Chemical Analysis of High Energy-Efficient Active Sorbents

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Abstract. In this article, the chemical composition of sorption materials activated from paulownia tree wood and bark waste was comprehensively analyzed, including the quantitative content of elements and oxides. Changes occurring in the raw material composition before and after activation were investigated through the observation of variations in mass composition. The identified structural and compositional changes were subjected to detailed analysis. Considering the requirements imposed on adsorbents, where the carbon content is expected to exceed 86%, the activation results demonstrate significant enrichment in carbon. Specifically, while the initial carbon content of the raw material was C = 54.2%, this value increased to C = 85.0% after thermal activation and reached C = 98.7% following steam activation. The research results were obtained based on energy-dispersive X-ray fluorescence (EDXRF) analysis, which enabled a comparative characterization of the elemental and oxide compositions of the raw materials before and after the activation process.

INTRODUCTION

At present, issues related to environmental protection have gained significant importance. This is primarily due to the fact that the environmental pollution indicator has reached critically high levels, which is characterized by a decrease in the oxygen content of the air and an increase in the concentration of heavy molecular gases. One of the most fundamental and pressing challenges in ecology and environmental protection today is air pollution, alongside emissions released into the atmosphere. One of the key factors determining human health and longevity is not only the consumption of clean water but also the inhalation of clean air. Currently, the rapid increase in the number of industrial enterprises has led to a sharp rise in atmospheric pollution. This is because industrial facilities release various toxic gases into the environment as waste emissions, resulting in air contamination by complex mixtures of heavy gases with diverse compositions. Such pollution poses a serious threat to human health and contributes to the increasing prevalence of respiratory and asthmatic diseases.

Taking into account the relevance of the problem, the main objective of this scientific research was defined as follows. In oil and gas production enterprises, zeolites are currently used as catalysts for drying natural gas supplied for public consumption and for removing toxic sulfur dioxide. However, the gases consumed by the population are not completely purified before being delivered for domestic use. As a result, during daily cooking activities, household users—particularly women—are exposed to partially combusted and harmful gases, which may lead primarily to gastric cancer as well as diseases of the upper respiratory tract. In this context, the use of kitchen ventilation hoods is considered a practical means of preventing health disorders that pose a threat to human well-being [1–2].

The present study focuses on investigating the adsorption mechanism of a carbon-based adsorbent with high adsorption capacity, derived from plant-based raw materials, as a substitute for imported adsorbents, and on evaluating its application in gas adsorption processes. Based on the selected raw materials used as sorption media, activated adsorbents were prepared, and comprehensive investigations were conducted on the physicochemical and colloidal properties of carbonaceous materials before and after activation. Furthermore, the application of these materials according to their properties and nature is considered an important factor in addressing large-scale challenges related to environmental protection and ensuring the chemical and biological safety of ecosystem infrastructure.

In industrial manufacturing enterprises, there is a high potential for producing activated adsorbents from large quantities of waste materials generated as a result of chemical and mechanical processing during production processes. In this context, sorption materials can be obtained by selecting appropriate activation methods for these waste masses. Initially, it is advisable to determine the carbon content of the selected raw materials in accordance with adsorbent requirements and, based on an investigation of their chemical composition, to organize the pyrolysis process in a manner compatible with the existing infrastructure of industrial enterprises.

Depending on the physicochemical properties of sorption materials during activation processes, carbon adsorbents are hereinafter referred to as activated carbon materials, abbreviated as ACMs (Activated Carbon Materials). For the production of adsorbents, surface solids with a highly porous internal structure and a high degree of dispersity, which are widely available in nature, are mainly selected. According to the analysis of the literature [116; pp. 3–13], various types of sorbents are produced from different raw material sources using a wide range of organic wastes, such as coconut shells, fruit stone shells, rice husk residues, and wood waste from the furniture industry, among many others. The principal role of sorbent materials in the adsorption process is associated with their high specific surface area (S). The parameters that characterize the ability of a material to function as an effective adsorbent include a high specific surface area, low ash content, and developed porosity. When developing adsorbents and recommending their application in specific industrial sectors, the composition of the selected raw materials and their carbon content are of critical importance [14–20]. It is well known that coal (both lignite and hard coal), which is naturally considered a semi-finished adsorbent, contains a high proportion of carbon. However, there are other types of raw materials that are widely distributed in nature, and the utilization of their waste as adsorbents can contribute to solving numerous economic and environmental problems. It is known that in countries with well-developed forestry sectors and extensive forested areas, such as Russia and Belarus, the raw material base for activated adsorbents is largely based on birch wood. Adsorbents produced from this raw material are purchased as imported products for use in certain industrial sectors of Uzbekistan [21–25]. Based on the analysis of the results obtained in this study, recommendations are proposed for the development of import-substituting materials [26–31].

