STRUCTURAL FEATURES AND MORPHOLOGICAL ANALYSIS OF CARBON-CONTAINING MATERIALS OBTAINED BY PYROLYSIS OF WORN-OUT AUTOMOBILE TIRES

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Annotatsiya. Ishlatilgan avtomobil shinalarini piroliz usulida qayta ishlash orqali olingan texnik uglerodning fizik-kimyoviy xususiyatlari kompleks o‘rganildi. Tahlil jarayonida asosiy ko‘rsatkichlar - yalpi zichlik, kul miqdori, vodorod ko‘rsatkichi (pH), namlikning massa ulushi hamda maydalangan uglerod moddasining granulometriya tarkibi aniqlandi. Tadqiqot natijalariga ko‘ra, texnik uglerod zarrachalarining o‘lchami kamayishi bilan uning yalpi zichligi, muhit kislotaliligi va namligi oshishi kuzatildi, ammo kul miqdorida sezilarli o‘zgarish qayd etilmadi. Rentgen fazaviy tahlil (RFA) ma’lumotlariga ko‘ra, tadqiq etilgan modda asosan amorf uglerod shaklidan (taxminan 90%) iborat bo‘lib, qolgan qismi mineral va oksid birikmalarining kam miqdori bilan ifodalanadi. Olingan natijalar piroliz uglerodining tuzilishi va fizik-kimyoviy xususiyatlari asosan maydalanish darajasi hamda xom ashyoning termik ishlov berish sharoitiga bog‘liqligini tasdiqlaydi.

Kalit so‘zlar: piroliz, texnik uglerod, rezina, zichlik, kul miqdori, namlik, rentgen fazaviy tahlil, mikroskopiya.

Аннотация. Проведено комплексное исследование физико-химических свойств технического углерода, полученного методом пиролиза изношенных автомобильных шин. В ходе анализа определены основные показатели — насыпная плотность, зольность, водородный показатель (pH), массовая доля влаги, а также гранулометрический состав измельчённого углеродного материала. Установлено, что с уменьшением размера частиц технического углерода наблюдается повышение его насыпной плотности, кислотности среды и содержания влаги, при этом показатель зольности остаётся практически неизменным. Согласно данным рентгенофазового анализа (РФА), исследуемый материал преимущественно состоит из аморфной формы углерода (около 90 %), при этом оставшаяся часть представлена небольшими количествами минеральных и оксидных соединений. Полученные результаты подтверждают, что структура и физико-химические

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характеристики пиролизного углерода зависят главным образом от степени измельчения и условий термической обработки исходного сырья.

Ключевые слова: пиролиз, технический углерод, резина, насыпная плотность, зольность, влажность, рентгенофазовый анализ, микроскопия.

Abstract. A comprehensive study of the physico-chemical properties of carbon black obtained by the pyrolysis of worn-out automobile tires has been carried out. During the analysis, the main parameters such as bulk density, ash content, hydrogen index (pH), moisture content, and the granulometric composition of the ground carbon material were determined. It was found that a decrease in the particle size of carbon black leads to an increase in its bulk density, acidity of the medium, and moisture content, while the ash content remains practically unchanged. According to the results of X-ray phase analysis (XRD), the studied material mainly consists of an amorphous form of carbon (about 90%), with the remaining portion represented by small amounts of mineral and oxide compounds. The obtained results confirm that the structure and physico-chemical characteristics of pyrolytic carbon are primarily dependent on the degree of grinding and the thermal treatment conditions of the raw material.

Keywords: pyrolysis, carbon black, rubber, bulk density, ash content, moisture, X-ray phase analysis, microscopy.

Introduction

In recent decades, there has been a steady global increase in the number of vehicles, which has led to a significant rise in the volume of used and worn-out tires. According to expert estimates, in the countries of the European Union alone, around 7–8 million tons of waste tires are accumulated annually. In the United States, this figure reached approximately 1.8 million tons in 2015, while in the United Kingdom the annual amount is about 450 thousand tons, of which nearly 40% are recycled, 20% are restored, 15% are incinerated, and the remainder is disposed of in landfills.

