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Studying the characteristics of iodine sorption in synthetized ion-exchangers

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ABSTRACT
Iodine is an essential micronutrient for humans and animals due to its important role as a component of thyroid hormones. Kazakhstan is a country, most regions of which suffer from a natural deficiency of iodine. At the same time, the country has a rich resource base for obtaining iodine and its compounds. These are formation waters of oil fields under development containing 0.001–0.005% iodine. To extract iodine from natural oil-field brines, we have proposed ion-exchange materials obtained from the waste of the oil refining industry. The article presents the results of studying the characteristics of iodine sorption in the synthesized ion exchangers. The synthesis of ion exchangers was carried out by polycondensation of dihydric phenols (1,3 dioxybenzene, 1,2 dioxybenzene, 1,4 dioxybenzene), hexamethylenediamine, and formaldehyde. The following sorption characteristics of iodine by the synthesized ion exchangers were studied: the influence of the pH of a solution on iodine sorption; kinetic curves of iodine sorption; iodine sorption isotherms; mechanism of iodine sorption. It has been established that the sorption of iodine on the synthesized samples is essentially independent of the pH of a medium, and the degree of its extraction in the entire area under study is 94–100%. Studies have been carried out on the dependence of the degree of extraction and the logarithm of the iodine distribution coefficient on the duration of the process. The sorption capacity of the synthesized ion exchangers with respect to iodine was estimated depending on the structure of the ion exchanger and sorption conditions. It has been established that sorbents based on 1,4 dioxybenzene, which are distinguished by high kinetic abilities and static exchange capacity (SEC = 2283.88 mg/g), are most preferable for iodine extraction. The mechanism of sorption of iodine by synthesized ion exchangers has been determined.

Keywords: *iodine sorption, sorption capacity, synthesis of ion exchangers, sorption isotherm, iodide leaching, iodine.*

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Introduction

Iodine is an essential micronutrient for humans and animals due to its important role as a component of thyroid hormones [1]. Kazakhstan is a country, most regions of which suffer from a natural deficiency of iodine. This is due to natural and climatic conditions, due to which iodine can scarcely be found in foods of plant and animal origin [[2], [3]]. At the same time, the country has a rich resource base for obtaining iodine and its compounds. These are formation waters of oil fields under development containing 0.001–0.005% iodine [4].

Interest in iodine sorption was caused by the possibility of using natural iodine-containing raw materials for processing, as well as the prospect of using iodide solutions in the technology of gold recovery [[4], [5]].

The implementation of the iodide method of gold leaching is possible in the development of effective, including sorption methods for extracting iodine from iodine-containing natural solutions. The efficiency of sorption strongly depends on soil types and their characteristics. While sorption of iodide dominates in mineral soils with a low content of organic carbon, soils rich in organic matter are more favorable for the sorption of iodate [6].

Various ion exchangers from activated carbons [[7], [8], [9], [10]] to synthesized ion exchangers [11, 12] are used for sorption recovery.

Thus, the organization of regeneration of iodine-containing solutions, as well as replenishment of irretrievable losses of iodine due to its extraction from natural sources by highly efficient sorption methods, is an important scientific and practical task.

Experimental Part

To extract iodine from natural oil-field brines, we have proposed ion-exchange materials obtained from the waste of the oil refining industry.

Sorption of iodine from aqueous iodine-containing solutions was carried out on synthesized ion exchangers.

The method for obtaining synthesized ion exchangers. The synthesis of amine-containing ion exchangers with a spatial structure of mixed basicity was carried out by polycondensation of dihydric phenols (1,3 dioxybenzene, 1,4 dioxybenzene, 1,2 dioxybenzene), hexamethylenediamine, and formaldehyde [13].

The method for obtaining synthesized ion exchangers. The synthesis of amine-containing ion Hexamethylenediamine was used in the synthesis process as a hardener for the resulting resins, and formaldehyde was used as a crosslinking agent. Ammonium hydroxide, nitric and phosphoric acids, and hydrogen peroxide were used as catalyzing and modifying additives. The latter are involved in the process not only as catalysts but also as reagents that facilitate the introduction of additional ionogenic groups into the polymer structure. The following sorption characteristics of iodine by the synthesized ion exchangers were studied: the influence of the pH of a solution on iodine sorption; sorption kinetics; sorption isotherms; mechanism of iodine sorption.

