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PREPARATION OF HIGHLY POROUS ACTIVATED CARBON WITH HIGH SPECIFIC SURFACE AREA FROM AGRICULTURAL WASTES

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Kazakhstan annually generates a significant amount of agro-industrial waste, which, due to the high content of carbonaceous compounds, can potentially become a valuable raw material for the production of activated carbon. This research considers the processing of rice husks, soya and sunflower husks into activated carbon. Potassium hydroxide (KOH) was used for activation to achieve high specific surface area. The obtained activated carbon samples from rice husk, soybean husk and sunflower husk were characterized by BET, SEM, XRD, energy dispersive analysis, TGA and DSC. The synthesized activated carbons possessed a developed porous structure with specific surface area: 2363.7 m^2/g (RH), 786.1 m^2/g (SfH), 642.4 m^2/g (SBH), which was confirmed by the above analyses. These findings highlight the potential of agro-industrial waste as a sustainable source for high-performance activated carbon production. This study demonstrates a sustainable pathway for converting agro-industrial residues into high-performance activated carbon suitable for environmental and industrial applications.

Keywords: *agricultural waste, activated carbon, pyrolysis, circular economy, waste recycling.*

INTRODUCTION

In Kazakhstan, the processing of plant-based agricultural wastes remains insufficiently developed. Meanwhile, their ongoing accumulation poses an increasingly significant environmental challenge, despite the high potential of these wastes as raw materials for the production of valuable carbon-based materials. According to the Concept of Kazakhstan's transition to a “green economy” it is planned to recycle at least 40% of waste by 2030 and up to 50% by 2050. This corresponds to the international Sustainable Development Goals [1]. Based on the above, in this work, we have considered the possibility of optimizing the method of obtaining activated carbon from agricultural waste to achieve high specific surface area and sorption capacity. This will allow us to expand the use of agricultural waste in Kazakhstan and create an effective carbon material for use in various fields.

Thus, in Kyzylorda region they have started to actively study ways of recycling rice husk, the annual volume of which is up to 80,000 tonnes. A number of reuse technologies are being introduced here. For example, Abai Daulet LLP turns husks into a feed additive that reduces the risk of gastrointestinal disorders in cattle. In addition, rice husks and straw are used to produce biofuel in the form of high-calorie pellets, which is extremely important for areas without gas [2]. Technologies have also been created for the production of biochar and silicon dioxide, which is used as a biofertiliser to improve soil structure and reduce its salinity, and silicon dioxide is used in industry, like glass, ceramics, abrasives [3].

Kazakhstan also recycles biomass waste such as sunflower husks. For example, the oil extraction plant of Altyn Shygys LLP processes about 1.4 thousand tonnes of sunflower husks and makes fuel pellets from the residues and uses them as biofuel for its own energy needs. This allows the plant to reduce fuel costs and reduces its carbon footprint. The plant workers note that there are no unpleasant odours when burning such fuel [4]. As for the processing of soybean husks, this area is still underdeveloped in Kazakhstan, which opens up the possibility of finding methods and ways of recycling.

Along with the discoveries already made, one of the promising ways of processing agricultural by-products is the production of activated carbon [5]. Activated carbon is one of the most popular sorbents, used as an effective carrier for catalysts in various chemical processes. Due to its high porosity and high specific surface area, it is widely used in various industries. For example, in the purification of various environments from heavy metals and pollutants, and can also find applications in medicine and as a carbon carrier for catalysts [6–9]. The relevance of finding alternative sources of raw materials is due to the depletion of traditional resources, which makes plant wastes from agriculture and food industries such as straw, husks of rice, soybean, corn and sunflower as promising replacement materials [10-12].

