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CONTENTS

Chemistry

Assembayeva E. K., Beisekhan A., Bozhbanov A. Zh., Nurmukhanbetova D.E., Gabdullina E.Zh. Effect of chia seeds (<i>Salvia Hispanica</i> l.) on the physicochemical and mineral properties of low-fat cottage cheese.....	11
Balkhashbay Sh.Zh., Azimbayeva G.E., Kudaibergenova G.N., Kamysbayeva A.K., Kurbanbayeva N.M. Determination of biologically active compounds in morphological parts of medicinal plants.....	24
Darmenbayeva A.S., Rajasekharan R. Preparation and characterization of nanocellulose biocomposites from agro-waste of the Zhambyl region.....	39
Demets O.V., Rakhimberlinova Zh.B., Zgardan V.V., Serykh N.V., Dyussekeyeva A.T. Qualitative and quantitative analysis of amino acids in Kyrgyz birch bark extract.....	55
Jumekeyeva A.I., Talgatov E.T., Auyezkhanova A.S., Kenzheyeva A.M., Naizabayev A.A. Complex formation of palladium (II) ions with organic polymers of various nature.....	70
Dmitriyeva E.A. Electrolytes of lithium-ion batteries.....	83
Yegemberdiyeva S., Abdurazova P., Turtabaev S., Shitybaev S., Kerimbayeva K. Catalytic properties of Ru- and Rh-promoted skeletal nickel catalysts in the hydrogenation of butyraldehyde.....	97
Yertayeva A.B., Adylbekova A.O., Toleubekova A.G. Production of emulsions stabilized by bentonite clay particles.....	112
Fischer D., Jumadilov T., Haponiuk J., Toilanbay G., Baishibekov A. Interpolymer KU-2-8: AV-17-8 systems for selective sorption of rhenium, molybdenum and tungsten.....	129
Zhanikulov N., Zhurgarayeva D. Investigation of the quality of cement clinker obtained from heap leaching waste.....	148
Zhoshybaeva A.A., Kozhanova K.K., Mombekov S.E., Barakova A.Sh. Pharmaceutical development of a medicinal product containing an isocitrate lyase inhibitor.....	162
Ivanov N.S., Abilmagzhanov A.Z., Nurtazina A.E., Adelbayev I.E., Kholkin O.S. Sequential electrochemical processes for the treatment of magnesium leaching solutions.....	176

Imangaliyeva B., Duzelbayeva S., Tolesinova I., Bukeykhan D., Turlanova A. Chemical and agronomic assessment of the use of mineral wool and coconut fiber as a substrate in a greenhouse.....	190
Kurmanaliev M.K., Shaikhova Zh.E., Abilkasova S.O., Kalimoldina L.M., Bugubaeva G.O. Crown esters immobilized on polymeric supports as novel interfacial catalysts.....	207
Mataev M.M., Ongarbek A.T., Sarsenbayeva Z.B., Nurbekova M.A., Abdraimova M.R. Synthesis and morphology of perovskite-structured $\text{CaMnO}_{2.98}$	221
Medeuova G.Zh., Azimbayeva G.E., Kaliyeva A.N.*, Sadykova D.A., Anuarova L.E. Determination of vitamins in <i>Polygonum Aviculare</i> L. using capillary electrophoresis.....	238
Mukusheva G.K., Jalmakhanbetova R.I., Seilkhanov T.M., Bakibaev A.A., Aliyeva M.R. Functional modification reactions at the nitrogen atom of salsolin and biological activity of the obtained derivatives.....	251
Muldakhmetov Z.M., Zhakina A.Kh., Arnt O.V., Vassilets Ye.P., Zhakin A.M. Composite materials modified with carbon filler.....	267
Nazarbek U., Raiymbekov Y., Abdurazova P., Kambarova G. Study on the efficiency of water treatment using nanostructured water.....	280
Nauanova A.P., Kassenov R.Z., Davrenbekov S.Zh., Bolatbay A.N., Altynbekkyzy A. Intensification of the process of extraction of humic substances from brown coal.....	295
Nurlybayeva A.N., Zharlykapova R.B., Taubaeva R.S., Matniyazova G.K., Rustem E.I. Study of physical, chemical and mechanical properties of acrylic terpolymer.....	309
Uali A., Omirzak U., Titanov A., Abilkanova F., Kunarbekova M. Waste biomass-derived Fe-modified biochar: structure and application in potentiometric analysis.....	323
Khamitova A.S., Nurmukhanbetova N.N., Ostretsova I.B., Kassenova N.B., Kuderina B.T. Synthesis of metal corrosion inhibitors based on ammonia.....	338

