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ISOLATION OF FULVIC ACID FROM COAL AND STUDY OF PHYSICAL AND CHEMICAL PROPERTIES

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Due to its high solubility and low molecular weight, fulvic acid is a very active participant in natural chemical processes. Due to its smaller molecule size than humic acid, it can act both inside and outside the cell. The work presents the method of isolation and purification of fulvic acid obtained from oxidized brown coal and the analysis of its physical and chemical properties by the methods of IR, NMR spectroscopy, and the amount of carboxyl and hydroxyl groups studied. Conducted work on calculation of equilibrium constant (pK) and concentration (C). Stages of purification: adsorption, ion purification and dialysis, where adsorbents obtained from brown (”Shoptykol”) and hard (”Shubarkol”) coal and coconut activated carbon were used as adsorbents.

Key words: oxidized carbon, fulvic acid, adsorbent, cation exchanger, dialysis, membrane cleaner.

КӨМІРДЕН ФУЛЬВОҚЫШҚЫЛЫН БӨЛІП АЛУ ЖӘНЕ ФИЗИКАЛЫҚ-ХИМИЯЛЫҚ ҚАСИЕТТЕРІН ЗЕРТТЕУ

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Жоғары ерігіштігі мен төмен молекулалық массасының әсерінен фульвоқышқылы табиғи химиялық процестердің өте активті қатысушысы болып табылады. Гумин қышқылына қарағанда молекулалық өлшемі кішкене болғандықтан жасушаның ішкі бөлігінен де, сыртқы бөлігінен да әсер ете алады. Жұмыста тотыққан қоңыр көмірден алынған фульвоқышқылын бөліп алу және тазарту, олардың физика-химиялық қасиеттерін ИҚ, ЯМР спектроскопиясы арқылы талдау және карбоксил және гидроксил топтарының санын зерттеу әдісі ұсынылған. «Майкөбен» бассейінінен алынған қоңыр көмірірнен алғаш рет фульвоқышқылы экстрацияланып бөлініп алынып, Форсит әдісімен тазартылып, тазалық дәрежесі жоғары фульвоқышқылы бөлініп алынды. Тепе-теңдік константаларын (pK) және концентрацияны (C) есептеу жұмыстары жүргізілді. Тазарту кезеңдері: адсорбция, ионды тазарту және диализ, мұнда адсорбент ретінде қоңыр («Шоптыкөл») және тас көмірден («Шұбаркөл») алынған адсорбенттер, сонымен қатар, кокосты белсендірілген көмірі пайдаланылды.

Түйін сөздер: тотыққан көмір, фульвоқышқылы, адсорбент, катонит, диализ, мембраналы тазалау.

ВЫДЕЛЕНИЕ ФУЛЬВОВОЙ КИСЛОТЫ ИЗ УГЛЯ И ИЗУЧЕНИЕ ФИЗИКО-ХИМИЧЕСКИХ СВОЙСТВ

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Благодаря высокой растворимости и низкой молекулярной массе фульвокислота является очень активным компонентом природных химических процессов. Благодаря меньшему размеру молекулы, чем гуминовая кислота, она может воздействовать как внутри, так и снаружи клетки. В работе представлена способ выделения и очистки фульвокислот полученного из окисленного бурого угля и анализ их физико-химических свойств методами ИК, ЯМР спектроскопии, изучены количество карбоксильных, гидроксильных груп. Впервые из бурого угля, полученного из бассейна «Майкубен», очищенного методом Форсита, выделена фульвокислота высокой степени чистоты. Проведены работы по расчету констант равновесии (pK) и концентрации (C). Стадии очистки: адсорбция, ионная очистка и диализ, где в качестве адсорбентов были использованы адсорбенты полученные из бурого («Шоптыколь») и каменного («Шубарколь») угля и кокосовый активированный уголь.

Ключевые слова: окисленный уголь, фульвокислота, адсорбент, катонит, диализ, мембранная очистка.