There are many studies that support the potential of utilising agricultural wastes to produce highly porous material. For example, *Mubinul Islam et al.* successfully synthesised microporous activated carbon from sunflower husk by chemical activation method using KOH, at 800 °C. The resulting activated carbon showed great potential as a solid desiccant material for capturing water vapour from humid air and also showed high efficiency in removing mercury ions from aqueous solutions [13]. Similar results were obtained by *Schneider Lara Talita et al.* who investigated the effect of different thermochemical activation conditions on the formation of meso- and microporous structure of activated carbon from soybean husk. The experimental results showed that optimisation of activation parameters promotes the production of carbon material with increased specific surface area and uniformly developed porosity, which ensures its high efficiency in metronidazole adsorption. The authors concluded that the synthesised activated carbon shows better sorption properties compared to commercial analogues, which confirms its significant potential for use in environmentally friendly water treatment methods [14]. In addition to its application in water treatment, biomass activated carbon can be used as a carbon carrier with high energy density [15]. Carbon carriers represent some of the most promising catalyst materials due to their high purity, well-developed porous structure, significant specific surface area and high reactivity. These carbon materials are resistant to acidic and alkaline environments, which makes them economically and environmentally advantageous, especially for the recovery of components from spent catalysts. A widely used carrier is activated carbon (AC), which has high porosity and specific surface area (500–2500 m^2/g), which is the optimum parameter for adsorption [16].

The obtained activated carbon was investigated by the following methods: BET analysis, scanning electron microscopy (SEM), energy dispersive analysis (EDA), thermogravimetric analysis and differential scanning calorimetry (TGA and DSC), X-ray diffraction method (XRD analysis).

1 MATERIALS AND METHODS

1.1 Characterization of raw materials

Rice husk from Kyzylorda oblast and soya bean and sunflower husks from East Kazakhstan oblast were selected as raw materials for the production of activated carbon. Potassium hydroxide (KOH) (KHIMPEK, Russia) in solid form was used as activating agent at the ratio of biomass and activator 1:4 (feedstock : KOH).

The primary processing steps include cleaning the biomass from impurities like dirt, sand, stones and organic contaminants. It was washed with distilled water and dried at 105 °C for 24 hours. The cleaned material was then ground in an analytical mill (A 11B, IKA, Germany) to a particle size of approximately 0.9±0.3 mm. The obtained material was used for the synthesis of activated carbon.

1.2 Method of production of activated carbon

Activated carbon (AC) has been produced by pyrolysis. Pyrolysis, the predominant method, involves heating the material in an oxygen-free environment, causing thermal decomposition and the formation of carbon, which is subsequently activated by chemical agents. This process not only optimises the adsorption properties of the coal, but also recycles the by-products of the production process, helping to reduce the environmental load.

The crushed feedstock is placed in a muffle furnace where it undergoes thermal decomposition and is converted into charcoal within an hour. To determine the optimum pyrolysis temperature, experiments were conducted at temperatures of 300 °C, 400 °C, 500 °C, 600 °C. After investigating different temperatures, the most optimum conditions were found, namely pyrolysis at 600 °C in an inert medium with nitrogen gas supply. To obtain activated carbon, the obtained product was mixed with potassium hydroxide. The mixture was then subjected to heat treatment in a muffle furnace at 750 °C under nitrogen atmosphere providing inert conditions [17, 18]. To remove alkali residues, the carbon was washed with a solution of 1% hydrochloric acid, for 2 hours at 50 °C. The material was then washed with hot water (~70-80 °C) and dried in a desiccator at 100 °C for 60 minutes (AC) [19, 20]. As a result of the thermal and chemical treatment, the modified activated carbon was obtained.

1.3 Research methods

The specific surface area and pore structure of activated carbon samples were determined using a high-performance BET analyser (BSD-660). The morphology and structure of activated carbon obtained from sunflower, soybean and rice husks were investigated using a Zeiss Auriga Crossbeam 540 high-vacuum scanning electron microscope (SEM) (Germany). The elemental composition of the materials was analysed using Thermo Fisher Scientific (USA) energy dispersive spectroscopy (EDS) system. Phase and structural analyses of the obtained samples were carried out on X'PertPRO diffractometer (Malvern Panalytical Empyrean, Netherlands). Monochromatised copper radiation (CuKα, $\lambda = 1.54060$ Å) at a scanning speed of 0.02° per second was used. The ICDD PDF-4/AXIOM database was used for phase identification. Thermal properties of activated carbon samples were analysed on a LabsysEvo instrument (Setaram, France) in argon atmosphere in accordance with GOST R 55134-2012 standards. Crystallisation and melting of the samples were studied under nitrogen flow.