МАЗМҰНЫ

ХИМИЯ

Асембаева Э.К., Бейсехан А., Божбанов А.Ж., Чиа дәндерінің (<i>Salvia Hispanica L.</i>) майсыздандырылған сүзбенің физика-химиялық және минералдық көрсеткіштеріне әсері.....	11
Балқашбай Ш.Ж., Азимбаева Г.Е., Қудайбергенова Г.Н., Қамысбаева А.К., Қурбанбаева М. Дәрілік өсімдіктердің морфологиялық мүшелеріндегі биологиялық белсенді заттарды анықтау.....	24
Дарменбаева А.С., Rajasekharan R. Жамбыл облысының агрокалдықты негізінде наноцеллюлозалық биокомпозиттерді алу және олардың қасиеттерін зерттеу.....	39
Демец О.В., Рахимберлинова Ж.Б., Згардан В.В.*, Серых Н.В., Дюсекеева А.Т., 2026. Қырғыз қайың қабығының сығындысындағы аминқышқылдарының сапалық және сандық құрамын талдау.....	55
Джумекеева А.И., Талғатов Э.Т., Ауезханова А.С., Кенжеева А.М., Найзабаев А.А. Палладий (II) иондарының табиғаты әртүрлі органикалық полимерлермен кешен түзуі.....	70
Дмитриева Е.А. Литий-ионды аккумуляторлардың электролиттері.....	83
Егембердиева С.Ж., Абдуразова П., Туртабаев С.К., Шитыбаев С.А., Керимбаева К.З. Ru және Rh промоторланған қаңқалы никель катализаторларының май альдегидін гидрлеу реакциясындағы каталитикалық қасиеттері.....	97
Ертаева А.Б., Адильбекова А.О., Төлеубекова А.Ғ. Бентонит сазының бөлшектерімен тұрақтандырылған эмульсияларды алу.....	112
Фишер Д., Джумадилов Т., Хапонюк Ю., Тойланбай Г., Байшибеков А. Рений, молибден және вольфрамды селективті сорбциялауға арналған KU-2-8:AV-17-8 интерполимерлі жүйелері.....	129
Жаникулов Н., Жургараева Д. Үйінді шаймалау қалдықтарынан алынған цемент клинкерінің сапасын зерттеу.....	148
Жошыбаева А.А., Кожанова К.К., Момбеков С.Е., Баракова А.Ш. Изоцитратлиаза ингибиторын қамтитын дәрілік препаратты фармацевтикалық әзірлеу.....	162

Иванов Н.С., Абиьмагжанов А.З., Нұртазина А.Е., Адельбаев И.Е., Холкин О.С. Магнийді шаймалау ерітінділерін қайта өңдеу технологиясындағы дәйекті электрхимиялық процестер.....	176
Имангалиева Б., Дүзелбаева С., Төлесінова И., Букейхан Д., Тұрланова А., Жылыжайда минералды жүн мен кокос талшығын субстарт ретінде қолданудың химия-агрономиялық бағасы.....	190
Құрманалиев М.Қ., Шанхова Ж.Е., Әбілқасова С.О., Калимолдина Л.М., Бугубаева Г.О. Полимерлік тасымалдаушыларда иммобилизацияланған краун-эфирлер — жаңа фазааралық катализаторлар ретінде.....	207
Матаев М.М., Оңғарбек А.Т., Сарсенбаева З.Б., Нурбекова М.А., Абдраимова М.Р. Перовскит құрылымды $\text{CaMnO}_{2.98}$ синтезі мен морфологиясы.....	221
Медеуова Г.Дж., Азимбаева Г.Е., Калиева А.Н., Садыкова Д.А., Ануарова Л.Е. <i>Polygonum Aviculare L.</i> өсімдігінің құрамындағы дәрумендерді капиллярлы электрофорез әдісімен анықтау.....	238
Мукушева Г.К., Джалмаханбетова Р.И., Сейлханов Т.М., Бакибаев А.А., Алиева М.Р. Сольсолиннің азот атомы бойынша функционалдық модификация реакциялары және алынған туындылардың биологиялық белсенділігі.....	251
Мулдахметов З.М., Жакина А.Х., Арнт О.В., Василец Е.П., Жакин А.М. Көміртекті толтырғышпен модификацияланған композициялық материалдар.....	267
Назарбек У., Райымбеков Е., Абдуразова П., Қамбарова Ғ. Наноқұрылымданған суды қолдану арқылы суды тазарту тиімділігін зерттеу.....	280
Науанова А.П., Касенов Р.З., Давренбеков С.Ж., Болатбай А.Н., Алтынбекқызы Ә. Қоңыр көмірден гуминдік заттарды бөліп алу процесін қарқындету.....	295
Нурлыбаева А.Н., Жарлыкапова Р.Б., Таубаева Р.С., Матниязова Г.К., Рустем Е.І Акрил терполимердің физика-химиялық және механикалық қасиеттерін зерттеу.....	309
Уәли А., Өмірзақ Ұ., Титанов А., Абилканова Ф., Қунарбекова М. Қалдық биомассадан алынған темірмен түрлендірілген биокөмір: құрылымы және потенциометриялық талдауда қолданылуы.....	323
Хамитова А.С., Нурмуханбетова Н.Н., Острцова И.Б., Касенова Н.Б., Кудерина Б.Т. Аммиак негізінде металдар коррозиясының ингибиторларын синтездеу.....	338