Introduction. The current state of the global coal industry and Kazakhstan shows that the competitiveness of coal has decreased compared to other energy sources [1-2]. Experience shows that it is possible to increase the profitability of coal enterprises and reduce environmental and economic damage provided that the full potential of coal is used, including the production of valuable products from it, as well as coal waste during production. Water-soluble polyelectrolyte acids are important because they are active biological and chemical substances [3-5].

Fulvic acid belongs to the group of humic acids, soluble in water, alkalis and acids. The salts are called fulvates. The Swedish chemist J. Berzelius separated, later in the 20th century S. Oden combined the two acids and called them by the common name fulvic acid [1].

Although it was discovered in the 20th century, an effective extraction method only became known in 2015. In particular, the isolation and purification of fulvic acid is a very expensive process, so the cost of 1 mg of fulvic acid averages US$59. Fulvic acid is a fraction of humic substances that is soluble in water in all pH ranges. Due to its high solubility and low molecular weight, fulvic acid is an active participant in many natural chemical processes. Due to its smaller size than humic acid, it can affect the cell both from the inside and outside. It contains 74 organic minerals, 18 amino acids and 10 vitamins. All minerals that make up fulvic acid are small ions that are easily absorbed by cells.

Humic substances contain 40-60% carbon, 30-50% oxygen, 3-7% hydrogen, 1-5% nitrogen. The elemental composition of humic substances varies due to the diversity of their nature. Humic acid contains 52-62% carbon, 3.0-5.5% hydrogen, 30-33% oxygen, 3.5-5.0% nitrogen. Fulvic acid contains 44-49% carbon, 3.5-5.0% hydrogen, 44-49% oxygen, 2.0-4.0% nitrogen. Due to different objects, the nitrogen content of humic acid varies. 20-50% of total nitrogen belongs to amino acids, 1-10% to amino sucroses. Variations in the elemental composition of humic acids (humic acids and fulvic acids) are explained by the fact that they are not chemically defined organic acids, but represent a group of high-molecular chemical compounds similar in composition and properties [6-7].

Fulvic acid has a wide range of uses in the medical field. Fulvic acid has antibacterial properties and affects replication, and also has the ability to destroy viral cells. This is an effective and safe drug in the treatment of various bacteriological and viral diseases. In addition, it has been established that fulvic acid exhibits immunomodulatory ability.

Fulvic acid and humic acid cannot be synthesized; they can only be obtained by isolating them from natural raw materials. However, the purity of the isolated fulvic acid is very low. Isolation and purification of high purity fulvic acids is a very expensive process [8-10]. The biological activity of fulvic acid is largely characterized by its physical and chemical properties. Fulvic acid with a purity of ≥95% is registered in the CAS catalog (CAS Registry Number) (No. 479-66-3) as a chemical compound with the molecular formula $C_{14}H_{12}O_8$ [11-13].

The purpose of the work is to isolate and purify fulvic acid from coal “Maykuben” (Kazakhstan) to a high degree of purity, to analyze the physicochemical composition of the resulting fulvic acid.

Materials and methods: 0.1 N HCl, 0.1 N NaOH, 5% HCl, acetone, KU-2-8 cation exchanger, potassium humate, distilled water, laboratory centrifuge TsN-12, laboratory stirrer IKA RH ”basic 2” , rotary evaporator RV 3 IKA, ion chromatograph ”Dionex” ICS 6000, FT-IR spectrometer Nicolet iS10, JEOL ECA-500 MHz NMR spectrometer.

Humic acid is formed in the form of an amorphous brown precipitate as a result of the neutralization reaction of potassium humate obtained from Maykuben oxidized brown coal with a 5% solution of nitric acid to pH=2-3. An unprecipitated organic compound is called a fulvic acid. The Forsyth method was used for purification. Purification of fulvic acid by the Forsyth method consists of several stages. As adsorbents for fulvic acid cleaning, sorbents «Shubarkol» and «Shoptykol» produced by LLP «Institute of Coal Chemistry and Technology» and sorbents «Coconut» presented on the market were used. The last stage of purification was carried out at the stage of membrane purification (dialysis) to pH = 4-5 using distilled water for complete purification of fulvic acid from ions during purification.

