

ISSN 2518-1483 (Online),
ISSN 2224-5227 (Print)

**ACADEMIC JOURNAL
OF PHYSICAL AND CHEMICAL SCIENCES**

**№3
2026**

ISSN 2518-1483 (Online),
ISSN 2224-5227 (Print)

2026 • 3



ACADEMIC JOURNAL OF PHYSICAL AND CHEMICAL SCIENCES

PUBLISHED SINCE JANUARY 1944

ALMATY, NAS RK

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ACADEMIC JOURNAL OF PHYSICAL AND CHEMICAL SCIENCES.

ISSN 2518-1483 (Online), ISSN 2224-5227 (Print)

Owner: «Central Asian Academic Research Center» LLP (Almaty).

The certificate of registration of a periodical printed publication in the Committee of Information of the Ministry of Information and Social Development of the Republic of Kazakhstan № **KZ93VPY00121157** issued **05.06.2025**

Thematic scope: *physics and chemistry*.

Periodicity: 4 times a year.

<http://reports-science.kz/index.php/en/archive>

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ACADEMIC JOURNAL OF PHYSICAL AND CHEMICAL SCIENCES

ISSN 2518-1483 (Online), ISSN 2224-5227 (Print)

Меншіктеуші: «Орталық Азия академиялық ғылыми орталығы» ЖШС (Алматы қ.).

Ақпарат агенттігінің мерзімді баспасөз басылымын, ақпарат агенттігін және желілік басылымды қайта есепке қою туралы ҚР Мәдениет және Ақпарат министрлігі «Ақпарат комитеті» Республикалық мемлекеттік мекемесі 05.06.2025 ж. берген № KZ93VPY00121157 Куәлік.

Тақырыптық бағыты: физика, химия.

Мерзімділігі: жылына 4 рет.

<http://reports-science.kz/index.php/en/archive>

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ACADEMIC JOURNAL OF PHYSICAL AND CHEMICAL SCIENCES

ISSN 2518-1483 (Online), ISSN 2224-5227 (Print)

Собственник: ТОО «Центрально-азиатский академический научный центр» (г. Алматы).

Свидетельство № KZ93VPY00121157 о повторной регистрации периодического печатного издания информационного агентства, информационного агентства и сетевого издания, выданное Республиканским государственным учреждением «Комитет информации» Министерства культуры и информации Республики Казахстан **05.06.2025**Тематическая направленность: *физика, химия*.

Периодичность: 4 раза в год.

<http://reports-science.kz/index.php/en/archive>

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ACADEMIC JOURNAL
OF PHYSICAL AND CHEMICAL SCIENCES
ISSN 2224-5227
Volume 3.
Number 359 (2026), 475-497

<https://doi.org/10.32014/2026.2518-1483.488>

UDC: 66.0
IRSTI: 61.67.33

©Talгатkyzy A.*, Nazhipkyzy M., Otenov A., Seitkazinova A.,
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WOOD WASTE-DERIVED ORGANOSOLV LIGNIN-BASED CARBON FIBERS FOR ENERGY STORAGE APPLICATIONS

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Abstract. This study examined the feasibility of recycling wood waste to produce organosolv lignin suitable as a precursor for carbon fibers. Mixed wood waste, pine, and elm sawdust were used as feedstock. Lignin was isolated using an organosolv method with a mixture of formic and acetic acids, which reduced the use of aggressive reagents and facilitated environmentally friendly biomass processing. The structural, morphological, and thermal properties of the resulting lignin samples were analyzed using scanning electron microscopy, infrared spectroscopy, nuclear magnetic resonance spectroscopy, thermogravimetric analysis, and Raman spectroscopy. The isolated lignin contained aromatic, phenolic, hydroxyl, methoxyl, and methyl functional groups. Nuclear magnetic resonance spectroscopy revealed a predominance of syringyl structural fragments over guaiacyl ones, indicating favorable thermal stability for subsequent carbonization. Composite fibers were fabricated from the resulting lignin using polyacrylonitrile as the electrospinning polymer. The influence of the spinning solution composition and heat treatment conditions on the fiber morphology and structure was studied. A lignin to polyacrylonitrile ratio of 30:70 proved optimal, ensuring the production of continuous, defect-free fibers with an average diameter of approximately 97 nm. After carbonization

at 800 °C under a nitrogen atmosphere, carbon nanofibers with an average diameter of approximately 47 nm and a developed porous structure were obtained. These results demonstrate the potential of organosolv lignin obtained from wood waste as a renewable precursor for carbon nanofibers and electrode materials for energy storage systems. The carbonized fibers exhibited a developed specific surface area of 549.7 m²/g, while the sample carbonized at 800 °C showed the highest electrochemical performance, with a specific capacitance of 136.4 F/g for supercapacitors and a discharge capacity of 797 mAh/g for lithium-ion batteries.

Keywords: lignin-based carbon fibers, electrospinning, energy storage systems, lithium-ion batteries, supercapacitors

Funding: *This research was conducted as part of the targeted funding program of the Science Committee of the Ministry of Education and Science of the Republic of Kazakhstan under Grant AP32721721, titled “Synthesis of lignin-based composite carbon nanofibers for lithium-ion battery electrodes.”*

For citations: Talgatkyzy A., Nazhipkyzy M., Otenov A., Seitkazanova A., Issanbekova A. Wood Waste-Derived Organosolv Lignin-Based Carbon Nanofibers For Energy Storage Applications. 2026. No.3. Pp. 475-497. DOI: <https://doi.org/10.32014/2026.2518-1483.488>

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АҒАШ ҚАЛДЫҚТАРЫНАН АЛЫНҒАН ОРГАНОСОЛЬВ ЛИГНИН НЕГІЗІНДЕГІ КӨМІРТЕКТІ ТАЛШЫҚТАРДЫ ЭНЕРГИЯ САҚТАУ ЖҮЙЕЛЕРІНДЕ ҚОЛДАНУ

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Аннотация. Бұл зерттеуде көміртекті талшықты материалдарға арналған прекурсор ретінде пайдалануға жарамды органосольв лигнинін алу үшін

ағаш қалдықтарын өңдеудің мүмкіндігі зерттелді. Шикізат ретінде аралас ағаш қалдықтары, қарағай және қарағаш үгінділері пайдаланылды. Лигнин құмырсқа және сірке қышқылдарының қоспасын қолданатын органосольв әдісімен бөлініп алынды, бұл агрессивті реагенттерді пайдалануды азайтып, биомассаны экологиялық қауіпсіз өңдеуге мүмкіндік береді. Алынған лигнин үлгілерінің құрылымдық, морфологиялық және термиялық қасиеттері сканерлеуші электрондық микроскопия, инфрақызыл спектроскопия, ядролық магниттік резонанс спектроскопиясы, термогравиметриялық талдау және Раман спектроскопиясы арқылы зерттелді. Бөлініп алынған лигнин құрамында ароматты, фенолдық, гидроксилдік, метоксильдік және метильдік функционалдық топтар бар екені анықталды. Ядролық магниттік резонанс спектроскопиясының деректері гваяцилдік құрылымдық фрагменттерге қарағанда синрингилдік фрагменттердің басым екенін көрсетті, бұл кейінгі карбонизация үшін қолайлы термиялық тұрақтылықты көрсетеді. Алынған лигнин негізінде иіру полимері ретінде полиакрилонитрилді пайдаланып, электроспиннинг әдісімен композиттік талшықтаралынды. Иіруерітіндісінің құрамымен термиялық өңдеу жағдайларының талшықтардың морфологиясы мен құрылымына әсері зерттелді. Лигнин мен полиакрилонитрилдің 30:70 қатынасы оңтайлы болып, орташа диаметрі шамамен 97 нм болатын үздіксіз, ақаусыз талшықтардың түзілуін қамтамасыз етті. Азот атмосферасында 800 °C температурада карбонизациядан кейін орташа диаметрі шамамен 47 нм және дамыған кеуекті құрылымы бар көміртекті наноталшықтар алынды. Нәтижелер ағаш қалдықтарынан алынған органосольв лигнинінің көміртекті наноталшықтар мен энергия сақтау жүйелеріне арналған электродтық материалдар алу үшін жаңартылатын прекурсор ретіндегі перспективалылығын көрсетеді. Карбонизациядан кейін алынған көміртекті талшықтар 549,7 м²/г болатын дамыған меншікті беткі ауданымен сипатталды, ал 800 °C температурада карбонизацияланған үлгі ең жоғары электрохимиялық көрсеткіштерді көрсетті: суперконденсаторлар үшін 136,4 Ф/г меншікті сыйымдылық және литий-ионды батареялар үшін 797 мА·сағ/г разрядтық сыйымдылық.

