ISSN 2786-4898

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COLLECTION OF RESEARCH PAPERS

of the 8th International Research and Practical Conference

CHEMICAL TECHNOLOGY: SCIENCE, ECONOMY AND PRODUCTION

ЗБІРНИК НАУКОВИХ ПРАЦЬ

VIII Міжнародної науково-практичної конференції

ХІМІЧНА ТЕХНОЛОГІЯ: НАУКА, ЕКОНОМІКА ТА ВИРОБНИЦТВО

Shostka, Ukraine 2024

ISSN 2786-4898

Міністерство освіти і науки України
Сумський державний університет
Шосткинський інститут Сумського державного університету
Центральний науково-дослідний інститут
озброєння та військової техніки збройних сил України
Публічне акціонерне товариство «Фармак»
Управління освіти Шосткинської міської ради
Виконавчий комітет Шосткинської міської ради

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(м. Шостка, 27-29 листопада 2024 року)

Суми
Сумський Державний Університет
2024

УДК 66.01

Збірник наукових праць VIII Міжнародної науково-практичної конференції «Хімічна технологія: наука, економіка та виробництво», м. Шостка, 27 - 29 листопада 2024 року. – Суми : Сумський державний університет, 2024. – 242 с.
ISSN 2786-4898.

Збірник містить наукові праці учасників VIII Міжнародної науково-практичної конференції «Хімічна технологія: наука, економіка та виробництво», що складаються з узагальнених матеріалів науково-дослідних робіт науковців різних галузей виробництв та наукових закладів України.

У збірнику висвітлюються актуальні питання спеціальної хімічної технології і виробництва боєприпасів, утилізації відходів виробництв різних галузей, енергозбереження, моделювання технологічних процесів, соціально-економічні аспекти виробництва та природокористування в умовах війни.

Збірник корисний робітникам хімічної промисловості, науковим співробітникам, аспірантам і студентам спеціальностей хіміко-технологічного та соціально-економічного профілів, фахівцям інформаційних технологій виробництва.

Наукові праці учасників конференції подаються в авторській редакції.

DEVELOPMENT OF TECHNOLOGY FOR OBTAINING NITROCELLULOSE FROM SEA GRASS

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Introduction

Coastal deposits of sea grass along the coastlines of the Black and Azov Seas deteriorate the appearance of beaches and complicate access to water. Large accumulations require constant removal from beach areas and further disposal. One direction for such disposal could be the utilization of sea grass deposits for obtaining cellulose and its chemical processing products, particularly nitrocellulose, which is in high demand in modern industry.

The existing problem of raw material supply for the production of demanded grades of nitrocellulose by industry requires the search for new raw material sources for their extraction. Traditionally, nitrocellulose is obtained from cotton cellulose, the cultivation of which requires significant resources. In addition to cotton, wood is also used for nitrocellulose production, leading to deforestation and environmental harm. Currently, cotton is an expensive imported raw material, and wood pulp production has reached a critical level.

One of the non-traditional sources of raw materials is sea grass. It is a renewable resource that does not require large areas for cultivation, grows in large quantities, does not compete with food crops, and has a sufficient amount of cellulose, which is the basis for nitrocellulose production. The use of sea grass in the chemical industry for nitrocellulose production is a relevant research direction due to the deepening environmental pollution issues and the search for alternative, environmentally friendly sources of raw materials.

The conditions of war require an increase in Ukraine's defense capabilities, but the absence of ammunition and domestic weapons production complicates the situation. Domestic production of ammunition, including gunpowder, can significantly increase the potential of the army.

The aim of the work is to develop the technology of using Zostera marinova grass as an alternative raw material for obtaining nitrocellulose. In accordance with this, the following tasks were set:

Conduct a literature search on the use of unconventional plant raw materials in nitrocellulose production.

Obtain cellulose samples from sea grass using the selected method based on previous research.

Determine the quality of the obtained cellulose using chemical and instrumental analysis methods.

Conduct nitration of cellulose samples according to the chosen technology.