METHODOLOGY

Based on energy-dispersive X-ray fluorescence (EDXRF) analysis, the elemental and oxide compositions of raw materials before and after activation were analyzed using a rapid qualitative and quantitative determination method for major and minor elements present in various samples. Owing to the multifunctionality of this method, the study enabled multi-element analysis over a wide concentration range—from parts per million (ppm) to high mass fractions (% by mass)—for elements ranging from sodium (^{11}Na) to uranium (^{92}U). This spectrometric technique is particularly suitable for the semi-quantitative determination of elemental composition in samples with completely unknown matrices. The spectrometer is characterized by high analytical performance, as well as versatility and ease of operation. In addition, it offers a broad range of applications, being effectively used in processes ranging from scientific research and development to industrial and factory-based quality control. The instrument developed by Rigaku represents a reliable method for determining fundamental research parameters and is based on algorithmic software designed for backscattered radiation. This software enables the automatic estimation of concentrations of unidentified low atomic number elements and the application of appropriate corrections. The use of this method for determining the compositional characteristics of raw materials provides essential information for identifying the composition of the primary raw material, which is a critical basis for implementing subsequent stages of the research [32].

EXPERIMENTAL AND RESEARCH RESULTS

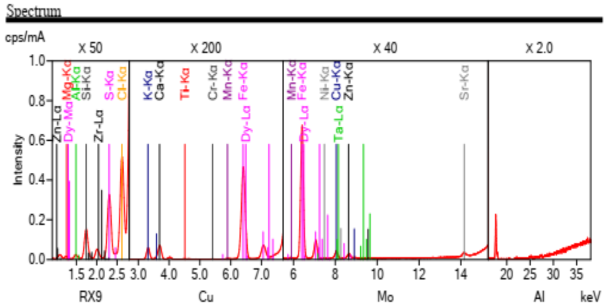
The experimental studies, aimed at the targeted utilization of paulownia tree waste, began with an investigation of its adsorptive properties by examining the elemental and oxide composition of the raw material. The elemental composition of the initial paulownia wood waste was determined using X-ray fluorescence (XRF) analysis (Figure 1). The results indicated that the sample composition is predominantly represented by light elements and alkali-earth metals, which is characteristic of natural lignocellulosic raw materials. The primary elements identified include silicon (Si) at 0.226 wt%, which constitutes a major component of the mineral phase. Calcium (Ca) at 0.363 wt% and potassium (K) at 0.503 wt% are biogenic elements that constitute a significant portion of the wood ash composition.

Aluminum (Al) at 0.104 wt% and magnesium (Mg) at 0.125 wt% indicate the presence of aluminosilicate and magnesium-containing mineral phases. In addition, iron (Fe) at 0.242 wt% occurs as a natural microcomponent. Elements such as chromium (Cr), manganese (Mn), nickel (Ni), copper (Cu), zinc (Zn), strontium (Sr), and zirconium (Zr) were detected in very low amounts (≤0.01 wt%), reflecting the natural geochemical origin of the raw material. Importantly, the absence of high concentrations of heavy metals indicates that this raw material is suitable for the environmentally safe synthesis of adsorbents. The XRF results expressed in oxide form clearly illustrate the composition of mineral phases in the raw material. The main oxides include SiO_2 at 0.483 wt%, confirming the dominance of silicon dioxide in the sample, which provides a basis for the future formation of silicon-carbon composite structures. CaO at 0.505 wt% and K_2O at 0.603 wt% represent the main components of the ash and exhibit strong alkaline properties. Al_2O_3 at 0.185 wt% and MgO at 0.207 wt% indicate the presence of aluminosilicate phases, while Fe_2O_3 at 0.349 wt% plays an important role in the formation of redox-active centers.