Discussion of the results. The functional groups of the fulvic acid passed through each step were determined by the Forsyth method using an acid-base titration. Table 1 shows the functional groups of the samples

Table 1 - Functional groups of samples

№	Name	Total acidity, mmol/g	Carboxyl group, mmol/g	Phenolic group, mmol/g
1	Initial FA before purification	0,411	0,027	0,384
2	FA after cleaning with sorbent «Coconut»	0,269	0,105	0,164
3	FA after cleaning with «Coconut» sorbent and cation exchanger KU-2-8	0,322	0,139	0,183
4	FA after «Coconut» sorbent, cation exchanger KU-2-8 and membrane treatment	0,366	0,152	0,187
5	FA after cleaning with the sorbent «Shoptykol»	0,378	0,096	0,282
6	FA after cleaning «Shoptykol» sorbent and cation exchanger KU-2-8	0,287	0,127	0,16
7	FA after «Shoptykol» sorbent, cation exchanger KU-2-8 and membrane treatment	0,292	0,146	0,159
8	FA after cleaning with the sorbent «Shubarkol»	0,298	0,078	0,22
9	FA after cleaning «Shubarkol» sorbent and cation exchanger KU-2-8	0,263	0,109	0,154
10	FA after «Shubarkol» sorbent, cation exchanger KU-2-8 and membrane treatment	0,26	0,128	0,152

Table 2 - Changing the pH at each stage of fulvic acid cleaning

Name	pH after sorbent	pH after cation exchanger KU-2-8	pH after membrane
FA after «Shubarkol» sorbent, cation exchanger KU-2-8 and membrane treatment	0,49	0,09	2,28
FA after «Shoptykol» sorbent, cation exchanger KU-2-8 and membrane treatment	0,95	0,04	2,24
FA after «Coconut» sorbent, cation exchanger KU-2-8 and membrane treatment	0,25	0,02	2,53

Determination of carboxyl groups is based on acid-base titration using 0.05 N sodium hydroxide as titrant using the acetate method. Total acidity is determined by acid-base titration using 0.1 N HCl as titrant using barium hydroxide. Phenolphthalein was used as an indicator. Phenolic groups were determined by the difference between the carboxyl group and total acidity.

As a result, it can be seen that the carboxyl groups of the fulvic acid isolated from the original coal and the fulvic acid that passed the purification stage increased, while the phenolic groups and the total acidity decreased. Fulvic acid contains many carboxyl functional groups. It can exchange or transfer electrons with the organic acid redox polymer, ions and molecules that react with fulvic acid [3]. Table 2 shows the pH changes at each stage of fulvic acid purification.

On a Dionex ICS 6000 ion chromatograph, the ionic composition of fulvic acid, which was completely purified by the Forsyth method, was studied. As a result, it was found that the amount of all ions, in comparison with the amount of the initial content of fulvic acid, significantly decreased after the last stage of purification the membrane (dialysis) method. Initially, fulvic acid contained a large amount of nitrate and chloride ions. It can be assumed that this is due to the use of 5% nitric acid in the extraction of fulvic acid from coal, and the presence of a large amount of chloride ions is due to the effect of cleaning the sorbent with 0.1 N hydrochloric acid solution before passing the fulvic acid through the sorbent. Shubarkol was chosen as an effective sorbent in terms of ion purification in the purification of fulvic acids, the degree of ion purification was 66.12-99.75%.

IR analyzes of fulvic acids were carried out in the laboratories of the «Nazarbayev University» (Kazakhstan) using a Nicolet iS10 FT-IR spectrometer. There are no stretching vibrations of C=O groups of saturated fatty acids, carboxyl, aldehyde and keto groups in the spectrum (although there is a very weak band at 1635 cm^{-1}). Stretching vibrations of OH bound by hydrogen bonds are poorly visible (very wide band at 3000-3500 cm^{-1}).