Investigate the cellulose samples using chemical and instrumental methods.

Draw conclusions about the possibility of using sea grass as a raw material for nitrocellulose production.

The object of the study is washed samples of sea grass deposits Zostera marina as an alternative raw material for obtaining nitrocellulose.

The subject of the study is the process of obtaining nitrocellulose samples and studying their properties.

To carry out the research work, literary sources on the research topic were processed. The following research methods were used: analysis and synthesis of literary sources, laboratory experiment, instrumental method of analysis.

The work has practical significance, and its results can lead to the creation of new directions for the utilization of sea grass by developing technological production lines for nitrocellulose. For the first time, the peculiarities of processing sea grass Zostera marina have been studied, which has not been previously considered as an alternative raw material for the production of energy-rich materials.

The possibility of using sea grass deposits Zostera marina as an alternative raw material for cellulose production can significantly reduce the production costs of nitrocellulose with its subsequent use as the energy basis of nitrocellulose powders, rocket fuels, components of paint and varnish products, for the production of optical transparent films, biological indicators, semi-permeable membranes, selective sorbents, and specialized nitrocoatings.

Literature review

Features of the Structure of Sea Grass Zostera Marina

Zostera marina, commonly known as eelgrass, grows in shallow waters along the coastlines of most countries worldwide. It is washed ashore by waves annually and accumulates in large quantities along the coastline. Currently, sea grass deposits in Ukraine are located along the shores of the Black and Azov Seas.

Figure 1 Deposits of seaweed on the coast of the Azov Sea

Kamka, which translates from Turkish as "spring," becomes resilient after drying and does not crumble. It possesses non-combustible properties and does not deteriorate. It is used for insulating roofs and buildings, and dried kamka is used to fill pillows and mattresses.

According to the analysis of the internal structure of sea grass fibers in [1], they consist of approximately 57% cellulose, 38% non-cellulosic polysaccharides (mainly xylan), and 5% residual substance, known as Klason lignin.

Given the above characteristics, particular attention is drawn to the possibility of using coastal deposits of Zostera marina for obtaining cellulose and products of its chemical processing, including nitrocellulose.

Among a wide range of polymers, nitrocellulose holds a leading position and has immense practical significance in both defense and civilian industries. The variety of applications for nitrocellulose is determined by its specific properties. High mechanical strength, the ability to be plasticized, good solubility, and compatibility with available plasticizers define the use of nitrocellulose as the energy basis of nitrocellulose powders, rocket fuels, components of paint and varnish products, for the production of optical transparent films, biological indicators, semi-permeable membranes, selective sorbents, and special-purpose nitro lacquers. The main raw material for the production of nitrocellulose is cotton cellulose.

Cellulose is a natural polymer obtained from wood cellulose or short fibers (lint) that adhere to cotton seeds. Hydroxyl groups form strong hydrogen bonds between cellulose molecules, as a result of which cellulose cannot be softened by heating or dissolved in solvents without chemical decomposition. However, during treatment with nitric acid in the presence of a sulfuric acid catalyst and water, hydroxyl groups are replaced by nitro groups (NO_2). Theoretically, all three hydroxyl groups can be replaced, resulting in the formation of cellulose trinitrate, which contains more than 14% nitrogen. In practice, however, most nitrocellulose compounds are dinitrates, containing on average 1.8-2.8 nitro groups per molecule and containing from 10.5 to 13.5% nitrogen. The degree of nitration determines the solubility and combustibility of the final product.

In practice, the degree of substitution is usually expressed as the nitrogen content, expressed as a percentage by weight. Depending on the nitrogen content, the following are distinguished:

Coloxylin (10.7 - 12.2% nitrogen)

Pyroxylin No. 2 (12.05 - 12.4% nitrogen)

Pyrocollodion (12.6% nitrogen) - a special type of nitrocellulose, soluble in alcohol and in a mixture of alcohol with ether.

