ISSN: 2224-5286 (Print)
ISSN: 2518-1491 (Online)

ACADEMIC SCIENTIFIC JOURNAL OF CHEMISTRY

№1
2026

ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)

CENTRAL ASIAN ACADEMIC
RESEARCH CENTER

ACADEMIC SCIENTIFIC JOURNAL OF CHEMISTRY

1 (466)

JANUARY – MARCH 2026

PUBLISHED SINCE JANUARY 1947
PUBLISHED 4 TIMES A YEAR

ALMATY

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ACADEMIC SCIENTIFIC JOURNAL OF CHEMISTRY
ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)

Owner: «Central Asian Academic Research Center» LLP (Almaty).

The certificate of registration of a periodical printed publication in the Committee of information of the Ministry of Information and Social Development of the Republic of Kazakhstan **№ KZ23VPY00121156,**
issued 05.06.2025

Thematic scope: *organic chemistry, inorganic chemistry, catalysis, electrochemistry and corrosion, pharmaceutical chemistry and technology.*

Periodicity: 4 times a year.

http://chemistry-technology.kz/index.php/en/arhiv

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«ACADEMIC SCIENTIFIC JOURNAL OF CHEMISTRY».
ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)
Собственник: Республиканское общественное объединение ТОО «Центрально-азиатский академический научный центр» (г. Алматы).
Свидетельство о постановке на учет периодического печатного издания в Комитете информации Министерства информации и общественного развития Республики Казахстан **№ KZ23VPY00121156,** выданное 05.06.2025 г.
Тематическая направленность: *органическая химия, неорганическая химия, катализ, электрохимия и коррозия, фармацевтическая химия и технологии.*
Периодичность: 4 раз в год.
http://chemistry-technology.kz/index.php/en/arhiv
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«ACADEMIC SCIENTIFIC JOURNAL OF CHEMISTRY»
ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)
Меншіктенуші: «Орталық Азия академиялық ғылыми орталығы» ЖШС (Алматы қ.).
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Тақырыптық бағыты: *органикалық химия, бейорганикалық химия, катализ, электрохимия және коррозия, фармацевтикалық химия және технологиялар.*
Мерзімділігі: жылына 4 рет.
http://chemistry-technology.kz/index.php/en/arhiv

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ACADEMIC SCIENTIFIC JOURNAL OF CHEMISTRY
ISSN 2224–5286
Volume 1.
Number 466 (2026), 323–337

https://doi.org/10.32014/2026.2518-1491.358

IRSTI 31.19.29
UDC 543.6, 544.63

© Uali A.[1*], Omirzak U.[1], Titanov A.[1], Abilkanova F.[2], Kunarbekova M.[3], 2026.
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WASTE BIOMASS-DERIVED FE-MODIFIED BIOCHAR: STRUCTURE AND APPLICATION IN POTENTIOMETRIC ANALYSIS

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Abstract. This study investigates a sustainable method for producing iron-modified biochar from wheat waste to fabricate a carbon-paste electrode for detecting iron in aqueous solutions. The focus lies on the dual objectives of waste utilisation and monitoring of heavy metal content in aqueous solutions. The biochar is synthesised via carbonisation, chemical modification with $FeCl_3$ and subsequent washing, and characterised using CHNS, FTIR, XRD, and SEM-EDS analyses, which reveal a carbon content of 76.46%. Furthermore, BET analysis indicates a specific surface area of 296.43 $m^2 \cdot g^{-1}$, with the pore distribution predominantly composed of micropores (66.98%) and mesopores (30.57%), which suggests promising textural properties. In this study, the synthesised biochar is used as an electroactive material in a carbon-paste electrode, enabling the measurement of Fe^{3+} content from $1.0 \cdot 10^{-6}$ mol·L^{-1} to $1.0 \cdot 10^{-1}$ mol·L^{-1} in aqueous solutions. Also, the relationship between electrode potential and the pH of the analysed solution is explored, demonstrating a stable electrode signal within the pH range of 3 to 6, along with temperature, both of which are crucial operational parameters for optimal performance. The selectivity tests have demonstrated that the electrode's

selectivity is limited by the type of interfering ions and the analyte concentration. The electrode testing in direct potentiometry for detecting Fe^{3+} ions (n=3) results in $5.00 \cdot 10^{-3}$ mol·L^{-1} (added), $4.43 \cdot 10^{-3}$ mol·$L^{-1} \pm 3.61 \cdot 10^{-4}$ mol·L^{-1} (found), absolute error $5.71 \cdot 10^{-4}$ mol·L^{-1}, relative error 11.42%. The evaluation of the analytical method using the AGREE metric indicates that the proposed approach is environmentally friendly. The findings imply that this technique has potential for sustainable analytical applications.

