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INFLUENCE OF ULTRASONIC TREATMENT ON THE STRUCTURE AND PHYSICOCHEMICAL PROPERTIES OF BROWN COAL HUMIC ACIDS

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Abstract. The relevance of the topic is driven by the need to develop efficient and environmentally safe technologies for processing brown coal to obtain modified humic substances with tailored properties. This work investigates the effect of ultrasonic treatment on the physicochemical properties and molecular structure of humic acids derived from brown coal of the Oy-Karagay deposit (Republic of Kazakhstan). The study aims to substantiate and optimize the time parameters of mechanoacoustic treatment for the targeted production of physiologically active and bioavailable humic substances. Ultrasonic treatment of brown coal was performed in a specialized sealed container using a "UZV-1.3 Sapphire" ultrasonic bath (frequency: 35 kHz, power: 50 W) within a time interval of 15-90 minutes. Humic acids were isolated from the coal via aqueous alkaline extraction with sodium hydroxide, followed by precipitation in an acidic medium. The dynamics of technical composition changes were investigated using a MOC63u (Shimadzu) automatic analyzer and according to GOST 11022-95.

The transformation of functional groups and the molecular framework was evaluated by Fourier-transform infrared spectroscopy (Bruker Tensor II), followed by the calculation of fundamental spectral indices. It was found that cavitation effects on the coal matrix proceed through a maximum and trigger coupled processes of ballast aliphatic periphery destruction and deep oxidation of the coal organic matter. Acoustic treatment for 45 minutes was shown to be optimal. This mode provides maximum hydrophilization of the matrix (sorption moisture increase up to 11.52%) and peak values of efficiency indices: aromaticity ($I_{ar/al} = 3.15$), hydrophilicity ($I_{hyd} = 1.48$), and carboxylation ($I_{carb} = 0.56$). This indicates maximum structural saturation with active groups ($-COOH$, $-OH$) that form chelating and biostimulating capacity. Extending the treatment time beyond 60 minutes is impractical due to decarboxylation and secondary particle reagglomeration. The results obtained can be applied in technological schemes for the comprehensive processing of solid fossil fuels to produce highly effective organomineral growth stimulants and environmentally safe fertilizers for agriculture.

Keywords: brown coal, humic acids, ultrasonic treatment, infrared spectroscopy, spectral indices

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УЛЬТРАДЫБЫСТЫҚ ӨНДЕУДІҢ ҚОҢЫР КӨМІР ГУМИН ҚЫШҚЫЛДАРЫНЫҢ ҚҰРЫЛЫМЫ МЕН ФИЗИКА-ХИМИЯЛЫҚ ҚАСИЕТТЕРІНЕ ӘСЕРІ

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Аннотация. Зерттеудің өзектілігі берілген қасиеттері бар модификацияланған гуминдік заттарды алу мақсатында қоңыр көмірді өндеудің тиімді әрі экологиялық қауіпсіз технологияларын әзірлеу қажеттілігімен анықталады. Жұмыста Қазақстан Республикасындағы Ой-Қарағай кен орнының қоңыр көмірінен алынған гумин қышқылдарының физика-химиялық қасиеттері мен молекулалық құрылымына ультрадыбыстық өндеудің әсері зерттелді. Зерттеудің мақсаты — физиологиялық белсенділігі мен биожетімділігі жоғары гуминдік заттарды мақсатты түрде алу үшін механоакустикалық өндеу ұзақтығын ғылыми негіздеу және оңтайландыру. Қоңыр көмірді ультрадыбыстық өндеу арнайы герметикалық ыдыста UZV-1.3 Sapphire ультрадыбыстық ваннасын пайдалана отырып, 35 кГц жиілікте және 50 Вт қуатта 15–90 минут аралығында жүргізілді. Гумин қышқылдары натрий гидроксидімен сулы-сілтілі экстракциялау және кейін қышқыл ортада тұндыру арқылы бөлініп алынды. Үлгілердің техникалық құрамының өзгеруі МОС63и (Shimadzu) автоматты анализаторында және ГОСТ 11022-95 талаптарына сәйкес анықталды. Функционалдық топтар мен молекулалық қаңқаның өзгерістері Bruker Tensor II құралында Фурье-түрлендірілген инфрақызыл спектроскопия әдісімен бағаланып, негізгі спектрлік индекстер есептелді. Нәтижелер көмір матрицасына кавитациялық әсер экстремум арқылы өтетінін және балластық алифаттық периферияның бұзылуы мен көмірдің органикалық массасының терең тотығуы қатар жүретінін көрсетті. 45 минуттық ультрадыбыстық өндеу оңтайлы режим ретінде анықталды. Осы жағдайда матрицаның ең жоғары гидрофилденуі байқалып, сорбциялық ылғалдылық 11,52%-ға дейін артты, ал негізгі спектрлік индекстер ең жоғары мәндерге жетті: ароматтылық $I_{ar/al} = 3,15$, гидрофилділік $I_{hyd} = 1,48$ және карбоксилдену $I_{carb} = 0,56$. Бұл нәтижелер құрылымның хелаттаушы және биостимуляциялаушы қасиеттерді қамтамасыз ететін белсенді –COOH және –ОН топтарымен барынша қаныққанын көрсетеді. Өндеу ұзақтығын 60 минуттан асыру декарбоксилдену және бөлшектердің қайталама агрегациясы салдарынан тиімсіз екені анықталды. Алынған нәтижелер ауыл шаруашылығына арналған жоғары тиімді органо-минералдық өсу стимуляторлары мен экологиялық қауіпсіз тыңайтқыштар алу үшін қатты жанғыш қазбаларды кешенді өндеудің технологиялық сызбаларында қолданылуы мүмкін.

