

STUDY OF WASTEWATER TREATMENT EFFICIENCY USING A COMBINED METHOD OF FORWARD AND REVERSE OSMOSIS INTEGRATED WITH ACTIVATED CARBON TREATMENT**¹K.A. Kurtibay[✉], ²Ye.Ye. Zhatkanbayev, ¹A. Kappassuly, ¹A.A. Ussenova ³Zh.K. Zhatkanbayeva, ¹N.B. Moldagulova, ¹E.B. Moldagulova**¹«Scientific and Production Center of Ecological and Industrial Biotechnology» LLP,
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In Kazakhstan, the issue of providing the population with quality drinking water remains one of the topical and important issues. Despite significant efforts in the field of infrastructure and water treatment technologies, many regions of Kazakhstan still face problems of pollution and insufficient water resources. In this regard, the development of effective water treatment methods is of particular importance. This study considers the issue of wastewater treatment by the combined method of forward and reverse osmosis, which is a key direction in modern water treatment. The relevance of the work is the use of integration of forward osmosis methods with the process of adsorption by powdered activated carbon in wastewater treatment. The use of powdered activated carbon in the process of wastewater treatment leads to a reduction in the content of organic matter, which are water pollutants. As a result of the experiment with pretreatment of wastewater with powdered activated carbon, a decrease in the level of chemical oxygen demand (COD) in wastewater from 538 mg O/dm³ to 256 mg O/dm³ was observed, as well as a relative decrease in the concentration of other indicators. The draw solution in the form of 1.5 M NaCl gradually drawing water molecules from the feed solution pretreated with activated carbon on the tenth day had a concentration of 0.425 M absorbing 2.529 liters of water entering through the membrane that is 27.6% more than the control version of the experiment. The effectiveness of integrated methods lies in the successful combination of different technologies to improve water treatment processes and ensure the availability of clean water not only in Kazakhstan, but also in different parts of the world.

Key words: wastewater treatment, forward osmosis, wastewater, powdered activated carbon, chemical oxygen demand (COD), organic pollutants, reverse osmosis.

**БЕЛСЕНДІРІЛГЕН КӨМІРМЕН ӨҢДЕУМЕН ИНТЕГРАЦИЯЛАНҒАН ТІКЕЛЕЙ ЖӘНЕ КЕРІ
ОСМОС ӘДІСІН ҚОЛДАНА ОТЫРЫП, АҒЫНДЫ СУЛАРДЫ ТАЗАРТУ ТИІМДІЛІГІН
ЗЕРТТЕУ****¹Қ.А. Куртибай[✉], ²Е.Е. Жатқанбаев, ¹Ә. Қаппасұлы, ¹А.Ә. Үсенова, ³Ж.К. Жатқанбаева, ¹Н.Б. Молдагулова, ¹Ә.Б. Молдагулова**¹«Экологиялық және өнеркәсіптік биотехнологияның ғылыми-өндірістік орталығы» ЖШС,
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Қазақстанда халықты сапалы ауыз сумен қамтамасыз ету мәселесі өзекті және маңызды мәселелердің бірі болып қала береді. Инфрақұрылым және су дайындау технологиялары саласындағы елеулі күш-жігерге қарамастан, Қазақстанның көптеген өңірлері әлі де судың ластануы мен жеткіліксіздігі проблемаларына тап болып отыр. Осыған байланысты суды тазартудың тиімді әдістерін жасау ерекше маңызға ие. Бұл зерттеу ағынды суларды тікелей және кері осмостың аралас әдісімен тазарту мәселесін қарастырады, бұл қазіргі заманғы суды дайындаудың негізгі бағыты болып табылады. Жұмыстың өзектілігі ағынды суларды тазарту кезінде ұнтақталған белсендірілген көмірмен адсорбциялау процесімен тікелей осмос әдістерін интеграциялау арқылы қолдану болып табылады. Ағынды суларды тазарту процесінде ұнтақталған белсендірілген көмірді пайдалану суды ластайтын органикалық заттардың азаюына әкеледі. Ағынды суларды ұнтақталған белсендірілген көмірмен алдын ала өңдеу экспериментінің нәтижесінде ағынды сулардағы оттегінің химиялық тұтынылу (ОХТ) деңгейінің 538 мгО/дм³-тен 256 мгО/дм³-ке дейін төмендеуі, сондай-ақ басқа көрсеткіштер концентрациясының салыстырмалы төмендеуі байқалады. 1,5 М NaCl түріндегі тартқыш ерітіндісі су молекулаларын алдын ала белсендірілген көмірмен өңделген бастапқы ерітіндіден оныншы күні біртіндеп тартып, 0,425 М концентрациясына ие болып, мембрана арқылы өткен су 2,529 литрді құрады. Бұл эксперименттің бақылау нұсқасына қарағанда 27,6% - ға көп. Интеграцияланған әдістердің тиімділігі суды тазарту процестерін жақсарту және таза судың тек Қазақстанда ғана емес, әлемнің әр түкпірінде де қолжетімділігін қамтамасыз ету мақсатында түрлі технологияларды сәтті комбинациялаудан құралады.