Keywords: waste wheat, Fe-modified biochar, carbon-paste electrode, potentiometry, greenness evaluation

For citations: Uali A., Omirzak U., Titanov A., Abilkanova F., Kunarbekova M. Waste biomass-derived fe-modified biochar: structure and application in potentiometric analysis. Academic Scientific Journal of Chemistry, 2026. — No.1. – P. 323–337. DOI: https://doi.org/10.32014/2026.2518-1491.358

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ҚАЛДЫҚ БИОМАССАДАН АЛЫНҒАН ТЕМІРМЕН ТҮРЛЕНДІРІЛГЕН БИОКӨМІР: ҚҰРЫЛЫМЫ ЖӘНЕ ПОТЕНЦИОМЕТРИЯЛЫҚ ТАЛДАУДА ҚОЛДАНЫЛУЫ

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Аннотация: Бұл жұмыс бидай қалдықтарынан темірмен түрлендірілген биокөмір алудың тұрақты әдісін зерттеуге, оны одан әрі су ерітінділеріндегі темірді анықтау мақсатында көміртекті-пасталы электродын жобалауға бағытталған. Негізгі назар екі жақты мақсатты көздейді: қалдықтарды утилизациялау және сулы ерітінділердегі ауыр металдардың құрамын бақылау. Биокөмір карбонизациялау, $FeCl_3$-мен химиялық түрлендіру және шаю процестері арқылы синтезделіп, CHNS, FTIR, XRD және SEM-EDS талданып, көміртек мөлшері 76,46% құрайтыны анықталды. Одан әрі BET талдау үлгінің меншікті бетінің ауданы - 296,43 $м^2 \cdot г^{-1}$,

ал кеуектер негізінен микро- (66,98%) және мезокеуектерден (30,57%) тұратынын көрсетті; бұл материалдың құрылыым құрделі дамыған екендігін білдіреді. Одан әрі осы жұмыста алынған биокөмір көміртекті-пасталы электродтың электроактивті материалы ретінде қолданылып, оның көмегімен сулы ерітінділер құрамындағы Fe^{3+} иондарының концентрациясын $1{,}0\cdot10^{-6}$ моль·л$^{-1}$-ден $1{,}0\cdot10^{-1}$ моль·л$^{-1}$ аралығында анықтау мүмкіндігі зерттелді. Сондай-ақ, электрод потенциалы мен аналит ерітіндінің pH мәні арасындағы байланыс қарастырылып, pH 3-тен 6-ға дейінгі аралықта электродтың аналитикалық сигналы тұрақты екені анықталды. pH-пен қатар, температура да электродың оңтайлы жұмысқа әсер ететін маңызды параметр екені көрсетілді. Селективтілік бойынша тестілер электродтың селективтілігі кедергі жасайтын иондардың түрімен және талданатын заттың концентрациясымен шектелетінін көрсетті. Fe^{3+} иондарын (n=3) анықтау үшін тікелей потенциометриядағы электрод сынақ нәтижелері $5{,}00\cdot10^{-3}$ моль·л$^{-1}$ (қосылған), $4{,}43\cdot10^{-3}$ моль·л$^{-1}\pm3{,}61\cdot10^{-4}$ моль·л$^{-1}$ (табылған), абсолютті қателік $5{,}71\cdot10^{-4}$ моль·л$^{-1}$, салыстырмалы қателік 11,42% көрсетті. AGREE метрикасына негізделген әдіс арқылы ұсынылған тәсіл экологиялық тұрғыдан қауіпсіз деп сипатталды. Осылайша, зерттеу нәтижелері бұл тәсілдің тұрақты аналитикалық қолданыс аясында потенциалы бар екенін көрсетті.

Түйін сөздер: бидай қалдықтары, темірмен түрлендірілген биокөмір, көміртекті-пасталы электрод, потенциометрия, экологиялық бағалау

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БИОУГОЛЬ, МОДИФИЦИРОВАННЫЙ ЖЕЛЕЗОМ, ИЗ ОТХОДОВ БИОМАССЫ: СТРУКТУРА И ПРИМЕНЕНИЕ В ПОТЕНЦИОМЕТРИЧЕСКОМ АНАЛИЗЕ

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Аннотация: В данном исследовании рассматривается устойчивый метод получения биоугля, модифицированного железом, из отходов пшеницы с целью его использования при создании угольно-пастового электрода для определения ионов железа в водных растворах. Основное внимание уделено двум аспектам: утилизации биомассы и мониторингу содержания тяжёлых металлов в водных средах. Биоуголь получали методом карбонизации с последующей химической модификацией с использованием $FeCl_3$ и промывкой. Полученные материалы были охарактеризованы методами CHNS-анализа, FTIR, XRD и SEM-EDS, которые показали содержание углерода на уровне 76,46%. Анализ BET выявил удельную поверхность 296,43 $м^2 \cdot г^{-1}$, при этом пористая структура представлена преимущественно микропорами (66,98%) и мезопорами (30,57%), что свидетельствует о благоприятных текстурных характеристиках. Синтезированный биоуголь использован в качестве электроактивного материала в угольно-пастовом электроде, способного определять концентрацию ионов Fe^{3+} в диапазоне от $1{,}0 \cdot 10^{-6}$ до $1{,}0 \cdot 10^{-1}$ моль·$л^{-1}$. Исследовано влияние pH раствора и температуры на потенциал электрода. Установлено, что стабильный отклик наблюдается в диапазоне pH 3–6, что является оптимальным для аналитических измерений. Проведённые исследования селективности показали, что точность определения зависит от природы мешающих ионов и концентрации аналита. Результаты потенциометрического анализа (n = 3) составили: $5{,}00 \cdot 10^{-3}$ моль·$л^{-1}$ (введено), $4{,}43 \cdot 10^{-3} \pm 3{,}61 \cdot 10^{-4}$ моль·$л^{-1}$ (найдено), абсолютная погрешность - $5{,}71 \cdot 10^{-4}$ моль·$л^{-1}$, относительная - 11,42%. Оценка аналитического метода с использованием метрики AGREE показала его экологическую безопасность. Полученные результаты свидетельствуют о высоком потенциале разработанного подхода для применения в устойчивых аналитических технологиях.

