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PHYSICOCHEMICAL PROPERTIES OF CARBON ADSORBENTS DERIVED FROM RENEWABLE BIOMASS

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Abstract: In recent years, the demand for environmentally friendly and economically efficient adsorbent materials has been steadily increasing. In this regard, the production of adsorbents from renewable resources—particularly agricultural and wood industry waste—has become a pressing issue. Poplar and willow trees are widely used in industrial applications, resulting in the generation of significant amounts of waste, such as stems, branches, and twigs. Instead of incinerating or discarding these residues, they can be recycled to obtain valuable adsorbent materials. This study investigates the thermal (TGA/DTA) and infrared (FTIR) properties of coal-based adsorbents prepared from the stem waste of poplar and willow trees. The wood residues were chemically activated with potassium hydroxide (KOH) at high temperatures. The resulting carbon materials were analyzed using thermogravimetric analysis (TGA) and differential thermal analysis (DTA) to determine their thermal stability, decomposition stages, and heat reactions. FTIR spectroscopy was employed to identify the presence of functional groups (O–H, C–H, C=C, C–O) and to evaluate their role in adsorption processes. The analysis revealed that the obtained carbon materials retained active functional groups capable of adsorbing pollutants. Such recycling of wood waste not only contributes to reducing environmental issues but is also economically advantageous. Therefore, these carbon adsorbents, derived from natural and low-cost raw materials, are considered promising candidates for applications in industrial and environmental fields.

Keywords: tree stem, biomass, activated carbon, adsorbent, waste recycling, FTIR spectrum, DTA, TGA, thermal analysis.

Introduction. The rapid development of modern industrial and agricultural production has led to a significant increase in the emission of harmful pollutants into the environment. In particular, the release of heavy metal ions, dyes, pharmaceutical residues, petroleum products, and various other organic compounds into water bodies disrupts ecological balance and poses serious risks to human health. Among the effective solutions to these problems, adsorption methods are gaining increasing attention due to their efficiency. Although traditional adsorbents—especially activated carbon materials—are known for their high performance, their high cost and sometimes non-renewable nature, as well as the complexity of regeneration processes, necessitate the search for new, sustainable, and economically viable alternatives [1-3].

In recent years, the development of carbon-based adsorbent materials derived from biomass waste has attracted growing interest as an environmentally friendly and cost-

effective approach. In particular, tree stem waste—including the bark, branches, and other woody residues of fruit-bearing, ornamental, or industrial trees—is naturally rich in lignin, cellulose, and other organic compounds [4]. These components can be transformed into high-quality carbon adsorbents through physical and chemical activation processes. Such materials possess high porosity, large surface area, and surface-active functional groups, enabling strong adsorption capacity for various pollutants [5,6].

Adsorbents derived from tree waste are valued for their affordability, environmental safety, and effectiveness, making them promising candidates for use in various industrial sectors, including catalysis, industrial wastewater treatment, and environmental protection. Moreover, their use promotes waste reduction, resource efficiency, and sustainable development. Future research should focus on the selectivity of these adsorbents toward different pollutants, their regeneration capabilities, and the feasibility of large-scale industrial application.

The quality and effectiveness of carbon adsorbents are directly related to their physicochemical properties, which can be thoroughly investigated using analytical methods such as infrared (IR) spectroscopy and differential thermal analysis (DTA). IR spectroscopy enables the identification of functional groups located on the surface of the adsorbent. Since these groups serve as active adsorption sites, their presence has a direct impact on the performance of the adsorbent. For instance, functional groups such as $-OH$, $-COOH$, $-C=O$, $-CH$, and $-C-O-C-$ play a significant role in the adsorption efficiency of carbon materials [7,8].

DTA, on the other hand, helps determine exothermic and endothermic events occurring in the sample under thermal influence, while thermogravimetric (TG) analysis, used in conjunction with DTA, provides insights into decomposition stages through mass loss.

Together, IR and DTA analyses offer valuable information about the active surface functional groups of the adsorbents, their chemical and thermal stability, and the efficiency of the activation and carbonization processes. These assessments provide a scientific basis for evaluating the quality and applicability of waste-derived adsorbents from both environmental and technological perspectives [9-10].

Methodology & Empirical analysis. The preparation of carbon-based adsorbents from locally available raw materials, including tree stem waste, is of particular importance. For this purpose, secondary biomass residues from poplar and willow trees—common species in various regions of Uzbekistan—were selected as research objects. These materials were chemically activated in a laboratory setup to obtain carbon adsorbents.