Analyzed result(FP method, Scatter)

No.	Component	Result	Unit	Stat. Err.	LLD	LLQ
1	Cl	0.0790	mass%	0.0004	0.0002	0.0007
2	Mg	0.125	mass%	0.0080	0.0180	0.0540
3	Al	0.104	mass%	0.0025	0.0026	0.0078
4	Si	0.226	mass%	0.0022	0.0018	0.0055
5	S	0.0993	mass%	0.0006	0.0004	0.0013
6	K	0.503	mass%	0.0051	0.0036	0.0107
7	Ca	0.363	mass%	0.0033	0.0028	0.0085
8	Ti	0.0060	mass%	0.0003	0.0006	0.0017
9	Cr	0.0015	mass%	0.0001	0.0002	0.0006
10	Mn	0.0056	mass%	0.0003	0.0005	0.0015
11	Fe	0.242	mass%	0.0013	0.0005	0.0015
12	Ni	0.0011	mass%	<0.0001	0.0001	0.0004
13	Cu	0.0051	mass%	0.0001	0.0001	0.0004
14	Zn	0.0029	mass%	<0.0001	<0.0001	0.0002
15	Sr	0.0006	mass%	<0.0001	<0.0001	0.0001
16	Zr	0.0592	mass%	0.0006	0.0001	0.0003
17	Ta	0.0013	mass%	0.0001	0.0004	0.0011

Spectrum

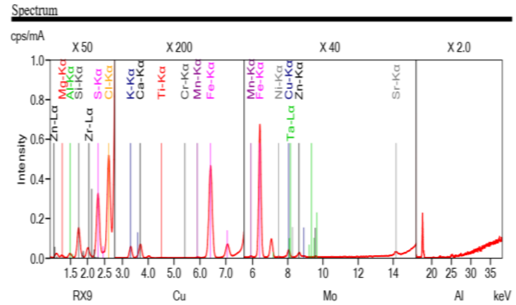


a)

Analyzed result(FP method, Scatter)

No.	Component	Result	Unit	Stat. Err.	LLD	LLQ
1	Cl	0.0786	mass%	0.0004	0.0002	0.0007
2	MgO	0.207	mass%	0.0132	0.0296	0.0889
3	Al2O3	0.195	mass%	0.0048	0.0049	0.0147
4	SiO2	0.483	mass%	0.0046	0.0039	0.0117
5	SO3	0.247	mass%	0.0014	0.0011	0.0032
6	K2O	0.603	mass%	0.0061	0.0043	0.0128
7	CaO	0.505	mass%	0.0046	0.0039	0.0118
8	TiO2	0.0100	mass%	0.0005	0.0009	0.0028
9	Cr2O3	0.0022	mass%	0.0001	0.0003	0.0009
10	MnO	0.0067	mass%	0.0004	0.0006	0.0018
11	Fe2O3	0.349	mass%	0.0018	0.0003	0.0010
12	NiO	0.0014	mass%	<0.0001	0.0002	0.0005
13	CuO	0.0064	mass%	0.0002	0.0002	0.0005
14	ZnO	0.0035	mass%	<0.0001	<0.0001	0.0002
15	SrO	0.0007	mass%	<0.0001	<0.0001	0.0001
16	ZrO2	0.0795	mass%	0.0008	0.0001	0.0004
17	Ta2O5	0.0015	mass%	0.0002	0.0005	0.0014

