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COMPOSITIONAL ANALYSIS OF RAW MATERIALS IN SORBENT PRODUCTION

JUMAYEVA DILNOZA
Professor, Institute of General and Inorganic Chemistry, Academy of Sciences of the Republic of Uzbekistan, Tashkent, Uzbekistan
Phone.: (0897) 773-1649, E-mail.: d.jumayeva@list.ru

ZARIPBAEV KUNKHOJA
Professor, Institute of General and Inorganic Chemistry, Academy of Sciences of the Republic of Uzbekistan, Tashkent, Uzbekistan
Phone.: (0893) 713-2093, E-mail.: z.kunxoja@gmail.com

OXUNJONOV ZUXRIDDIN
Senior lecturer, Fergana State Technical University, Fergena, Uzbekistan
Phone.: (0890) 304-7847, E-mail.: zuhridin12345ohunjonov@gmail.com

NOMONOVA ZILOLA
PhD student, Namangan State Technical University, Namangan, Uzbekistan
Phone.: (0893) 544-8898, E-mail.: nomonovazilola7@gmail.com
*Corresponding author

Abstract: This article presents the results of obtaining an adsorbent based on carbon-containing soot waste through chemical activation using hydrochloric acid and sodium hydroxide. As a result, improvements were achieved in the compositional purity of the material and its sorption properties. Data are presented on the elemental and oxide composition of the waste after chemical activation, which enables a reduction in reagent consumption and the removal of harmful compounds prior to subsequent activation stages. The analytical characteristics of the resulting sorption-active adsorbent are substantiated.

Keywords: acetylene soot, chemical activation, hydrochloric acid, sodium hydroxide, chemical composition, elemental content, oxide content.

Introduction. In today's world, the rapid development of global industry has led to the generation of vast amounts of waste, which continues to exert a negative impact on ecology and environmental protection. Addressing the waste problem requires research based on compositional analysis, through which key factors—particularly the quantitative indicators of carbon content, a primary parameter in adsorbent development—can be identified. Utilizing such waste as a raw material for the production of new products contributes not only to solving ecological challenges but also to addressing economic issues. One such type of waste belongs to the category of technical carbon and is known as acetylene soot waste. After conducting a preliminary chemical composition analysis of this waste, the processes of acidic and alkaline treatment using various reagents for the separation of organic substances have been described in this article.

A range of carbon-based raw waste materials, commonly categorized under the term "technical carbon," includes acetylene soot and tar-containing by-products. These materials primarily consist of carbon and represent a collection of substances with structures resembling various degrees of ordered graphite. Their wide availability and the need to reduce their negative environmental impact as waste underscore the importance of targeted processing-specifically, converting them into sorption materials for industrial applications. Investigating the chemical composition of these materials is critically important for the development of effective adsorbents. The selection of appropriate activation methods based on chemical composition analysis has been validated in several scientific studies [1–3].

The analysis of waste generated during industrial processes allows for the selection of carbon-rich raw materials, which can be utilized to produce sorption materials. This approach not only contributes to solving environmental issues but also offers economic benefits by enabling the production of low-cost and highly efficient sorbents. According to current statistical data, acetylene soot—classified as a technical carbon waste and characterized by a high carbon content—represents a significant reserve. For instance, in Navoi alone, the annual volume of acetylene soot generated from calcium carbide production reaches approximately 110 tons [4–6].

Due to its high electrical conductivity within a polymer matrix, acetylene black is used as a conductive agent in batteries. It is produced in the form of fine powders and is therefore typically granulated to facilitate handling and transportation. Simultaneously meeting these two essential requirements-high conductivity and ease of handling-is challenging; thus, granulation is considered a critical step for the industrial application of acetylene black in production processes [7–10].

Experimental Section. In the course of the research [11], chemical activation methods were employed to eliminate interfering compounds present in the waste raw material, with the aim of increasing the carbon content in the sample through activation in the presence of water vapor. The resulting quantitative data, obtained through chemical composition analysis, was substantiated by analytical results. The chemical composition of the raw materials, both before and after treatment, was analyzed using a Rigaku NEX CG X-ray fluorescence (XRF) spectrophotometer, which enables the detection of elements ranging from $_{11}Na$ to $_{92}U$ [12-13]. Figure 1 presents the elemental and oxide composition spectra of the initial chemical content, while Table 1 provides the corresponding quantitative values of the elements and oxides. Elemental analysis was conducted using the XRF method to investigate the chemical composition. For this purpose, acetylene soot waste—classified as technical carbon—was selected as the waste raw material under investigation.