2 RESULTS AND DISCUSSIONS

2.1 Physicochemical structure of activated carbon (AC)

2.1.1 Textural characteristics of AC: BET analysis

The textural characteristics of the initial activated carbon according to nitrogen adsorption isotherms are presented in Table 1 and Figure 1 (a, b, c). According to the data obtained, AC from rice husk can be classified as mesoporous, and AC from soybean and sunflower husks can be classified as microporous. From Table 1 and the graph, it can be seen that rice husk (a) has a specific pore surface area of 574.6 m^2/g. And the specific pore surface area of activated carbon from sunflower husk (b) and soya husk (c) was 464.4 m^2/g and 255.6 m^2/g respectively. The average pore size of activated carbon from rice husk lies within 2.52 nm, from sunflower husk 2.86 nm and from soya husk 1.55 nm (Table 1).

Table 1. Characteristics of porous structure of initial AC

Sample	Specific pore surface SW, m²/g	Pore radius Dv(r), mm	Total pore volume V_p, cm³/g
AC (RH)	574.6	2.52	0.50
AC (SFH)	464.4	2.86	0.19
AC (SbH)	255.6	1.55	0.11

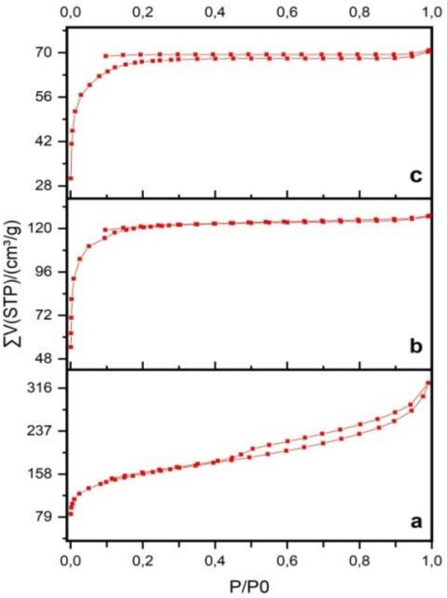

Figure 1. N2 adsorption-desorption isotherms of activated carbon: (a) rice husk; (b) sunflower husk; (c) soy husk

2.1.2 SEM analysis of activated carbon

The results of SEM with energy dispersive analysis are shown in Figure 2 (a, b, c) and Table 2. Based on the SEM analysis, it was found that the obtained activated carbon has a clearly distinguishable porous structure, which includes meso- and micropores and a minor amount of macropores, which is comparable to the results of BET analysis.

EDS analysis identified the elemental composition of coal samples obtained from rice, soya and sunflower husks. The elemental composition of the obtained coals is presented in Table 2. Peaks corresponding to carbon, oxygen, and impurities such as potassium, magnesium, calcium and iron are observed on the EDS spectra (Figure 2). All samples have a high carbon content, which is characteristic of activated coals. SEM-EDS allowed visualising the surface of the material at the macro- and nano-level, and also allowed analysing its elemental composition (Figures 3).

Table 2. Result of energy dispersive analysis of activated carbon obtained from the feedstock: rice husk (RH), sunflower husk (SFH) and soya husk (SbH)

Sample	Elements (mass, %)					
	C	O	K	Mg	Ca	Fe
AC (RH)	91.92±0.07	6.86±0.07	0.64±0.01	0.08±0.01	0.37±0.01	0.13±0.01
AC (SFH)	91.09±0.06	7.88±0.07	0.75±0.01	—	0.28±0.01	—
AC (SbH)	89.85±0.06	8.39±0.07	0.76±0.01	0.24±0.01	0.57±0.01	0.19±0.01

