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PRODUCTION OF SODIUM CARBOXYMETHYLCELLULOSE FROM PINE WOOD WASTE AND INVESTIGATION OF ITS PHYSICOCHEMICAL PROPERTIES

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Abstract. With the increasing use of wood resources, wood waste disposal is becoming a pressing issue. Wood waste can be effectively processed and reused, thus reducing environmental impact. Our study proposes using wood waste to produce sodium carboxymethyl cellulose (Na-CMC) with enhanced physical and chemical properties. The paper explores a promising approach to processing Scots pine (*Pinus sylvestris L.*) wood waste via the suspension method to obtain the biopolymer Na-CMC. The ability to convert pine sawdust into Na-CMC meets the demand for high-quality wood raw materials, expands the biopolymer's applications, and reduces the environmental and economic burden of wood consumption. The aim of this study is to produce Na-CMC from Scots pine sawdust at 60°C and 80°C and investigate the biopolymer's physical and chemical properties. In laboratory experiments, Na-CMC samples were synthesized at 60°C and 80°C using sodium hydroxide, propanol-2, and sodium monochloroacetate (Na-MCA). The physical and chemical properties of Na-CMC were studied such as functional groups (hydroxyl, methylene, carbonyl), alcohol and ether bonds by IR spectrophotometry; molecular mass, polymerization degree, and viscosity of polymer solutions by viscometry; carboxymethyl groups by conductometric titration; and product yield and solubility by gravimetry. The scientific and practical significance of the chemical modification of wood sawdust cellulose has been established, with carboxymethylation proving effective at 60°C.

Keywords: carboxymethylation, sodium carboxymethyl cellulose, biopolymer, carboxymethyl groups, molecular weight, degree of polymerization, viscosity.

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ҚАРАҒАЙ АҒАШЫНЫҢ ҚАЛДЫҚТАРЫНАН НАТРИЙ КАРБОКСИМЕТИЛЦЕЛЛЮЛОЗА АЛУ ЖӘНЕ ОНЫҢ ФИЗИКА-ХИМИЯЛЫҚ ҚАСИЕТТЕРІН ЗЕРТТЕУ

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Аннотация. Бұл мақалада натрий карбоксиметилцеллюлоза (Na-КМЦ) биополимерін алу мақсатында кәдімгі қарағай (*Pinus sylvestris L.*) ағаш қалдықтарын суспензия әдісімен өңдеудің тиімді бағыты зерттелген. Қарағай ағаш үгінділерін Na-КМЦ-ға қайта өңдеу, синтездеуге қажетті жоғары сапалы ағаш шикізатына деген сұранысты қанағаттандырады және биополимердің қолдану аясын кеңейтіп қана қоймай, сонымен қатар ағаш шығынының экологиялық және экономикалық ауыртпалығын азайтуға мүмкіндік береді. Жұмыстың мақсаты – кәдімгі қарағай (*Pinus sylvestris L.)* ағашының үгінділерінен 60°С және 80°С температурада суспензиялық әдіспен Na-КМЦ алу және биополимердің физика-химиялық қасиеттерін зерттеу. Зерттеу әдістері. Лабораториялық тәжірбиеде 60°С және 80°С температурада натрий гидроксиді, карбоксиметилдеу реагенті ретінде монохлорсірке қышқылының натрий тұзы (Na-МХСҚ) және пропанол-2 қолданылып, Na-КМЦ үлгілері синтезделді. Нәтижелер және талқылау. Na-КМЦ-нің физика-химиялық қасиеттері зерттелді: функционалды топтары (гидроксил, метилен, карбонил), спиртті және қарапайым эфирлі химиялық байланыстары ИҚ-спектрофотометрия әдісімен; үлгілердің молекулалық массасы, полимеризация дәрежесі, полимер ерітінділерінің тұтқырлығы вискозиметрия әдісімен; карбоксиметил топтары кондуктометриялық титрлеумен; өнімнің шығымы және ерігіштігі гравиметрия әдісімен анықталды. Қорытынды. Ағаш үгінділердің целлюлозасын химиялық модификациялау әдісінің ғылыми және практикалық маңыздылығы ұсынылады, сондай-ақ 60°С температурада карбоксиметилдеудің тиімділігі жоғары екені анықталды.