СОДЕРЖАНИЕ

ХИМИЯ

Асембаева Э. К., Бейсехан А., Божбанов А.Ж., Нурмуханбетова Д.Е., Габдуллина Е.Ж. Влияние семян чиа (<i>Salvia Hispanica L.</i>) на физико-химические и минеральные показатели обезжиренного творога.....	11
Балкашбай Ш.Ж., Азимбаева Г.Е., Кудайбергенова Г.Н., Камысбаева А.К., Курбанбаева Н.М. Определение биологически активных веществ в морфологических органах лекарственных растений.....	24
Дарменбаева А.С., Rajasekharan R. Получение и свойства наноцеллюлозных биокомпозитов на основе агроотходов Жамбылской области.....	39
Демец О.В., Рахимберлинова Ж.Б., Згардан В.В., Серых Н.В., Дюсекеева А.Т. Качественный и количественный анализ аминокислот в экстракте коры берёзы киргизской.....	55
Джумекеева А.И., Талгатов Э.Т., Ауезханова А.С., Кенжеева А.М., Найзабаев А.А. Комплексообразование ионов палладия (II) с органическими полимерами различной природы.....	70
Дмитриева Е.А. Электролиты литий-ионных аккумуляторов.....	83
Егембердиева С.Ж., Абдуразава П., Туртабаев С.К., Шитибаев С.А., Керимбаева К.З. Каталитические свойства скелетных никелевых катализаторов, промотированных Ru и Rh, в реакции гидрирования масляного альдегида.....	97
Ертаева А.Б., Адильбекова А.О., Төлеубекова А.Ғ. Получение эмульсий, стабилизированных частицами бентонитовой глины.....	112
Фишер Д., Джумадилов Т., Хапонюк Ю., Тойланбай Г., Байшибеков А. Интерполимерные системы KU-2-8:AV-17-8 для селективной сорбции рения, молибдена и вольфрама.....	129
Жаникулов Н., Жургараева Д. Исследование качества цементного клинкера, полученного из отходов кучного выщелачивания.....	148
Жошыбаева А.А., Кожанова К.К., Момбеков С.Е., Баракова А.Ш. Фармацевтическая разработка лекарственного препарата, содержащего ингибитор изоцитратлиазы.....	162

Иванов Н.С., Абильмагжанов А.З., Нуртазина А.Е., Адельбаев И.Е., Холкин О.С. Последовательные электрохимические процессы в технологии переработки растворов выщелачивания магнезия.....	176
Имангалиева Б., Дүзелбаева С., Төлесінова И., Букейхан Д., Турланова А. Химико-агрономическая оценка использования минеральной ваты и кокосового волокна в качестве субстрата в теплице.....	190
Курманалиев М.К., Шаихова Ж.Е., Абилкасова С.О., Калимолдина Л.М., Бугубаева Г.О. Краун-эфиры, иммобилизованные на полимерных носителях, как новые межфазные катализаторы.....	207
Матаев М.М., Онгарбек А.Т., Сарсенбаева З.Б., Нурбекова М.А., Абдраимова М.Р. Синтез и морфология перовскитной структуры $\text{CaMnO}_{2.98}$	221
Медсұова Г.Дж., Азимбаева Г.Е., Калиева А.Н., Садыкова Д.А., Ануарова Л.Е. Определение витаминов, содержащихся в растении <i>Polygonum aviculare L.</i> , методом капиллярного электрофореза.....	238
Мукушева Г.К., Джалмаханбетова Р.И., Сейлханов Т.М., Бакибаев А.А., Алиева М.Р. Реакции функциональной модификации хлорида аммония по атому азота и биологическая активность полученных производных.....	251
Мулдахметов З.М., Жакина А.Х., Арнт О.В., Василец Е.П., Жакин А.М. Композитные материалы, модифицированные углеродным наполнителем.....	267
Назарбек У., Райымбеков Е., Абдуразова П., Камбарова Г. Исследование эффективности очистки воды с применением наноструктурированной воды.....	280
Науанова А.П., Касенов Р.З., Давренбеков С.Ж., Болатбай А.Н., Алтынбекқызы А. Интенсификация процесса выделения гуминовых веществ из бурого угля.....	295
Нурлыбаева А.Н., Жарлыкапова Р.Б., Таубаева Р.С., Матниязова Г.К., Рустем Е.И. Изучение физико-химических и механических свойств акрилового терполимера.....	309
Уали А., Омирзак У., Титанов А., Абилканова Ф., Кунарбекова М. Биоуголь, модифицированный железом, из отходов биомассы: структура и применение в потенциометрическом анализе.....	323
Хамитова А.С., Нурмуханбетова Н.Н., Острцова И.Б., Касенова Н.Б., Кудерина Б.Т. Синтез ингибиторов коррозии металлов на основе аммиака.....	338

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COMPOSITE MATERIALS MODIFIED WITH CARBON FILLER

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Abstract. Currently, one of the most promising and dynamically developing areas in materials science and nanotechnology is the synthesis of composite materials containing carbon nanofillers. The integration of such nanoscale carbon structures into the composite matrix allows for significant modification of its physicochemical properties, including thermal and chemical stability. The aim of the work was to obtain new composite materials based on the processing product of coal waste modified with graphene oxide, as well as to study the influence of various factors on the process of their production, and to study their composition and physical and chemical properties. The methodology of the work includes the synthesis of composite materials based on HA using GO was carried out under the action of ultrasound. Synthesis of composite

materials based on humic acids modified with graphene oxide was carried out at different ratios of the initial reagents (HA:GO=10:0.1, 20:0.1) and different times of ultrasonic exposure (0, 15, 30 minutes). The results of the study showed that ultrasonic treatment has a significant impact on the chemical structure and functional properties of humic acid-based composites. The optimal ultrasonication time was found to be 30 minutes; further increases in time lead to destructive processes in the humic acid structure and a reduction in the number of functional groups. The introduction of graphene oxide into the humic acid shifts thermal degradation processes to higher temperatures. The resulting composite materials are characterized by increased thermal stability and a developed surface structure, making them promising materials for use in wastewater treatment processes and as components of catalytic systems.

