



Investigation of the Characteristics of Humic Acids Based on Kazakhstan's Local Coal Mining Waste

Ainash Nauanova,¹ Tolkyn Khamitova,^{1,*} Nursanat Parmanbek,^{2,*} Sairagul Tyanakh³ and Fengyun Ma⁴

Abstract

This study presents the synthesis and comparative characterization of humic acids extracted from brown coals of the Kuznetsk and Kumyskuduk deposits in Kazakhstan. Advanced analytical techniques, including infrared (IR) spectroscopy, elemental analysis, thermogravimetric (TGA/DSC) analysis, scanning electron microscopy (SEM), UV-Vis spectroscopy, and band gap analysis, were employed to evaluate their physicochemical properties. The results revealed marked differences in elemental composition, functional group distribution, thermal stability, surface morphology, and optical properties, reflecting the distinct geological origins of each deposit. Notable variations in pH, titratable acidity, and the ratio of carboxyl to phenolic hydroxyl groups suggest differences in buffering capacity and sorption behavior. The higher concentrations of carboxyl and hydroxyl groups in some samples enhance their potential for complexation and adsorption processes. This comparative analysis highlights the potential of these humic acids for use in agriculture, environmental remediation, and wastewater treatment. The findings contribute to a deeper understanding of how geological factors influence the structural and functional characteristics of humic substances, broadening their scope of practical applications and guiding future material development.

Keywords: Brown coal; Humic acid; Kumyskuduk deposit; Kuznetsk deposit; Infrared spectroscopy.

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1. Introduction

Kazakhstan is among the world's leading coal producers, boasting substantial lignite and sub-bituminous coal reserves, particularly in regions such as Karaganda, Ekibastuz, Kuznetsk and Kumyskuduk deposits as well. These coal deposits are abundant in organic matter, presenting an excellent resource for extracting humic acids. With its extensive reserves, Kazakhstan provides a cost-effective and sustainable raw material base for humic substance production, positioning the country to address both domestic and international demands. Humic substances play a crucial role in enhancing soil fertility, improving water retention, and remediating polluted soils by binding heavy metals and organic pollutants. Given Kazakhstan's legacy of industrialization and mining activities, environmental issues like soil and water contamination are prominent challenges. The utilization of humic substances derived from local coal deposits offers a viable pathway for mitigating environmental

degradation and fostering a sustainable market for these materials.^[1-3]

The rising demand for humic substances in Kazakhstan aligns with the country's rich coal reserves and the need for sustainable agricultural and ecological solutions. With strategic investments and supportive policies, Kazakhstan is well-poised to expand its production and export capabilities, contributing to a greener economy. Humic acids (HA), as key constituents of natural organic matter, are ubiquitously present in soils, sediments, and aquatic environments.^[4] These heterogeneous macromolecules, originating from the prolonged decomposition of plant and animal matter, play an integral role in numerous environmental and biological processes. Humic acids exhibit distinct physicochemical properties, including metal ion chelation, adsorption of organic contaminants, and soil structure enhancement. These attributes make humic acids highly relevant for applications in agriculture, environmental restoration, and industrial processes. However, the properties and efficacy of humic acids are significantly influenced by their source material and formation conditions, necessitating comprehensive research into humic acids derived from diverse natural origins.^[5,6]

Lignite, or brown coal, is particularly promising for humic acid extraction due to its elevated organic content and lower

¹Department of Soil Science and Agrochemistry, Faculty of Agronomy, S. Seifullin Kazakh Agro Technical Research University, Astana, 010011, Kazakhstan

²The Institute of Nuclear Physics of the Republic of Kazakhstan, Almaty, 050032, Kazakhstan

degree of carbonization. The Kuznetsk Basin in central Kazakhstan and the Kumuskuduk field represent two significant lignite deposits, each characterized by unique geological and environmental conditions. Despite their abundance, humic acids derived from these coal deposits remain insufficiently studied, particularly in terms of their physicochemical attributes and practical potential. A deeper understanding of the relationship between the coal source and the resulting humic acid properties is essential for optimizing their applicability across various fields.^[1,5]

To date, a substantial body of research has been devoted to the investigation of the structural and functional characteristics of humic acids (HAs) extracted from coal deposits across various regions of the world, including Germany, Russia, China, and the United States.^[7-9] These studies have contributed to the identification of correlations between the geological age of coals, their macro- and microcomposition, and the properties of the resulting humic acids. The contributions of Kazakhstani researchers to this field should also be noted. Several studies^[10,11] have examined the physicochemical properties of HAs derived from the Oy-Karagai and Kiyakty coal deposits. Investigations into their antioxidant activity and elemental composition have demonstrated the potential of domestic coal as a source of humic substances. Moreover, technologies have been developed for producing humic-based fertilizers from brown coals of the Karaganda region, exhibiting plant growth-stimulating effects.^[12] Other studies have highlighted the efficiency of HAs in binding heavy metals and radioactive elements.^[13] Nevertheless, the available scientific literature still contains limited data on humic acids extracted from the Kumuskuduk coal deposit and the Kuznetsk Basin, despite their considerable potential as sources of functional organic matter. Isolated studies, such as those exploring the interactions of heavy metal ions with HAs extracted from the Ekibastuz Basin, further demonstrate the promise of Kazakhstani coal as a valuable feedstock for humic substance production^[14] However, to date, there have been no comparative studies that provide a structural and chemical characterization of HAs from different Kazakhstani deposits using modern analytical techniques (FTIR, TGA/DSC, UV-Vis spectroscopy, elemental analysis), nor studies that place these findings in an international context. The present study aims to address this gap. Its objective is to extract and comparatively analyze humic acids from brown coals of the Kumuskuduk and Kuznetsk deposits in Kazakhstan using a

suite of physicochemical methods. The results obtained will enhance the understanding of the structural features of Kazakhstani humic acids, allow for the assessment of their potential practical applications, and facilitate comparison with counterparts described in international literature.

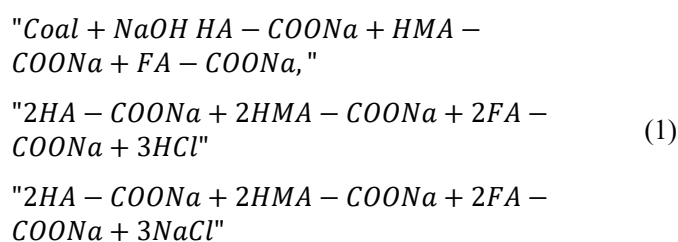
This study aims to synthesize and compare humic acids extracted from brown coal of the Kuznetsk (KUZ) and Kumuskuduk (KUM) deposits. The research involves detailed characterization of their physicochemical properties through techniques such as elemental analysis, functional group identification, thermal characterizations, and structural surface morphology assessment as well. By elucidating the factors that govern the structure and reactivity of humic acids from these distinct coal sources, this study seeks to advance the development of efficient humic acid-based solutions for agricultural, environmental, and industrial applications. Furthermore, the findings will contribute to the broader understanding of the influence of geological factors on humic acid properties, supporting innovations in sustainable technologies and resource management.^[15,16]

2. Materials and methods

2.1 Materials

In the preliminary stage of the experiment, coal samples from the Kumuskuduk (KUM) and Kuznetsk (KUZ) deposits were selected for the study as feedstock for the extraction of humic acids. Materials included sodium hydroxide (NaOH, AR), deionized water, and hydrochloric acid (HCl, 20%). The above reagents were purchased from Sigma Aldrich (Schnelldorf, Germany).

