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Production and optimization of activated carbon from plant waste with high specific surface area for moisture-saving applications in agriculture

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ABSTRACT

In conditions of water shortage, sustainable agricultural development requires the use of water-saving technologies, including the use of water-retaining substrates based on activated carbon. In this work, the textural and adsorption characteristics of activated carbon obtained from plant waste were studied at different mass ratios of the sorbent and KOH (1:1, 1:2, 1:3 and 1:4). The aim of the study was to determine the optimal activation conditions for creating a material with a high specific surface area and a developed porous structure. The results showed that the largest pore volume (1.6 cm^3/g) and a high degree of microporosity are achieved at a ratio of 1:3, which is confirmed by the analysis of pore distribution using the DFT and BJH methods. FTIR spectroscopy revealed the presence of functional groups (O–H, C=O and C–O) that contribute to water conservation. The differential pore volume distribution (dv(r), $cm^3/Å/g$) also demonstrated that at a ratio of sorbent and KOH (1:3), the sample structure optimally combines micropores and mesopores, which increases the adsorption capacity of carbon.

Keywords: activated carbon, agricultural waste, water-retaining substrate, porous structure, adsorption properties

1. Introduction

Ensuring sustainable agricultural production in the context of climate change and water shortage is one of the most important challenges for the agro-industrial sector worldwide [1-3]. In Kazakhstan, where more than 80% of agricultural land faces a deficit of atmospheric precipitation, the development of technologies that help conserve moisture and improve plant growing conditions is of particular importance. In this regard, effective water management and the introduction of moisture-saving technologies is a key area for the sustainable development of the agricultural sector and ensuring food security in the country. One of the promising approaches to solving the problem of irrigation water shortage is the use of water-retaining substrates based on nanostructured sorbents [4]. These materials have a unique ability to retain significant volumes of water and nutrients, creating favorable conditions for plant growth and simultaneously reducing the need for irrigation [5-7]. The most accessible source for the production of such sorbents is plant waste, such as stems, husks and shells, which can be converted into activated carbons with a high specific surface area and developed porous structure [8-12]. The process of obtaining activated carbon includes the stages of carbonization and activation of biomass using potassium hydroxide (KOH). Activation parameters, such as the mass ratio of KOH and carbon raw materials, significantly affect the textural and adsorption characteristics of carbon, determining its ability to conserve moisture and adsorb nutrients [13-15]. In this work, the surface and structural characteristics of activated carbon obtained at different mass ratios of the sorbent and KOH were studied.

The aim of the study is to optimize the activation conditions to achieve a high specific surface area and improved porous structure of activated carbon, which will allow its use in moisture-saving substrates for agriculture. During the work, the obtained sorbents were analyzed using FTIR spectroscopy to determine the functional groups. The pore structure of the

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activated carbons was analyzed using two well-established methods: Density Functional Theory (DFT) and and the Barrett-Joyner-Halenda (BJH) method [16,17]. The results obtained allow a deeper understanding of the effect of activation conditions on the properties of activated carbon and its prospects as an effective moisture-saving material.

2. Experimental part

2.1 Raw materials

The activated carbons in this study were produced from rice husks, a byproduct of rice milling. Rice husks are rich in cellulose, hemicellulose, and lignin, which contribute to the carbon yield and porous structure upon activation.

2.2 Synthesis of Activated Carbon from Rice Husk Biomass

The production of activated carbon (AC) from plant biomass was carried out in four stages, as shown in Fig. 1: milling, carbonization, impregnation and preheating, and chemical activation.

In the first stage, the rice husks were ground into small chips to increase surface area, which enhances the uniformity of heat transfer during subsequent pyrolysis. This preparatory step promotes more efficient and consistent carbonization, thereby improving the quality and structure of the resulting activated.

The carbonization process was carried out at a temperature of 550 °C for 120 min with a linear heating rate of 5 K/min. The experiment was carried out in an inert atmosphere maintained by feeding N_2 gas at a rate of 150 cm^3/min in a vertical steel reactor.