Ключевые слова: пшеничные отходы, Fe-модифицированный биоуголь, угольно-пастовый электрод, потенциометрия, оценка экологичности

Introduction. Traditionally, activated carbon (AC) and biochar (BC) production relied on fossil resources such as coal; however, recent shifts favour renewable biomass, particularly agricultural waste, as sustainable precursors. These carbon materials possess high surface areas and porosity, making them vital for environmental remediation (Adylkanova et al., 2025) and energy storage (Chaudhary et al., 2024). Their properties can be optimised carefullyby selection selecting feedstocks, activation techniques, and pyrolysis conditions, thereby tailoring them for applications such as water treatment and air pollution control. Of particular interest is the application of such activated carbon in carbon paste electrodes (CPEs)—devices used in electrochemical analysis, namely potentiometric analysis (Amerkhanova et al., 2022). These electrodes, composed of carbonaceous material mixed with binding agents, exhibit high electrical conductivity, chemical stability, and the potential for modification. As a result, they are practical for a range of analytical tasks. Herein, one significant application of CPEs is the determination of metal ions in aqueous environments, which is crucial for several reasons. Excessive iron concentrations, as a potential ecological risk factor, can

exert toxic effects on aquatic organisms, including humans who rely on these water sources, thereby disrupting ecosystems and causing adverse health impacts (Teschke, 2024). Therefore, developing affordable, environmentally friendly sensor systems for monitoring iron concentrations is a pivotal challenge (Amerkhanova et al., 2022).

Literary review. The potentiometric method is a well-established, widely used technique for detecting metal ions in aqueous solutions. It offers advantages such as high selectivity, a broad linear response, excellent reproducibility and precision, real-time analysis, non-destructive testing, and overall simplicity and robustness. Its Nernstian behaviour facilitates straightforward calibration and reliable results. Notably, potentiometry is among the most sensitive methods for Fe^{3+} ion detection, achieving a detection limit of $1.0 \cdot 10^{-6}$ mol·L^{-1}, surpassing spectrophotometry (Cheng et al., 2021), fluorometry (Shahat et al., 2022), ratiometry (Yan et al., 2019), and voltammetry (Mittal et al., 2019).

Table 1 – Comparison of different methods for the quantitative content of Fe^{3+} ions

№	Method	Concentration range, mol·L^{-1}	LOD, mol·L^{-1}	Selectivity	Cost	Environmental friendliness	Ref.
1	Spectrophotometry	$2.0 \cdot 10^{-7} – 9.5 \cdot 10^{-5}$	$6.7 \cdot 10^{-8}$	Good-Moderate	Low-Moderate	Moderate (toxic chemicals)	(Cheng et al., 2021)
2	Fluorometry	$< 3.1 \cdot 10^{-5}$	$4.12 \cdot 10^{-7}$	Good-Moderate	Moderate	Good (reusability of sensors)	(Shahat et al., 2022)
3	Ratiometry	$2 \cdot 10^{-6} – 1.4 \cdot 10^{-5}$	$1.81 \cdot 10^{-8}$	Moderate	High	Moderate (toxic chemicals)	(Yan et al., 2019)
4	Voltammetry	$1.6 \cdot 10^{-5} – 4.4 \cdot 10^{-5}$	$5.2 \cdot 10^{-8}$	Good	Low	Good (low energy costs)	(Mittal et al., 2019)
5	Potentiometry	$1.0 \cdot 10^{-6} – 1.0 \cdot 10^{-1}$	$1.01 \cdot 10^{-7}$	Moderate	Low	Good (cheap, non-toxic, recycled materials)	(Kadyrbayeva et al., 2026)
6	Potentiometry	$1.0 \cdot 10^{-6} – 1.0 \cdot 10^{-1}$	$1.0 \cdot 10^{-6}$	n.r.	Low	Good (cheap, non-toxic, recycled materials)	(Amerkhanova et al., 2022)

This method covers a broader concentration range, from $1.0 \cdot 10^{-6}$ to $1.0 \cdot 10^{-1}$ mol·L^{-1}, than most other techniques. However, it may be less effective at very low concentrations than ratiometry. Its advantages include lower cost and eco-friendliness. Unlike some methods listed in Table 1, which can be expensive and involve toxic substances, potentiometry uses inexpensive, non-toxic, and recyclable materials. It also doesn't require complex sensors or hazardous chemicals, unlike fluorometry and spectrophotometry. Therefore, this study explores the production of biochar from local wheat waste, aligning with the UN Sustainable Development Goals, and examines its structural properties and potential as a carbon paste electrode for Fe(III) detection in water. A key innovation is the construction of the entire carbon electrode solely from wheat-waste-derived biochar, thereby avoiding the use of commercial carbon materials.