Түйін сөздер: карбоксиметилдену, натрий карбоксиметилцеллюлоза, биополимер, карбоксиметил топтары, молекулалық масса, полимерлену дәрежесі, тұтқырлық.

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ПОЛУЧЕНИЕ НАТРИЙ КАРБОКСИМЕТИЛЦЕЛЛЮЛОЗЫ ИЗ ОТХОДОВ ДРЕВЕСИНЫ СОСНЫ И ИЗУЧЕНИЕ ЕЕ ФИЗИКО-ХИМИЧЕСКИХ СВОЙСТВ

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Аннотация: В данной статье исследовано перспективное направление по переработке отходов древесины сосны обыкновенной *(Pinus sylvestris*

L.) суспензионным методом с целью получения биополимера натрий карбоксиметилцеллюлозы (Na-КМЦ). Возможность переработки опилок древесины сосны в Na-КМЦ позволит удовлетворить потребность синтеза в высококачественном древесном сырье и расширить сферу применения биополимера, а также снизить экологическую и экономическую нагрузку расхода древесины. Цель работы – получение Na-КМЦ суспензионным методом из опилок древесины сосны обыкновенной *(Pinus sylvestris L.)* при температурах 60°С и 80°С и исследование физико-химических свойств биополимера. Методы исследования. В лабораторном эксперименте синтезированы образцы Na-КМЦ при температурах 60^0С и 80°С с применением гидроксида натрия, растворителя пропанола-2 и карбоксиметилирующего реагента натриевой соли монохлорукусной кислоты (Na-МХУК). Исследованы физико-химические свойства Na-КМЦ: обнаружены функциональные группы (гидроксильная, метиленовая, карбонильная), спиртовые и простые эфирные химические связи ИК-спектрофотометрией; молекулярная масса, степень полимеризации образцов и вязкость растворов полимера визкозиметрией; реалогия; карбоксиметильные группы кондуктометрическим титрованием; выход продукта и растворимость гравиметрией. Рекомендовано научное и практическое значение предложенного способа химической модификации целлюлозы опилок древесины, а также установлена эффективность карбоксиметилирования при температуре 60 ^{0}С.

Ключевые слова: карбоксиметилирование, натрий карбоксиметилцеллюлоза, биополимер, карбоксиметильные группы, молекулярная масса, степень полимеризации, вязкость

Introduction. In recent years, there has been a widespread increase in the generation of industrial waste. The total volume of accumulated solid household waste in Kazakhstan has reached approximately 103 million tons. Each year, an average of 5-6 million tons of such waste is generated, and according to estimates, this figure could rise to 8 million tons per year by 2025. with an annual growth rate of 20% (Bekezhanov, 2017). This issue is particularly significant for enterprises engaged in the processing of plant raw materials, including wood. In the production of certain types of woodworking products, waste exceeds 75% of the volume of wood used (Kolesnikova, et all, 2020). The main types of waste from the woodworking industry include chunky residues, paper waste, shavings, sawdust, and dust. In both global and domestic practices of wood processing waste utilization, sawdust has found the least application, with no more than 30% of its total volume being used for the production of eco-fuel, construction materials, complex fertilizers, zoo products, and feed additives. Inefficient use of sawdust leads to environmental and economic problems, such as environmental pollution by wood dust and toxic substances (phenol, formaldehyde, etc.), irrational use of forest resources, and increased fire hazards, among others (Kostyleva, et all, 2020), (Klara, 2022). Addressing the issue of sawdust processing, it is essential to consider the quality characteristics of the raw material and the economic factor (Kalyuta, 2020).

In modern conditions, the use of sawdust expands the raw material base and reduces production costs by minimizing the number and duration of individual technological stages. (Bazarnova, et all, 2004), (Olaiya, et all, 2023).

One the possible solution for the utilization of wood waste is the chemical modification (carboxymethylation) of plant raw materials to obtain water-soluble polymers with a range of useful mechanical, rheological, bioactive, and other operational properties (Markin, et all, 2013; Markin, et all, 2010).