Түйін сөздер: лигнин негізіндегі көміртекті талшықтар, электроспиннинг, энергия сақтау жүйелері, литий-ионды батареялар, суперконденсаторлар

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УГЛЕРОДНЫЕ ВОЛОКНА НА ОСНОВЕ ОРГАНСОЛЬВ ЛИГНИНА, ПОЛУЧЕННОГО ИЗ ДРЕВЕСНЫХ ОТХОДОВ, ДЛЯ ПРИМЕНЕНИЯ В СИСТЕМАХ ХРАНЕНИЯ ЭНЕРГИИ

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Аннотация. В данном исследовании изучена возможность переработки древесных отходов для получения органосольв лигнина, пригодного в качестве прекурсора для углеродных волокнистых материалов. В качестве сырья использовали смешанные древесные отходы, опилки сосны и опилки вяза. Лигнин выделяли органосольв-методом с использованием смеси муравьиной и уксусной кислот, что позволяет снизить использование агрессивных реагентов и способствует экологически безопасной переработке биомассы. Структурные, морфологические и термические свойства полученных образцов лигнина исследовали с использованием сканирующей электронной микроскопии, инфракрасной спектроскопии, спектроскопии ядерного магнитного резонанса, термогравиметрического анализа и Раман-спектроскопии. Выделенный лигнин содержал ароматические, фенольные, гидроксильные, метоксильные и метильные функциональные группы. Данные спектроскопии ядерного магнитного резонанса выявили преобладание сиригильных структурных фрагментов над гваяцильными, что указывает на благоприятную термическую стабильность для последующей карбонизации. На основе полученного лигнина методом электроспиннинга были изготовлены композитные волокна с использованием полиакрилонитрила в качестве полимера для формования. Исследовано влияние состава прядильного раствора и условий термической обработки на морфологию и структуру волокон. Оптимальным оказалось соотношение лигнина и полиакрилонитрила 30:70, обеспечивающее формирование непрерывных бездефектных волокон со средним диаметром около 97 нм. После карбонизации при 800 °С в атмосфере азота были получены углеродные нановолокна со средним диаметром около 47 нм и развитой пористой структурой. Полученные результаты демонстрируют перспективность органосольв лигнина из древесных отходов в качестве возобновляемого прекурсора для получения углеродных нановолокон и электродных материалов для систем хранения энергии. Углеродные волокна после карбонизации характеризовались развитой удельной поверхностью 549,7 м²/г, при этом образец, карбонизированный при 800 °С, продемонстрировал наилучшие электрохимические характеристики: удельную ёмкость 136,4 Ф/г для суперконденсаторов и разрядную ёмкость 797 мА·ч/г для литий-ионных батарей.

Ключевые слова: углеродные волокна на основе лигнина, электроспиннинг, системы хранения энергии, литий-ионные батареи, суперконденсаторы.



Introduction. Global climate change, depletion of fossil resources, and the increasing demand for sustainable energy technologies have accelerated the search for environmentally friendly functional materials. In recent years, considerable attention has been focused on renewable biomass-derived materials capable of reducing environmental impacts while maintaining high technological performance. In this context, carbon materials obtained from renewable resources have emerged as promising candidates for advanced energy storage applications.

Carbon occupies a special place among the elements in nature and is characterized by a variety of allotropes and functional properties. Carbon occurs in various structural forms, such as graphite, diamond, fullerenes, carbon nanotubes, and nanofibers, each of which has unique physicochemical properties (Shokrani Havigh and Mahmoudi Chenari, 2022). Carbon materials are of particular interest due to their high electrical conductivity, thermal and chemical stability, mechanical strength, low weight, and flexibility for functionalization (Zhu et al., 2025). These properties are especially important for electrochemical energy storage systems, where electrode materials require high conductivity, structural stability, and efficient ion transport. For this reason, research has increasingly focused on synthesizing carbon materials not only from traditional raw materials but also from biomass, polymers, and industrial waste (Hossen et al., 2026; Wang et al., 2026; Jang et al., 2022).

One of the important structures among carbon materials is carbon fibers, which are characterized by high modulus, mechanical strength, corrosion resistance, and high electrical and thermal conductivity (Jiao et al., 2025). In recent years, carbon nanofibers obtained by electrospinning have attracted particular attention. This method allows fibers with nanometer-scale diameters to be obtained from polymer solutions or melts under a high-voltage electric field. The resulting fibers are characterized by a high specific surface area, controllable porosity, and nanoscale structure (Jeon et al., 2025). These properties make electrospun carbon nanofibers promising electrode materials for electrochemical energy storage devices, particularly lithium-ion batteries and supercapacitors (Sun et al., 2025).

One important approach to producing carbon fibers is the use of biomass-based precursors. Lignocellulosic biomass is considered a promising raw material because of its abundance, renewability, and low environmental impact. Lignocellulose is the main structural component of plants and consists primarily of cellulose, hemicellulose, and lignin. Among these components, lignin is the main aromatic polymer and contains a high proportion of carbon.

Lignin is one of the most widely distributed but least efficiently utilized biopolymers. Globally, 50-70 million tons of lignin are produced annually, but about 95% is burned for energy production or used as a low-value fuel (Guo et al., 2025). Such utilization limits the economic value of lignin and contradicts modern strategies for biomass valorization and sustainable resource management. However, its carbon-rich aromatic structure makes lignin a promising precursor for carbon materials, particularly carbon fibers (Di Martino et al., 2025; Cao et al., 2024). Therefore, converting lignin into high-

value carbon nanofibers represents an important approach for both resource utilization and sustainable electrode development.

Among the different types of lignin, organosolv lignin is particularly attractive for carbon fiber production. Organosolv lignin is relatively pure, sulfur-free, and has a low molecular weight, making it suitable for electrospinning (Parot et al., 2023). In contrast, kraft lignin has a highly condensed structure and may be limited in some applications because of its sulfur content (Karppi et al., 2025). These differences in lignin structure and composition can directly affect the morphology, porosity, conductivity, and electrochemical properties of the resulting carbon fibers (Li et al., 2025).

One of the most effective methods for converting lignin into carbon fibers is electrospinning (Su et al., 2026). Lignin-based fibers obtained by this method are subsequently converted into carbon nanofibers through stabilization and carbonization. However, the low molecular weight and insufficient viscosity of pure lignin prevent its direct electrospinning (Yan et al., 2025). Therefore, lignin is often blended with polymers such as polyacrylonitrile (PAN), polyethylene oxide (PEO), or polyvinyl alcohol (PVA), or subjected to chemical modification (Dai et al., 2025; Martínez Cánovas et al., 2026; Desidério et al., 2026). These limitations remain important challenges for the large-scale fabrication of lignin-derived carbon fibers.

In recent years, lignin-based carbon fibers have attracted increasing attention as electrode materials for advanced energy storage devices, particularly lithium-ion batteries and supercapacitors. Their high specific surface area, porous structure, and ability to be modified with heteroatoms can improve their electrochemical characteristics (Sun et al., 2025). For lithium-ion batteries, the electrical conductivity, structural stability, pore structure, and surface chemistry of carbon fibers are important for lithium-ion storage and transport. In supercapacitors, high specific surface area, accessible pores, and good electrical conductivity are essential for efficient charge storage and rapid ion transport.