Despite biochar's environmental benefits, its application in electrochemical analysis is limited by poor selectivity and understanding of surface functional groups. This research aims to address these gaps by investigating iron-oxide-modified biochar for selective potentiometric detection of Fe^{3+} ions.

Methods and materials.

Chemicals and Materials: The reagents used in this study were of chemically pure, analytical-grade, and high-purity quality: $FeCl_3$ (1%, 10^{-6}-10^{-1} mol·L^{-1}), KCl (3.5 M), and KNO_3 (1%) solutions. Deionised water (0.055 μS) was used as the solvent for all solution preparations.

Preparation of BC: To prepare BC from wheat husks, the raw material was sequentially washed, dried, and grinded, followed by a three-step treatment process consisting of primary carbonisation (temperature of 600 °C, heating rate of 10 °C·min^{-1}, 1 h under the Ar atmosphere), chemical activation (using 1% $FeCl_3$ solution) as the activating agent, with a precursor-to-agent mass ratio of 1:10), and secondary carbonisation (temperature of 600 °C, heating rate of 10 °C·min^{-1}, 2 h under the Ar atmosphere). The intermediate product was washed with water until the pH was neutral, then dried at 105 °C to a constant weight. The final product yield was 25.64%.

Determination of the ash content: The biochar samples were dried in an oven at 105°C and then heated in a closed crucible in a muffle furnace at 750°C for 6 h. The remaining material after incineration is called ash. All lab tests, including the ashness test and others, were performed three times to ensure accuracy and calculate the standard deviation (SD).

Material Characterisation: A UNICUBE® elemental analyser for organic compounds was used for the CHNS analysis. The oxygen content (%) is obtained by recording the difference between 100 and the (CHNS+ash) content (in %) *(1)*:

$$O_{subs} = 100 - (N + C + H + S + A) \qquad (1)$$

where:

A is ash content, %

Information on the micro- and mesoporous texture (ranging from 1.7 nm to 300 nm) of carbon samples was obtained using the low-temperature nitrogen adsorption method on the device BSD-66OS A3 (China, 2024), after pretreatment at 200 °C and under a residual pressure of at least 0.001 bar. Further measurements of nitrogen adsorption/desorption isotherms were made at 77 K in the relative pressure range 0.005-0.991 bar. Standard data processing was performed using the BJH method, which applies a conventional cylindrical pore model. The total surface area (ΣS) and micropore surface area (S_μ) were calculated using the BET method. Also, the total pore volume (ΣV), micropore volume (V_μ), and average pore diameter (D_{avg}) (accounting for both micro- and mesopores) were determined using DFT calculations.

FTIR-spectroscopy was performed on the IR Tracer-100 (Shimadzu) spectrometer in the 4000-400 cm^{-1} region. The sample was prepared by mixing KBr with the BC sample in a 99.5%:0.5 % weight ratio and pressing the mixture into a disc for analysis, which

consisted of 32 scans at a resolution of 1 cm^{-1}. The background was collected before each measurement.

XRD analysis was performed using a Bruker D8 Advance Eco diffractometer over a 2θ range of 10–100° with a step size of 0.05°. The acquisition time was 1.5 seconds per step at a rotation speed of 15 rpm. The X-ray source used was Cu Kα radiation (λ = 1.54060 Å).

The SEM-EDX analysis was performed using a Phenom ProX Scanning Electron Microscope (voltage 15 kV, detector BSD Full, FW 519 μm, WD 7.089 mm, Vac. 1.2 Pa).

Potentiometric analysis: All potentiometric measurements were carried out in the following cell: Ag/AgCl//analyte/CPE/Copper. The carbon-paste electrode (CPE) (the designed one in this study) and the Ag/AgCl electrode served as the working and reference electrodes, respectively. All EMF measurements were performed using a pH-150 MI ionomer (n = 3). In direct potentiometry, CPE was immersed in a Fe(III) solution with concentrations ranging from $1.0 \cdot 10^{-6}$ mol·L^{-1} to $1.0 \cdot 10^{-1}$ mol·L^{-1} at room temperature (21.0°C ± 0.5°C) under continuous stirring. All analyte solutions maintained a consistent pH of 3.0. Each EMF measurement was performed in triplicate. Using the recorded data, a plot of the stabilised electrode potentials vs. pFe^{3+} was constructed.

For pH tests, HCl and NaOH solutions (both 0.1 M) were added to adjust the pH of the analyte solutions. In the selectivity tests, the fixed-potential method (FPM) was used to determine the selectivity coefficients for the interfering species (Na^{+}, Ni^{2+}, or Cr^{3+}).