The raw materials were first pulverized to a particle size of 0.5–1 mm using a "Vibrotechnik ShKD-6" crusher in order to increase the surface area and optimize the extraction process. Following this, the ground samples were subjected to drying in an oven at 80°C for 8 hours to ensure complete moisture removal.^[17,18] Humic acids were isolated from the brown coals through an aqueous-alkaline extraction process, wherein sodium hydroxide (NaOH) was used to solubilize the humic acids, followed by their precipitation in an acidic medium. The procedure relies on the reaction of humic acids with NaOH, enabling their extraction and subsequent separation via acid-induced precipitation, as represented by the following Eq. 1.



2.2 HA Extraction

Humic acids were extracted from the coal samples using sodium hydroxide (NaOH) followed by acid precipitation.

³Department of Chemical Technologies and Petrochemicals, Faculty of Chemistry, Karaganda Buketov University, Karaganda, 100028, Kazakhstan

⁴Xinjiang University, Xinjiang Uy-gur Autonomous Region (XUAR), Urumqi, 830046, People's Republic of China

*Email: khamitova.t@inbox.ru (T. Khamitova), parmanbek.nursanat@gmail.com (N. Parmanbek)



Fig. 1: Flow chart of the humic acid preparation.

Initially, 10.0–20.0 g of coal was precisely weighed to an accuracy of 0.0001 g and transferred into a 250 cm³ flask. Subsequently, 100 cm³ of a 4% NaOH solution was added to the flask. Centrifugation was carried out at a speed of 4000 rpm for 15 minutes. Washing was performed using distilled water at a volume of 50 mL per 1 g of sample. The mixture was heated to 80°C and continuously stirred on a shaker for 2 hours to facilitate extraction. The resulting suspension was filtered or centrifuged to separate undissolved carbon residues, which were then rinsed with a small volume of alkaline solution. The residual solid carbon was dried and weighed for further evaluation. The volume of the filtered alkaline solution was recorded, and the humic acid concentration in the resulting product was determined.^[19] Humic acid was precipitated by acidifying the solution to a pH of 2–3 using a 5% hydrochloric acid (HCl) solution. The acidic mixture was left to settle for 60 minutes, after which the precipitated humic acid was collected on pre-weighed (blue strip) filter paper. To eliminate residual impurities and reagents, the precipitate was thoroughly rinsed with distilled water. The filter paper containing the humic acid precipitate was carefully removed from the Büchner funnel, folded, and pre-dried. It was then placed into a pre-weighed beaker and further dried in an oven at 80°C until a constant weight was achieved, ensuring complete moisture removal for accurate determination of the humic acid yield, as schematically illustrated in Fig.1, and, finally, the HA solids obtained were sealed in a sample bag and placed in a dry and cool place for storage.

2.3 Characterization of the properties of coal samples and HA

(1) The elemental composition of the samples was determined using a CHNS-O UNICUBE organic elemental analyzer

manufactured by Elementar Analysensysteme GmbH (Germany). In this paper, the instrument employs the classical Dumas-Pregl method, which involves combustion of the samples in the presence of an oxidizing agent within a controlled inert gas stream. During combustion, oxygen was continuously supplied, facilitating the generation of analytical gases, including carbon dioxide (CO₂), water vapor (H₂O), molecular nitrogen (N₂), and sulfur dioxide (SO₂). To ensure high accuracy in sample preparation, a Mettler Toledo XPR6U Ultra-Microbalance was used for precise weighing. Each sample was placed into disposable tin boats, less than 0.01 mm thick, and carefully sealed using tweezers to minimize any potential sample loss prior to analysis.^[20,21]

(2) Fourier infrared spectroscopy (FSM 1202 FT-IR) was investigated to characterize the identification of functional groups in the coal and HA samples. The samples were finely ground into a homogeneous powder to ensure uniform distribution within the tablet and minimize light scattering. Approximately 2 mg of the sample was used for FTIR analysis. A very small amount of the sample was mixed with 200 mg of KBr to prevent excessive absorption. The resulting mixture was placed into a tablet press mold and compressed under a pressure of approximately 5 tons for several minutes. This high pressure facilitated the formation of a transparent, durable, and uniform tablet with a thickness of approximately 1 mm, allowing effective transmission of infrared radiation. The prepared KBr tablet containing the sample was then positioned in a specialized holder within the FTIR spectrometer. The spectrometer scanned the sample in the infrared range (4000–400 cm⁻¹), recording the absorption spectrum. The peaks observed in the spectrum corresponded to the vibrational frequencies of chemical bonds present in the analyzed molecules.^[15,22,23]

(3) Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) were performed using a LABSYS Evo TG-DTA/DSC analyzer (SETARAM, France) to assess the thermal stability and decomposition behavior of the samples during heating.^[24,25] The coal samples were placed in aluminum oxide crucibles and heated at a constant rate of 10°C/min under an air atmosphere, with the temperature increasing up to 800°C. Throughout the process, the changes in sample mass were continuously monitored to identify the temperatures corresponding to key stages of organic matter decomposition. This analysis enabled the determination of moisture content, volatile matter, fixed carbon, and ash content. Furthermore, the TGA results provided valuable insights into the thermal stability of the coal's organic components and their reactivity potential.

(4) The investigation of the optical properties of the samples in the ultraviolet (UV) range was conducted using a Shimadzu UV-1800 spectrophotometer, developed by Shimadzu Corporation (Japan). The analyses were carried out in the laboratory of the Faculty of Chemistry at Karaganda University named after E.A. Buketov. The working principle of the instrument is based on measuring light absorption in the ultraviolet and visible regions of the spectrum. Absorption occurs due to electronic transitions within the sample molecules under UV radiation exposure. This method enables the quantitative assessment of composition and the study of molecular structures. Coal samples were dissolved in a 5% NaOH solution at a concentration of 0.1 mg/mL, ensuring solution transparency within the required wavelength range. The solutions were placed into quartz cuvettes, which possess high transmittance in the UV region (190–400 nm). A 5% NaOH solution was used as a reference sample and was also placed in a cuvette. The UV spectrophotometer was calibrated using the reference solution. To minimize measurement errors, the instrument performed an automatic signal baseline correction before analysis. The sample cuvette was then placed into the holder, and the spectrophotometer scanned the sample by measuring absorption at various wavelengths, automatically generating an absorption spectrum as a function of wavelength.

(5) A scanning electron microscope (SEM) MIRA3 (TESCAN, Czech Republic) was applied to investigate the surface morphology of the samples of coal and humic acids. This microscope features a high resolution, enabling detailed

microstructural analysis of samples at micron and submicron scales. Particle size and morphology of the samples of coal and humic acids were analyzed at magnifications of $\times 6920$ and $\times 17300$.

