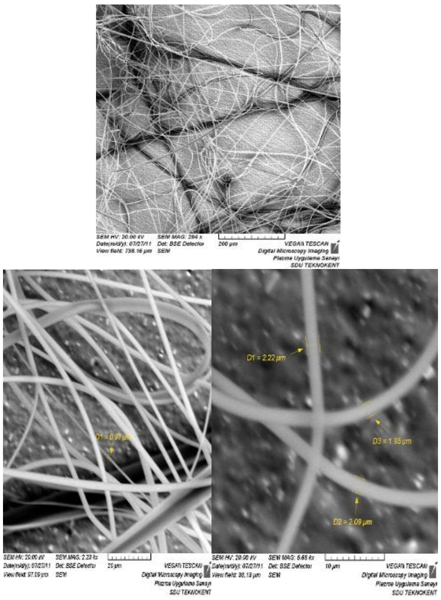

Figure 12. After electrospinning LB2 SEM Image.

LB$_3$: Blend was included PVA, lignin and Ethyl alcohol and Ethylene glycol. Electrospinning occurred. Fiber formation was observed in SEM images after electrospinning from this blend. Fiber diameters are seen in Figure 13. They are 0.61 µm, 0.69 µm and 2.22 µm.

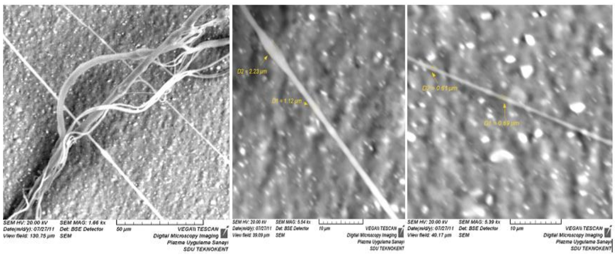

Figure 13. After electrospinning LB3 SEM Image.

LB$_4$: Blend was included PVA, lignin, and ethylene glycol as a plasticizer for lignin. Electrospinning occurred. Fiber formation was observed in SEM images after electrospinning from this blend. It is seen in Figure 14. Fiber diameters were 0.21 µm, 1.78 µm, and 2.38 µm.

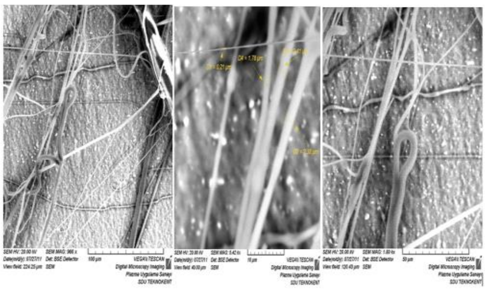

Figure 14. After electrospinning LB4 Sem Image.

4.1.1. Fiber image is taken from the system obtained by modifying the melt fiber extraction method

In the study, after the examination of the drawability of lignin by electrospinning, the spinnability of the lignin by the melt fiber extraction method was also examined. The solution was formed by mixing lignin and ethylene glycol in certain proportions. The solution was heated to 205ºC and fiber outputs were observed under the air atmosphere with 1000 rpm spinneret. Obtained fiber is shown in figure 15.

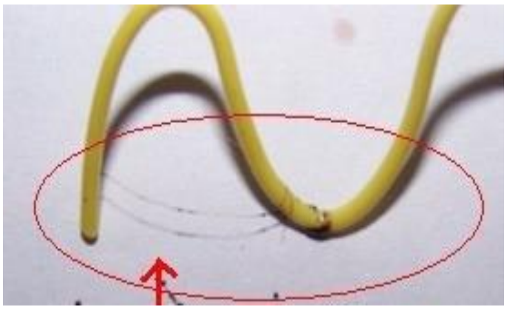

Figure.15 Lignin Fiber from the spinneret.

5. Results and Discussion

As a result of the literature studies, it was aimed to produce the high performance carbon fibers obtained from raw materials such as polyacrylonitrile, pitch, rayon, lyocell but due to high production costs, it is aimed to use a material which is in waste position for paper industry such as lignin in carbon fiber production. Studies so far and the fiber diameters of the fibers produced in the study are given in Table 3.

Table 3. Comparison of Carbon Fiber Raw Materials and Production Methods with Fiber Diameters.

