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CONTENTS

Abdugaliyev A.Sh., Jalmakhanbetova R.I., Mukusheva G.K. Atomistic insight into solvent effects on the chemical structure of betulonic acid.....	11
Assembayeva E.K., Shadi D.K., Nurmukhanbetova D.E., Bozhbanov A.Zh., Gabdullina E.Zh. Effect of inulin on the quality characteristics of a fermented cheese whey-based beverage	25
Asylbekova D.D., Toktibayeva Kh.R., Kaldybayeva A.R., Zhurkhabayeva L., Tukibayeva A.S. Electrochemical behavior of phosphorous acid on a platinum electrode and its galvanostatic anodic oxidation using a lead anode	39
Auyeshov A.P., Yeskibayeva Ch.Z., Ibrayeva A.M., Alzhanov K.B., Dzholdasova E.M. Investigation of the adsorption characteristics of silica obtained from serpentinite by a combined acid–alkali method	58
Galymzhan A., Kozhabekov S., Kuanyshev A., Sansyzbay N., Abdrasilova A. Effect of octadecyl methacrylate–maleic anhydride copolymer composition on crude oil rheological properties	78
Darmenbayeva A.S., Rajasekharan R. Investigation of the structural properties of nanocellulose membranes derived from agricultural waste	95
Dmitriyeva E.A., Medvedev A., Kemelbekova A. Development of a low-temperature gas sensor based on SnO ₂ thin films and a precision measurement system for high-resistance detection	109
Dzheldybaeva I.M., Kairbekov Zh., Esenalieva M.Z., Dossumova B.T., Suimbaeva S.M. Influence of ultrasonic treatment on the structure and physicochemical properties of brown coal humic acids	125
Jumadilov T.K., Khimersen Kh., Imangazy A. Features of heavy metal ion sorption by interpolymer systems formed from homogeneous polyelectrolytes	140
Zhylykybek M., Orazbekova R.S., Yerkibaeva M.K., Aidarova A.O., Baizhumanova T.S. Co–Mg oxide catalysts for deep oxidation of methane: synthesis, structure, and catalytic activity.....	154
Imangaliyeva B., Rakhmetova G., Dossanova B., Sovet N. Technology of biological fertilizer production by hydrolysis treatment of polymer waste.....	169
Karar M.E., Beisenbayev O.K., Alibekov R.S., Shamsudin R. Starch extraction from Baidaly rice and Gala potatoes for application in biodegradable materials.....	186
Kudaibergenova G.N., Azimbayeva G.E., Kamysbayeva A.K., Izteleu B.M., Kurbanbayeva N.M. Fatty acid composition of <i>Helianthus tuberosus</i> flowers	204
Massenova A.T., Sass A.S., Torlopov I.I., Rakhmetova K.S., Malgazhdarova M.K. Tuning of surface acidity in Zn–Me-modified shaped ZSM-5–Al ₂ O ₃ alkylation catalysts.....	219
Nurislamov R.M., Nefedov A.N., Beissenbayev D.K., Leontyeva X.A., Imashev B.K. Effect of oxide films on the corrosion resistance of iron: modeling in COMSOL Multiphysics 6.2.....	238

Nurlybayev O., Kudaibergenova R., Taubayeva R., Bekaulova A., Seitmanbetova A. Textural properties of bentonite–sawdust carbon composites after metal-chloride-assisted carbonization.....	250
Puzikova D.S., Bessonova G.V., Kenzin N.R., Zhurinov M.Zh., Torlopov I.I. Modeling cavitation of oil-containing media in various hydrodynamic configurations.....	263
Rakhimov A., Zhanikulov N., Taimasov B., Potapova E., Sarsenbaev B. Calculation of the raw material composition for cement clinker with the addition of lead slag processing waste.....	285
Ryskalieva D., Baibulanova A., Yessenamanova M., Mustafina A., Samat A. Hydrogen sulfide pollution in the atmosphere of an industrial city: the potential role of evaporation fields (Atyrau, Kazakhstan)	299
Serikbayeva K.T., Chalov T.K., Sadykov K.A. Rubidium sorption from aqueous solutions by VEMEA–AGE–PEI anion exchangers.....	322
Seitkazinova A.R., Nazhipkyzy M., Yeleuov M.A., Zhaparov A.A., Kalybayeva A.K. Orange peel-derived porous activated carbon	334
Samadun A.I., Taussarova B.R., Asrakulova D.I., Nurmukhanbetova D.E. Biodegradable packaging films with copper nanoparticles: effect of NaBH ₄ concentration on morphology and antifungal properties	351
Khamitova T.O., Tyanakh S., Bolatbay A.N., Ma Fengyun, Aitbekova D.E. Linking geological genesis to the molecular and optical traits of humic acids from coal wastes.....	370
Talgatov E., Zamanbekova A., Abilmagzhanov A., Auyezkhanova A., Akhmetova S. Polymer-modified supported catalysts for the oxidation of cyclohexane with hydrogen peroxide.....	389

МАЗМҰНЫ

Абдугалимов А.Ш., Джалмаханбетова Р.И., Мукушева Г.К.

Бетулон қышқылының химиялық құрылымына еріткіштер әсерін атомистикалық тұрғыдан зерттеу11

Асембаева Э.К., Шәді Д.Қ., Нурмуханбетова Д.Е., Божбанов А.Ж., Габдуллина Е.Ж.

Инулиннің ірімшік сарысуы негізіндегі ашытылған сусынның сапа көрсеткіштеріне әсері.....25

Асылбекова Д.Д., Токтыбаева Х.Р., Қалдыбаева А.Р., Журхабаева Л., Түкібаева А.С.

Фосфорлы қышқылдың платина электродындағы электрохимиялық қасиеттері және оны қорғасын анодын қолданып гальваностатикалық анодты тотықтыру.....39

Ауешов А.П., Ескибаева Ч.З., Ибраева А.М., Алжанов К.Б., Джолдасова Э.М.

Қышқылды-сілтілі комбинациялық әдіспен серпентиниттен алынған кремнеземнің адсорбциялық сипаттамаларын зерттеу58

Ғалымжан А., Қожабеков С., Қуанышев А., Сансызбай Н., Абдрасилова А.

Октадецилметакрилат пен малеин ангидридi сополимерлері құрамының мұнайдың реологиялық қасиеттеріне әсері78

Дарменбаева А.С., Rajasekharan R. Агроқалдықтардан алынған наноцеллюлозалы

мембраналардың құрылымдық қасиеттерін зерттеу.....95

Дмитриева Е.А., Медведев А., Кемелбекова А.