Түйін сөздер: ағынды суларды тазарту, тікелей осмос, ағынды сулар, ұнтақталған белсендірілген көмір, оттегінің химиялық тұтынылуы (ОХТ), органикалық ластаушы заттар, кері осмос.

ИССЛЕДОВАНИЕ ЭФФЕКТИВНОСТИ ОЧИСТКИ СТОЧНЫХ ВОД С ИСПОЛЬЗОВАНИЕМ КОМБИНИРОВАННОГО МЕТОДА ПРЯМОГО И ОБРАТНОГО ОСМОСА, ИНТЕГРИРОВАННОГО С ОБРАБОТКОЙ АКТИВИРОВАННЫМ УГЛЕМ

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В Казахстане вопрос обеспечения населения качественной питьевой водой остается одним из актуальных и важных. Несмотря на значительные усилия в области инфраструктуры и технологий водоподготовки, многие регионы Казахстана по-прежнему сталкиваются с проблемами загрязнения и недостаточности водных ресурсов. В связи с этим особое значение приобретает разработка эффективных методов очистки воды. В данном исследовании рассматривается вопрос очистки сточных вод комбинированным методом прямого и обратного осмоса, который является ключевым направлением в современной водоподготовке. Актуальностью работы является использование интеграции методов прямого осмоса с процессом адсорбции порошкообразным активированным углем при очистке

сточных вод. Использование порошкообразного активированного угля в процессе очистки сточных вод приводит к уменьшению содержания органических веществ, которые являются загрязняющими воду. В результате эксперимента с предварительной обработкой сточных вод порошкообразным активированным углем наблюдается снижение уровня химического потребления кислорода (ХПК) в сточной воде с 538 мг О/дм³ до 256 мг О/дм³, а также относительное уменьшение концентрации других показателей. Вытяжной раствор в виде 1,5 М NaCl постепенно вытягивая молекул воды с исходного раствора предварительно обработанного активированным углем на десятый день имел концентрацию 0,425 М поглощая 2,529 литров поступающей через мембрану воды что на 27,6% больше, чем на контрольном варианте эксперимента. Эффективность интегрированных методов заключается в успешном сочетании различных технологий с целью улучшения процессов очистки воды и обеспечения доступности чистой воды не только в Казахстане, но и в различных уголках мира.

Ключевые слова: очистка сточных вод, прямой осмос, сточные воды, порошкообразный активированный уголь, химическое потребление кислорода (ХПК), органические загрязнители, обратный осмос.

Introduction. In the modern world, the problem of access to clean drinking water remains one of the most urgent, affecting the health and well-being of mankind. In Kazakhstan, the issue of providing the population with quality drinking water remains one of the urgent and important.

The main pollutants in both natural and polluted water are microorganisms, as well as organic and inorganic substances. Wastewaters are solid and liquid substances in water that enter the sewer system as a by-product of society's activities. They include dissolved and suspended organic solids that undergo decomposition or biodegradation [1]. Over time, domestic and industrial wastewater increasingly contains non-biodegradable organic compounds such as humic substances, which reduce the effectiveness of low-cost wastewater treatment methods [2].