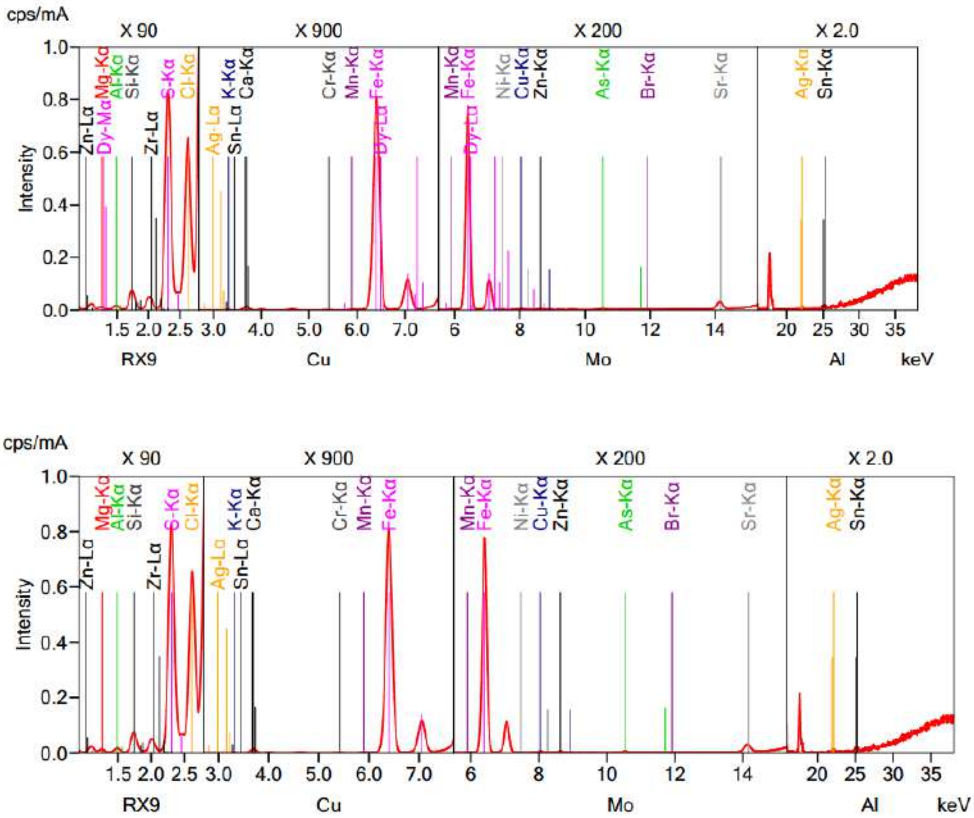


Figure 1. Initial chemical composition of acetylene soot waste raw material in the form of: 1) elements and 2) oxides.

Based on the analysis, the initial chemical composition was found to exhibit a high ash content. The amount of ash was evaluated through kinetic studies involving both chemical and thermal activation processes.

Table 1. Initial chemical composition of acetylene soot raw material in elemental and oxide forms.

No.	Element	Quantity	Oxide	Quantity, wt/%
1	Cl	0.154	Cl	0.153
2	Br	0.0003	Br	0.0003
3	Mg	0.169	MgO	0.281
4	Al	0.112	Al_2O_3	0.211
5	Si	0.171	SiO_2	0.367
6	S	0.377	SO_3	0.939
7	K	0.0227	K_2O	0.0272
8	Ca	0.291	CaO	0.406
9	Cr	0.0021	Cr_2O_3	0.0030
10	Mn	0.0053	MnO	0.0069
11	Fe	1.11	Fe_2O_3	1.61
12	Ni	0.0010	NiO	0.0014
13	Cu	0.0036	CuO	0.0045
14	Zn	0.0023	ZnO	0.0029