The main advantages of lignin-based carbon fibers include their renewable origin, low cost, environmental benefits, and high carbon content. Among lignin types, organosolv lignin is particularly promising because of its relatively high purity and suitability for electrospinning. Despite these advantages, challenges associated with lignin heterogeneity, limited electrospinnability, structural changes during thermal treatment, and electrochemical performance remain. Therefore, further research on precursor composition, electrospinning, and thermal treatment is necessary to develop organosolv lignin-derived carbon nanofibers with improved performance for lithium-ion batteries and supercapacitors.

Materials and Methods. Pine and elm sawdust collected from furniture manufacturing plants in Almaty were used as lignocellulosic raw materials for lignin extraction. Polyacrylonitrile (PAN, molecular weight 100,000 g/mol, Sigma-Aldrich) was used as a spinning polymer to produce fibrous materials. Dimethylformamide (DMF, 99.8%, Sigma-Aldrich) was used as a solvent for preparing spinning solutions.

The morphology of the isolated lignin and electrospun lignin/PAN fibers was studied using scanning electron microscopy. Lignin samples were examined using a Quanta

200i 3D microscope (FEI, USA), and the morphological features of the resulting fibers were further analyzed using a JEOL JSM-6490LA scanning electron microscope.

The chemical composition and functional groups of the isolated lignin were determined using Fourier transform infrared spectroscopy on a PerkinElmer Spectrum 65 spectrometer. Spectra were recorded in the range of 450-4000 cm^{-1} at a resolution of 4 cm^{-1} . The obtained data were used to identify characteristic functional groups and assess the purity of the isolated lignin.

The structural features of the carbonized fibers were studied using Raman spectroscopy on a Solver Spectrum (NT-MDT) spectrometer using a 473 nm laser. The signal accumulation time was 30 s. Analysis of the characteristic D- and G-bands allowed us to evaluate the degree of ordering of the carbon structure and changes in graphitization after heat treatment.

The thermal stability and degradation characteristics of the lignin/PAN fibers were studied using thermogravimetric analysis. Measurements were carried out under a nitrogen atmosphere over a temperature range from room temperature to 1000°C at a heating rate of 20°C/min.

The molecular structure of the isolated lignins was further studied using proton nuclear magnetic resonance spectroscopy (^1H NMR) on a JNM-ECA Jeol400 spectrometer operating at 399.78 MHz. DMSO- d_6 was used as a solvent.

The combined use of SEM, IR spectroscopy, Raman spectroscopy, TGA, and ^1H NMR allowed us to characterize the morphology, chemical structure, thermal behavior, and structural changes of lignin and the carbon fibers obtained from it.

Extraction of lignin from wood waste using the organosolv method. Lignin was isolated from wood waste using the organosolv method, using a mixture of formic and acetic acids. Pine, elm, and mixed wood waste sawdust were used as feedstock. An 8.5-gram sample of sawdust was placed in a reaction vessel, after which a mixture of formic and acetic acids in a 70:30 volume ratio was added. The reaction mixture was maintained with constant stirring at 115°C for 2.5 hours.

During the process, the lignocellulosic matrix of the wood gradually degraded, and the lignin dissolved. After completion of the process, the reaction mixture was cooled to room temperature, after which the insoluble residue was separated by filtration. To precipitate lignin, distilled water was added to the resulting extract in a 5:1 volume ratio, and the mixture was heated to 105°C. Dilution with water resulted in the formation and subsequent precipitation of lignin particles.

After 30 minutes of holding time, the resulting precipitate was separated by centrifugation at 5000 rpm for 5 minutes. The precipitated lignin was washed repeatedly with distilled water until a neutral pH was achieved, then dried at room temperature for 24 hours. To remove residual moisture, the samples were further held in a vacuum drying oven at 60°C for 1 hour (Figure 1).

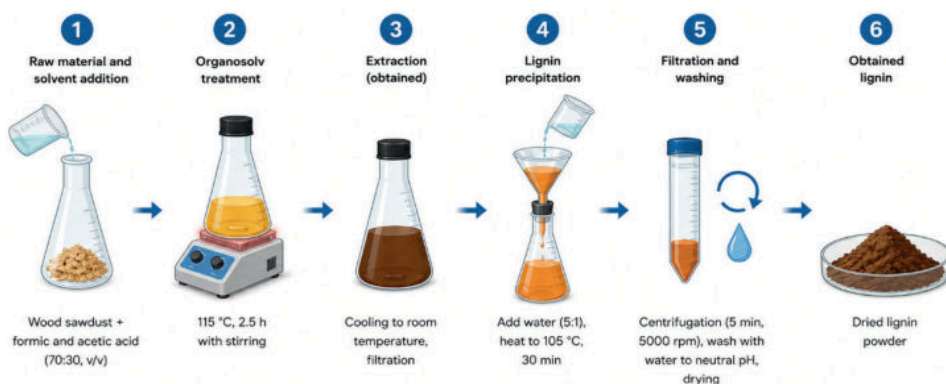


Figure 1. The process of obtaining lignin from wood waste.

The use of the organosolv process produces lignin that is virtually free of sulfur compounds and residual carbohydrates, which is an important advantage for its subsequent processing into carbon fiber materials.

As a result of the process, three samples of organosolv lignin were obtained: from mixed wood waste with a yield of 19.3%, from elm wood with 18.0%, and from pine sawdust with 16.7%. To evaluate the structure, morphology, and physicochemical properties, the obtained samples were analyzed using scanning electron microscopy, infrared spectroscopy, thermogravimetric analysis, and proton NMR spectroscopy.

Production of Lignin-Based Fibers by Electrospinning. Lignin-based composite fibers were produced by electrospinning using a NanoFiber laboratory setup (AME Energy, Shenzhen, China, model AME-11). The general schematic of the experimental setup is shown in Figure 2.

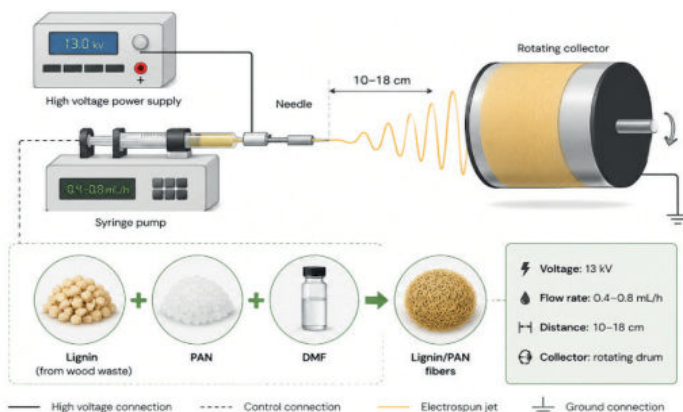


Figure 2. Diagram of the experimental installation for electrospinning.

During fiber formation, the operating voltage was maintained at 13 kV. The spinning solution feed rate was varied between 0.4 and 0.8 ml/h, and the distance between

the capillary and collector was varied from 100 to 180 mm. The formed fibers were deposited on a rotating drum, which served as a collector.

Polyacrylonitrile (PAN), organosolv lignin, and dimethylformamide (DMF) were used to prepare the spinning solution. The lignin content was varied over a wide range—from 0 to 100% by weight relative to the PAN mass (0, 10, 20, 30, 40, 50, 60, 70, 80, 90, and 100%)-which allowed us to evaluate the influence of solution composition on the fiber formation process and their morphological characteristics.

The solutions were prepared at room temperature by continuously stirring the components until a homogeneous, viscous system without visible undissolved particles was obtained. The resulting spinning solutions were used to subsequently form continuous lignin-containing fibers by electrospinning.

Thermal Stabilization and Carbonization of Fibers. The resulting lignin-polyacrylonitrile composite fibers were subjected to a two-stage heat treatment, including preliminary stabilization in an air atmosphere and subsequent carbonization in a nitrogen atmosphere.

The heat treatment was performed in a PT-T1600-L6012CB3 three-zone tube furnace (Zhengzhou Protech Technology Co., Ltd., China) with a maximum operating temperature of 1600°C and a temperature control accuracy of $\pm 1^\circ\text{C}$. The samples were heated at a rate of $5^\circ\text{C}/\text{min}$ to final temperatures of 800, 900, and 1000°C. Once the set temperature was reached, the samples were held for 60 minutes.