SnO₂ жұқа қабықшалары негізіндегі төмен температуралы газ сенсорын және жоғары кедергілерді тіркеуге арналған прецизионды өлшеу жүйесін әзірлеу.....109

Джелдыбаева И.М., Қайырбеков Ж., Есеналиева М.З., Досумова Б.Т., Суймбаева С.М.

Ультрадыбыстық өңдеудің қоңыр көмір гумин қышқылдарының құрылымы мен физика-химиялық қасиеттеріне әсері.....125

Джумадилов Т.К., Химэрсэн Х., Иманғазы А.

Біртекті полиэлектролиттерден құрылған интерполимерлік жүйелердің ауыр металл иондарын сорбциялау ерекшеліктері.....140

Жылқыбек М., Оразбекова Р.С., Еркибаева М.К., Айдарова А.О., Байжұманова Т.С.

Метанды терең тотықтыруға арналған Co–Mg оксидті катализаторлар: синтезі, құрылымы және каталитикалық белсенділігі.....154

Иманғалиева Б., Рахметова Г., Досанова Б., Совет Н.

Полимер қалдықтарын гидролиздік өңдеу арқылы биологиялық тыңайтқыш алу технологиясы.....169

Қарар М.Е., Бейсенбаев О.К., Алибеков Р.С., Роснах Шамсудин.

Байдалы күрішпен және Гала сортты картоптан крахмал алу және оны биоыдырайтын материалдарда қолдану.....186

Құдайбергенова Г.Н., Азимбаева Г.Е., Камысбаева А.К., Ізтелеу Б.М., Құрбанбаева Н.М.

Топинамбур гүлдерінің май қышқылдық құрамы204

Масенова А.Т., Сасс А.С., Торлопов И.И., Рахметова К.С., Малгаждарова М.К. Zn–Me-модификацияланған қалыпталған ZSM-5–Al ₂ O ₃ алкилдеу катализаторларының беттік қышқылдығын реттеу.....	219
Нурисламов Р.М., Нефедов А.Н., Бейсенбаев Д.К., Леонтьева К.А., Имашев Б.К. Темірдің коррозияға төзімділігіне оксидтік қабықшалардың әсері: COMSOL Multiphysics 6.2 бағдарламасында модельдеу.....	238
Нурлыбаев О., Кудайбергенова Р., Таубаева Р., Бекаулова А., Сейтманбетова А. Металл хлоридтері қатысында карбонизацияланған бентонит–ағаш үгіндісі көміртекті композиттерінің текстуралық қасиеттері.....	250
Пузикова Д.С., Бессонова Г.В., Кензин Н.Р., Жұрынов М.Ж., Торлопов И.И. Өртүрлі гидродинамикалық конструкциялардағы мұнай құрамды орталардың кавитация процесін модельдеу	263
Рахимов А., Жаникулов Н., Таймасов Б., Потапова Е., Сәрсенбаев Б. Қорғасын қожын өндеу қалдықтары қосылған цемент клинкерінің шикізат шихтасының құрамын есептеу	285
Серікбаева К.Т., Чалов Т.К., Садықов К.А. Сулы ерітінділерден рубидийді ВЭМЭА–АГЭ–ПЭИ аниониттерімен сорбциялау.....	299
Сейтказинова А.Р., Нажипқызы М., Елеуов М.А., Жапаров А.А., Қалыбаева А.Қ. Апельсин қабығынан алынған кеуекті белсендірілген көмір.....	322
Самадун А.И., Таусарова Б.Р., Асракулова Д.И., Нурмұханбетова Д.Е. Мыс нанобөлшектері бар биоыдырайтын қаптамалық үлдірлер: NaBH ₄ концентрациясының морфологияға және саңырауқұлаққа қарсы қасиеттеріне әсері.....	334
Хамитова Т.О., Тянах С., Болатбай А.Н., Ма Фэньюнь, Айтбекова Д.Е. Көмір қалдықтарынан бөлінген гумин қышқылдарының геологиялық генезисі мен молекулалық және оптикалық қасиеттерінің байланысы	351
Рысқалиева Д.К., Байбуланова А.А., Есенаманова М.С., Мұстафина А.Ж., Самат А.С. Өнеркәсіптік қала атмосферасының күкіртсутекпен ластануы: булану алаңдарының ықтимал рөлі (Атырау, Қазақстан)	370
Талғатов Э.Т., Заманбекова А.Т., Абильмагжанов А.З., Әуезханова А.С., Ахметова С.Н. Циклогександы сутек пероксидімен тотықтыруға арналған полимермен модификацияланған тасымалдағышқа бекітілген катализаторлар	389

СОДЕРЖАНИЕ

Абдугалимов А.Ш., Джалмаханбетова Р.И., Мукушева Г.К. Атомистическое исследование влияния растворителей на химическую структуру бетулоновой кислоты.....	11
Асембаева Э.К., Шади Д.К., Нурмуханбетова Д.Е., Божбанов А.Ж., Габдуллина Е.Ж. Влияние инулина на показатели качества ферментированного напитка на основе сырной сыворожки	25
Асылбекова Д.Д., Токтибаева Х.Р., Калдыбаева А.Р., Журхабаева Л., Тукибаева А.С. Электрохимическое поведение фосфористой кислоты на платиновом электроде и её гальваностатическое анодное окисление с использованием свинцового анода.....	39
Ауешов А.П., Ескибаева Ч.З., Ибраева А.М., Алжанов К.Б., Джолдасова Э.М. Исследование адсорбционных характеристик кремнезёма, полученного из серпентинита комбинированным кислотно-щелочным методом	58
Галымжан А., Кожабеков С., Куанышев А., Сансызбай Н., Абдрасилова А. Влияние состава сополимеров октадецилметакрилата и малеинового ангидрида на реологические свойства нефти	78
Дарменбаева А.С., Rajasekharan R. Исследование структурных свойств наноцеллюлозных мембран, полученных из агроотходов.....	95
Дмитриева Е.А., Медведев А., Кемелбекова А. Разработка низкотемпературного газового сенсора на основе тонких плёнок SnO_2 и прецизионной измерительной системы для регистрации высокоомных сопротивлений.....	109
Джелдыбаева И.М., Каирбеков Ж., Есеналиева М.З., Досумова Б.Т., Суймбаева С.М. Влияние ультразвуковой обработки на структуру и физико-химические свойства гуминовых кислот бурого угля	125
Джумадилов Т.К., Химэрсэн Х., Имангазы А. Особенности сорбции ионов тяжёлых металлов интерполимерными системами, сформированными из однородных полиэлектролитов.....	140
Жылкыбек М., Оразбекова Р.С., Еркибаева М.К., Айдарова А.О., Байжуманова Т.С. Co–Mg оксидные катализаторы для глубокого окисления метана: синтез, структура и каталитическая активность	154
Имангалиева Б., Рахметова Г., Досанова Б., Совет Н. Технология получения биологического удобрения путём гидролизной обработки полимерных отходов	169
Карар М.Е., Бейсенбаев О.К., Алибеков Р.С., Роснах Шамсудин. Получение крахмала из риса «Байдалы» и картофеля сорта «Гала» и его применение в биоразлагаемых материалах	186
Кудайбергенова Г.Н., Азимбаева Г.Е., Камысбаева А.К., Изтелеу Б.М., Курбанбаева Н.М. Жирнокислотный состав цветков топинамбура	204