Results. As described by Kadyrbayeva et al. (2026), iron chloride undergoes the following chemical reactions at different temperature ranges during carbonisation and chemical activation of BC with iron chloride (chem. eq.-s 1-7):

200°C-300°C

$$FeCl_3 + 2H_2O = FeOC \cdot H_2O + 2HCl \quad (1)$$

$$FeOCl \cdot H_2O = FeOOH + HCl \quad (2)$$

330°C-700°C

$$2FeOOH = Fe_2O_3 + H_2O \quad (3)$$

$$3Fe_2O_3 + C = 2Fe_3O_4 + CO \quad (4)$$

>700°C

$$2Fe_2O_3 + 3C = 4Fe + 3CO_2 \quad (5)$$

$$Fe_3O_4 + 2C = 3Fe + 2CO_2 \quad (6)$$

$$Fe_3O_4 + 4C = 3Fe + 4CO \quad (7)$$

As a result, highly porous BC containing highly stable Fe(II, III) oxides is formed. The elemental composition, ash content, and H/C and N/C ratios of BCs formed via this mechanism are given in Table 2.

Table 2 – Elemental composition and ash content in BC

Initial Raw Material	**Biochar** (Caban et al., 2019)	**BC7-washed** (Najafi-Ghiri et al., 2022)	**BC of the current interest**
Conditions of carbonization			
Atmosphere	Under limited oxygen conditions	Air	Ar
Temperature, °C	400	650	600
Heating rate, °C·min^{-1}	5	20	10
Composition			
C, %	51.50	70.09	76.46
H, %	2.50	3.78	1.683
N, %	0.00	4.23	4.01
S, %	7.01	n.r.	0.00
O_{subs}, %		19.72	8.33
A, %	27.00	n.r.	9.52
Aromaticity and polarity indices			
H/C	0.049	0.054	0.022
N/C	-	0.060	0.052
O/C	-	0.281	0.109
(O+N)/C	-	0.342	0.161
(O+N+S)/C	-	-	0.161

As shown in Table 2, the carbon content of biochar, which depends on synthesis conditions such as an inert medium, higher temperature, and lower heating rate, is 76.46%, a value higher than that reported for similar material (Turarbek et al., 2025). The atomic ratios of hydrogen to carbon (H/C) and oxygen to carbon (O/C) are widely recognised as crucial determinants of biochar characteristics. For instance, the H/C ratio indicates the conversion of hydrocarbon carbon structures into aromatic rings, serving as a measure of aromaticity and an indicator of lifetime (Wood et al., 2024). As pyrolysis temperatures rise, the H/C ratio drops significantly, indicating depolymerisation and increased oxidation resistance. Biochar with an H/C ratio below 0.7 primarily aromatises above 400°C, forming more fused aromatic rings that boost stability and soil resistance.

The O/C ratio of 0.109 is low, indicating a significant reduction in oxygen-containing functional groups in the biochar (BC), making it less polar and more hydrophobic (Kadyrbayeva et al., 2026). During carbonisation at temperatures above 450°C, these compounds decrease and eventually disappear. As the temperature rises, the decreasing O/C ratio reflects decarboxylation, indicating more advanced carbonisation and the formation of solid carbon structures.

The atomic ratio (O + N)/C indicates the material's overall polarity. The BC produced in this study yields a low value for this indicator (0.161), indicating a low overall polarity. According to the CHNS results, the biochars' low H/C and (O + N)/C ratios—indicating high aromaticity and low polarity—suggest a greater proportion of aromatic carbon and hydrophobic sites. Higher nitrogen and oxygen levels characterise biochars with a greater proportion of polar functional groups and a higher (O + N)/C ratio. Overall, these

findings align with the FTIR and Raman spectroscopy data (Fig. 1a).

As shown, no peaks corresponding to oxygen-containing functional groups are observed in the spectrum. According to Kadyrbayeva *et al.* (2026), the absence of obvious functional groups in the AC spectrum prepared at 800°C indicates that it is graphitic C in nature. This is most likely due to the formation of condensed aromatic structures, as observed in pure graphite and in graphite-rich activated carbons.

XRD analysis was performed to analyse the phase composition of biochar (Fig. 1b). It is known that the diffraction angle (2θ) of the 002 peak of crystalline graphite is 26.56º. Nevertheless, according to Lee *et al.* (2021), the XRD 002 peak of biochar is located at approximately 25.10°. This showed its microcrystallites differed from those of graphite. In the current case, a flat-convex peak was observed at 20-30º, which, based on the above information, is attributed to the graphite 002 phase. Additionally, the sharp, high peaks recorded at 32º, 35º, and 46º (2θ) align with the XRD patterns reported for hematite (Fe_2O_3) and magnetite ($Fe_2O_3 \cdot FeO$), particularly the peaks at 32º, 35º, and 46º (2θ) (Kadyrbayeva et al., 2026).

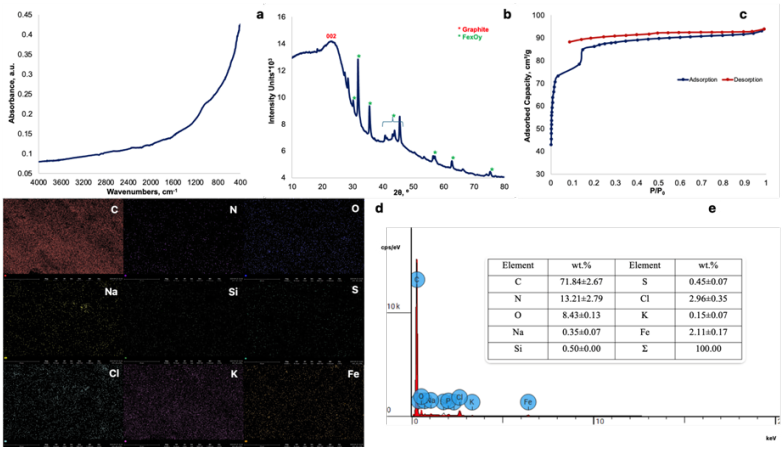


Figure 1 – (a) FTIR spectrum, (b) X-Ray diffractogram, (c) BET adsorption–desorption isotherms recorded for synthesised biochar; (d) the distribution map and (e) the content of elements on the BC's surface

