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# REGULATION OF THE COMPOSITION AND PROPERTIES OF HUMIC SUBSTANCES DURING ACID TREATMENT

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**Abstract**. *Introduction.* Humic substances (HS) are promising natural sorbents that contain carboxyl, phenolic, hydroxyl and other functional groups, which ensures their high reactivity and tendency to complex with heavy and transition metals, influencing their migration, bioavailability and toxicity.*The aim of the work* is to investigate the influence of various factors on the production of humic acids from the domestic natural raw materials. *Methods*: standardized methods, elemental and functional analysis, IR spectroscopy, and thermogravimetry. *Results and discussion*. Research has been conducted on the production of humic acids (HA) by reacting sodium humate with hydrochloric acid. It has been shown that the regulation of the HCl concentration, liquid phase ratio, reaction time and temperature allows one to control the yield of $HA^{daf}$ (up to 78.31%), the content of COOH and $OH_{phen.}$ groups (up to 1.20 and 1.62 mmol/g), the total pore volume (up to 0.58 $cm^3/g$) and static exchange capacity (up to 21.10 mg-eq/g). The determined optimal synthesis parameters (sodium humate concentration of 3.0%, liquid phase ratio 1:1, treatment for 15 min at 20°C) ensure reproducible properties of the HA and their high efficiency in the sorption of metal ions. The elemental analysis and the calculation of the H/C and O/C atomic ratios have shown that the structure of the synthesized the HA is heterogeneous, including aromatic and aliphatic fragments. It has been found that an increase in the HCl concentration initiates an oxidative-hydrolytic destruction of the peripheral structures with a simultaneous increase in the oxygen-containing functional groups, which determine the sorption activity of the HAs. The data of the thermal analysis and IR spectroscopy confirm the preservation of the functional groups up to 350°C and are consistent with the degradation processes at higher temperatures. *Conclusion*. A process flow diagram for producing humic acids under the "mild" conditions has been developed. It is characterized by flexibility, low waste generation, and the ability to produce humic sorbents with a specified composition. The synthesized HA have sorption, ion exchange and other properties and are promising for practical application.

**Key words:** humic acids, hydrochloric acid, oxygen-containing functional groups, heavy metal ions, sorbent, process flow diagram

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## 1. Introduction

Contamination of the liquid media with heavy metal ions (HMI) poses a serious threat to human health and the biosphere due to their high toxicity, stability, and tendency to bioaccumulate [1-3]. Various methods have been developed for the removal of the HMI from the aquatic systems [2-5], among which sorption is considered one of the most effective and versatile approaches [6-8]. Among synthetic and natural sorbents, HS, natural organic compounds with the oxygen-containing functional groups that provide high complexation and sorption capacity, are of particular interest for the HMI removal. The role of the HS as selective sorbents for $Pb^{2+}$ in the multi-element systems with pH regulation and the introduction of activating groups into the sorbent structure has been demonstrated in [9]. It has been established that a combination of alkaline extraction of brown coal with membrane ultrafiltration makes it possible to obtain the HS with a low impurity content [10]. Biosolubilization of a lignite, using a cell-free fungal enzyme filtrate has demonstrated significantly higher humic acid yields [11]. The authors [12] have found that the alkaline extraction of a lignite, combined with the fermentation and subsequent organic decomposition, have improved the functional properties of humic acids.

Despite these results, the impact of the humic acid production methods on their properties, reproducibility, and industrial applicability remains poorly understood, which justifies the objective of this study - to investigate the influence of various factors on the process of obtaining humic acids from the domestic natural raw materials.

## 2. Experimental part

Humic acids were synthesized using sodium humate, extracted from brown coal from the Oikaragai deposit (the Almaty region). The yield of free humic acids ($HA^{daf}$) was 47.0%, with the ash content ($A^a$) of 24.80% and the moisture content ($W^a$) of 12.80%.