Масенова А.Т., Сасс А.С., Торлопов И.И., Рахметова К.С., Малгаждарова М.К. Регулирование поверхностной кислотности Zn–Me-модифицированных формованных ZSM-5–Al ₂ O ₃ катализаторов алкилирования	219
Нурисламов Р.М., Нефедов А.Н., Бейсенбаев Д.К., Леонтьева К.А., Имашев Б.К. Влияние оксидных плёнок на коррозионную стойкость железа: моделирование в COMSOL Multiphysics 6.2	238
Нурлыбаев О., Кудайбергенова Р., Таубаева Р., Бекаулова А., Сейтманбетова А. Текстурные свойства бентонит–углеродных композитов после карбонизации в присутствии хлоридов металлов	250
Пузикова Д.С., Бессонова Г.В., Кензин Н.Р., Журинов М.Ж., Торлопов И.И. Моделирование процесса кавитации нефтесодержащих сред в различных гидродинамических конструкциях.....	263
Рахимов А., Жаникулов Н., Таймасов Б., Потапова Е., Сарсенбаев Б. Расчёт состава сырьевой шихты для цементного клинкера с добавлением отходов переработки свинцового шлака	285
Серикбаева К.Т., Чалов Т.К., Садыков К.А. Сорбция рубидия из водных растворов анионитами ВЭМЭА–АГЭ–ПЭИ.....	299
Сейтказинова А.Р., Нажипкызы М., Елеуов М.А., Жапаров А.А., Калыбаева А.К. Пористый активированный углерод, полученный из апельсиновой кожуры.....	322
Самадун А.И., Таусарова Б.Р., Асракулова Д.И., Нурмуханбетова Д.Е. Биоразлагаемые упаковочные плёнки с наночастицами меди: влияние концентрации NaBH ₄ на морфологию и антигрибковые свойства	334
Хамитова Т.О., Тянах С., Болатбай А.Н., Ма Фэньюнь, Айтбекова Д.Е. Связь геологического генезиса с молекулярными и оптическими характеристиками гуминовых кислот из угольных отходов	351
Рыскалиева Д.К., Байбуланова А.А., Есенаманова М.С., Мустафина А.Ж., Самат А.С. Загрязнение атмосферы промышленного города сероводородом: потенциальная роль полей испарения (Атырау, Казахстан)	370
Талгатов Э.Т., Заманбекова А.Т., Абилямагжанов А.З., Ауезханова А.С., Ахметова С.Н. Полимер-модифицированные нанесённые катализаторы окисления циклогексана пероксидом водорода	389

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ORANGE PEEL-DERIVED POROUS ACTIVATED CARBON

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Abstract. The conversion of renewable biomass waste into porous carbon materials is a promising approach for both efficient waste valorization and the development of environmentally sustainable electrode materials for modern electrochemical energy-storage systems. Food waste, particularly orange peel, is of considerable interest as an abundant, renewable, and carbon-rich precursor. This study aimed to synthesize porous carbon materials from orange peel waste and to determine the effects of carbonization and chemical activation temperatures on their structural, elemental, and electrochemical properties. Porous carbon materials were prepared using a two-step process consisting of pre-carbonization followed by chemical activation. Carbonization was performed at 500, 600, and 700 °C, while activation was conducted at 700, 800, and 900 °C. The morphology and elemental composition of the resulting materials were characterized using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX), respectively, while their carbon structure was investigated

by Raman spectroscopy. The electrochemical characteristics and specific capacitance of the prepared electrode materials were also evaluated. SEM analysis revealed that increasing the activation temperature promoted the development of a heterogeneous and well-developed porous structure. EDX analysis confirmed carbon as the predominant element (83–90 wt%), with an oxygen content of 10–14 wt% and a small amount of residual potassium. Raman spectroscopy demonstrated that the carbon structure varied depending on the synthesis conditions. Among the investigated materials, OPA500/700 exhibited the best electrochemical performance, delivering a specific capacitance of 367.5 F g⁻¹ at 0.1 A g⁻¹ and 259.6 F g⁻¹ at 2 A g⁻¹, corresponding to retention of more than 70% of the initial value. The results demonstrate that orange peel waste is a promising, readily available, and renewable precursor for producing high-capacitance porous carbon electrodes for supercapacitor applications.

Keywords: food waste, activated carbon, supercapacitor, orange peel, electrode material.

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Аннотация. Жаңартылатын биомасса қалдықтарынан кеуекті көмір материалдарын алу қалдықтарды тиімді кәдеге жарату және заманауи электрохимиялық энергия сақтау жүйелеріне арналған экологиялық тұрақты электродтық материалдарды әзірлеу тұрғысынан өзекті бағыт болып табылады. Тағам қалдықтары, соның ішінде апельсин қабығы, көміртекке бай, қолжетімді және жаңартылатын шикізат көзі ретінде ерекше қызығушылық тудырады. Зерттеудің мақсаты — апельсин қабығы негізінде кеуекті көмір материалдарын алу және карбонизация мен химиялық белсендіру температураларының олардың құрылымдық, элементтік және электрохимиялық қасиеттеріне әсерін бағалау. Кеуекті көмір материалдары екі сатылы әдіспен алынды: алдын ала карбонизация және кейінгі химиялық белсендіру. Карбонизация 500, 600 және 700 °C температураларда, ал белсендіру 700, 800 және 900 °C температураларда жүргізілді. Алынған үлгілердің морфологиясы мен элементтік құрамы SEM және EDX әдістерімен, ал көміртекті құрылымның ерекшеліктері Раман спектроскопиясы арқылы зерттелді. Сонымен қатар электродтық материалдардың электрохимиялық сипаттамалары мен меншікті сыйымдылығы анықталды. SEM деректері белсендіру температурасы жоғарылаған сайын кеуекті құрылымның айқын әрі біртекті емес сипатқа ие болғанын көрсетті. EDX талдауы бойынша көміртек мөлшері 83–90 масс.%, ал оттек мөлшері 10–14 масс.% аралығында болды; кейбір үлгілерде аз мөлшерде қалдық калий анықталды. Раман спектроскопиясы көміртекті құрылымның реттелу дәрежесі карбонизация және белсендіру жағдайларына тәуелді екенін көрсетті. Зерттелген үлгілердің ішінде ОРА500/700 ең жоғары электрохимиялық көрсеткіштерге ие болды: оның меншікті сыйымдылығы 0,1 А/г ток тығыздығында 367,5 Ф/г, ал 2 А/г кезінде 259,6 Ф/г болды. Осылайша, ток тығыздығы артқан кезде бастапқы сыйымдылықтың 70%-дан астамы сақталды. ОРА500/700 үлгісінің меншікті бет ауданы 1968 м²/г құрады. Алынған нәтижелер апельсин қабығы қалдықтарын суперконденсаторларға арналған жоғары сыйымдылықты кеуекті көмір электродтарын өндіруге қолжетімді және жаңартылатын шикізат ретінде пайдаланудың перспективалы екенін көрсетті.