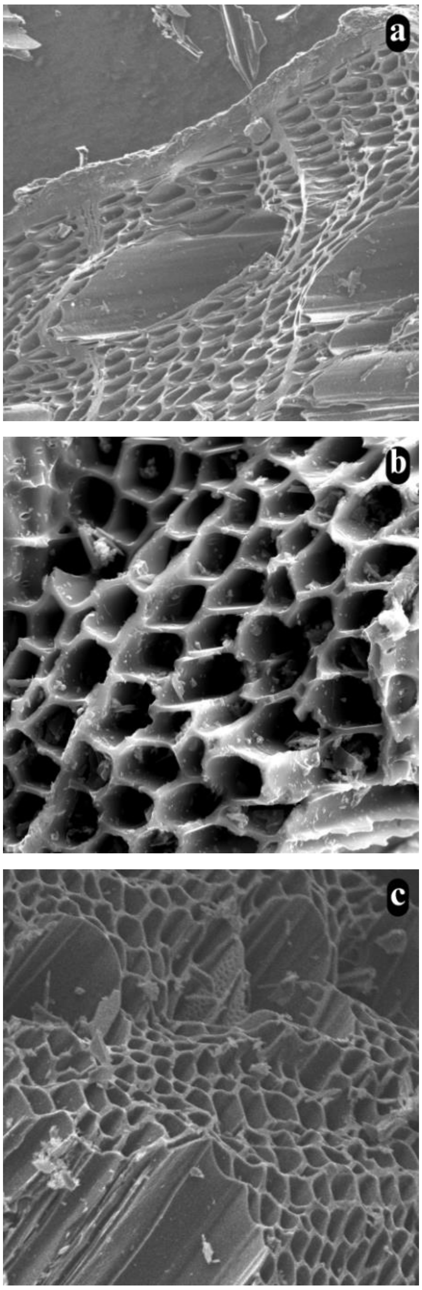

Figure 2. SEM micrographs with EDS analysis of activated carbon: (a) rice husk; (b) sunflower husk; (c) soya husk

2.1.3 XRD of activated carbon

The SEM data with energy dispersive analysers and textural characteristics were confirmed by X-ray phase analysis (XRD) results. The XRD analysis of activated carbon is presented in Table 3 and Figure 3. The activated carbon is amorphous in structure, which we verified by performing X-ray phase analysis (XRF). The spectrum of the sample from rice husk (a) shows broad peaks in the region of 24–26° 2θ and 45° 2θ, indicating the presence of a partially ordered graphite-like layer, and the moderate intensity of the peaks indicates a small number of crystalline regions. And the peak in the 73–75° region may indicate non-carbonaceous residual minerals, which we observed in the EDS analysis. The sunflower husk sample (b) shows more pronounced peaks at 23° 2θ and 43° 2θ, this indicates a more pronounced degree of crystallinity, and also small peaks in the range of 60–65°, may indicate minor residual mineral inclusions. In the case of soya husk sample (c), the peaks in the 23° 2θ and 43° 2θ region are weaker, indicating a more chaotic amorphous structure. And the additional weak peak in the range of 50–55° 2θ may be due to residual non-carbon impurities or microcrystalline inclusions, which was confirmed with EDS analysis.

Table 3. Values of interfacial and D-interfacial AC distances between atoms

Pos. [°2θ]	Height, cts	FWHM Left, °2θ	d-spacing, Å	Rel. Int., %
26.625	3.85	0.0900	3.78073	3.35
45.675	18.45	0.5904	3.04879	16.06
43.7044	26.91	0.3936	2.07122	23.43
55.9856	15.81	0.7872	1.79122	13.76

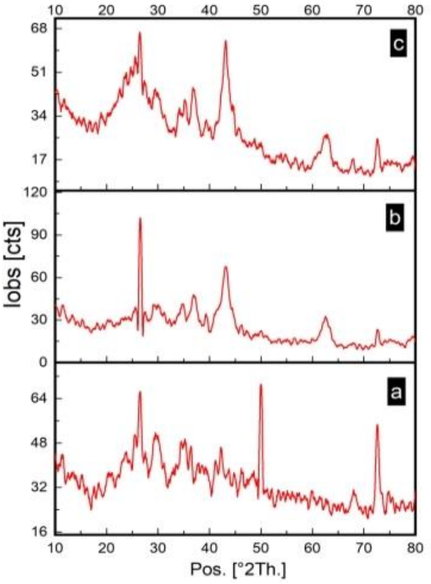

Figure 3. X-ray phase analysis of activated carbon: (a) rice husk; (b) sunflower husk; (c) soya husk

2.1.4 Thermogravimetric analysis

Figure 4 shows the results of thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) of activated charcoal obtained from different types of biomass. For coals from rice husk, a gradual decrease in mass is observed starting at 70 °C and temperature up to 500 °C, followed by a sharp decrease in mass at an average of 500–630 °C. In the case of sunflower husk charcoal, thermal degradation is observed in three temperature ranges: 55–160 °C, 340–500 °C and 550–750 °C. In the temperature range (up to 160 °C), the reduction is due to low release of hygroscopic moisture and desorption of adsorbed water. Two intervals of thermal mass loss are observed for the applied soya husk charcoal: 50–190 °C and 380–780 °C. The design of the mass media is related to moisture removal, the main decomposition process of components and minerals occurs at 380–780 °C.