Humic acids were synthesized, using 1.0-5.0% HCl at 20°C for 5-35 min with a liquid phase ratio (sodium humate:hydrochloric acid) of 1:0.4÷2 and a drying temperature of 20-150°C. The elemental composition of the HA (C, H, N, O) was analyzed on a FlashSmart elemental analyzer (Thermo Fisher Scientific Inc., USA), the yield of $HA^{daf}$, the content of COOH and $OH_{phen.}$-groups, the static exchange capacity (SEC), and the total pore volume were determined according to standard methods [13-16]. The IR spectra were recorded on a Nicolet 5700 Fourier-transform IR spectrometer (Thermo Electron, USA) in the range of 4000-400 $cm^{-1}$ in the KBr tablets; the absorption bands were interpreted according to the literature [14, 17]. A thermogravimetric analysis was performed on an SKZ1053 device (SKZ Industrial Co., Limited, China) in the air atmosphere up to 600°C at a heating rate of 1-80°C/min [18, 19].

## 3. Results and discussion

It follows from the results shown in Figure 1 that an increase in the HCl concentration is accompanied by a change in $HA^{daf}$, the content of the COOH- and $OH_{phen}$-groups, the total pore volume and the SEC of the obtained HA samples, reaching 79.10%, 1.23 and 1.63 mmol/g, 0.59 $cm^3/g$ and 21.23 mg-eq/g, respectively, at L:L=1:1, the reaction time of 35 min and the temperature of 20°C. An acid treatment of sodium humate probably initiates the oxidative-hydrolytic destruction of the organic macromolecule, accompanied by a structural rearrangement of the HA and the formation of the additional functionally active centers. An increase in the content of the COOH- and $OH_{phen}$-groups, characteristic of the HS [9, 10, 14], causes an increase in the number of active sites and, as a consequence, an increase in the sorption properties of the synthesized HA.

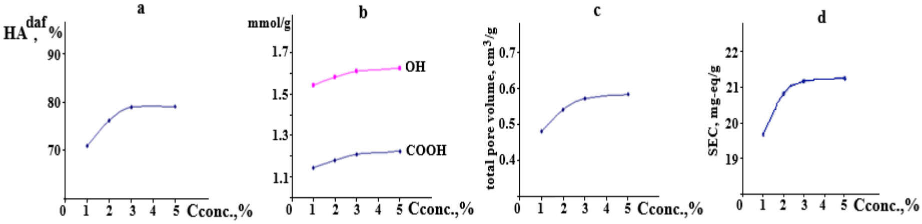


**Figure 1** – An isotherm of the dependence of the $HA^{daf}$ (a), acidic groups (b), total pore volume (c) and SEC (d) on the concentration of hydrochloric acid.

An analysis of the data presented in Figures 2 and 3 indicates that an increase in the L:L ratio from 1:0.4 up to 1:2, as well as an increase in time from 5 to 35 min, leads to an increase in $HA^{daf}$ from 70.50 up to 78.75%, the content of the COOH groups from 1.09 up to 1.21 mmol/g and the $OH_{phen}$ groups from 1.44 up to 1.61 mmol/g, the total pore volume from 0.46 up to 0.57 $cm^3/g$ and SEC from 19.50 up to 21.14 mg-eq/g, which is due to the dilution of the reaction suspension and intensification of the mass transfer, contributing to a more complete transition of the HA into the solution, acceleration of the decomposition of organic fragments of the humic sorbent and deepening of the oxidation of aliphatic structures, resulting in the formation of a more developed and porous structure of the material.

The obtained experimental data (Figure 4) indicate the presence of a pronounced temperature pattern in the formation of the structural and functional characteristics of the HA: an increase in the drying temperature from 20 up to 80°C promotes the removal of the physically bound moisture and stabilization of the porous structure, which leads to an increase in the yield of $HA^{daf}$ up to 78.27%, the content of the COOH and $OH_{phen}$-groups – up to 1.19 and 1.59 mmol/g, respectively, the total pore volume – up to 0.55 $cm^3/g$ and SEC – up to 21.0 mg-eq/g. At the same time, a further increase in the drying temperature up to 150°C changes the direction of the process and causes a decrease in the above indicators, due to the intensification of the decarboxylation processes, partial oxidation and destruction of the aliphatic fragments of the HA macromolecules, leading to a decrease in the number of the oxygen-containing functional groups and degradation of the porous structure of the material. An elemental analysis of the HA characterizes their composition, transformation, aromaticity, and functional activity [14, 20]. The elemental analysis (Table 1) and calculated H/C and O/C atomic ratios demonstrate structural changes in the synthesized HA, affecting the ratio of the central and peripheral fragments of the molecules. A decrease in the content of C, H, N, and O with an increase in the concentration of HCl indicates the oxidative-hydrolytic destruction of the peripheral organic part. The H/C (0.92-1.08) and O/C (0.39-0.46) ranges reflect the presence of the aromatic and aliphatic fragments and enrichment of the HA with the oxygen-containing functional groups.