Түйін сөздер: тағам қалдықтары, белсендірілген көмір, суперконденсатор, апельсин қабығы, электрод материалы

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ПОРИСТЫЙ АКТИВИРОВАННЫЙ УГЛЕРОД, ПОЛУЧЕННЫЙ ИЗ АПЕЛЬСИНОВОЙ КОЖУРЫ

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Аннотация. Получение пористых углеродных материалов из возобновляемых отходов биомассы является актуальным направлением с точки зрения эффективной утилизации отходов и разработки устойчивых электродных материалов для современных систем электрохимического накопления энергии. Пищевые отходы, в частности апельсиновая кожура, представляют интерес как доступный, возобновляемый и богатый углеродом источник сырья. Целью работы являлись получение пористых углеродных материалов из апельсиновой кожуры и оценка влияния температур карбонизации и химической активации на их структурные, элементные и электрохимические свойства. Материалы получали двухстадийным методом, включающим предварительную карбонизацию и последующую химическую активацию. Карбонизацию проводили при 500, 600 и 700 °С, а активацию - при 700, 800 и 900 °С. Морфологию и элементный состав образцов исследовали методами SEM и EDX, а структурные характеристики углеродных материалов - методом рамановской спектроскопии. Также определяли электрохимические характеристики и удельную ёмкость полученных электродных материалов. По данным SEM, повышение температуры активации сопровождалось развитием более выраженной и неоднородной пористой структуры. Результаты EDX показали, что содержание углерода составляло 83–90 масс.%, а кислорода - 10–14 масс.%; в отдельных образцах выявлено небольшое количество остаточного калия. Рамановская спектроскопия показала зависимость

степени структурной упорядоченности углеродных материалов от условий карбонизации и активации. Наиболее высокие электрохимические показатели получены для образца ОРА500/700: удельная ёмкость составила 367,5 Ф/г при плотности тока 0,1 А/г и 259,6 Ф/г при 2 А/г, что соответствует сохранению более 70% исходной ёмкости. Удельная поверхность данного образца составила 1968 м²/г. Полученные результаты свидетельствуют о перспективности использования отходов апельсиновой кожуры в качестве доступного и возобновляемого сырья для производства высокоёмкостных пористых углеродных электродов для суперконденсаторов.

Ключевые слова: пищевые отходы, активированный уголь, суперконденсатор, кожура апельсинов, материал электрода

Introduction. The rapid growth of the global population and the increasing demand for energy have led to a serious energy crisis. The generation of energy from non-renewable resources over the past centuries has caused significant environmental damage. To address issues related to excessive fossil fuel consumption, severe environmental pollution, and global warming, the development of renewable and eco-friendly energy resources has become an urgent priority. Considerable efforts have been devoted to the production and storage of energy from renewable materials with minimal CO₂ emissions. Among various energy types, electrochemical energy is considered one of the cleanest and most sustainable solutions. As a safe and environmentally friendly energy form, electrochemical energy can effectively contribute to solving environmental pollution problems. Significant research has focused on enhancing both the generation and storage of electrochemical energy from renewable sources.

Literary review. Electrochemical energy storage devices are typically classified into three types: batteries, supercapacitors, and hybrid systems (Ehsani et al., 2020). Modern electronic devices often require rapid energy delivery that conventional batteries cannot provide due to their limited charge–discharge rates. In such cases, supercapacitors are preferred. Although supercapacitors operate on principles similar to conventional capacitors, their fabrication methods and performance characteristics are significantly different. They combine the energy storage capacity of batteries with the high-power density and fast charge–discharge properties of capacitors, offering advantages such as long cycle life, compactness, cost-effectiveness, and rapid energy transfer (Ajay et al., 2021).

Based on the energy storage mechanism, supercapacitors can be classified into two main types: electric double-layer capacitors (EDLCs), which utilize carbon-based materials, and pseudocapacitors, which employ metallic compounds and conductive polymers. Pseudocapacitors store and deliver energy through reversible redox reactions at the electrode–electrolyte interface, whereas EDLCs store energy via electrostatic attraction of opposite charges at the interface. EDLCs exhibit exceptional cyclic stability, high performance, low cost, and straightforward preparation methods (Kumar et al., 2022; Singh et al., 2025; Patel et al., 2024).

The performance of EDLCs strongly depends on the electrode materials, particularly their porosity, which determines the effective surface area available for charge storage

(Lobato-Peralta et al., 2024). Carbon-based materials, including activated carbon have been extensively studied as electrode materials for EDLCs. Among these, activated carbon has attracted significant attention due to its abundant raw material sources, excellent electrochemical properties, and scalability for industrial applications has attracted significant attention due to the wide availability of its precursor materials and its suitable electrochemical properties for EDLC applications.

Activated carbon electrodes can be synthesized from biowaste, making them economically viable and environmentally sustainable. offering a practical way to make use of biomass residues. Chemical activation is the most widely used method to produce activated carbon, with common activating agents including ZnCl_2 (Mardina, et al., 2025), KOH (Li et al., 2016; Nazhipkyzy et al., 2024; Arkhipova et al., 2022), and H_3PO_4 (Fan et al., 2025; Quesada-Plata et al., 2016). Various biomass precursors have been employed, such as rice husk (Anusonthiwong et al., 2025; Gao et al., 2015), corn cobs (Dong et al., 2024; Andem et al., 2024), nut shells (Zou et al., 2024), and fruit peels (Yang et al., 2023; Ahmad et al., 2024; Nazhipkyzy et al., 2024). Carbon materials derived from biowaste often demonstrate excellent electrochemical properties, with high specific surface area and well-developed porosity being key factors for enhanced supercapacitor performance.

