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DEVELOPMENT OF A PURIFICATION TECHNOLOGY FOR PHARMACEUTICAL-GRADE SODIUM CARBOXYMETHYL CELLULOSE (NA-CMC) FROM MASHAR STEM CELLULOSE FOR MEDICAL GYPSUM APPLICATIONS

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Abstract. *This study presents a technology for producing highly purified sodium carboxymethyl cellulose (Na-CMC) from cellulose extracted from locally available mashar stems for medical applications, particularly in the manufacture of medical gypsum. Technical Na-CMC was purified by ethanol extraction using a 53% aqueous ethanol solution, and the influence of extraction time on the purity and quality of the final product was systematically investigated. The optimum extraction time was found to be 40 min, resulting in a maximum main substance content of 99%. The physicochemical properties of the purified Na-CMC, including degree of substitution, dynamic viscosity, water solubility, and pH, were determined and compared with the requirements of relevant technical standards. X-ray diffraction (XRD) analysis confirmed the successful structural transformation of cellulose into Na-CMC through a reduction in crystallinity after carboxymethylation. The results demonstrate that mashar stem cellulose is a promising renewable local raw material for the production of pharmaceutical-grade Na-CMC with properties suitable for medical gypsum and other biomedical applications.*

Keywords: *Na-CMC, mashar stem, cellulose, ethanol extraction, medical gypsum, X-ray diffraction, physicochemical properties.*

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РАЗРАБОТКА ТЕХНОЛОГИИ ПОЛУЧЕНИЯ И ОЧИСТКИ НАТРИЕВОЙ КАРБОКСИМЕТИЛЦЕЛЛЮЛОЗЫ (NA-CMC) ФАРМАЦЕВТИЧЕСКОГО КАЧЕСТВА ИЗ ЦЕЛЛЮЛОЗЫ СТЕБЛЕЙ МАШАРА ДЛЯ МЕДИЦИНСКОГО ГИПСА

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Аннотация. *В данной работе разработана технология получения высокоочищенной натриевой карбоксиметилцеллюлозы (Na-CMC) из целлюлозы стеблей машара для применения в медицинской промышленности, в частности при производстве медицинского гипса. Очистка технической Na-CMC осуществлялась методом экстракции в 53%-ном растворе этилового спирта, при этом исследовано влияние продолжительности экстракции на содержание основного вещества и качество конечного продукта. Установлено, что оптимальная продолжительность процесса составляет 40 минут, при которой содержание основного вещества достигает 99%. Определены основные физико-химические характеристики очищенной Na-CMC, включая степень замещения, вязкость, растворимость в воде и значение pH, а также проведено их сравнение с требованиями действующих технических стандартов. Рентгенодифракционный анализ (XRD) подтвердил успешное превращение целлюлозы в карбоксиметилцеллюлозу. Полученные результаты свидетельствуют о перспективности использования стеблей машара в качестве местного возобновляемого сырья для производства Na-CMC фармацевтического качества.*

Ключевые слова: *Na-CMC, стебель машара, целлюлоза, этанольная экстракция, медицинский гипс, XRD-анализ, физико-химические свойства.*

MASHAR POYASI SELLYULOZASI ASOSIDA TIBBIY GIPS UCHUN FARMATSEVTIK SIFATDAGI NATRIY KARBOKSIMETILSELLYULOZA (NA-CMC) OLISH VA TOZALASH TEXNOLOGIYASINI ISHLAB CHIQISH

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Annotatsiya. *Ushbu tadqiqotda mahalliy qishloq xo‘jaligi xom ashyosi – mashar poyasi sellyulozasi asosida tibbiyot sohasi, xususan, tibbiy gips ishlab chiqarish uchun mo‘ljallangan yuqori tozalikdagi natriy karboksimetilsellyuloza (Na-CMC) olish texnologiyasi ishlab chiqildi. Texnik Na-CMC ning tozalash jarayoni 53% etil spirti muhitida ekstraksiyalash usuli yordamida amalga oshirildi hamda ekstraksiya davomiyligining mahsulot sifati va asosiy modda miqdoriga ta’siri o‘rganildi. Tadqiqot natijalariga ko‘ra, ekstraksiyaning optimal davomiyligi 40 minutni tashkil etib, bunda asosiy modda miqdori 99% ga yetdi. Olingan Na-CMC namunalarining fizik-kimyoviy xossalari, jumladan, almashinish darajasi, qovushqoqligi, suvda eruvchanligi va pH ko‘rsatkichlari aniqlandi hamda amaldagi texnik talablar bilan taqqoslandi. Rentgen difraksion (XRD) tahlili sellyulozaning Na-CMC ga muvaffaqiyatli aylanganligini tasdiqladi. Tadqiqot natijalari mashar poyasi sellyulozasining farmatsevtik sifatdagi Na-CMC ishlab chiqarish uchun istiqbolli mahalliy xom ashyo ekanligini ko‘rsatdi.*

