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ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
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АО «Институт топлива, катализа и
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ELECTROFLOTATION EXTRACTION OF POWDERED CARBON-BASED MATERIALS FROM AQUEOUS SOLUTION WITH USING OF SURFACTANTS

Abstract. Comparative electroflotation studies of powdered carbon materials from an aqueous solution of sodium sulfate electrolyte were carried out.

Kinetic electroflotation dependences of various carbon materials particles (carbon nanoflakes and activated carbons OU-B and BAU-A) are shown. It is established that the particles of carbon nanoflakes are most effectively extracted in the presence of nonionic surfactants. Whereas the extraction degree of activated carbons does not reach 20% in the same conditions.

Electroflotation studies of powdered carbon materials from aqueous solutions with the addition of coagulants Al^{3+} and Fe^{3+} were carried out to improve the efficiency and intensity of particles extraction. We studied aqueous solutions containing different types of surfactants: anionic, cationic and nonionic. Studies have shown that carbon nanoflakes are extracted quite effectively regardless of nature either the coagulants or the surfactants.

The extraction degree of activated carbon particles OU-B reached 90-95% if iron ions were present in an aqueous solution of cationic or nonionic surfactant. But the efficiency of the process was reduced in the presence of Al^{3+} due to the formation of a large amount of insoluble aluminum hydroxide.

We also studied the surface characteristics of powdered carbon materials (electrokinetic potential (ζ-potential) and hydrodynamic radius of the particles) for a better understanding of the electroflotation process.

Keywords: carbon materials, electroflotation, surfactants, coagulant, ζ-potential, hydrodynamic radius.

Introduction. Activated carbon is a highly porous carbon adsorbent, which is obtained from various carbon-containing organic materials [1]. Activated carbon is widely used for treatment, separating and extracting gaseous and liquid substances [2-6]. In the Russian Federation and Kazakhstan, as well as in world practice, the largest share in the consumption of activated carbon is a processing of drinking and technical water.

Activated carbons are unique adsorbents [7-9] due to their hydrophobic properties in comparison with such adsorption materials as silica gels, zeolites, ionites and etc.

Activated carbons with a particle size less than 0.1 mm are considered powdered materials. This type of coal is often used for the liquid phase; it is thoroughly mixed with the liquid to be purified. After adsorption of impurities, the coal is removed from the solution by filtration or precipitation [10, 11].

The particle size distribution, which is carefully controlled by modern grinding processes, greatly influences the rate of adsorption, filtration, and precipitation. In addition, the small particle size makes it difficult to extract the formed dispersed phase "carbon material - adsorbate".

One of the most popular methods of particle extraction is presently flotation [12-16]. Studies of the electroflotation extraction of powdered carbon materials, such as activated carbons [17], and carbon nanomaterials [18-20] have been published before.

This article presents the experimental results of electroflotation extraction of powdered carbon materials, namely, activated carbons BAU-A and OU-B, as well as carbon nanoflakes (CNF), from aqueous solutions of different composition.

Methods and researching objects. Carbon nanoflakes (bulk density 0.27 g/cm^3; ash content 0.5%; specific surface 243 m^2/g), activated carbon OU-B (GOST 4453-74) and BAU-A (GOST 6217-74) were selected as objects of research. Colloid-dispersed systems of carbon materials in aqueous solutions with surfactants (anionic surfactant — NaDDS; cationic surfactant — Katinol; nonionic surfactant – Triton X-100), metals salts $Al_2(SO_4)_3$, $Fe_2(SO_4)_3$ and electrolytes Na_2SO_4, NaCl were prepared for conducting experiments.

Laboratory studies of the efficiency of the electroflotation extraction were carried out in a non-flowing electroflotation unit with an insoluble anode at a constant pH. The column of electroflotation unit is made of glass with the cross-sectional area 10 cm^2 and the volume of the treated solution 500 ml. The height of the unit is 800 mm, the sampling valve is located at a height of 40 mm above the electrode unit.

The electrode unit includes an insoluble anode made of ORTA (titanium base with a film coating of cobalt and ruthenium oxides caused by thermal decomposition of a mixture of their salts) and a cathode made of stainless steel mesh with a cell size of 0.5 × 0.4 mm (wire thickness 0.3 mm). The cathode is located above the anode in order to allow free passage of the anodic oxygen bubbles into the column of electroflotation unit. A rubber packing separates electrodes. The device is powered by a DC power source B5-48. The range of bulk current densities is 0.1-0.5 A/l. Gas saturation occurs due to hydrogen and oxygen released at the cathode and anode.

In fine of the treatment, sampling is performed through the valve in order to determine the residual concentration of the pollutant in the treated solution. When studying the kinetics of the process, sampling is carried out every 5 min.

