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CHEMICAL CHARACTERIZATION OF RAW MATERIALS USED FOR ADSORBENT PRODUCTION

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Abstract: This study investigates the initial and post-activation oxide and elemental composition of tar material selected as the raw feedstock for adsorbent production. The research provides a foundation for determining the subsequent stages of chemical activation, including the selection criteria and consumption rates of activating agents. The obtained results offer critical insights into the overall composition of the material and support informed decisions regarding the optimal pathway for further activation processes.

Keywords: tar product, particle size distribution, temperature, thermal activation, adsorbent, porosity, chemical analysis, elemental and oxide composition.

Introduction. In modern chemistry and technology, the recycling of industrial waste and its conversion into value-added products represents one of the most pressing challenges. Among these, adsorbents are widely utilized in the oil and gas industry, chemical processing, environmental monitoring, and medical applications. The selection of suitable raw materials for adsorbent production is critically dependent on their chemical composition, structural properties, and environmental safety. In this study, a waste tar product generated during the polyethylene production process at the "Uz-Kor Gas Chemical" plant was selected as the raw material. This by-product is often discarded without further processing; however, the presence of carbon-based, metallic, and oxide components in its composition suggests its potential for reutilization as an adsorbent material. In the chemical analysis of the raw material, a Rigaku NEX CG spectrometer was employed to detect elements ranging from Na_{11} to U_{92}. Based on the results of this analysis, compositional changes in the material before and after the pyrolysis process were observed, and its potential adsorptive properties were evaluated.

Today, issues related to environmental protection, the use of renewable resources, and the rational management of industrial waste are at the forefront of research among scientists and engineers across various fields. One of the major industrial enterprises in Uzbekistan, the Uz-Kor Gas Chemical Plant, generates thousands of tons of tar waste annually as a by-product of polyethylene production. If not properly processed, this material poses not only a serious environmental threat but also represents a significant economic loss.

From this standpoint, utilizing tar waste as a precursor for adsorbent production offers an opportunity not only to reduce industrial waste but also to obtain high-performance functional materials. This represents a highly relevant direction from both

scientific and practical perspectives. Accurate determination of the chemical composition of the raw material and monitoring its transformation after pyrolysis are essential steps in evaluating its adsorption capacity. Using a modern analytical device—an EDXRF spectrometer (Rigaku NEX CG)—various elements and oxides were identified, their quantitative distribution was assessed, and key factors influencing process efficiency were established.

Currently, adsorbent materials play a vital role both in industrial applications and in environmental protection by enabling the removal of harmful compounds from various gases and liquids. The development of novel types of adsorbents, enhancement of their adsorption efficiency, and the utilization of locally available industrial waste as precursor materials are among the key priorities of modern science and technology [1–4]. Several researchers have achieved significant scientific and practical outcomes in this field, including: the synthesis of porous carbon-based nanospherical particles from coal residues [5–6]; the preparation of activated carbon sorbent materials from waste products [7]; and the design of composite sorbents—specifically mineral–carbon sorbents—based on a dual-waste raw material selection strategy [8].

He research conducted by the author [9] demonstrated the application of carbon-based composite materials for the adsorption of NO_x gases and tar compounds present in synthesis gas. Tar products derived from biomass pyrolysis exhibited high adsorption capacity. These findings support the notion that tar-based materials have potential for conversion into effective adsorbents.

In the study by the authors [10], the structure and composition of pyrolyzed polyethylene and other polymeric wastes were analyzed using XRF, FTIR, and GC/MS techniques. Such investigations provide reliable data on the compositional changes that occur in raw materials during and after pyrolysis.

Researchers in [11] applied the XRF method to analyze the elemental composition and evaluate the physicochemical properties of various types of sorbents. This research provides a direct scientific basis for assessing tar-derived products as carbon-based adsorbent materials.

The reduction in catalyst activity and its regeneration during the pyrolysis process, along with elemental analysis via EDXRF, were examined by the authors in [12]. Their findings contribute to the understanding of compositional transformations occurring in materials during pyrolysis reactions.

Carbon materials obtained from raw materials via pyrolysis have been evaluated as adsorbents in the studies conducted by the authors [13]. The reliability of the data was significantly enhanced through the integration of XRF results with SEM and BET analyses, as demonstrated in [14].