Impregnation was carried out using 5 g of carbonized mass, which was saturated with a 1M aqueous KOH at different mass ratios of the sorbent to alkali: (1:1), (1:2), (1:3), and (1:4). Accordingly, 5 ml of 1M KOH was used for the 1:1 ratio, 10 ml for 1:2, 15 ml for 1:3, and 20 ml for 1:4. This process included preheating from 30 °C to 80 °C, followed by a 3-hour hold to ensure complete penetration of the KOH into the carbonized samples. The impregnated sorbents were then transferred to a vertical electric furnace with a constant argon supply.

At the chemical activation, the samples were pyrolyzed at a temperature of 300 °C to 850 °C with a linear heating rate of 5 °C/min. After reaching the maximum temperature, the samples were held for 3 hours, after which they were left to cool naturally to room temperature. Argon supply was continued during the cooling period, preventing oxidation and maintaining the integrity of the AC. The resulting product was washed with a 0.1M hydrochloric acid aqueous solution and then with distilled water until the optimal pH value of the washing solution was reached (6–7). The washed AC samples were dried at a temperature of 100–105 °C.

3. Results and discussion

3.1 Morphological structure of the obtained activated carbons

Figure 2 shows the microstructural changes in the activated carbon samples obtained at different mass ratios of the sorbent and KOH: (a) 1:1, (b) 1:2, (c) 1:3, and (d) 1:4. These images illustrate the effect of the alkali amount on the porosity and texture of the activated carbon.

At the 1:1 ratio, a dense structure with a relatively low degree of porosity is observed. Probably, at this ratio, the amount of KOH is insufficient for significant activation and the creation of a developed porous structure, which leads to the preservation of large, less branched structural elements. The 1:2 ratio demonstrates an increase in porosity and a looser structure compared to the 1:1 ratio. The sample shows

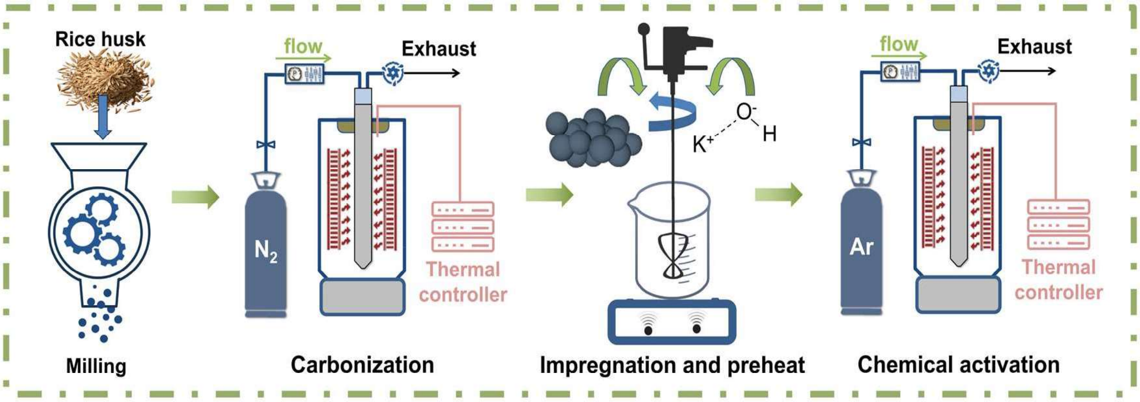

Fig. 1. Schematic illustration of the preparation of AC.