In this regard, there is both scientific and practical interest in natural polysaccharides, such as cellulose and its derivatives, within the field of polymer chemistry. (Rahman, et all, 2021; Kukrety, et all, 2018). The most common cellulose derivatives are carboxymethyl cellulose (CMC), a simple water-soluble cellulose ether, and its sodium salt (Na-CMC), which exhibit excellent film-forming properties, complexation abilities, and non-toxicity. These properties enable the creation of interpolymer complexes based on Na-CMC in combination with various polymers for a wide range of applications in medicine, the oil industry, food production, pharmaceuticals, and agriculture.

The process of wood carboxymethylation is well-studied and has been extensively covered in scientific journals and patent literature (Joshi N, et all, 2024; Müller, et all, 2018).

Wood carboxymethylation is based on the Williamson ether synthesis - the O-alkylation of alkaline cellulose with monochloroacetic acid (MCA) or its sodium salt (Na-MCA) in a two-stage process (Figure 1).

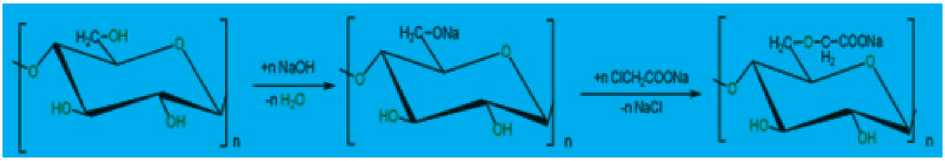

Figure 1. Chemical reactions of cellulose carboxymethylation.

The synthesis of Na-CMC involves the alkaline treatment of cellulose fibers (mercerization) and the carboxymethylation of alkaline cellulose with MCA in the presence of sodium hydroxide. In this process, the hydroxyl groups of the polymer's elementary unit are predominantly substituted at the C6 position, due to the reactivity order of C3 < C2 < C6 (Gaisina, et all, 2022).

In recent research, the modification of the three main methods of cellulose carboxymethylation reactions is particularly relevant: suspension (liquid-phase or heterogeneous, in a solvent medium), solid-phase (without a solvent), and homogeneous (in solution) methods (Markin, 2010). In the production of Na-CMC by the solid-phase method, technical cellulose undergoes various mechanical treatments such as mechanical grinding, mechanochemical processing, and extrusion (Cheprasova, **et all,** 2012; Qi, **et all,** 2023). Homogeneous carboxymethylation involves the use of different solvents and mechanisms for dissolving cellulose. In the suspension method, during the initial stage, wood is treated with various reagents such as ethanol, propanol-1, propanol-2, toluene, tertiary butyl alcohol, benzene, acetone, and mixtures of alcohols and water. These reagents interact with the reactive functional groups of the main biomass components, forming additional covalent bonds.

The suspension method for producing Na-CMC is of particular interest due to its advantages: the alkylating agent (MCA or Na-MCA) is dissolved and uniformly distributed throughout the reaction mixture in the swollen alkaline cellulose; the low viscosity of the reaction medium maintains the desired reaction temperature; it prevents local overheating; it uses simple and inexpensive equipment for mixing the reaction mixture; and it allows for the removal of by-products such as sodium chloride, sodium carbonate, and the sodium salt of glycolic acid by extraction with 60% ethanol (Kalyuta, et all, 2006).

The production of Na-CMC with specific properties (viscosity, solubility, uniformity of substituent distribution, and degree of substitution) is determined by the synthesis conditions: the type of plant raw material, the ratio and order of introduction of the reacting agents, the temperature of mercerization and carboxymethylation, the duration of the overall synthesis and individual stages, the nature and volume of the solvent, and the intensity of side reactions (saponification of MCA forming sodium glycolate).

The purpose of this study is to obtain sodium carboxymethyl cellulose (Na-CMC) using a suspension method from Scots pine (*Pinus sylvestris L.*) sawdust at temperatures of 60 and 80°C, and to investigate the physicochemical properties of the resulting biopolymer.