Taking the above into account, preliminary chemical analyses were carried out to assess the potential of utilizing the brown-black tar waste—produced as a by-product during polyethylene manufacturing—as a precursor for adsorbent synthesis.

II. Experimental Section. For this study, the raw material was selected from tar waste generated during the polyethylene production process at the "Uz-Kor Gas

Chemical" plant. Visually, the material appears as a dark black solid with a rough, irregular surface and a glassy, brittle structure (Figure 1a). Its tendency to fracture into thin flakes or nut-like fragments facilitates its processing. The research began with crushing the bulk tar material into smaller particle sizes. The crushed material was sieved using laboratory sieves with varying mesh sizes (0.315–0.071 mm), and the fraction with a particle size of 0.071 mm was selected as the most suitable for further analysis.

Subsequently, the initial chemical composition of the raw material was determined using a Rigaku NEX CG spectrometer, employing the EDXRF (Energy-Dispersive X-ray Fluorescence Spectrometry) method [15]. This instrument uses polarized X-ray beams to reduce background noise and enables the detection of elements present at low concentrations.

The tar material was mechanically ground and fractionated using laboratory sieves with different mesh sizes (0.315 mm, 0.2 mm, 0.125 mm, and 0.071 mm) to evaluate its density and adsorption characteristics. The sieving process was carried out for 15 minutes using an electric vibratory sieve shaker.

Figure 1. a) Tar product **b) Tar product ground to a particle size of 0.071 mm**

Among the selected fractions, the 0.071 mm particle size was identified as the most suitable. This is due to the higher surface reactivity of the material at this size, which contributes to more effective adsorption performance. Additionally, during the pyrolysis process, this fraction demonstrates uniform heating and efficient gas release, and provides more accurate results in spectroscopic analysis (EDXRF). The tar material at this size appears dark brown, with a soft texture, and is considered ready for both physical and chemical analysis (Figure 1b).

Reducing the material to a fine fraction not only enhances the efficiency of the pyrolysis process but also enables more complete and accurate determination of its chemical composition. Therefore, the 0.071 mm fraction was selected as the optimal choice for all subsequent analyses and adsorption experiments.

As shown in Table 1, the tar material contains alkali and alkaline earth metals, as well as transition metals such as Fe, Zn, Cu, and Al. The highest elemental concentrations were observed for sulfur (S – 0.253 g/%) and its oxide form SO_3 (0.630 g/%). Furthermore, the presence of silicon and its oxide in the tar material can contribute to improved surface area, porosity, and chemical functionality of the resulting adsorbents. This, in turn, may enhance the performance of the adsorbent in various industrial applications.

In the next stage of the chemical process, the tar material was thermally activated at 600 °C for 2 hours under an oxygen-free (inert) atmosphere. Upon completion of the process, the elemental and oxide composition was determined using the EDXRF (Energy-Dispersive X-ray Fluorescence Spectrometry) method.

Table 1. Preliminary chemical composition of the tar product

№	Elemental composition	Quantity (wt.%)	Oxide composition	Quantity (wt.%)
1	Cl	0.0488	Cl	0.0488
2	Br	0.0004	Br	0.0004
3	Mg	0.0703	MgO	0.117
4	Al	0.0556	Al_2O_3	0.105
5	Si	0.0662	SiO_2	0.142
6	S	0.253	SO_3	0.630
7	K	0.0146	K_2O	0.0176
8	Ca	0.0233	CaO	0.0326
9	Ti	0.0016	TiO_2	0.0026
10	V	0.0008	V_2O_5	0.0015
11	Cr	0.0015	Cr_2O_3	0.0022
12	Fe	0.0129	Fe_2O_3	0.0185
13	Cu	0.0014	CuO	0.0017
14	Zn	0.0030	ZnO	0.0037
15	As	0.0002	As_2O_3	0.0003
16	Zr	0.0514	ZrO_2	0.0694
17	Ag	0.0003	Ag_2O	0.0004
18	Hf	0.0012	HfO_2	0.0015
19	Ta	0.0013	Ta_2O_5	0.0015
20	Au	0.0006	Au_2O	0.0006
21	Tb	(0.0019)	Tb_4O_7	(0.0022)
22	Dy	0.0030	Dy_2O_3	0.0035
23	Er	(0.0012)	Er_2O_3	(0.0013)

As shown in Table 1, the initial composition of the raw material selected for adsorbent production contains relatively high concentrations of several key components: S – 0.253 mass%, MgO – 0.117 mass%, Al_2O_3 – 0.105 mass%, SiO_2 – 0.142 mass%, and SO_3 – 0.630 mass%. These compounds are notably more abundant compared to other constituents present in the material. However, during the thermal activation process, a portion of these compounds may be lost due to the influence of high temperatures,

potentially through vaporization or transition into the gas phase. In particular, sulfur-containing compounds such as SO_2 may volatilize during the process.

