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Physico-Chemical Analysis of the Obtained Solid and Liquid Phases from Worn Tires and Rubber Materials

**Juraev Shokhrukh, Mukhiddinov Bahodir, Tailakov Utkir, Uktam Temirov, Jurabek
Shodikulov, Shodiyev Asliddin**

Navoi State University of Mining and Technology, PhD, Head of the Department of "Agronomy",
Navoi State University of Mining and Technology, Professor of the Department of Chemical Technology,
JSC "NGMK" UMTS, Dep. Procurement of works and services (POZRIU).
Navoi State University of Mining and Technology, Doctor of Technical Sciences, Professor of the Department of
Agronomy.
Navoi State University of Mining and Technology, PhD, associate professor of the Department of Agronomy.
Navoi State University of Mining and Technology, PhD, associate professor of the Department of Agronomy.

ABSTRACT: Every year in the world, the number of used car tires continues to grow, which leads to an increase in the number of waste tires, leaving behind the problem of their disposal. The European Association reports that in Europe alone, tire recycling generates more than 9 million tons of waste tires. In the US this number is about 1.5 million tons, and in the UK almost 500 thousand tons, with partial processing, recovery, incineration and landfilling. Japan generates approximately 96 million waste tires, most of which are recycled. In France and Germany, this number is also significant, amounting to more than 400 and 460–510 thousand tons, respectively. [1-3] In Russia, the annual amount of waste tires exceeds 1 million tons, but only a small part of them can be recycled. According to statistics in our republic, the NMMC alone accumulates about 1 thousand tons of waste tires annually (as of 2018). Among the existing methods for recycling waste tires, thermal decomposition - pyrolysis - is considered the most effective. Using these wastes as a source of raw materials has both economic and environmental benefits. Car tires are a valuable secondary raw material containing approximately 65-70% rubber, 15-25% carbon material and 10-15% metal cord. Among these components, carbonaceous materials are especially important, so their physicochemical characteristics have been studied in detail. [4]

KEYWORDS: Pyrolysis, rubber, carbon material, physicochemical characteristics, raw materials, car tires.

I. INTRODUCTION

Based on the results obtained, a technological procedure was developed for processing waste rubber products and tires. The raw materials are first cleaned and then loaded into a retort (special crucible). In the retort, the raw materials are decomposed at a temperature of 500 to 550 °C, resulting in various products: gas, pyrolysis liquid, carbonaceous material and metal cord. The pyrolysis process is carried out in a hermetically sealed retort, where the rubber heats up but does not burn. The released gases are then sent to the nozzle and pyrolysis unit to be burned along with the wood waste to maintain operating temperature. The gas is diluted with air in a certain ratio to ensure the required temperature. Pyrolysis gases are discharged through a pipe that heats the raw material. During the heating of the raw material and bringing it to operating temperature, thermal decomposition occurs, releasing a large amount of gas, which is then directed to the heat exchanger through a connecting pipe and condenses. The resulting hydrocarbon fractions are cooled and condensed into liquid pyrolysis fuel. The non-condensed fractions are partially sent back to the retort and partially discarded. Water used for cooling is reused. The resulting liquid fuel, metal cord and carbon-containing material are sent to a warehouse for further use. After the liquid is collected in the product sump, it is transferred to a storage tank as it accumulates. The water generated in the process is drained from the bottom of the sump through installed pipes.[5-7] The heat exchanger is cooled by process water, which is then returned to the chilled water pool for reuse. After the process is completed, the furnace cools down, the crucible is removed from it and cools naturally. The lid is then removed from the crucible and the solid residue is discharged. The furnace is prepared for the next cycle by installing the next crucible, which is then loaded with new raw materials.

II. SIGNIFICANCE OF THE SYSTEM

The gas generated during the process is returned to the furnace firebox to maintain temperature. Excess gas can be released through a special pipe. The carbon-containing residue after quenching and cooling is sifted through a sieve to separate the metal cord. Pyrolysis products are mainly represented by gas, liquid and solid phases.

III. METHODOLOGY

The objects of study were carbon-containing material obtained by pyrolysis of worn-out car tires. Some physical and chemical characteristics have been determined, such as bulk density (Pn) - GOST 16190-70; pH was determined according to the method [8], ash content (Ad) - GOST 1022-95; mass fraction of moisture (Wa) - GOST 52917-2008; granulometric composition - GOST 2093-82. Diffractograms were obtained on a Shimadzu XRD-6100 powder X-ray diffractometer equipped with a copper (Cu) tube ($K_{\infty 1} = 1.5406 \text{ \AA}$, $K_{\infty 2} = 1.5443 \text{ \AA}$, $K_{\infty 2} / K_{\infty 1} = 0.5$). Scintillation detector.