Keywords: composite materials, carbon filler, humic acids, graphene oxide, ultrasound

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КӨМІРТЕКТІ ТОЛТЫРҒЫШПЕН МОДИФИКАЦИЯЛАНҒАН КОМПОЗИЦИЯЛЫҚ МАТЕРИАЛДАР

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Аннотация. Қазіргі таңда материалтану мен нанотехнология саласындағы ең қарқынды дамып келе жатқан әрі болашағы зор бағыттардың бірі — құрамында көміртекті нанотолтырғыштар бар композиттік материалдарды синтездеу болып табылады. Осындай наноөлшемді көміртекті құрылымдарды композиттік матрицаға енгізу оның физика-химиялық қасиеттерін, соның ішінде жылулық және химиялық тұрақтылығын айтарлықтай өзгертуге мүмкіндік береді. Жұмыстың мақсаты — графен оксидімен модификацияланған көмір қалдықтарын өңдеу өнімдері негізінде жаңа композиттік материалдар алу, сондай-ақ олардың алыну процесіне әртүрлі факторлардың әсерін зерттеу және олардың құрамы мен физика-химиялық қасиеттерін жан-жақты талдау болып табылады. Зерттеу әдістемесіне сәйкес, гумин қышқылдары (НА) негізіндегі композиттік материалдарды графен оксидін (GO) қолдана отырып синтездеу ультрадыбыс әсерінде жүзеге асырылды. Синтез бастапқы реагенттердің әртүрлі қатынасында (НА:GO = 10:0.1, 20:0.1) және ультрадыбыстық өндеудің әртүрлі уақыт аралықтарында (0, 15, 30 минут) жүргізілді. Зерттеу нәтижелері ультрадыбыстық өндеудің гумин қышқылдары негізіндегі композиттердің химиялық құрылымы мен функционалдық қасиеттеріне елеулі әсер ететінін көрсетті. Ультрадыбыстық өндеудің оңтайлы уақыты 30 минут екені анықталды; уақытты одан әрі ұзарту гумин қышқылдарының құрылымында деструктивті өзгерістерге әкеліп, функционалдық топтардың санының азаюына себеп болады. Графен оксидін енгізу нәтижесінде термиялық ыдырау процестері жоғары температура аймағына ығысады. Алынған композиттік материалдар жоғары жылулық тұрақтылықпен және дамыған беткі құрылымымен ерекшеленеді. Бұл оларды ағынды суларды тазарту процестерінде және каталитикалық жүйелердің компоненттері ретінде қолдануға перспективалы материалдар қатарына жатқызуға мүмкіндік береді.

Түйін сөздер: композиттік материалдар, көміртекті толтырғыш, гумин қышқылдары, графен оксиді, ультрадыбыстық

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КОМПОЗИТНЫЕ МАТЕРИАЛЫ, МОДИФИЦИРОВАННЫЕ УГЛЕРОДНЫМ НАПОЛНИТЕЛЕМ

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Аннотация: В настоящее время одним из наиболее перспективных направлений в области материаловедения и нанотехнологий является разработка композитных материалов, содержащих углеродные наполнители. Интеграция наноразмерных углеродных структур в состав композитной матрицы позволяет существенно модифицировать их физико-химические свойства, включая термическую устойчивость, химическую стабильность и структурные характеристики. Целью работы является получение композитных материалов на основе продуктов переработки углеотходов, модифицированных оксидом графена, а также исследование влияния технологических параметров на их структуру и свойства. Методология исследования включает синтез композитов на основе гуминовых кислот, модифицированных оксидом графена, с использованием ультразвуковой обработки. Синтез проводился при различных соотношениях компонентов (ГК:ОГ = 10:0,1 и 20:0,1) и времени ультразвукового воздействия (0, 15 и 30 минут). Результаты исследования показали, что ультразвуковая обработка оказывает значительное влияние на химическую структуру и функциональные свойства полученных композитов. Установлено, что оптимальное время ультразвуковой обработки составляет 30 минут, тогда как дальнейшее увеличение времени приводит к деструкции структуры гуминовых кислот и снижению содержания функциональных групп. Введение оксида графена способствует повышению термической устойчивости композитов за счёт смещения процессов термической деструкции в область более высоких температур. Полученные материалы характеризуются развитой поверхностной структурой и повышенной

термостойкостью по сравнению с исходными гуминовыми кислотами. Практическая значимость работы заключается в возможности применения разработанных композитов в процессах очистки сточных вод, а также в качестве компонентов каталитических систем.