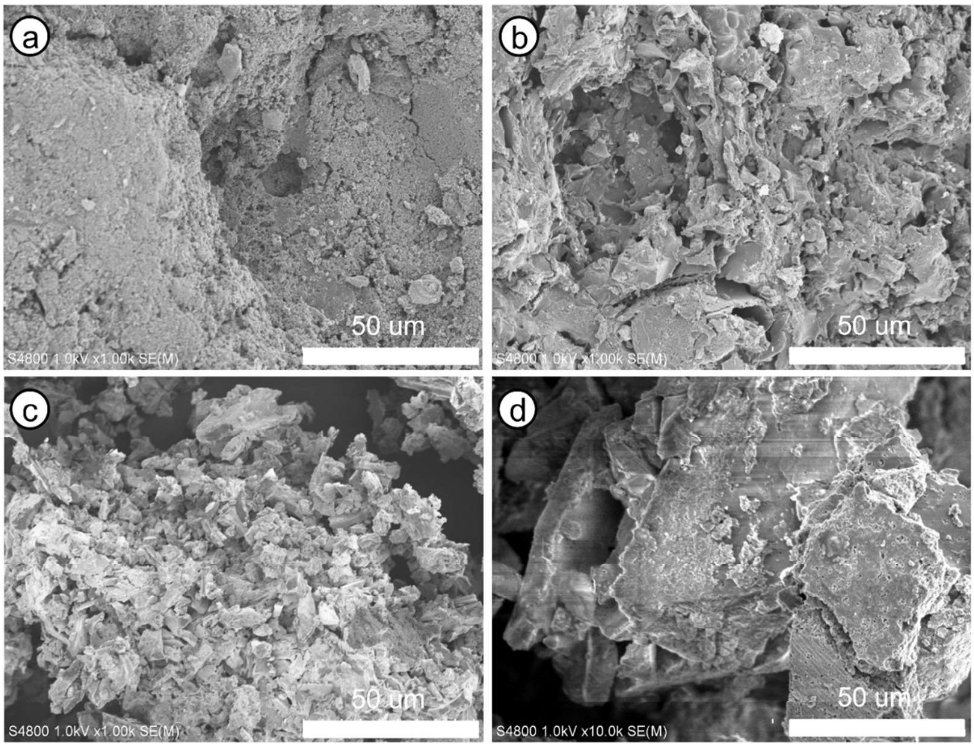

Fig. 2. Microstructure of activated carbon obtained at different mass ratios of sorbent and KOH: (a) 1:1, (b) 1:2, (c) 1:3 and (d) 1:4.

signs of active destruction of large fragments and the formation of micropores, which indicates a greater efficiency of KOH in creating a porous network in the carbon matrix.

With a further increase in the ratio to 1:3, the porosity of the structure becomes even more pronounced, characterized by a uniform distribution of pores and a high degree of loosening. This indicates an optimal ratio for activation, which achieves a good combination of high porosity and structural integrity. However, at a ratio of 1:4, the structure of the sample becomes extremely loose with signs of excessive surface erosion. Here, excess KOH leads to excessive destruction of the carbon framework, which causes a partial loss of structure and a significant decrease in the density of the material. Thus, an increase in the KOH ratio promotes the development of the porous structure of activated carbon, but too high concentrations (1:4) can lead to excessive destruction and a decrease in the mechanical stability of the sample.

Figure 3 shows the energy dispersive X-ray (EDS) spectra of activated carbon obtained at different mass ratios of sorbent and KOH: (a) 1:1, (b) 1:2, (c) 1:3, and (d) 1:4. The spectra show peaks corresponding to the main elements present in the structure of activated carbon, including carbon (C), oxygen (O), sodium (Na), magnesium (Mg), aluminum (Al), and silicon (Si). At a 1:1 ratio, moderate peaks of carbon and oxygen are observed, indicating the initial stage of activation with a limited number of active sites. The presence of additional elements, such as Na and Mg, may be attributed to incomplete removal of natural residues, which is typical of lower activation levels. For the 1:2 ratio, there is a significant increase in the intensity of carbon and oxygen peaks, suggesting more active interaction with KOH and an increase in porosity within the structure. The high of silicon (Si) peak likely reflects the natural silica content of the rice husk sorbent, stabilized during the activation process. At the 1:3 ratio, there is a marked predominance of carbon and oxygen in the spectrum, indicating optimal activation of the carbon matrix. Peaks of other elements are minimized, suggesting a cleaner structure and higher activation level, making this ratio preferable for producing activated carbon with a highly developed porous structure. At a 1:4 ratio, carbon and oxygen remain the primary elements, but the intensity of Na and Mg slightly increases. This suggests that an excess of KOH may lead to excessive structural degradation of the carbon and is not fully removed during washing, potentially resulting in an accumulation of alkaline residues within the activated carbon structure.