Chemical activation was carried out using a potassium hydroxide (KOH) solution. Initially, the raw biomass was ground and subjected to pyrolysis at 500°C under an inert atmosphere. The resulting carbonizate was then chemically treated with a KOH solution. The effect of varying mass ratios of carbonizate to activating agent (1:1, 1:2, 1:3, 1:4, and

1:5) was studied. The mixtures were subsequently filtered, and the obtained carbon materials were thermally activated at 800°C with a heating rate of 10°C/min.

The resulting carbon adsorbents were treated with 0.5 N hydrochloric acid until a neutral pH was achieved. After acid washing, the samples were thoroughly rinsed with distilled water multiple times and dried at 100±5°C.

The prepared samples were designated as follows: FU-1 (willow + KOH, 1:1 ratio), FU-2 (willow + KOH, 1:3 ratio), FU-3 (willow + KOH, 1:5 ratio), FU-4 (poplar + KOH, 1:1 ratio), FU-5 (poplar + KOH, 1:3 ratio), FU-6 (poplar + KOH, 1:5 ratio).



Figure 1. Conventional schematic diagram for obtaining coal-based adsorbents from plant biomass

The qualitative and quantitative composition of functional groups in the obtained coal-based adsorbents was analyzed using infrared (IR) spectroscopy (Shimadzu IRTracer-100, Japan). The high sensitivity of the instrument (signal-to-noise ratio of 60,000:1) allowed for precise identification of absorption bands even with relatively low intensities, enabling step-by-step analysis of minor components in the samples. The IRTracer-100 spectrometer, with a spectral resolution of 0.25 cm⁻¹, provided high accuracy in identifying characteristic vibrations, particularly in gaseous-phase species. Additionally, the internal interferometer optimization system, coupled with automated self-diagnostics, ensured stable and reproducible performance throughout the measurements.

Thermal behavior of the samples was investigated using the differential thermogravimetric method (DTG) on a DTG-60 derivatograph (Shimadzu), operating in the Paulik F., Paulik I., and Erdey L. system. The analysis was conducted in the temperature range of 293–1093 K at a heating rate of 2–5 K/min, based on changes in thermal effects occurring during the decomposition of compounds.

The effective kinetic parameters of the thermal degradation of stabilized samples were calculated using the Freeman–Carroll method based on TGA data. The decomposition rate of the samples is described by the following equation:

$$dW/dt = (A_0/RH) \cdot e^{(-E/RT)} \cdot W^n$$

where:

RH – heating rate

W – sample mass

A_0 – pre-exponential factor

n – reaction order

E – effective activation energy of thermal decomposition

R – universal gas constant

T – absolute temperature

The reaction order n is determined from the slope ($\tan \theta$) of the linear plot in logarithmic coordinates, and the effective activation energy E is obtained from the intercept on the ordinate axis.

Results and Discussion. Thermal analyses are widely used to study the thermal characteristics of materials. Common techniques include differential scanning calorimetry (DSC), thermogravimetric analysis (TG), and combined thermoanalytical methods (TGA/DSC). For coal adsorbents derived from plant biomass, properties such as thermal conductivity and heat retention capacity are investigated during thermal treatments, such as the pyrolysis process. These parameters are essential for enhancing the energy efficiency of the coal and optimizing its processing conditions.

The thermal stability of coal adsorbents obtained from plant biomass waste is often influenced by the pyrolysis process. During pyrolysis, the organic matter in the biomass undergoes thermal decomposition at elevated temperatures, leading to the formation of coal. The thermal stability of these adsorbents determines their applicability under high-temperature and chemically reactive conditions. Figures 1 and 2 present the thermogravimetric (TGA) and differential thermal analysis (DTA) data of local poplar and willow tree stem waste. Figure 1 shows the thermogram of the sample derived from poplar biomass.

In this sample, the first stage (35.56–209.64 °C) is associated with the evaporation of moisture and other volatile components. This process lasts approximately 18.36 minutes and results in a mass loss of 4.369%, mainly due to the removal of physically bound water and low-molecular-weight substances.

The second stage, occurring between 209.64 °C and 399.11 °C, shows a significant mass loss of 74.983%, indicating the primary decomposition phase. This substantial weight loss is likely due to oxidative and pyrolytic reactions, including combustion and thermal degradation of organic constituents. According to literature and various studies, the decomposition of hemicellulose occurs predominantly in the 250–350 °C range as an endothermic process, while the degradation of cellulose and lignin occurs between 350–450 °C, accompanied by energy release. This interpretation is consistent with the observed exothermic peak in the DTA curve between 315.30 °C and 388.33 °C, with a maximum at 375.78 °C. This exothermic event is likely associated with carbonization and coking processes occurring in the material. Such exothermic behavior reflects the heat released during oxidation and decomposition of the coal structure.