Therefore, the utilization of biowaste-derived activated carbon represents a sustainable, promising approach for electrode materials in supercapacitor applications.

Orange peel is one of the fruit-based biomass residues that can be used to produce activated carbon electrodes. As a readily available food waste, it can be converted into porous carbon through chemical activation. KOH -activated carbon prepared from orange peel has previously been investigated as an electrode material for supercapacitors (Ajay et al., 2021). The characteristics of biomass-derived activated carbon are closely related to the activation conditions, particularly the temperature, which can influence the morphology, pore structure, and electrochemical behavior of the resulting material. For this reason, examining the effect of activation temperature is useful for understanding how the properties of orange-peel-derived carbon change during its preparation.

In this work, activated carbon was prepared from orange peel using KOH activation at 700, 800, and 900 °C. The resulting materials were examined using morphological, elemental, structural, and electrochemical analyses to determine how the preparation conditions affected their properties and their performance as supercapacitor electrode materials.

Materials and methods. The preparation of porous carbon materials was carried out in two stages: preliminary carbonization and chemical activation. To determine the optimal conditions for carbonization, parameters such as temperature and duration were systematically investigated.

Pyrolysis is an important method for the decomposition of organic matter—typically cellulose, hemicellulose, and lignin—from biowaste and its conversion into unique carbon materials. Carbon materials from various biowaste sources can be obtained either via a single-step high-temperature carbonization or a two-step process involving preliminary carbonization followed by activation. The activation process generates a high volume of pores—micro-, meso-, and macropores—which are differently associated with electrochemical properties.

For preliminary carbonization, orange peels collected as household food waste after consumption of oranges in Kazakhstan were used as the raw material. The raw materials were washed several times with distilled water to remove impurities. The washed material was dried at 80 °C for 16 hours in a drying oven. The dried material was then mechanically ground to homogeneity using a porcelain mortar and pestle.

Experimental studies showed that the optimal temperatures for preliminary carbonization under an inert argon atmosphere were 500, 600, and 700 °C, with an average argon flow rate of approximately 0.005 L/min. Heating was performed at a controlled rate of 7.5 °C/min in all experiments. To determine the optimal duration, preliminary carbonization was carried out for 60–120 minutes, and it was found that 2 hours was optimal for producing porous carbon materials. The carbonization time was fixed at 2 h based on conditions reported for biomass-derived carbon materials and was kept constant for all samples.

Thus, the optimal conditions for synthesizing porous carbon material (carbonizate) from orange waste were established as follows: carbonization temperature – 500, 600, 700 °C; duration – 2 hours; heating rate – 7.5 °C/min; argon flow rate – ~0.005 L/min.

Chemical activation of the pre-carbonized orange peel samples was performed under the following conditions: activation temperature ranged from 700 to 900 °C, KOH was used as the chemical activating agent with a carbonizate-to-KOH ratio of 1:2, argon gas served as the inert atmosphere, and the activation duration was 2 hours. Activation was carried out in the same setup used for carbonization. The resulting activated carbon (AC) was cooled to room temperature and ground. To remove residual KOH, the product was washed with 1 M HCl, followed by multiple washes with distilled water until neutral pH was achieved. Finally, the product was dried at 60 °C for 16 hours in a drying oven.

The carbonization yield was calculated using the following equation:

$$Y_s(\%) = \frac{m_c}{m_o} \times 100 \quad (1)$$

where (m_c) is the mass of the carbonized material (g) and (m_o) is the initial mass of orange peel (g). The carbonization yields were 27.3%, 24.7%, and 32.2% at 500, 600, and 700 °C, respectively.

The activation yield was calculated using the following equation:

$$Y_a(\%) = \frac{m_a}{m_{c,a}} \times 100 \quad (2)$$

where (m_a) is the mass of the activated carbon (g) and ($m_{c,a}$) is the mass of carbonized material used for activation (g). For the samples carbonized at 500 °C, the activation yields at 700, 800, and 900 °C were 51.8%, 48.3%, and 45.2%, respectively. For the samples carbonized at 600 °C, the corresponding yields were 59.8%, 60.5%, and 53.3%, respectively. For the samples carbonized at 700 °C, the activation yields were 42.9%, 42.5%, and 42.0% at 700, 800, and 900 °C, respectively.

The obtained activated carbon materials were characterized by scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), Raman spectroscopy, and nitrogen adsorption–desorption analysis. SEM images were obtained using a JSM-6490LA scanning electron microscope (JEOL Ltd., Tokyo, Japan), while EDX analysis was performed using an EDX system (JEOL Ltd., Tokyo, Japan). Raman spectra were recorded using a SENTERRA II Raman spectrometer (Bruker Optik GmbH, Ettlingen, Germany) with 532 nm excitation. The specific surface area of the selected sample was determined from N₂ adsorption–desorption isotherms using a TriStar II 3020 analyzer (Micromeritics Instrument Corp., Norcross, GA, USA).

The electrodes were prepared as follows: activated carbon (75 wt%) was mixed with polyvinylidene fluoride (PVDF) as a binder (15 wt%), acetylene black (10 wt%), and 2–3 mL of N-methyl-2-pyrrolidone. The resulting mixture was thoroughly ground in an agate mortar until a homogeneous mass was obtained. The suspension was then rolled into thin sheets, cut into pieces on titanium foil with an area of 2 cm², and dried at 130 °C for 12 hours.

An R-40X potentiostat was used to determine the capacitive characteristics of the samples. A 6 M KOH solution was selected as the electrolyte for the two-electrode cell. Two identical electrodes, separated by a separator (Whatman™ filter paper, Cat. No. 1001-110), were placed in a symmetric electrochemical cell. Cyclic voltammetry (CV) tests were conducted at scan rates ranging from 5 to 160 mV s⁻¹ within a potential window of 0.0–1.0 V. Galvanostatic charge-discharge tests were performed at current densities of 0.1, 0.25, 0.5, 1.0, and 2.0 A g⁻¹ within a potential window of 0.0–1.0 V.

Specific capacitance (C_s) was calculated from the cyclic voltammetry curves of the two-electrode cell using the following equation:

$$C_s = \frac{A}{m \times k \times (V_2 - V_1)} \quad (3)$$

where A is the integral of the CV curve (C);

m is the mass of the active material of a single electrode (g);

k is the scan rate (mV s⁻¹);

$V_2 - V_1$ is the potential window (V).