Thus, increasing the KOH mass ratio promotes an increase in the level of carbon activation, however, at a ratio above 1:3, alkaline elements accumulate, which remain in the structure of activated carbon and can negatively affect its properties.

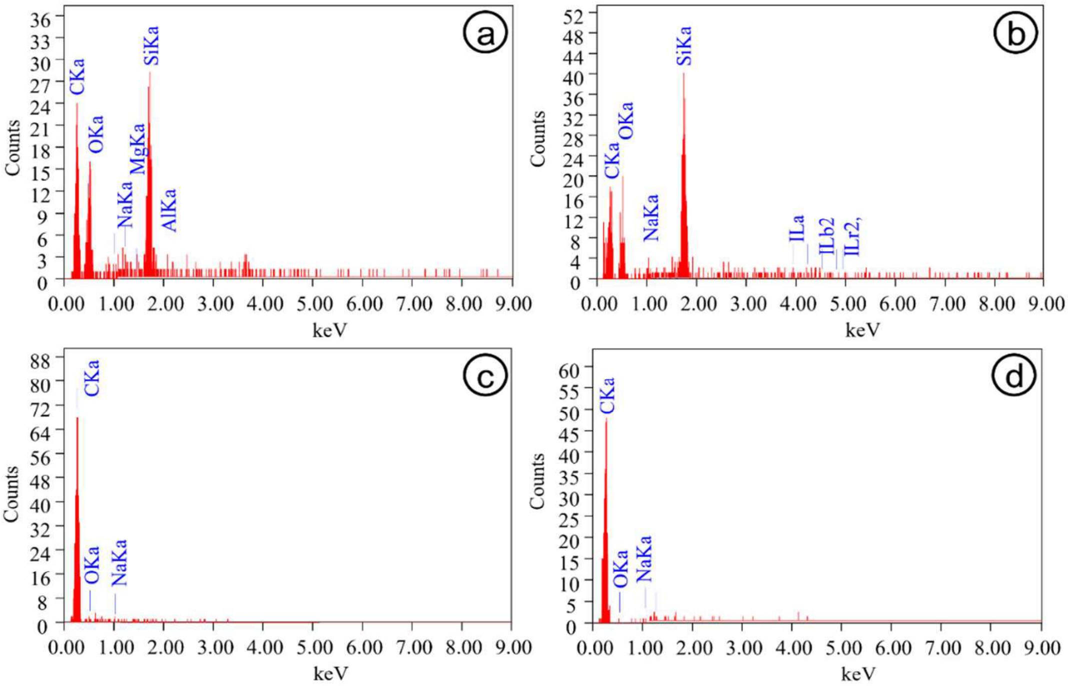

Fig. 3. Energy dispersive spectra (EDS) of activated carbon obtained at different mass ratios of sorbent and KOH: (a) 1:1, (b) 1:2, (c) 1:3 and (d) 1:4.