Raw material	Fiber diameter	Production Method	References
Polyacrylonitrile	40-100	Wet spinning	[19]
Lignin	100	Melt spinning	[24]
Polyacrylonitrile	5-10	Melt spinning	[28]
Pitch	10-11	Melt spinning	[29]
Rayon	5-10	Melt spinning	[30]
LÇ$_{11}$	2.52	Electrospinning	Study
LB$_1$	0.52-1.14	Electrospinning	Study
LB$_2$	1-2.5	Electrospinning	Study
LB$_3$	0.6-2.25	Electrospinning	Study
LB$_4$	0.2-2.38	Electrospinning	Study

Within the scope of the study, fiber extraction difficulties with the electrospinning method with direct solvents of brown powder polymer lignin have not been overcome yet. Fiber withdrawal was observed in 20% NaOH solution of lignin. Fiber formation was not observed with other solvents. The finest fiber diameter in the blends formed by PVA polymer by lignin solutions is 0.21µm and microfiber can be produced with 2.5µm diameter PVA blends. Compared with fiber diameters finer fibers by electrospinning spinnability was seen.

6. Conclusion

Lignin is a waste for the paper industry and it is burned in the heat recovery units in factories. This method causes environmental problems. Instead of being burned down, using as a raw material alternative for carbon fiber will contribute to the environment and economy.
In this study, the first step was testing to lignin if it can be spinnable or not. There is some plasticizer to make a spinnable solution with lignin. Blends were prepared in certain proportions and the solution is heated to 205 ^{0}C under the air atmosphere. Lignin got the best spinnability feature with ethylene glycol. Two methods were used for lignin fiber production. One was electrospinning and the second method was a new experimental melt spinning. Waste for the paper industry will be a renewable and sustainable resource for the textile industry with the advancement of these studies.

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Authors' Contributions

All authors contributed equally to this manuscript and all authors reviewed the final manuscript.

Ethics

There are no ethical issues after the publication of this manuscript.

References

[1]. M. Demir *et al.*, "Graphitic biocarbon from metal-catalyzed hydrothermal carbonization of lignin," *Industrial & Engineering Chemistry Research*, vol. 54, no. 43, pp. 10731–10739, 2015.

[2]. S. Bilek, A. Y. Melikoğlu, and S. Cesur, "Tarımsal Atıklardan Selüloz Nanokristallerinin Eldesi, Karakteristik Özellikleri ve Uygulama Alanları," *Akademik Gıda*, vol. 17, no. 1, pp. 140–148.

[3]. N. KOLANKAYA and N. SAĞLAM, "Biyodelignifikasyon ve biyoteknolojik önemi," *Hacettepe Üniversitesi Eğitim Fakültesi Dergisi*, vol. 3, no. 3, 1988.

[4]. J. F. Kadla, S. Kubo, R. A. Venditti, R. D. Gilbert, A. L. Compere, and W. Griffith, "Lignin-based carbon fibers for composite fiber applications," *Carbon*, vol. 40, no. 15, pp. 2913–2920, 2002.

[5]. M. Zhang and A. A. Ogale, "Carbon fibers from dry-spinning of acetylated softwood kraft lignin," *Carbon*, vol. 69, pp. 626–629, Apr. 2014, doi: 10.1016/j.carbon.2013.12.015.

[6]. K. Sudo, K. Shimizu, N. Nakashima, and A. Yokoyama, "A new modification method of exploded lignin for the preparation of a carbon fiber precursor," *Journal of Applied Polymer Science*, vol. 48, no. 8, pp. 1485–1491, 1993, doi: 10.1002/app.1993.070480817.

[7]. H. Mainka *et al.*, "Lignin – an alternative precursor for sustainable and cost-effective automotive carbon fiber," *Journal of Materials Research and Technology*, vol. 4, no. 3, pp. 283–296, Jul. 2015, doi: 10.1016/j.jmrt.2015.03.004.

[8]. I. Norberg, "Carbon Fibres from Kraft Lignin," 2012, Accessed: Nov. 22, 2019. [Online]. Available: http://urn.kb.se/resolve?urn=urn:nbn:se:kth:diva-92256.

[9]. J. Hayashi, A. Kazehaya, K. Muroyama, and A. P. Watkinson, "Preparation of activated carbon from lignin by chemical activation," *Carbon*, vol. 38, no. 13, pp. 1873–1878, Jan. 2000, doi: 10.1016/S0008-6223(00)00027-0.

[10]. M. Demir *et al.*, "Lignin-derived heteroatom-doped porous carbons for supercapacitor and CO2 capture applications," *International Journal of Energy Research*, vol. 42, no. 8, pp. 2686–2700, 2018.

[11]. Adıgüzel Ali Osman, "LİGNOSELÜLOZİK BİYOKÜTLEDEN BİYOETANOL ÜRETİMİ." Jan. 2011.

[12]. A. B. Hazar Yoruç and V. Uğraşkan, "Green Polymers and Applications," *Afyon Kocatepe University Journal of Sciences and Engineering*, vol. 17, no. 1, pp. 318–337, Mar. 2017, doi: 10.5578/fmbd.53940.