Түйін сөздер: қоңыр көмір, гумин қышқылдары, ультрадыбыстық өндеу, инфрақызыл спектроскопия, спектрлік индекстер

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ВЛИЯНИЕ УЛЬТРАЗВУКОВОЙ ОБРАБОТКИ НА СТРУКТУРУ И ФИЗИКО-ХИМИЧЕСКИЕ СВОЙСТВА БУРОУГОЛЬНЫХ ГУМИНОВЫХ КИСЛОТ

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Аннотация: Актуальность исследования обусловлена необходимостью разработки эффективных и экологически безопасных технологий переработки бурого угля для получения модифицированных гуминовых веществ с заданными свойствами. В работе исследовано влияние ультразвуковой обработки на физико-химические свойства и молекулярную структуру гуминовых кислот, выделенных из бурого угля месторождения Ой-Карагай в Республике Казахстан. Цель исследования заключалась в обосновании и оптимизации продолжительности механоакустического воздействия для направленного получения физиологически активных и биодоступных гуминовых веществ. Ультразвуковую обработку бурого угля проводили в герметичной ёмкости с использованием ультразвуковой ванны UZV-1.3 Sapphire при частоте 35 кГц и мощности 50 Вт в течение 15–90 мин. Гуминовые кислоты выделяли методом водно-щелочной экстракции гидроксидом натрия с последующим осаждением в кислой среде. Изменения технического состава исследовали с помощью автоматического анализатора МОС63u (Shimadzu) и в соответствии с ГОСТ 11022-95. Трансформацию функциональных групп и молекулярного каркаса оценивали методом ИК-Фурье-спектроскопии на приборе Bruker Tensor II с последующим расчётом основных спектральных индексов. Установлено, что кавитационное воздействие на угольную матрицу имеет экстремальный характер и инициирует сопряжённые процессы разрушения балластной алифатической периферии и глубокого окисления органического вещества угля. Оптимальной

оказалась продолжительность ультразвуковой обработки 45 мин. При данном режиме достигалась максимальная гидрофилизация матрицы: сорбционная влажность увеличивалась до 11,52%, а спектральные индексы достигали максимальных значений — ароматичности $I_{ar/al} = 3,15$, гидрофильности $I_{hyd} = 1,48$ и карбоксилирования $I_{carb} = 0,56$. Полученные данные свидетельствуют о максимальном насыщении структуры активными группами $-COOH$ и $-OH$, определяющими хелатирующие и биостимулирующие свойства гуминовых веществ. Увеличение продолжительности обработки свыше 60 мин оказалось нецелесообразным вследствие процессов декарбоксилирования и вторичной агрегации частиц. Результаты исследования могут быть использованы в технологических схемах комплексной переработки твёрдых горючих ископаемых для получения высокоэффективных органоминеральных стимуляторов роста и экологически безопасных удобрений для сельского хозяйства.

Ключевые слова: бурый уголь, гуминовые кислоты, ультразвуковая обработка, инфракрасная спектроскопия, спектральные индексы

Introduction. In the context of the modern transition to environmentally friendly and resource-saving technologies, the deep and comprehensive processing of solid fossil fuels is a highly relevant task (Saroja, et al., 2026). Solid fossil fuels encompass organic-derived minerals ranging from peat and oil shale to anthracite (Skursky et al., 2026). Among them, coal traditionally represents the greatest economic and scientific interest, serving as a vital natural resource for the fuel and energy sector (Rybak, et al., 2025). Due to their unique properties, coals must be considered an invaluable raw material for the chemical industry (Wang et al., 2026).

Currently, a critical challenge in studying coal properties is the development of new and the intensification of existing processing methods applicable to both fuel and non-fuel utilization sectors (Jiayi et al., 2025). Brown coal is traditionally regarded as a low-grade fuel. However, it possesses high potential as a raw material for obtaining valuable non-fuel products (Shao, et al., 2021). Among these, humic acids (HAs) occupy a special place (Zhang et al., 2020). HAs are complex, high-molecular-weight natural compounds (Pal et al., 1985). They possess a unique complex of physicochemical properties (Leoš Doskočil, et al., 2018), which enables their effective application as environmentally safe fertilizers, plant growth stimulants, and for other purposes (Kunanbayeva, et al., 2020; Shah et al., 2018).