3.2 BET surface area and porosity analysis

To evaluate the surface characteristics of activated carbon obtained at different ratios of sorbent and KOH, nitrogen sorption parameters were measured using the BET method. The sample with a ratio of 1:1 has a correlation coefficient r equal to 0.9990, which indicates a high degree of linearity in the dependence of nitrogen adsorption. The constant C is -55.456, and the specific surface area is 910 m^2/g, which is a moderate value among all samples. At a ratio of 1:2, the correlation coefficient slightly decreases to 0.9967, and the constant C reaches 195.234, which is significantly higher compared to the other samples. The specific surface area of this sample is 1210 m^2/g, which indicates a significant increase in porosity. The sample with a ratio of 1:4 is characterized by a decrease in the correlation coefficient to 0.9521, which indicates a slight deviation in the linearity of adsorption. The constant C at this ratio is -57.389, and the specific surface area is 924 m^2/g, which is within the range of values for other samples, but lower than that of the sample with a ratio of 1:2.

The highest specific surface area (2900 m^2/g) was achieved at a ratio of 1:3, which indicates the maximum porosity and developed surface of this sample. The correlation coefficient in this case is 0.9973, and the constant C is 101.356, which also indicates significant activity of the sorbent. These results demonstrate that an increase in the KOH ratio contributes to an increase in the specific surface area of activated carbon, but under certain conditions, such as a ratio of 1:3, deviations in adsorption properties may occur.

Figure 4 illustrates the pore volume distribution of activated carbon obtained at different mass ratios of sorbent and KOH: (a) 1:1, (b) 1:2, (c) 1:3 and (d) 1:4. The graphs show the total pore volume (Pore Volume, cc/g) and the differential pore volume (dv(r), cc/Å/g) as a function of pore diameter, which allows us to evaluate the effect of activation conditions on the porous structure of the carbon. At a ratio of 1:1, a moderate pore volume is observed, mainly consisting of micropores with a diameter of about 2 nm. The peak of dv(r) for this sample increases sharply at this diameter, indicating the dominance of micropores, while mesopores and macro-pores are practically absent. At a ratio of 1:2, the total pore volume increases to 0.7 cc/g, while micropores with a diameter of about 2 nm also predominate. This ratio shows higher porosity compared to 1:1, indicating improved activation with increasing KOH proportion. The most significant pore volume (1.6 cc/g) was achieved at the 1:3 ratio, where micropores also dominate, but a significant amount of mesopores is also present.

The dv(r) peak confirms a high concentration of micropores, while the pore distribution becomes more uniform, which makes this ratio optimal for creating a structure with maximum adsorption capacity.

