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Sodium carboxymethyl cellulose as an inhibitor of scale formation in nanofiltration of hard artesian waters

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ABSTRACT

The ability of sodium carboxymethyl cellulose (Na-CMC) to inhibit scale formation in nanofiltration (NF) of hard artesian waters have been studied. Na-CMC inhibition ability was examined in comparison with ACUMER scale formation inhibitor. We used three types of membranes: ESPA1, OPMN-P, and AMN-P. It was revealed that the event of absence of the inhibitor in the hard artesian water membranes' flux is decreasing. Level of flux leads to decrease in virgin membranes that depend on their type. Scale formation process gradually decreases when Na-CMC or ACUMER was added to the water. Na-CMC inhibition ability is comparable with ACUMER with the use of less dense membranes OPMN-P, AMN-P. Na-КМЦ и ACUMER models of inhibition action in NF of hard artesian water are proposed when different types of membranes are used.

Keywords: Hard artesian waters; Inhibitors; Nanofiltration; Sodium carboxymethyl cellulose; Scaling

1. Introduction

Currently, nanofiltration (NF) finds expanding application in drinking water supply systems all over the world. However, this method can ensure water quality meeting the world standards for drinking water only if the membranes are cleaned of scale forming impurities present in the water treated. As the analysis of the results for using NF to obtain drinking water [1–4] shows the membrane contamination is the main reason which hinders the large-scale implementation of this method in drinking water supply systems.

To reduce the formation of calcium carbonate and calcium sulfate on membranes, the inhibitors of scaling formation are suggested to be used. According to the data [5], the application of scaling formation inhibitors is the most efficient way to reduce the cost of water treatment provided that the cost of inhibitors is also decreased. Anti-scalant sodium hexameta phosphate [6] is the most known and studied agent. In recent years, new types of inhibitors have been synthesized. Yet, mechanism of many inhibitors has not been studied enough. This paper [7] shows the example of Calgon—its derivatives and the difficulties which occur when studying inhibition mechanisms.

The copolymers of new generation so-called polymer scale dispersants are of special interest as inhibitors. ACUMER by Rohm and Haas, Co. is one of them. According to the manufacturer's data, it is a

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high performance scale inhibitor. There are at least three mechanisms by which ACUMER performs: increases solubility of crystals, deforms crystals, and disperses crystals [8].

The papers [9–11] show that the efficient inhibitor to crystallize calcium sulfate is sodium carboxymethyl cellulose (Na-CMC). The main mechanism of Na-CMC inhibiting action with regard to calcium sulfate was found to be related to its adsorption on the calcium sulfate microcrystals formed in the solution filtered. Its blocking stops the further growth of calcium sulfate crystals.

The purpose of this study is to investigate inhibition ability of sodium carboxymethyl cellulose in NF of hard artesian waters.

2. Material and methods

2.1. NF procedure

All the experiments were carried out using a unit, the schematic view is shown in Fig. 1.

Recycled artesian water or sodium chloride brine was taken in container *1*. Permeate and concentrated products were returned to container *1*. The speed of solution circulation was about 1.5 m s^{-1}. NF was carried out at a pressure of 1.5 MPa and at the temperature of 21 ± 1°C. The recycling experiments enabled the separation of concentration polarization effects from scaling effects.