Ключевые слова: композитные материалы, углеродный наполнитель, гуминовые кислоты, оксид графена, ультразвук

Introduction. In recent decades, the rapid development of nanotechnology has led to increased interest in the development of composite materials using nanoparticles. One of the most promising areas is the creation of composites based on organic polymer matrices and nanosized fillers (Chai et al., 2021; Lima et al., 2021). Such materials are widely used in various fields of science and technology due to their improved physicochemical, mechanical, and functional properties compared to traditional polymers (Li et al., 2024). Natural polymers, which are highly available, low cost, and have pronounced stabilizing properties, are attracting particular attention from researchers.

One of the promising natural polymers capable of serving as a matrix for the creation of composites are humic acids (HA), products of the transformation of organic matter from coals of varying degrees of metamorphism. HA are a complex set of high-molecular compounds that differ in structure, degree of aromaticity, and content of functional groups. Depending on the formation conditions, such as the degree of metamorphism, genesis, and oxidation level, their composition may be dominated by both low-molecular-weight and high-molecular-weight components, including aromatic and aliphatic fragments in various ratios (Feng et al., 2024; Sazawa et al., 2026). Recent studies have shown the effectiveness of using humic acids as sorbents for organic pollutants and heavy metals, as well as in membrane technologies for wastewater treatment (Zhakina et al., 2024; Papadaki et al., 2023).

Literary review. In Kazakhstan, where coal remains one of the main energy sources, a significant share of the coal balance consists of low-energy coals and coal waste, which are not suitable for efficient use in traditional power plants. The disposal and processing of such carbon-containing materials represent both an environmental and technological challenge that requires modern solutions. One promising area is the extraction of humic acids from them – natural high-molecular substances with a wide range of functional properties. Due to their ability to sorb heavy metals, organic pollutants, and ions, humic acids are used in wastewater treatment technologies, the creation of sorbents and nanocomposites, and in electrochemical systems. The use of coal waste as a raw material for the production of humic acids not only promotes rational environmental management but also increases the added value of low-grade carbon raw materials. Graphene and its derivatives serve as a promising source for the creation of nanocomposite materials (Brahma et al., 2025; Ibragimova et al., 2026). Graphene and its oxidized form (graphene oxide, GO) have become new promising materials in the field of nanotechnology due to their unique thermal, chemical, mechanical, electronic, electrical, and optical properties (Haque et al., 2025; Lin et al., 2024). This material is applicable in various fields, from medicine to the oil industry and nanoelectronics

(Huang et al., 2025; Milka et al., 2026; Amigues et al., 2026). Additionally, its sensory qualities make it a potential sensor for detecting hazardous elements (Elhaes and Ibrahim, 2025; Harini and Ponraj, 2024).

Graphene oxide is small islands of graphene with sp²-hybridized bonds, randomly distributed over the surface of the substrate, surrounded by large areas with sp³-bonds, functionalized with oxygen-containing groups (epoxy, hydroxyl, carbonyl and carboxyl) (Jirickova et al., 2022). Graphene oxide has a relatively high exchange capacity (from 5 to 8 mmol-eq/g) due to the ion-exchange properties of its carboxyl groups, which effectively extract cations of transition metals, rare earth elements, actinides, and radionuclides.

In the future, this material can be used in tap water purification systems or portable bactericidal filters, avoiding chemical oxidation processes (Gayen et al., 2024; Tiwary et al., 2024). The significant advantage of this material is its low consumption and easy regeneration (Avila-Aviles, 2025).

The combination of natural polymers and graphene oxide is a promising direction in the field of creating multifunctional nanocomposite systems. However, there are still open questions related to optimizing the synthesis conditions, ensuring uniform distribution of nanoparticles, and studying the physical and chemical properties of the composite.

The primary objective of this study is to produce composites based on humic acids and graphene oxide, as well as to investigate the influence of various factors on the production process, their composition, structure, and physicochemical properties. This work is a logical continuation of research conducted in the “Polymer Chemistry” laboratory of the “Institute of Organic Synthesis and Coal Chemistry of the Republic of Kazakhstan”, aimed at creating new functional materials based on man-made carbon sources.

Materials and methods. Materials: Composite materials modified with graphene oxide were obtained from humic acids (HA) isolated from samples of weathered coal from the Shubarkol deposit (Karaganda, Kazakhstan) using a method we developed earlier (Zhakina et al., 2020).

The following were used as a composite modifier:

– graphene oxide (GO) from Sigma Aldrich (USA, 796034-1G) with the following characteristics: powder, 15-20 sheets, 4-10% oxidation at the edges, average number of layers 15-20.

Methods: As a source of ultrasonic radiation, an IL 100-6/2 ultrasonic unit with a maximum power of 1200 W and a cylindrical waveguide, equipped with an IL10-1.0 ultrasonic generator with a magnetostrictive transducer operating at a frequency of 22 kHz, was used. An “Anion-4100” (Infraspec-Analyte, Novosibirsk, Russia) laboratory conductivity meter was used to measure the total content of oxygen-containing functional groups $\Sigma(\text{COOH}+\text{OH})$. Measurements were performed sequentially with three sample weights, and the average value of the three runs was taken as the final value.

The elemental composition (C, H, O) of the starting humic acids, GO, and composites was determined by elemental analysis using an Elementar Unicube analyzer (Germany).

Fourier-transform infrared spectroscopy was used to study the chemical composition of the composites and identify functional groups. Spectra were recorded in the 400-4000 cm^{-1} range with an error of no more than 2 cm^{-1} using an FSM-1201 spectrometer (Infraspec Company, St. Petersburg, Russia).