There is a band at 1350 cm^{-1} with a shoulder in higher wave numbers (may correspond to combinations of O-H bend, CH_2 and CH_3 deformation, C-H alkene bend, C-N stretching vibrations of carboxamide, and symmetrical stretching vibrations of the -CO_2- carboxylate ion (assuming 1635 cm^{-1} asymmetrical), 832 cm^{-1} (out-of-plane defarmation vibrations of C-H in substituted aromatics). The IR spectrum of fulvic acid purified by the Shubarkol sorbent, obtained from potassium humate, shows the OH group (broad band 3000-3500 cm^{-1}), and the C=O stretching vibrations (1717 cm^{-1}; C=O stretching vibrations of saturated fatty acids, carboxyl, aldehyde and keto groups) and C=C stretching (1653 cm^{-1}).The bands at 1361 cm^{-1} may correspond to combinations bending vibrations of CH_2 and CH_3 groups, C-H alkene bend), at 1103 cm_{-1} - stretching vibrations of C-O, C-O-C stretching.

The IR spectrum of fulvic acid purified by the sorbent ”Shoptykol” obtained from potassium humate is very similar to the spectrum of fulvic acid from potassium humate purified by the sorbent ”Shubarkol”, but the bands for vibrations of OH groups and at 1718 cm^{-1} related to stretching vibrations of C=O are less intense . At the same time, there is a significant broadening of the bands in the range of 1300-1450, 1400 cm^{-1} (with reduced intensity) and in the range of 1100-1200 cm^{-1} (all these bands are typical for bending vibrations of CH_2 and CH_3 groups, C-H alkene as well as C-O valence vibrations). A clearly pronounced band appears at 953 cm^{-1}, which may be related to out-of-plane bending vibrations of the C–H bond in the substituted aromatic.

The following bands are clearly visible in the IR spectrum of fulvic acid purified by the “Coconut” sorbent, obtained from potassium humate: 3000-3650 cm^{-1} (stretching vibrations of various OH groups), 1716 cm^{-1} (stretching vibrations of C=O), approximately 1620 cm^{-1} (stretching C=C), 1316 cm O–C stretching), 1043 cm^{-1} (stretching vibrations of C-O in polysaccharides or polysaccharide-like substances), 821 cm^{-1} (out-of-plane deformation vibrations of C-H in a substituted aromatic) (Figure 1).

NMR analysis of fulvic acid was carried out on a JEOL ECA-500 MHz NMR spectrometer. In the spectrum of the original potassium salt sample (before cleaning with sorbents), resonances of the methyl group (1.20 ppm) and aliphatic protons in the alpha position to aromatic, alkenyl and carbonyl groups (2.04 and 2.18 ppm) are visible. In the range from 3.0 ppm to 4.0 ppm, there are resonances corresponding to aliphatic protons in ester groups and in the alpha position to OH and OR groups, as well as aliphatic OH protons. In the range of 6.90-9.00 ppm, there are resonances of aromatic and heteroaromatic protons. Also, a very broad signal of 6.50–9.50 ppm can correspond to phenolic and carboxyl OH groups (broadening due to hydrogen bonds).

