



## STRUCTURE AND PROPERTIES OF INTERPOLYMER COMPLEXES BASED ON SODIUM CARBOXYMETHYLCELLULOSE POLYSACCHARIDE AND CARBOPOL

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In this paper, the structure and properties of interpolymer complexes (IPC) based on sodium carboxymethylcellulose with a linear carbopol were studied. Interpolymer complexes were obtained by mixing aqueous solutions of Na-CMC and carbopol components in various ratios of components and pH of the medium. The structure of the obtained products was determined using the methods of IR spectroscopy and X-ray diffraction analysis. IR spectra in the range of 400-4000  $\text{cm}^{-1}$  were recorded on spectrophotometers "NIKOLET Magna-560 IR" and "Specord-75 IR" (Karl Zeiss, GDR). X-ray diffraction analysis of IPC films was carried out on a Rigaku X-Ray installation with an X-ray generator with a rotating copper anode, at a voltage of 40 kV, with a current strength of 15 mA and using characteristic Cu-K $\alpha$  radiation in the area of angles  $0 < 2\theta < 40$ . IR spectroscopic data show that the interpolymer complexes based on Na-CMC and carbopol obtained in moderately acidic regions are stabilized due to the cooperative hydrogen bond between the carboxyl groups of Na-CMC and the carbonyl groups of carbopol. X-ray diffraction analysis has shown that a change in the composition of the interpolymer complex leads to a change in the structure, which depends on the structure and nature of the interchain bonds. It is ascertained that an increase in the number of hydrogen bonds leads to a more ordered state of the resulting interpolymer complex. It is revealed that the formation of an interpolymer complex due to hydrogen bonds provides additional stability. This can serve as one of the means of controlling the structure and properties of the IPC of sodium carboxymethylcellulose with carbopol.

**Keywords:** Sodium carboxymethylcellulose; Carbopol; Polycomplex; Interpolymer complex; Films; Structure; Properties; IR spectroscopy; X-ray diffraction analysis; Hydrogen bond

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### INTRODUCTION

Interpolymer complexes (IPC) are promising products in pharmacy and are increasingly being used as thickeners and stabilizers of suspensions, prolongators of the action of drugs, film-forming agents for capsules and tablets, as bases for ointments and other soft dosage forms, since they reveal a number of unique and most valuable properties [1-4].

IPC are products of the interaction of chemically complementary macromolecules – polyanions and polycations or proton donors and acceptors. Unlike conventional chemical reactions between low molecular weight substances, the interaction between macromolecules is of a cooperative nature. The formation of a bond between the links of complementary chains, the strength of which coincides with the strength of the corresponding bond between small molecules, greatly facilitates the formation of subsequent interpolymer contacts. This ensures exceptionally high stability of IPC, even if the free energy of a single bond formation is small [4].

IPC are divided into two groups – stoichiometric - interpolymer complexes (S-IPC), in which chemically complementary links are included in an equimolar (1:1) ratio of components (Figure 1, *a*) and non-stoichiometric interpolymer complexes (N-IPC) containing an excess of one of the components (Figure 1, *b* and *c*) [1.5-8]. In the structure of the IPC, it is possible to distinguish uniformly connected areas that have formed with each other (section A), defective areas that are not connected with each other (section B) and an excess area of the IPC (section C) of one or another component (Figure 1, *b* and *c*).

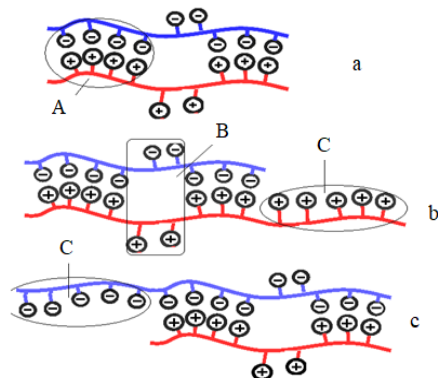
It is interesting to note that IPC particles have colloidal properties similar to protein structures, with a change in the ratio of interacting components, the structure charge, solubility of the hydrogel structure, pH, controlled permeability through water and solutions, including components of physiological fluids and other properties of the products obtained can be regulated [8]. The consequence of this is an unusually good biocompatibility and hemocompatibility of interpolymer complexes.

These features of the structure and properties of IPC open up wide opportunities for their use in various fields of practice, including pharmacy [8].