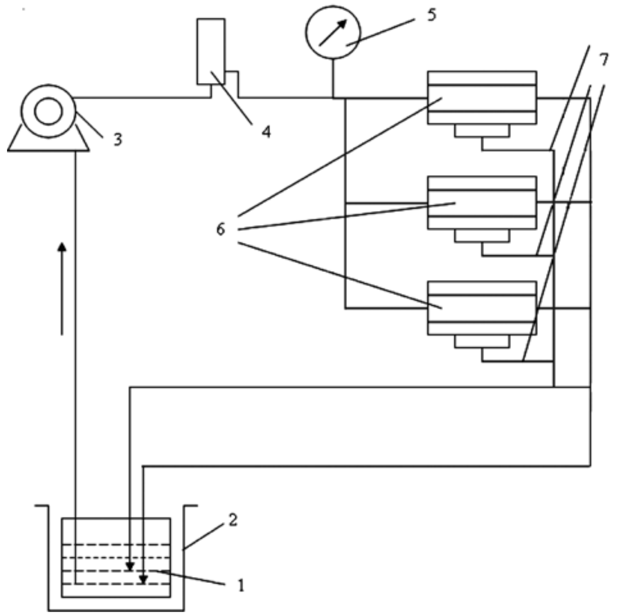

Fig. 1. Schematic view of laboratory facility with recycling solution: *1*—container with recycling solution (artesian water or sodium chloride brine); *2*—temperature regulator; *3*—pump; *4*—damping element; *5*—pressure detector; *6*—cells with membranes; and *7*—permeate sampling.

After each experiment carried out on artesian water, membranes were replaced with virgin membranes. Water flux of virgin membranes (J_0) and rejection (R_0) as to sodium ions are provided in Table 1.

Water flux with the use of artesian water (J_p) and J_0 was calculated by the following formulas (1) and (2):

$$J_0 = V_0/S{\cdot}\tau \quad (1)$$

$$J_p = V_p/S{\cdot}\tau \quad (2)$$

where V_0, V_p—volume of permeate samples in the course of NF in proportion to sodium chloride brine and artesian water, m^3; S—membrane's square, m^2; τ—time of flux's intake, s.

The change of membranes' water flux was calculated by formula (3):

$$\alpha = J_p/J_0 \quad (3)$$

where J_0—water flux in the course of NF of sodium chloride brine, (l $m^{-2}h^{-1}$); J_p—water flux in the course of NF of artesian water, (l m^{-2} h^{-1}).

Membranes' rejection (R) was calculated by formula (4):

$$R = \frac{C_0 - C}{C_0} 100\% \quad (4)$$

where C_0—concentration of sodium ions in artesian water, mg l^{-1}—concentration of sodium ions in permeate, mg l^{-1}.

2.2. Membranes

The NF membranes OPMN-P and AMN-P by "Polimersintez" (Russia) and ESPA1 by "Hydranautics" (USA) were used. OPMN-P membrane is manufactured on the basis of piperazine [12]. According to the manufacturer's data, AMN-P membrane is made on the basis of modified cellulose acetate. ESPA1 membrane is made on the basis of composite polyamide [13]. Zeta potential of membranes is one of the factors having an essential influence on the membranes' characteristics of the hard water NF [13].

We have measured zeta potential of virgin membranes (see Table 1).

Table 1
Membrane characteristics of in standard conditions of NF process of a sodium chloride solution

Parameter	ESPA1	AMN-P	OPMN-P
Sodium chloride solution $1.5\,\mathrm{g\,l^{-1}}$			
Operating pressure = 1.5 MPa; the speed of solution circulation = $1.5\,\mathrm{m\,s^{-1}}$; $T = 21 \pm 1$ °C			
Average pure water flux, $J_0 \times 10^6$, $\mathrm{m\,s^{-1}}$	17 ± 1	40 ± 5	39 ± 2
Rejection (Na^+) R_0 (%)	92	48	52
ξ_0 (mV; pH)	-4.9 ± 0.4; pH = 6.38	-4.2 ± 0.2; pH = 6.47	-4.9 ± 0.2; pH = 6.54

Further, structural properties of virgin ESPA1 membrane were defined as a dense one. It has the lowest J_0. OPMN-P and AMN-P membranes having higher and practically similar J_0 were recognized to be less dense.

2.3. Artesian water

The target of survey was artesian water in Kiev. The total hardness of the artesian water did not exceed $5.4\,\mathrm{mmol\,l^{-1}}$. During the tests, the carbonate hardness of water was raised up to $9.2–10.8\,\mathrm{mmol\,l^{-1}}$. For this purpose, the following salts were added to the artesian water kept in container *1*: $CaC1_2 = 6.66$ g; $NaHCO_3 = 10.08$ g. The sequence of adding salts to artesian water was as follows: artesian water → $CaCl_2$ → $NaHCO_3$.