Literature review. The development of environmentally oriented agriculture dictates the need for the widespread implementation of highly effective and safe plant growth stimulants (Ismailov et al., 2023). Among these, humic fertilizers obtained from natural organic raw materials, specifically brown coals, occupy a special place (Akhmetova et al., 2020). Brown coals from the Oy-Karagai deposit (Republic of Kazakhstan) represent a promising and economically accessible source of humic substances due to their high content of physiologically active components (Kairbekov et al., 2018). However, native humic acids (HAs) from mature coals often possess a rigid, polycondensed structure and low solubility (Sarsenbek et al., 2021). This

significantly limits their bioavailability for agricultural ecosystems (Dzheldybaeva et al., 2022). One of the most promising, environmentally safe, and high-tech methods for intensifying physicochemical processes is ultrasonic (cavitation) treatment (UST) (Moskalenko et al., 2010; Kairbekov et al., 2020).

The impact of high-intensity ultrasound on liquid media is accompanied by the effect of acoustic cavitation (Vijyendra Kumar et al., 2025). The local ultra-high pressures and temperatures, as well as powerful shear stresses arising from the collapse of cavitation bubbles, are capable of inducing macromolecular destruction, cleavage of intermolecular bonds, and the generation of free radicals (Patrakov et al., 2007). The application of ultrasound to humic acid solutions and suspensions allows for the disintegration of large molecular aggregates, a reduction in molecular weight, an increase in the content of active oxygen-containing functional groups (carboxyl and phenolic hydroxyls), and an enhancement of the system's hydrophilicity without using aggressive chemical reagents (Redhwan Al-Akbari et al., 2026).

Despite the existence of individual studies (R. Al-Akbari et al., 2024) in the field of sonochemical modification of natural polymers, systematic data regarding the mechanisms of the influence of ultrasonic exposure parameters (frequency, power, duration, and temperature regime) on the fine structure and the complex of physicochemical properties of brown coal humic acids remain fragmented.

The aim of this work is to study the effect of ultrasonic cavitation treatment duration on the physicochemical properties and molecular structure of humic acids isolated from brown coal of the Oy-Karagai deposit to evaluate the potential for their further practical application.

Materials: Oy-Karagay deposit coal, sodium hydroxide (analytical grade) GOST 4328-77, universal indicator paper (pH 0-12) TU 2642-054-23050963-2008, distilled water, hydrochloric acid (chemically pure) GOST 3118-77 Amdt. 1, "Blue Ribbon" filter paper TU 2642-001-68085491-2011.

Methods: The analytical moisture content (W^a) of the initial and sonicated coal samples, as well as their humic acids, was determined using a MOC63u automatic moisture analyzer (Shimadzu, Japan). Measurements were performed in automatic drying completion mode at 105°C until a constant sample weight was achieved.

The analytical ash content (A^a) was determined in accordance with GOST 11022-95 "Solid mineral fuels. Determination of ash content."

Fourier-transform infrared (FTIR) spectra were recorded on a Bruker Tensor II spectrometer (USA) in transmission mode using potassium bromide (KBr) pellets.

Calculation of FTIR Spectral Indices:

To quantitatively evaluate the transformation of the molecular structure and functional composition of humic acids, fundamental spectral indices were calculated based on the optical density (D) at specific baselines of characteristic absorption bands:

Aromaticity Index ($I_{ar/al}$): Evaluates the ratio of aromatic structures to aliphatic fragments.

$$I_{ar/al} = \frac{D_{1600}}{D_{2920}}; \quad (1)$$

Where D_{1610} corresponds to $C = C$ stretching vibrations of aromatic rings, and D_{2920} corresponds to asymmetric $C - H$ stretching of aliphatic $-CH_2-$ groups.

Hydrophilicity Index (I_{hyd}): Reflects the degree of enrichment of the molecular structure with polar hydrophilic groups relative to the hydrophobic framework.

$$I_{hyd} = \frac{D_{3400}}{D_{1600}}; \quad (2)$$

Where D_{3400} corresponds to $O-H$ stretching vibrations of hydrogen-bonded phenolic and alcoholic hydroxyl groups.

Carboxylation Index (I_{carb}): Indicates the density of carboxylic acid functional groups.

$$I_{carb} = \frac{D_{1710}}{D_{1600}}; \quad (3)$$

Where D_{1710} corresponds to $C=O$ stretching vibrations of un-ionized carboxylic groups ($-COOH$).

Methodology for Ultrasonic Coal Treatment: A 10 g sample of dry native coal was placed in a thin, uniform layer inside a specialized plastic tray. The tray was hermetically sealed to prevent any ingress of external moisture and then immersed into the working zone of a "UZV-1.3 Sapphire" ultrasonic bath (Russia). Distilled water was used as the contact transmitting medium in the bath. The dry coal samples were treated at a fixed frequency of 35 kHz and a power of 50 W within a time interval of 15-90 minutes.