BET analysis shows that the specific surface area (SSA) is 296.43 $m^2 \cdot g^{-1}$, with the pore distribution as micropores (0.35-2 nm) – 66.98%, mesopores (2-10 nm) – 30.57% and (10-50 nm) – 1.76%, large pores (50-200 nm) – 0.68%. The hysteresis loop most likely represents an H4 type (Fig. 1c) (Kadyrbayeva et al., 2026). In this loop, the adsorption branch exhibits a sharp increase at low relative pressure ($p/p_0 < 0.1$), typical of micropore filling. As the pressure increases to medium levels, the curve rises more gradually, indicating the presence of mesopores. On the desorption side, the lower limit usually corresponds to cavitation. This pattern is common in microporous carbon materials.

The EDX analysis shows that the elements on the surface of the BC are evenly distributed (Fig. 1d). Specifically, carbon makes up 71.84% of the composition,

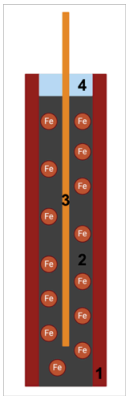
nitrogen accounts for 13.21%, and oxygen constitutes 8.43% (Fig. 1e). Additionally, minor amounts of Na, Si, S, Cl and K were identified: they are most likely derived from the inherent inorganic fraction of the wheat biomass precursor; and are typical for plant-derived biochars (Vassilev et al., 2010).

Further in this study, the synthesised biochar was employed as an electroactive material to construct a carbon-paste electrode (Fig. 2a), where 1 denotes the Teflon body, 2 denotes the modified biochar, 3 denotes the wire, and 4 denotes the glue. The results of the direct potentiometric measurements are presented in Figs. 2 b and 2c.

As shown in Fig. 1b, over the concentration range from $1.0 \cdot 10^{-6}$ mol·L^{-1} to $1.0 \cdot 10^{-1}$ mol·L^{-1}, a linear dependence is observed. The measured potential was 732.33 mV ±4.71 mV (pFe^{3+}=1), 683.00 mV ±3.39 mV (pFe^{3+}=2), 656.00 mV ±5.66 mV (pFe^{3+}=3), 623.33 mV ±5.10 mV (pFe^{3+}=4), 547.67 mV ±5.70 mV (pFe^{3+}=5) and 469.67 mV ±1.31 mV (pFe^{3+}=6) (95% confidence interval, n=3). It is also characterised by an intercept of 793.87 mV ± 10.31 mV based on three parallel measurements, and a Nernstian slope of 50.06 mV·decade^{-1}±1.28 mV·decade^{-1}. This approximately corresponds to a one-electron transfer process (chem. eq. 8):

$$Fe^{3+} + e^{-} \rightarrow Fe^{2+} \quad (8)$$

The dependence of the electrode potential on analyte pH indicates that the electrode signal is relatively stable across the pH range 3 to 6. Structural changes occur in the electrode in a strongly acidic medium (pH < 3), destabilising the analytical signal. Conversely, at pH > 7, the decrease in electrode potential is associated with the accumulation of less-soluble forms of iron (III). In these measurements, the predicted electrode potential across the pH range was accompanied by a 95% confidence band, with a maximum deviation not exceeding ±8.49 mV (n=3). Regarding the influence of temperature on electrode performance, experiments indicate that the electrode signal remains relatively stable over the temperature range of 21°C ± 0.5°C to 40°C ± 0.5°C (Fig. 2d).



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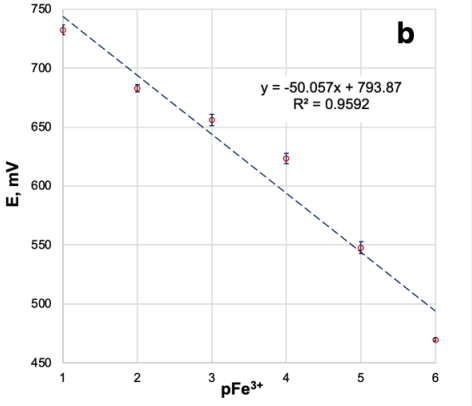