Specific capacitance was also calculated from the galvanostatic charge-discharge curves of the two-electrode cell using the equation:

$$C_s = \frac{2I \times t}{m \times (V_2 - V_1)} \quad (4)$$

where I is the current density (A g⁻¹);

t is the discharge time (s);

m is the mass of the active material of a single electrode (g);

$V_2 - V_1$ is the potential window (V).

Results

To investigate the structural and morphological characteristics of the obtained activated carbon materials, studies were conducted using scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), and Raman spectroscopy.

The effect of carbonization and subsequent activation temperatures on the morphology and microstructure of activated carbons derived from orange peel was investigated using scanning electron microscopy (SEM). The orange peel was carbonized at 500, 600, and 700 °C, and the resulting carbon materials were subsequently activated at 700, 800, and 900 °C (Figure 1, a-i).

For the samples carbonized at 500 °C and activated in the range of 700–900 °C, the SEM images revealed a higher number of pores with increasing activation temperature and a more heterogeneous pore distribution. In addition, the proportion of smaller pores increased, and the material surface exhibited noticeable morphological changes, forming a more wrinkled and rough structure.

SEM images of the samples carbonized at 600 °C (OPA600/700 and OPA600/800) showed a large number of pores of various sizes ranging from approximately 4 to 13 μm . In contrast, the OPA600/900 sample demonstrated a more heterogeneous structure with a predominance of amorphous carbon and the presence of smaller pores with sizes ranging from 880 nm to 1.5 μm in the synthesized material.

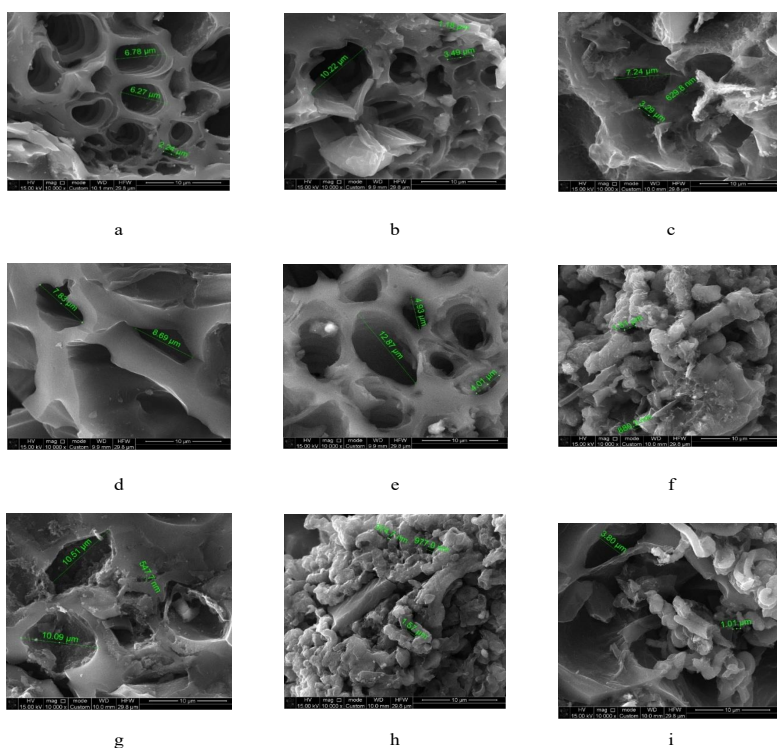


Figure 1 — SEM images of activated carbon samples derived from orange peel: a) OPA500/700; b) OPA500/800; c) OPA500/900; d) OPA600/700; e) OPA600/800; f) OPA600/900; g) OPA700/700; h) OPA700/800; i) OPA700/900

All images of samples OPA700/700, OPA700/800, and OPA700/900 revealed irregular and non-uniform porous structures.

Table 1 shows the elemental composition of activated carbons derived from orange peel.

Table 1 — Elemental composition of activated carbons derived from orange peel (EDX analysis)

Sample	Carbon C (wt%)	Oxygen O (wt%)	Potassium K (wt%)
OPA500/700	88.12	11.88	—
OPA500/800	89.91	10.09	—
OPA500/900	87.05	11.12	1.83
OPA600/700	87.89	11.34	0.77
OPA600/800	86.89	11.72	1.39
OPA600/900	84.04	14.38	1.58
OPA700/700	88.18	11.11	0.71
OPA700/800	85.02	11.84	3.14
OPA700/900	83.65	13.54	2.81

The elemental composition of the activated carbon samples derived from orange peel was investigated using energy-dispersive X-ray spectroscopy (EDX).

The results indicate that the carbon content in the activated carbons varies between 83 and 90 wt%. Oxygen content ranges from 10 to 14 wt%, confirming that carbon and oxygen are the main elements present in all synthesized samples.

For the samples carbonized at 500 °C, the carbon content remained relatively high (87–90 wt%), while oxygen was detected at approximately 10–12 wt%. A small amount of potassium appeared in the sample activated at 900 °C, likely originating from the activating agent used during the chemical activation process.

In the samples carbonized at 600 °C, a gradual decrease in carbon content was observed with increasing activation temperature, accompanied by a slight increase in oxygen and potassium concentrations. The carbon content decreased from 87.9 wt% to 84.0 wt%, while oxygen increased up to 14.38 wt%.

A similar trend was observed for the samples carbonized at 700 °C. The carbon content decreased from 88.18 wt% to 83.65 wt% as the activation temperature increased from 700 °C to 900 °C, whereas oxygen content increased to approximately 13.5 wt%. Potassium was detected in all samples with concentrations up to 3 wt%.

Figure 2 presents the Raman spectra of activated carbon samples prepared at different carbonization (500–700 °C) and activation (700–900 °C) temperatures. All samples show two characteristic peaks: the D band at around 1350 cm⁻¹ and the G band at approximately 1580–1600 cm⁻¹. And all samples except OPA 600/700 and OPA 700/700 show a 2D peak at approximately 2700 cm⁻¹. As can be seen in the figure, there are virtually no defects in the OPA 500/700 °C sample. Of all the samples, the 2D peak is clearly visible in the OPA 700/900 sample.