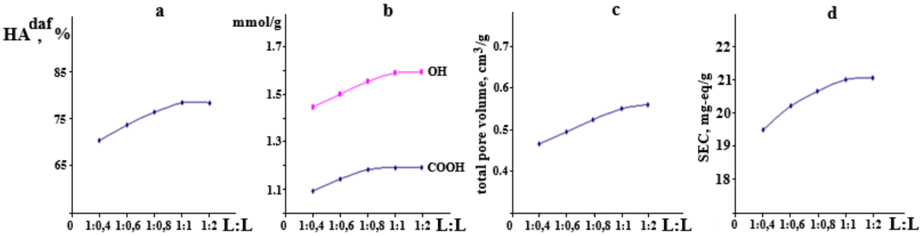


**Figure 2** – Changes in the HA$^{daf}$ (a), acidic groups (b), total pore volume (c) and SEC (d) depending on the L:L ratios.

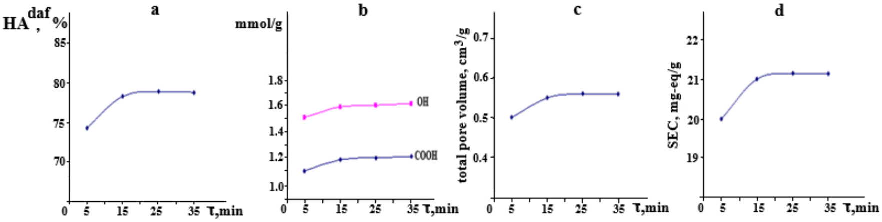


**Figure 3** – Effect of time on the HA$^{daf}$ (a), acidic groups (b), total pore volume (c) and SEC (d).

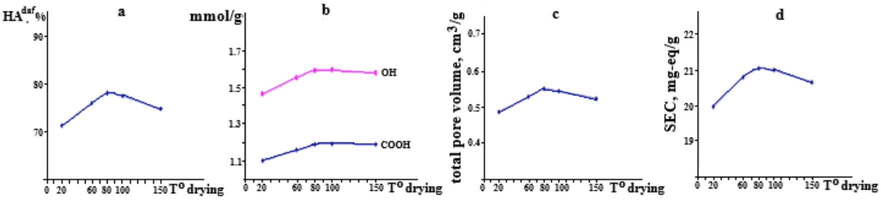
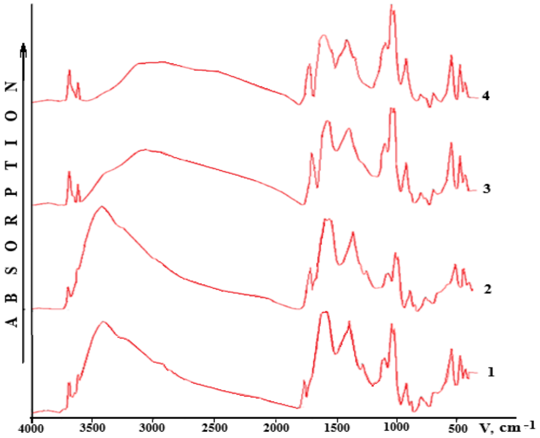


**Figure 4**– Dependence of the HA$^{daf}$ (a), acidic groups (b), total pore volume (c) and SEC (d) on the drying temperature.

An analysis of the IR spectra of the synthesized HA (Figure 5) has revealed the characteristic absorption bands of ν-vibrations of the unbound OH groups at 3695–3690 and 3620–3615 cm$^{-1}$, as well as the hydrogen-bonded OH groups in the region of 3415–3410 and 3235–3230 cm$^{-1}$ [14, 17]. The shoulder in the range of 3065–3000 cm$^{-1}$ is due to the vibrations of the aromatic =C–H bonds. The bands at 2965–2960, 2930–2925, and 2855–2850 cm$^{-1}$ correspond to the $\nu_s$-vibrations of the aliphatic -CH$_2$ and -CH$_3$ groups, and at 1710–1700 cm$^{-1}$ to the ν-vibrations of the carbonyl groups of carboxylic acids and their derivatives, while the absorption in the region of 1580–1555 cm$^{-1}$ indicates the presence of secondary amides. The bands at 1390–1385 cm$^{-1}$ are associated with ν- and δ-vibrations of the C–O and O–H groups of carboxylic acids, esters, and phenols, and the range of 1095–1010 cm$^{-1}$ is associated with the ν-vibrations of the C–O bonds in alcohols and esters. The bands in the region of 915-425 cm$^{-1}$ are due to the out-of-plane δ-vibrations of the aromatic rings. An increasing in the HCl concentration is accompanied by an increase in the intensity of the carboxyl group bands in the region of 1710-1700 cm$^{-1}$.