Pyroxylin No. 1 (13.0 - 13.5% nitrogen)

The best raw material for the production of nitrocellulose is considered to be long-fiber varieties of hand-picked cotton. Machine-picked cotton and wood cellulose contain significant impurities, which complicate the preparation and reduce the quality of the product. Nitrocellulose is obtained by treating purified, fluffed, and dried cellulose with a mixture of sulfuric and nitric acids, known as the nitrating mixture. The concentration of the nitric acid used is usually above 77%, and the ratio of acids to cellulose can vary from 30:1 to 100:1. The product obtained after nitration undergoes multi-stage washing and treatment with various stabilizers to reduce degradation under the influence of light and heat, followed by drying. To reduce the risk of ignition, nitrocellulose is usually stored and transported in water or alcohol at a concentration of alcohol not less than 20%.

Use of unconventional raw materials for the production of cellulose nitrates

Due to the shortage of cotton raw material in many countries, the use of unconventional cellulose sources for the production of nitrates has become the subject of scientific research. Materials from renewable sources are receiving increased attention, as leading industrial sectors and manufacturers seek to replace valuable raw materials with agricultural waste, such as grain straw. In the study referenced [5], cellulose nitrate polymer was produced from Nigerian rice husks using a method that included alternative alkaline treatment and chlorination to remove non-cellulosic components, followed by nitration reaction. The variation in the composition of a certain nitrating acid mixture, relative acid strength of the nitrating mixture, nitration time, and the proportion of nitric acid to cellulose material influenced the yield and solubility of cellulose nitrate, the nitrogen content of which ranged from 11.06 to 13.12%.

In another study [6], bacterial cellulose was used as a raw material–an organic material synthesized extracellularly by microorganisms. A symbiotic culture of Medusomyces gisevii, consisting of various species of acetic acid bacteria and yeasts, was used as the producer. Nitrate of bacterial cellulose (NBC) was obtained by nitrosulfuric acid under heterogeneous conditions.

The results of the study [7] showed that esparto grass fibers can be considered a valuable alternative raw material for the synthesis of cellulose-rich polymers for potential use in solid fuel compositions.

Rheinmetall Denel Munition [8] exclusively uses cotton linters for the production of military and industrial grades of nitrocellulose, while wood cellulose from coniferous trees is used for the production of mining-type nitrocellulose.

Flax waste [9], wheat straw [10], rapeseed straw [11], and corn stalks [12] are proposed as raw materials. Cellulose extraction from agricultural waste is carried out by organo-solvent [11] and sodium hydroxide methods with prior hydrolysis [13].

In patent [14], the initial raw material was selected from a group consisting of paper, paper products, wood, wood-related materials, straw, rice husks, bagasse, cotton, jute, reeds, flax, bamboo, sisal, manila hemp, straw, cornstalks, coconut fibers, algae, seaweed, microbial materials, synthetic cellulose, and their mixtures. The technologies for processing non-traditional raw materials were included in the educational manual based on the results of these studies [15].

The cellulose content, as well as α- and β-cellulose, were evaluated in thirty-four species of algae belonging to fifteen orders of Chlorophyta, Phaeophyta, and Rhodophyta in the waters of India [16]. The cellulose content ranged from 20 to 1%. Combined studies confirmed that Chaetomorpha aerea, Acrosiphonia orientalis, Caulerpa taxifolia, Sargassum tenerrimum, Hydroclathrus clathratus, and Gelidiella acerosa have a relatively high (> 10%) cellulose content, which could be potentially useful.

The process of obtaining pure microcrystalline cellulose (MCC) from brown algae Posidonia oceanica through delignification and alkali treatment followed by acid hydrolysis is described in the work [17].

Research [18] enumerates optimization possibilities to increase cellulose yield from algae biomass and the current state of its conversion into nanocellulose. The results of this review provide insights into existing knowledge and future directions in the field of algal cellulose industry based on life cycle assessment studies.