Electroflotation efficiency was evaluated by the degree of extraction (α, %), which was calculated as the ratio of the difference between the initial (C_0) and final (C_1) content of pollutants in the solution to their initial content: $\alpha = \frac{C_0 - C_1}{C0} \times 100\%$.

The analysis of the carbon material concentration was carried out using an adapted quantitative analysis method (with calibration graph) on a PROMEKOLAB PE-5300B spectrophotometer. The determination of the hydrodynamic radius of the particles and the electrokinetic potential was carried out by the method of dynamic light scattering with the use of equipment PHOTOCOR Compact-Z.

The experiments were conducted using analytical equipment of the MUCTR resource-sharing center.

Experimental results and discussion. Figure 1 shows the kinetic dependence of the degree of extraction of particles of carbon materials from an aqueous solution of the electrolyte Na_2SO_4 in the presence of a nonionic surfactant.

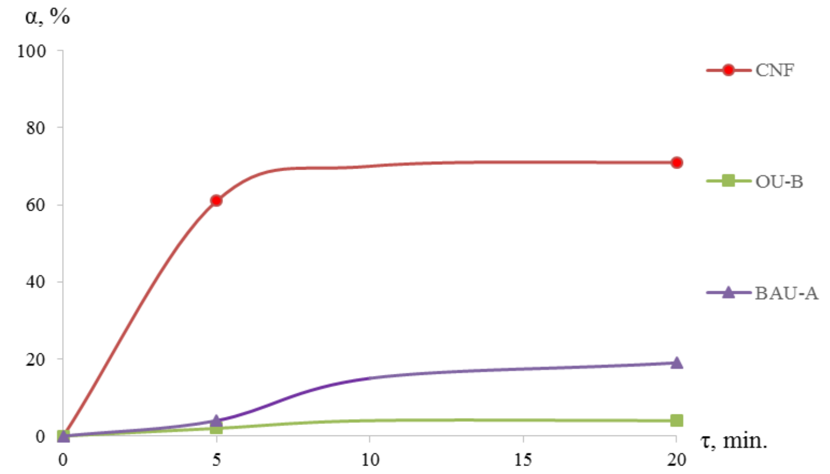

Figure 1 - Kinetic dependence of the degree of extraction of particles of carbon materials from an aqueous solution in the presence of a nonionic surfactant: C (CNF, OU-B) = 100 mg/L; C (BAU-A) = 1000 mg/L; C (TX-100) = 100 mg/L; C (Na_2SO_4) = 500 mg/L; i_v = 0.2 A/l; pH 7.0

The analysis showed that CNF particles are most efficiently extracted from an aqueous solution of a nonionic surfactant — after 10 minutes of electroflotation the recovery rate is 69%. While the degree of extraction of activated carbon reaches 5-20%.

Below are the studies of the electroflotation extraction of carbon materials from aqueous solutions of electrolytes of different composition.

Table 1 shows the results of the study of electroflotation of carbon materials (CN) from an aqueous solution of anionic surfactant in the presence of metal hydroxides Al^{3+} и Fe^{3+}.

Table 1 - Effect of the nature of carbon materials on the degree of extraction of the floatation complex "CN-$Me(OH)_3$" from an aqueous solution in the presence of metal hydroxides and anionic surfactant NaDDS

Me^{3+}	Recovery rate "CN-$Me(OH)_3$" α, %					
	CNF		OU-B		BAU-A	
	5 min	30 min	5 min	30 min	5 min	30 min
*without Me^{3+}	3	5	2	2	5	26
Al(III)	85	94	22	36	27	75
Fe(III)	83	95	15	20	5	15

i_v = 0.2 A/L; pH 7.0; C(CNF, OU-B) = 100 mg/L; C(BAU-A) = 1000 mg/L;
C(NaDDS) = 100 mg/L; C(Na_2SO_4) = 500 mg/L; *C(Na_2SO_4) = 500 mg/L

Analysis of the experimental data showed that after 5 minutes of electroflotation, the degree of extraction of ULF reaches 83-85% regardless of the nature of the added coagulant. After completion of the extraction process, the recovery rate reached 94-95%.

Unlike carbon nanomaterial, OU-B activated carbon was not efficiently removed (20-36%). Whereas the extraction process of BAU-A carbon in the presence of Al^{3+} coagulant did not show effective results in the first minutes, and upon completion of the purification process, the degree of particle recovery reached 75%.

The results of the study of the electroflotation of activated carbons and carbon nanoflakes from an aqueous solution of a cationic surfactant in the presence of metal hydroxides Al^{3+} и Fe^{3+} are presented in Table 2.