(6) Methods for analyzing acid-base properties of humic acids. Measurement of pH in aqueous solutions: The pH of humic acid aqueous solutions was measured using a calibrated pH meter. Samples were dissolved in distilled water at a 1:10 (w/w) ratio, and measurements were taken after a 30-minute stabilization period.^[26] Determination of titratable acidity: Titratable acidity was determined by acid-base titration using 0.1 M NaOH until the endpoint was reached. This allowed estimation of the content of carboxylic and phenolic groups.^[27] Determination of the -COOH/-OH ratio: The ratio of carboxyl to phenolic groups was calculated based on titration data and subsequent determination of group concentrations.^[28]

3. Results and discussion

3.1 Extraction of HA

Humic acid extraction is shown in Fig. 1. From 10 g of coal obtained from the Kuznetsk deposit, 4.3 g of humic acids (yield: 43%) was extracted. The drying control was performed sequentially on two balances until the mass difference did not exceed 0.001 g. Considering the mass of the bag and the filter, the precipitate mass was determined. To optimize the existing technology for the complete extraction of humic acids from coal, various concentrations (1–4%), temperatures (20–80°C), and extraction durations (30–120 minutes) were investigated. It was established that the most effective separation of sodium humates occurs when extraction is performed using a 4% alkali solution at 80°C. The optimal reaction time was determined to be 120 minutes. A high alkali concentration significantly enhances the extraction efficiency of humic acids. However, applying high temperatures to the raw material leads to a decrease in sodium humate yield (from 4% to 2%) and causes significant alterations in the composition of the resulting products. Specifically, hydrolysis and leaching of carboxyl and, particularly, polysaccharide fragments occur, leading to an increased relative content of aromatic fragments (44–45%). Extending the heating duration beyond two hours does not further enhance the extraction efficiency of humic acids. Thus, the most efficient extraction of sodium humate is achieved using a 4% alkali solution at 80°C with an extraction time of two hours.

Table 1: Average Content of Oxygen (O), Carbon (C), Hydrogen (H), Nitrogen (N), and Sulfur (S) in the brown coal samples and HA derived from them.

Name	Oxygen (O), %	Carbon (C), %	Hydrogen (H), %	Nitrogen (N), %	Sulfur (S), %
Kuz-2024	25,07	56,02	5,085	0	0,155
Ash Kuz-2024	11,56	12,33	0,16	0,43	0
Kum-2024	25,07	50,855	5,294	0,455	0,138
Ash Kum-2024	11,56	11,14	0,136	0,36	0
Humic acid (Kuznetsk)	49,605	3,071	0,93	0,391	45,263
Humic acid (Kumuskuduk)	46,69	3,079	0,63	0,172	48,26

3.2 Analysis of the elemental composition of the coal samples and HA derived from them

The elemental composition analysis provided in order to characterize the average content values of oxygen (O), carbon (C), hydrogen (H), nitrogen (N), and sulfur (S) in the brown coal obtained from the Kuznetsk deposit (sample KUZ-2024) and from the Kumuskuduk deposit (sample KUM-2024) as well as in the ash's residue generated after combustion (sample Ash KUZ-2024 and sample Ash KUM-2024). As shown in Table 1, the brown coal from the Kuznetsk coal deposit exhibits a high carbon content (56.02%), which confirms its significant calorific value, making it suitable for use as a fuel source. The elevated oxygen (25.07%) and hydrogen (5.085%) contents indicate a substantial presence of volatile matter and high reactivity during combustion, enhancing its efficiency as a fuel. The absence of nitrogen in the sample suggests a low risk of nitrogen oxide (NO_x) formation, which contributes to its environmental safety. Additionally, the low sulfur content (0.155%) implies a minimal likelihood of sulfur dioxide (SO₂) emissions; however, monitoring SO₂ emissions during combustion remains crucial to mitigating atmospheric pollution. Meanwhile, as presented in Table 1, the average carbon content was 50.855%, which is characteristic of low-grade brown coal from the Kumuskuduk coal mining. This carbon level indicates a relatively moderate calorific value, as carbon serves as the principal source of energy during combustion. Nevertheless, the significant oxygen content (25.07%) suggests the presence of considerable moisture and oxygenated compounds, which reduce the overall energy efficiency of the coal. Furthermore, the elemental composition of carbon (C), hydrogen (H), nitrogen (N), sulfur (S), and oxygen (O) in humic acids (HA) extracted from brown coals of the Kumuskuduk and Kuznetsk deposits was also analyzed. This analysis aimed to determine the elemental profile of the humic acids, offering valuable information about their chemical characteristics and potential applications in agriculture. The summarized results are shown in Table 1 and Fig. 2.

The humic acid from the Kuznetsk deposit exhibits a higher carbon content (49.605%) compared to that from the Kumuskuduk deposit (46.69%), indicating a greater degree of

aromaticity or polymerization in the organic material of Kuznetsk coals. The hydrogen content is nearly identical in both samples, measuring 3.071% in Kuznetsk and 3.079% in Kumuskuduk, suggesting similar hydrogen-containing structural groups. The nitrogen content differs, with Kuznetsk humic acid containing 0.93% and Kumuskuduk 0.63%, potentially reflecting a higher presence of amino groups or proteins in the Kuznetsk sample. Sulfur content is also higher in the Kuznetsk humic acid (0.391%) than in Kumuskuduk (0.172%), possibly due to an increased concentration of sulfide or organic sulfur compounds. In contrast, Kumuskuduk humic acid has a higher oxygen content (48.26%) compared to Kuznetsk (45.263%), which may suggest a greater abundance of carboxyl groups, contributing to its higher acidity and reactivity.

3.3 Characterization of the functional groups of the coal samples and HA derived from the coal samples

The analysis focused on identifying absorption bands associated with key functional groups, such as carboxyl (-COOH), hydroxyl (-OH), aromatic (C=C), and aliphatic hydrocarbon (C-H) groups. As shown in Fig. 3a, the absorption band in the range of 3800–3600 cm⁻¹ indicates the presence of water associated with the coal surface or hydroxyl groups formed during chemical reactions. In the 2000–1600 cm⁻¹ range, absorptions corresponding to C=O bond vibrations are observed. These may represent carbonyl-containing compounds such as ketones, aldehydes, or carboxyl groups.

Fourier-transform infrared (FTIR) spectroscopy further supports these findings. For instance, strong absorption bands near 1720 cm⁻¹ indicate C=O stretching from carboxylic groups, while broad peaks around 3400 cm⁻¹ correspond to O–H stretching in hydroxyl groups. The intensity and breadth of these peaks were most pronounced in the Kumuskuduk sample, reinforcing its oxygen-rich character. These spectral features corroborate the sample's higher acidity and suggest a robust ability to interact with soil minerals and nutrients, crucial for soil fertility enhancement.