**Table 1** – An elemental analysis of the humic acid samples

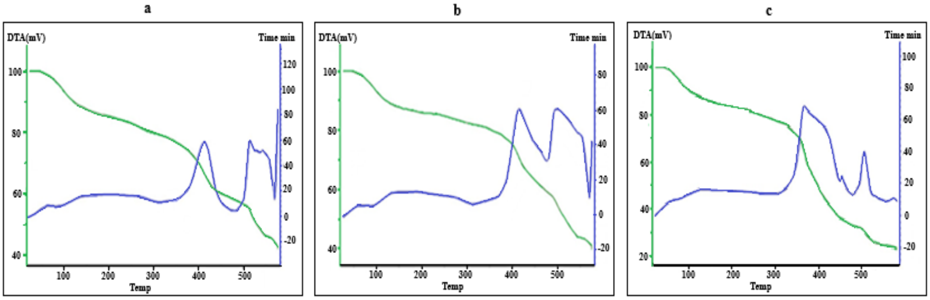
| The concentration of hydrochloric acid, % | By weight % | | | | Atomic % | | | | Atomic relations | |
|---|---|---|---|---|---|---|---|---|---|---|
| | C | H | N | O | C | H | N | O | H/C | O/C |
| 1 | 60.15 | 4.63 | 2.21 | 31.53 | 42.60 | 39.37 | 1.27 | 16.75 | 0.92 | 0.39 |
| **2** | 59.91 | 4.35 | 2.13 | 30.11 | 35.75 | 43.81 | 1.51 | 18.93 | 1.22 | 0.53 |
| **3** | 58.53 | 4.16 | 2.09 | 27.82 | 44.59 | 38.09 | 1.37 | 15.93 | 0.85 | 0.36 |
| **5** | 58.10 | 4.01 | 1.96 | 27.71 | 38.68 | 41.81 | 1.46 | 18.04 | 1.08 | 0.46 |



humic acids synthesized at different concentrations of hydrochloric acid, %: 1 – 1.0; 2 – 2.0; 3 – 3.0; 4 – 5.0

**Figure 5** – The IR spectra of the HA samples.

The thermal analysis data (Figure 6) are in good agreement with the IR spectroscopy results: the thermal stability of humic acids up to 350°C correlates with the presence of the intense bands of the carboxyl, hydroxyl, and aromatic groups in the IR spectra. The onset of degradation at 350-360°C corresponds to the degradation of the aliphatic fragments and carboxyl groups, indicated by the bands at 1710-1700 and 2960-2850 cm$^{-1}$, while the exothermic effect at 510-550°C is consistent with the destruction of the aromatic ring, confirmed by the bands in the range of 1600-1500 and 915-425 cm$^{-1}$.



humic sorbents obtained at different concentrations of hydrochloric acid, %: a – 1,0; b – 2,0; c - 3,0

**Figure 6** – The thermograms of the synthesized samples of humic sorbents.

Based on the experimental data, a process flow diagram for producing humic acids (Figure 7), implemented under the “mild” conditions, has been proposed. The process does not require high temperatures or pressures or the use of expensive or hazardous reagents, and the low reaction temperature (20°C) eliminates the need for additional energy. The technology is highly flexible due to the ability to vary parameters and use various mineral acids, enabling the production of humic sorbents with a specified composition and controlled physicochemical properties. An additional advantage is the low level of waste generation due to the ability to reuse the liquid phase in the process cycle.