Results and Discussion

Influence of the pH of a solution on the results of iodine sorption.

The sorption of iodine was studied from aqueous solutions with a concentration of ~200 mg/dm^3 in a wide range of pH values for 24 hours.

The results of the sorption of iodine from its solutions by synthesized ion exchangers depending on the pH of the medium are shown in Table 1 and Figure 1.

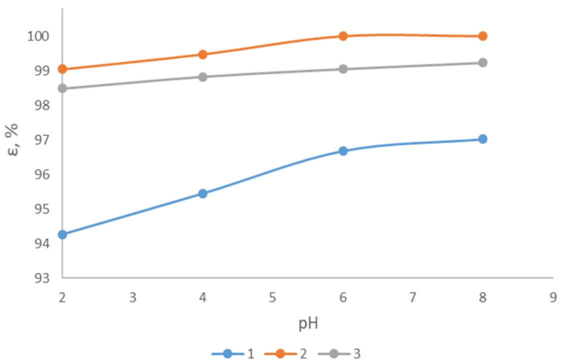
Studies have shown that the sorption of iodine on the synthesized samples is essentially independent of the pH of the medium, and the degree of its extraction in the entire area under study is 94–100% (Table 1, Figure 1).

Influence of the duration of the sorption process on the extraction of iodine.

Sorption of iodine (kinetic curves of iodine sorption) was conducted under the following conditions: C_{I_2} = 200.58 mg/dm^3; $V_{Solution}$= 500 cm^3; $m_{Ion\text{-}Exchanger}$ = 0.0998 g; pH 7.4. The results of the influence of the duration of the sorption process on the extraction of iodine are shown in Table 2 and Figure 2.

Table 1 – Influence of the pH of the solution on the results of iodine sorption by amine-containing ion exchangers

Ion exchanger code	pH of the initial solution	$C_{Equilibrium}$, mg/dm^3	Kp, mL/g	lg K_p	Static Exchange Capacity, mg/g	ε, %
1	2.25	11.48	82199.95	4.92	943.66	94.27
	4.04	9.11	104884.03	5.02	955.49	95.45
	5.11	6.65	145531.01	5.16	967.78	96.68
	7.85	5.97	162676.35	5.21	971.18	97.02
2	2.25	1.90	521846.05	5.72	991.51	99.05
	4.04	1.04	957503.07	5.98	995.80	99.48
	5.11	–	–	–	1001.00	100.00
	7.85	–	–	–	1001.00	100.00
3	2.25	3.02	326461.29	5.51	985.91	98.49
	4.04	2.34	422781.92	5.63	989.31	98.83
	5.11	1.90	521846.05	5.72	991.51	99.05
	7.85	1.54	645003.70	5.81	993.31	99.23
Note: polymeric ion exchangers are synthesized on the basis of: 1 – 1,3 dioxybenzene, 2 – 1,4 dioxybenzene, 3 – 1,2 dioxybenzene						

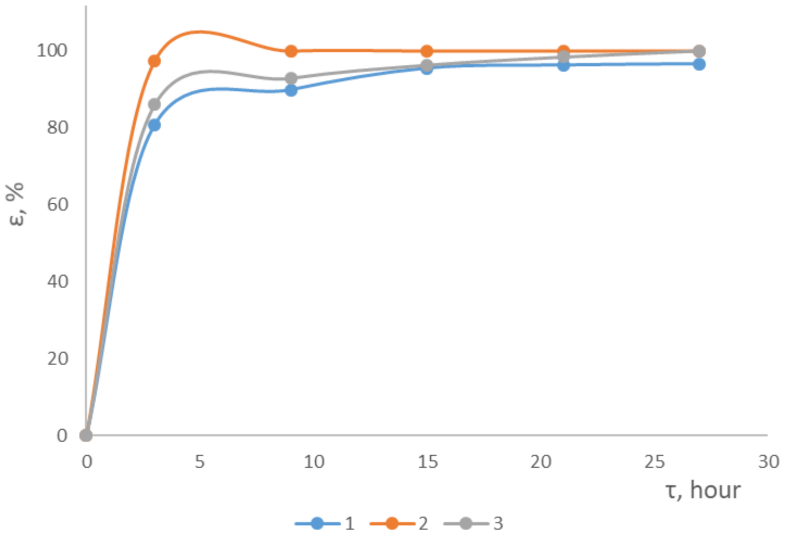


1 – 1,3 dioxybenzene; 2 – 1,4 dioxybenzene; 3 – 1,2 dioxybenzene

Figure 1 – Influence of the pH of stock solutions on the sorption of iodine by amine-containing ion exchangers