When producing interpolymer complexes, swelling polyelectrolytes of natural origin are used as polyanions: alginic acid and its sodium salt, agar-agar, gums, pectin, polymers containing sulfogroups: heparin, dextran sulfonic acid, chondroitin sulfate, hyaluronic acid; and polyelectrolytes of synthetic origin: polymers and copolymers of acrylic and methacrylic acid, copolymers of methoxyethylene with maleic anhydride and its hydrolysates, partial esters of vinyl acetate copolymer with maleic anhydride, vinyl acetate copolymers with crotonic acid and cellulose derivatives: methylcellulose, sodium carboxymethylcellulose, etc.

Cationic polyelectrolytes include gelatin, chitosan, as well as polydimethyldiallylammonium chloride, polyethylene glycol, polyethylenimine, collagen, biopolymers, urea-formaldehyde oligomers, etc.

When mixing aqueous solutions of the above natural and synthetic polyelectrolytes under certain technological conditions, IPC stabilized by ionic, hydrogen and other types of bonds are formed. Depending on the ratio of interacting components, stoichiometric and non-stoichiometric interpolymer complexes or polycomplex gels can be obtained. These obtained products are successfully used in pharmacy as carriers of drugs with prolonged actions [9-16].



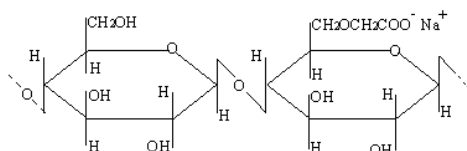
**Figure 1.** Schematic representation of the structure of S-IPC (a) and N-IPC (b, c) with an excess of polycation (b) and polyanion (c)

as the basis for ointments with controlled diffusion membrane properties, which have significant scientific and applied significance [20-22].

## EXPERIMENTAL

### Materials

**Sodium Carboxymethylcellulose (Na-CMC).** The main object of the study was purified Na-CMC of the Namangan Chemical Plant, obtained by heterogeneous solid-phase esterification of sulfite wood pulp with monochloroacetic acid (MCHAA) of the following structure:



with a degree of substitution (SD) 70 and a degree of polymerization (PD) 450, according to SSt 5.588-79 and BA 6-05-386-80. When using Na-CMC, it was repeatedly purified from low molecular weight salts according to the procedure given in the paper [23].

Na-CMC is a weak polyacid, its dissociation constant depends on the SD. When the SD changes from 10 to 80, the dissociation constant changes from  $5.25 \times 10^{-7}$  to  $5 \times 10^{-5}$  [1,2]. Na-CMC is a white or slightly yellowish powdery or fibrous odorless product with a bulk weight of 400-800 kg/m<sup>3</sup>, density of 1.59 g/cm<sup>3</sup>. The refractive index is 1.515. The softening temperature is Na-CMC T = 443 K, at a higher temperature it decomposes. Na-CMC is soluble in cold and hot water. They form highly viscous aqueous solutions. In aqueous solutions, it is a polyelectrolyte. Na-CMC is approved for wide use in medicine and pharmacy [23].

**Carbopol.** The second component of IPC is carbopol, a white, powdery polymer obtained by polymerization of acrylic acid. Carbopol does not dissolve in nonpolar organic solvents, but it swells strongly in water and polar solvents and forms a gel. The bulk density of carbopol is approximately 208 kg/m<sup>3</sup>, the glass transition temperature is 373-378 K. On average, the particle size of a solid polymer is approximately 2-7 microns, and each particle is a three-dimensional mesh structure of woven polymer chains. The density of polymers is 1.39-1.41 kg/m<sup>3</sup>. The pH value of 1% of the aqueous dispersion of acidic forms is 2.5 - 3.5. At pH values >6, the carboxyl groups of the polymer are ionized, as a result of which repulsion occurs between negatively charged particles, leading to swelling of the polymer and straightening of the chain [24-26].

**Synthesis of interpolymer complexes based on Na-CMC with carbopol.** Solutions of Na-CMC in bidistilled water with concentrations from 0.01 to 0.1 basic mol/l were used. Reaction mixtures of the required concentrations were prepared by mixing reagent solutions in an appropriate proportion at room temperature and pH 3.8-7.2. Under these conditions, water-soluble interpolymer complexes with stabilized hydrogen bonds are formed [27, 28].