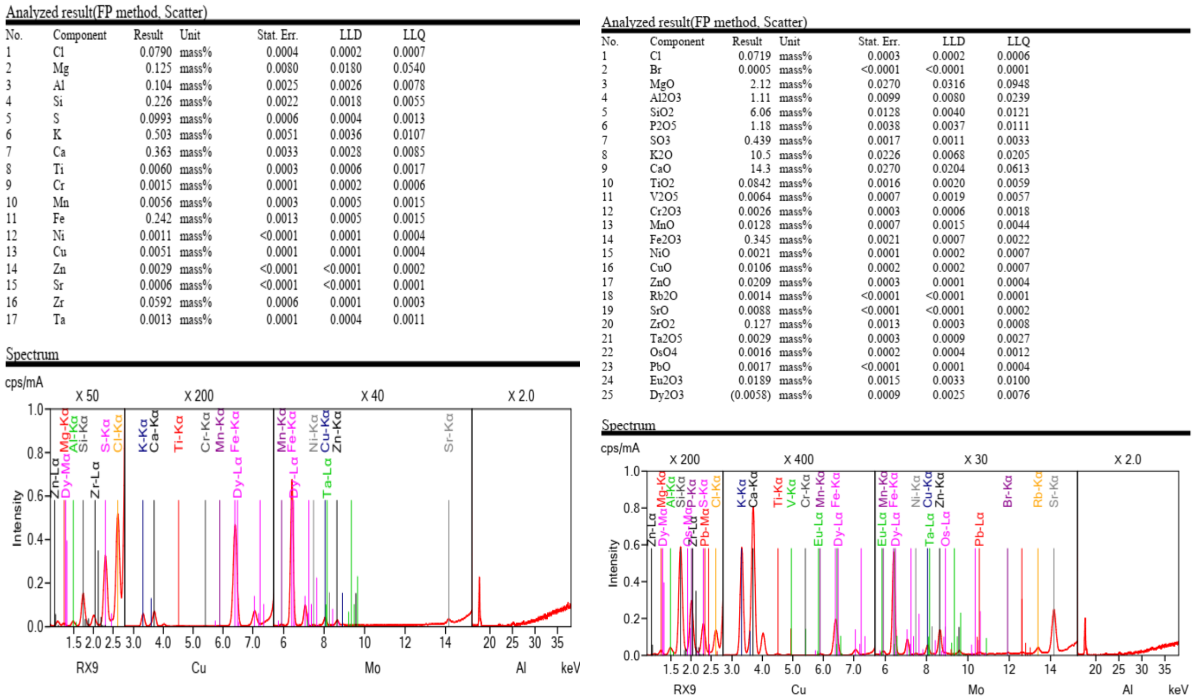
Spectrum



b)

FIGURE 1. Elemental (a) and oxide (b) composition of the initial Paulownia wood waste

Additionally, small amounts of oxides such as TiO_2, MnO, Cr_2O_3, NiO, CuO, and ZnO were detected. Oxides like Fe_2O_3 and TiO_2 can exhibit catalytic effects during the activation process, potentially accelerating the formation of a porous structure in the raw material. The presence of K_2O and CaO acts as natural agents that promote gas release and carbonization processes at high temperatures. Paulownia wood waste can be considered a mineral–carbon hybrid system. The SiO_2–Al_2O_3–CaO–K_2O system provides a favorable environment for the subsequent formation of micro- and mesoporous structures. The presence of natural alkali and transition metal oxides indicates that this raw material is highly suitable for chemical or thermal activation. Low concentrations of heavy metals increase the potential for environmentally safe application of the resulting adsorbents. In the course of the study, the chemical composition of the selected raw material after activation—specifically, the content of elements and oxides—was investigated (Figure 2). After activation, the paulownia wood waste is abbreviated as PPAU-A, i.e., Paulownia pyrolysis activated carbon adsorbent. Its elemental (1) and oxide (2) compositions are presented in mass%. Among the mineral components in the sample, the following elements were predominant: K = 0.503% and Ca = 0.363%, which constitute the main part of the ash–mineral residual phase in the wood matrix. These elements can subsequently form surface alkaline/basic centers. Silicon (Si) at 0.226% corresponds to the siliceous phase—primarily SiO_2/silicates—contributing to structural rigidity and mechanical stability in the carbon matrix. Iron (Fe) at 0.242% was also observed, providing the potential for the formation of redox-active centers during activation and subsequent applications. Other elements, such as Mg (0.125%), Al (0.104%), and S (0.0993%), were present at moderate levels, while Ti, Cr, Mn, Ni, Cu, and Zn were detected in very low concentrations. In the wood waste, the total content of mineral additives is low and is mainly concentrated within the K–Ca–Si–Fe system. The oxide composition of the raw material, expressed in terms of phase distribution, is as follows: K_2O = 0.603% and CaO = 0.505%. The predominance of these alkali-earth oxides enhances the fundamental nature of the material's surface.



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4	Si	0.226	mass%	0.0022	0.0018	0.0055
5	S	0.0993	mass%	0.0006	0.0004	0.0013
6	K	0.503	mass%	0.0051	0.0036	0.0107
7	Ca	0.363	mass%	0.0033	0.0028	0.0085
8	Ti	0.0060	mass%	0.0003	0.0006	0.0017
9	Cr	0.0015	mass%	0.0001	0.0002	0.0006
10	Mn	0.0056	mass%	0.0003	0.0005	0.0015
11	Fe	0.242	mass%	0.0013	0.0005	0.0015
12	Ni	0.0011	mass%	<0.0001	0.0001	0.0004
13	Cu	0.0051	mass%	0.0001	0.0001	0.0004
14	Zn	0.0029	mass%	<0.0001	<0.0001	0.0002
15	Sr	0.0006	mass%	<0.0001	<0.0001	0.0001
16	Zr	0.0592	mass%	0.0006	0.0001	0.0003
17	Ta	0.0013	mass%	0.0001	0.0004	0.0011

Analyzed result(FP method, Scatter)