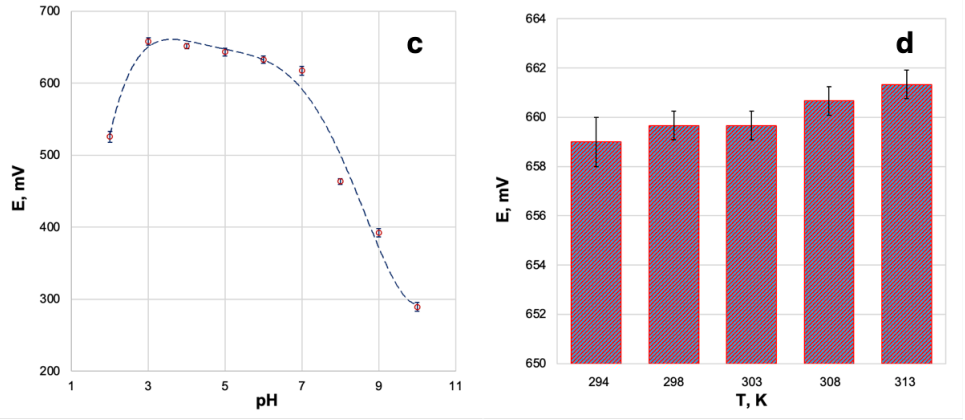

Figure 2 – Image of the carbon paste electrode made from BC (a), Nernst dependence recorded in $FeCl_3$ (0.001 M) solution for the electrode (b), the pH-electrode potential dependence (c) and the temperature-electrode potential dependence (d)

Over the examined temperature range, the measured electrode potential fell within a 95% confidence band, with deviations limited to ±1.13 mV (n=3).

Discussion. The electroanalytical performance of the Fe-modified biochar-based carbon-paste electrode is compared with that of other iron-selective electrodes (Table 2). Unlike electrodes that use commercial carbon materials, this electrode is entirely derived from waste-biomass biochar, underscoring its novelty. The study demonstrates that a renewable, waste-based carbon material can be effectively employed for simplified electrode fabrication and sustainable sensor development.

A comparison between the new electrode (№5) and the previously reported electrodes (№1–4) reveals notable differences in key testing parameters, including slope, concentration range, and detection limit (LOD). A review of the literature indicates that the slope of electrodes used to measure Fe^{3+} ions in aqueous solutions typically ranges from 18.6 to 56 mV·decade^{-1} (Table 3).

Table 3 – Comparison of the CPE of interest with the reference electrodes designed for detecting Fe^{3+} ions in aqueous solutions

№	Electrode-active material/ Binder	Slope, mV·decade^{-1}	Concentration range, mol·L^{-1}	LOD, mol·L^{-1}	Ref.
1	1,10 -((ethane-1,2- diylbis(oxy)) bis(2,1-phenylene))bis(N-p-tolylmethanimine) /Paraffin	18.6 ± 0.2	$1.0\cdot10^{-7}$-$1.0\cdot10^{-2}$	$5.0\cdot10^{-8}$	(Kaur et al., 2021)
2	1,4-Diaminoanthraquinone (DAQ)/Paraffin	19.7 ± 0.7	$1.0\cdot10^{-8}$-$1.0\cdot10^{-2}$	$5.0\cdot10^{-9}$	(Ghohari et al., 2018)
3	Tetradentate ligand/ NPOE	19.5	$4.9\cdot10^{-6}$-$1.3\cdot10^{-2}$	$1.2\cdot10^{-6}$	(Shafaatian et al., 2018)
4	Fe(II)-morin/ DOS	56.14 ± 0.22	$1.0\cdot10^{-6}$-$1.0\cdot10^{-1}$	$4.5\cdot10^{-7}$	(Ozer & Isildak, 2018)
5	Fe-modified BC/Paraffin	50.06 ± 1.28	$1.0\cdot10^{-6}$-$1.0\cdot10^{-1}$	$1.0\cdot10^{-6}$	This study

Electrodes 1-3 exhibit Nernstian slopes between 18.57 mV·decade^{-1} and 19.9 mV·decade^{-1}, typical of monovalent ion detection, whereas electrodes 4 and 5 show significantly higher slopes of 56.14 mV·decade^{-1} and 50.06 mV·decade^{-1}, indicating greater sensitivity and improved charge transfer, likely due to differences in active materials and design. The LOD of the studied electrode ($1.0 \cdot 10^{-6}$ mol·L^{-1}) aligns with reported values. While electrodes 1–3 utilise organic ligands and binders such as paraffin, DAQ, or NPOE, electrode 5 employs Fe-modified BC with paraffin, thereby enhancing conductivity, electron transfer, and active-site density and improving analytical performance.

The electrode testing in direct potentiometry for the determination of Fe^{3+} ions using the added-found method (n=3): $5.00 \cdot 10^{-3}$ mol·L^{-1} (added), $4.43 \cdot 10^{-3}$ mol·L^{-1}±$3.61 \cdot 10^{-4}$ mol·L^{-1} (found), absolute error $5.71 \cdot 10^{-4}$ mol·L^{-1}, relative error 11.42%.