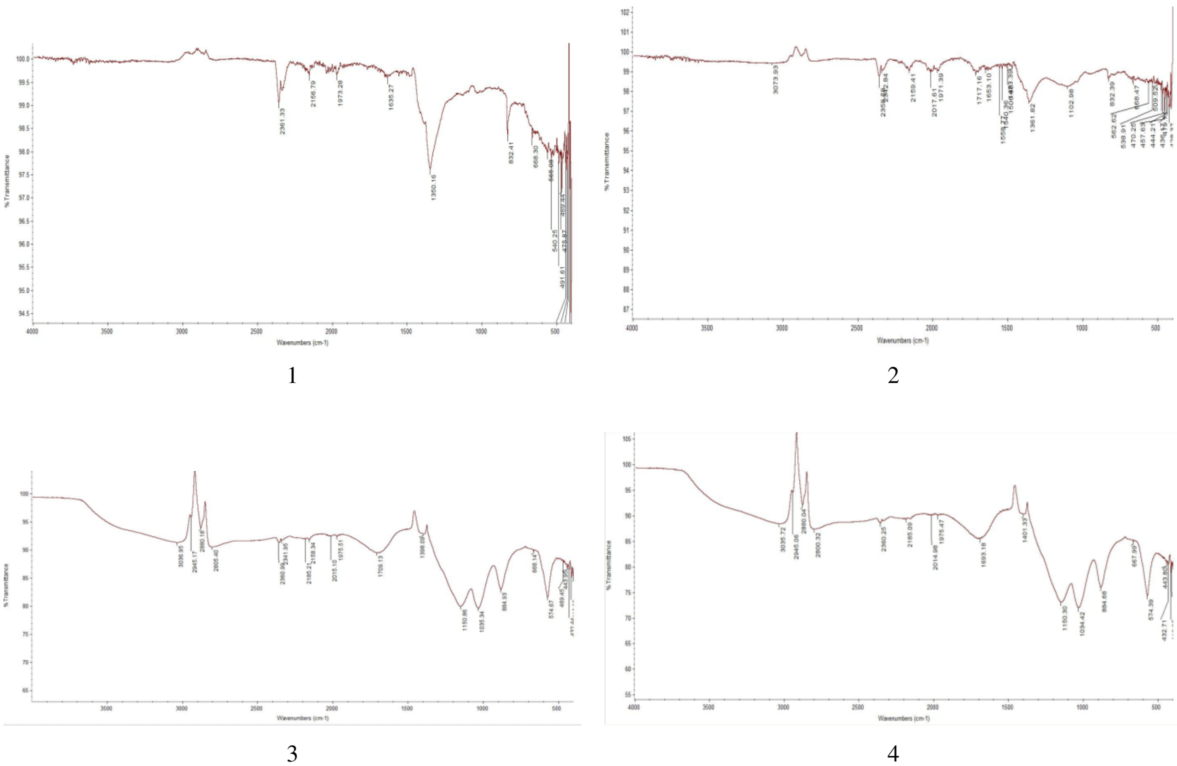

Fig. 1 - Initial fulvic acid obtained from potassium humate, 2- FA, passed through the «Shubarkol» sorbent, 3- FA passed through the «Shoptykol» sorbent, 4- FA passed through the «Coconut» sorbent