Forward osmosis is used to treat municipal wastewater that is treated in municipal wastewater treatment plants [3]. Forward osmosis membrane systems can remove large ions and concentrate wastewater 10-15 times [4]. Forward osmosis can be integrated with reverse osmosis in a hybrid system. In this case, forward osmosis is used to pre-treat wastewater to produce high quality water, which is then used to dilute seawater before the reverse osmosis step. Another study [5] used a hybrid process combining direct osmosis and membrane distillation to remove tetracycline from wastewater. This method provided a purification

rate of 99.9%, and the water recovery efficiency was 15-22%. In addition to water recovery, forward osmosis can be used to treat wastewater to extract nutrients and generate energy [6]. Examples of such recovery include biogas production and recovery of valuable components such as phosphates, ammonia, and potassium. Moreover, forward osmosis can help to improve the environmental sustainability of treatment processes by reducing energy costs and decreasing the burden on the environment [6].

As a result, numerous physicochemical treatment methods such as coagulation, flocculation, adsorption and accelerated oxidation have been proposed and investigated for effective wastewater treatment [7]. However, physicochemical treatment methods alone are not sufficient to completely remove various organic pollutants from wastewater. To address these limitations, the use of membrane filtration processes in combination with these methods has been proposed. Membrane filtration methods such as nanofiltration (NF) and reverse osmosis (RO) have been successfully used for wastewater treatment. However, operation at high transmembrane pressure increases operating costs and leads to significant organic fouling of the membrane [8,9].

From an economic point of view, the forward osmosis (FO) process is superior to nanofiltration (NF) and reverse osmosis (RO) due to several advantages. These advantages include efficient removal of organic and inorganic contaminants,

no need for external hydraulic pressure, and less membrane fouling with better reversibility [10]. As previously stated, the application of membrane filtration techniques in integration with pretreatment by physicochemical methods such as, coagulation, flocculation, magnetic ion exchange resins and powdered activated carbon to avoid membrane fouling shows high efficiency [11].

Pretreatment of wastewater to remove organic pollutants with powdered activated carbon has attracted attention due to its advantages in conjunction with membrane filtration processes. Powdered activated carbon provides improved membrane surface cleaning, effective reduction of irreversible fouling and removal of organic matter.

Currently, biochar stands out as a promising alternative with additional advantages such as environmental sustainability, low production cost, soil fertility enhancement and carbon sequestration. Biochar also possesses additional cationic functional groups, which makes it easy to modify its surface properties to improve functionality [12].

The use of adsorption pretreatment for partial removal of organic pollutants provides reduction of chemical oxygen demand (COD) of wastewater.

Methods and Materials. As an object of study for wastewater treatment by direct osmosis method, incoming wastewater was taken. The wastewater sample was collected in the volume of 10 liters at the sewage treatment plant (STP) located in the vicinity of Astana, Kazakhstan.

To determine the efficiency integrated using powdered activated carbon, experiments were performed without adsorption step and with pretreatment with powdered activated carbon for adsorption to investigate a larger flow of clean water. 1.5 M NaCl was used as a draw solution with higher osmotic pressure than the stock solution. The concentration of the stock and draw solutions was measured every 24 hours for 10 days.

Adsorption of wastewater using powdered activated carbon

For adsorption, untreated wastewater is filtered through filter paper to remove coarse suspended solids and the filtrate in a total volume of 500

cm³ is placed in Erlenmeyer flasks, then 3.5 g/L powdered activated carbon is added to the filtrate. The flasks were then placed on a horizontal shaker for intensive-continuous stirring at 150 r/min for 24 hours at room temperature (25-28°C). During the experiment, the pH of the solution was maintained at 7 by adding 5M HCl or 5M NaOH every 4 hours. After adsorption, the solution was filtered through a paper filter with a pore diameter of 0.45 µm and the resulting filtrate was stored for the following analyses [13]. After treatment of the initial sample by adsorption with powdered activated carbon, a general physicochemical analysis was performed.

Physico-chemical methods of research

Refractometry. To determine the concentration of sodium chloride and sucrose solutions, the refractometry method was used using a refractometer of Abbemat 350/550 Performance Plus series (Anton Paar, Austria). All analyses were performed in triplicate.

Determination of chemical oxygen demand (COD). COD was measured according to GOST 31859-2012 “Water. Method for determination of chemical oxygen demand” on the Expert 003 device (spectrophotometer with thermoreactor). Calibration of the device was carried out with solutions of the standard sample of chemical oxygen consumption GSO 7552-99. The device has a function of built-in construction of calibration curve and automatic determination of COD value.