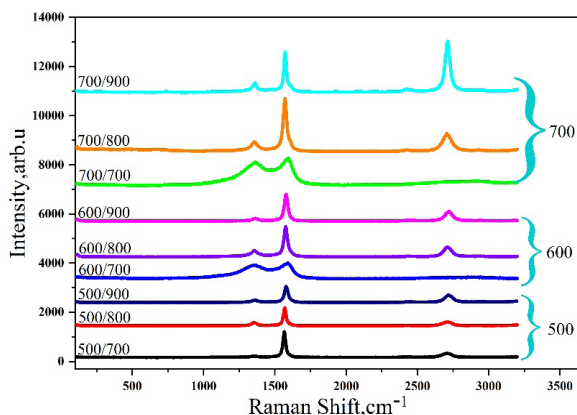


Figure 2 — Raman spectra of samples

Table 2 presents the Raman parameters of activated carbons derived from orange peel. Samples carbonized at 600 °C exhibit the highest ID/IG ratios (0.89–0.94), indicating a predominantly amorphous carbon structure. In contrast, samples carbonized at 500 °C and 700 °C and activated at 900 °C show relatively lower ID/IG values, suggesting the development of more ordered graphitic domains. The increased intensity of the 2D band ($\sim 2700\text{ cm}^{-1}$) in the OPA500/900 and OPA700/900 samples further supports the formation of a more graphitized carbon structure at higher activation temperatures.

Table 2 — Raman parameters of activated carbons derived from orange peel

Sample	D band (cm^{-1})	G band (cm^{-1})	I_D/I_G	Structural interpretation
OPA500/700	~ 1350	~ 1560	0.17	Higher graphitization
OPA500/800	~ 1350	~ 1560	0.62	More disordered structure
OPA500/900	~ 1350	~ 1560	0.27	Relatively ordered carbon structure
OPA600/700	~ 1350	~ 1560	0.93	Highly amorphous structure
OPA600/800	~ 1350	~ 1560	0.94	Highly disordered carbon
OPA600/900	~ 1350	~ 1560	0.89	Amorphous carbon structure
OPA700/700	~ 1360	~ 1580	0.86	High structural disorder
OPA700/800	~ 1360	~ 1580	0.89	High defect density
OPA700/900	~ 1360	~ 1580	0.48	Higher graphitic ordering

The CV and GCD curves of the activated carbon samples derived from orange peel are shown in Figure 3.

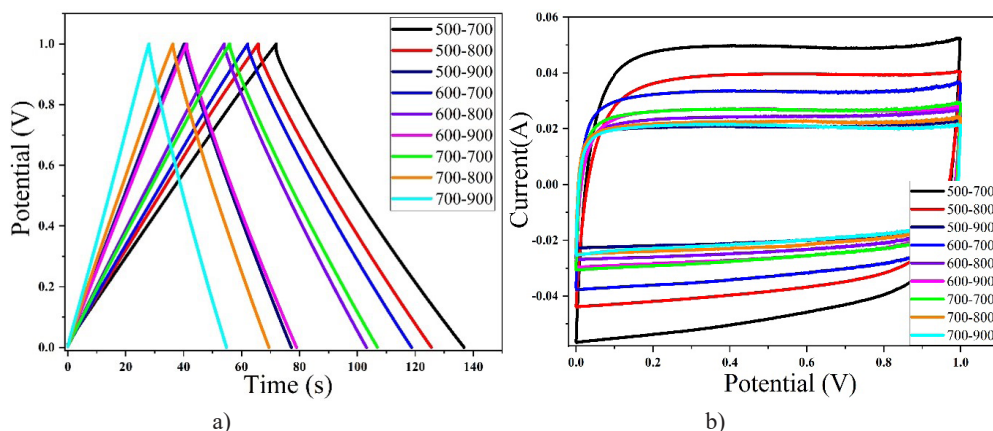


Figure 3 — Electrochemical characterization of the samples: a) Galvanostatic charge–discharge curves of the electrode materials at 2 A g^{-1} ; b) Cyclic voltammetry at 160 mV s^{-1}

The electrodes were evaluated using the galvanostatic charge–discharge (GCD) method at current densities of 0.1 , 0.25 , 0.5 , 1 , and 2 A g^{-1} . Figure 3a shows the GCD curves of the samples at 2 A g^{-1} .

The electrochemical performance of the electrodes was assessed using cyclic voltammetry (CV) at scan rates of 5 , 10 , 20 , 40 , 80 , and 160 mV s^{-1} . The results at 160 mV s^{-1} are shown in Figure 3, b.

The highest specific capacitance of 281.9 F g^{-1} at a scan rate of 5 mV s^{-1} , and 213.2 at 160 mVs^{-1} is achieved when the orange peel is first carbonized at 500°C and then activated at 700°C .

The highest specific capacitance of 367.5 F g^{-1} at a current density of 0.1 A g^{-1} was achieved for the electrode sample OPA500/700. The specific capacitance of the same sample at 2 A g^{-1} was 259.6 F g^{-1} , corresponding to more than 70% capacitance retention at increased current density. The average capacitance retention for the other electrodes in this series, synthesized from orange peel, was 94.4% , indicating high stability under high charge–discharge current densities.

OPA500/700 showed the highest specific capacitance among the prepared samples. Therefore, this sample was selected for nitrogen adsorption–desorption analysis. Its specific surface area was $1968 \text{ m}^2 \text{ g}^{-1}$.

Discussion

The SEM analysis showed that with increasing activation temperature, the porosity of the material significantly increased, and the surface structure became more complex. Overall, the results indicate that increasing both carbonization and activation temperatures significantly influences the microstructure of the carbon materials, promoting the development of porosity, altering pore size distribution, and increasing the structural complexity of the surface.

The EDX results confirm that carbon is the dominant element in all synthesized activated carbons, while oxygen and residual potassium originate from surface

functional groups and activation reagents. The carbon content confirms the successful carbonization of the biomass precursor. The oxygen content can be attributed to the presence of oxygen-containing functional groups on the carbon surface. The detected oxygen may be associated with oxygen-containing surface functional groups; however, EDX analysis alone does not allow the chemical or structural state of oxygen to be determined. The potassium detected in the samples may be attributed to incomplete neutralization or washing of the alkaline activating agent after the activation process.

The D-band is associated with defects and structural disturbances in the carbon matrix. The G-band is associated with vibrations of sp^2 -bonded graphitic carbon atoms. The shape and position of the 2D peak provide information about the degree of graphitization and the multilayered nature of the material's structure.

The presence of the D and G peaks indicates that all samples contain both amorphous and graphitic carbon structures. Although the peak positions are identical in all samples, slight differences in peak intensity indicate that heat treatment has affected the structural properties.

The Raman analysis demonstrates that the structural ordering of the activated carbons strongly depends on both carbonization and activation temperatures.

Overall, the combined SEM, EDX, and Raman analyses demonstrate that increasing carbonization and activation temperatures significantly affect the microstructure, surface composition, and structural ordering of the activated carbons derived from orange peel. Higher activation temperatures promote the development of porous structures and may enhance the formation of graphitic domains within the carbon matrix.