In coals, such bands may arise due to the presence of oxygen-containing functional groups involved in oxidation processes. The band in the 1600-1300 cm⁻¹ region corresponds

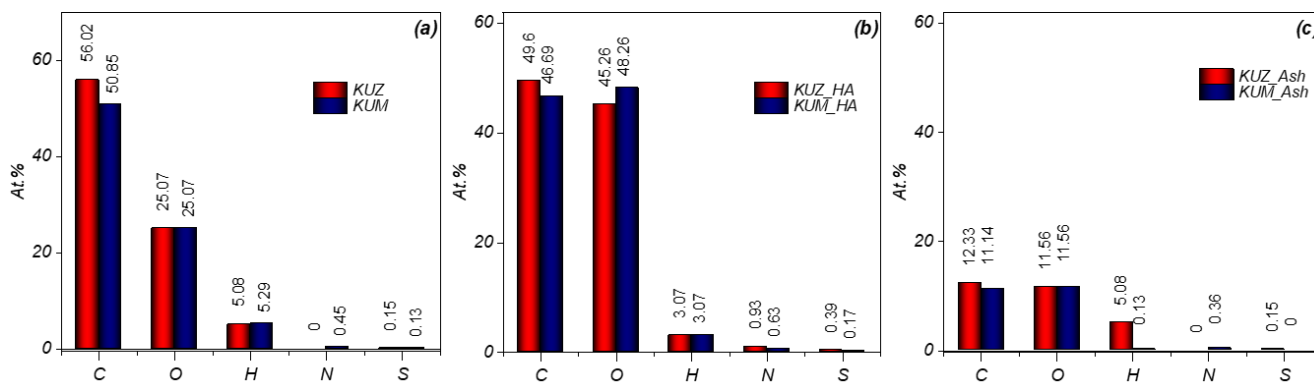


Fig. 2: Average content of oxygen (O), carbon (C), hydrogen (H), nitrogen (N), and sulfur (S) in brown coals (a), humic acids (b) and ashes derived from Kumuskuduk deposit and Kuznetsk deposit (c).

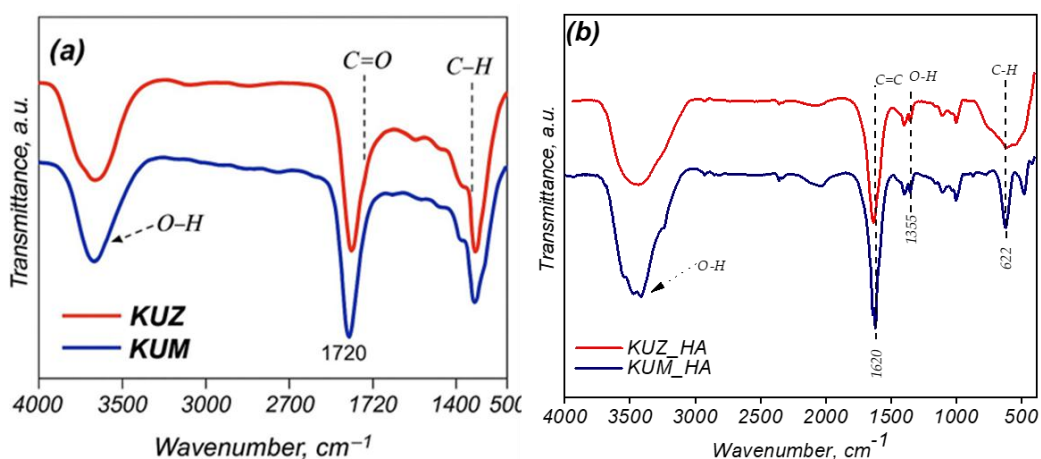


Fig. 3: FTIR spectrum of the samples derived from the Kuznetsk and Kumuskuduk coals (a) and FTIR spectra of the obtained humic acids (b).

to the deformation vibrations of C–H and C=C bonds in deformation vibrations of C–H in aromatic rings and C–O–C aromatic rings (Fig. 3a). Coals contain a significant amount of bonds in esters and ether groups are observed. The low-aromatic structures, and this region can indicate the degree of frequency region (600–500 cm^{-1}) is associated with out-of-aromaticity of the carbon skeleton. The more intense the bands plane C–H vibrations in aromatic systems and deformation in this region, the higher the content of aromatic carbon vibrations of C–C bonds in carbon chains. Key absorption compounds. The 1000–750 cm^{-1} range may indicate the presence of substituted benzene rings (aromatic hydrocarbons) and in-plane C–H vibrations of the ring. Additionally, Kumuskuduk sample showing the strongest intensity at vibrations of carbon-oxygen bonds in structures such as ethers or alcohols may also appear in this region, provided these compounds are present in the coal.

Fig. 3b illustrates an absorption band within the range of 3800–3550 cm^{-1} , associated with O–H group vibrations. These bands are characteristic of hydroxyl groups, present both in free water and hydrogen bonds within the structure of humic acids. Absorption bands in the range of 2100–1600 cm^{-1} correspond to carboxylic acids, aldehydes, ketones, and esters.^[29] As illustrated in the Fig. 3b, the range of 1600–1250 cm^{-1} is attributed to aromatic C=C bond vibrations, reflecting the presence of aromatic rings in humic acid structures. Additionally, this range may include deformation vibrations of C–O bonds in carboxyl and phenolic groups, indicative of complex oxygen-containing functional groups in humic acids. In the range of 1000–600 cm^{-1} , absorption bands related to

3.4 Differential scanning calorimetry (TGA)/DSC analysis of the samples of coals and HA

As illustrated in Fig. 4a, the initial phase of mass loss, occurring between 100–150°C, is attributed to moisture evaporation. The subsequent phase, observed between 200–400°C, corresponds to the release of volatile components, including low-molecular-weight hydrocarbons and various organic compounds. The third phase, extending from 400–600°C, is associated with the decomposition of the coal's primary organic matrix, involving the breakdown of complex macromolecules such as humic substances and hydrocarbons. The residual mass remaining between 600–800°C represents the mineral content (ash), as further confirmed by the residue observed in the TG analysis.

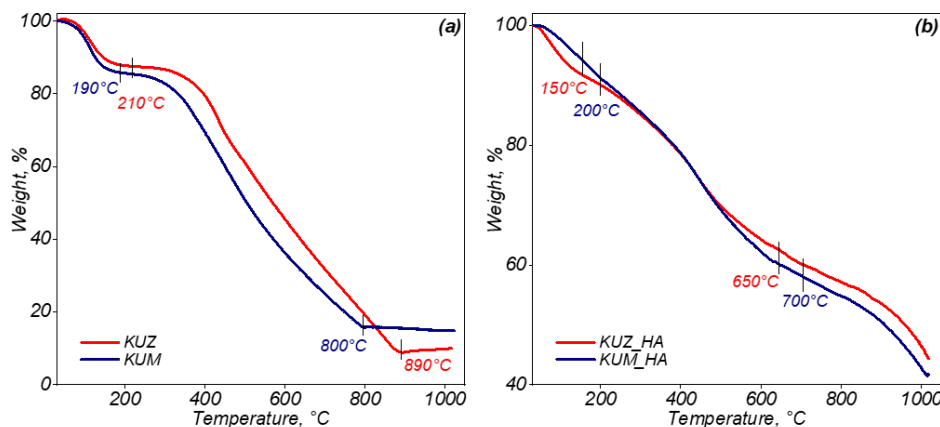


Fig. 4: TGA curves of the coal (a) and humic acids (b) samples derived from the Kuznetsk and Kumuskuduk deposits.