No.	Component	Result	Unit	Stat. Err.	LLD	LLQ
1	Cl	0.0719	mass%	0.0003	0.0002	0.0006
2	Br	0.0005	mass%	<0.0001	<0.0001	0.0001
3	MgO	2.12	mass%	0.0270	0.0316	0.0948
4	Al2O3	1.11	mass%	0.0099	0.0080	0.0239
5	SiO2	6.06	mass%	0.0128	0.0040	0.0121
6	P2O5	1.18	mass%	0.0038	0.0037	0.0111
7	SO3	0.439	mass%	0.0017	0.0011	0.0033
8	K2O	10.5	mass%	0.0226	0.0068	0.0205
9	CaO	14.3	mass%	0.0270	0.0204	0.0613
10	TiO2	0.0842	mass%	0.0016	0.0020	0.0059
11	V2O5	0.0064	mass%	0.0007	0.0019	0.0057
12	Cr2O3	0.0026	mass%	0.0003	0.0006	0.0018
13	MnO	0.0128	mass%	0.0007	0.0015	0.0044
14	Fe2O3	0.345	mass%	0.0021	0.0007	0.0022
15	NiO	0.0021	mass%	0.0001	0.0002	0.0007
16	CuO	0.0106	mass%	0.0002	0.0002	0.0007
17	ZnO	0.0209	mass%	0.0003	0.0001	0.0004
18	Rb2O	0.0014	mass%	<0.0001	<0.0001	0.0001
19	SrO	0.0088	mass%	<0.0001	<0.0001	0.0002
20	ZrO2	0.127	mass%	0.0013	0.0003	0.0008
21	Ta2O5	0.0029	mass%	0.0003	0.0009	0.0027
22	OsO4	0.0016	mass%	0.0002	0.0004	0.0012
23	PbO	0.0017	mass%	<0.0001	0.0001	0.0004
24	Eu2O3	0.0189	mass%	0.0015	0.0033	0.0100
25	Dy2O3	(0.0058)	mass%	0.0009	0.0025	0.0076

a) b)

FIGURE. 2. Elemental (a) and oxide (b) composition of Paulownia wood waste after activation (PPAU-A)

This is particularly beneficial for capturing acidic gases or ions, such as H_2S and other acidic components. The SiO_2 content is 0.483%, forming a siliceous phase that acts as a mineral skeleton, enhancing structural stability. Although Fe_2O_3 is present at 0.349%, it is important for catalytic and redox properties, especially in adsorption–oxidation mechanisms. SO_3 is retained at 0.247%, which may correspond to natural organic-sulfate fragments or residues from the processing procedure. Other oxides, such as MgO (0.207%), Al_2O_3 (0.185%), and Cr_2O_3, MnO, NiO, CuO, ZnO, are observed in very low concentrations. Overall, the oxide composition confirms the presence of alkali (K_2O), alkaline-earth (CaO), siliceous (SiO_2), and iron oxide (Fe_2O_3) phases. Although the mineral content is low, it is functionally significant, with the main mineral components being K, Ca, Si, and Fe. The predominance of K_2O and CaO increases the likelihood of forming active surface centers, which enhances interactions with acidic adsorbates. The presence of Fe_2O_3 enables the formation of redox-active sites, which can contribute not only to adsorption but also to catalytic pathways. Very low concentrations of heavy metal oxides such as NiO, CuO, ZnO, and Cr_2O_3 increase the environmental safety of the material and its practical application potential. The numerous peaks in the spectrum indicate a multi-component system with a predominantly low-mineral ash phase.

CONCLUSION

The carbon content is a critical factor in both conventional and non-traditional activated sorbents, and their chemical analyses indicate a high carbon concentration. As a result, the observed changes, mass losses, and the quantity of particles that volatilize or melt into a resinous form within the activation temperature range have been analyzed, allowing important conclusions to be drawn regarding the yield of the resulting adsorbent. Prior to activation, the mineral content in paulownia wood waste is low, with K, Ca, Si, and Fe elements predominating. This reflects a low-mineralized, organic-rich structure characteristic of the wood tissue. In oxide form, K_2O, CaO, SiO_2, and Fe_2O_3 were detected, indicating that the mineral phase initially had a limited share. After activation, the relative concentration of mineral phases sharply increased due to the decomposition and volatilization of organic components. As a result, a K_2O–CaO–SiO_2 dominant system was formed in the material, and the significant increase in CaO and K_2O content indicates enhanced basic (alkaline) properties of the material surface. Furthermore, the retention of Fe_2O_3

(≈0.35 wt%) confirms the presence of redox-active centers in the activated sample. Trace elements (Ti, Mn, Cr, Ni, Cu, Zn, etc.) and their oxides were observed at very low concentrations, enhancing the environmental safety and practical applicability of the material. As a result of activation, paulownia wood waste transforms from a low-mineralized organic raw material into a mineral–carbon active material with alkaline properties. The resulting K_2O–CaO–SiO_2–Fe_2O_3-based system makes the material promising for both adsorption and catalytic applications.

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