**Determination of the pH value of IPC solutions.** Determination of the pH value of the base: 5 g of the base was mixed with 50 ml of purified water heated to a temperature of 323-333 K, after careful shaking, filtered through filter paper. The pH values of the resulting aqueous extract were measured using a potentiometric method on a universal

pH meter "Bante 210 Bentctop pH Meter" with glass (measuring) and silver chloride (comparative) electrodes at a temperature of 297 K [29].

**Obtaining IPC films.** Films from IPC were obtained by mixing aqueous solutions of Na-CMC and carbopol components in equinormal ratios at different component contents and pH of the medium. The solutions were poured onto an optical glass substrate and evaporated at room temperature. The solid dry films were washed with bidistilled water to a neutral pH value, then dried at room temperature [1,2].

**Viscometric properties.** The viscosity of solutions of interpolymer complexes was determined on a Ubbelode viscometer ( $d=2$  mm), at various temperatures under thermostatically controlled conditions, and the expiration time of the solution from the capillary was determined. The method for determining the viscosity of solutions is described in detail in [1,2].

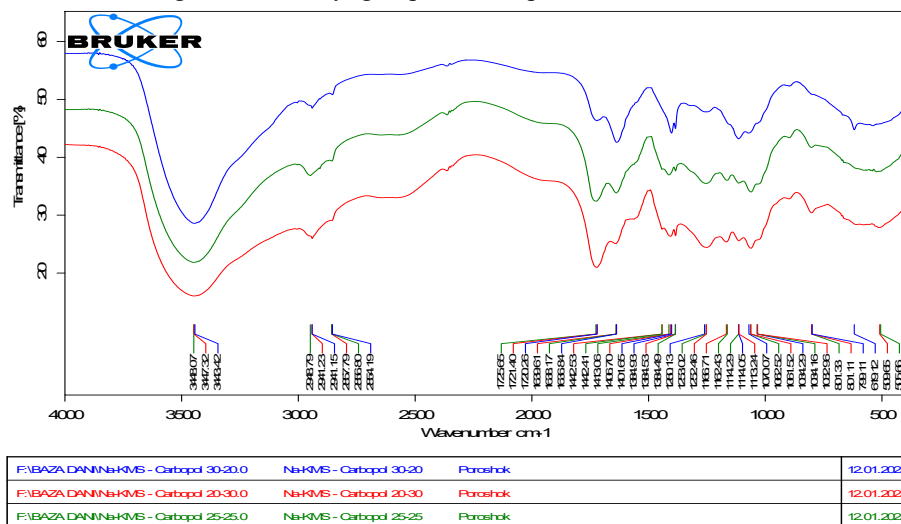
**IR spectroscopy.** IR spectra in the range of  $400 - 4000\text{ cm}^{-1}$  were recorded on "Specord-75 IR" spectrophotometers (Karl Zeiss, Germany) and NIKOLET Magna-560 IR. The samples for IR spectroscopy were prepared in the form of tablets with KBr, films on a KRS-5 plate and films with a thickness of 8-12 microns. Films on the KRS-5 plate were obtained by evaporation of a solvent (water) at room temperature (295-297 K) [3,4].

**X-ray diffraction analysis [30, 31].** X-ray diffraction analysis was performed on a Rigaku X-Ray installation with an X-ray generator with a copper rotating anode (maximum power 18 kW), at a voltage of 40 kV, a current of 15 mA and with a goniometer "Miniflex 300/600". D/teX Ultra 2 X-ray diffraction detector, 1D scanning mode(scan), scanning speed is  $10^\circ/\text{min}$ , step width is  $0.02^\circ$ , scanning axes are  $\theta/2\theta$ , scanning range is  $5-40^\circ$ , X-ray wavelength  $\lambda=1.5418$  nm, using characteristic Cu-K $\alpha$  radiation in the region angles  $0 < 2\theta < 40$  without a filter and at a temperature  $T = 298$  K [30,31].

### RESULTS AND DISCUSSION

The IPC obtained on the basis of Na-CMC and carbopol were studied by IR spectroscopy and X-ray diffraction analysis. IR spectra in the range of  $400-4000\text{ cm}^{-1}$  were recorded on Specord -75 IR spectrophotometers (Carl Zeiss) and UR-20 (GDR). The samples for IR spectroscopy were prepared in the form of tablets with KBr, films on a KRS - 5 plate and films with a thickness of 8-12 microns obtained by the above method. Films on the KRS-5 plate were obtained by evaporation of a solvent (water) at room temperature (295-297 K) [3,4].