These results are consistent with previously published studies in which TGA accounts for the three- or four-stage nature of biochar decomposition due to continuous moisture desorption, pyrolysis of volatile methods, degradation of carbonised structures and thermal effects of inorganic components.

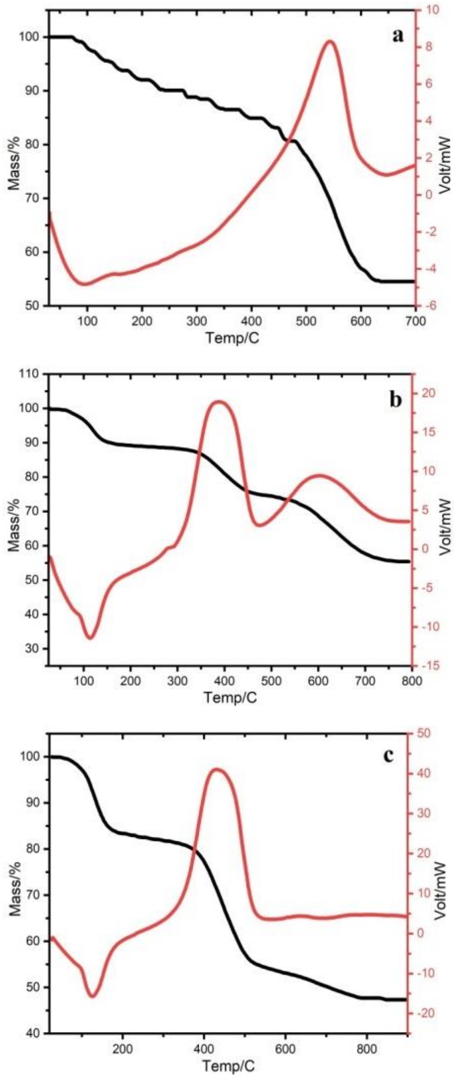

Figure 4. Thermogravimetric curve and differential scanning calorimetry of the initial coal from the starting materials: a) rice husk; b) sunflower husk; c) soya husk

2.2 Physicochemical structure of modified activated carbon (AC)

2.2.1 Textural characterisation of the activated carbon. BET analysis

The textural characteristics of the modified activated carbon obtained from nitrogen adsorption isotherms are presented in Table 4 and Figure 5. Prior to the BET measurements, all samples were degassed at 300 °C for 180 min. According to the analysis, after modification with potassium hydroxide, the structure of activated carbon from rice husk disappeared micro pores and more mesopores and the appearance of macropores is observed. This is confirmed by the high specific pore surface area of 2363.7 m^2/g and average pore radius of 1.43 nm. At the same time, the modified activated carbon from soya and sunflower husks retains a predominantly microporous structure with few mesopores, as reflected by their specific surface area of 642.4 m^2/g and 786.1 m^2/g, respectively, and pore radii of 1.37 nm and 1.27 nm. The total pore volume also differs, with the activated carbon from rice husk reaching 1.61 cm^3/g, whereas the activated carbon from soybean and sunflower husk is 0.25 cm^3/g and 0.35 cm^3/g, respectively. Thus, modification with potassium hydroxide significantly increased the specific pore surface area and pore volume of activated carbon, favouring the formation of meso- and microporous structure.

Table 4. Characteristics of the porous structure of ACm modified activated carbon obtained from rice husk (RH), sunflower husk (SFH) and soya husk (SbH).

Sample	Specific pore surface SW, m^2/g	Pore radius Dv(r), nm	Total pore volume V_p, cm^3/g
AC_m (RH)	2363.7	1.43	1.61
AC_m (SFH)	786.1	1.27	0.35
AC_m (SbH)	642.4	1.37	0.25