Existing tire recycling technologies are generally divided into two main groups:

1. Physical methods – mechanical grinding and reuse of rubber crumb.
2. Chemical methods – degradation processes, among which pyrolysis is considered the most promising [1].

Worn-out tires are a valuable secondary raw material since they contain, on average, 60–65% rubber, 18–22% carbon black, and 10–12% metal cord. Therefore, their recycling has both economic and environmental significance.

Under modern conditions, with the tightening of environmental regulations, pyrolysis is recognized as the most efficient and environmentally sound method of tire recycling. This process makes it possible to obtain a wide range of products-gas, liquid fuel, and carbon black. Among these, carbon black occupies a special

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place, as its properties are determined by its physico-chemical parameters. Unfortunately, in many technical descriptions, only basic characteristics such as moisture, ash content, bulk density, sulfur content, and medium acidity (pH) are mentioned, which limits the potential applications of the material.

At the Central Mining Administration of the Navoi Mining and Metallurgical Combine, a pyrolysis plant with a capacity of 1.2 tons per day is currently in operation, providing opportunities for the production and detailed study of carbon black. The investigation of its properties is essential for expanding the industrial applications of this product [2].

LITERATURE REVIEW

The recycling of vulcanized rubber waste to obtain secondary carbon materials is one of the priority directions of modern environmentally oriented chemical technology. According to several researchers [3-4], the **low-temperature pyrolysis method** allows for the effective decomposition of rubber macromolecules and the production of carbon-containing products with a high degree of purity. It has been established that the **physico-chemical properties** of the resulting carbon black-such as **bulk density, ash content, acidity (pH), and moisture**-depend significantly on the **pyrolysis conditions**, including temperature and the composition of the original raw material. Studies conducted by foreign authors show that under **optimal pyrolysis temperatures (450–550 °C), amorphous carbon** is formed with a highly developed **porous structure**, which makes it a promising filler for **polymer composites.** Investigations employing **X-ray diffraction (XRD), infrared (IR),** and **Raman spectroscopy** confirm the presence of **oxygen-containing functional groups** in the structure of pyrolytic carbon, which enhance its **adhesive activity** [5].

Both domestic and foreign sources emphasize the importance of **modification and activation** of such carbon materials to improve their **dispersion, sorption capacity,** and **compatibility with polymer matrices**. Thus, the analysis of the literature confirms the **promising potential of pyrolytic carbon fillers** as effective ingredients for the development of **environmentally friendly and resource-efficient composite materials**.

MATERIALS AND METHODS

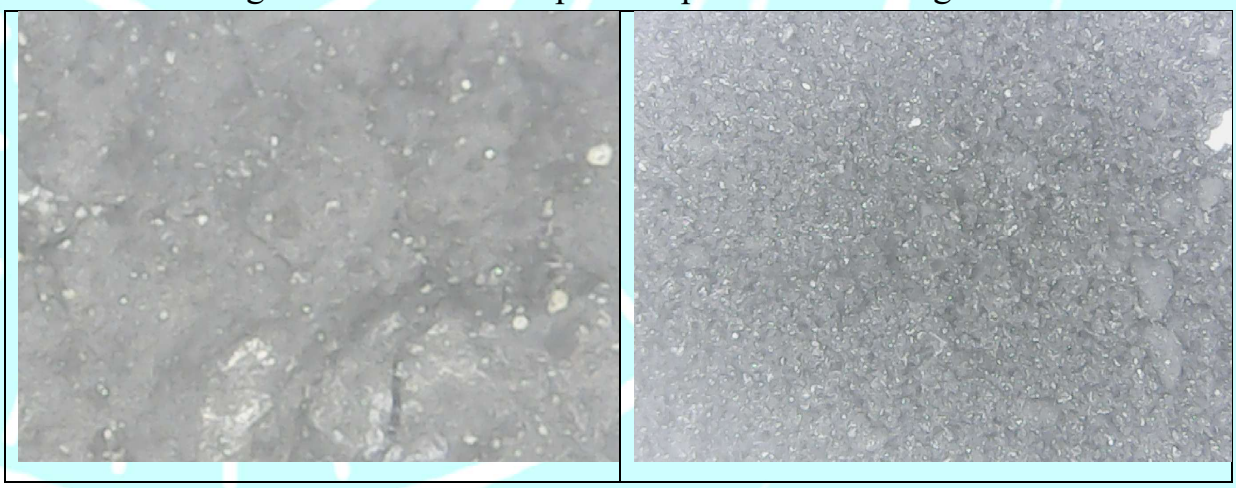
As the object of study, **carbon black obtained by the pyrolysis of worn-out automobile tires** was selected. Several **physico-chemical characteristics** of the obtained material were determined, including: **Bulk density (ρn)** - in accordance with **GOST 16190-70; pH value-** determined according to the methodology described in [6]; **Ash content (Ad)**-determined according to **GOST 1022-95; Moisture content (Wa)**-determined according to **GOST 52917-2008; Granulometric composition**-determined according to **GOST 2093-82**. The **X-ray**