Table 2 – Effect of the nature of carbon materials on the degree of extraction of the “CN-$Me(OH)_3$” flotation complex from an aqueous solution in the presence of metal hydroxides and a cationic surfactant Katinol

Me^{3+}	Recovery rate "CN-$Me(OH)_3$" α, %					
	CNF		OU-B		BAU-A	
	5 min	30 min	5 min	30 min	5 min	30 min
*without Me^{3+}	68	73	9	52	3	36
Al(III)	90	95	14	81	3	51
Fe(III)	92	96	71	94	23	81

i_v = 0.2 A/L; pH 7.0; C(CNF, OU-B) = 100 mg/L; C (BAU-A) = 1000 mg/L;
C(Katinol) = 100 mg/L; C(Na_2SO_4) = 500 mg/L; *C(Na_2SO_4) = 500 mg/L

It was established that, regardless of the nature of the particles in the presence of the Fe^{3+} coagulant, the extraction degree reached 81-96%. However, the addition of Al^{3+} coagulant makes it possible to efficiently remove only CNF and OU-B carbon after 30 minutes of the electroflotation.

Similar results were shown by experiments on the extraction of carbon materials from an aqueous solution of nonionic surfactants in the presence of metal hydroxides Al^{3+} и Fe^{3+} (Table 3).

Differences in the extraction of particles of carbon materials are primarily associated with the methods of obtaining these materials, which affect their textural and surface characteristics. In addition, the size of particles and their charge is important in electroflotation.

Table 4 presents the results of the study of the electrokinetic potential and the hydrodynamic radius of the CNF particles.

Table 3 – Effect of the nature of carbon materials on the degree of extraction of the "CN-Me(OH)$_3$" flotation complex from an aqueous solution in the presence of metal hydroxides and nonionic surfactant TX-100

Me^{3+}	Recovery rate "CN-Me(OH)$_3$" α, %					
	CNF		OU-B		BAU-A	
	5 min	30 min	5 min	5 min	30 min	30 мин
*without Me^{3+}	69	71	3	4	4	25
Al(III)	80	93	21	86	3	3
Fe(III)	94	95	58	92	66	91

i_v = 0,2 A/l; pH 7.0; C(CNF, OU-B) = 100 mg/L; C(BAU-A) = 1000 mg/L;
C(TX-100) = 100 mg/L; C(Na_2SO_4) = 500 mg/L; *C(Na_2SO_4) = 500 mg/L

Table 4 – Effect of the nature of surfactants on the electrokinetic potential ζ and the hydrodynamic radius R of CNF particles in an aqueous solution in the presence of metal hydroxides

Type of surfactant	NaDDS			Katinol			TX-100		
Additives	Na_2SO_4	$Fe(OH)_3$	$Al(OH)_3$	Na_2SO_4	$Fe(OH)_3$	$Al(OH)_3$	Na_2SO_4	$Fe(OH)_3$	$Al(OH)_3$
ζ, mV	-24	-3	+7	+1	+7	+12	-18	+10	+12
R, μm	14	28	57	11	67	10	22	39	69

C(CNF) = 100 mg/L; C(surfactant) = 100 mg/L; C(element) = 500 mg/L; i_v = 0.2 A/l; pH 7.0; τ (electroflot.) = 30 min

In the presence of cationic surfactant Katinol, CNF particles recharge (ζ = +1-12 mV) and acquire more hydrophobic properties, which positively affects their electroflotation extraction. In addition, in the presence of iron (III) coagulant, the particles have a maximum size of 69 microns.

The same results and changes in the particle surface properties were observed in the presence of anionic surfactant and Al^{3+} coagulant, as well as in the presence of nonionic surfactant and Fe^{3+}, Al^{3+} coagulants. In the absence of coagulants, the surface of the particles acquired a high negative charge, which could complicate the electroflotation extraction of CNF.

The characteristics of OU-B activated carbon in the presence of various electrolytes were also studied (Table 5).

Table 5 – The effect of the pH of the electrolyte solution on the magnitude of the electrokinetic potential ζ and the hydrodynamic radius R of the particles of OU-B

	OU-B					
	Na_2SO_4			NaCl		
Specifications \ pH	3	7	11	3	7	11
ζ, mV	-5	-16	-37	-10	-18	-18
R, μm	17	6	8	10	12	11

C(OU-B) = mg/L; C(electrolyte) = 1 mg/L

Analysis of the experimental data made it possible to establish that the maximum particle size of OU-B (10-17 μm) can be achieved in an acidic medium of sulfate background, as well as at any pH of the chloride background. However, the high negative electrokinetic potential of the particles of activated carbon can affect and hinder their extraction.