Table 2 – Dependence of the results of iodine sorption by synthesized ion exchangers on the duration of the process

Ion exchanger code	τ, hour	$C_{Equilibrium}$, mg/dm^3	Kp, mL/g	lg K_p	Static Exchange Capacity, mg/g	ε, %
1	3	38.71	20949.85	4.32	810.97	80.7
	9	20.46	44105.63	4.64	902.40	89.8
	15	9.23	103863.86	5.02	958.66	95.4
	21	7.62	126614.17	5.10	964.80	96.2
	27	7.02	138138.97	5.14	969.74	96.5
2	3	5.22	187500.68	5.27	978.75	97.4
	9	–	–	–	1004.91	100.0
	15	–	–	–	1004.91	100.0
	21	–	–	–	1004.91	100.0
	27	–	–	–	1004.91	100.0
3	3	28.08	30777.24	4.49	864.23	86.0
	9	14.24	65559.23	4.82	933.56	92.9
	15	7.42	130422.04	5.12	967.73	96.3
	21	3.21	308044.76	5.49	988.82	98.4
	27	–	–	–	1004.91	100.0
Note: polymeric ion exchangers are synthesized on the basis of: 1 – 1,3 dioxybenzene, 2 – 1,4 dioxybenzene, 3 – 1,2 dioxybenzene.						



1 – 1,3 dioxybenzene; 2 – 1,4 dioxybenzene; 3 – 1,2 dioxybenzene

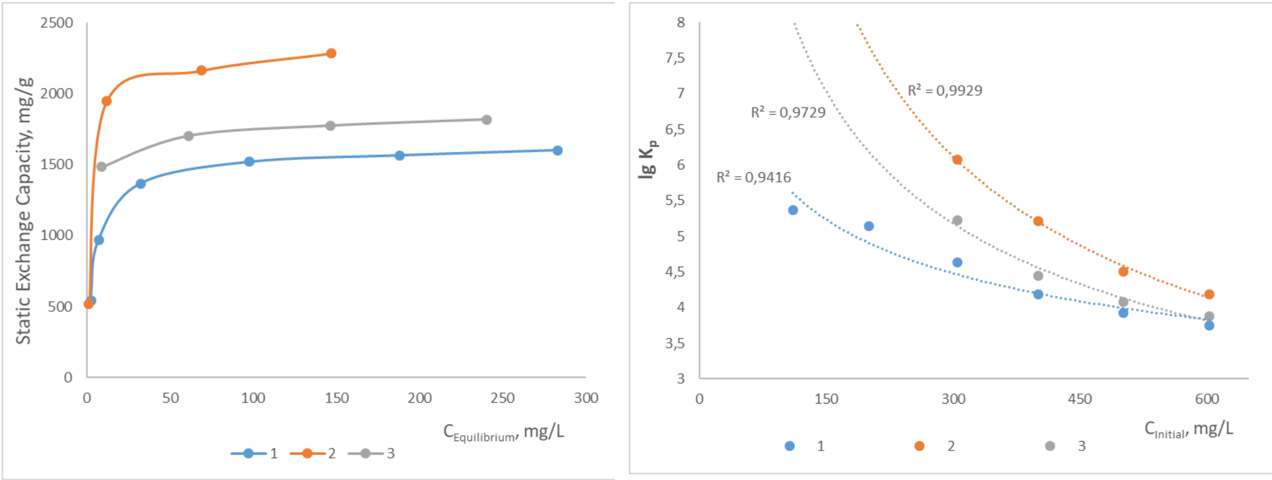
Figure 2 - Dependence of the degree of iodine extraction on the duration of the process

Table 2 and Figure 3 show that preference should be given to the ion exchanger based on 1,4 dioxybenzene, whose sorption kinetic curves are the highest. The maximum extraction of iodine by sorption by the ion exchanger based on 1,4 dioxybenzene is achieved in 9 hours, while it takes 27 hours or more to obtain the same result with 1,3 dioxybenzene, 1,2 dioxybenzene ion exchangers.