Carbonization was performed in a continuous nitrogen flow at a gas flow rate of 120 ml/min, which provided an inert atmosphere and prevented oxidation of the material during high-temperature treatment. After heat treatment, the samples were cooled to room temperature under a nitrogen atmosphere. Schematic illustration of the transformation of lignin/PAN fibers during thermal stabilization and carbonization is shown in Figure 3.

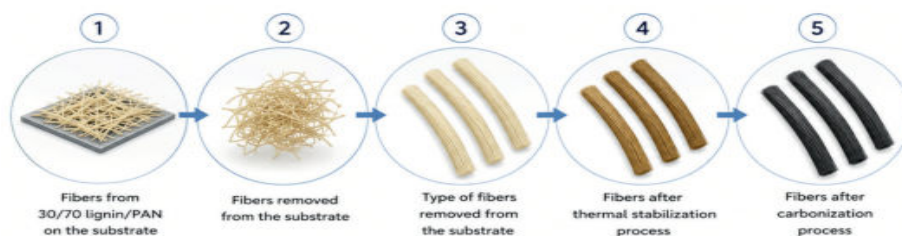


Figure 3. Schematic illustration of the transformation of lignin/PAN fibers during thermal stabilization and carbonization.

The morphological features and structural characteristics of the resulting carbon fibers were studied using scanning electron microscopy, thermogravimetric analysis, elemental analysis, and Raman spectroscopy.

Electrode Fabrication and Electrochemical Testing. To evaluate the electrochemical characteristics of the resulting carbon nanofibers, electrodes for supercapacitors and

lithium-ion batteries were fabricated. A 0.4 mm thick SS316L stainless steel foil was used as the current collector. Before applying the active material, the substrate surface was mechanically treated with P1000 abrasive paper and then polished with GOI No. 3 paste, which increased surface roughness and improved the adhesion of the electrode coating. The substrates were then sequentially cleaned in an ultrasonic bath using ethanol and distilled water, followed by drying at 120°C for 2 hours.

The active mass was prepared by mixing carbon nanofibers, acetylene black, and polyvinylidene fluoride (PVDF) in a 70:15:15 weight ratio. N-methyl-2-pyrrolidone (NMP) was used as a solvent, gradually added while vigorously stirring the components until a homogeneous suspension was obtained. The resulting mixture was uniformly applied to the surface of the current collector, forming an active layer approximately 400 μm thick with an area of 1 x 2 cm^2 . After coating, the electrodes were dried in a vacuum oven at 120°C for 12 hours to completely remove the solvent.

The electrochemical characteristics of the fabricated electrodes were initially studied in supercapacitors using cyclic voltammetry and galvanostatic charge-discharge.

To evaluate the potential of the obtained carbon nanofibers as anode material for lithium-ion batteries, CR2032 half-cells were assembled in a sealed glovebox filled with argon (99.99% purity). Metallic lithium served as the counter and reference electrodes. A single-layer Celgard 2500 polypropylene membrane served as the separator, and a 1 M LiPF_6 solution served as the electrolyte. The carbon nanofibers fabricated in this study served as the working electrode (Figure 4).

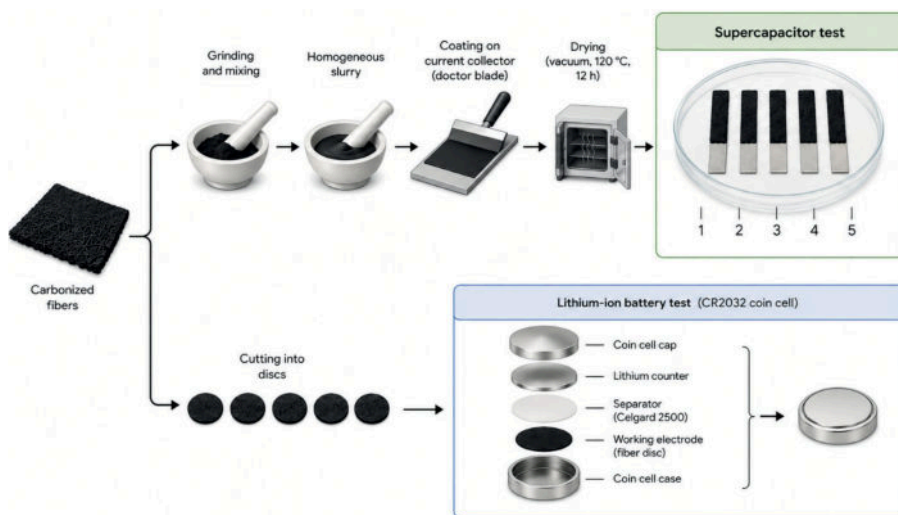


Figure 4. Schematic illustration of the preparation of carbon nanofiber electrodes for supercapacitor and lithium-ion battery applications.

The electrochemical characteristics of the lithium-ion half-cells were evaluated by cycling in charge-discharge modes, determining their specific capacity and cyclic stability.

Results and Discussion. *Study of the Physicochemical Properties of Organosolv Lignin Obtained from Wood Waste.* The morphology of organosolv lignin isolated from mixed wood waste (MWW), pine, and elm sawdust was analyzed using scanning electron microscopy. As shown in Figure 5, all samples exhibited a developed porous structure with irregularly shaped macropores. The largest pores were observed in the lignin sample obtained from pine sawdust, with pore sizes exceeding 100 μm . For lignin from mixed wood waste and elm, the macropore sizes were approximately 100 μm . The formation of this structure is due to the specific characteristics of organosolv extraction and subsequent precipitation of the lignin, resulting in the formation of a loose, porous material. This morphology is favorable for the subsequent production of carbon fibers, as it facilitates more uniform dissolution of the precursor and the formation of a homogeneous spinning solution.

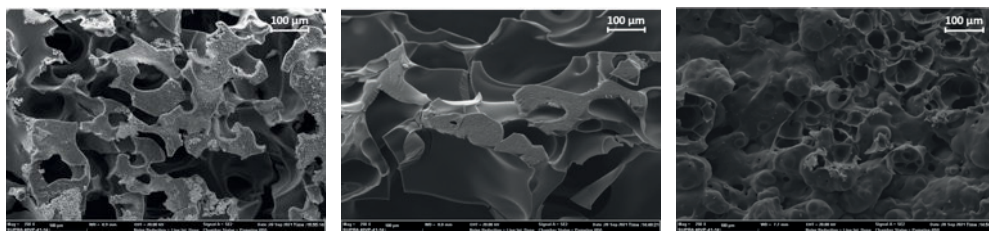


Figure 5. SEM images of the obtained lignins: (a) lignin from MWW; (b) pine lignin; (c) lignin from elm.

The resulting lignin samples were further analyzed using FTIR spectroscopy to determine functional groups and assess the purity of the isolated product. The spectra of samples obtained from various types of wood raw material are shown in Figure 6. Regardless of the wood source, all samples exhibit a similar set of characteristic absorption bands, indicating successful lignin isolation while preserving its chemical structure.

The broad absorption band at 3435 cm^{-1} corresponds to the stretching vibrations of hydroxyl groups found in the phenolic and aliphatic fragments of the lignin molecule. The band at 2926 cm^{-1} is attributed to the stretching vibrations of the C-H bonds of methyl and methoxyl groups. Intense bands at 1603 and 1512 cm^{-1} are due to vibrations of the aromatic ring and confirm the presence of the aromatic framework characteristic of lignin. Furthermore, absorption bands at 1375 and 1270 cm^{-1} indicate the presence of syringyl (S) and guaiacyl (G) structural units in the lignin structure.

Despite the similarity of the spectra, the intensities of individual bands vary slightly, due to the different ratios of syringyl and guaiacyl fragments depending on the type of wood raw material. As is known, hardwood lignin contains more syringyl units, while softwood lignin is dominated by guaiacyl structures. The increased content of guaiacyl units promotes the formation of more intermolecular bonds, which positively affects the thermal stability of the material and is an important factor during subsequent carbonization.