Kalit so‘zlar: *Na-CMC, mashar poyasi, sellyuloza, etanol ekstraksiyasi, tibbiy gips, XRD tahlili, fizik-kimyoviy xossalar.*

Introduction. Cellulose is the most abundant renewable natural polymer on Earth and constitutes approximately 30–50% of plant biomass and more than 50% of wood biomass. Due to its biodegradability, biocompatibility, low toxicity, and chemical versatility, cellulose has become one of the

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most important renewable resources for producing value-added polymeric materials [17]. Among numerous cellulose derivatives, sodium carboxymethyl cellulose (Na-CMC) is one of the most widely manufactured anionic, water-soluble polymers because of its excellent water retention, viscosity, film-forming ability, and chemical stability.

Na-CMC is produced by introducing carboxymethyl (-CH_2COOH) groups into the hydroxyl groups of cellulose through an etherification reaction. The physicochemical properties of Na-CMC, including its degree of substitution, molecular weight, viscosity, and purity, determine its suitability for industrial applications. Owing to these characteristics, Na-CMC has found extensive applications in pharmaceuticals, medical gypsum, drug delivery systems, wound dressings, tissue engineering, cosmetics, food products, paper manufacturing, textile processing, detergents, oil drilling, and construction materials [3].

The global demand for Na-CMC has increased continuously due to the rapid development of pharmaceutical and biomedical industries. Conventional industrial production mainly relies on wood pulp and cotton linters as cellulose sources. However, increasing production costs, depletion of forest resources, and growing environmental concerns have stimulated the search for renewable, inexpensive, and sustainable non-wood lignocellulosic materials [21]. Agricultural residues have therefore become attractive alternative feedstocks because of their wide availability, low cost, and environmental benefits.

Recent studies have demonstrated that cellulose suitable for Na-CMC production can be successfully extracted from numerous agricultural residues, including corn cobs, corn stalks, corn husks, sugarcane bagasse, rice straw, cotton stalks, cotton linter waste, oil palm biomass, bamboo, waste paper, pineapple peel, orange peel, and other lignocellulosic wastes [8]. These renewable resources not only reduce dependence on wood-derived cellulose but also contribute to agricultural waste valorization and environmental protection [14].

Mashar stem represents an abundant agricultural biomass in Uzbekistan and contains a considerable amount of cellulose that can serve as an alternative raw material for producing cellulose derivatives. Nevertheless, information regarding the production of high-purity pharmaceutical-grade Na-CMC from mashar stem cellulose remains very limited. In particular, purification technologies capable of increasing the active substance content to pharmaceutical standards have not been sufficiently investigated.

Therefore, the objective of the present study was to develop a technology for producing highly purified Na-CMC from mashar stem cellulose for medical applications, particularly medical gypsum [1]. The purification process was performed by ethanol extraction to remove impurities from technical Na-CMC, followed by physicochemical characterization of the purified product. The obtained results demonstrate the feasibility of utilizing locally available agricultural biomass for producing high-quality Na-CMC suitable for pharmaceutical and biomedical applications [5, 19, 23].

Research Methodology. Mashar stem cellulose was used as the raw material for producing highly purified sodium carboxymethyl cellulose (Na-CMC). Technical Na-CMC was synthesized by the conventional alkalization and etherification process and subsequently purified by ethanol extraction. For purification, 150 g of technical Na-CMC was added to 650 mL of 53% aqueous ethanol in a 3000 mL glass reactor and continuously stirred [11]. The extraction time was varied from 10 to 50 min to determine the optimum purification conditions. After extraction, the purified product was filtered, pressed, and dried at 86 °C until constant weight was achieved.

The purified Na-CMC was characterized by determining the content of the main substance, moisture content, degree of substitution, dynamic viscosity, water solubility, and pH [6]. The obtained results were compared with the requirements of TSh 88.2-12-2005 and TSh 2231-001-53535770-01 to evaluate its suitability for medical applications.

Results and Discussion. The purification of technical sodium carboxymethyl cellulose (Na-CMC) obtained from mashar stem cellulose was carried out using ethanol extraction. The extraction conditions significantly affected the quality of the final product [20]. The purification efficiency was

evaluated by determining the content of the main substance under different extraction times.