Conclusions

Consequently, conducted experiments show that electroflotation extraction of carbon materials depends on their nature, method of preparation, as well as on texture and surface specifications (potential and particle size).

In addition, the presence of cationic and nonionic surfactants, Al^{3+}, Fe^{3+} coagulants in solutions contribute to the change of particle properties due to the formation of flotation complexes, which leads to effective electroflotation extraction of carbon materials.

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ҚҰРАМЫНДА БАЗ БАР БОЛҒАН СУЛЫ ЕРІТІНДІЛЕРДЕН ҰНТАҚТӘРІЗДІ КӨМІРТЕКТІ МАТЕРИАЛДАРДЫ ЭЛЕКТРОФЛОТАЦИЯЛЫҚ ЖОЛМЕН АЛУ

Аннотация. Натрий сульфатының сулы электролиттерінен электрофлотация жолымен алынған ұнтақтәрізді көміртекті материалдардың салыстырмалы зерттеулері жүргізілді.

Әртүрлі көміртекті материалдардан тұратын бөлшектерді бөліп алудың, яғни көміртекті наноқабыршақ пен ОУ-Б және БАУ-А белсенді көмірлердің кинетикалық тәуелділігі көрсетілді. Ионогенді емес БАЗ бар кезде көміртекті наноқабыршақ бөлшектері ең тиімді алынатындығы, ал белсенді көмірдің алыну дәрежесі 20 % жетпейтіндігі анықталды.

Электрофлотацияның интенсивтілігін және тиімділігін жоғарылату мақсатында Al^{3+} және Fe^{3+} коагулянттарын қосылған сулы ерітінділерден ұнтақтәрізді көміртекті материалдарды бөліп алу бойынша зерттеулер жүргізілді. Құрамында әртурлі БАЗ болған (анионды, катионды және ионогенді емес) сулы ерітінділер зерттелді. Зерттеулер БАЗ және коагулянттың табиғатына қарамастан көміртекті наноқабыршақтар анағұрлым тиімді бөлініп алынатындығын көрсетті.

Құрамында катионды немесе ионогенді емес БАЗ бар болған сулы ерітіндіде темір иондары болғанда ОУ-Б белсенді көмір бөлшектерінің алыну дәрежесі 90-95 %-ға жетті. Al^{3+} бар болған кезде қиын еритін алюминий гидроксиді түзілу себебті процесс тиімділігі төмендейді.

Электрофлотация процессін жақсы түсіну мақсатында ұнтақтәрізді көміртекті материалдардың беттік сипаттамасы, яғни электрокинетикалық потенциал (ζ-потенциал) және бөлшектердің гидродинамикалық радиусы зерттелді.

Түйін сөздер: көміртекті материалдар, электрофлотация, беттік-активті заттар, коагулянт, ζ-потенциал, гидродинамикалық радиус

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ЭЛЕКТРОФЛОТАЦИОННОЕ ИЗВЛЕЧЕНИЕ ПОРОШКООБРАЗНЫХ УГЛЕРОДНЫХ МАТЕРИАЛОВ ИЗ ВОДНЫХ РАСТВОРОВ В ПРИСУТСТВИИ ПАВ

Аннотация. Проведены сравнительные исследования электрофлотационного извлечения порошкообразных углеродных материалов из водного раствора электролита сульфата натрия.

Показаны кинетические зависимости извлечения частиц различных углеродных материалов: углеродных наночешуек и активированных углей ОУ-Б и БАУ-А. Установлено, что в присутствии неионогенного ПАВ

наиболее эффективно извлекаются частицы углеродных наночешуек, тогда как степень извлечения активированных углей не достигала 20%.

Для повышения эффективности и интенсивности электрофлотации были проведены исследования по извлечению порошкообразных углеродных материалов из водных растворов при добавлении коагулянтов Al^{3+} и Fe^{3+}. Были изучены водные растворы, содержащие различные типы ПАВ: анионное, катионное и неионогенное. Исследования показали, что углеродные наночешуйки извлекаются достаточно эффективно, независимо от природы коагулянта и ПАВ.

Степень извлечения частиц активированного угля ОУ-Б достигала 90-95%, если в водном растворе катионного или неионогенного ПАВ присутствовали ионы железа. Тогда как в присутствии Al^{3+} эффективность процесса снижалась за счет образования большого количества осадка труднорастворимого гидроксида алюминия.

Для лучшего понимания процесса электрофлотации были изучены поверхностные характеристики порошкообразных углеродных материалов: электрокинетический потенциал (ζ-потенциал) и гидродинамический радиус частиц.

Ключевые слова: углеродные материалы, электрофлотация, поверхностно-активные вещества, коагулянт, ζ-потенциал, гидродинамический радиус.

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