Ion exchange isotherms. Ion exchange isotherms were built by the method of variable concentrations using the same volumes of iodine-containing solutions (0.5 dm^3) and weighed portions of ion exchangers (0.1 g) for 27 hours. The pH value of the solutions was maintained in the found optimal range. Sorption isotherms and dependences of the logarithm of the iodine distribution coefficient are presented in Table 3 and Figure 3.

Table 3 - Results of iodine sorption by synthesized ion exchangers depending on its initial concentration

Ion exchanger code	$C_{Initial}$, mg/L	$C_{Equilibrium}$, mg/L	K_p, mL/g	lg K_p	Static Exchange Capacity, mg/g	ε, %
1	110.41	2.32	233185.8	5.37	540.99	97.9
	200.58	7.02	138139.0	5.14	969.74	96.5
	305.32	32.15	42526.2	4.63	1367.22	89.5
	401.13	97.48	15590.6	4.19	1519.77	75.7
	500.88	188.06	8325.4	3.92	1565.67	62.5
	603.24	283.14	5658.3	3.75	1602.10	53.1
2	110.41	–	–	–	552.60	100.0
	200.58	–	–	–	1003.60	100.0
	305.32	1.26	1207795	6.08	521.82	99.6
	401.13	11.90	163705.7	5.21	1948.10	97.0
	500.88	68.78	31443.2	4.50	2162.66	86.3
	603.24	146.92	15545.1	4.19	2283.88	75.6
3	110.41	–	–	–	552.60	100.0
	200.58	–	–	–	1003.60	100.0
	305.32	8.76	169438.4	5.23	1484.28	97.1
	401.13	60.90	27961.5	4.45	1702.85	84.8
	500.88	146.54	12102.3	4.08	1773.47	70.7
	603.24	240.32	7558.3	3.88	1816.42	60.2
Note: polymeric ion exchangers are synthesized on the basis of: 1 – 1,3 dioxybenzene, 2 – 1,4 dioxybenzene, 3 – 1,2 dioxybenzene, $pH_{initial}$ ~ 7.4						



1 – 1,3 dioxybenzene; 2 – 1,4 dioxybenzene; 3 – 1,2 dioxybenzene

Figure 3 - Isotherms of iodine sorption by amine-containing ion exchangers

It can be seen from the data obtained that the isotherms of sorption of iodine on ionites have a convex ($C_{Equilibrium} > 1$) shape. The slopes of the logarithmic dependences of the distribution coefficients of iodine on its initial concentration in an aqueous solution slightly differ from each other, which allows us to make an assumption that the mechanism of halogen sorption by all synthesized ion exchangers is identical. The apparent equilibrium constants for iodine can be arranged in the following series:

$$C_{Equilibrium\ 2} > C_{Equilibrium\ 3} > C_{Equilibrium\ 1},$$

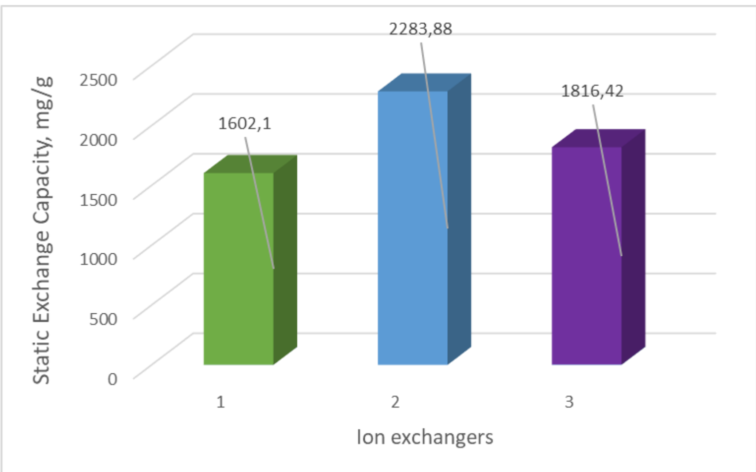
where indices 1–3 denote the corresponding codes of the synthesized ion exchangers.

Figure 4 shows the comparative sorption characteristics of the synthesized ion exchangers.

It can be seen that the sorption capacity of all synthesized ion exchangers for iodine is high and exceeds 1600 mg/g. The ion exchanger synthesized on the basis of 1,4 dioxybenzene has the best sorption characteristics (Static Exchange Capacity = 2283.88 mg/g).