Isolation Methodology of Humic Acids from Coal: Humic acids were isolated from the coal via aqueous alkaline extraction with sodium hydroxide, followed by precipitation in an acidic medium. A coal sample of 5.0 g ($\leq 1\text{mm}$), weighed to the nearest 0.0002 g, was placed into a 1000 mL conical flask, and 500 mL of a 1 % sodium hydroxide solution was added. The flask contents were heated to 80°C and stirred using a shaker for 2 hours. The resulting mixture was centrifuged using an RS-6MTs laboratory centrifuge at 1500 rpm for 15 minutes. The insoluble coal residue was washed with a small amount of alkali. The filter containing the coal residue was dried and weighed. Subsequently, the volume of the obtained extract was measured, and the humic acid content was determined by precipitation with 5% HCl until a pH of 2-3 was reached. The mixture with the precipitate was kept on a water bath for 30 minutes. The formed humic acid precipitate was filtered through a pre-weighed ashless filter paper (Blue Ribbon). The precipitate was then washed with distilled water. The filter with the precipitate was removed from the Büchner funnel, folded several times, and placed into pre-dried and pre-weighed weighing bottles. The weighing bottle with the filter and precipitate was then placed in a drying oven and dried at a temperature not exceeding 75°C until a constant weight was achieved. Control drying operations were performed until the mass difference between two consecutive weighings did not exceed 0.001 g. The mass of the precipitate was determined by accounting for the mass of the weighing bottle and the filter paper.

Results and discussion

Effect of Ultrasonic Treatment on Sample Moisture and Ash Content.

Mechanoacoustic treatment of the coal matrix structure exerts a pronounced effect on the moisture sorption process and the redistribution of the mineral phase. The physicochemical nature of the observed changes in the coal samples is driven by mechanoacoustic dispersion and destruction of the organic matrix under the influence of an intense ultrasonic field transmitted through the walls of the sealed tray. The increase in analytical moisture content from 10.06% to a maximum of 11.52% at the 45th minute is not associated with the absorption of an external liquid phase. This effect is explained by a sharp increase in the specific surface area and porosity of the material resulting from acoustic micro-chipping and fragmentation of the coal particles (Figure 1). Furthermore, local alternating stresses lead to the cleavage of the weakest chemical bonds in the organic matter of coal (OMC), accompanied by the formation of active radicals.

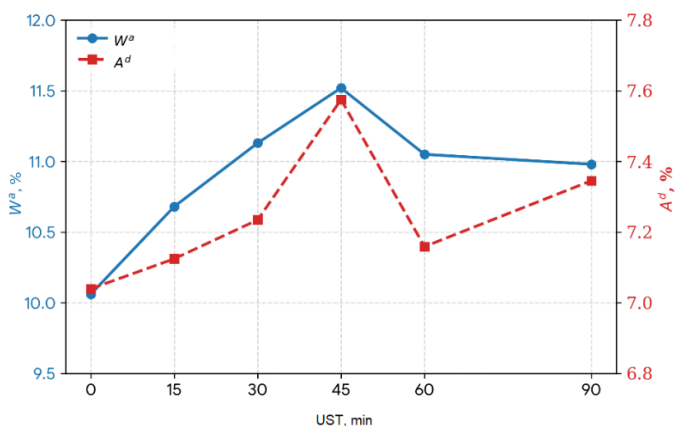


Figure 1 - Influence of ultrasonic treatment (UST) duration on the analytical moisture content (W^a) and ash content (A^d) of coal.

The increase in the relative ash residue content from 7.0395% to 7.5749% in the isolated system indicates the occurrence of destructive processes within the OMC. Intense ultrasonic treatment initiates the mechanochemical destruction of the peripheral fragments of coal macromolecules, which is accompanied by the evolution of volatile gaseous products (CO , CO_2 , CH_4 , H_2). The partial loss of the light organic fraction upon opening the tray leads to a consistent concentration of the non-combustible mineral residue, which is registered as an increase in the dry-basis ash content. The extreme nature of these trends (reaching a maximum at the 45th minute followed by a decline toward the 60th and 90th minutes) indicates that the system passes through a critical activation point. Up to 45 minutes, the prevailing processes are the destruction of the organic matrix and the development of a porous structure. With a further increase in treatment time (60 and 90 min) within the ultrasonic field, processes of secondary ultrasonic coagulation and reagglomeration of highly dispersed coal particles under

the influence of acoustic pressure forces likely begin to dominate. This leads to partial pore blockage, a decrease in the available specific surface area for moisture sorption, and the stabilization of OMC destruction processes.

Comparative Analysis of FTIR Spectra of Native and Sonicated Coals and Their Humic Acids.

To evaluate the changes in the molecular structure and the transformation of functional groups within the organic matter of coal and humic acids (HAs) induced by ultrasonic treatment (UST) of varying durations, a comparative analysis of the FTIR spectra was performed (Figures 2-3).