Figure 1. Tar product subjected to thermal activation at 600°C for 2 hours under an inert (oxygen-free) atmosphere

Thermal activation of the tar material can enhance its adsorption characteristics and improve its efficiency as an adsorbent. This treatment contributes to an increase in surface area, the development of a porous structure, and improved chemical reactivity of the material (Figure 2).

As shown in Table 2, the amount of magnesium increased from 0.0703 g/% (as Mg) and 0.117 g/% (as MgO) to 0.113 g/% and 0.186 g/%, respectively, following activation. Similarly, aluminum increased from 0.0556 g/% (Al) and 0.105 g/% (Al_2O_3) to 0.228 g/% and 0.429 g/%. Silicon content also showed a substantial increase, rising from 0.0662 g/% (Si) and 0.142 g/% (SiO_2) to 0.723 g/% and 1.54 g/%.

In addition, noticeable changes in the mass fractions of other elements such as K, Ca, Cr, and Mn were also observed, indicating a significant alteration in the material's elemental and oxide composition as a result of thermal treatment.

Table 2. Post-activation chemical composition of the tar product subjected to thermal treatment at 600°C for 2 hours

№	Elemental composition	Quantity (wt.%)	Oxide composition	Quantity (wt.%)
1	Cl	0.0426	Cl	0.0425
2	Br	0.0015	Br	0.0015
3	Mg	0.113	MgO	0.186
4	Al	0.228	Al_2O_3	0.429
5	Si	0.723	SiO_2	1.54
6	S	0.0997	SO_3	0.248
7	K	0.376	K_2O	0.451
8	Ca	0.0679	CaO	0.0947
9	Ti	0.0079	TiO_2	0.0131

10	V	(0.0008)	V_2O_5	(0.0014)
11	Cr	0.0151	Cr_2O_3	0.0220
12	Mn	0.0043	MnO	0.0055
13	Fe	0.113	Fe_2O_3	0.161
14	Ni	0.0030	NiO	0.0038
15	Cu	0.0019	CuO	0.0023
16	Zn	0.0023	ZnO	0.0028
17	As	0.0003	As_2O_3	0.0004
18	Rb	0.0002	Rb_2O	0.0002
19	Sr	0.0003	SrO	0.0003
20	Zr	0.0519	ZrO_2	0.0700
21	Ag	0.0003	Ag_2O	0.0003
22	Hf	0.0014	HfO_2	0.0017
23	Ta	0.0013	Ta_2O_3	0.0016
24	Dy	(0.0016)	Dy_2O_3	(0.0018)
25	Yb	0.0018	Yb_2O_3	0.0020

These changes indicate chemical modifications within the material that may contribute to improved performance as an adsorbent. The chemical transformations observed between elements and their oxides during thermal activation lead to the development of new physical and chemical properties in the material.

These results suggest that the residual tar product obtained after pyrolysis should not be classified as waste, but rather as a material with high concentrations of components such as SiO_2, Al_2O_3, and Fe_2O_3—indicating a dense, mineral-rich adsorbent structure. The tar by-product from polyethylene production, when subjected to pyrolysis, has the potential to serve as an inexpensive, stable, and environmentally safe adsorbent for industrial applications.

The analysis of post-pyrolysis changes is illustrated in the diagram presented in Figure 3. Among the detected elements, silicon (Si) exhibited the most significant increase (~+0.66%), indicating the high thermal stability of silicon oxide during the pyrolysis process. In addition, potassium (K) and aluminum (Al) accumulated in the residue as mineral phases derived from decomposed organic matter, while iron (Fe) and calcium (Ca) were likely present in the form of oxides or sulfates, which are commonly found in active adsorbent materials.Smaller increases were also observed for elements such as Mg, Cr, and Ti, suggesting their relative resistance to thermal decomposition. The order of increasing elemental content ($\Delta > 0$) after pyrolysis is as follows: **Si > K > Al > Fe > Ca > Mg > Cr > Ti > Br > Zr.** Conversely, the elements that showed a decrease in concentration ($\Delta < 0$) are ordered as: **Cl > S > Au > Er > Dy > Tb > V**, with the changes being relatively minor in magnitude.