The third stage, from 399.11 °C to 700.22 °C, results in an additional mass loss of 14.300%, during which the carbon-rich framework of the adsorbent is formed.

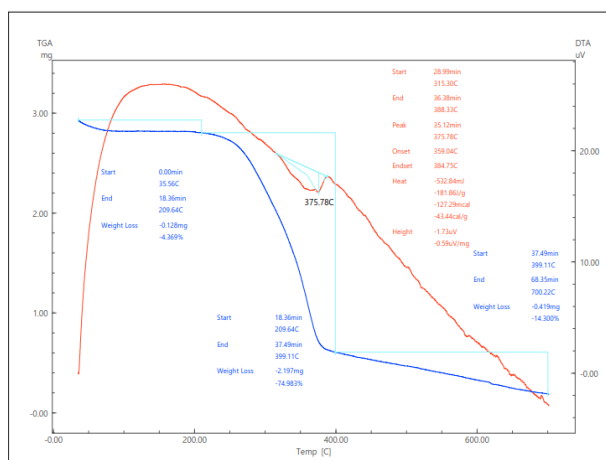


Figure 2. Thermogram of poplar tree stem waste

The thermogram presented in Figure 2 illustrates the thermal behavior of willow tree stem waste. Compared to the poplar stem waste sample, the thermal stability of this sample does not differ significantly.

For this sample, the first stage occurs between 38.51 °C and 233.65 °C, with a mass loss of 3.759%. This stage corresponds to the evaporation of moisture and other light volatile compounds.

The second stage, from 233.65 °C to 401.72 °C, shows a significant mass loss of 76.435%, which is the largest weight reduction observed across all stages. This loss is attributed to the decomposition of hemicellulose, cellulose, and lignin, consistent with major pyrolytic and oxidative reactions.

The third stage occurs between 401.72 °C and 700.32 °C, during which an additional mass loss of 7.276% is recorded. This phase reflects the final degradation processes and the formation of the carbonaceous structure of the adsorbent.

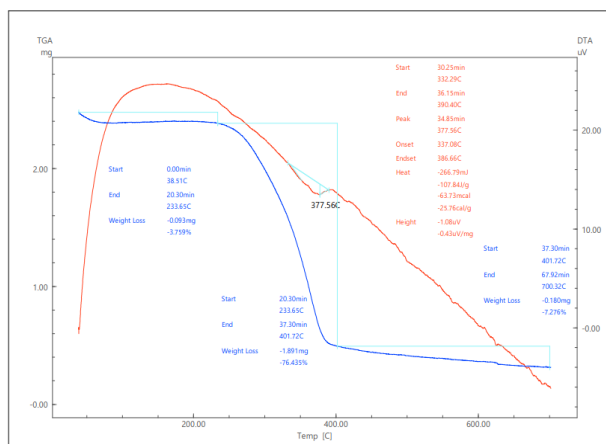


Figure 3. Thermogram of willow tree stem waste

It can be observed that the total mass losses in all stages are nearly identical for both samples. Furthermore, both samples exhibit exothermic peaks at nearly the same temperatures—375.78 °C and 377.56 °C, respectively. This suggests a close similarity in the composition and structural characteristics of the two samples.

Thermal analysis of coal adsorbents derived from plant biomass waste is crucial for evaluating their physical and chemical properties. Through thermal analysis, key parameters such as thermal stability, porous structure, combustion behavior, and other thermal properties of the coal can be determined. These insights are essential for improving the performance of coal adsorbents and optimizing their use for various applications. A detailed understanding of these processes enhances the potential for producing high-quality coal-based adsorbents from plant biomass.

The infrared (IR) spectra of the carbon materials derived from tree stems were obtained using Fourier Transform Infrared Spectroscopy (FTIR). FTIR is a powerful analytical technique for investigating the physicochemical properties of biochar obtained from tree stem biomass. This method allows for the identification of functional groups such as -OH , C=O , C=C , C-H , C-O , and others present in the material.

The presence and nature of surface functional groups are critical, as they directly influence the adsorption capacity of the biochar. In particular, the surface chemistry of biochar produced from biomass determines its interaction with adsorbates and hence its effectiveness as an adsorbent.

FTIR analysis provides insights into the active functional groups on the surface of the carbon materials, as well as structural transformations resulting from carbonization and chemical activation processes. It helps predict the potential adsorption performance of the material and explains the conversion of biomass into a value-added product.