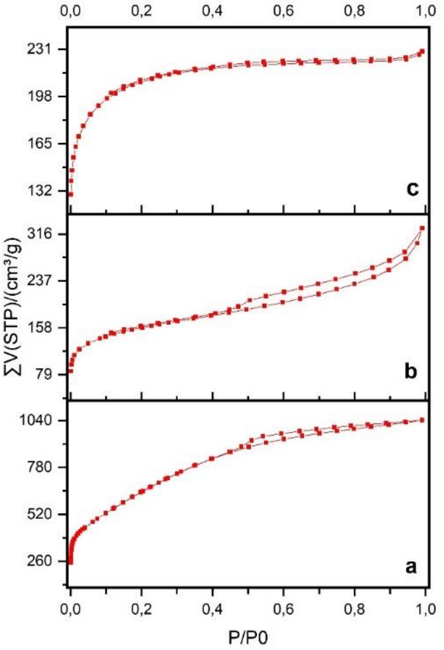

Figure 5. N_2 adsorption-desorption isotherms of modified activated carbon ACm: (a) rice husk; (b) sunflower husk; (c) soya husk

2.2.2 SEM analysis

Figures 7 and 8 and Table 5 show the results of SEM with energy dispersive analysis. When the activated carbon was modified with potassium hydroxide, slight changes in the morphology of the carbon material were observed (Figure 6).

As a result, a decrease in the number of different debris is observed, the structure becomes more cellular and organised, the lines become clearer, and the number of white particles on the pore surface decreases. An increase in the number and size of pores is observed, which acquire a regular shape and uniform size [8, 21, 22]. In places, the formation of macropores can be observed (Figure 6a, c).

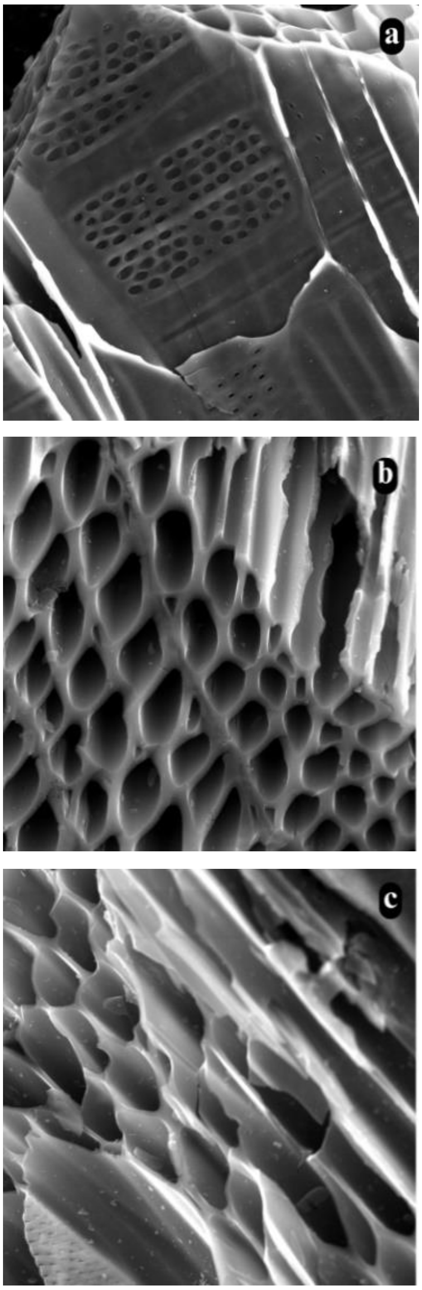

Figure 6. SEM micrographs with EDS analysis of modified activated carbon: (a) rice husk; (b) sunflower husk; (c) soya husk

The EDS analysis showed that after modification such elements as magnesium and iron are absent in the activated carbon (Table 5).

Table 5. Result of energy dispersive analysis of activated carbon modified with hydrochloric acid

Sample	Elements (mass, %)					
	C	O	K	Mg	Ca	Fe
AC_m (RH)	89.90±0.04	9.86±0.05	0.09±0.00	–	0.15±0.00	–
AC_m (SFH)	80.89±0.08	18.60±0.13	0.11±0.01	–	0.40±0.01	–
AC_m (SbH)	88.81±8.81	10.79±0.79	0.2390.01	–	0.1790.01	–

3.2.3 XRD X-ray diffraction analysis

Analysis of the data obtained by XRD analysis confirmed that the modified AC is an amorphous solid. It possesses a large internal surface area and pores of a certain size. It can be seen that the original AC and the modified ACm have a similar appearance from the XRD diffraction result (Figures 3 and 7).