The possibility of using Zostera marina seagrass in the pulp and paper industry was previously investigated at the Shostka Institute of Sumy State University [19,20]. Based on the research results, a patent "Method for making paper from seagrass" was obtained [21]. Further development of this direction involves the development of technology for obtaining energy-rich materials from cellulose from sea grass.

Experiment

Materials and methods

The research object consisted of dry stems of marine seagrass Zostera marina, collected on the coast of the Karkinitska Bay. They represent a resilient mixture of ribbon-like brownish leaves. For the extraction of cellulose from the seagrass, an organo-solvent method was chosen, using a mixture of acetic acid and hydrogen peroxide as reagents [22].

The advantages of organo-solvent methods include:

- Elimination of toxic sulfur-containing compounds from the cellulose boiling process;
- Ability to switch to bleaching methods with almost complete or complete exclusion of chlorine-containing compounds;
- Conducting the delignification process under mild conditions to obtain high-quality cellulose;
- Simplification of the regeneration of cooking solutions that do not contain significant amounts of mineral substances;
- Possibility of creating closed-loop processes without environmental pollution by harmful emissions;
- Relatively low capital investment and ensuring profitability of small and medium-sized enterprises.

The determination of alpha-cellulose content was conducted through gravimetric analysis of the insoluble residue after treatment of cellulose with a 17.5% NaOH solution, followed by washing with a 9.5% NaOH solution

The formation of cellulose nitrates occurs through the reaction:

$$[C_6H_7O_2(OH)_3]_n + 3nHNO_3 \leftrightarrow [C_6H_7O_2(ONO_2)_3]_n + 3nH_2O$$

Nitration is carried out using a ternary mixture: nitric acid - sulfuric acid - water. The nitration process is reversible, so the less water in the mixture, the more complete and faster the reaction proceeds. If the water content exceeds 25%, the reaction does not occur at all. Sulfuric acid is used to bind water. However, this slows down the reaction and reduces the nitrogen content in the final product. Increasing the amount of sulfuric acid reduces the viscosity of nitrates, especially with an increase in the temperature of the process.

Experimental procedure:

From the recommended compositions for the nitrating mixture for nitration with a module of 1:100, the ratio by weight was chosen as follows: HNO_3 - 17; H_2SO_4 - 64; H_2O - 19.

Afterwards, the nitrocellulose was stabilized with hot 1% soda solution (50-100 ml) for 10-15 minutes.

The determination of nitrogen content in nitrocellulose was carried out by decomposing the cellulose nitrates with concentrated sulfuric acid and then reducing the nitric acid formed to nitrogen oxide using a solution of iron(II) sulfate. The excess of the latter reacts with nitric oxide to form the complex compound $Fe(NO)SO_4$, imparting a yellow-red color to the solution.

The obtained samples of cellulose and nitrocellulose were analyzed using Fourier-transform infrared spectroscopy (FTIR). The research was conducted at the State Scientific Research Institute of Chemical Products with the assistance of scientist Serhiy Tyshchenko. Additionally, the obtained samples of nitrocellulose were further analyzed using thermogravimetric analysis (TGA) at the laboratory of the Shostka Institute of Sumy State University. [23].

Results and Discussion

During the experiment, samples of cellulose were obtained. Chemical analysis revealed that the content of α-cellulose was 75%. The samples were analyzed using Fourier-transform infrared spectroscopy (FTIR). In parallel, an FTIR spectrum of cotton cellulose was obtained.

Figure 2 shows the results of the investigation of cellulose samples obtained by the organo-solvent method from Zostera Marina seaweed using Fourier-transform infrared spectroscopy (FTIR) in comparison with cotton cellulose.

Comparison of the FTIR spectra of cotton cellulose and cellulose from Zostera Marina seaweed showed the presence of all characteristic peaks for cellulose [24]. The spectra are characterized by the following frequencies: 3570-3125 cm^{-1} – stretching vibrations (ST) of OH groups involved in intermolecular and intramolecular H-bonds; 2940-2860 cm^{-1} - ST of C-H and CH_2 groups; 1650 cm^{-1} - deformation vibrations (DV) of H-O-H bonds, attributed to the presence of bound water; 1430 cm^{-1}, 1370 cm^{-1} - DV of CH_2 groups; 1340 cm^{-1} - DV of O-H in CH_2OH; 1160 cm^{-1}, 1110 cm^{-1}, 1060 cm^{-1} - ST of C-O bonds. However, the spectrum of the investigated cellulose exhibits vibrations in the 1600 cm^{-1} region, characteristic of aromatic compounds of residual lignin.