Thermogravimetric analysis curves of humic acids reveal a minor mass loss in the temperature range of 30–175°C, likely attributed to residual water. The sample decomposition occurs in two distinct stages as illustrated in the Fig. 4b. The first stage, between 200–500°C, involves the breakdown of volatile compounds and functional groups such as carboxyl, phenolic, and methoxyl groups. Additionally, small organic molecules associated with aromatic structures decompose, resulting in significant mass loss. This stage is characterized by the release of water, carbon dioxide, and other low-molecular-weight gases. In the second stage, from 550–1000°C, more stable aromatic structures and condensed polycyclic systems undergo decomposition. This leads to further mass loss and the formation of carbonaceous residues. The main processes during this stage include carbonization (conversion of organic matter into carbon) and the formation of a stable residue resembling coke. These stages reflect the sequential thermal decomposition of humic acids, progressing from light functional groups to stable carbon structures.

The thermogravimetric (TG) and differential scanning calorimetry (DSC) analyses provide valuable insights into the thermal stability, decomposition characteristics, and energy release behavior of coal and humic acids from the Kuznetsk and Kumuskuduk deposits.

As depicted in the Fig. 5a, b, the TG curves for coal samples exhibit a multi-step mass loss process, reflecting the sequential degradation of volatile components, fixed carbon, and inorganic residues. The initial weight loss observed at lower temperatures (~100–200°C) is attributed to the evaporation of moisture and weakly bound volatiles. The most significant decomposition occurs between 300°C and 600°C,

corresponding to the release of volatile matter and the thermal breakdown of organic macromolecules, such as aliphatic and aromatic structures. The DSC curves of the coal samples show prominent endothermic and exothermic peaks. The major exothermic peak around 400–500°C is associated with the combustion of volatile compounds and oxidation of carbonaceous material, indicating the high energy release during combustion. The differences in peak intensities between the Kuznetsk and Kumuskuduk coal samples suggest variations in their composition, possibly due to differences in carbon content, volatile matter, and mineral impurities.

Compared to coal, the humic acid samples exhibit distinct thermal behavior with relatively lower decomposition temperatures, according to the results shown in Fig. 5c, d. The initial weight loss in the TG curves (below 150°C) corresponds to the evaporation of residual water and physically adsorbed molecules, a common characteristic of humic substances due to their hydrophilic nature. The major decomposition occurs between 200°C and 450°C, associated with the breakdown of oxygen-containing functional groups, such as carboxyl (-COOH) and hydroxyl (-OH) groups, as well as the degradation of aliphatic and polysaccharide components.

The DSC profiles of the humic acid samples reveal multiple endothermic and exothermic events. The exothermic peaks in the range of 250–450°C indicate the oxidation of organic components, while a more gradual heat flow suggests a less pronounced combustion process compared to coal. The lower exothermic peak intensity in humic acids compared to coal samples suggests a lower energy release, which is consistent with their lower fixed carbon content and higher oxygen functionalities.

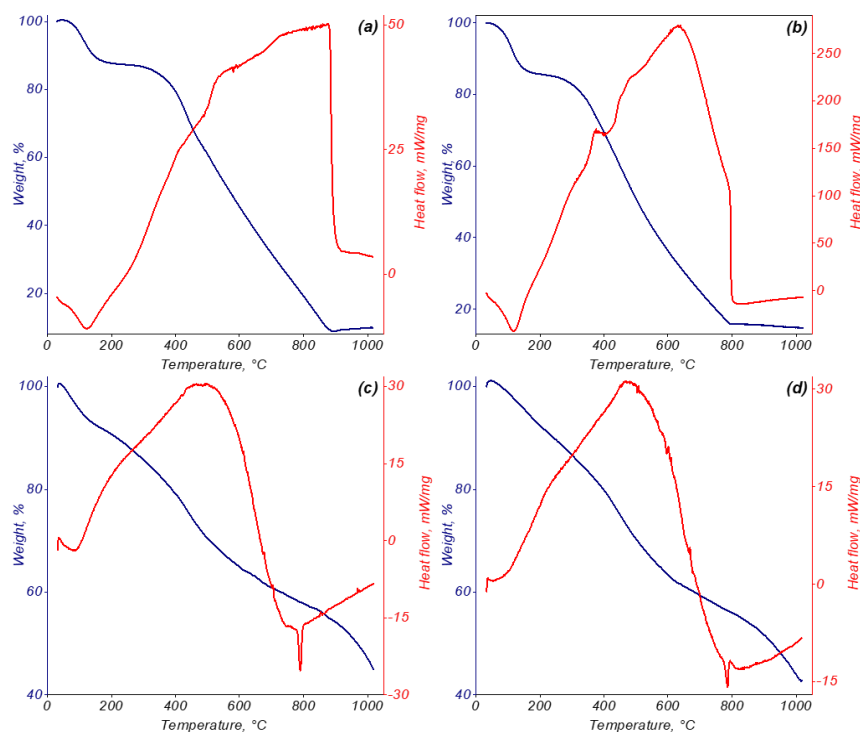


Fig. 5: Results of TG/DSC analyses of the coal (a, b) and humic acids (c, d) samples derived from the Kuznetsk and Kumuskuduk deposits.

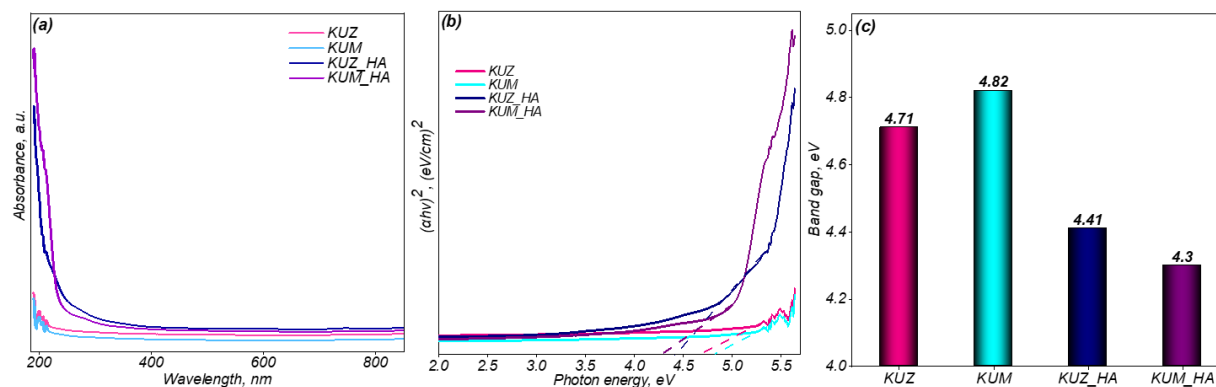


Fig. 6: UV-vis spectra (a), Tauc's plot (b) and band gap (c) of the samples derived from the Kuznetsk and Kumuskuduk coals and obtained humic acids.

A comparison between the two deposits reveals differences in thermal stability and combustion characteristics. The Kuznetsk coal sample demonstrates a more significant mass loss at higher temperatures and a sharper exothermic peak, indicative of a higher fixed carbon content and a greater energy yield upon combustion. In contrast, the Kumuskuduk coal sample appears to undergo decomposition at slightly lower temperatures, suggesting a higher volatile matter content and increased reactivity. For humic acids, the TG/DSC profiles from both deposits exhibit similar trends, but the differences in thermal events indicate slight variations in their structural composition. The Kuznetsk humic acids show a broader decomposition range, suggesting a more complex molecular structure with diverse oxygen-containing functional groups. Meanwhile, the Kumuskuduk humic acids appear to degrade more rapidly, indicating a higher content of thermally labile components.