2.4. Anti-scalants

Two polymers, i.e. commercial product of Na-CMC of 75/400 type, ACUMER 2200 by Rohm and Haas, Co. were investigated as inhibitors to prevent scaling formation of calcium carbonate on the membranes.

The characteristics of Na-CMC are given in Table 2, Fig. 2 shows structural fragment of macromolecule.

Before using Na-CMC in experiments, the solution of Na-CMC with the concentration of $1.0\,\mathrm{g\,l^{-1}}$ was prepared. The solution was prepared by diluting weighed portion of polyelectrolyte in the distilled water. Then, this solution was further diluted with distilled water up to the concentration of $0.1\,\mathrm{g\,l^{-1}}$. To provide equilibrium conformation of the polymer molecules, the solution was held for a day. This solution was used as a dosing solution in artesian water tests. The ACUMER 2200 characteristics are summarized in Table 3.

Table 2
The sodium carboxymethyl cellulose characteristics

Average molecular weight	9×10^4
Polymerization degree	400
Substitution degree	0.75
Content of the basic substance (%)	50

Preparation technique for dosing solution of ACUMER 2200 was similar to that of Na-CMC.

2.5. Quantitative determination of ξ-potential membranes

The measurement of samples ξ-potential was carried out using electrokinetic analyzer by Anton Paar GMBH. The calculation of ξ-potential is based on the results of measurement of electrolyte streaming potential and its electroconductivity. Based on this data and using the Fairbrother-Mastin method ξ-potential was calculated by the following formulae (5) and (6).

$$\xi = \frac{\mathrm{d}U}{\mathrm{d}p} \times \frac{\eta}{\varepsilon \times \varepsilon_0} \times \kappa \times 10^{-8} \tag{5}$$

where ξ—potential, mV, $\mathrm{d}U/\mathrm{d}p$—streaming potential derivative of pressure, η—electrolyte viscosity, mPa, ε—electrolyte dielectric constant, ε_0—specific conductivity, and κ—electrolyte electroconductivity, $\mathrm{mS\,m^{-1}}$.

$$\xi = \frac{\mathrm{d}I}{\mathrm{d}p} \times \frac{\eta}{\varepsilon \times \varepsilon_0} \times \kappa \times R_z \times 10^{-14} \tag{6}$$

where $\mathrm{d}I/\mathrm{d}p$—streaming current derivative of pressure, R_z—cell resistance when using electrolyte, Ohm.

2.6. Quantitative determination of total iron, Ca^{2+}, Mg^{2+}, total hardness, and alkalinity

The total content of iron ions dissolved in the filtrate was measured after water sample containing *o*-phenanthroline was filtered through a fine-mesh filter. The content of ions of Fe_{total} was determined using *o*-phenanthroline technique. Error of method was 2.0–3.5%.

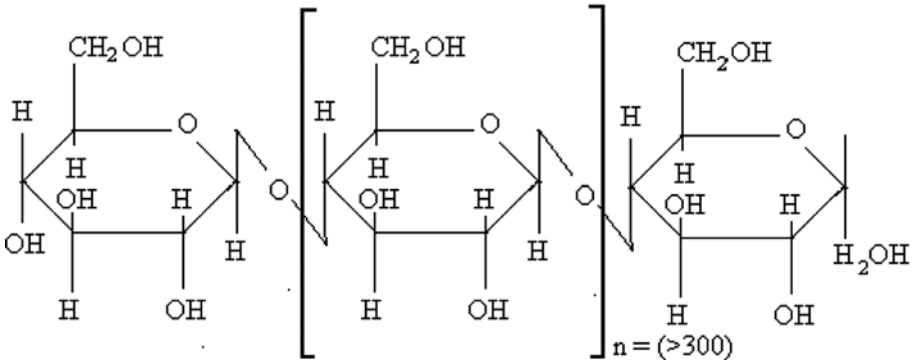

Fig. 2. The structural fragment of Na-CMC.