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diffraction (XRD) patterns were obtained using a **Shimadzu XRD-6100 powder diffractometer,** equipped with a **copper (Cu) radiation tube** ($K\alpha_1$ = 1.5406 Å, $K\alpha_2$ = 1.5443 Å, $K\alpha_2/K\alpha_1$ = 0.5) and a **scintillation detector**. The **microscopic analysis** of the samples was carried out using a **Diotox 1500 microscope**, which enabled the observation of the morphological features and particle structure of the carbon material [7–8].

RESULTS AND DISCUSSION

During the process of **thermo-oxidative pyrolysis, carbon black** is formed, which represents a relatively **brittle, lumpy black substance** with a **slightly grayish hue** and a **distinct unpleasant odor** (Fig. 1a). In some fragments of the obtained material, **metallic inclusions** were observed (Fig. 1b), which are residues of the reinforcing cord and steel components present in the original tire structure.

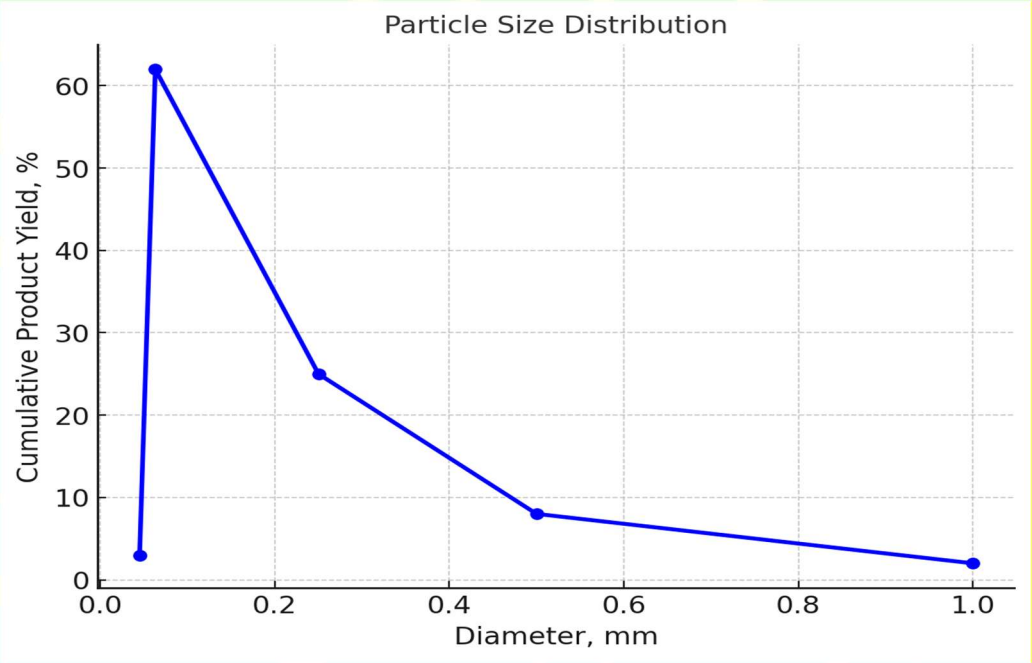


a b

Fig. 1. Microscopic general view of carbon black obtained after pyrolysis

Before use, the carbon black was **ground using a laboratory jaw crusher (model BB 600)**. The **particle size (granulometric) distribution** of the ground carbon black was determined, and the obtained results are presented in **Figure 2.**