3.5 Performance analysis of the UV-Vis spectroscopy and band gap analysis of the samples

The results of UV-VIS and band gap characterization of the coal and HA samples are shown in Fig. 6. The UV-VIS spectroscopy and Tauc's plot analyses provide insight into the electronic transitions, optical properties, and band gap energies of the coal and humic acid samples derived from the Kuznetsk and Kumuskuduk deposits. These results are essential for understanding the molecular structure, aromaticity, and potential applications of the studied materials.

Higher absorbance in the 260–280 nm region indicates greater aromaticity in Kumuskuduk HA. Tauc plots show decreasing band gap energies with increasing structural complexity.

As we can see from the Fig. 6a, the UV-Vis spectra of the samples exhibit characteristic absorption features, which can be attributed to the presence of conjugated π -electron systems and various chromophoric groups. The coal samples show broad absorption bands extending from the UV to the visible range, indicating a complex mixture of aromatic and aliphatic structures. The strong absorption at lower wavelengths (below 300 nm) is associated with π - π^* electronic transitions in aromatic rings, while the shoulder in the visible region

suggests the presence of n - π^* transitions involving oxygen-containing functional groups. The humic acid samples display distinct absorption characteristics compared to the coal samples. The spectra of humic acids typically show a monotonic decrease in absorbance with increasing wavelength, a behavior attributed to their polydisperse macromolecular structure and high content of oxygen functional groups. The higher absorbance in the UV region suggests the presence of extensive conjugated systems, likely due to the breakdown of larger aromatic clusters during humification.

The Tauc's plot is used to determine the optical band gap (E_g) of the materials by extrapolating the linear region of the $(\alpha h\nu)^2$ versus photon energy ($h\nu$) curve, as shown in the Fig. 6b. The band gap values provide insight into the electronic structure and potential applications of the materials in photocatalysis, optoelectronics, and environmental remediation.

The band gap energies of the coal-derived samples and the extracted humic acids were determined using UV-Vis diffuse reflectance spectroscopy and analyzed using Tauc's method. The Tauc Eq. 2, expressed as:

$$(\alpha h\nu)^n = A(h\nu - E_g) \quad (2)$$

was employed, where α is the absorption coefficient, h is Planck's constant, ν is the photon frequency, A is a proportionality constant, and E_g is the optical band gap energy. The exponent n depends on the nature of the electronic transition, with $n=2$ for direct allowed transitions and $n=1/2$ for indirect allowed transitions. To estimate the band gap, Tauc's plots $(\alpha h\nu)^n$ vs. $h\nu$ were generated, and the linear portion of the curve was extrapolated to the x-axis, where $(\alpha h\nu)^n$ approaches zero. The intersection of this extrapolation with the energy axis (photon energy, $h\nu$) provides the band gap value. The results (Figure 6c) indicate variations in the band gap energies between the raw coal samples and their extracted humic acids, suggesting structural and electronic modifications during the extraction process. The lower band gap values observed for humic acids in comparison to coal suggest increased electronic delocalization due to aromatic and conjugated systems.

The UV-Vis absorption spectra provided insights into the

degree of aromaticity and conjugation within the HAs. The Kumuskuduk HA exhibited a more intense absorption in the 260–280 nm range, indicative of greater aromatic content. The E4/E6 ratio, a classical index derived from UV-Vis spectra, showed lower values for this sample, consistent with higher molecular complexity and condensation. These optical properties not only reflect the structural maturity of the HA but also suggest its stronger interaction with light and metal ions, relevant for environmental remediation applications such as heavy metal binding. Band gap analysis using the Tauc plot revealed values in the range of 2.1 to 2.6 eV across the samples, with Kumuskuduk HA showing a slightly narrower band gap. In the context of HA applications, this band gap is related to the material's semiconducting properties, influencing its electron-donating and -accepting behavior. A narrower band gap implies higher electronic conductivity, which could enhance redox activity in soil systems. This makes such HAs suitable for advanced roles, such as in biochar-enhanced soils or as components in environmental electrochemical sensors and bioremediation systems.

A comparison between the Kuznetsk and Kumuskuduk samples reveals notable differences in their optical properties. The Kuznetsk coal sample exhibits a slightly lower band gap than the Kumuskuduk coal, suggesting a higher degree of aromatic condensation and a more extended conjugation system. In contrast, the Kumuskuduk humic acids demonstrate lower band gap values compared to the Kuznetsk humic acids, indicating structural variations in their molecular composition. These differences can be attributed to the geological conditions and the degree of coalification in the respective deposits. The Kumuskuduk deposit appears to have undergone more extensive carbonization, resulting in a structure with increased aromaticity and lower band gap energy. Conversely, the Kuznetsk samples exhibit higher band gaps, indicating a more

heterogeneous molecular composition with a greater presence of oxygen-functionalized moieties.

3.6 Study of the structure and particle morphology of coal samples and HA using microscopy to assess structural characteristics

The surface morphology of coal from the Kuznetsk deposit is characterized by the presence of fine-dispersed pores and particles on the nanometer scale (Fig. 7a, b). Small pores with diameters of approximately 5 nanometers are observed on the coal surface, indicating its microporous structure. These pores play a crucial role in the adsorption properties of coal, as they efficiently capture and retain molecules of various substances, including gases and liquids. Additionally, individual particles about 1 nanometer in size are visible on the coal surface. These particles may represent fragments of organic matter preserved during coal formation or the products of interactions between coal and minerals. Nanoparticles of this size can also influence the coal's reactivity.

The morphology of humic acids derived from Kuznetsk deposit coals exhibits a complex and heterogeneous structure, characterized by formations resembling petal-like shapes, as characterized in the Fig. 7c, d. These petal-like structures are composed of thin, elongated plate-like elements that overlap to form a layered and porous texture. This morphological feature provides a large specific surface area, enhancing the sorption and reactive properties of humic acids. Such formations may result from the specific molecular organization of humic substances, driven by their high content of aromatic and carboxyl groups. Structural heterogeneity is further evidenced by the presence of micropores, and pores of varying sizes distributed across the surface of the humic acids. These pores typically facilitate the adsorption of various ions and molecules, making humic acids effective natural sorbents.

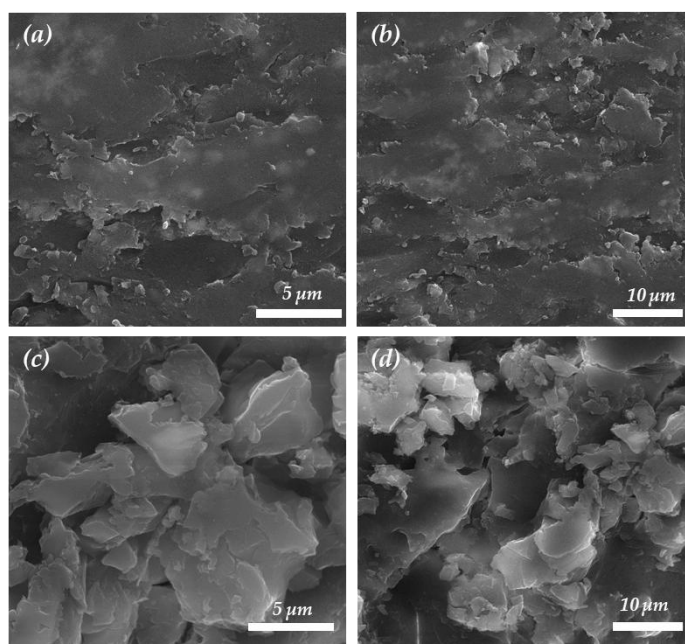


Fig. 7: SEM images of the surface morphology of coals (a, b) and obtained humic acids (c, d) from the Kuznetsk deposit.