The pH value of 0.2% carbopol solution is 3.5. With the help of organic solutions, the pH of the solution can be changed from 5 to 10. IR spectroscopic data showed that there are from 50 to 68.5% carboxyl groups in the carbopol structure. In addition to the carboxyl group, the carbopol structure contains such functional groups having absorption bands of  $2960\text{ cm}^{-1}$ ,  $1720\text{ cm}^{-1}$ ,  $1455\text{ cm}^{-1}$ ,  $1415\text{ cm}^{-1}$ ,  $1250\text{ cm}^{-1}$ ,  $1175\text{ cm}^{-1}$ ,  $800\text{ cm}^{-1}$  (Fig. 2). The most active, intense bands are  $1720\text{ cm}^{-1}$ , which belong to the carboxyl groups of carbopol.

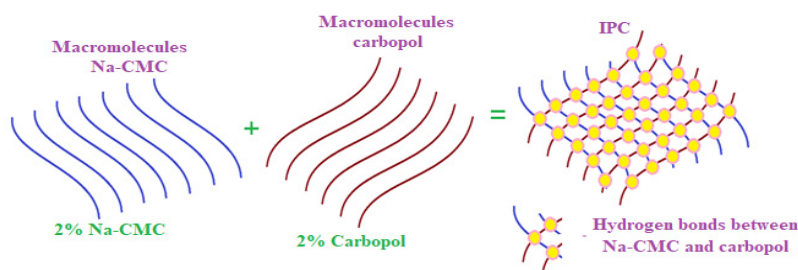


**Figure 2.** IR spectra of interpolymer complexes based on Na-CMC and carbopol polysaccharides

Furthermore, the second component of the Na-CMC polycomplex, in addition to the polydispersity usual for high-molecular compounds, has significant compositional chemical heterogeneity [2], i.e. it has a different quantitative ratio of functional groups in the chain and a different distribution of these groups in the link. Therefore, it can be considered as a copolymer consisting of two types of units: D – glucopyranose with glucopyronosoglycolic acid. In neutral media at a pH of about 6-7, both unsubstituted hydroxyl groups and a mixture of ionized carboxyl groups are present in the Na-CMC macromolecule. Quantitative analysis of the Na-CMC spectra using data on the characteristic frequencies of individual functional groups [1,2] made it possible to assign all absorption bands and ascertain structural patterns. The analysis of the IR spectra of Na-CMC and carbopol shows that the constituent components of the polycomplex are multifunctional. The presence of OH- ( $3200-3500\text{ cm}^{-1}$ ), COO- ( $1590-1620\text{ cm}^{-1}$ ,  $1410\text{ cm}^{-1}$ ) and COOH ( $1700\text{ cm}^{-1}$ )

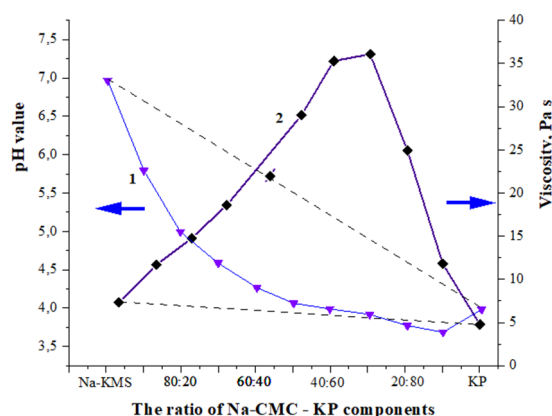
functional groups in their macromolecules gives these polymers the characteristic properties of polyelectrolytes [3,4]. According to the results of IR spectroscopic studies, it can be argued that, apparently, the Na-CMC polycomplex with carbopol obtained in moderately acidic regions is stabilized by hydrogen bonds between the carboxyl groups of Na-CMC with carbonyl groups of carbopol [3,4].

When aqueous solutions of Na-CMC and carbopol are mixed in neutral and slightly acidic media, water-soluble PC stabilized by hydrogen bonds are formed (Fig. 3).