Optimization of the humic acid synthesis parameters has been conducted under the laboratory conditions, resulting in the establishment of the optimal process conditions: the sodium humate concentration of 3.0%, phase ratio of 1:1, duration of 15 min, temperature of 20°C, and drying temperature of 80°C. A chemical analysis of the obtained test samples has been used to determine the contents of HA$^{daf}$, COOH and OH$_{phen}$ group, the total pore volume, and COE (Table 2). The results confirm the high efficiency of this technology, which ensures the production of humic sorbents with the reproducible physicochemical characteristics, suitable for the sorption of metal ions.

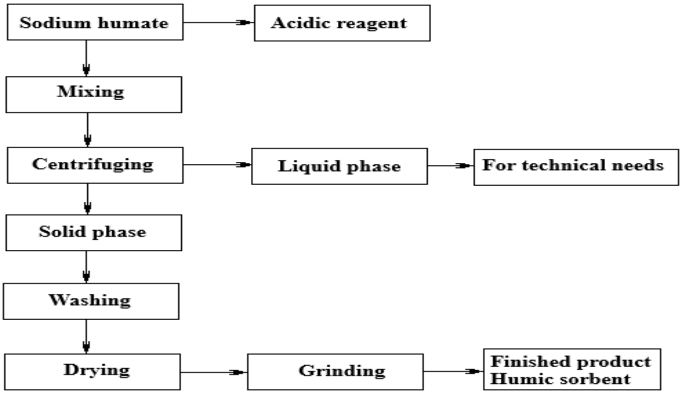


**Figure 7** – The basic technological scheme for obtaining humic acids.

**Table 2** – The characteristics of the prepared humic acid samples.

| $HA^{daf}$, mass.% | The content of the acidic groups, mmol/g | | The total pore volume, $cm^3/g$ | COE, mg-equ./g |
|---|---|---|---|---|
| | COOH | $OH_{phen.}$ | | |
| 78.31 | 1.20 | 1.62 | 0.58 | 21.10 |

## 4. Conclusion

The experimental results have demonstrated the feasibility of producing humic acids by reacting sodium humate with a low-concentration HCl solution. The composition and properties of the synthesized humic acids have been determined to depend on hydrochloric acid concentrations, drying time, temperature, and the ratios of the starting components. The elemental and functional analysis, IR spectroscopy, and differential thermal analysis (DTA) data indicate that the humic acids are characterized by the thermal stability, a high yield of the active ingredient, and a high content of the carboxyl groups and phenolic hydroxyls. They can participate in ion exchange and complexation reactions. The proposed humic acid production technology utilizes various types of acid reagents, variable process parameters, and the composition and properties of the humic acids, making it more flexible and enhancing their sorption capacity. The resulting humic acids can be used as effective natural sorbents for purifying the liquid and solid media from various contaminants.

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**Conflict of Interest:** The authors declare no conflicts of interest requiring the disclosure in this paper.