15	As	0.0012	As_2O_3	0.0016
16	Sr	0.0029	SrO	0.0034
17	Zr	0.0561	ZrO_2	0.0758
18	Ag	0.0002	Ag_2O	0.0003
19	Sn	0.0005	SnO_2	0.0006

Based on the initial chemical composition of acetylene soot, its ash content was determined, and preliminary treatment was conducted using an acid solution of a specific concentration. The concentration of the acid used for treatment was selected according to the initial chemical composition, and chemical activation was carried out using acid solutions of varying concentrations. To reduce the ash content and increase the carbon content of the acetylene soot, the effect of hydrochloric acid concentration during acid treatment was investigated, and the chemical activation process was optimized accordingly. Among the tested concentrations, a 15% hydrochloric acid solution was identified as the most effective activating agent. As a result of the chemical treatment, a significant reduction in ash content was achieved, along with an increase in the carbon content of the acetylene soot.

The activation of raw materials through acidic and alkaline treatment is carried out to enhance their adsorption capacity, increase surface area, and develop a porous structure. Although no specific national standard (GOST) exists for these activation processes, the selection of reagents was performed in accordance with relevant GOST standards. Specifically, hydrochloric acid (HCl) was used according to GOST 3118-77, sulfuric acid (H_2SO_4) according to GOST 4204-77, nitric acid (HNO_3) according to GOST 4461-77, and hydrofluoric acid (HF) was also considered. However, hydrochloric acid at a 15% concentration was identified as the most effective agent for acid treatment, and subsequent studies were conducted using this concentration. During the acid activation process, the raw material is immersed in an acid solution with a concentration range of 1–20 M at a temperature of 50–90°C for a duration of 30 minutes to 2 hours. During this period, the dissolution of metal oxides and an increase in surface porosity are observed. Following treatment, the samples are washed with distilled water and neutralized until a pH of 7 is reached, then dried at 120°C until the moisture content is reduced to 15–18%.

In the subsequent stage of chemical activation, alkaline treatment was carried out. For this process, alkaline reagents were selected in accordance with relevant GOST standards: sodium hydroxide (NaOH) in accordance with GOST 4328-77, potassium hydroxide (KOH) according to GOST 9285-78, and ammonium hydroxide (NH_4OH) as per GOST 3760-79. Experimental studies were conducted using these selected alkaline reagents. However, sodium hydroxide at a 10% concentration was determined to be the most effective, and further experiments were conducted using this solution. The activation process involved treating the raw material with the selected alkaline solution at a concentration of 1–10 M, maintained at a temperature of 50–100°C for 1 to 2 hours. During this process, the dissolution of silicate and aluminate compounds leads to the formation of a macroporous structure. After treatment, the samples were washed with distilled water and neutralized until the pH reached 7. The materials were then dried at 110–120°C until the moisture content was reduced to 15–18%. It is important to note that specific GOST standards may not exist for certain types of raw materials and their activation methods. Therefore, in each case, it is recommended to study the relevant standards and ensure compliance accordingly.

As a result of chemical activation via acid treatment, the chemical composition of the sorption material is presented in spectral form in Figure 2, and the quantitative values of its elements and oxides are provided in Table 2.