Methods and materials. The raw material for obtaining Na-CMC was common pine wood sawdust (*Pinus sylvestris L.)* with a particle size of 0.315-0.630 mm and a moisture content of 5.5-6%. For the identification of the laboratory-produced Na-CMC, a commercial sample of Na-CMC polymer (Laborpharma, Kazakhstan) was also used. A thermostat LAB-TJ-TB-01/19 (Russia), a conductivity meter Agilent 3200c (USA), a spectrophotometer Infealum FT-801 (Russia), a capillary viscometer VPJ-2 with a diameter of 0.56 mm (Russia), rotational viscometer Haake VT-550 (Thermo Scientific, Germany) a membrane filter with a diameter of 0.45 µm, and a Schott filter were used in this study.

Suspension method of Carboxymethylation. Equal portions of air-dried wood sawdust were weighed and mixed with sodium hydroxide to obtain a homogeneous mass. Then, propanol-2 was added as a solvent, and the first sample was thermostated at 60°C, while the second sample was thermostated at 80°C for 1 hour. Subsequently, a mixture of Na-MCA and water was added to each sample, and they were thermostated under the same conditions. The resulting products were separated by decantation, washed with a mixture of 96% ethanol and 90% acetic acid at pH 5 until negative reactions to alkali (using phenolphthalein) and chloride ions (using a silver nitrate solution) were observed, and then air-dried. From the obtained carboxymethylated wood (CMW) product, Na-CMC was isolated by boiling in a conical flask with a reflux condenser for 15 minutes in a water bath with 15% peracetic acid. The precipitate was then filtered, washed first with 70% ethanol from acid (to remove the acid, as verified by titanyl sulfate), then with a hot mixture of ethanol and acetone (1:1), and dried to a constant weight (Markin, et all, 2013).

The absorption spectra of functional groups in Na-CMC in the infrared region were determined using a spectrophotometer in the range of 4000-400 cm^{-1}, according to the methodology. Potassium bromide (KBr) was used as the immersion medium. Tablets of

Na-CMC and KBr were prepared in a press mold under a pressure of 100 kg/cm². The spectrum of pure KBr was subtracted from the obtained spectra.

The samples of Na-CMC synthesized at different temperatures were analyzed for carboxymethyl group content (CMG, %) using the conductometric titration method.

The solubility (S, %) of the obtained samples was determined according to the methodology, which is based on dissolving the product in distilled water and subsequently filtering this solution through a Schott filter.

The molecular masses (M, kDa) of the biopolymers were determined using the viscometric method and calculated according to the Mark-Houwink-Kuhn-Sakurada equation (Formula 1):

$$M = \left(\frac{[\eta]}{K}\right)^{\frac{1}{\alpha}}$$

where [η] - the intrinsic viscosity, cm^3/g;

$K = 5.37 * 10^{-3}$ and $\alpha = 0.73$ (K and α are empirical constants for Na-CMC).

The degree of polymerization (n) was determined by dividing the molecular mass of the polymer (M) by the molecular mass of the monomer (M_m) (Klivenko, et all, 2023).

The rheological properties of synthesized and commercial samples of Na-CMC were studied using a rotational viscometer Haake VT-550 (Thermo Scientific, Germany) (Berikbol, et all, 2024). The flow behavior of polymers with a concentration of 0.01 mol/L in aqueous solution was examined. The measurement system used was the NV rotor-cup configuration. In the rotational viscometry method, the torque exerted on the rotor is a measure of viscosity. The measurements were performed in the CS mode (stationary flow curve) at shear rates ranging from 50 to 4000 s^{-1} and 50 to 1000 s^{-1}. Data acquisition and processing were carried out using RheoWin 4.0 and Excel software. For comparison, the solution of a commercial Na-CMC sample in the same concentrations was also studied.

Results and discussion. Samples of Na-CMW and Na-CMC were obtained using the suspension method at 60°C and 80°C (Figures 2a and 2b).

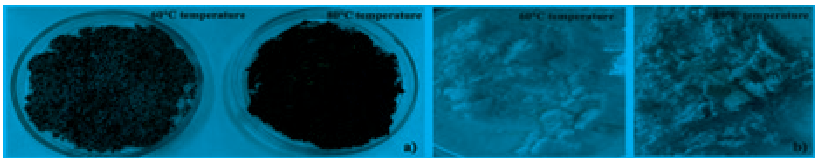

Figure 2. Samples of Na-CMW (a) and Na-CMC (b) obtained at temperatures of 60°C (left) and 80°C (right).