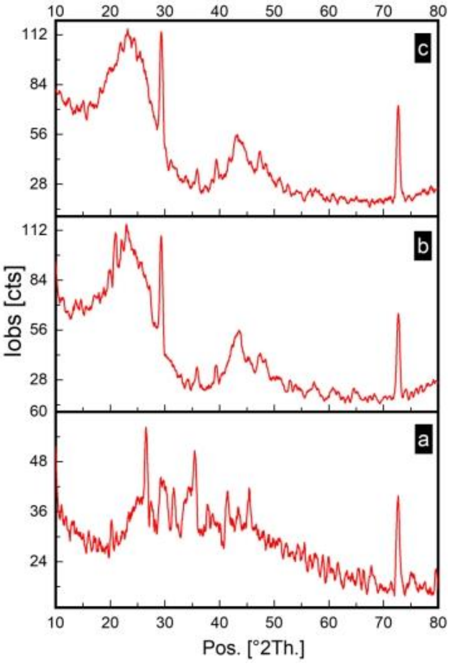

Figure 7. XRD X-ray diffraction analysis of ACm

The spectra of all three samples (Figure 7) showed approximately the same peaks [21], indicating their similar structural organisation. X-ray diffraction analysis of the activated carbon revealed broad asymmetric peaks in the ranges of 15~35 and 40~50, indicating the shape of typical amorphous carbon. This suggestion is consistent with the scanned micrograph images of the activated carbon shown in Figure 6. This showed that the activated carbon AC originates from carbon material and is an amorphous carbon with many pores.

Table 6. Values of interfacial distances of ACm and d-between atoms

Pos., °2θ	Height, cts	FWHM Left, °2θ	d-spacing, Å	Rel. Int., %
23,8687	1.96	0.0900	3.72502	1.59
44,0859	2.68	0.0900	2.05248	2.17
51,4593	1.75	0.0900	1.77437	1.42

2.2.4 Thermogravimetric analysis

Figure 8 shows the results of thermogravimetric analysis of the modified activated carbon ACm. As can be seen from the figure, the decrease in the bulk of the primary activated carbon occurs in the temperature range of 90–120 °C to 420–700 °C for the rice husk sample, and in the ranges of 60–150 °C and 440–670 °C for the sunflower husk sample.

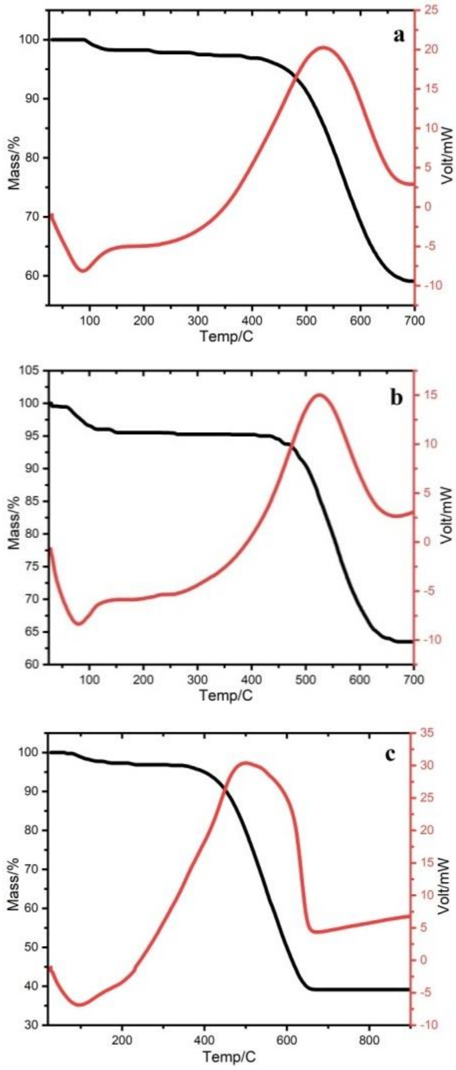

Figure 8. Thermogravimetric curve and differential scanning calorimetry of modified activated carbon ACm: (a) rice husk; (b) sunflower husk; (c) soya husk

The low temperatures, i.e. the sharp decrease in mass are directly related to the evaporation of moisture, in particular surface and adsorption water. In the temperature range of 420–700 °C (RH) and 440–670 °C (SFH), a gradual decrease in ACm mass occurs and organic compounds are burnt (Figure 8a). This may be due to the reduction of mineral content in ACm, and the formation of additional carboxyl, lactone and quinone functional groups after modification. Generally, the degree of thermal decomposition depends on the functional groups located on the surface of the activated carbon. Hence, an increase or decrease in the thermal decomposition index of activated carbon indicates the removal or formation of functional groups on its surface [22, 23]. Carboxylic acid, anhydride and lactone groups decompose at temperatures around 400 °C, while other surface functional groups (phenolic, carbonyl, quinone and pyronic structures) require more energy (above 400 °C) for decomposition.