Figure 3 shows that the CV curves of the prepared electrodes retain a quasi-rectangular shape, which is characteristic of electric double-layer capacitors. No distinct redox peaks are observed within the investigated potential range. This indicates that charge storage occurs mainly through the formation of an electric double layer at the electrode–electrolyte interface, although a minor contribution from surface functional groups cannot be completely excluded.

The higher capacitance observed at lower scan rates is associated with more effective ion transport within the porous carbon structure. When the potential is varied slowly, electrolyte ions have sufficient time to enter the pores and reach a larger portion of the internal electrode surface. At higher scan rates, access to narrow or less accessible pores becomes limited. As a result, only part of the available surface contributes to charge storage, leading to a decrease in the measured capacitance.

The galvanostatic charge–discharge curves are nearly symmetric and triangular and show an approximately linear relationship between potential and time. These features are consistent with predominantly capacitive behaviour and good reversibility during repeated charging and discharging. Among the investigated samples, OPA500/700 exhibited the longest discharge time at the same current density, in agreement with its higher specific capacitance.

The activation temperature has a direct influence on the morphology and pore structure of biomass-derived activated carbon and, therefore, on its electrochemical properties. During KOH activation, increasing the temperature intensifies the interaction

between KOH and the carbon matrix, resulting in the formation and enlargement of pores. At excessively high temperatures, however, intensive etching may damage the carbon framework and reduce the number of pores that are suitable for electrolyte-ion transport and charge storage. When the temperature becomes too high, however, excessive etching of the carbon framework can occur, which may adversely affect the resulting porous structure. A similar temperature dependence was reported for corncob-derived activated carbon (Dong et al., 2024), where changes in KOH activation temperature altered the pore structure; micropores contributed to charge storage, while mesopores favored electrolyte-ion transport. Thus, the activation temperature needs to be controlled to promote pore formation without causing excessive modification of the carbon framework. The differences in morphology, structural characteristics, and electrochemical behavior observed for the samples activated at 700, 800, and 900 °C may therefore be related to temperature-dependent changes occurring during KOH activation.

The specific surface area of OPA500/700 was 1968 m² g⁻¹, confirming the highly porous nature of the material. A large surface area can increase the contact between the carbon surface and electrolyte ions, which may contribute to the high specific capacitance observed for this sample.

To evaluate the electrochemical performance of the obtained activated carbon, its specific capacitance was compared with those of biomass-derived activated carbon electrodes reported in the literature (Table 3).

Table 3 — Comparison of the electrochemical performance of this work with reported biomass-derived activated carbons.

Biomass precursor	Activation / preparation method	BET surface area, m ² g ⁻¹	Electrolyte	Specific Capacitance (F g ⁻¹) and current density	Ref.
Rice husk	KOH activation; carbonization at 800 °C; hydro-thermal post-treatment	1847	3 M H ₂ SO ₄	242.1 F g ⁻¹ at 0.5 A g ⁻¹	Anusonthiwong et al., 2025
Corn cob	KOH activation; carbonization/activation at 700–900 °C;	1945,7	6 M KOH	260 F g ⁻¹ at 1 A g ⁻¹	Dong et al., 2024
Rice husk	KOH activation; carbonization	3145	6 M KOH	367 F g ⁻¹ at 5 mVs ⁻¹	Gao et al., 2015
Corn cob	Hydrothermal treatment + H ₃ PO ₄ activation; carbonization at 400 °C	68.54	—	41 F g ⁻¹ at 1 A g ⁻¹	Andem et al., 2024,
Pecan shell	Self-activation/ carbonization at ~1050 °C	1554	6 M KOH	269 F g ⁻¹ at 2 A g ⁻¹	Zou et al., 2024
Watermelon peel	H ₃ PO ₄ -assisted carbonization; 700 °C, 2 h; N/P-doped	—	6 M KOH	135 F g ⁻¹ .2 at 1 A g ⁻¹	Yang et al., 2023

Melon peel	Pretreatment + activation; hydrothermal pretreatment	500	LiCF ₃ SO ₃ /EMIMCF ₃ SO ₃ gel electrolyte	161 F g ⁻¹ at 1 A g ⁻¹	Ahmad et al., 2024
Orange peel	KOH activation	—	0,5 M H ₂ SO ₄	41,6 F g ⁻¹ at 1 A g ⁻¹	Madhusre et al., 2023
Orange peel	Carbonization; KOH activation	1968	6 M KOH	259.6 F g ⁻¹ at 2 A g ⁻¹	This study

As shown in Table 3, the obtained activated carbon exhibits a specific capacitance of 259.6 F g⁻¹ at a current density of 2 A g⁻¹ in 6 M KOH. This value is comparable to those reported for several biomass-derived activated carbon electrodes under similar electrochemical conditions.

Thus, electrode materials were fabricated from activated carbons synthesized via chemical activation of carbonizates at 700 °C, 800 °C, and 900 °C. All samples exhibited good capacitive performance when used as supercapacitor electrodes.

Conclusion

In this study, porous activated carbons were obtained from orange peel waste by combining carbonization with subsequent KOH activation. The carbonization temperature was varied between 500 and 700 °C, while activation was performed at 700, 800, and 900 °C. This allowed us to examine how the selected synthesis conditions affected the structure and electrochemical behavior of the resulting carbons.

SEM observations showed that the materials developed a porous structure during activation. Changes in activation temperature were accompanied by noticeable differences in the pore morphology, including the appearance of a more heterogeneous network with a larger number of smaller pores. EDX analysis confirmed that carbon was the main component of all samples, with a content of 83–90 wt%. Oxygen and small amounts of residual potassium were also present. The oxygen detected by EDX may be related to oxygen-containing groups on the carbon surface, although this technique does not provide information about their specific chemical or structural state. The Raman results also showed differences in the degree of structural ordering depending on the synthesis conditions. In particular, the samples activated at higher temperatures showed a more pronounced graphitic character.

All of the obtained carbons showed electrochemical behavior typical of electric double-layer capacitors. The best result was obtained for OPA500/700, which delivered 367.5 F g⁻¹ at 0.1 A g⁻¹ and 259.6 F g⁻¹ at 2 A g⁻¹. The relatively high capacitance retained at the higher current density also indicates good rate performance of this sample.

The differences between the samples suggest that the carbonization and activation temperatures affect both the carbon structure and its electrochemical response. During KOH activation, temperature influences the development of the porous carbon framework, which in turn affects the accessibility of the electrode surface to electrolyte ions. At the same time, excessive activation can alter the pore structure of the carbon matrix. Under the conditions investigated here, the OPA500/700 sample provided the highest specific capacitance. Its combination of porous structure and electrochemical performance makes this material a promising candidate for supercapacitor electrodes.

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