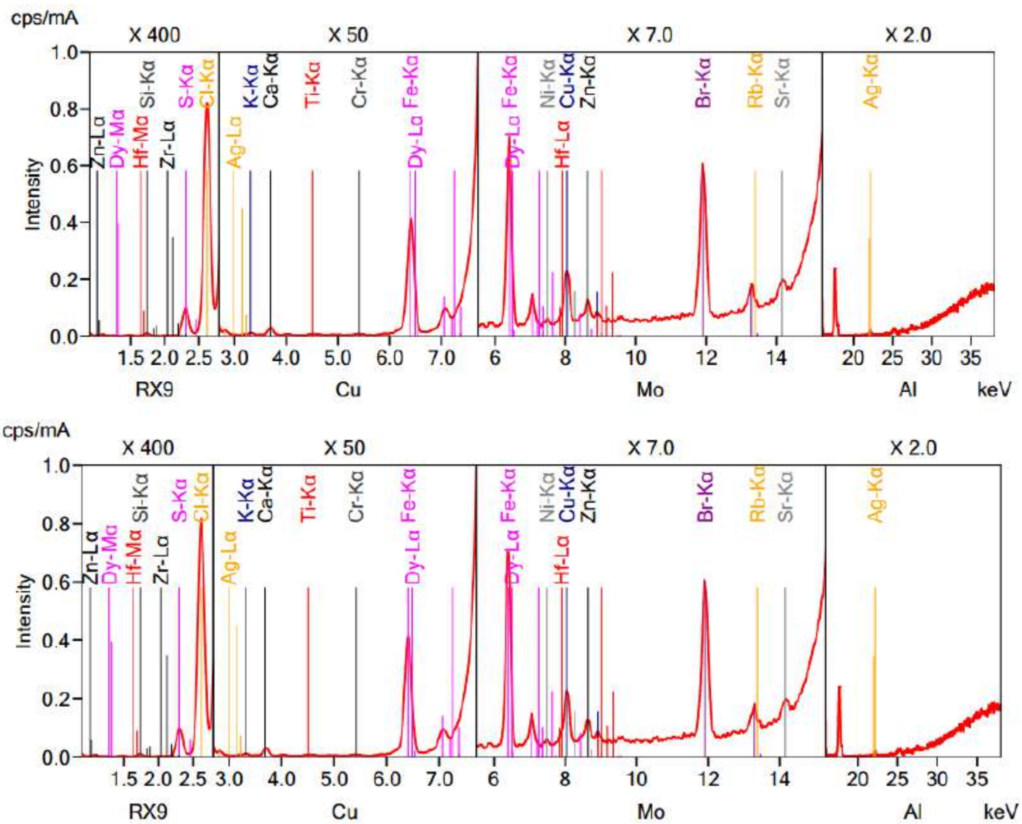


Figure 2. Chemical composition of acetylene soot raw material after hydrochloric acid treatment, presented as: 1) elemental form, and 2) oxide form

The analysis of the chemical composition reveals that iron, sulfate, silicon, and calcium are the primary components, with smaller amounts of zinc, aluminum, and other compounds also present. In the chemical treatment process, analysis of the soot treated with dilute hydrochloric acid showed the following changes in composition as a result of hydrochloric acid exposure, when compared to the initial composition.

Table 2. Chemical composition of acetylene soot before and after treatment with 15% hydrochloric acid (HCl), expressed in weight percent (%).

Element	Initial	Acidic	Oxides	Initial	Acidic
Fe	1.11	0.0317	Fe_2O_3	1.61	0.0452

S	0.377	0.188	SO_2	0.939	0.468
Si	0.171	0.0901	SiO_2	0.367	0.193
Ca	0.291	0.0278	CaO	0.406	0.0389
Zn	0.0023	0.0014	ZnO	0.0029	0.0017

Following chemical interaction with hydrochloric acid, a significant decrease was observed in the concentrations of Fe (1.11% → 0.0317%), Ca (0.291% → 0.0278%), and Si (0.171% → 0.0901%). In contrast, the concentration of Cl (0.154% → 0.805%) increased sharply, indicating the formation of chloride compounds. Additionally, the amount of SO_3 (0.939% → 0.468%) decreased, suggesting its decomposition in the acidic medium. The content of oxides such as Fe_2O_3 (1.61% → 0.0452%) and CaO (0.406% → 0.0389%) also dropped significantly. The analysis shows that, during the chemical reaction, major metal oxides decomposed in the acidic environment, and metals such as Fe, Ca, Si, and Al were converted into soluble chloride compounds [8]. The presence of Fe, Si, S, and Ca elements confirms the impact of hydrochloric acid. The increase in Cl and Br concentrations (Cl: 0.154% → 0.805%) further indicates the formation of new compounds as a result of acid reactions. The following reactions may occur during the acid treatment process:

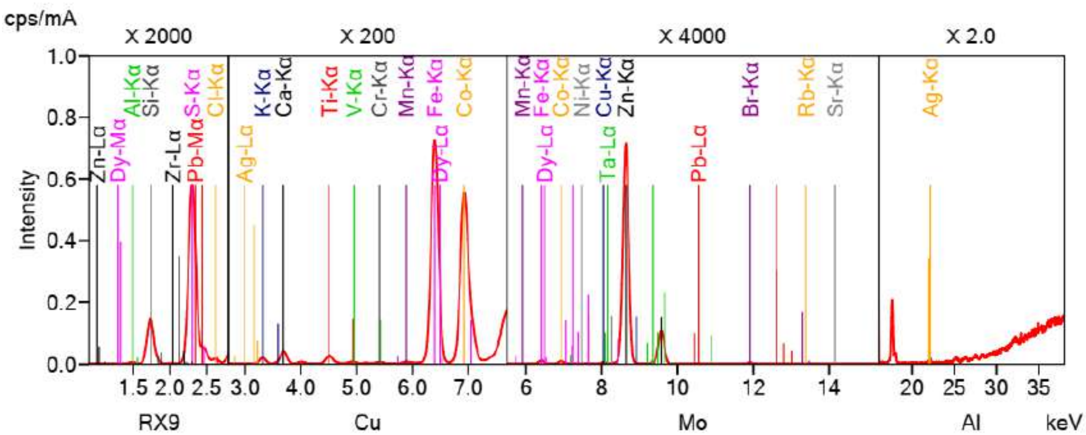
$Fe_2O_3 + 6HCl \rightarrow 2FeCl_3 + 3H_2O$

$SiO_2 + 6HF \rightarrow H_2SiF_6 + 2H_2O$

$CaO + 2HCl \rightarrow CaCl_2 + H_2O$

In the next stage of the study, alkaline activation was carried out using a 10% sodium hydroxide (NaOH) solution. The chemical composition of the products obtained after this activation stage was analyzed. Based on the results, the chemical composition of the sorption materials is presented in spectral form in Figure 3, while Table 3 provides the quantitative values of the elements and oxides.

The analysis of the elemental and oxide composition of the initial soot raw material shows that the zinc (Zn) content accounts for 5.32 g/%, while the silicon dioxide (SiO_2) content among the oxides is 11.8 g/% by mass.



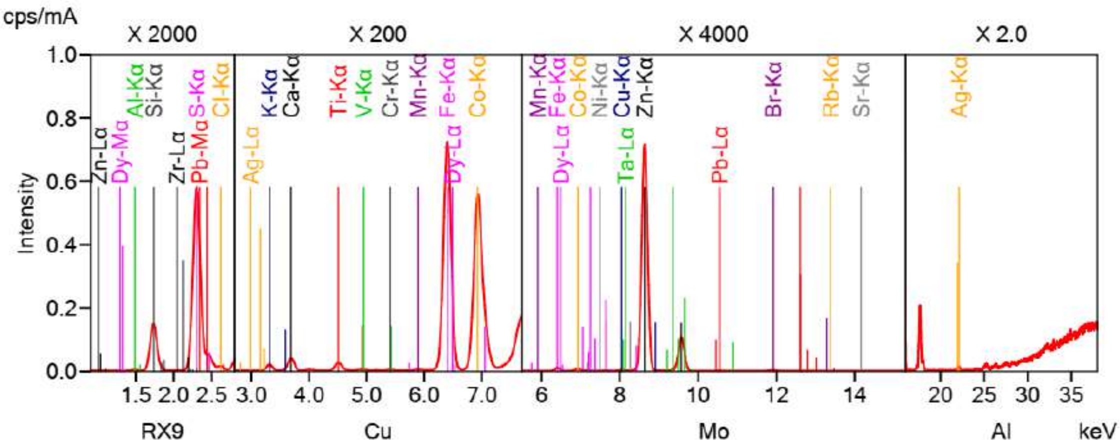


Figure 3. Chemical composition of acetylene soot raw material after alkaline (NaOH) treatment, presented as: 1) elemental composition, and 2) oxide composition

The following changes were observed after the acetylene soot underwent alkaline (NaOH) treatment following the acid treatment stage. A significant increase in the content of Fe, S, Si, and Zn in the raw material indicates that recrystallization or regeneration processes occurred under the influence of the alkali.