CONCLUSION

In conclusion, the study confirms the high efficiency of agro-industrial wastes such as rice husk, soya husk and sunflower husk as raw materials for the production of activated carbon. The optimised heat treatment method, including carbonisation at 600 °C and subsequent activation with potassium hydroxide at 750 °C, allowed to obtain a material with a developed porous structure, which is confirmed by BET analysis showing a specific surface area (2363.7 m^2/g in the case of rice husk). X-ray diffraction (XRD) showed that the activated carbon from rice husk retains a predominantly amorphous character with the presence of meso- and macropores. This expands its application in industrial filtration processes, water and air purification, adsorption of heavy metals and organic pollutants, and as a carbon carrier in fuel cells, where the porous structure favours efficient catalyst distribution and improved electrochemical performance.

The obtained results demonstrate that the use of renewable sources of raw materials for the production of activated carbon not only contributes to the effective utilisation of agro-industrial waste, but also provides the creation of environmentally safe and economically feasible adsorbents with high adsorption capacity and structural stability.

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АУЫЛ ШАРУАШЫЛЫҚ ҚАЛДЫҚТАРЫНАН ЖОҒАРЫ МЕНШІКТІ БЕТІ БАР ЖОҒАРЫ КЕУЕКТІ БЕЛСЕНДІРІЛГЕН КӨМІР АЛУ

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Қазақстанда жыл сайын көміртекті қосылыстарға бай агроөнеркәсіптік қалдықтардың үлкен көлемі түзіледі, олар активтелген көмір өндіруге перспективалы шикізат бола алады. Бұл жұмыста күріш қабығы, соя және күнбағыс қалдықтарынан активтелген көмір алу мүмкіндігі зерттелді. Белсендіру үшін калий гидроксиді (KOH) қолданылды. Алынған үлгілер BET, SEM, XRD, EDS, TGA және DSC әдістерімен зерттелді. Алынған активтелген көмірдің кеуектілігі жоғары және меншікті беті айтарлықтай үлкен: 2363,7 м^2/г (күріш қабығы), 786,1 м^2/г (күнбағыс), 642,4 м^2/г (соя). Зерттеу нәтижелері агроөнеркәсіптік қалдықтарды жоғары тиімді активтелген көмірдің тұрақты көзі ретінде пайдалануға болатынын дәлелдейді.

Түйінді сөздер: *агроқалдықтар, белсендірілген көмір, пиролиз, көміртекті материалдар, жасыл экономика.*

ПОЛУЧЕНИЕ ВЫСОКОПОРИСТОГО АКТИВИРОВАННОГО УГЛЯ С ВЫСОКОЙ УДЕЛЬНОЙ ПОВЕРХНОСТЬЮ ИЗ СЕЛЬСКОХОЗЯЙСТВЕННЫХ ОТХОДОВ

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В Казахстане ежегодно образуется значительный объём агропромышленных отходов, которые благодаря высокому содержанию углеродсодержащих соединений являются перспективным сырьём для получения активированного угля. В работе исследованы рисовая шелуха, соевая и подсолнечная шелуха. Для активации использовался гидроксид калия (KOH). Полученные образцы охарактеризованы методами BET, SEM, XRD, EDS, TGA и DSC. Активированный уголь обладает развитой пористой структурой и высокой удельной поверхностью: 2363,7 м^2/г (рисовая шелуха), 786,1 м^2/г (подсолнечная), 642,4 м^2/г (соевая). Результаты подтверждают потенциал аграрных отходов как устойчивого источника для производства высокоэффективного активированного угля.

Ключевые слова: *аграрные отходы, активированный уголь, пиролиз, углеродные материалы, зелёная экономика.*