The FTIR spectra of the carbon materials derived from willow and poplar tree stem waste are presented in Figures 4 and 5, respectively.

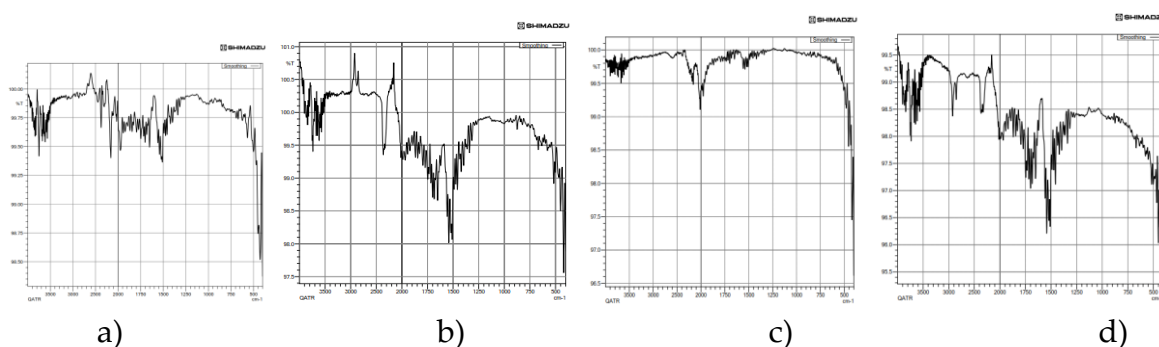


Figure 4. FTIR spectra of coal adsorbents derived from willow tree stem waste: (a) raw willow stem material; (b) FU-1; (c) FU-2; (d) FU-3

The table below presents a comparative analysis of the infrared (FTIR) spectra for samples obtained from willow tree stem waste, including the raw material and activated carbon samples FU-1, FU-2, and FU-3, as shown in Figure 4.

Sample	Main Peaks (cm ⁻¹)	Spectral Assignment	Analytical Interpretation
Raw Material	~3410, ~2920, ~1730, ~1600, ~1030	–OH (hydroxyl), C–H (aliphatic), C=O (carbonyl), C=C (aromatic), C–O (ether/alcohol)	Presence of alcohols, acids, and cellulose/lignin-based structures. High intensity of hydroxyl and carbonyl bands indicates rich oxygen-containing groups.
FU-1	~3390, ~2920, ~1700, ~1580, ~1030	O–H, C–H, C=O, aromatic C=C, C–O	Post-activation, the number of functional groups decreases, though still detectable. The weakened O–H peak suggests reduced hydroxyl/water content.
FU-2	~3400, ~1620, ~1030	Weak O–H, C=C (aromatic), C–O	Hydroxyl and carbonyl bands are significantly diminished. Oxidized functionalities decrease while aromatic carbon matrix becomes more dominant.
FU-3	~3400, ~1620, ~1030	Very weak O–H, C=C, C–O	A high degree of carbonization is evident. Most functional groups are eliminated. Main absorption is due to aromatic carbon and C–O bonds, indicating enhanced adsorptive activity.

The FTIR spectra clearly demonstrate that significant structural transformations occurred in the samples as a result of chemical activation with KOH. The decrease in the –OH absorption band (~3400 cm⁻¹) corresponds to the decomposition of hydroxyl groups and moisture, which occurs during the carbonization phase under the influence of heat and chemical reactions. The weakening of aliphatic C–H bands (~2920 cm⁻¹) suggests the degradation of lipid-like components present in the raw biomass, likely due to high-temperature interactions with KOH.

A reduction in the carbonyl (C=O) peak intensity (~1700 cm⁻¹) indicates possible reactions of these groups with the alkaline agent or their volatilization as CO₂ during pyrolysis. In contrast, the enhancement of the aromatic C=C signal (~1600 cm⁻¹) reflects an increased degree of graphitization, implying that the aromatic carbon framework becomes more ordered and consolidated during activation. The intensified C–O absorption bands (~1000–1100 cm⁻¹) imply the formation or retention of ether and acid-type oxygen-containing functional groups on the surface, which are beneficial for adsorption applications.

Overall, the FTIR analysis confirms that KOH-induced chemical activation of willow biomass-derived precursors leads to the modification and partial preservation of oxygen-containing functional groups (such as C–O and –OH), while simultaneously promoting the development of a stable aromatic carbon structure. These features are indicative of a highly functional and efficient adsorbent surface, which is essential for practical adsorption applications.