Table 3
The ACUMER 2,200 typical properties [8]

Appearance	Clear light amber solution
Chemical nature	Carboxylate copolymer
Molecular weight	2.000
Total solids (%)	55
pH as is (at 25°C)	4
Density (at 25°C)	1.3
Brookfield viscosity (mPa.s/cps at 25°C)	500

Table 4
Carbonate hardness change with time of a solution of calcium carbonate with concentration of 10.8 mmol l^{-1}sence and with addition of Na-CMC in static conditions

No	Time (min)	Water hardness (mmol l$^{-1)}$	
		Without Na-CMC	With addition of 1 mg l^{-1} Na-CMC
1	5	10.8	10.8
2	35	10.1	10.6
3	60	9.6	10.4
4	120	8.4	10.0
5	150	7.6	10.0
6	180	6.7	10.0
7	240	5.4	10.0
8	1,440	4.6	8.8

Content of Ca^{2+} and Mg^{2+} ions, and total hardness were determined by complexometric method. Total alkalinity was determined by titrimetric method. Errors of methods were 1.5–2.3%. Relative error in the experiments amounted to 10%.

3. Results and discussion

Na-CMC as a scale inhibitor of calcium carbonate was studied under static conditions. It was found (Table 4) that as little as 1 mg l^{-1} concentration of Na-CMC considerably inhibits the process of calcium carbonate crystallization in the volume of solution. As can be seen from the data provided, in the presence of inhibitor, the hardness of the solution reduces only by 7.4% within 4 h. When no inhibitor was used it reduced by 50% within the same time.

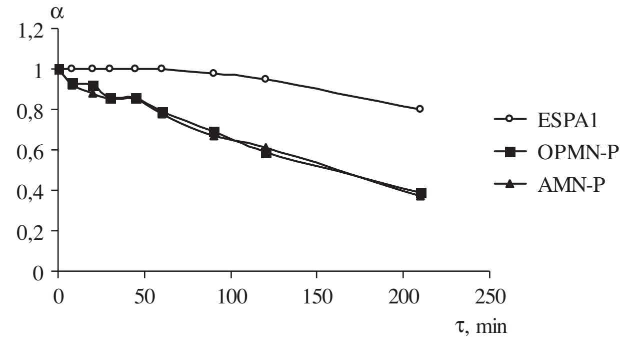

Fig. 3. The change of membranes' water flux in NF of artesian water with high carbonate hardness in absence of a scaling inhibitor ($P = 1.5$ MPa; $\upsilon = 1.5\ \mathrm{m\,s^{-1}}$; and $T = 21 \pm 1$°C).

Fig. 3 shows a change in the membranes' water flux when no inhibitor of scaling formation was used. Membranes AMN-P and OPMN-P demonstrate practically the same α value. Approximately after 10 min from the beginning of NF, this parameter decreased by 0.92 and after 3.5 h reached ≈0.38. The α value of ESPA1 membrane started to decrease only after 1.5 h. After 3.5 h, this parameter decreased by 0.80. Meanwhile, membranes' J_p value remained the same and was within the range of $1.60–1.78 \times 10^{-6}\ \mathrm{m\,s^{-1}}$. At the same time, the quality of filtrates obtained, as shown in Table 5, differed essentially.

Table 5
The characteristics of initial artesian water and permeate, after NF of artesian water without addition of scaling inhibitor

Parameter	Water composition			
	Initial	Permeate		
		ESPA1	AMN-P	OPMN-P
Hardness (mmol l^{-1})	9.2	1.0	2.0	1.4
Ca^{2+} (mg l^{-1})	178.4	16.0	32.0	20.0
Mg^{2+} (mg l^{-1})	21.5	2.4	4.1	4.9
Alkalinity (mmol l^{-1})	14	1.8	3.6	3.0
Fe_{total} (mg l^{-1})	0.24	0	0	0
Na^+ (mg l^{-1})	170.0	37.5	125.0	112.5
Rejection (Na^+) (%)	–	77.8	26.5	33.8
pH	7.8*; 8.1***	7.2**; 7.3***	7.6**; 7.7***	7.5**; 7.7***

Notes: *At the beginning in NF of artesian water; **pH values of permeate after 35 min in NF of artesian water; ***pH values of the permeate at the end (3.5 h) in NF of artesian water.