Thus, the IR spectroscopy results confirm that the organosolv method effectively

extracts lignin from various wood wastes while preserving its characteristic chemical structure. The absence of bands corresponding to carbohydrate and sulfur-containing impurities indicates a high degree of purity of the obtained samples and confirms their suitability as precursors for producing carbon nanofibers.

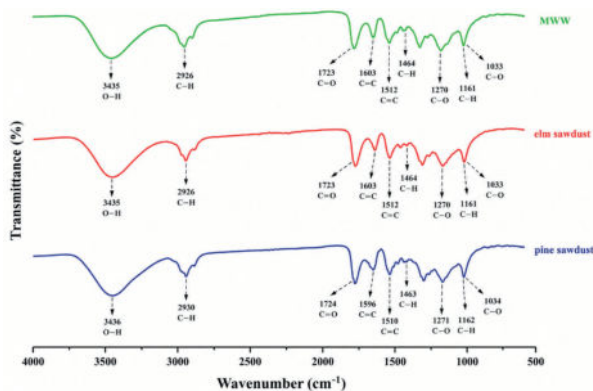
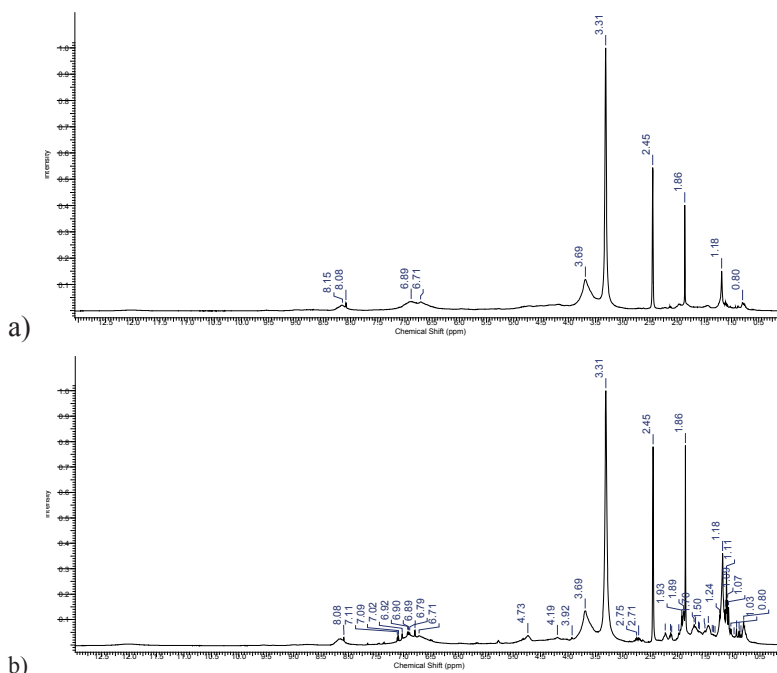


Figure 6. IR spectra of the obtained lignins.

Proton nuclear magnetic resonance spectroscopy (^1H NMR) was used to study the molecular structure of the isolated lignin samples. The resulting spectra of lignin isolated from mixed wood waste (MWW), pine, and elm sawdust are shown in Figure 7. The main chemical shifts corresponding to the characteristic structural fragments of lignin, along with their assignments, are listed in Table 1.



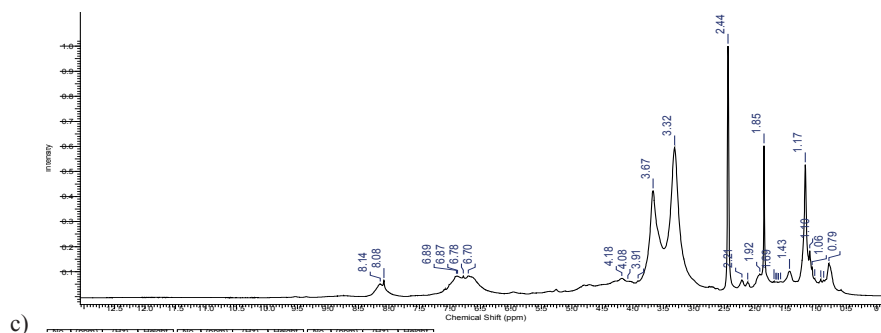


Figure 7. NMR spectra of the obtained lignins: (a) lignin from MWW; (b) pine lignin; (c) lignin from elm.

Table 1. Characteristic signal assignment and chemical shifts in the ^1H NMR spectra of the extracted lignins.

Chemical shift (ppm)	Appointment
0.80-1.2	Methyl and methylene protons
2.21	Aromatic acetate
2.45-4.73	Signals in this region refer to two aromatic protons in the structures of syringil and guaiacylpropane
6.70-7.11	Aromatic protons

Analysis of the NMR spectra revealed that all studied samples have a similar set of characteristic signals, indicating a similar chemical composition of the organosolv lignins, regardless of the type of original wood feedstock. Signals corresponding to protons of methyl and methylene groups of aliphatic side chains are observed in the 0.80-1.20 ppm range. The signal at 2.21 ppm corresponds to aromatic acetate fragments, while the region between 2.45 and 4.73 ppm characterizes protons of syringyl and guaiacyl structural units. Intense signals in the range of 6.70 and 7.11 ppm confirm the presence of aromatic protons characteristic of the aromatic backbone of lignin.

These results indicate the preservation of the main structural elements of lignin after organosolvent extraction. The presence of syringyl (S) and guaiacyl (G) units is consistent with FTIR spectroscopy data and confirms that the extraction process does not lead to significant changes in the chemical structure of the biopolymer. Moreover, the slightly higher signal intensity in the region of syringyl structures may indicate their predominance in the studied samples, potentially improving the thermal processability of lignin and producing more uniform carbon fibers.

A comparison of the spectra of lignin isolated from mixed wood waste, pine, and elm sawdust reveals no significant differences in either the location or intensity of the main signals. This indicates a similar chemical structure for the obtained organosolv lignins and confirms the effectiveness of the chosen extraction method for various types of wood raw materials.

A comprehensive analysis of the results obtained using SEM, FTIR spectroscopy, and ^1H NMR revealed that lignin isolated from mixed wood waste is virtually

indistinguishable in its morphological and structural characteristics from lignin obtained from individual wood species. Given these comparable physicochemical properties, as well as the urgency of the problem of recycling mixed wood waste, mixed wood waste (MWW) was chosen as the primary raw material for the subsequent production of carbon nanofibers.

This choice has not only scientific but also practical significance. In the wood processing industry, waste from various wood species is typically not separated but accumulates as a single mass, occupying significant space and creating additional environmental burden. Using mixed wood waste as a raw material simultaneously addresses the issue of its sustainable recycling and produces a promising precursor for the synthesis of carbon nanofibers.

Physicochemical Characteristics of Lignin/PAN-Based Carbon Nanofibers. Optical microscopy and scanning electron microscopy (SEM) were used to evaluate the morphology and structure of the resulting fibers. The results are presented in Figure 8.