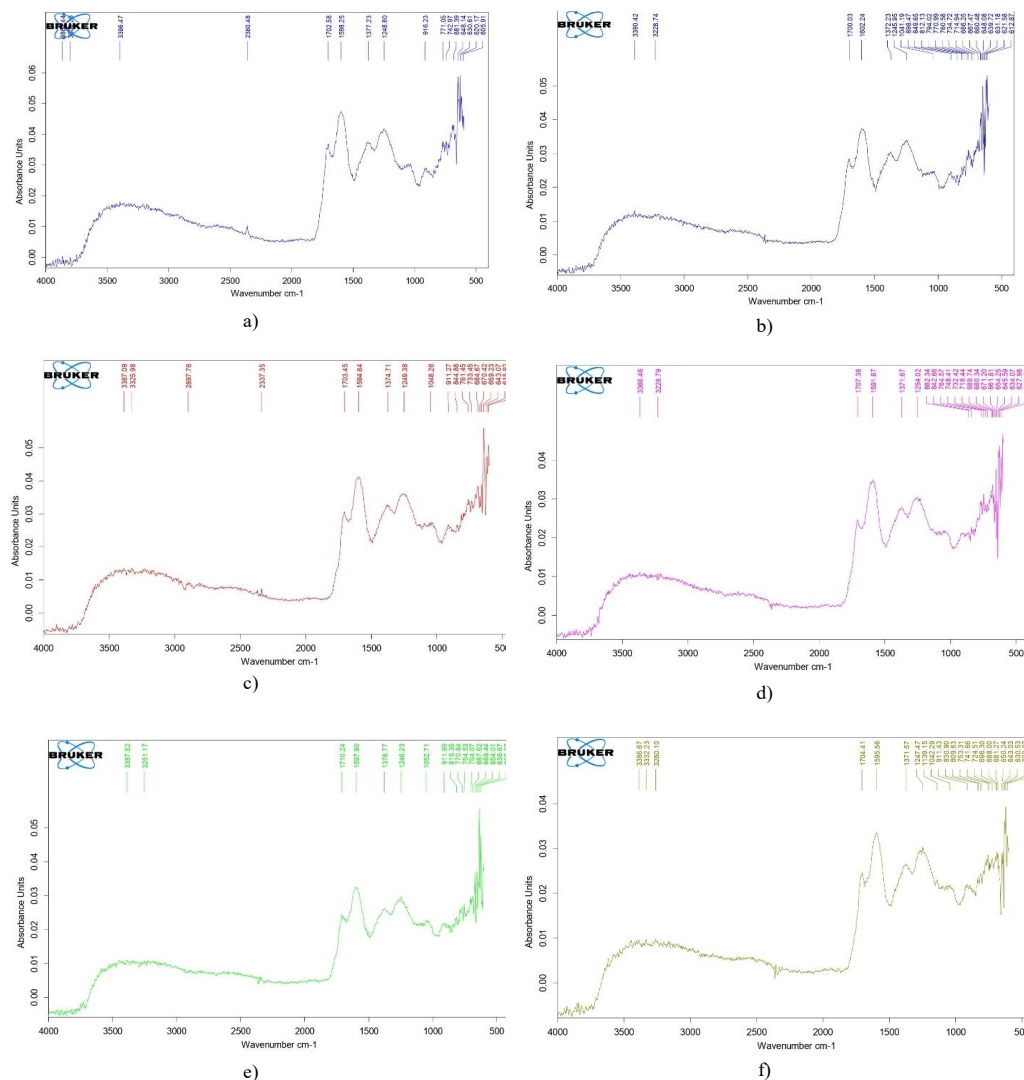


Figure 2 – FTIR spectra of the native and ultrasound-treated coal samples at different exposure times: a) native; b) 15 min; c) 30 min; d) 45 min; e) 60 min; f) 90 min