Through nitration of the obtained cellulose with a ternary nitrating mixture, samples of nitrocellulose were obtained. According to chemical analysis, the nitrogen content was found to be 11.95 %. The nitrocellulose samples were also investigated using FTIR spectroscopy. In parallel, the FTIR spectrum of coloxylin was obtained.

Figure 3 presents the results of the investigation of nitrocellulose samples obtained from Zostera Marina seaweed using Fourier-transform infrared spectroscopy, compared to coloxylin.

The main characteristic frequencies observed in the FTIR spectra of nitrocellulose from Zostera Marina seaweed are: 1660-1630, 1280-1270, 823-817, 745-738, 689-680 cm^{-1}, corresponding to the vibrations of nitro groups, indicating that the synthesized product is nitrocellulose.

Comparison of the obtained spectra of nitrocellulose samples with the spectrum of standard coloxylin H indicates structural similarity between these polymers. The higher peak heights of the listed peaks indicate a higher nitrogen content in the obtained nitrocellulose compared to coloxylin.

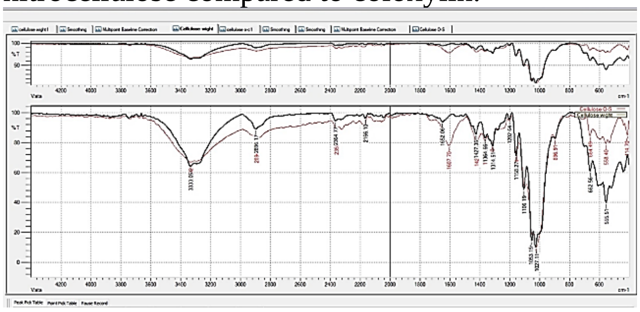

Figure 2 FTIR spectrum of cellulose obtained from Zostera Marina seaweed (red line) compared with the FTIR spectrum of cotton cellulose (black line).

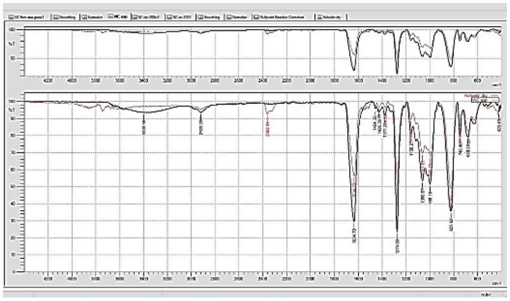

Figure 3 FTIR spectrum of nitrocellulose from Zostera Marina seaweed (black line) compared to the FTIR spectrum of coloxylin (red line).

Conclusions

In the course of research, a technology was developed for obtaining nitrocellulose from the coastal sediments of the sea grass Zostera marina. The relevance of the issue is due to the need to dispose of coastal deposits of dried algae and, at the same time, the need of the chemical industry for alternative raw materials for the production of nitrocellulose.

During the study, samples of cellulose and nitrocellulose from seagrass Zostera Marina were obtained. Comparison of the obtained spectra of nitrocellulose samples with the spectrum of standard colloxilin N shows the coincidence of the main characteristic frequencies: 1660-1630, 1280-1270, 823-817, 745-738, 689-680 cm^{-1}, corresponding to the vibrations of nitro groups. However, the results of the chemical analysis showed that the content of α-cellulose in the obtained samples is insufficient to obtain a high-quality product. Therefore, the technology requires further improvement.

Possible further research in this direction is aimed at establishing the optimal parameters of the processing of seagrass into cellulose nitrates to obtain high-quality products.

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