Further, the selectivity of the electrode ($k_{A,B}^{pot}$) was evaluated with respect to interfering monovalent (Na^+), divalent (Ni^{2+}) and trivalent (Cr^{3+}) ions using the fixed-potential method (Amerkhanova et al., 2022). It has been shown that the electrode of interest maintains its electroanalytical performance over the concentration range of 10^{-4} mol·L^{-1} to 10^{-1} mol·L^{-1} for Na^+ and Ni^{2+} ions, with potentiometric selectivity coefficients of $2.98 \cdot 10^{-3}$ and $7.29 \cdot 10^{-3}$, respectively. For Cr^{3+} ions, as the interfering ions, the selectivity coefficient is $2.39 \cdot 10^{-6}$ over the analyte concentration range of 10^{-2} mol·L^{-1} to 10^{-1} mol·L^{-1}. Based on this ($k_{A,B}^{pot}$ <1 in the specified concentration ranges), it might be concluded that the selectivity of the electrode is limited by the type of interfering ions as well as the analyte concentration. The selectivity coefficient's dependence on ion type likely reflects variations in hydration energies and partitioning behaviours among monovalent, divalent, and trivalent ions, influencing their competition with Fe^{3+} ions at the electrode interface. The concentration dependence arises from the conditional nature of the fixed-potential method: higher Fe^{3+} activities favour ion dominance at the interface, reducing interference, whereas lower concentrations increase the impact of coexisting ions. Potentiometry is highly sensitive for Fe^{3+} detection; the proposed CPE surpasses the referenced methods in sensitivity (Table 1) with a detection limit of $1.0 \cdot 10^{-6}$ mol·L^{-1}, though it is less suitable for ultra-trace analysis than the compared methods.

The greenness evaluation of the method: The Eco-Scale is an ecological assessment tool designed to evaluate the environmental impact of analytical methods (Kadyrbayeva et al., 2026). It facilitates the comparison and selection of more environmentally friendly approaches. According to Table 4, the ecological assessment of the proposed potentiometric method yielded an excellent green analysis score.

Table 4 - Penalty points (PPs) for the proposed potentiometric method

Reagents and Penalty points		**Instruments and Penalty points**	
$FeCl_3$	4	Tube furnace	3
HCl	4	Potentiometry	1

KCl	0	Occupational hazard	3
KNO_3	3		
DI water	0		
Biomass	0		
Paraffin oil	1		
NaOH	2		
Epoxy	3		
Total:17		Total: 7	
Total Penalty points: 24		Analytical Eco-Scale total score: 76	

To comprehensively evaluate the environmental impact of our analytical methodology, the entire process was divided into three distinct stages: BC synthesis, carbon-paste electrode preparation, and analytical measurement. The greenness was independently assessed at each stage using the AGREE metric, yielding three separate circular diagrams (Fig. 3).

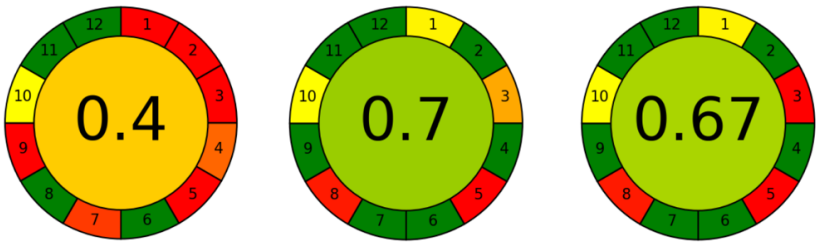

Figure 3 - The AGREE evaluation of the three-stage analytical process: (a) BC synthesis, (b) CPE preparation and (c) analytical measurement

The AGREE assessment indicated variable environmental friendliness across the analytical method, with an average score of 0.59. The biochar synthesis phase scored lower, while subsequent steps aligned well with green chemistry principles. Key sustainability features include using waste wheat biomass as a precursor, reducing reliance on non-renewable resources, and doping with Fe_xO_γ to avoid noble metals and hazardous reagents. Fabrication of the carbon paste electrode is straightforward and energy-efficient. Additionally, potentiometric analysis requires minimal sample preparation, no derivatisation, and small volumes of organic solvent, collectively enhancing the method's eco-friendliness and efficacy.

Conclusion. It might be suggested that integrating iron oxide into the biochar matrix creates specific surface sites capable of reversible Fe^{3+} interactions, thereby enhancing the sensitivity and selectivity of potentiometric measurements. The detected analytical performance, including a near-Nernstian response, satisfactory detection limits, relatively rapid response time, and a relatively tolerant response to interfering ions (within the specific concentration range), indicates that waste wheat-derived biochar has the potential to compete with sensors based on synthetic carbon-based materials. This improvement is evidenced by a higher slope of 50.06 mV·decade^{-1}, a working

concentration range of $1.0 \cdot 10^{-1}$ mol·L^{-1} to $1.0 \cdot 10^{-1}$ mol·L^{-1}, and a relatively low detection limit of $1.0 \cdot 10^{-6}$ mol·L^{-1}. The reported sensitivity and efficiency of the CPE of interest may be attributed to the unique properties of the iron-modified biochar; further studies of conductivity, electron transfer rates, and the density of active centres are warranted. These findings represent a significant advance in sustainable electrochemical sensing, highlighting the potential to replace resource-intensive materials with biomass-derived alternatives without sacrificing analytical performance.

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Ответственный редактор *А. Ботанқызы*
Редакторы: *Д.С. Аленов, Т. Апендиев*
Верстка на компьютере *Г.Д. Жадырановой*

Подписано в печать 31.03.2026.
Формат 60x88$^{1}/_{8}$.
22,0 п.л. Заказ 1.

«Central Asian Academic Research Center» LLP
Алматы, Қонаев к-сі, 142