**Figure 3.** The scheme of interpolymer complexes formation based on Na-CMC and carbopol

Reaction mixtures were prepared by mixing concentrated ( $C = 0.1$  basic mol/l) solutions of Na-CMC and carbopol under certain technological conditions and in certain component ratios. The pH of electrolyte solutions and their mixtures was carried out on a pH meter "210 Benchtop pH/mV meter" using combined electrodes. The accuracy of the pH measurement is 0.01 pH units. Before measuring, the device was adjusted using standard solutions. Titration was carried out with constant stirring and at a temperature of 22-24<sup>0</sup>C. When mixing solutions of Na-CMC and carbopol, gel-like, transparent IPCs are formed that can be used as a base for soft drugs in the production of gels, ointments, creams, pastes and liniments. When aqueous solutions of Na-CMC and carbopol are mixed at a pH of a moderately acidic region, a transparent water-soluble polycomplex composite is formed, stabilized by hydrogen bonds between the carboxyl groups of Na-CMC with carbonyl groups of carbopol (Fig.4, curve 1).



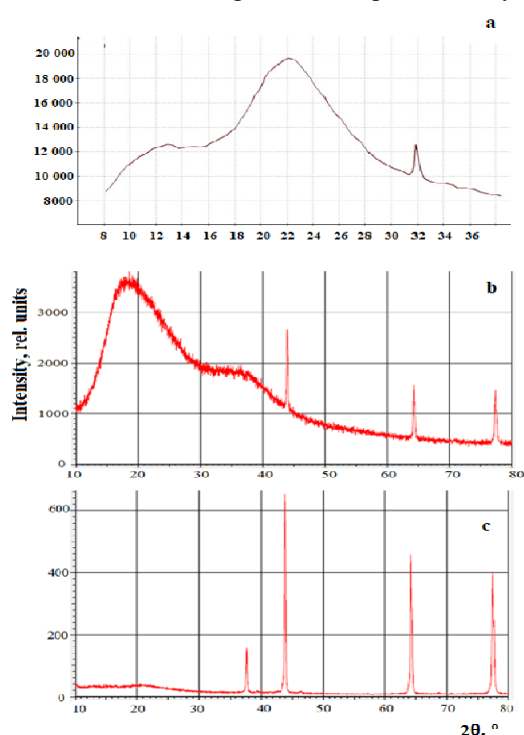
**Figure 4.** Graph of the pH (1) dependence and viscosity of IPC (2) solutions on the ratio of Na-CMC-carbopol components

To study the equilibrium of the Na-CMC-carbopol reaction, a potentiometric titration method was used, which is widely used to study the reactions of polycomplex formation. The results indicate the formation of IPC in neutral and slightly acidic environments. Mixing of Na-CMC and carbopol solutions is accompanied by a decrease in pH, which is typical for reactions between polyelectrolytes. The maximum output of the polycomplex corresponds to the equimolar ratio of the interacting components. [1,2] (Fig.4, curve 1). Experimental data have shown that the pH decrease for mixtures of Na-CMC and carbopol is the highest value of  $\Delta\text{pH} = 0.7- 1.0$ , which indicates a weak intermolecular interaction of the reacting components (Fig.4, curve 1).

Experimental data on the study of the viscosity of solutions of interpolymer complexes have shown that the viscosity value depends on the ratio of interacting components. The addition of a carbopol solution under certain technological conditions to the Na-CMC solution leads to an increase in viscosity and, with an equimolar ratio of interacting components, reaches its maximum value. A further increase in the amount of carbopol leads to a decrease in the viscosity of the polycomplex solution. The maximum change in the viscosity of solutions of polycomplexes from additivity with an equimolar composition is 15-20 Pa·s (Fig. 4, curve 2). To confirm the above data, the structure of the obtained IPC was studied by X-ray diffraction analysis (Fig. 5).

X-ray diffraction analysis allows you to objectively determine the structure of polymers, polycomplexes and other complex coordination compounds, etc. A complete structural study of IPCs often allows you to solve purely chemical problems, for example, ascertaining or clarifying a chemical formula, type of bond, molecular weight at a known density or density at a known molecular weight, symmetry and configuration of molecules and molecular ions.

X-ray diffraction analysis is most successfully used to ascertain the atomic structure of amorphous crystalline bodies. This is due to the fact that the crystals have a strict periodicity of structure and represent a diffraction grating for X-rays created by nature itself. Based on the width, shape and intensity of the diffractogram, conclusions can be drawn about the features of the near-range order in a particular crystalline or amorphous structure [30,31].



**Figure 5.** X-ray diffractograms of Na-CMC – (a), carbopol – (b) and IPC at a molar ratio of Na-CMC : Carbopol 1:1 - (c)

and nature of the interchain bonds. An increase in the number of hydrogen bonds apparently leads to a more ordered state of the resulting interpolymer complex.