The morphology and surface structure of the starting compounds and the resulting composites were examined using a MIRA 3 scanning electron microscope (Tescan Orsay Holding, Brno-Kohoutovice, Czech Republic).

Synthesis of composite materials.

Synthesis of HA-based composite materials using GO was accomplished using ultrasound. The synthesis was performed using various ratios of the starting reagents (HA:GO = 10:0.1, 20:0.1) and different ultrasonic exposure times (0, 15, 30 min). To form the composite material, the mixture after ultrasonic treatment was subjected to thermal holding at 90°C for 6 hours. Ultrasonic treatment has a significant impact on the chemical structure and functional properties of humic acid-based composites. One of the key indicators is the content of oxygen-containing groups, which determine the sorption and reactivity properties of the composite.

Results. Table 1 evaluates the change in the total content of oxygen-containing functional groups $\Sigma(\text{COOH}+\text{OH})$ under the influence of ultrasonic irradiation (0, 15, 30 minutes) at different mass ratios of the HA:GO components. The composition of the HA:GO composites varied in HA:GO mass ratios of 10:0.1 and 20:0.1, with ultrasonic treatment durations of 0, 15, and 30 minutes. A study examining the effect of ultrasonication on the production of HA:GO composites revealed that ultrasonic irradiation leads to a slight but consistent increase in the total oxygen-containing functional groups $\Sigma(\text{COOH}+\text{OH})$. The most pronounced effect is observed after 30 minutes of treatment, after which the $\Sigma(\text{COOH}+\text{OH})$ values stabilize.

Table 1 – Effect of the ratio of initial components and the time of ultrasonic treatment on the product yield and other characteristics

Composite (mass%)	US, min	$\Sigma(\text{COOH}+\text{OH})$, mg-eq/g	Yield, %
HA:GO 10:0.1	–	4.2±0.2	82.15
	15	4.2±0.2	82.74
	30	4.4±0.2	83.07
HA:GO 20:0.1	–	4.5±0.2	87.64
	15	4.5±0.2	87.99
	30	4.6±0.2	88.53

This effect is explained by the fact that ultrasound improves the dispersion of graphene oxide in the humic acid matrix and activates the surface through cavitation processes, which create localized zones of high temperatures and pressures. With an increase in the content of graphene oxide from 10 to 20 wt.%, an increase in $\Sigma(\text{COOH}+\text{OH})$ to 4.6 mg-eq/g and an increase in the product yield to 88.53% are observed with a ratio of HA:GO=20:0.1 and an ultrasonic treatment duration of 30 minutes, which can be considered the optimal combination of synthesis parameters.

Table 2 presents the results of elemental analysis of composites based on humic acid with graphene oxide.

Table 2 – Characteristics of synthesized composites.

Composite (mass, %)	C ^g , %	H ^g , %	O ^g , %
HA:GO 10:0.1 (US=30 min)	48.91±0.2	3.04±0.1	48.05±0.2
HA:GO 20:0.1 (US=30 min)	45.63±0.2	3.33±0.1	51.04±0.2

Elemental analysis revealed that all composites contain three main elements: carbon, hydrogen, and oxygen, the ratio of which reflects the characteristics of the chemical interaction of HA with GO. The oxygen content varies from O^g=48.0 to 51.0%, which is explained by the presence of numerous oxygen-containing groups in the structure of graphene oxide (C–OH, C=O, C–OOH).

The structure of the starting compounds and the HA:GO composite was evaluated using IR spectroscopy. Figure 1 shows the IR spectra of the starting components and the HA:GO composite (20:0.1) obtained by ultrasonic treatment for 30 min. The IR spectrum of HA (a) is characterized by a broad, intense band in the 3000-3600 cm⁻¹ region, corresponding to the stretching vibrations of hydroxyl groups (–OH) involved in the hydrogen bond system. The band at ~1710 cm⁻¹ is due to the stretching vibrations of carbonyl groups (C=O) of carboxyl fragments. A set of bands in the 1595-1500 cm⁻¹ range is due to vibrations of aromatic C=C bonds, as well as asymmetric vibrations of COO⁻ groups. The bands at 1270-1230 and 1030-1010 cm⁻¹ correspond to the stretching vibrations of C–O in phenolic, alcohol, and ester groups, reflecting the polyfunctional nature of the humic matrix.

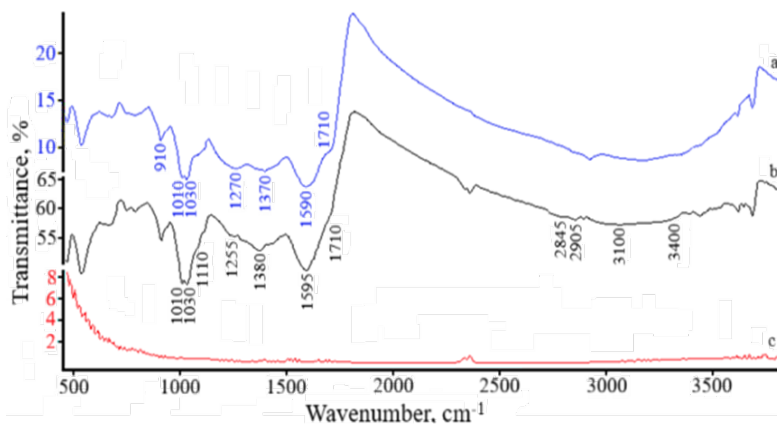


Figure 1 - IR spectra: graphene oxide (c), humic acids (b), composite HA:GO (20:0.1, US=30 min) (a).