Table 1

Effect of extraction time on the purity of Na-CMC obtained from mashar stem cellulose

№	CH_3-CH_2-OH Ethyl alcohol, ^0C	Mas'har pulp Na-CMC	
		The content of the main materials is 55%.	
		Extraction time, min	Content of the main substance, %
1.	55	10	74
2.	55	20	87
3.	55	30	98
4.	55	40	99
5.	55	50	98

As shown in Table 1, the purity of Na-CMC increased continuously with increasing extraction time. The content of the main substance increased from 74% after 10 min to 87% after 20 min, reaching 98% after 30 min and a maximum value of 99% after 40 min. Extending the extraction time to 50 min did not improve the purification efficiency, and the purity slightly decreased to 98%. This behavior indicates that most impurities were removed within the first 40 min of extraction, making 40 min the optimum extraction time for obtaining highly purified Na-CMC [2, 9, 16].

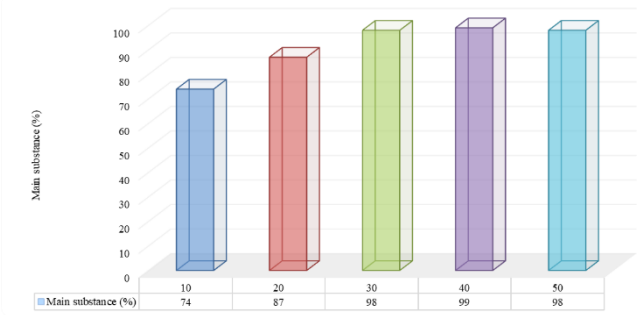

Fig.1. Effect of ethanol extraction time on the main substance content of Na-CMC obtained from mashar stem cellulose.

To evaluate the quality of the purified product, its physicochemical properties were determined and compared with those of conventionally produced Na-CMC.

The results presented in Table 2 demonstrate that the proposed purification method significantly improved the quality of Na-CMC. The purified sample obtained from mashar stem cellulose exhibited a degree of substitution of 0.88, 98.6% water solubility, dynamic viscosity of 178.2 mPa·s, and pH 9, while the moisture content decreased compared with conventionally produced Na-CMC. The increase in the degree of substitution and viscosity indicates that ethanol extraction effectively removed residual salts and low-molecular-weight impurities without damaging the polymer structure. These characteristics are particularly important for pharmaceutical and biomedical applications, where high purity and stable rheological properties are required [13].

Table 2

Physicochemical properties of Na-CMC obtained from mashar stem cellulose by the INNO-CELL-MONO method

Na-CMC samples	Indicators of Na-CMC						
	Wet-quantity, %	Degree of substitution with carboxyl groups	Main amount of substance, %	Dynamic viscosity of a 2% aqueous solution, mPa	Water solubility, %	pH	PD
Na-CMC based on PTKTCH cellulose							
1*	11.	81	45	109.3	97.2	12	4.20.
2+	7.	85	50	140.4	98.7	9	600
Na-CMC based on Pavlonia tree cellulose							
1*	10.	82	50	98.6	97.8	12	360
2+	8.	85	55	124.2	98.8	9	550
Na-CMC based on marshmallow stem cellulose							
1*	11.	83	50	124.2	97.7	12	500
2+	10.	88	56	178.2	98.6	9	750

**Note: 1*- Physicochemical indicators of CMC obtained on the basis of currently available production technology*

2^+Physicochemical indicators of CMC obtained by the "INNO-CELL-MONO" method.

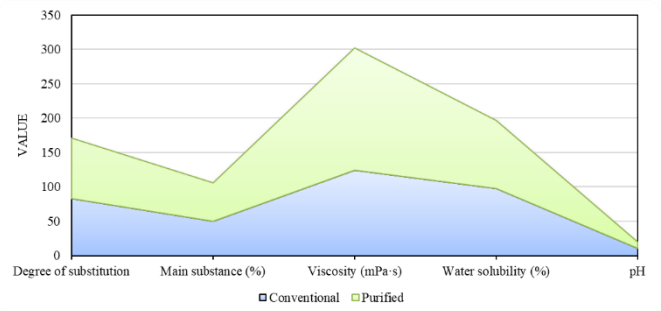

Fig.2. Comparison of the physicochemical properties of conventional and purified Na-CMC obtained from mashar stem cellulose.

The quality of the purified Na-CMC was further evaluated by comparing its properties with the technical requirements specified for commercial pharmaceutical-grade products.

As shown in Table 3, the obtained Na-CMC satisfies the quality requirements specified in TSh 88.2-12-2005 and TSh 2231-001-53535770-01. The degree of substitution, content of the main

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substance, water solubility, viscosity, and pH values all fall within the acceptable ranges of the technical specifications [22]. These findings confirm that mashar stem cellulose can serve as an effective alternative raw material for producing pharmaceutical-grade Na-CMC.