Determination of chloride ions. Determination of chloride content was carried out according to GOST 26425-85 “Method for determination of chloride in water extract”, the method of determination - titration by silver nitrate solution with potassium bichromate indicator.

Determination of ammonium nitrogen. Determination of ammonium and nitrate nitrogen according to GOST 15476-2013 “Fertilizers. Determination of nitrate and ammonium nitrogen by the Devard method”.

Determination of nitrates and nitrites. Determination of nitrates and nitrites in water was carried out according to GOST 33045-2014 “Water. Methods of determination of nitrogen-

containing substances”, the method of determination by photocalorimetry.

Determination of phosphate ions. Determination of phosphate content in water according to GOST 18309-2014 “Water. Methods of determination of phosphorus-containing substances”.

Determination of suspended solids. Suspended solids were determined according to PND F 14.1:2:4.254-09 “Methods of measuring mass concentrations of suspended and calcined suspended solids in drinking, natural and waste water samples by gravimetric method”.

Processing of the obtained results

The osmotic pressure of solutions with known concentration was determined by the osmotic pressure (π) Vant-Goff equation:

$$\pi = C(x)RT \quad (1)$$

The osmotic pressure gradient of the initial and extraction solutions was determined by the equation:

$$\Delta\pi = \pi_{DS} - \pi_{FS} \quad (2)$$

Water flux is determined by water transport across the semipermeable membrane due to osmotic pressure difference by equation [14]:

$$J_w = A(\pi_{DS} - \pi_{FS}) \quad (3)$$

The analysis and processing of the obtained data was carried out through calculations and equations, and visualizations in the form of graphs and charts were constructed using Microsoft Excel software (Office 16)

Results and discussion. Before the beginning of all experiments the initial general physico-chemical analysis of the selected sample of incoming wastewater of the sewage treatment plant of Astana city was carried out. All chemical analyses were carried out in accordance with the State standards. The results of the general physico-chemical analysis are given in Table 1.

Table 1 - Indicators of physico-chemical analysis of incoming wastewater of the sewage treatment plant of Astana city

Name of physico-chemical parameters	Results
pH	7,83
Temperature	22
COD, mg O/dm ³	538
Suspended solids, mg/ dm ³	2584
Phosphorus (PO ₄), mg/ dm ³	3,69
Ammonium nitrogen, mg/ dm ³	18,34
Nitrite nitrogen, mg/ dm ³	1,51
Nitrate nitrogen, mg/ dm ³	8,26
Chlorides, mg/ dm ³	13,2
Ash content, %	128

According to the results of the general physico-chemical analysis we can notice a high level of water pollution by organic substances, which is estimated by the value of chemical oxygen demand (COD) of the object under study. In this case, the incoming wastewater of the sewage treatment plant of Astana city has COD - 538 mg O/dm³. Also relatively high content of nitrate, nitrite,

ammonium, phosphate and chloride ions, exceeding the maximum permissible concentration (MPC). The hydrogen index of this wastewater is 7.83, which characterizes the alkalinity of this sample.

Adsorption of wastewater using powdered activated carbon

For adsorption, the untreated wastewater is

filtered through filter paper to remove coarse suspended solids and the filtrate in a total volume of 500 cm³ is placed in Erlenmeyer flasks, then 3.5 g/L powdered activated carbon is added to the filtrate. The flasks were then placed on a horizontal shaker for intensive-continuous stirring at 150 r/min for 24 hours at room temperature (25-28°C). During the experiment, the pH of the solution was maintained at 7 by adding 5M HCl or 5M NaOH every 4 hours.

After adsorption, the solution was filtered through a paper filter with a pore diameter of 0.45 µm and the resulting filtrate was stored for the following analyses [13]. After treatment of the initial sample by adsorption with powdered activated carbon, a general physicochemical analysis was carried out.

The results obtained after adsorption are shown in Table 2.