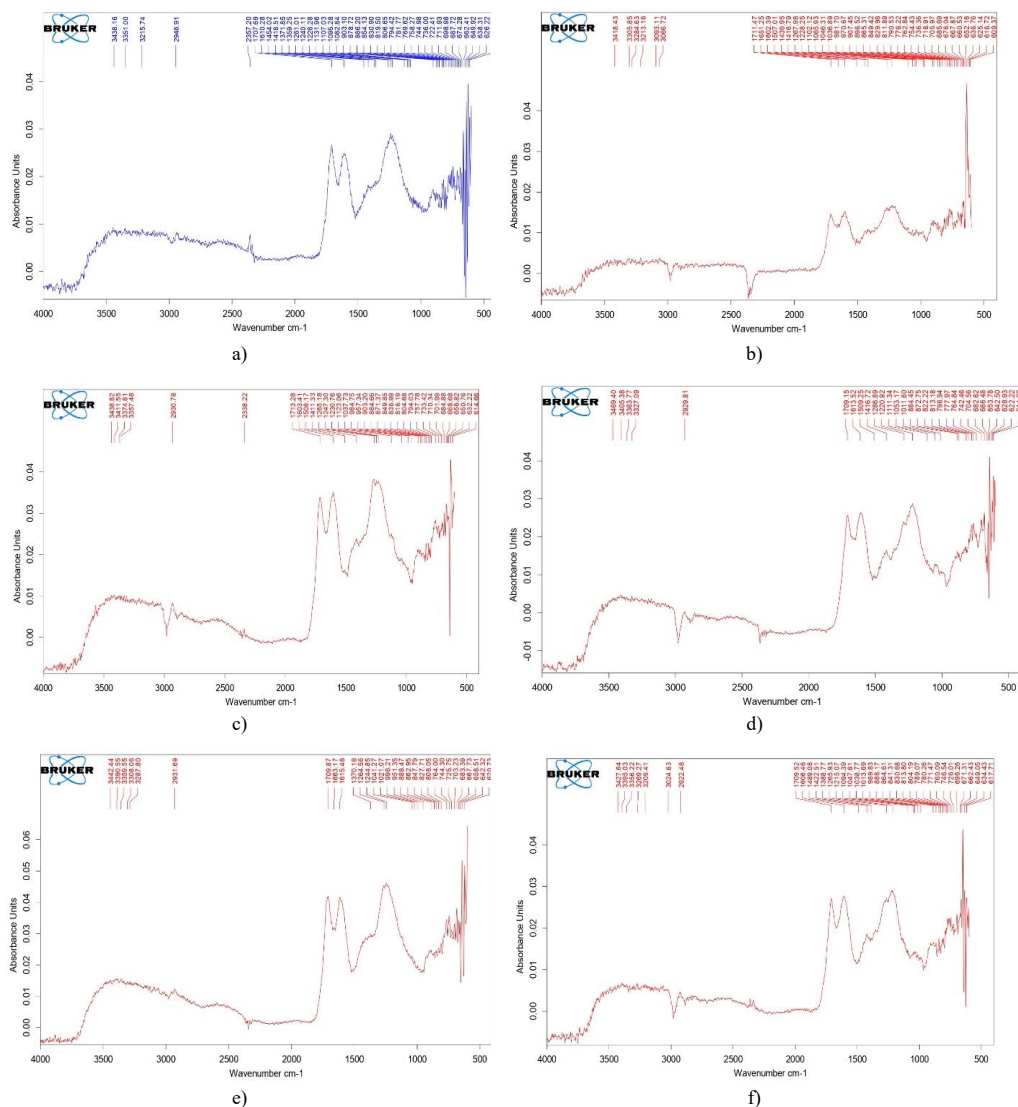


Figure 3 – FTIR spectra of HAs obtained from native and sonicated coals at different exposure times: a) native; b) 15 min; c) 30 min; d) 45 min; e) 60 min; f) 90 min.

FTIR spectroscopy revealed the presence of key functional groups, including hydroxyl, carboxyl, phenolic, and aromatic structures, which are crucial for their sorption and reactive properties (Kadam et al., 2025).

For a comprehensive comparative analysis of the FTIR spectra of HAs, a combined frequency assignment table has been prepared. It clearly illustrates the spectral differences between the native coal matrix and the target component isolated from it - humic acids (Table 1).

Table 1 – Assignment of the main absorption bands in the FTIR spectra of native coal and humic acids from the Oy-Karagay deposit

Spectral range (cm ⁻¹)	Main coal wavenumber (ν, cm ⁻¹)	Main HA wavenumber (ν, cm ⁻¹)	Band assignment and mode of vibration	Functional groups and structural fragments
4000 – 3000	3390 – 3200	3438, 3351, 3215	Broad, strong; stretching (ν _{OH} , ν _{NH})	Hydroxyl (phenols, alcohols) and amino groups involved in hydrogen bonding. In HA, the peaks are more differentiated.
3000 – 2800	~2920 (shoulder)	2946	Weak; asymmetric stretching (ν _{CH2} , ν _{CH3})	Methylene and methyl groups within aliphatic side chains (–CH ₂ , –CH ₃)
1750 – 1650	1700 – 1710	1707	Medium / shoulder; stretching (ν _{C=O})	Unconjugated carbonyl groups within carboxylic groups (–COOH) and ketones.
1650 – 1580	1602 – 1595	1610	Strong; stretching (ν _{C=C} , ν _{as COO-})	Skeletal vibrations of the aromatic ring conjugated with carbonyls; asymmetric vibrations of carboxylates.
1580 – 1500	~1540 (weak)	1541	Medium; skeletal ring and deformation (δ _{NH})	Aromatic core; amide fragments of the periphery (Amide II band, more pronounced in HAs).
1450 – 1350	1454, 1377	1454, 1418, 1371, 1359	Medium / weak; deformation (δ _{CH2} , δ _{CH3}), (symmetric stretching ν _{s COO-})	In-plane vibrations of alkyl groups; symmetric stretching vibrations of ionized –COO ⁻ groups.
1300 – 1150	1260 – 1220	1261, 1240, 1226	Medium; stretching (ν _{C-O}), deformation (δ _{OH})	Ether bridges Ar–O–R; hydroxyls of phenolic and carboxylic fragments.
1150 – 1000	1092 – 1035	1131, 1107, 1082	Strong, complex; stretching (ν _{C-O} , ν _{Si-O/Al-O})	Alcohol groups, carbohydrate residues; silicate/clay mineral impurities of coal.
1000 – 700	916, 800 – 720	903 – 722 (серия пиков)	Weak; out-of-plane deformation (γ _{C-H})	Methine groups of the aromatic ring with various substitution types (from 1 to 4 isolated H-atoms).
700 – 400	662 – 530	662, 649, 626	Narrow, strong in coal; inorganic deformation in HAs	Mineral (ash) component of coal (quartz, aluminosilicates) and sulfur-containing compounds.