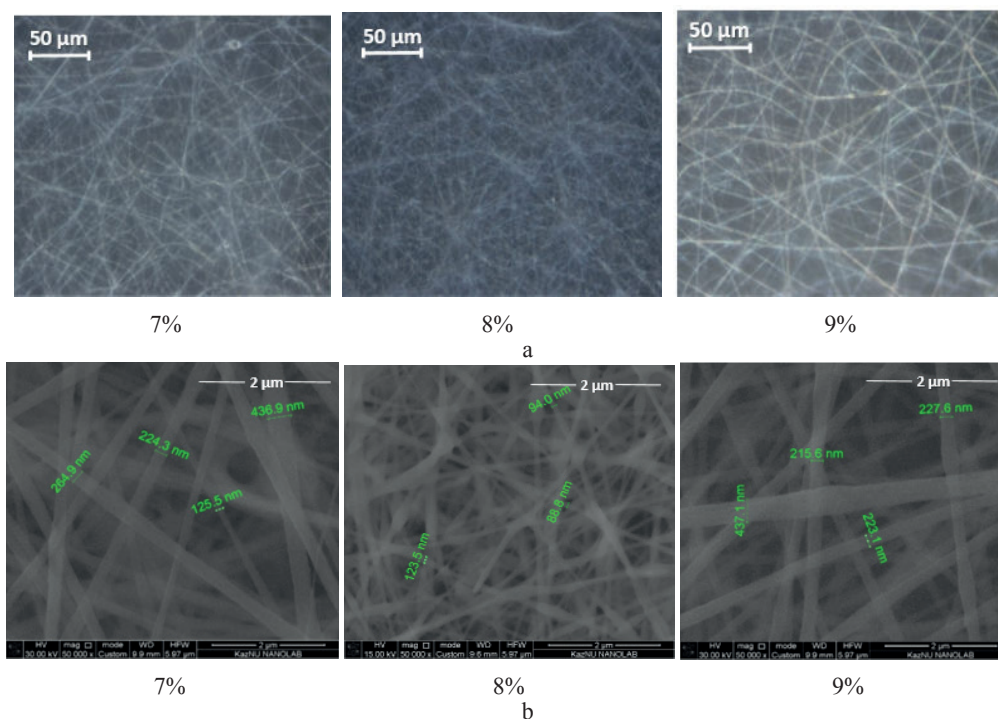
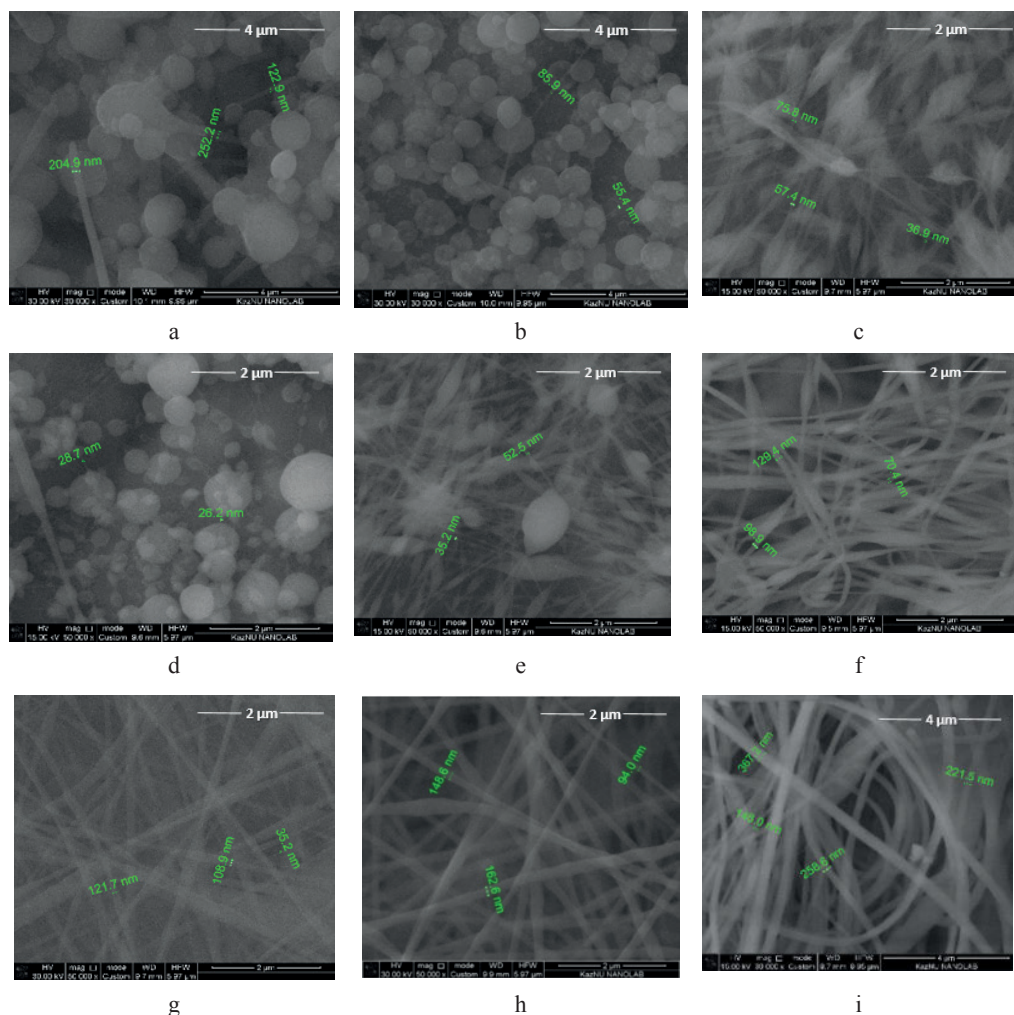


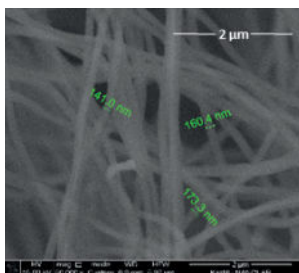
Figure 8. Optical and SEM images of lignin/PAN fibers: (a) optical images; (b) SEM images.

According to the optical images (Figure 8a), the resulting lignin/PAN fibers are continuous one-dimensional structures with a uniform surface. A more detailed SEM study (Figure 8b) revealed the effect of the spinning solution concentration on the morphology of the resulting fibers. At a solution concentration of 7 wt.%, the fiber diameter ranged from 125 to 440 nm, at 8 wt.%, it ranged from 88 to 123 nm, and at 9

wt.%, it increased to 215 to 440 nm. The thinnest and most uniform fibers were obtained at a concentration of 8 wt.%, indicating an optimal combination of solution viscosity and electrospinning conditions.

After determining the optimal spinning solution concentration, the effect of the lignin-to-PAN weight ratio on fiber formation was studied. For this purpose, composites with lignin contents ranging from 0 to 100 wt.% relative to the PAN weight were prepared. The morphology of the resulting samples was examined by SEM, and the average fiber diameter was determined using ImageJ software. The results are shown in Figure 9.





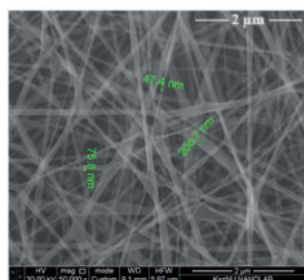
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Figure 9. SEM images of lignin/PAN fibers: (a) pure lignin; (b) 90/10 lignin/PAN; (c) 80/20 lignin/PAN; (d) 70/30 lignin/PAN; (e) 60/40 lignin/PAN; (f) 50/50 lignin/PAN; (g) 40/60 lignin/PAN; (h) 30/70 lignin/PAN; (i) 20/80 lignin/PAN; (j) 10/90 lignin/PAN.

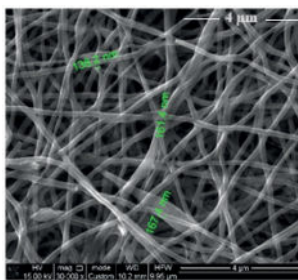
As can be seen in Figure 9, composites with lignin-to-PAN ratios of 10/90, 20/80, and 30/70 form continuous nanofibers with a smooth surface and no noticeable defects. As the lignin content increases to 40–50 wt.%, isolated spherical beads begin to appear, but the fibrous structure is maintained. Further increases in lignin content (60–80 wt%) are accompanied by the intense formation of "beads on a string" defects, indicating a decrease in jet stability during electrospinning. At a lignin content of 90 wt%, as well as when using pure lignin without the addition of PAN, the formation of continuous fibers is virtually absent; predominantly microspheres and agglomerates are observed.

The results show that increasing the lignin content leads to deterioration in the spinning properties of the solution due to the insufficient molecular weight of the lignin and the weak intertwining of the macromolecular chains. As a result, the influence of surface tension increases, promoting the formation of spherical defects. At the same time, increasing the PAN content ensures stable formation of continuous fibers, although accompanied by a slight increase in their diameter. Based on morphological analysis, it was established that a 30/70 (lignin/PAN) composition is optimal for producing defect-free nanofibers.