The synthesized Na-CMD and Na-CMC exhibited the following physical properties: in dry form, they were yellow, white, or light-yellow powders with no odor, soluble in cold water, forming transparent viscous solutions that, upon drying, produced stable films.

A universal and accessible method for identifying Na-CMC samples is IR spectroscopy. Figure 3 shows the IR spectra of the synthesized polymers and the commercial sample.

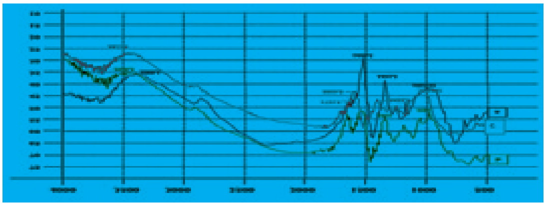

Figure 3. IR spectra of the studied samples; a) commercial Na-CMC sample; b) Na-CMC sample synthesized at 60°C; c) Na-CMC sample synthesized at 80°C.

The obtained IR spectra have absorption bands that differ slightly from each other. The IR spectra (ν, cm^{-1}) show absorption peaks characteristic of the vibrations of functional groups: the absorption band in the range of 1611-1601 characterizes the stretching vibrations of the carbonyl group (C=O); peaks in the range of 3478-3300 correspond to the vibrations of the hydroxyl group (O-H); peaks in the range of 3100-2750 correspond to the stretching vibrations of the methylene group (C–H); and in the range of 1067-1062, there are intense bands characterizing the vibrations of the alcohol and simple ether bonds (C–O). The presented results indicate that the studied samples have the same structure and confirm their correspondence to the Na-CMC polymer structure.

The yield and characteristics of the obtained Na-CMC from common pine sawdust, including the presence of carboxymethyl groups and water solubility, are presented in Table 1.

Table 1. Quantity of Carboxymethyl Groups and Solubility of Carboxymethylated Derivatives Obtained in Propanol-2

Sample	Temperature, °C	Yield, %	CMG, %	S, %
Na-CMC (synthesized)	60	57,4±0,7	21,2±0,4	50±1
	80	51,8±0,3	24,6±0,2	34±1
Commercial Sample*	-	-	12,5±0,3	98±1
*Note: Dash indicates absence of data.				

As shown in Table 1, the quantity of CMG in the synthesized Na-CMC samples is 21.2% and 24.6%, which is twice as much as that of the commercial sample.

The solubility of Na-CMC obtained by the suspension method was 35-50%, which is 2-3 times lower compared to the commercial sample. The decrease in Na-CMC solubility with increasing carboxymethyl group content contradicts the expected increase in polymer hydrophilicity due to the higher number of polar and ionic groups. However, this phenomenon can be explained by changes in molecular conformation, enhanced intermolecular interactions, and the potential formation of gel structures, which limit the ability of Na-CMC molecules to interact with water. The solubility of polymers also depends on the molecular weight and degree of polymerization. To study the molecular weights, relative, specific, and intrinsic viscosities were determined. Based on the calculation results, graphs of the dependence of intrinsic viscosity on polymer solution concentration were constructed (Figure 4).

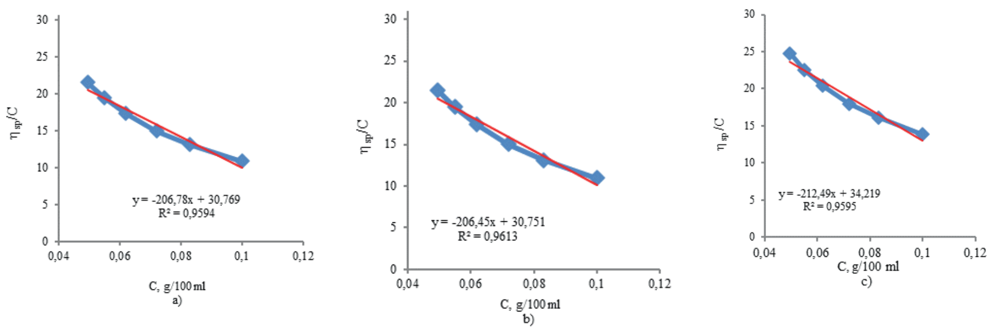

Figure 4. Concentration dependence of intrinsic viscosity of solutions:
a) Na-CMC synthesized at 60°C, b) Na-CMC synthesized at 80°C, c) commercial Na-CMC.