[13]. P. Bajpai, "Lignin," in *Carbon Fibre from Lignin*, Singapore: Springer Singapore, 2017, pp. 11–15.

[14]. Suhas, P. J. M. Carrott, and M. M. L. Ribeiro Carrott, "Lignin – from natural adsorbent to activated carbon: A review," *Bioresource Technology*, vol. 98, no. 12, pp. 2301–2312, Sep. 2007, doi: 10.1016/j.biortech.2006.08.008.

[15]. W. O. S. Doherty, P. Mousavioun, and C. M. Fellows, "Value-adding to cellulosic ethanol: Lignin polymers," *Industrial Crops and Products*, vol. 33, no. 2, pp. 259–276, Mar. 2011, doi: 10.1016/j.indcrop.2010.10.022.

[16]. T. Q. Hu, *Chemical modification, properties, and usage of lignin*. Springer, 2002.

[17]. S. Huş, "Lignin kullanış yerleri," *Journal of the Faculty of Forestry Istanbul University| İstanbul Üniversitesi Orman Fakültesi Dergisi*, pp. 15–23, 1971.

[18]. R. Ding, H. Wu, M. Thunga, N. Bowler, and M. R. Kessler, "Processing and characterization of low-cost electrospun carbon fibers from organosolv lignin/polyacrylonitrile blends," *Carbon*, vol. 100, pp. 126–136, Apr. 2016, doi: 10.1016/j.carbon.2015.12.078.

[19]. E. Frank, L. M. Steudle, D. Ingildeev, J. M. Spörl, and M. R. Buchmeiser, "Carbon Fibers: Precursor Systems, Processing, Structure, and Properties," *Angewandte Chemie International Edition*, vol. 53, no. 21, pp. 5262–5298, May 2014, doi: 10.1002/anie.201306129.

[20]. J. F. Kadla, S. Kubo, R. D. Gilbert, and R. A. Venditti, “Lignin-based carbon fibers,” in *Chemical Modification, Properties, and Usage of Lignin*, Springer, 2002, pp. 121–137.

[21]. A. L. Compere, W. L. Griffith, C. F. Leitten Jr, and S. Petrovan, “Improving the fundamental properties of lignin-based carbon fiber for transportation applications,” in *Proceedings of the 36th International SAMPE Technical Conference*, 2004, pp. 2246–2254.

[22]. A. G. Dumanlı and A. H. Windle, “Carbon fibres from cellulosic precursors: a review,” *Journal of Materials Science*, vol. 47, no. 10, pp. 4236–4250, May 2012, doi: 10.1007/s10853-011-6081-8.

[23]. S. P. Maradur, C. H. Kim, S. Y. Kim, B.-H. Kim, W. C. Kim, and K. S. Yang, “Preparation of carbon fibers from a lignin copolymer with polyacrylonitrile,” *Synthetic Metals*, vol. 162, no. 5–6, pp. 453–459, Apr. 2012, doi: 10.1016/j.synthmet.2012.01.017.

[24]. I. Norberg, *Carbon fibres from kraft lignin*. Stockholm: Chemical Science and Engineering, Royal Institute of Technology (KTH), 2012.

[25]. Omid Hosseinaei, David Harper, Joseph Bozell, and Timothy Rials, “Improving Processing and Performance of Pure Lignin Carbon Fibers through Hardwood and Herbaceous Lignin Blends,” *International Journal of Molecular Sciences*, vol. 18, no. 7, p. 1410, Jul. 2017, doi: 10.3390/ijms18071410.

[26]. D. A. Baker and T. G. Rials, “Recent advances in low-cost carbon fiber manufacture from lignin,” *Journal of Applied Polymer Science*, vol. 130, no. 2, pp. 713–728, Oct. 2013, doi: 10.1002/app.39273.

[27]. Y. Uraki, A. Nakatani, S. Kubo, and Y. Sano, “Preparation of activated carbon fibers with large specific surface area from softwood acetic acid lignin,” *Journal of Wood Science*, vol. 47, no. 6, pp. 465–469, 2001.

[28]. “Carbon Fiber Composite Materials | TORAY.” http://www.toray.com/products/prod_004.html (accessed Feb. 10, 2018).

[29]. “Carbon Fiber | CYTEC SOLVAY GROUP.” http://www.cytec.com/businesses/aerospace-materials/products/carbon-fiber (accessed Feb. 10, 2018).

[30]. S. Peng, H. Shao, and X. Hu, “Lyocell fibers as the precursor of carbon fibers,” *Journal of applied polymer science*, vol. 90, no. 7, pp. 1941–1947, 2003.