Upon transition from coal to HAs, the band at 1610 cm⁻¹ becomes narrower and more intense, which proves the successful concentration of humic substances and the isolation of the aromatic core from the bulk coal matrix. In the HAs spectrum, the 3438-3215 cm⁻¹ region (hydroxyl groups) and the peaks around 1418-1359 cm⁻¹ (carboxylates) are much more pronounced than in coal. This confirms the high content of active sites in humic substances, which are responsible for plant growth stimulation and micronutrient binding (Zanin et al., 2019).

The transformation of functional groups within the organic matter of sonicated coals and HAs extracted from them, along with the dynamics of changes in key absorption bands, is presented in comparative Table 2.

Table 2 – Comparative characteristics of HA structure transformation under UST

Sample (UST)	Key Changes in the FTIR Spectrum	Structural interpretation and significance for fertilizers (Lamar et al., 2024)
Native HA	A classic profile. Intense bands of O–H ($\sim 3400\text{ cm}^{-1}$), conjugated C=C, and carboxylates (1610 cm^{-1}). Distinct aliphatic peaks (2946 cm^{-1}) and a carbohydrate tail ($1100\text{--}1000\text{ cm}^{-1}$)	High molecular weight, a rigid skeleton, and a moderate number of accessible hydrophilic centers.
UST 15-30 min	An increase in intensity and broadening of the $3438\text{--}3215\text{ cm}^{-1}$ band. The formation and elevation of a carbonyl shoulder at 1707 cm^{-1} . A slight decrease in the 2946 cm^{-1} peak	Initial activation stage: hydrolytic cleavage of macromolecules and oxidation of the periphery occur. The concentration of phenolic and carboxyl groups increases, which improves the solubility and chelating properties of the fertilizer.
UST 45 min	Maximum intensity of the O–H absorption bands (3418 cm^{-1}) and carboxyl groups ($1710\text{--}1704\text{ cm}^{-1}$). Minimum relative intensity of aliphatic C–H bonds ($2946\text{--}920\text{ cm}^{-1}$). Clear resolution of the bands in the 1610 and 1540 cm^{-1} region	Optimal activation regime: deep destruction of ballast aliphatic chains is achieved (consistent with the analytical moisture maximum, $W^a = 11,52\%$). The structure becomes maximally saturated with active centers ($-\text{COOH}$ and $-\text{OH}$) responsible for the transport of micronutrients into plants.
UST 60-90 min	A decrease in the intensity of the O–H band (down to 3366 cm^{-1}). Partial smearing of the carbonyl shoulder (1704 cm^{-1}). An increase in the peaks within the inorganic residue region ($662\text{--}626\text{ cm}^{-1}$).	Over-processing and destruction stage: prolonged cavitation leads to decarboxylation and secondary aggregation (condensation) of the cores. The relative proportion of mineral impurities in the HAs increases, which is confirmed by ash content data.

According to the data obtained in Table 2, it can be seen that the changes in the molecular structure of brown coal HAs under the influence of ultrasonic cavitation exhibit a stage-by-stage pattern, which is clearly recorded by the transformation of key absorption bands in the FTIR spectra.

Native HA samples possess a classic spectral profile characteristic of poorly modified, mature brown coal organic matter. Its structure is dominated by intense absorption bands of O–H groups ($\sim 3400\text{ cm}^{-1}$), which are incorporated into an extensive system of inter- and intramolecular hydrogen bonds, as well as bands at 1610 cm^{-1} caused by the vibrations of conjugated C=C bonds of the aromatic skeleton and carboxylate ions. The peripheral part of native HAs is represented by long hydrophobic alkyl chains, as confirmed by distinct aliphatic peaks (2946 cm^{-1}), and a labile carbohydrate tail in the $1100\text{--}1000\text{ cm}^{-1}$ region.