The NF raises the pH of artesian water from 7.8 to 8.1 while the pH of permeate of all membranes was lower than that pH of artesian water (see Table 5). It points to the negative selectivity of the negatively charged membranes to H^+. The pH of ESPA1 membrane permeate was lower than the pH of AMN-P and OPMN-P membranes permeates (see Table 5). It means that ξ-potential of ESPA1 membrane is higher than ξ-potential of AMN-P and OPMN-P membranes. It was confirmed by higher rejection (Na^+) of ESPA1 membrane which was 77.8%. The rejection (Na^+) of OPMN-P and AMN-P membranes was 33.8 and 26.5%, respectively (see Table 5).

Low rejection of fouled membranes compared to the rejection of virgin membranes may be explained by the decrease of the membranes' effective ξ-potential. It may be associated with two reasons: (1) the presence of multicharged ions in artesian water results in the compression of the electrical double layer of the membrane pores charged surface reducing its ξ-potential; (2) the blocking of carboxyl groups by Ca^{2+} ions, specifically, in AMN-P membrane with further formation of weak ionizing calcium salts of carboxylic acids reduces ξ-potential. That is the reason why, in our opinion, the rejection of AMN-P membrane is the lowest one (see Table 5). In addition, the probability of the formation of such salts increases with the increase in pH.

Preferential transfer of H^+ which results in the increase in artesian water pH contributes to the acceleration of calcium carbonate crystallization. Besides, the presence of iron in artesian water (see Table 5) and its inevitable coagulation during NF also helps accelerate calcium carbonate crystallization. The particles of iron hydroxide may act as the centers of the salt crystal nucleus.

Therefore, in the absence of inhibitor of calcium carbonate crystallization, the decrease in the membranes' water flux results from the intensive crystallization of calcium carbonate in artesian water and formation of its sediments in the pores and on the surface of membranes. Under such conditions, the characteristics of the fouled membranes depend on the properties of virgin membranes pore structure.

When Na-CMC was added to the artesian water NF took place with considerable decrease in scaling formation on the membranes (see Fig. 4).

Fig. 4 shows that J_0 of membranes OPMN-P and AMN-P decreased by 0.81 and 0.71 correspondingly, while in case of absence of an antioxidant inhibitor by 0.38. Water flux of the membrane ESPA1 decreased by 0.86 within the first 20 min, then it increased and stabilized at the value $\alpha = 0.90$. On the whole, quality of permeate improved considerably for all the membranes (Table 6). Comparing data provided in the Tables 5 and 6, we can see that membranes' rejection

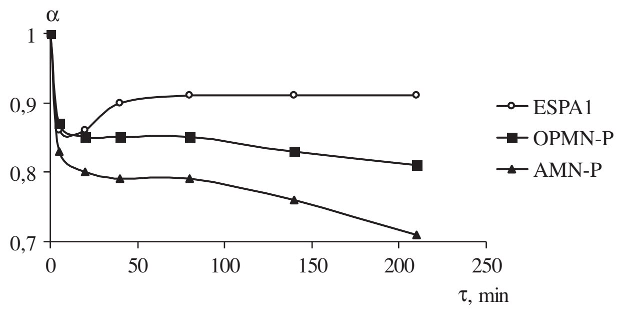

Fig. 4. The change of membranes' water flux in NF of artesian water with high carbonate hardness in the Na-CMC presence ($P = 1.5$ MPa; $\upsilon = 1.5\, m\, s^{-1}$; and $T = 21 \pm 1$°C).