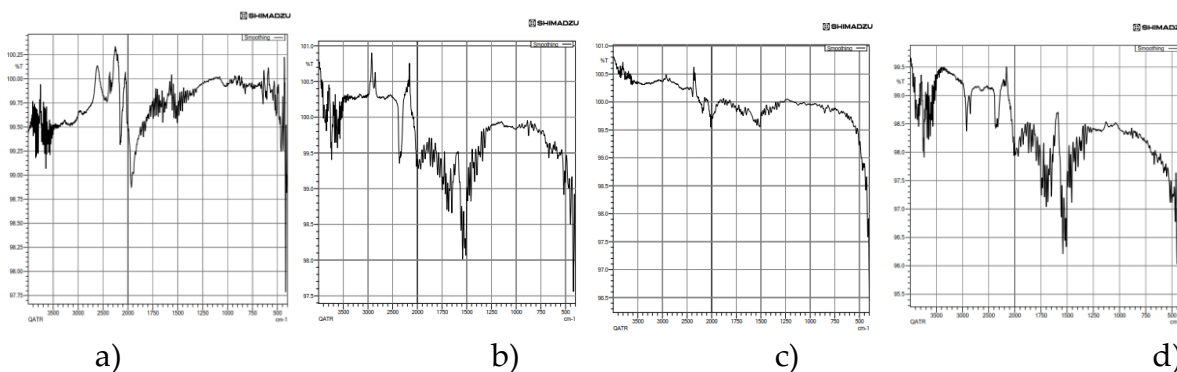


Figure 5. FTIR spectra of coal adsorbents derived from poplar stem waste: a) raw poplar biomass; b) FU-4; c) FU-5; d) FU-6.

For the raw poplar biomass, the broad absorption band around 3400 cm^{-1} is attributed to O–H stretching vibrations, indicative of hydroxyl groups present in alcohols, phenols, and adsorbed water. The band at approximately 2900 cm^{-1} corresponds to C–H stretching vibrations of aliphatic chains. The absorption near 1730 cm^{-1} is assigned to C=O stretching vibrations from carbonyl-containing groups such as carboxylic acids, esters, ketones, or aldehydes. The signal around 1600 cm^{-1} is likely due to aromatic C=C bonds or carboxylate groups. The region between 1000 and 1200 cm^{-1} is characteristic of C–O stretching vibrations, commonly found in alcohols, ethers, and acids.

In the case of sample FU-1, the O–H peak slightly diminishes, suggesting a reduction in hydroxyl functionalities due to the activation process. The C=O and C–O groups are still present but with reduced intensity, indicating partial degradation of oxygen-containing functionalities. Additionally, the formation of new aromatic and carbonaceous structures is likely initiated at this stage.

For sample FU-2, the O–H peak has nearly disappeared, implying the loss of most water molecules and hydroxyl-containing functional groups. A noticeable decrease in the intensity of C=O and other oxygenated groups further confirms the progressive removal of polar functionalities. The sample predominantly exhibits carbonaceous and aromatic structural features.

In sample FU-3, the structural transformation is more pronounced. The IR spectrum shows very weak signals, primarily associated with aromatic carbon networks and a minimal presence of residual functional groups. This indicates a highly carbonized adsorbent material.

Overall, the FTIR spectra reveal that increasing the amount of KOH during chemical activation leads to a gradual reduction in surface functional groups, while enhancing the development of aromatic and carbon-rich structures. This transformation contributes to the formation of a highly porous material with improved adsorption capacity.

Conclusion/Recommendations. The physicochemical properties of activated carbon adsorbents derived from poplar and willow tree stem waste were investigated. The samples obtained through carbonization and thermal treatment processes were analyzed using Fourier-transform infrared spectroscopy (FTIR) and thermal analysis

(TGA/DTA). FTIR analysis confirmed the presence of oxygen-containing functional groups, indicating that the adsorbents possess active surface sites. Thermal analysis demonstrated their stability under heat exposure. The results of the study validate that the adsorbents produced from biomass waste are environmentally friendly, cost-effective, and efficient materials that can be applied in industrial purification processes. This approach allows for reduced environmental impact by utilizing renewable resources.

From an ecological standpoint, this method is based on the secondary use of biological waste, contributing to the reduction of environmental pollution, waste disposal, and conservation of natural resources. The fact that these carbon adsorbents are derived from renewable, inexpensive, and widely available biomass sources makes them environmentally safe and economically viable products.

In conclusion, activated carbon adsorbents obtained from poplar and willow tree waste demonstrate high efficiency, thermal stability, and environmental benefits. Therefore, they can be recommended as promising materials for addressing environmental challenges and for the treatment of industrial effluents.

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