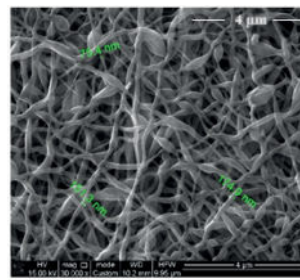
For further studies, fibers of the 30/70 (lignin/PAN) composition were selected and subjected to thermal stabilization and carbonization at temperatures of 800, 900, and 1000°C. The morphology of the resulting carbon nanofibers after carbonization is shown in Figure 10.



a



b



c

Figure 10. SEM micrographs of carbon fibers from 30/70 lignin/PAN carbonized at temperatures of: (a) 800 °C; (b) 900 °C; (c) 1000 °C.

SEM images show that after carbonization, the fiber diameter decreases compared to the original lignin/PAN fibers. While the average diameter before thermal treatment was over 88 nm, after carbonization, it varied depending on the process temperature and was approximately 47 nm at 800°C, 130 nm at 900°C, and 75 nm at 1000°C. This decrease in diameter is due to the removal of volatile components and non-carbon elements during thermal decomposition of the organic matrix.

Along with the change in size, changes in the surface area of the carbon fibers are observed. Carbonization at 800°C maintains a relatively uniform structure, whereas increasing the temperature to 900-1000°C is accompanied by the appearance of localized defects and increased surface roughness. This is due to further aromatization processes and carbon structure rearrangement, accompanied by the removal of oxygen-containing functional groups and the formation of a more ordered carbon matrix.

Thus, the results of morphological studies showed that the optimal conditions for producing continuous, defect-free carbon nanofibers are the use of a spinning solution with a concentration of 8 wt.%, a 30/70 (lignin/PAN) weight ratio, and subsequent carbonization in the selected temperature range. The resulting fibers possess a well-developed continuous structure and were used for further electrochemical studies.

The thermal stability of the resulting lignin/PAN fibers was studied using thermogravimetric analysis (TGA). Differential thermograms (DSC) were also constructed for a more detailed analysis of the thermal degradation processes. The results are presented in Figure 11.

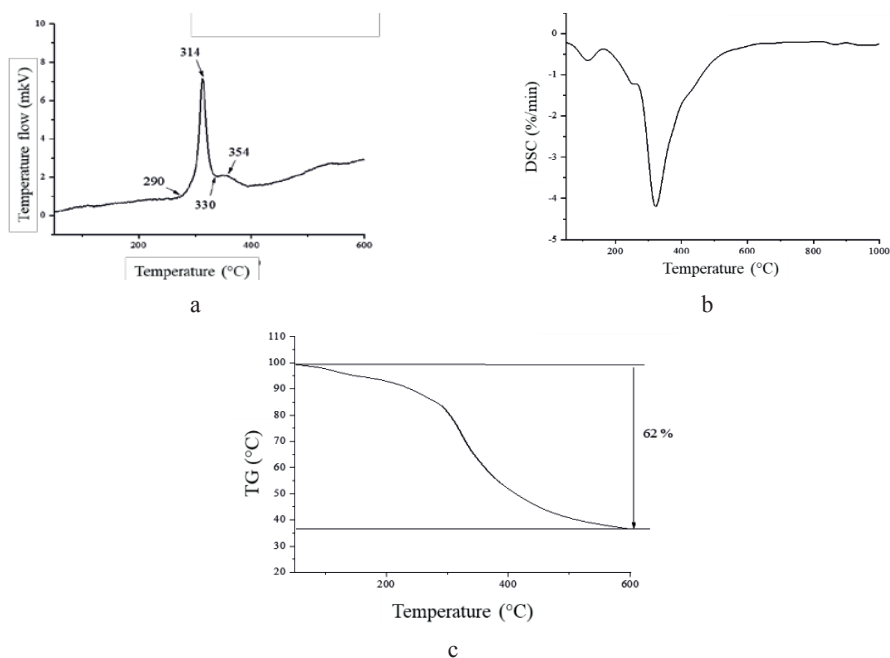


Figure 11. Curves of TGA and DSC nanofibers from 30/70 lignin/PAN: (a) curves of TGA (b) curves of DSC (c).

According to the thermogravimetric curves (Figure 11a), the thermal degradation of the composite fibers occurs in several sequential stages. In the temperature range up to 100°C, a slight weight loss is observed due to the removal of physically adsorbed moisture. The main weight loss occurs in the range of 100-500°C, where the thermal degradation of the composite components occurs sequentially. Polyacrylonitrile degradation begins at approximately 290°C, while intense lignin degradation is observed at approximately 400°C.

As Figure 11b shows, the two-stage nature of thermal decomposition is due to the different thermal stability of the composite components and the presence of oxygen-containing functional groups in the lignin structure. At this stage, dehydration reactions, side chain cleavage, and removal of volatile products occur, accompanied by the formation of a porous carbon structure.

According to TGA data, the total mass loss of the studied lignin/PAN fibers was approximately 62%, while the residual yield of the carbon product (semi-coke) reached 38%. The relatively high yield of carbon residue indicates good thermal stability of the resulting composite and confirms the potential of using organosolv lignin as a precursor for the synthesis of carbon nanofibers.

These results are consistent with published data and demonstrate that softwood lignin provides a comparatively high yield of carbon residue after carbonization. This represents an important advantage in the development of carbon materials for electrochemical energy sources.

To quantitatively evaluate the morphological characteristics of the electrospun fibers, their diameters were analyzed using ImageJ software. The fiber diameter distribution is shown in Figure 12.

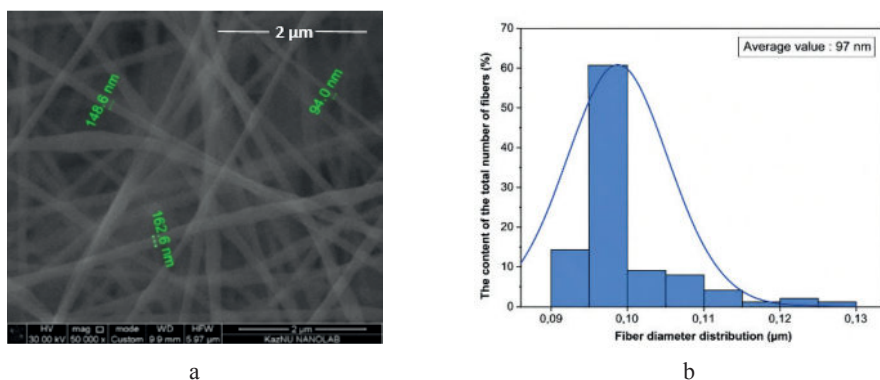


Figure 12. SEM micrography (a) of lignin/PAN fiber with fiber size distribution (b).

To plot the histogram, 100 individual fibers were analyzed, ensuring statistical reliability of the results. According to the data presented in Figure 12, the diameter of the fibers obtained at a lignin/PAN mass ratio of 30/70 varied between 90 and 128 nm, with an average diameter of 97 nm. The resulting distribution is characterized by a

relatively narrow size range, indicating high homogeneity of the formed fibers and the stability of the electrospinning process under the chosen synthesis conditions.

To study the crystal structure of the obtained carbon nanofibers, X-ray diffraction (XRD) analysis was performed using Cu K α radiation ($\lambda = 1.54 \text{ \AA}$). The diffraction patterns of the samples carbonized at temperatures of 800, 900, and 1000 °C are shown in Figure 13.

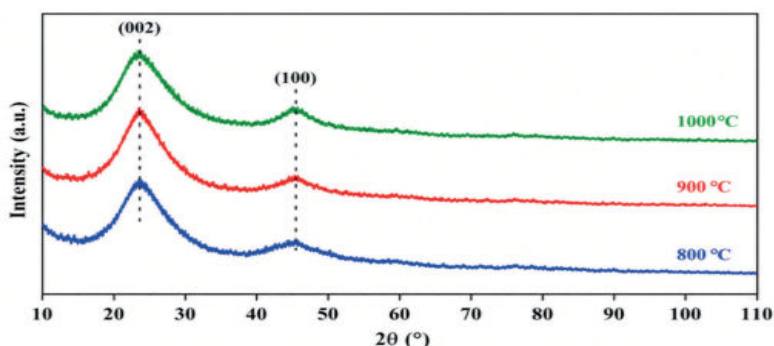


Figure 13. X-ray patterns of the carbon fibers.