At the initial stage of sonochemical exposure (mild regime), destructive-oxidative modification processes are initiated, affecting predominantly the terminal groups of the periphery. In the FTIR spectrum, this is manifested by an increase in intensity and broadening of the $3438\text{--}3215\text{ cm}^{-1}$ band, indicating the accumulation of hydroxyl (phenolic and alcoholic) fragments and the complication of the hydrogen bond network. Concurrently, the formation and elevation of a shoulder of free carbonyl and carboxyl groups at 1707 cm^{-1} is recorded, serving as a direct marker of oxidation. At the same

time, the rigid carbon skeleton and the bulk of the aliphatic chains still maintain their integrity, as indicated by only a slight decrease in the intensity of the 2946 cm^{-1} peak.

Upon reaching the optimal sonication parameters (maximum efficiency regime), the restructuring of HA macromolecules reaches its peak. At this point, the maximum intensity of the absorption bands of hydrophilic O–H groups (3418 cm^{-1}) and carboxyl groups (1710–1704 cm^{-1}) is registered, which testifies to the ultimate degree of sonochemical oxidation of the structure and a sharp increase in the cation exchange capacity. Deep destruction of the hydrophobic periphery is confirmed by the decline to a minimum of the relative intensity of aliphatic C–H bonds (2946–2920 cm^{-1}). An important structural consequence of the "cleansing" of the aromatic core from side chains is the clear resolution of the bands in the 1610 and 1540 cm^{-1} region, reflecting the differentiation of vibrations between the skeletal C=C bonds of the core and the ionized carboxylate groups.

Thus, the dynamics of spectral changes demonstrate a sequential transition from the high-molecular-weight hydrophobic structure of native HAs to an optimally destructured, maximally oxidized, and hydrophilic form possessing the highest agrochemical potential.

Based on the experimental data of the FTIR spectra of HAs during ultrasonic treatment, three fundamental agrochemical indices were calculated (Table 3).

Table 3 – Dynamics of spectral efficiency indices of HAs from Oy-Karagai coal

UST time (min)	$I_{ar/al}$	I_{hyd}	I_{carb}	Sample Characterization
0 (Native)	1.85	1.12	0.31	Rigid hydrophobic structure, low bioavailability.
15	2.10	1.18	0.35	Onset of hydrolysis, partial loss of light aliphatic chains.
30	2.45	1.29	0.42	Destruction of ballast periphery, exposure of active centers.
45	3.15	1.48	0.56	Peak efficiency: Maximum amount of OH- and COOH-групп.
60	2.90	1.35	0.48	Onset of decarboxylation and oxidative destruction of groups.
90	2.62	1.21	0.40	Secondary condensation (densification), drop-in activity.

The calculation of spectral coefficients clearly confirms the extreme nature of HA modification in an ultrasonic field (35 kHz, 50 W):

Activation period (0–45 min): The aromaticity index $I_{ar/al}$ increases from 1.85 to 3.15 (a 70% growth). This is due to the selective cavitation-induced destruction of thermally unstable, loose aliphatic fragments (decrease in the 2946 cm^{-1} peak). Concurrently, the hydrophilicity index I_{hydr} reaches its maximum (1.48), while the carboxylation index I_{carb} reaches a value of 0.56. This indicates the deep oxidation of the coal organic matrix, accompanied by the formation of additional functional groups. Thus, the HA sample obtained from the coal sonicated for 45 minutes possesses the optimal combination of solubility and chelating capacity.

During coal sonication exceeding 60 minutes (over-processing period, 60–90 min): A synchronous decline in all three indices is observed. The decrease in I_{carb} to 0.40 testifies to thermal and radical decarboxylation (cleavage of CO_2 from HA molecules), while the drop in $I_{ar/al}$ indicates partial destruction of the aromatic skeleton

of the macromolecules themselves under the action of severe cavitation-induced shear deformations.

Conclusion. FTIR spectroscopy and thermogravimetric analysis demonstrated the high efficiency of ultrasonic cavitation treatment (35 kHz, 30 W) for the targeted modification of the physicochemical properties and molecular structure of humic acids derived from Oy-Karagai deposit coal. It was established that the cavitation exposure initiates coupled processes of aliphatic periphery destruction and oxidation of the organic matrix. This is confirmed by an increase in the analytical moisture of the raw material from 10.06% to 11.52%, accompanied by a simultaneous redistribution of the stretching vibration intensities of aliphatic C–H bonds (2946 cm^{-1}) and carboxyl groups (1707 and 1610 cm^{-1}). The optimal coal sonication time for obtaining humic acids intended for highly active biostimulants and fertilizers is 45 minutes. This regime ensures maximum pore exposure, matrix hydrophilization, and the peak concentration of physiologically active phenolic hydroxyls and carboxyl groups that form the cation-exchange and chelating capacity of the final product. Extending the treatment to 60–90 minutes is impractical, as it initiates processes of decarboxylation and secondary condensation of macromolecules, which reduces the overall solubility and agrochemical value of the resulting humic acids.

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