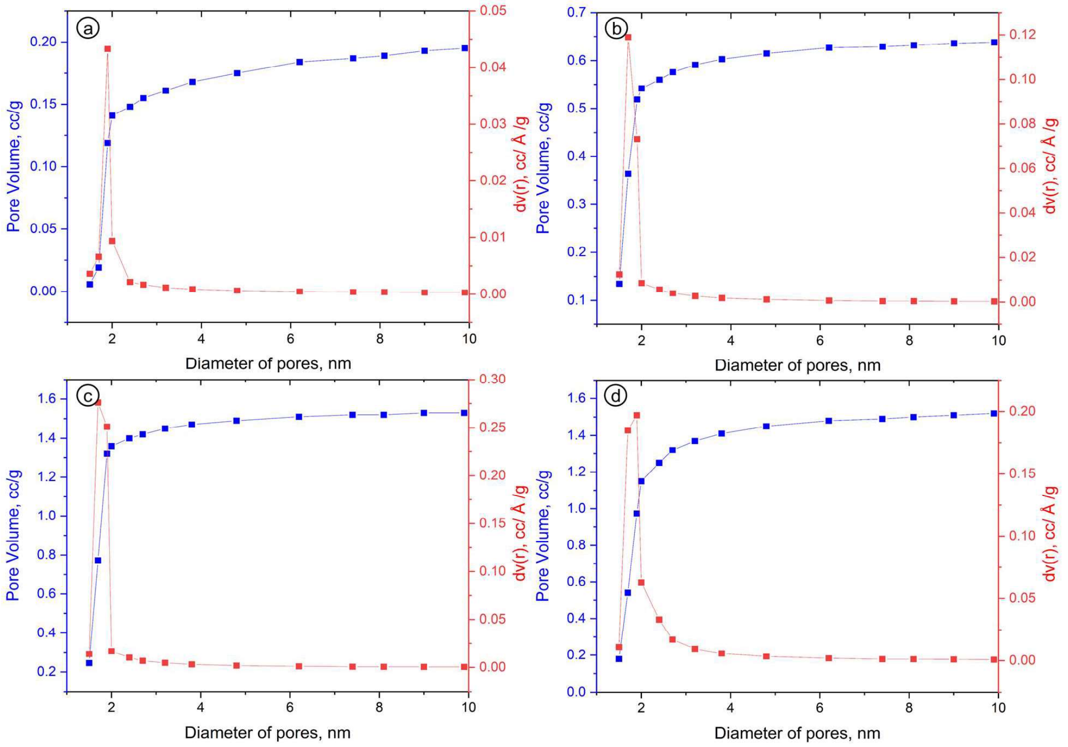

Fig. 4. Distribution of the pore volume of activated carbon at different mass ratios of the sorbent and KOH: (a) 1:1, (b) 1:2, (c) 1:3 and (d) 1:4.

At the 1:4 ratio, the pore volume remains at the level of 1.6 cc/g, but the dv(r) peak is less pronounced, indicating the appearance of larger pores and partial destruction of the microporous structure. This indicates that an excessive amount of KOH leads to the formation of macropores and a decrease in the strength of the structure.

The 1:3 and 1:4 ratios exhibit similar activation behaviors, as evidenced by comparable carbon and oxygen peak intensities, indicating high activation levels and developed porosity. However, visually, these samples differ significantly in structural appearance. At the 1:3 ratio, the activated carbon demonstrates a cleaner and more uniform structure, with minimized presence of additional elements, suggesting optimal activation without excess residue. In contrast, the 1:4 ratio shows slight increases in Na and Mg peaks, which may indicate incomplete removal of residual KOH, leading to a more heterogeneous structure with visible alkaline residues. This suggests that while both ratios promote high activation, the 1:4 ratio may introduce structural inconsistencies due to excessive KOH.

In general, an increase in the KOH ratio contributes to the development of the porous structure of activated carbon, but the 1:3 ratio is optimal for obtaining carbon with a developed microporous structure and a high specific surface area.

3.3 Physicochemical properties of the obtained activated carbons

Figure 5 shows the FTIR spectra of activated carbon obtained at different mass ratios of the sorbent and KOH: (a) 1:1, (b) 1:2, (c) 1:3 and (d) 1:4. The spectra show the main functional groups formed during the activation process, which can affect the adsorption properties of carbon. The main absorption bands, such as O–H, C=O, C=C and C–O, change depending on the activation conditions and demonstrate patterns similar to those found in previous studies.

The band at 3430 cm^{-1}, associated with hydroxyl groups (O–H), is present in all spectra and indicates the presence of hydrogen-containing functional groups. The intensity of this band is slightly higher at higher KOH ratios, which is consistent with increased surface hydrophilicity. The band at 1740 cm^{-1}, corresponding to carbonyl groups (C=O), is most intense at a ratio of 1:3, indicating optimal formation of these groups at this ratio. This is consistent with the results of previous studies, which noted that KOH ratios above 1:2 promote an increase in oxygen-containing functional groups, which positively affect the adsorption characteristics of carbon.