As can be seen in Figure 13, all the studied samples exhibited two broad diffraction maxima located at $2\theta \approx 24.7^\circ$ and $2\theta \approx 43.6^\circ$, corresponding to the (002) and (100) planes, respectively. The broad shape of the (002) peak indicates a predominantly amorphous structure of the carbon material and a low degree of graphitization, while the (100) peak indicates the presence of a turbostratic carbon structure formed during carbonization.

As the carbonization temperature increases, no significant change in the shape of the diffraction maxima is observed; however, a slight shift is observed, indicating a gradual restructuring of the carbon structure and an increase in its degree of order.

To quantify the structural changes, the interplanar spacings $d(002)$ and $d(100)$ were calculated using the Bragg equation. The calculation results are presented in Table 2.

Table 2. Parameters of the interlayer distance of carbon nanofibers.

Samples	{002}		{100}	
	2 θ	Distance, D (nm)	2 θ	Distance, D (nm)
CNF 800	24.7251	0.356245	43.6751	0.207083
CNF 900	24.9251	0.357655	43.8751	0.26185
CNF 1000	24.9751	0.35979	44.2251	0.204634

As shown in Table 2, the interplanar spacing for the (002) reflection ranges from 0.356 to 0.360 nm, while for the (100) plane it ranges from 0.205 to 0.262 nm. These values are typical of carbon materials with a turbostratic structure and are in good agreement with the X-ray diffraction data.

Thus, the XRD results confirm that carbonization produces carbon nanofibers

consisting predominantly of an amorphous carbon phase with individual ordered graphene-like regions. Increasing the carbonization temperature promotes partial structural ordering of the material, which is an important factor for increasing its electrical conductivity and its future use as an electrode material for electrochemical energy storage devices.

To assess the degree of structural ordering of the obtained carbon nanofibers, Raman analysis was performed using a Solver Spectrum spectrometer (NT-MDT). The Raman spectra of samples carbonized at temperatures of 800, 900, and 1000°C are shown in Figure 14.

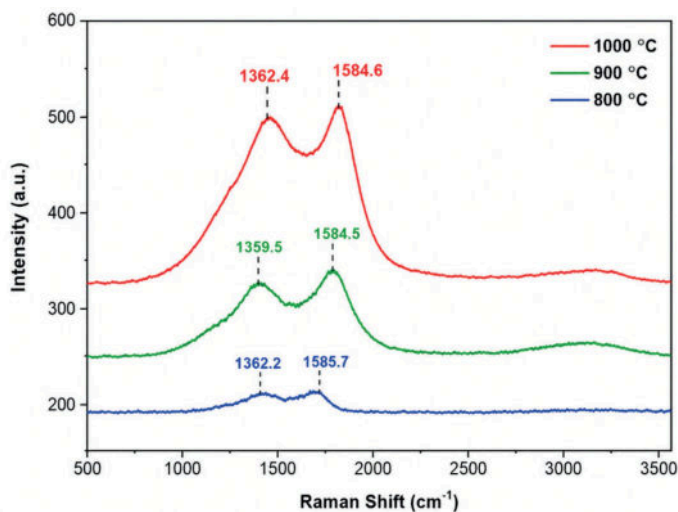


Figure 14. Raman spectra of carbon fibers.

As can be seen in Figure 14, all studied samples exhibit two broad bands at ≈ 1360 cm^{-1} (D band) and ≈ 1585 cm^{-1} (G band). The D band is associated with the presence of structural defects and symmetry breaking in the carbon lattice, while the G band corresponds to vibrations of sp^2 -hybridized carbon atoms and characterizes the degree of structural ordering in the material.

Analysis of the spectra revealed that increasing the carbonization temperature leads to an increase in the degree of graphitization of the carbon nanofibers. During carbonization at 800 and 900°C, the degree of graphitization was approximately 22%, while at 1000°C it increased to 26.7%. The obtained results indicate a gradual ordering of the carbon structure with increasing heat treatment temperature, which is in good agreement with the X-ray diffraction analysis results.

Additionally, the specific surface area of the carbon nanofibers was determined using the BET method. For the 30/70 (lignin/PAN) sample, its value was 549.7 m^2/g , indicating the formation of a developed porous structure. A high specific surface area is an important factor ensuring the efficient transfer of electrolyte ions and increasing the contact area between the electrode and electrolyte.

Thus, a comprehensive study using optical microscopy, SEM, EDS analysis, thermogravimetric analysis, X-ray diffraction, Raman spectroscopy, and BET confirmed the successful production of continuous carbon nanofibers based on a 30/70 (lignin/PAN) composite. The resulting fibers are characterized by an average diameter of 97 nm, a high specific surface area, and signs of partial graphitization, making them promising electrode materials for supercapacitors and lithium-ion batteries.

Electrochemical Characteristics of Carbon Nanofiber-Based Electrodes. To evaluate the practical potential of the obtained carbon nanofibers, electrodes for supercapacitors and lithium-ion batteries were fabricated. The electrode fabrication process is described in detail in the "Materials and Methods" section.

The electrochemical characteristics of the supercapacitor electrodes were determined using cyclic voltammetry and galvanostatic charge-discharge. For the CNF 30/70 sample carbonized at 800°C, the specific capacitance, calculated from galvanostatic charge-discharge curves at a current density of 0.1 A/g, was 136.4 F/g. According to cyclic voltammetry at a scan rate of 10 mV/s, the specific capacitance was 92.3 F/g. The obtained values demonstrate the excellent capacitive properties of the developed electrode material and its potential for use in high-power energy storage systems.

In addition to supercapacitors, electrodes for CR2032 lithium-ion batteries were fabricated, enabling the effect of carbonization temperature on the electrochemical characteristics of the material to be assessed. It was found that the sample carbonized at 800°C exhibited the highest discharge capacity, with a specific capacity of 797 mAh/g. Increasing the carbonization temperature to 900°C reduced the discharge capacity to 668 mAh/g, while for the sample processed at 1000°C, it was 594 mAh/g.

These results demonstrate that increasing the carbonization temperature does not always improve electrochemical characteristics. Despite the increased degree of graphitization at higher temperatures, the decrease in specific capacity is likely due to a reduction in the number of structural defects and active centers involved in charge accumulation. Thus, the optimal combination of structure and electrochemical properties was achieved for the CNF-800 sample, which demonstrated high specific capacity and stable operation during long-term cycling.

Conclusion. In this study, we developed an integrated approach to producing carbon nanofibers from mixed wood waste using organosolv lignin as a carbon precursor. It was found that lignin isolated from mixed wood waste, pine, and elm sawdust possesses similar morphological and structural characteristics, making it possible to use mixed wood waste as a promising raw material for producing carbon materials.

Optimal electrospinning conditions were achieved with a spinning solution concentration of 8 wt% and a lignin/PAN weight ratio of 30/70, ensuring the production of continuous, defect-free nanofibers with an average diameter of 97 nm. After carbonization, porous carbon nanofibers were formed with a developed surface area (549.7 m²/g) and signs of partial graphitization, confirmed by XRD and Raman spectroscopy.

Electrochemical studies revealed that the sample carbonized at 800°C exhibited the best performance, achieving a supercapacitor specific capacitance of 136.4 F/g and a

lithium-ion battery discharge capacity of 797 mAh/g. These results confirm the potential of using carbon nanofibers synthesized from mixed wood waste as electrode materials for modern energy storage systems and demonstrate the feasibility of efficiently recycling wood waste into high-value-added materials.

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Managing Editor: T. Apendiev
Editors: D.S. Alenov, A.Shormakova
Computer layout: G.D. Zhadyranova

Signed for print: September 29, 2026
Format: 70×90 1/16. 34.5 printed sheets. Order No. 3.

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