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Title: Particle Size Distribution
X-axis: Diameter, mm
Y-axis: Cumulative Product Yield, %

Fig. 2. Granulometric composition of ground carbon black

The analysis of the experimental results (Fig. 2) shows that the **carbon black particles exhibit a heterogeneous particle size distribution**. Among the particles, those with diameters **greater than 0.045 mm and less than 0.25 mm** (approximately **0.063 mm**) make up about **63.0 %** of the total mass. Particles ranging from **0.063 mm to 0.5 mm** (approximately **0.25 mm**) account for **24.0 mass %**, while particles of **0.5 mm and larger** constitute around **9.0 mass %**. The use of carbon black containing **finely dispersed metallic inclusions** as a filler in **rubber-technical products** can negatively affect the quality of the final materials. Therefore, **magnetic separation** was employed to remove these metallic impurities.

After grinding, the carbon black appears as a **dark black, finely dispersed powder**. The ground carbon black can be recommended as a **filler for polymers and polymer-based materials**. In general, **carbon black** is widely used as a **structural filler** in **polyethylene, polypropylene, polyvinyl chloride**, and other polyolefins. The addition of carbon black significantly improves the **durability** of polymer materials and enhances their **resistance to photo-aging**.

The **physico-chemical characteristics** of the carbon black before grinding (sample TU-1) and after grinding (sample TU-2) are presented in **Figure 3**.

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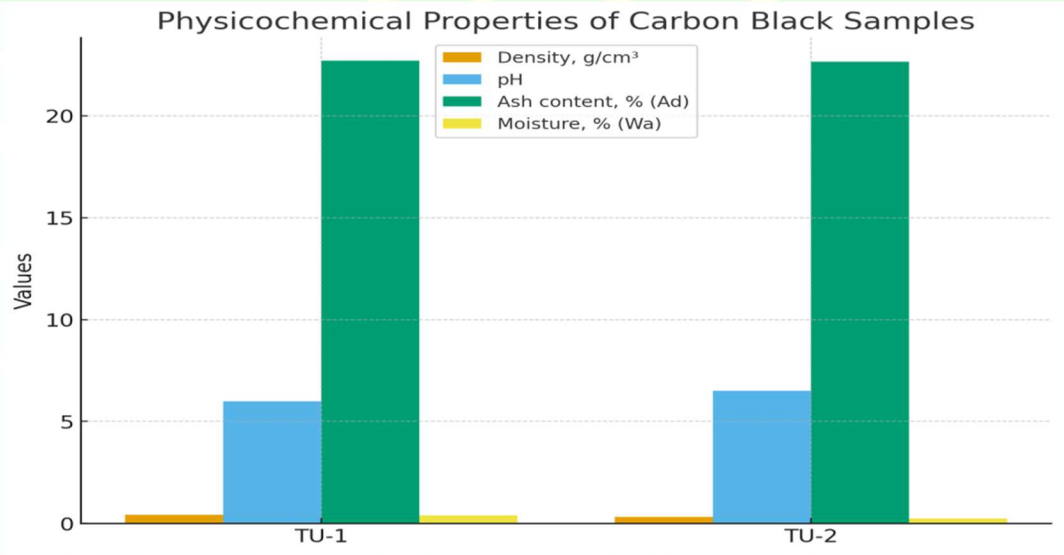


Fig. 3. Physico-chemical characteristics of carbon black before grinding (TU-1) and after grinding (TU-2)

The analysis of the research results (Table 1) shows that **a decrease in the particle size of carbon black leads to an increase in bulk density, acidity, and moisture content**, while **the ash content remains practically unchanged**.

The **increase in acidity and moisture** can be explained by the fact that **smaller particles**, having a **larger specific surface area**, are more capable of **adsorbing oxygen from the surrounding environment**, resulting in the formation of **complex oxygen-containing compounds** on the particle surface. **X-ray diffraction (XRD) analysis** makes it possible to determine the **atomic and molecular structure** of the substance, including the **arrangement of atoms within the crystalline lattice**. When a substance contains carbon, this method helps to identify **which groups or functional groups** are present within its molecular or crystalline structure. For example, carbon can form part of various organic compounds such as **carboxylic acids, amides, esters**, and others.