Table 6
The characteristics of initial artesian water and permeate, after NF of artesian water at the presence of $1\,\mathrm{mg\,l^{-1}}$ Na-CMC

Parameter	Water composition			
	Initial	Permeate		
		ESPA1	AMN-P	OPMN-P
Hardness ($\mathrm{mmol\,l^{-1}}$)	9.4	0.5	1.5	1.0
Ca^{2+} ($\mathrm{mg\,l^{-1}}$)	165.3	10.0	28.0	22.0
Mg^{2+} ($\mathrm{mg\,l^{-1}}$)	22.0	2.0	3.5	4.5
Alkalinity ($\mathrm{mmol\,l^{-1}}$)	10.4	1.0	2.7	2.1
Fe_{total} ($\mathrm{mg\,l^{-1}}$)	0.30	0	0	0
Na^{+} ($\mathrm{mg\,l^{-1}}$)	154.0	23.0	91.0	68.0
Rejection (Na^{+}) (%)	–	85.1	40.9	56.0

increased with the presence of Na-CMC. Rejection of the membranes ESPA1, OPMN-P, and AMN-P increased from 77.8 to 85.1%, from 33.8 to 56%, and from 26.5 to 40.9% correspondingly.

We believe that the following factors determine reasons for the improvement of water flux and membranes' rejection. The graining process decreased in the presence of Na-CMC, as Na-CMC gets adsorbed on the originating water volumes of calcium carbonate microcrystals. This blocks their further growth and slows down calcium carbonate formation. In this case, according to the study [14], the surface of microcrystals is hydrophilizating. Na-CMC can also partially tie calcium as a result of ion exchange. Apart from the above, we can also explain that alongside with inhibitive effect as to the formation of calcium carbonate, polymer proves itself as a flocculant with regard to iron hydroxide. Interaction of the polymer's macromoles with colloidal ions of iron hydroxide which inevitably forms in NF of the artesian water, results in formation of the large colloidal ions and their sedimentation on the membranes. In consequence of this, complex, as to its composition, layer of sediment has formed on the membranes. Furthermore, structure and properties of the sediment depend on properties of virgin membranes. J_0 of the dense membrane ESPA1 decreased only by 0.90. This is indicative of formation of the coarse-porous spongy scale which does not demonstrate considerable hydrodynamic resistance. Increase of membrane's rejection is probably caused by the hydrophilic nature of the scale and presence of functional groups in its structure which have negative charge. In this particular case, these are carboxylic groups.

J_0 of the less dense membranes decreases much more (see Fig. 4). Difference of artesian waters kinetics during NF in the presence of Na-CMC of membranes OPMN-P and AMN-P can be explained by their different porous structure. J_0 of virgin membranes practically does not differ; rejection of the membrane AMN-P is lower than OPMN-P (see Table 6). It can be expected that in the event of membranes' equal effective porosity within the structure of the membrane AMN-P, there are some larger by diameter not selective pores. Meanwhile, there are less such pores in the membrane OPMN-P and predominate pores of small diameter. Therefore, J_0 decrease of the membranes OPMN-P and AMN-P can be caused first of all by the emergence of sediment in the membranes' pores. This happens if sludging particles are of the comparable or less size than pores' size. In this event, increase of membranes' rejection is achieved at the expense of new physicochemical and structural properties of the porous membranes' structure.

Therefore, virgin membranes acquire new properties as a result of formation of the sediment in the presence of Na-CMC. Water flux and membranes' rejection are defined by the properties of the new membranes' pore structure.

Fig. 5 shows change membranes' water flux in the artesian waters during NF with addition of inhibitor ACUMER low-molecular polymer. In the presence of ACUMER, water flux of the membrane ESPA1 does not decrease: $J_P = J_0$. Value α of the membrane OPMN-P and AMN-P are close to the value α in the presence of Na-CMC (Figs. 4 and 5). Thus, during 3.5 h in the presence of ACUMER α of the membranes OPMN-P and AMN-P is 0.81 and 0.71 correspondingly. It is 0.85 and 0.78 correspondingly with addition of Na-CMC. However, as Table 7 shows membranes' rejection has decreased in the presence of ACUMER.

In our opinion, the main reason of the difference of ACUMER and Na-CMC influence on the membranes' characteristics is different inhibition action mechanism of scale formation by the given polymers.