Table 3. Chemical composition of acetylene soot raw material before treatment, after acid treatment, and after alkaline treatment (elemental and oxide content in % by weight).

Element	**Initial**	**Acidic**	**Alkaline**	**Oxides**	**Initial**	**Acidic**	**Alkaline**
Fe	**1.11**	**0.0317**	**0.187**	Fe_2O_3	**1.61**	**0.0452**	**0.254**
S	**0.377**	**0.188**	**3.98**	SO_2	**0.939**	**0.468**	**9.50**
Si	**0.171**	**0.0901**	**4.30**	SiO_2	**0.367**	**0.193**	**8.82**
Ca	**0.291**	**0.0278**	**0.123**	CaO	**0.406**	**0.0389**	**0.165**
Zn	**0.0023**	**0.0014**	**4.65**	ZnO	**0.0029**	**0.0017**	**5.52**

The amounts of Si and S elements, which had decreased after acid treatment, were restored upon subsequent alkaline treatment. The recovery of zinc and iron during the alkaline treatment suggests that these elements could potentially be utilized in purification processes. This indicates that the following alkaline reactions may occur during the treatment:

$$Al_2O_3 + 2NaOH + 3H_2O \rightarrow 2Na[Al(OH)_4]$$
$$ZnO + 2NaOH + H_2O \rightarrow Na_2[Zn(OH)_4]$$
$$Fe_2O_3 + 2NaOH \rightarrow 2FeO(OH) + Na_2O$$

Table 4. Chemical composition of acetylene soot raw material after treatment with 10% NaOH solution.

No.	Element	Quantity, wt/%	Oxide	Quantity, wt/%
1	Cl	0.0706	Cl	0.0676
2	Br	0.0173	Br	0.0165
3	AL	0.413	Al_2O_3	0.749
4	Si	4.30	SiO_2	8.82
5	S	3.98	SO_3	9.50
6	K	0.105	K_2O	0.120
7	Ca	0.123	CaO	0.165
8	Ti	0.0319	TiO_2	0.0509
9	V	0.0018	V_2O_5	0.0031
10	Cr	0.21	Cr_2O_3	0.0030
11	Mn	0.0018	Fe_2O_3	0.254
12	Fe	0.187	MnO	0.0022
13	Co	0.0885	Co_2O_3	0.118
14	Ni	0.0031	NiO	0.0037
15	Cu	0.0241	CuO	0.0290
16	Zn	4.65	ZnO	5.52
17	Rb	0.0007	Rb_2O	0.0007
18	Sr	0.0006	SrO	0.0006
19	Zr	0.0766	ZrO_2	0.0989
20	Ag	0.0005	Ag_2O	0.0005
21	Ta	0.0308	Ta_2O_5	0.0347
22	Pb	0.0132	PbO	0.0106
23	Dy	0.0066	Dy_2O_3	0.0069

The elements and oxides with the lowest mass content in the acetylene soot raw material were observed to be silver (Ag), strontium (Sr), and rubidium (Rb), with concentrations of 0.0005 g/%, 0.0006 g/%, and 0.0007 g/%, respectively. Among the key valuable components present in acetylene soot, iron (Fe) is widely used in metallurgy, while silicon dioxide (SiO_2) is applied as a sorbent and in the production of filter materials. Zinc (Zn) may serve as a raw material for zinc metallurgy, and aluminum (Al) is an important material for recycling. The acid and alkali treatment processes affect the chemical composition in specific ways: acid treatment decomposes metal oxides and forms soluble chlorides, whereas alkali treatment enhances the concentration of aluminum and silicate compounds.

Conclusion. Thus, the activation of soot raw material through acid and alkaline treatment was carried out to enhance its adsorption capacity, increase surface area, and develop a porous structure. Based on the results obtained, the application of chemical activation using reagents (acidic and alkaline) was scientifically substantiated as a method for adapting the raw material to meet the requirements for adsorbent production. The analysis of the raw material's composition demonstrated a reduction in the quantities of certain elements and oxides, which in turn led to an increase in its carbon content—this was interpreted as an improvement in the material's purity level.

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