Figure 4 shows that the change in synthesis temperature of Na-CMC did not affect the viscosity of the polymer solutions. Additionally, the viscosity of the synthesized samples is 10% lower compared to the commercial sample. The reduction in viscosity can be explained by gel formation due to the aforementioned hydrophobicity of the CMG.

Measuring intrinsic viscosity is used to determine molecular weight, as the rheological properties of the solution depend on the number of units in the chain. The results of the calculations for the molecular weight and degree of polymerization of the Na-CMC samples are presented in Table 2.

Table 2. Molecular weight and degree of polymerization of synthesized and commercial Na-CMC

Sample	Intrinsic viscosity [η], cm^3/g	Molecular weight M, kDa	Degree of polymerization
Na-CMC synthesized at 60°C	30,8	181	750±20
Na-CMC synthesized at 80°C	30,7	181	750±20
Commercial Na-CMC	34,2	163	670±20

As seen from the results in Table 2, the synthesized polymers are high-molecular-weight and, therefore, possess sufficient mechanical strength, making them highly promising for practical applications.

The study of the rheological properties of polymer solutions allows for the evaluation of the chemical structure and the arrangement of functional groups that determine the intermolecular interactions of polyelectrolyte macromolecules in aqueous solutions and their stability over time. Figure 5 shows the dependence of the viscosity of polymer solutions on their shear rate.

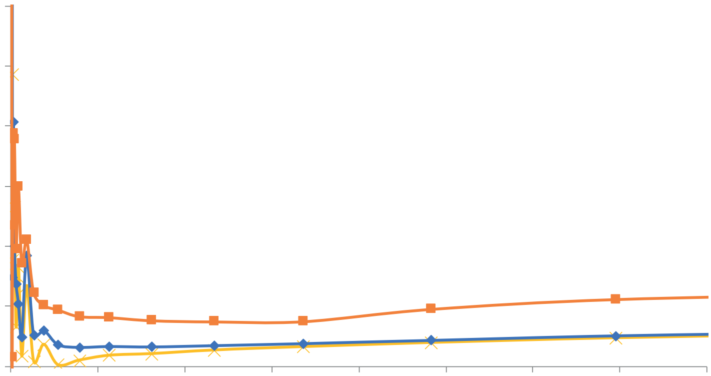

Figure 5. Dependence of the viscosity of polymer solutions on their shear rate.

The flow curve of Na-CMC polymer samples is characteristic of pseudoplastic fluids, where shear stress increases and viscosity decrease with increasing shear rate [24]. In general, the investigated non-Newtonian fluids exhibit flow behavior in a static state but demonstrate solid-like properties under abrupt force. When interacting with solid surfaces, the polymer solutions form a protective polymer film that shields against destructive mechanical impact.

Conclusion.

The synthesis of Na-CMC using the suspension method demonstrated significant advantages in controlling the physicochemical properties of the product and ensuring its environmental safety, while also addressing the global issue of wood waste recycling. The Na-CMC biopolymer samples were analyzed using physicochemical methods, including IR spectroscopy, viscometry, conductometric titration, and gravimetry. Based on the results, the Na-CMC sample obtained at 60°C exhibited more optimal characteristics: yield (57.4±0.7%), carboxymethyl group content (21.2±0.4%), solubility (50±1%), intrinsic viscosity (30.8 mL/g), molecular weight (181 kDa), and degree of polymerization (750±20). Thus, the high mechanical strength, good solubility, and optimal viscosity of Na-CMC synthesized from wood waste highlight the environmental and economic potential of this modern waste recycling method.

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