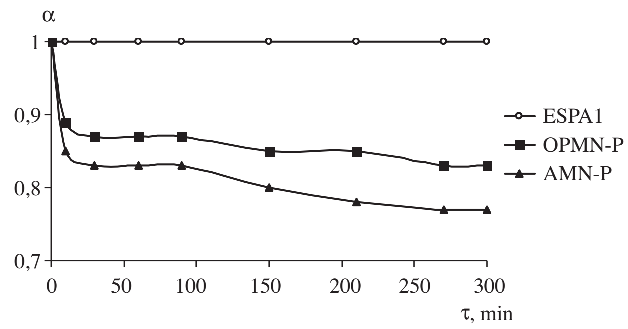

Fig. 5. The change of membranes' water flux in NF of artesian water with high carbonate hardness in the ACUMER presence ($P = 1.5$ MPa; $\upsilon = 1.5\,\mathrm{m\,s^{-1}}$; and $T = 21 \pm 1$°C).

ACUMER inhibition action, according to study [8], is carried out simultaneously by three mechanisms: (1) dispergating of calcium carbonate crystals; (2) polymer's adsorption on them; and (3) lysing of calcium carbonate. Inhibition action by the mechanism 1 increases dispersiveness, but leads to the pore blocking of the less dense membranes OPMN-P and AMN-P with the small calcium carbonate crystals. This decreases J_0 of the virgin membrane. J_0 of the dense membrane ESPA1 does not worsen as the size of its pores is smaller than the size of calcium carbonate crystals. Inhibition action by mechanism 3 leads to the emergence of Ca^{2+} in the water. Blocking of Ca^{2+} of carboxylic groups leads to the decrease in rejection of the membrane AMN-P (Table 7). Because water flux of the less dense membranes OPMN-P and AMN-P in the presence of ACUMER and Na-CMC is comparable, it can be explained that out of three mechanisms, the second (polymer's adsorption) makes main contribution to the inhibition action of the deposit formation with ACUMER. Decrease in their rejection is caused by the formation mainly crystalline sediment in the membranes' pores.

Thus, ACUMER's high inhibition ability in NF of hard artesian waters shows itself with the use of the dense membrane ESPA1. Membrane's water flux and rejection do not worsen. If less dense membranes are used inhibition of deposit formation ACUMER and Na-CMC is comparable by membranes' water flux.

4. Conclusions

(1) If inhibitor of calcium carbonate crystallization is absent, decrease in the membranes' water flux and rejection is caused by the intensive calcium carbonate crystallization in artesian waters and formation of its sediment in the pores and on the surface of the membranes. Under such conditions, properties of the virgin membranes pore structure have an effect on the characteristics of fouled membranes.

(2) Virgin membranes acquire new properties in the presence of Na-CMC due to formation of the complex by composition sediment layer. Membranes' water flux and rejection are determined by the properties of the membranes' pore structure, which is formed in NF.

(3) Process of scaling formation on the membranes in the presence of ACUMER depends on virgin membranes pore structure. ACUMER high inhibition ability shows itself with the use of dense membrane.

(4) Na-CMC inhibition ability with regard to water flux of the less dense membranes is comparable with ACUMER. Best inhibition ability has Na-CMC as to the rejection of these membranes.

(5) Models of inhibition action Na-CMC and ACUMER in NF of hard artesian waters are proposed with the use of different types of membranes.

Table 7
The characteristics of initial artesian water and permeate, after NF purification at the presence of $1\,\mathrm{mg\,l^{-1}}$ ACUMER

Parameter	Water composition			
	Initial	Permeate		
		ESPA1	AMN-P	OPMN-P
Hardness ($\mathrm{mmol\,l^{-1}}$)	9.3	0.5	1.5	1.0
Ca^{2+} ($\mathrm{mg\,l^{-1}}$)	175.4	12.0	32.0	22.0
Mg^{2+} ($\mathrm{mg\,l^{-1}}$)	21.0	2.0	3.5	4.5
Alkalinity ($\mathrm{mmol\,l^{-1}}$)	10.1	1.0	2.7	2.1
Fe_{total} ($\mathrm{mg\,l^{-1}}$)	0.35	0	0	0
Na^{+} ($\mathrm{mg\,l^{-1}}$)	165.0	25.0	103.5	94.4
Rejection (Na^{+}) (%)	–	84.8	37.3	42.8

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