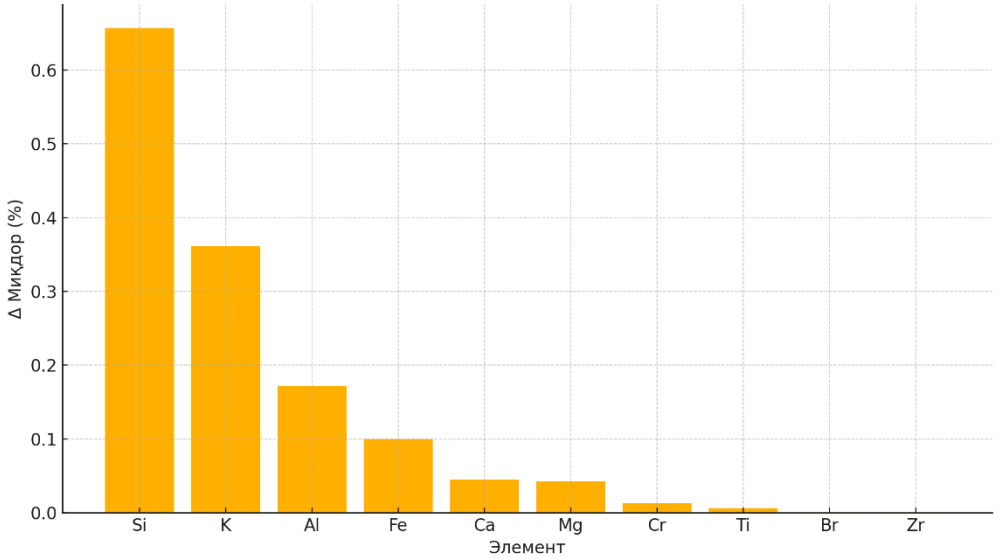

Figure 3. Post-pyrolysis variation in elemental content of the raw material

As a result of the conducted experiments, it was observed that despite the thermal activation process being carried out in an oxygen-free atmosphere, significant changes occurred in the concentrations of Mg, Al, Si and their respective oxides (MgO, Al_2O_3, SiO_2). Compared to the initial state, the relative amounts of these elements and their oxides increased. These changes are primarily attributed to several factors: thermal decomposition of organic compounds and volatile substances led to their release from the material, which in turn increased the relative concentration of the remaining inorganic elements. Structural redistribution within the material and the development of porosity contributed to enhanced surface activity, thus improving the detectability of elements during analysis. Although direct oxidation reactions did not occur in the inert atmosphere, existing oxides were stabilized due to thermal reactivity and structural transformations, leading to a higher proportion of detectable oxide forms. These results indicate that thermal activation can effectively enrich the composition of the material and improve its sorbent properties. In particular, the formation of MgO-, Al_2O_3-, and SiO_2-based structures is considered highly promising for enhancing surface activity and adsorption capacity. Activation in an oxygen-free environment promotes chemical reactivity, while increased oxide concentrations improve the material's ability to capture gases and liquids. Overall, comprehensive chemical and environmental evaluation of tar-based products demonstrates strong potential for practical applications.

III.Conclusions. The results of the pyrolysis process show that the presence and elevated concentration of elements such as Si, Al, K, and Fe enable the formation of carbon–mineral composites with high adsorption capacity. This provides a scientific basis for recommending tar waste as a viable raw material for environmentally and economically beneficial reuse.

Pyrolysis causes a redistribution in the concentration of elemental components, notably increasing the amounts of Si, K, Al, and Fe. This can be attributed to their thermal stability and persistence in oxide or active mineral forms at elevated temperatures. These

elements—especially SiO_2, Al_2O_3, and Fe_2O_3—are commonly found in natural adsorbents and are considered key contributors to enhanced adsorption performance.

Meanwhile, the significant reduction in volatile elements such as S, Cl, and Br—due to their transition into the gas phase—indicates that the post-pyrolysis material is enriched in mineral and carbonate components, making it a promising candidate with high adsorption activity.

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