The IR spectrum of graphene oxide is inert and lacks any clearly defined peaks. Weak vibrations in the spectrum are due to the minimal content of oxygen-containing reaction centers. The IR spectrum of the HA:GO composite (b) retains the main spectral signatures of both initial components but exhibits a number of significant changes.

A redistribution of the –OH band intensity is noted in the 3100-3400 cm^{-1} region, indicating increased hydrogen bonding between the humic acid functional groups and the oxygen-containing groups of graphene oxide. A slight shift of the carbonyl band in the 1710 cm^{-1} region in the composite spectrum demonstrates a change in intensity and indicates the involvement of the carboxyl groups of HA and GO in interfacial interactions. The weakening and smoothing of the bands in the 1200-1000 cm^{-1} region may be due to partial shielding of the C–O groups of the humic matrix during adsorption on the graphene oxide surface. Overall, the combination of spectral changes confirms the formation of a HA:GO composite system.

Discussions. The thermal stability of the resulting HA:GO composite and a comparative assessment with the original components (HA and GO) were determined using thermogravimetric analysis (TGA). Thermograms of all samples are shown in Figure 2. As Figure 2 shows, the thermogravimetric curve combining data for the three samples (HA, GO, and HA:GO) reveals marked differences in thermal behavior, reflecting the specific features of their chemical composition and structural organization. The curve for humic acids is characterized by a three-stage mass loss. The first stage records moisture removal of ~5%. In the range of 180-600°C, intense decomposition of organic matter is observed, with a mass loss of approximately 45%, corresponding to the destruction of aliphatic and aromatic fragments containing oxygen functional groups. At the final stage (600-1000°C), further destruction of heat-resistant structural fragments occurs, accompanied by an additional mass loss of approximately 25%. The total mass loss is approximately 75%, reflecting the relatively low thermal stability of humic acids.

Graphene oxide is characterized by a multi-step curve shape. In the range of 30-770°C, adsorbed moisture is removed and oxygen-containing functional groups (hydroxyl, carboxyl, and epoxy) undergo sequential degradation, accompanied by a total mass loss of approximately 25%. An additional loss (~5%) in the range of 770-1000°C is due to the destruction of residual functional groups and the onset of oxidation of the carbon core. The total residue after heating is approximately 70%, indicating a high degree of ordering of the carbon layers and good thermal stability of GO.

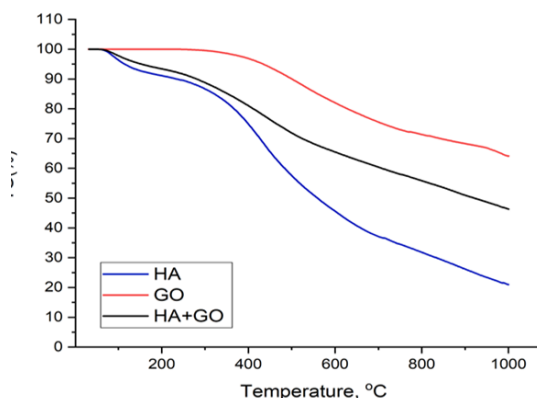


Figure 2 - Thermograms: HA, GO and HA:GO (20:0.1, US=30 min).

The TGA curve of the HA:GO composite demonstrates an intermediate behavior between the initial components. At the first stage (30-190°C), moisture evaporation (~5%) is recorded. The second stage (190-570°C) corresponds to the main stage of destruction, including the decomposition of the organic components of humic acids and partial destruction of the functional groups of graphene oxide, with a mass loss of about 40%. At the final stage (570-1000°C), a number of less pronounced processes associated with the oxidation of stable structural fragments are observed, accompanied by an additional mass loss of about 12%. The total mass loss is approximately 57%, the remainder is 43%. A combined analysis of the TGA curves showed that the thermal stability of the studied samples increases in the following order: HA < HA:GO < GO.

Thus, the introduction of graphene oxide into the humic acid structure reduces overall mass loss and shifts thermal degradation processes to higher temperatures. This indicates a stabilizing effect of the graphene filler, associated with enhanced interfacial interactions and the formation of a denser compo-site structure. The resulting composite exhibits improved heat-resistant properties compared to the original humic acids, making it a promising material for the creation of heat-resistant sorbents and functional coatings.

Figure 3 shows topographic images of the surface morphology of the initial components and the composite of the composition HA:GO (20:0.1, US=30 min), obtained using scanning electron microscopy (SEM). Humic acids (Figure 3a) exhibits a complex porous structure with a distinctly uneven and rough surface containing numerous pores and cracks. HA particles have an irregular, predominantly rounded shape, often indicative of its natural origin and complex chemical composition. At $\times 100,000$ magnification, pores of varying sizes are observed, which is important for the sorption properties of the composite.