The peak at 1640 cm^{-1}, associated with aromatic bonds (C=C), appears consistently across all samples, indicating the stability of the aromatic matrix under varying activation conditions, as previously

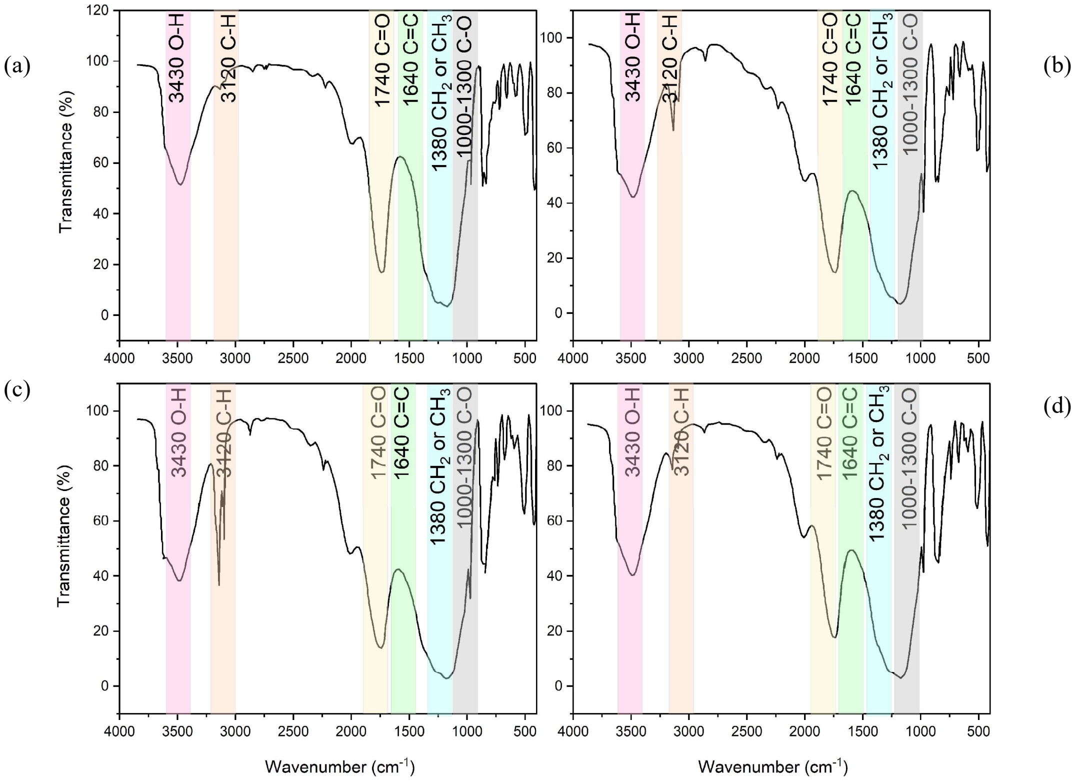

Fig. 5. FTIR spectra of activated carbon obtained at different mass ratios of sorbent and KOH: (a) 1:1, (b) 1:2, (c) 1:3 and (d) 1:4.

established. The band at 1380 cm^{-1}, attributed to methyl and methylene groups (CH_2 and CH_3), is most pronounced at ratios of 1:2 and 1:3, aligning with prior findings that these groups form optimally under certain activation conditions, enhancing the hydrophobic properties of the carbon. Additionally, the peak at 3120 cm^{-1}, present especially at the 1:3 ratio, corresponds to C–H stretching vibrations, suggesting substantial hydrocarbon group formation. This may indicate extensive decomposition of the original plant polymers, contributing to a highly developed carbon structure. The band within the 1000–1300 cm^{-1} range, corresponding to ether and alcohol groups (C–O), is most intense at the 1:3 ratio, suggesting active formation of oxygen-containing groups, consistent with studies linking high KOH activity to enhanced adsorption of polar molecules. Overall, these findings confirm that a KOH ratio of about 1:3 is optimal for producing activated carbon with a high density of functional groups, enhancing its adsorption properties for pollutant removal and catalytic applications.

4. Conclusion

This work investigated the textural and adsorption properties of activated carbon obtained from plant waste at different mass ratios of the sorbent and KOH. The results showed that the activation conditions significantly affect the porous structure and functional groups present on the surface of the sorbents. The most optimal parameters were achieved at a ratio of 1:3, which was characterized by a high specific surface area and developed porosity, which provides activated carbon with improved adsorption characteristics. FTIR spectra confirmed the presence of functional groups such as O–H, C=O and C–O, which contribute to moisture conservation and potential nutrient retention.