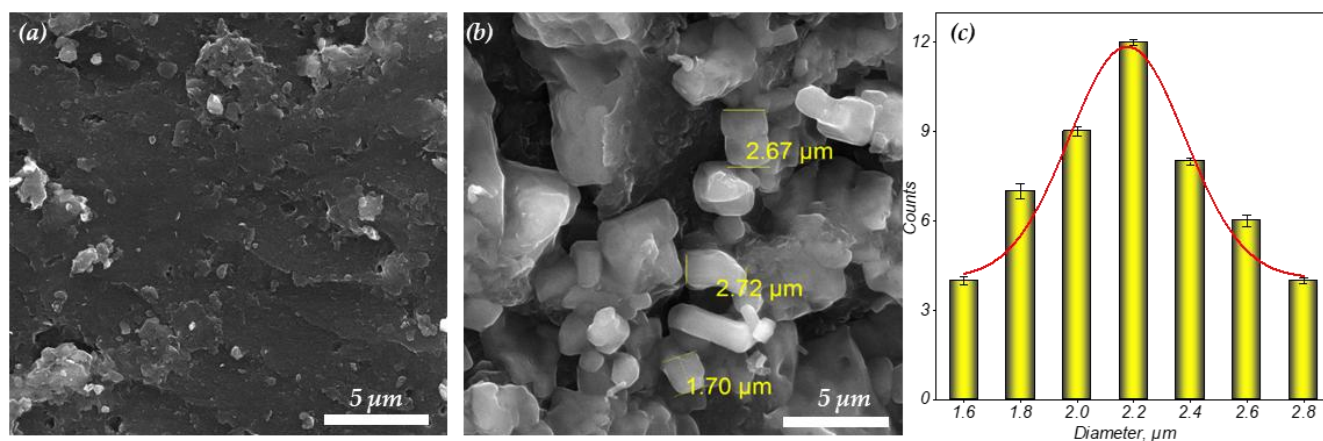


Fig. 8: SEM images of the surface morphology of coals (a), obtained humic acids (b) from the Kumuskuduk deposit and corresponding size distributions (c).

The observed changes in morphology confirm that the extraction process effectively disrupts the coal's structural integrity, leading to the release of humic substances with increased surface area and reactivity. The enhanced porosity and irregular surface of humic acids are expected to improve their interaction with water and other chemical species, making them more suitable for applications in soil amendment, adsorption, and catalysis.

As illustrated in Fig. 8a, captured using a scanning electron microscope, the structure of the Kumuskuduk brown coal deposit is depicted. Typically, brown coal has a complex porous structure, visible as grains and fractures with varying degrees of smoothness and roughness. The SEM images reveal the presence of micropores, ranging in size from a few nanometers to several micrometers. This porous texture is crucial for understanding the coal's adsorption properties and behavior during combustion, as the pores can act as channels for the movement of gases and liquids. The SEM images also show coal particle aggregates formed through processes such as compaction or gasification. The shape and size of these aggregates can vary, influencing the coal's technological properties.

The SEM images provide a comparative analysis of the surface morphology of the Kumuskuduk coal sample (Fig. 8a) and the extracted humic acids (Fig. 8b).

The coal structure appears relatively compact, with a smooth and dense morphology. The presence of few visible pores and fractures suggests a highly cross-linked and polymerized carbonaceous matrix. This compact nature is characteristic of lignite and sub-bituminous coals, where the organic matter is densely packed, resulting in limited accessibility for chemical interactions. The low porosity of the coal matrix likely contributes to its lower reactivity in alkaline extraction processes. In contrast, the extracted humic acids exhibit a significantly more fragmented and porous morphology. The SEM image shows the presence of irregularly shaped, loose, and disaggregated particles, indicating successful breakdown of the coal's dense structure. The measured particle sizes range from approximately 1.70

μm to 2.72 μm, confirming the substantial reduction in particle dimensions compared to the original coal matrix. The increased porosity and surface roughness are indicative of a higher degree of oxidation and functionalization, leading to improved solubility and reactivity in aqueous environments.

The observed differences confirm that alkaline extraction effectively disrupts the original coal matrix, resulting in humic acid particles with enhanced surface area, which is beneficial for applications in agriculture, adsorption, and environmental remediation.

The particle size distribution analysis, as depicted in the Fig. 8c, provides additional insights into the dispersion and aggregation characteristics of the obtained humic acids:

- 1) The particle size distribution follows a Gaussian-like trend, indicating a relatively uniform size reduction during the extraction process.
- 2) The majority of humic acid particles fall within the submicron to low-micron range (0.7–2.67 μm), confirming effective fragmentation.
- 3) The presence of larger particles suggests partial aggregation, which may be due to intermolecular interactions between humic substances through hydrogen bonding and van der Waals forces.

The reduction in particle size enhances the dispersibility and reactivity of humic acids, making them more suitable for applications such as soil amendments, water purification, and controlled-release fertilizers.

3.7 Study of acid-base properties of humic acids

Our results showed that the pH of humic acid aqueous solutions ranged from 4.2 to 5.5, indicating a weakly acidic medium. Notably, humic acids extracted from Kuznetsk coals exhibited significantly higher titratable acidity compared to those from the Kumuskuduk deposit, due to the higher content of carboxylic groups. The -COOH/-OH ratio, derived from quantitative analysis, ranged from 1.4 to 2.1, suggesting a predominance of acidic fragments in the structure. These characteristics determine the strong buffering capacity of humic acids, their interaction with soil minerals, and their role

Table 2: Comparison of Kumuskuduk HA with Literature and Commercial HA Samples.

Parameter	Kumuskuduk HA (This study)	Leonardite HA (Commercial)	Peat-derived HA	Low-rank Coal HA (China)
C (%)	~ 54-56	58-62	52-55	55-60
O (%)	~ 36-38	30-35	38-42	32-36
H/C ratio	~ 1.0	0.7-0.9	1.2-1.5	0.8-1.1
O/C ratio	~ 0,5-0,6	0,4-0,5	0,6-0,7	0,4-0,5
FTIR (C=O, ~ 1720 cm ⁻¹)	Strong	Moderate	Weak-Moderate	Moderate
FTIR (O=H, ~ 3400 cm ⁻¹)	Broad, strong	Moderate	Strong	Moderate
FTIR (C=C, ~ 1600 cm ⁻¹)	Present	Present	Present	Present
E4/E6 ratio	Low (~3-4)	Moderate (~5-6)	High (~7-9)	Moderate (~5)
Thermal stability (°C)	> 500 (major mass loss after 400°C)	~ 300-500	~ 200-450	~ 250-500
Notes	High acidity and oxidation; suitable for soil amendment and environmental application	Widely used in agriculture; less oxygenated	Less stable; more aliphatic	Variable properties; moderate performance

in the formation of soil colloids.