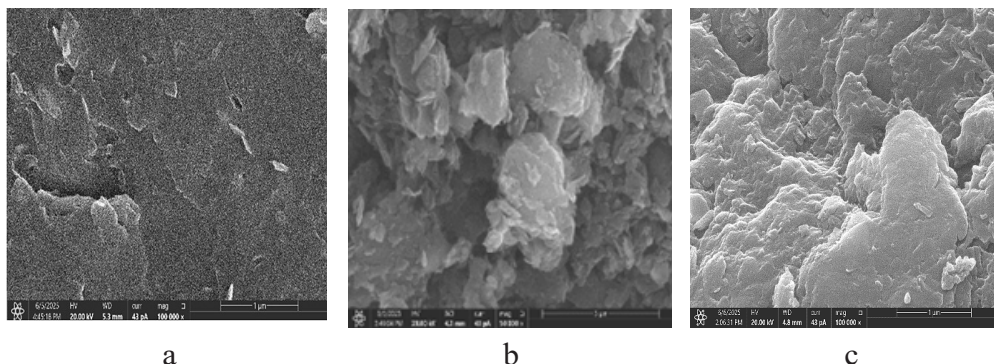


Figure 3 - Electron microscopic images: HA (a), GO (b) and composite HA:GO (20:0.1, US=30 min) (c).

Graphene oxide (Figure 3b) appears in microscopic images as a multilayered, flaky structure typical of graphene. The plates vary in size and thickness, forming loose agglomerates. The particle surface appears rough and wavy, due to the presence of oxygen-containing functional groups (hydroxyl, carboxyl, and epoxy) that deform the

graphene crystal lattice. The samples exhibit non-uniform particle sizes, which may be due to incomplete oxidation of the original graphite and partial aggregation of GO sheets. Micrographs confirm that the synthesized graphene oxide is a thin-platelet, porous material with a high degree of exfoliation and a developed specific surface area, suitable for modification and the formation of composites.

The presented micrograph (Figure 3c) shows the surface morphology of a composite HA:GO (20:0.1, US=30 min). The sample exhibits a dense, uniform surface morphology with aggregated particles of predominantly rounded and irregular shapes. Unlike pure graphene oxide, which is characterized by a pronounced layered, flaky structure, this composite exhibits a reduced degree of delamination and a more compact structure, indicating the intercalation of GO layers into the organic HA matrix. The particle surface appears relatively smooth and compact, indicating the formation of strong interactions between the functional groups of HA and the oxygen-containing centers of GO. Small pores and microcracks are locally present, which could act as active sites for the sorption of metal ions or other target molecules. Composite formation is accompanied by chemical and physical modification of the surface, during which the layered GO structure is partially degraded, and amorphous humic acids fill the interlayer spaces and bind individual graphene oxide sheets. This results in a stable hybrid structure with a uniform distribution of components, a high degree of compaction, and a developed specific surface area. Ultrasonic treatment also has an additional effect, promoting the breakdown of agglomerates and uniform dispersion of GO nanoparticles within the organic matrix of the HA.

This results in the formation of a stable hybrid structure with a uniform distribution of components, a high degree of compaction, and a developed specific surface area. Ultrasonic treatment has an additional effect, promoting the breakdown of agglomerates and the uniform dispersion of GO nanoparticles within the organic matrix of the HA. Cavitation effects, arising from the propagation of ultrasonic waves in a liquid medium, improve the homogeneity of the composition and activate interfacial interactions. According to SEM data, it can be concluded that the obtained HA:GO composite is a dense but porous nanostructured material with a developed surface microrelief, which makes it promising for use in sorption and catalytic systems.

Conclusion. During the study, composites based on humic acids and graphene oxide were synthesized. Using modern physicochemical methods, the composition and structure of the resulting composites were studied. The effect of ultrasonic irradiation on the structure and properties of the resulting composites was examined. The optimal ultrasonication time was found to be 30 minutes; further increases in time lead to destructive processes in the humic acid structure and a reduction in the number of functional groups.

The introduction of graphene oxide into the humic acid structure reduces overall mass loss and shifts thermal degradation processes to higher temperatures. This indicates a stabilizing effect of the graphene filler, associated with enhanced interfacial interactions and the formation of a denser composite structure. A composite of HA:GO, 20:0.1, with a 30-minute ultrasonic treatment, exhibits improved heat-resistant properties compared

to the original humic acids, making it a promising material for creating heat-resistant sorbents and functional coatings.

It was found that at low HA content, ultrasound causes partial destruction of oxygen-containing groups, whereas with an increase in the HA proportion to the optimal ratio, activation of the functional groups is observed without their destruction. Thus, regulating the composition and duration of ultrasonic exposure allows one to control the chemical properties of the material.

IR spectroscopic analysis confirmed the formation of physicochemical interactions between the HA and GO components, including hydrogen and electrostatic bonds, which ensure the structural stability and homogeneity of the material.

Micrographs of the HA:GO composite are characterized by a dense yet porous nanostructure with a developed surface microrelief, which increases its effectiveness in sorption and catalytic processes.

Thus, modifying a humic matrix with graphene oxide results in the formation of structurally ordered composites with increased thermal stability, a developed surface area, and stable functional groups. These characteristics combined ensure high activity and thermal stability, making the developed materials promising for use in wastewater treatment processes and catalytic systems.

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