IV. EXPERIMENTAL RESULTS

During the process of thermal oxidative pyrolysis, carbon black is formed, which is a relatively brittle, lumpy material that is black in color with a grayish tint and has an unpleasant odor (see Figure 1a). Metallic inclusions can be found in some pieces of this carbon (see Figure 1b).

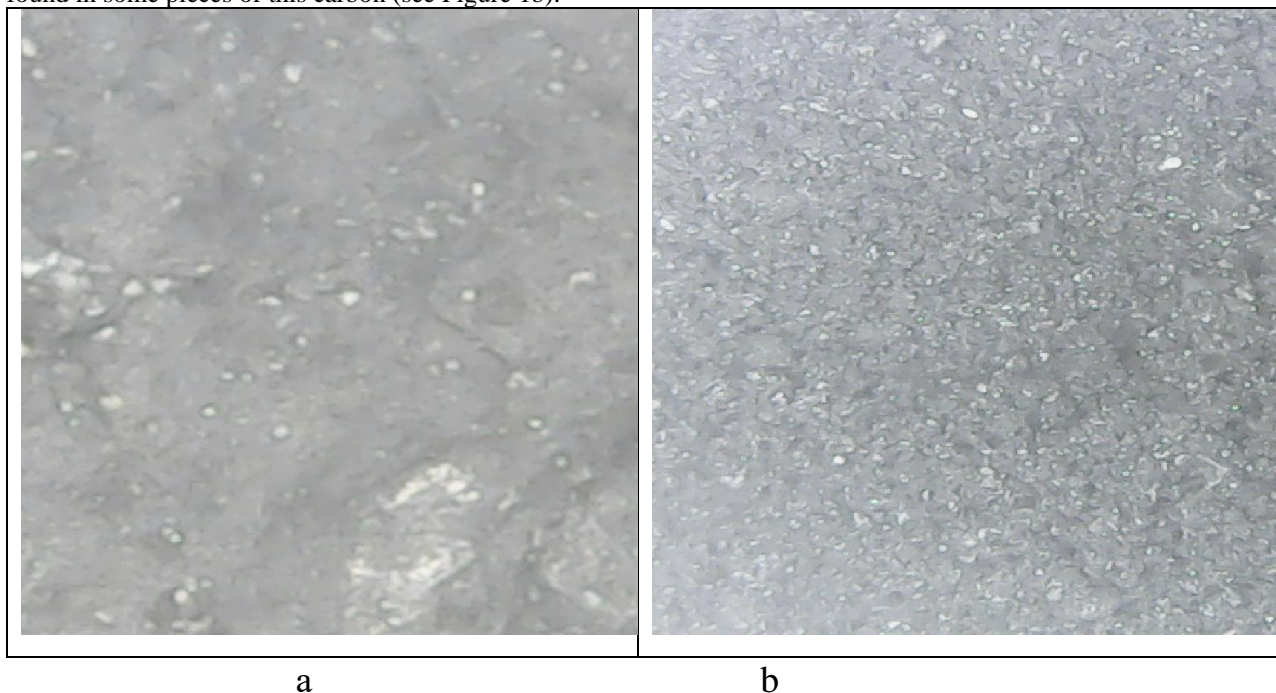


Fig.1. Microscopic general view of carbon black after pyrolysis

Before use, carbon black was crushed using a BB 600 laboratory jaw crusher. The granulometric composition of crushed carbon black was determined, the results of which are shown in Fig.2.