Figure 5 shows the diffractograms of Na-CMC (a), carbopol (b) and the interpolymer complex with an equimolar component ratio (c), from which it can be seen that all the studied systems belong to amorphous crystalline polymers. The diffractogram revealed an intense reflex of  $2\theta = 22^\circ$  ( $d = 4.4 \text{ \AA}$ ), which occurs due to the superposition of reflexes (002 and 101) characteristic of cellulose II (Fig. 5, a, b), [30-32]. This Na-CMC reflex reflects the presence of a pronounced short-range order along the chain of a rigid-chain polymer.

In the IPC diffractogram, Na-CMC – carbopol can be found to be significantly ordered compared to Na-CMC and carbopol. The amorphous state of the initial constituent components of Na-CMC and carbopol, during interaction, passes into a more ordered state due to the formation of a hydrogen bond between the constituent components of the IPC. The diffractogram of the IPC Na-CMC – carbopol of equimolar composition clearly shows a change in the intensity of reflexes 101 and 002 and a new band is formed in the region  $2\theta = 38^\circ$  (Fig.5, c). This fact can be explained by the interaction of Na-CMC with carbopol and it is typical for a mixture of two interacting components.

Thus, X-ray examination shows that a change in the composition of the interpolymer complex leads to a change in the structure, which depends on the structure

## CONCLUSIONS

1. It follows from the above research results that a new interpolymer complex has been obtained based on Na-CMC and carbopol.
2. The structure and properties of the obtained product were studied by IR spectroscopy, potentiometric titration, viscosity and X-ray diffraction analysis.
3. The relationship between the structure of carbopol and Na-CMC, as well as the structure formed during their interaction, is revealed, and the possibility of forming an interpolymer complex due to hydrogen bonds providing additional stability is shown.
4. The formation of an interpolymer complex due to hydrogen bonds can serve as one of the means of controlling the structure and properties of the IPC of sodium carboxymethylcellulose with carbopol.

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## БУДОВА ТА ВЛАСТИВОСТІ ІНТЕРПОЛІМЕРНИХ КОМПЛЕКСІВ НА ОСНОВІ ПОЛІСАХАРИДУ КАРБОКСИМЕТИЛЦЕЛЮЛОЗИ НАТРИЇ ТА КАРБОПОЛІ

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У роботі досліджено структуру та властивості інтерполімерних комплексів (ІПК) на основі натрійкарбоксиметилцелюлози з лінійним карбополіом. Інтерполімерні комплекси отримували змішуванням водних розчинів Na-КМЦ і компонентів карбополу в різних співвідношеннях компонентів і рН середовища. Структуру отриманих продуктів визначали методами ІЧ-спектроскопії та рентгеноструктурного аналізу. ІЧ-спектри в діапазоні 400–4000 см<sup>-1</sup> записували на спектрофотометрах «NIKOLET Magna-560 IR» та «Specord-75 IR» (Karl Zeiss, НДР). Рентгеноструктурний аналіз плівок ІПС проводили на установці Rigaku X-Ray з генератором рентгенівського випромінювання з обертовим мідним анодом, напругою 40 кВ, силою струму 15 мА та характеристикою Cu-Kα. випромінювання в області кутів 0 <2θ <40. Дані ІЧ-спектроскопії показують, що отримані в помірних кислотних областях інтерполімерні комплекси на основі Na-КМЦ і карбополу стабілізуються за рахунок кооперативного водневого зв'язку між карбоксильними групами Na-КМЦ і карбонільними групами карбополу. Рентгеноструктурний аналіз показав, що зміна складу інтерполімерного комплексу призводить до зміни структури, яка залежить від структури та природи міжланцюгових зв'язків. Встановлено, що збільшення числа водневих зв'язків призводить до більш упорядкованого стану отриманого інтерполімерного комплексу. Виявлено, що утворення інтерполімерного комплексу за рахунок водневих зв'язків забезпечує додаткову стабільність. Це може служити одним із засобів контролю структури і властивостей ІПК натрійкарбоксиметилцелюлози з карбополіом.

**Ключові слова:** натрій карбоксиметилцелюлоза; карбопол; полікомплекс; інтерполімерний комплекс; плівки; структура; властивості; ІЧ-спектроскопія; рентгеноструктурний аналіз; водневий зв'язок