Table 2 - Indicators of general physicochemical analysis after adsorption

Name of physico-chemical parameters	Results
pH	7,71
Temperature	25
COD, mg O/dm ³	256
Suspended solids, mg/ dm ³	367
Phosphorus (PO ₄), mg/ dm ³	3,25
Ammonium nitrogen, mg/ dm ³	17,67
Nitrite nitrogen, mg/ dm ³	1,42
Nitrate nitrogen, mg/ dm ³	8,11
Chlorides, mg/ dm ³	12,84
Ash content, %	84

According to the results obtained after the analysis with pretreatment with powdered activated carbon we can observe a decrease in COD level of wastewater from 538 mg O/dm³ to 256 mg O/dm³. And also, relative decrease of concentration of other indicators.

Integration of the adsorption process with a forward osmosis system

The forward osmosis system used was a plant that was designed for desalination of sea salt water. The process of concentrating wastewater to produce clean secondary water was carried out in a similar way to the forward osmosis desalination method, since the basic principle of this method is the same for all types of treatment, recovery and concentration. The methods differ only in that, depending on the purpose, chemical composition, concentration and osmotic pressure of the feed solution, different draw solutions are used, respectively higher osmotic pressure than the feed solution for greater water flux through

the semipermeable membrane. And also, different membranes of different nature are applied relative to the purpose of the solution.

To determine the efficiency integrated using powdered activated carbon, experiments were performed without adsorption step and with pretreatment with powdered activated carbon for adsorption to investigate the higher flow of pure water. 1.5 M NaCl was used as a draw solution with higher osmotic pressure than the feed solution. Concentration measurements of feed and draw solutions were made every 24 hours for 10 days. The water flux was measured by evaluating the change in concentration and osmotic pressure of sodium chloride in the feed and draw solutions, as well as the change in the volume of solution in the draw solution compartment. The concentration of sodium chloride was measured by refractometry.

The results of experiments on water treatment by forward osmosis method without pretreatment and by complex method are given in Tables 3-4.

Table 3 - Wastewater treatment by forward osmosis without pretreatment with powdered activated carbon

Time, days	0	2	4	6	8	10
COD, mg O/dm ³	538	1216	1932	2367	2527	2714
NO ₃ ⁻ , mg O/dm ³	8,26	18,66	29,96	36,7	39,18	42,08
NH ₄ ⁺ , mg O/dm ³	18,34	41,45	66,52	81,48	86,99	93,43
PO ₄ ⁻ , mg O/dm ³	3,69	8,34	13,38	16,39	17,5	18,8
NO ₂ ⁻ , mg O/dm ³	1,51	3,41	5,47	6,7	7,16	7,69
Cl ⁻ , mg O/dm ³	13,2	29,81	47,82	58,57	62,59	67,22
Concentration of draw solution, mol/L	1,5	1,252	1,128	0,985	0,912	0,884
Osmotic pressure of draw solution π , Pa	–	3,102*10 ³	2,795*10 ³	2,440*10 ³	2,260*10 ³	2,143*10 ³
Gradient of osmotic pressure $\Delta\pi$, Pa	–	2,054*10 ³	1,850*10 ³	1,616*10 ³	1,497*10 ³	1,450*10 ³
Water flux J _w , L*m ⁻² *h ⁻¹	–	20,54	18,5	16,15	14,97	14,5
Volume of clean water, ml	–	0,198	0,131	0,198	0,122	0,054

Table 4 - Wastewater treatment by integrated forward osmosis method with pretreatment with powdered activated carbon

Time, days	0	2	4	6	8	10
COD, mg O/dm ³	256	1024	1792	2612	2965	3288
NO ₃ ⁻ , mg O/dm ³	8,11	32,44	56,77	82,75	93,9	104,16
NH ₄ ⁺ , mg O/dm ³	17,67	70,68	123,69	180,29	204,58	226,94
PO ₄ ⁻ , mg O/dm ³	3,25	13	22,75	33,16	37,63	41,74
NO ₂ ⁻ , mg O/dm ³	1,42	5,68	9,94	14,48	16,43	18,23
Cl ⁻ , mg O/dm ³	12,84	51,36	89,88	130,93	148,56	167,84
Concentration of draw solution, mol/L	1,5	1,056	0,884	0,693	0,512	0,425
Osmotic pressure of draw solution π , Pa	–	2,616*10 ³	2,190*10 ³	1,717*10 ³	1,269*10 ³	1,053*10 ³
Gradient of osmotic pressure $\Delta\pi$, Pa	–	2,386*10 ³	1,998*10 ³	1,566*10 ³	1,157*10 ³	0,961*10 ³
Water flux J _w , L*m ⁻² *h ⁻¹	–	23,86	19,98	15,66	11,57	9,61
Volume of clean water, ml	–	0,42	0,249	0,498	0,763	0,599

A forward osmosis system without adsorbent pretreatment of the feed solution was taken into consideration as a control for this experiment, which allowed us to compare the water flux through the membrane and membrane fouling potential with

the integrated forward osmosis method with pre-adsorption by powdered activated carbon.