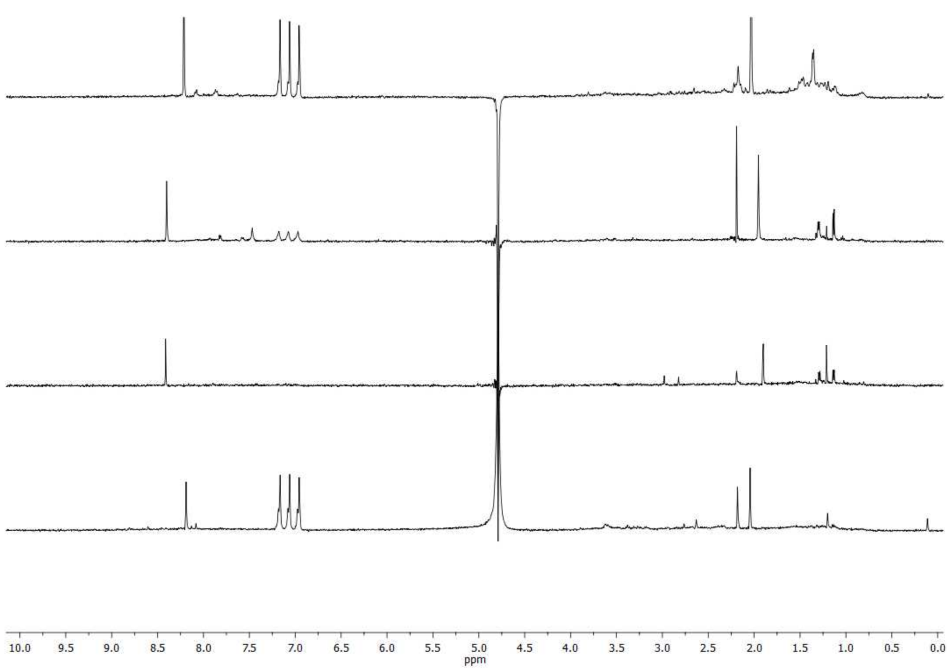

Fig. 2 - Initial fulvic acid obtained from potassium humate, 2- FA, passed through the «Shubarkol» sorbent, 3- FA passed through the «Shoptykol» sorbent, 4- FA passed through the «Coconut» sorbent

Table 3 - Equilibrium constants (pK) and sample concentrations

Name	Equilibrium constant (pK)	Concentration, mg/l
Initial FA before purification	7,58	163
FA after «Shubarkol» sorbent, cation exchanger KU-2-8 and membrane treatment	8,62	137
FA after «Shoptykol» sorbent, cation exchanger KU-2-8 and membrane treatment	12,31	127
FA after «Coconut» sorbent, cation exchanger KU-2-8 and membrane treatment	-	125

When cleaning with the Shubarkol sorbent, a significant part of the aromatic signals disappears, while the aliphatic part of the spectrum changes, where several resonances of terminal methyl groups (1.13-1.30 ppm) can be distinguished, as well as proton signals in the alpha position to aromatic, alkenyl and carbonyl groups (1.90 and 2.19 md) and protons in ester groups, in the alpha position to OH and OR groups and aliphatic OH protons (2.82 and 2.98 md). When cleaning with «Shoptykol» and «Coconut» sorbents, changes are also observed in the aliphatic and aromatic regions of the spectra. In the first case («Shoptykol»), there are fewer protons of ester groups and aliphatic protons in the alpha position to OH and OR groups (range 3.0-4.0 ppm), although there are more aromatic and heteroaromatic protons (6.97-8.40 ppm). In the second case («Coconut»), the number of terminal saturated hydrocarbon groups (1.05-1.60 ppm) and protons in the alpha position to aromatic and carbonyl groups (2.03-2.17) increases, and the number of protons of ester groups and in the alpha position to OH and OR groups decreases (range 3.0–4.0 ppm), while almost no changes compared to the original sample are observed in the aromatic region of the spectrum (6.90–9.00 ppm) (Figure 2).

The titration curves of fulvic acid, determined by the pH-metric titration method, were constructed, and the equilibrium constant was determined. Table 3 shows the equilibrium constant and concentration of the samples.

Research work continues to study the physicochemical properties of the obtained FA and the possibility of their application.

Conclusion: Fulvic acid was isolated from «Maykuben» oxidized brown coal (Kazakhstan). The resulting fulvic acid was purified by the Forsyth method. Purified fulvic acid can be used in medicine as a medicine, as an antiviral drug, and also as a flavoring agent for drinks in the food industry. Fulvic acid is widely used as a plant growth stimulant, as a drug and dye, as an antiallergic drug, as an immunostimulant and as an antiviral drug. Based on the results of the study, it was proven that the molecular structure of fulvic acids is an aromatic structure with aliphatic substituents. As a result, the amount of all ions compared to the amount of the initial fulvic acid content was significantly reduced at the final stage of purification - membrane purification. Fulvic acid improves metabolic processes, restores energy reserves of cells, has antioxidant properties, neutralizes heavy and toxic metals, and helps remove toxins from the living body. Fulvic acid has antibacterial properties and affects replication, and also has the ability to destroy viral cells. This is an effective and safe drug in the treatment of various bacterial and viral diseases. Fulvic acid is also known to be used for agricultural purposes such as fertilizers and soil amendments, as fulvic acid has the highest degree of biological activity and nutrient complexing ability among humic substances and does not bind to many pesticides. Fulvic acid can be used by people living in environmentally polluted areas for gastrointestinal diseases, allergic conditions, poisoning and weakened immunity.

Due to its high solubility and low molecular weight, fulvic acid is a very active participant in natural chemical processes.

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