Thus, XRD analysis enables the determination of **atomic configurations within these functional groups**, providing insight into the **composition and structure** of the material. Using this method, we analyzed the **structural components and functional groups** of the **carbon-containing material obtained by pyrolysis**, revealing the characteristic features of its carbon framework.

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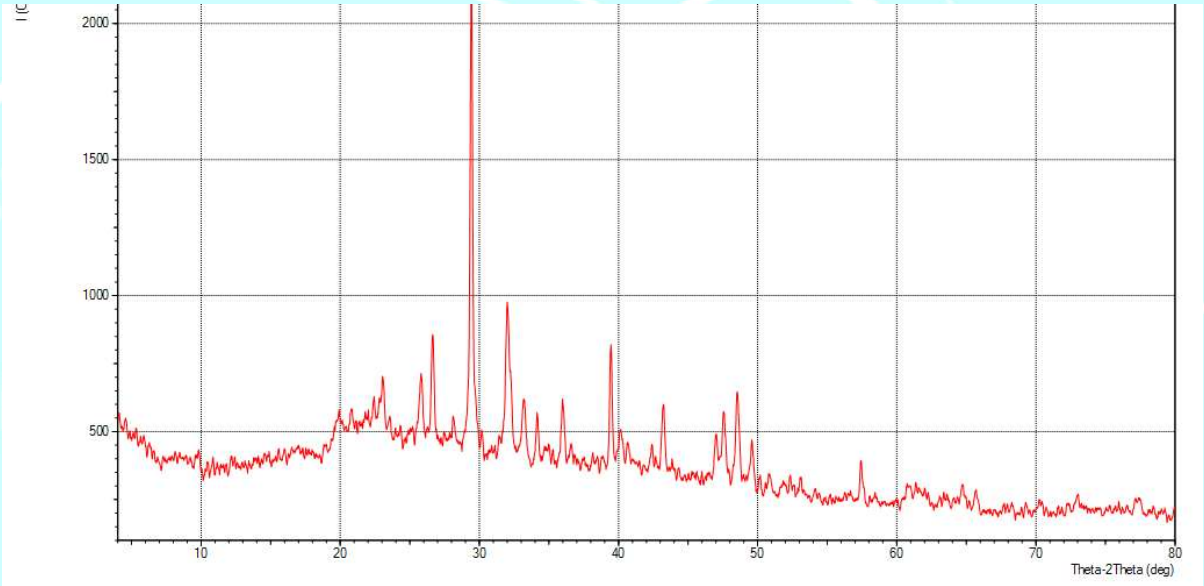


Fig. 4. X-ray diffraction pattern of the carbon-containing material obtained by pyrolysis of worn-out automobile tires.

The **increase in acidity and moisture content** can be attributed to the fact that **smaller carbon particles**, possessing a **larger specific surface area**, are more capable of **adsorbing oxygen from the surrounding environment**. This process promotes the formation of **complex oxygen-containing compounds** on the surface of the material, which in turn affects its **surface chemistry and functional activity**.

CONCLUSION AND RECOMMENDATIONS

As a result of the conducted research, it was established that carbon black obtained by the **pyrolysis of worn-out automobile tires** possesses distinctive structural and physico-chemical characteristics that depend on both **particle size** and **thermal treatment conditions**. The analysis showed that **a reduction in particle size from 0.5 mm to 0.063 mm** led to a **noticeable increase in bulk density**- from **0.323 g/cm³ (TU-1)** to **0.408 g/cm³ (TU-2).** The **pH value** decreased slightly from **6.5 to 5.4**, indicating an increase in surface acidity. Meanwhile, **moisture content** rose from **0.24 % to 0.40 %**, whereas **ash content** remained nearly constant at around **22.7 %**, confirming the chemical stability of the mineral phase. These findings indicate that decreasing particle size enhances the sorption capacity of carbon black due to the enlargement of its specific surface area and oxygen-binding activity. The resulting material, with optimized parameters of **ρ = 0.408 g/cm³**, **pH ≈ 5.4**, and **moisture ≈ 0.4 %**, can be effectively used as a **filler in polymer composites**, as well as a **sorbent or catalyst base** in chemical and environmental technologies.

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