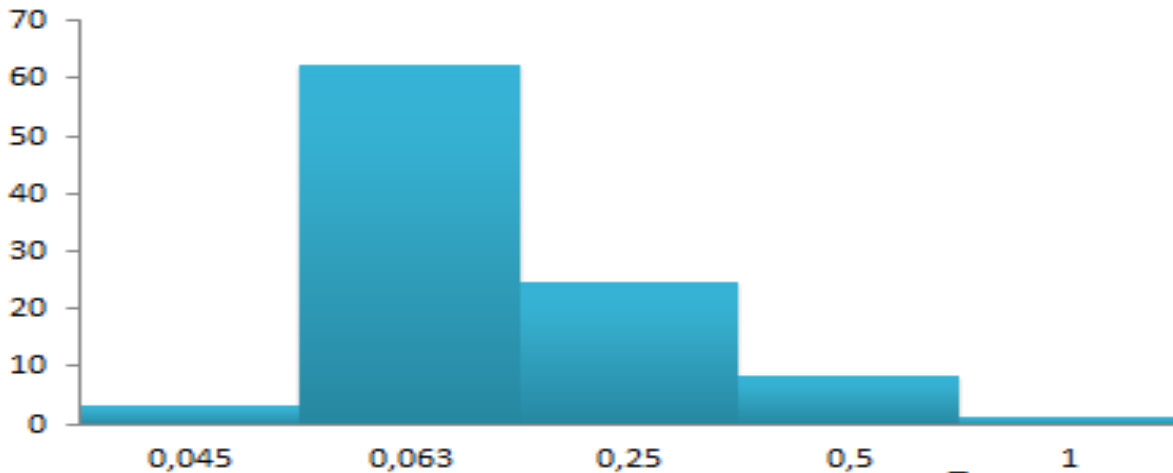


Fig.2. Granulometric composition of crushed carbon black

The analysis results show (see Figure 2) that carbon black particles have different sizes. More than half of the carbon particles range in size from 0.045 mm to 0.25 mm (0.063 mm to 0.25 mm), accounting for 63.0% of the total particle content. Particles ranging in size from 0.063 mm to 0.5 mm (including particles measuring 0.25 mm) constitute 24.0% of the mass. There are also particles of about 0.5 mm in size, which make up about 9.0% of the mass. The use of carbon black with metallic inclusions as a filler for rubber products negatively affects their quality. Magnetic separation is often used to remove these metallic inclusions. After grinding, carbon black is a dispersed powder of dark black color. Crushed carbon black can be recommended as a filler for polymers and polymeric materials. Carbon black is often used as a structural filler for polyethylene, polypropylene, polyvinyl chloride and other polyolefins. The introduction of carbon black helps improve the durability of polymer materials and increases their resistance to light aging.

The physicochemical characteristics of the original carbon black (before grinding U-1) and crushed carbon black (UM-2) are presented in Table 1.

**Table-1
Physico-chemical characteristics of carbon-containing material, initial (before crushing UM-1) and crushed (UM-2)**

Characteristics	$\rho_H, \text{ g/cm}^3$	pH	A ^d , %	W ^a , %
UM-1	0,408 ± 0,02	6,5-5,4	22,70 ± 0,44	0,40 ± 0,05
UM-2	0,323 ± 0,02	6,5	22,65 ± 0,44	0,24 ± 0,05

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, humidity and has virtually no effect on the ash content. The increase in acidity and moisture is due to the fact that smaller particles with a large specific surface area more easily absorb oxygen from the environment to form a complex compound.

Table-2
Characteristics of thermo-oxidative pyrolysis products
worn car tires

№	Name	Exit, %	Product Feature	Recommended application
1.	Pyrolysis gas	13,0	Gas composition: methane-45.0%, ethane-14.9%, propane-5.0%, butane-3.5%, propylene-1.5%, ethylene-2.2%, butene-1.3 %, hydrogen - 17.5%, carbon monoxide - 4.2%, carbon dioxide - 4.9%.	In mini boiler systems, for combustion in pyrolysis reactors.
2.	Liquid fraction	41,0	Density=16948 kg/m ³ Viscosity=1.7957	For the production of aromatic hydrocarbons, petroleum products and heating oil.
3.	Carbon containing material	39,0	Density=0.408±0.02 Viscosity=0.4 ± 0.05 Humidity= 22.7 ± 0.44	For the production of activated carbon, coke and thermal energy and as a filler
4.	Metal cord	7,0	Wires with a partially shiny surface, dark gray color. Presses well.	As scrap metal

Research shows that the gas phase resulting from pyrolysis mainly consists of 45% methane, 14.9% ethane and 17.5% hydrogen. This allows the gas mixture to be used as fuel for car tires in the reactors of pyrolysis plants. Currently, such a gas mixture is reused in reactors as fuel.

Particular attention is paid to the liquid fraction, which is one of the main products of pyrolysis. Work is underway to develop technology for producing alternative diesel fuel from this liquid fraction.

V. CONCLUSION AND FUTURE WORK

The physicochemical characteristics of carbon-containing material obtained by pyrolysis of worn-out automobile tires were studied. The following parameters were determined: bulk density, ash content, pH, mass fraction of moisture and particle size distribution of crushed carbon-containing material. It has been established that a decrease in the particle size of carbon-containing material leads to an increase in bulk density and acidity, and also increases the moisture content, while the ash content remains practically unchanged. Using X-ray phase analysis, it was determined that the carbon-containing material mainly consists of amorphous carbon (88.4%), calcite (7.59%), ankerite (1.21%), zinc oxide (1.14%) and other components. Based on the research conducted, it is recommended to use carbon-containing material as a filler for rubber products.

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