3.8 Characterization comparison of humic acids from various coal and commercial sources based on elemental composition, functional group profiles, and thermal behavior

To further emphasize the novelty and practical significance of the humic acids (HAs) studied, especially the sample from the Kumuskuduk deposit, a comparative evaluation was conducted against literature-reported and commercially available HAs. Humic substances can vary widely in their chemical composition and structural features depending on their origin. Comparing key parameters—such as elemental composition, functional groups, and thermal stability—offers critical insight into their performance in various environmental and agricultural applications. This contextual comparison helps validate the distinctive properties of the Kumuskuduk HA and supports its potential use as a high-quality soil amendment or multifunctional organic material.

Table 2 highlights the elemental composition, key functional groups (based on FTIR), and thermal stability of the Kumuskuduk HA sample from your study alongside humic acids from other coal sources or commercial products, based on commonly reported literature values.

Table 2 presents a comparative summary of the elemental composition, key functional group indicators (based on FTIR spectra), and thermal stability of the Kumuskuduk humic acid (HA) sample against literature-reported HAs derived from various coal and organic sources, including commercial Leonardite products, peat, and low-rank Chinese coal. The Kumuskuduk HA stands out due to its higher oxygen content and lower C/O ratio, indicating a higher degree of oxidation and abundance of acidic functional groups such as carboxylic and phenolic moieties. These characteristics are further confirmed by strong FTIR bands near 1720 cm⁻¹ and 3400 cm⁻¹, associated with C=O and O–H stretching vibrations, respectively.

Compared to Leonardite and peat derived HAs, the Kumuskuduk HA exhibits a lower E4/E6 ratio, suggesting a higher molecular weight and greater aromatic condensation. Its thermal stability also exceeds that of several reference materials, with significant decomposition occurring above 400 °C, which implies higher structural resilience. Collectively, these features highlight the superior potential of Kumuskuduk HA for applications in soil conditioning, environmental remediation, and possibly even in composite material formulations due to its robustness and functional diversity.

4. Conclusion

The comprehensive investigation of humic acids derived from brown coal, as well as the brown coals of the Kuznetsk and Kumuskuduk deposits, provided valuable insights into their structural, thermal, and morphological characteristics. Utilizing advanced analytical techniques such as IR spectroscopy, elemental analysis, TGA/DSC characterization, The UV-Vis and band gap analyses, and SEM analyses as well, the study highlighted significant differences and similarities in the properties of these materials.

The properties of humic acids are largely determined by the geological origin of the carbonaceous source material. This study focuses on two coal deposits in Kazakhstan—Kumuskuduk and the Kuznetsk open-pit mine—which differ in age, coalification degree, and lithological features. The Kumuskuduk deposit, located in southern Kazakhstan, contains Neogene brown coals with a low degree of metamorphism. These coals were formed in swampy and lacustrine environments, leading to the accumulation of organic matter rich in humic fractions. The sedimentary rocks are predominantly fine-grained with clay components. The resulting humic acids are characterized by a loose structure, high solubility, and colloidal stability. The Kuznetsk open-pit mine, situated in the Karaganda region, contains Carboniferous coals with a higher metamorphic grade. Formed during earlier geological epochs, these coals yield humic acids

with more condensed aromatic structures and a higher content of oxygen-containing functional groups, particularly carboxylic ones. As a result, they exhibit stronger acid-base behavior, enhanced sorption activity, and chelating capacity.

Thus, geological differences, including age and coalification degree, account for the structural and functional variability of humic acids, which is critical for their applications in agronomy and environmental technologies.^[30-32]

Infrared spectroscopy revealed the presence of key functional groups, including hydroxyl, carboxyl, phenolic, and aromatic structures, which are crucial for their sorption and reactive properties. Elemental analysis confirmed the high carbon content and the presence of oxygen-containing functional groups, indicative of the complex molecular structure of humic acids and coal. Thermal analysis through TGA and DSC demonstrated the distinct decomposition stages of volatile compounds, functional groups, and stable aromatic structures, emphasizing the thermal stability and carbonization potential of these materials. The SEM analysis elucidated the surface morphologies, with Kuznetsk humic acids exhibiting petal-like structures with high specific surface areas and Kumuskuduk humic acids displaying granular and porous textures with larger particle sizes. The UV-Vis and band gap analyses reveal distinct optical and electronic characteristics of the coal and humic acid samples. The lower band gap in coal samples suggests their potential utility in applications requiring conductive and semiconducting materials, while the higher band gap in humic acids highlights their potential in environmental and chemical applications. The observed differences between the Kuznetsk and Kumuskuduk samples emphasize the influence of geological formation on the optical properties of coal and humic substances.

The structural and chemical characteristics of humic acids extracted from coals of the Kuznetsk and Kumuskuduk deposits exhibit substantial differences due to lithological-stratigraphic and geochronological features of the original organogenic material, as well as the degree of coalification. The observed variations in the content of oxygen-containing functional groups—particularly carboxyl and phenolic groups—are directly reflected in the acid-base properties of the humic acids, including titratable acidity and the pH of aqueous extracts. Nevertheless, the issue of functional activity remains equally important. This includes sorption capacity, complexation ability, antioxidant properties, as well as water solubility and colloidal stability—parameters that are closely correlated with the molecular composition and spatial organization of humic macromolecules. However, the present study focused primarily on structural-analytical indicators. It is therefore advisable to extend the scope of future research by including correlation analyses that would quantitatively relate FTIR spectra, thermogravimetric data, and elemental composition to functional performance. Special attention should be paid to the water solubility and colloidal stability of humic acids, as these are critical factors determining their behavior in agroecosystems, soil solutions, and natural water

bodies. Expanding research in these directions is promising for achieving a comprehensive evaluation of the applied potential of humic substances in agriculture and environmental protection.

These findings underscore the potential applications of humic acids and brown coals in environmental remediation, agriculture, and energy sectors. The high sorption capacity, reactivity, and thermal stability make them suitable for roles such as natural sorbents, soil conditioners, and carbon precursors. The observed structural and compositional variations between deposits suggest that the specific applications of these materials can be optimized based on their unique properties. Further studies focusing on their interaction mechanisms, environmental impacts, and scalability are recommended to enhance their practical utility in diverse fields.

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Data Availability Statement

The original contributions presented in the study are included in the article, further inquiries can be directed to the corresponding authors.

Conflicts of Interest

The authors declare no conflict of interest.

Supporting Information

Not applicable

Abbreviations

The following abbreviations are used in this manuscript:

KUM	Kumuskuduk
KUZ	Kuznetsk
HA	humic acid
IR	infrared spectrum
SEM	scanning electron microscope
TGA	thermogravimetric analysis

CRedit Statement

Ainash Nauanova and **Tolkyn Khamitova:** Conceptualization. **Nursanat Parmanbek:** Writing - Original draft preparation, Writing - Review and editing, Methodology, Investigation. **Tolkyn Khamitova:** Software and visualization. **Ma Feng Yun, Sairagul Tyanakh** and **Nursanat Parmanbek:** Validation. **Sairagul Tyanakh:** Formal analysis. **Tolkyn Khamitova:** Resources. **Ma Feng Yun:** Data curation. **Ainash Nauanova:** Supervision, Project administration, Funding acquisition. All authors have read and agreed to the published version of the manuscript.

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