The integrated method with pre-adsorption with powdered activated carbon resulted in a significant

increase in the maximum water flux, which is observed after 2 days from the beginning of the experiment compared to the control by 16.17%. In addition, judging by the results obtained, it can be seen that after pretreatment of wastewater with activated carbon, the membrane fouling potential decreases, at the expense of this wastewater in the compartment for feed water is more quickly concentrated losing water molecules, and the draw solution is diluted reducing its osmotic pressure to the onset of osmotic pressure equilibrium in the two compartments. The draw solution in the form of 1.5 M NaCl gradually drawing water molecules from the feed solution on day 10 had a concentration of 0.425 M absorbing 2.529 liters of water entering through the membrane, which is 27.6% more than the control version of the experiment.

Concentrated wastewater with a significantly higher content of organic matter can serve as an alternative substrate for anaerobic digestion [15], most often carried out for biogas production. The high content of organic matter in concentrated wastewater is evidenced by COD values, which reached 2714 mg O/dm³ in the control, and in the experiment with pretreatment due to high water flux concentrated to 3288 mg O/dm³ despite the fact that during adsorption part of the organic matter remained on the sorbent.

Pure water recovery by reverse osmosis method

To obtain clean secondary water for domestic and agricultural use, a number of methods are used today. One of these methods is the reverse osmosis method. In comparison with forward osmosis, reverse osmosis has a number of disadvantages such as high energy consumption, high degree of membrane fouling, scaling, etc. Therefore, modern researchers have proposed a hybrid method “FO-RO”, a method of forward and reverse osmosis [16].

After completion of wastewater treatment by forward osmosis, a dilute draw solution remains from which pure water must be extracted. Extraction of pure water by thermal distillation requires a lot of electrical energy and takes more time. Thus, it was decided to recover pure water from dilute solutions using «Atoll» reverse osmosis systems with a pure water flux capacity of 7.5 liters

per hour.

Conclusions. Application of adsorption method by powdered activated carbon in wastewater treatment allows to reduce the concentration of organic substances that pollute water. According to the results of the experiment with pretreatment of wastewater with powdered activated carbon we can observe a decrease in the level of COD of wastewater from 538 mgO/dm³ to 256 mgO/dm³. As well as a relative decrease in the concentration of other indicators.

The use of the integrated method of pre-adsorption of wastewater by powdered activated carbon led to a significant increase in the maximum water flux, which is observed after 2 days from the beginning of the experiment compared to the control by 16.17%. In addition, judging by the results obtained, it can be seen that after pretreatment of wastewater with activated carbon, the membrane fouling potential decreases, at the expense of this wastewater in the compartment for feed water is more quickly concentrated losing water molecules, and the draw solution is diluted reducing its osmotic pressure to the onset of osmotic pressure equilibrium in the two compartments. The draw solution in the form of 1.5 M NaCl gradually drawing water molecules from the feed solution on day 10 had a concentration of 0.425 M absorbing 2.529 liters of water entering through the membrane, which is 27.6% more than the control version of the experiment. After completion of wastewater treatment by forward osmosis method, there remains a dilute draw solution from which pure water should be extracted. Extraction of pure water by thermal distillation requires a lot of electricity and takes more time. Therefore, it was decided to recover pure water from diluted solutions by reverse osmosis method using reverse osmosis systems «Atoll», the capacity of pure water flux of 7.5 l/h. Which proves the effectiveness of the combined method «FO-RO».

Thus, the results obtained at the end of the experiments indicate that wastewater treatment using the forward osmosis method, as well as the integration of this process with the activated carbon adsorption method, represent a cost-effective

and efficient approach to obtaining clean water processes and increase the availability of clean water for reuse in various industries and agriculture. not only in Kazakhstan, but also in other regions of These integrated methods can successfully combine the world. different technologies to improve water treatment

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