Table 3

Comparison of the physicochemical properties of Na-CMC with the requirements of TSh 88.2-12-2005 and TSh 2231-001-5353-5770-01

No.	Indicators	PTKTCHS, Na-CMC (1Th)	Standard	PTKTCHS, Na-CMC (2Th)	Standard	Pavlonia, Na-CMC (1Th)	Standard	Clown, Na-CMC (1Th)	Standard
1	Polymerization rate, not less than	500	600	650	920	500	500	750	500
2	Degree of substitution with carboxyl groups	85	80–100	85	85	80–100	65–85	88	80–100
3	Content of the main substance, %	50	50	53	55	50	48	50	50
4	Dynamic viscosity of a 2% aqueous solution, mPa·s	124.0	100	215.8	115.2	100	90–150	122.8	100
5	Water solubility, %	98.4	97	98.8	99.2	97	98	98.6	97
6	pH	11	8–12	9	11	8–12	8–12	9	8–12

**Note: 1Tsh-88.2-12-2005; 2Tsh-2231-001-5353-5770-01*

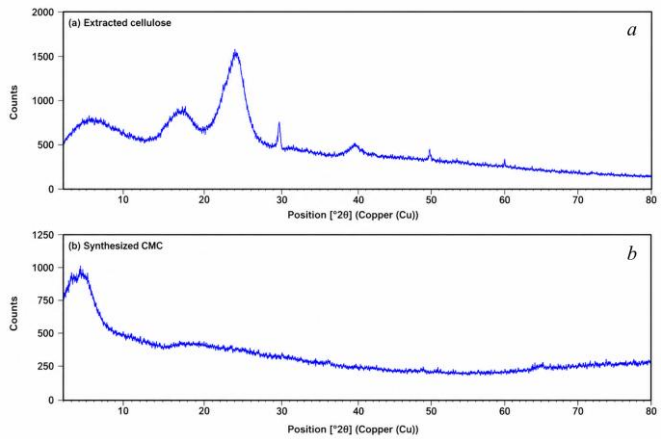

Fig.3. X-ray diffraction (XRD) patterns of (a) extracted cellulose and (b) synthesized Na-CMC obtained from mashar stem.

As shown in Figure 3(a), the extracted cellulose exhibits a distinct diffraction peak at approximately 2θ = 22°, which is characteristic of the crystalline structure of native cellulose. Additional weak diffraction peaks observed at higher diffraction angles correspond to the ordered arrangement of cellulose chains.

Following carboxymethylation, the XRD pattern of the synthesized Na-CMC (Figure 3(b)) becomes considerably broader and less intense. The characteristic crystalline peak is significantly reduced, indicating a decrease in cellulose crystallinity due to the substitution of hydroxyl groups with carboxymethyl groups. The disruption of the intermolecular hydrogen-bonding network results in a more amorphous polymer structure, which is a typical characteristic of Na-CMC.

These structural changes confirm the successful conversion of cellulose extracted from mashar stem into sodium carboxymethyl cellulose. The reduction in crystallinity is consistent with the improved water solubility and physicochemical properties presented in Table 2 and Figure 2, demonstrating the effectiveness of the proposed synthesis and purification process for producing pharmaceutical-grade Na-CMC.

The results demonstrate that the proposed purification technology is an effective approach for producing pharmaceutical-grade Na-CMC with enhanced physicochemical properties [4]. The purified product exhibited improved purity, water solubility, viscosity, and degree of substitution, satisfying the requirements for medical applications, including medical gypsum [18]. Furthermore, the utilization of locally available mashar stem as a cellulose source provides a sustainable and value-added strategy for the conversion of agricultural biomass into high-quality functional materials, while reducing reliance on conventional wood-based cellulose resources.

Conclusion. In this study, a technology for producing highly purified sodium carboxymethyl cellulose (Na-CMC) from locally available mashar stem cellulose was successfully developed. The ethanol-assisted purification process significantly improved the quality of the synthesized product, and the optimum extraction conditions were established. The highest main substance content (99%) was achieved after 40 min of extraction, while the purified Na-CMC exhibited enhanced physicochemical properties, including a degree of substitution of 0.88, water solubility of 98.6%, dynamic viscosity of 178.2 mPa·s, and pH 9. XRD analysis confirmed the successful structural transformation of cellulose into Na-CMC through a reduction in crystallinity after carboxymethylation. The obtained results demonstrate that the proposed purification method effectively removes impurities while preserving the functional characteristics of the polymer.

Furthermore, the quality characteristics of the purified Na-CMC complied with the requirements of TSh 88.2-12-2005 and TSh 2231-001-5353-

5770-01, confirming its suitability for pharmaceutical applications, particularly medical gypsum production. The developed technology provides a sustainable approach for the efficient utilization of locally available agricultural biomass and offers an alternative to conventional wood-derived cellulose. Consequently, mashar stem can be considered a promising renewable raw material for the production of high-value pharmaceutical-grade cellulose derivatives with potential applications in biomedical and pharmaceutical industries.

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