# ҚЫШҚЫЛМЕН ӨҢДЕУ КЕЗІНДЕГІ ГУМИНДІ ЗАТТАРДЫҢ ҚҰРАМЫ МЕН ҚАСИЕТТЕРІН РЕТТЕУ

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**Түйіндеме.** *Кіріспе.* Гуминді заттар (ГЗ) – карбоксил, фенолды, гидроксил және басқа да функционалдық топтарды қамтитын перспективалы табиғи сорбенттер, бұл олардың жоғары реакциялық және ауыр және өтпелі металдармен кешен түзуге бейімділігін қамтамасыз етеді, олардың миграциясына, биожетімділігіне және уыттылығына әсер етеді. *Жұмыстың мақсаты –* отандық табиғи шикізаттан гумин қышқылдарын алу үдерісіне әртүрлі факторлардың әсерін зерттеу. *Әдістер*: стандартталған әдістер, элементтік және функционалдық талдау, ИҚ-спектроскопия және термогравиметрия. *Нәтижелер және талқылау.* Натрий гуматын тұз қышқылымен әрекеттесу арқылы гумин қышқылдарын (ГҚ) алу бойынша зерттеулер жүргізілді. HCl концентрациясын, сұйық фаза қатынасын, реакция уақытын және температурасын реттеу $\text{HA}^{daf}$ шығымын (78.31%-ға дейін), COOH және $\text{OH}_{\text{фен.}}$ топтарының мөлшерін (1.20 және 1.62 ммоль/г дейін), жалпы кеуек көлемін (0.58 $\text{см}^3$/г дейін) және статикалық алмасу сыйымдылығын (21.10 мг-экв/г дейін) бақылауға мүмкіндік беретіні көрсетілді. Анықталған оңтайлы синтез параметрлері (натрий гуматының концентрациясы 3.0%, сұйық фаза қатынасы 1:1, 20°С, 15 мин) ГҚ-ның қайталанатын қасиеттерін және металл иондарын сорбциялаудағы жоғары тиімділігін қамтамасыз етеді. Элементтік талдау және Н/С және О/С атомдық қатынастарын есептеу синтезделген ГҚ құрылымының гетерогенді екенін көрсетті, оның ішінде ароматты және алифатты фрагменттер бар. HCl концентрациясының жоғарылауы перифериялық құрылымдардың тотығу-гидролитикалық ыдырауына, ГҚ сорбциялық белсенділігін анықтайтын оттегі бар функционалдық топтардың артуына әкелетіндігі анықталды. Термиялық талдау және ИҚ-спектроскопия деректері функционалдық топтардың 350°С дейін сақталуын растайды және жоғары температурадағы ыдырау үдерістеріне сәйкес келеді. *Қорытынды.* «Жұмсақ» жағдайларда ГҚ алудың технологиялық сызбасы әзірленді, бұл икемділігімен, қалдықтардың аз түзілуімен және құрамы белгіленген гумин сорбенттерін алу мүмкіндігімен сипатталады. Синтезделген ГҚ сорбциялық, ион алмасу және басқа да қасиеттерге ие және практикада қолдану үшін перспективалы.

**Түйінді сөздер:** гумин қышқылдары, тұз қышқылы, оттегі бар функционалдық топтар, ауыр металл иондары, сорбент, технологиялық сызба

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# РЕГУЛИРОВАНИЕ СОСТАВА И СВОЙСТВ ГУМИНОВЫХ ВЕЩЕСТВ ПРИ КИСЛОТНОЙ ОБРАБОТКЕ

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**Резюме.** *Введение.* Гуминовые вещества (ГВ) – перспективные природные сорбенты, обладающие карбоксильными, фенольными, гидроксильными и другими функциональными группами, что обеспечивает их высокую реакционную способность и склонность к комплексообразованию с тяжелыми и переходными металлами, влияя на их миграцию, биодоступность и токсичность. *Цель работы* – исследование закономерностей влияния различных факторов на процесс получения гуминовых кислот из отечественного природного сырья. *Методы:* стандартизованные методы, элементный и функциональный анализ, ИК-спектроскопия и термогравиметрия. *Результаты и обсуждение.* Проведены исследования по получению гуминовых кислот (ГК) путем взаимодействия гумата натрия с соляной кислотой. Показано, что регулирование концентрации HCl, соотношения жидкой фазы, времени реакции и температуры позволяет управлять выходом $HA^{daf}$ (до 78.31%), содержанием COOH- и $OH_{фен.}$-групп (до 1.20 и 1.62 ммоль/г), суммарным объемом пор (до 0.58 $см^3$/г) и статической обменной емкостью (до 21.10 мг-экв/г). Определенные оптимальные параметры синтеза (концентрация гумата натрия 3.0%, соотношение жидких фаз 1:1, обработка 15 мин при 20°С) обеспечивают воспроизводимые свойства ГК и их высокую эффективность в сорбции ионов металлов. Элементный анализ и расчет атомных соотношений H/C и O/C показали, что структура синтезированных ГК является гетерогенной, включающей ароматические и алифатические фрагменты. Установлено, что повышение концентрации HCl инициирует окислительно-гидролитическое разрушение периферийных структур с одновременным ростом кислородсодержащих функциональных групп, определяющих сорбционную активность ГК. Данные термического анализа и ИК-спектроскопии подтверждают сохранность функциональных групп до 350 °С и согласуются с деградационными процессами при более высоких температурах. *Заключение.* Разработана технологическая схема получения ГК в «мягких» условиях, характеризующаяся гибкостью, низким уровнем отходообразования и возможностью получения гуминовых сорбентов с заданным составом. Синтезированные ГК обладают сорбционными, ионообменными и др. свойствами и перспективны для практического применения.

**Ключевые слова:** гуминовые кислоты, соляная кислота, кислородсодержащие функциональные группы, ионы тяжелых металлов, сорбент, технологическая схема

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