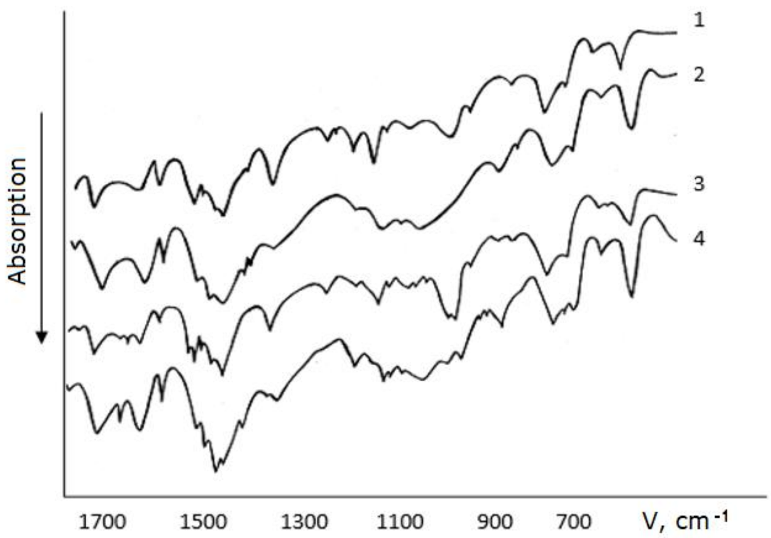
The results obtained significantly exceed the sorption rates of the highly basic anion exchanger AV-17 (Static Exchange Capacity = 300 mg/g), which is widely used in the iodine-bromine industry, as well as those of EDE-10P (Static Exchange Capacity = 500 mg/g) [13].

To establish the mechanism of iodine sorption, the IR spectra of sorbents were studied (Figure 5).



1 – 1,3 dioxybenzene; 2 – 1,4 dioxybenzene; 3 – 1,2 dioxybenzene

Figure 4 - Sorption characteristics of synthesized ion exchangers in relation to iodine



1,4 dioxybenzene saturated with water (1) and iodine (2);
1,2 dioxybenzene saturated with water (3) and iodine (4)

Figure 5 - IR spectra of sorbents

After the contact of ion exchangers with a sulphate solution of potassium iodide, the spectra show changes at 1700–1300 cm^{-1}, where C=C valence bonds and deformation –C–H groups appear.

The increase in the intensities of the absorption bands of bending vibrations of $-CH_3$ and $-CH_2$ groups (3000–2800 cm^{-1}) is also associated with iodine sorption. The coordination of iodine to the nitrogen of the amino group is shown by changes in the spectra at 1100–1200 cm^{-1}.

Thus, it can be concluded that the sorption of iodine by ion exchangers is carried out through a mixed mechanism: ion exchange mechanism, i.e. the extraction of iodide ion, and the coordination one, i.e. the sorption of elemental iodine, which leads to a significant increase in the capacitive properties of sorbents compared to known industrial ion exchangers.

Conclusions

The results obtained made it possible to draw the following conclusions:

– the sorption of iodine on the synthesized samples is essentially independent of the pH of the medium, and the degree of its extraction in the entire area under study is 94–100%.

– sorption kinetic curves of the synthesized ion exchange resin based on 1,4 dioxybenzene are the highest in comparison with 1,2 dioxybenzene and 1,3 dioxybenzene ion exchangers. The maximum extraction of iodine by sorption with an ion exchanger based on 1,4 dioxybenzene is achieved in 9 hours.

– the sorption capacity of all synthesized ion exchangers for iodine is high and exceeds 1600 mg/g. The ion exchanger synthesized on the basis of 1,4 dioxybenzene has the best sorption characteristics (Static exchange capacity = 2283.88 mg/g).

– the sorption of iodine by ion exchangers is carried out through a mixed mechanism: ion exchange mechanism, i.e. the extraction of iodide ion, and the coordination one, i.e. the sorption of elemental iodine, which leads to a significant increase in the capacitive properties of sorbents compared to known industrial ion exchangers.

Conflict of interests. On behalf of all authors, the correspondent author declares that there is no conflict of interest.