The study showed that activated carbon obtained under optimal conditions can be used as a water-retaining substrate, which is especially important for agriculture under water scarcity conditions. These results open up prospects for the development of water-retaining substrates based on activated carbon, which can increase crop productivity and reduce the need for water resources.

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Получение и оптимизация активированного угля из растительных отходов с высокой удельной поверхностью для влагосберегающих применений в сельском хозяйстве

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АННОТАЦИЯ

В условиях дефицита водных ресурсов устойчивое развитие сельского хозяйства требует применения влагосберегающих технологий, включая использование влагоудерживающих субстратов на основе активированного угля. В данной работе исследованы текстурные и адсорбционные характеристики активированного угля, полученного из растительных отходов при различных массовых соотношениях сорбента и KOH (1:1, 1:2, 1:3 и 1:4). Целью исследования было определение оптимальных условий активации для создания материала с высокой удельной поверхностью и развитой пористой структурой.

Результаты исследования показали, что наибольший объем пор (1.6 см^3/г) и высокая степень микропористости достигаются при соотношении 1:3, что подтверждено анализом распределения пор методами «Теория функционала плотности» и «Метод Барретта-Джойнера-Халенды». ИК-Фурье спектроскопия выявила наличие функциональных групп (O–H, C=O и C–O), способствующих влагосбережению. Дифференциальное распределение объема пор (dv(r), см^3/Å/г) также продемонстрировало, что при соотношении сорбента и KOH (1:3) структура образца оптимально сочетает микропоры и мезопоры, что повышает адсорбционную способность угля.

Ключевые слова: активированный уголь, растительные отходы, влагосберегающий субстрат, пористая структура, адсорбционные свойства

Ауыл шаруашылығында ылғал үнемдеу үшін жоғары беттік ауданы бар өсімдік қалдықтарынан белсендірілген көмірді дайындау және оңтайландыру.

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АҢДАТПА

Су ресурстарының тапшылығы жағдайында ауыл шаруашылығының тұрақты дамуы ылғал үнемдейтін технологияларды, оның ішінде белсендірілген көмір негізіндегі ылғал сақтайтын субстраттарды қолдануды талап етеді. Бұл жұмыста сорбент пен KOH (1:1, 1:2, 1:3 және 1:4) әртүрлі массалық қатынасында өсімдік қалдықтарынан алынған белсендірілген көмірдің текстуралық және адсорбциялық сипаттамалары зерттелді. Зерттеудің мақсаты жоғары меншікті бетінің ауданы мен дамыған кеуекті құрылымы бар материалды жасау үшін оңтайлы белсендіру шарттарын анықтау болды.

Нәтижелер ең үлкен кеуек көлеміне (1,6 см^3/г) және микрокеуектіліктің ең жоғары дәрежесіне сорбент пен KOH (1:3) қ атынасында қол жеткізілгенін көрсетті, бұл кеуекті бөлудің Тығыздықтың функционалдық теориясы және Баррет-Джойнер-Халенда әдісі талдауымен расталды. FTIR спектроскопиясы ылғалдың сақталуына ықпал ететін функционалдық топтардың (O–H, C=O және C–O) болуын анықтады. Кеуек көлемінің дифференциалды таралуы (dv(r), см^3/Å/г) сонымен қатар 1:3 қатынасында үлгі құрылымы микрокеуектер мен мезокеуектерді оңтайлы біріктіретінін көрсетті, бұл көмірдің адсорбциялық қабілетін арттырады.

Түйін сөздер: белсендірілген көмір, өсімдік қалдықтары, ылғал сақтайтын субстрат, кеуекті құрылым, адсорбциялық қасиеттер