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ТҮЙІНДЕМЕ
Иод адам және жануарлардың қалқанша безінің маңызды компоненті ретінде маңызды микроэлемент болып табылады. Қазақстанның көптеген аймақтарында йодтың табиғи тапшылығы байқалады. Әйтседе, республикада йод пен оның қосындыларын алудың бай ресурстық базасы бар – бұл құрамында 0,001–0,005% йод бар өндірілетін мұнай кен орындарының қабат сулары. Табиғи бұрғылау суларынан йод алу үшін біз мұнай өңдеу өнеркәсібінің қалдықтарынан алынған ион алмастырғыш материалдарды ұсындық. Мақалада синтезделген иониттердегі иод сорбциясының сипаттамаларын зерттеу нәтижелері келтірілген. Ион алмастырғыштардың синтезі екі атомды фенолдарды (1,3 диоксибензол, 1,2 диоксибензол, 1,4 диоксибензол), гексаметилендиамин және формальдегидті поликонденсациялау арқылы жүзеге асырылды. Синтезделген ион алмастырғыштармен иодтың келесі сорбциялық сипаттамалары зерттелді, олар: иодтың сорбциялануына ерітіндінің pH әсері; иодтың сорбциялануының кинетикалық қисықтары (сызықтары); иодтың сорбция изотермалары; иодтың сорбциялану механизмі. Синтезделген үлгілердегі йодтың сорбциясы іс жүзінде ортаның pH-на тәуелді емес екені, ал оның бүкіл зерттеу аймағындағы бөліп алу дәрежесі 94–100% құрайтыны анықталды. Йодтың бөліп алу дәрежесінің және таралу коэффициентінің логарифмінің процесс ұзақтығына тәуелділігі зерттелді. Синтезделген ион алмастырғыштардың йодқа қатысты сорбциялық қабілеттілігі ионалмастырғыштың құрылымына және сорбция жағдайларына байланысты бағаланды. Йодты бөліп алу үшін жоғары кинетикалық және статикалық алмасу қабілеттілігіімен (САҚ = 2283,88 мг/г) ерекшеленетін 1,4 диоксибензол негізіндегі сорбенттер қолайлы болатыны анықталды. Синтезделген ион алмастырғыштармен иодты сорбциялау механизмі анықталды.
Түйін сөздер: иод сорбциясы, сорбциялық сыйымдылық, ионалмастырғыштардың синтезі, сорбция изотермасы, иодидті шаймалау, иод.

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Изучение характеристики сорбции иода в синтезированных ионитах

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АННОТАЦИЯ
Иод является важным микроэлементом для людей и животных из-за его важной роли в качестве компонента гормонов щитовидной железы. Казахстан является страной, в большинстве регионов которого наблюдается природный дефицит иода. В то же время республика обладает богатой сырьевой базой для получения иода и его соединений – это пластовые воды разрабатываемых нефтяных месторождений, содержащие 0,001–0,005 % иода. Для извлечения иода из природных буровых вод нами были предложены ионообменные материалы, полученные из отходов нефтеперерабатывающей промышленности. В статье приводятся результаты изучение характеристики сорбции иода в синтезированных ионитах. Синтез ионитов осуществляли поликонденсацией двухатомных фенолов (1,3 диоксибензол, 1,2 диоксибензол, 1,4 диоксибензол), гексаметилендиамина и формальдегида. Были изучены следующие сорбционные характеристики иода синтезированными ионитами: влияние pH раствора на сорбцию иода; кинетические кривые сорбции иода; изотермы сорбции иода; механизм сорбции иода. Установлено, что сорбция иода на синтезированных образцах практически не зависит от pH среды, и степень извлечения его во всей исследуемой области составляет 94–100 %. Проведены исследования зависимости степени извлечения и логарифма коэффициента распределения иода от продолжительности процесса. Оценена сорбционная способность синтезированных ионитов по отношению к иоду в зависимости от структуры ионита и условий сорбции. Установлено, что при извлечении иода наиболее предпочтительны сорбенты на основе 1,4 диоксибензола, отличающиеся высоким кинетическими способностями и статической обменной емкостью (СОЕ = 2283,88 мг/г). Определен механизм сорбции иода синтезированными ионитами.

Ключевые слова: сорбция иода, сорбционная емкость, синтез ионитов, изотерма сорбции, иодидное выщелачивание, иод.

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