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MUNDARIJA

MUNDARIJA * СОДЕРЖАНИЕ * CONTENTS

THERMAL-OXIDATIVE PYROLYSIS OF WASTE TIRES: PRODUCT CHARACTERIZATION AND POTENTIAL FOR RESOURCE RECOVERY AND BITUMEN MODIFICATION

Sanalar
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Abstract. This study investigates the thermal-oxidative pyrolysis of waste automobile tires as a route for converting secondary raw materials into valuable products. The process was carried out at 550 °C in a batch reactor, yielding gas, liquid, and solid fractions. The gas phase was found to be rich in combustible components, indicating its potential for energy recovery within the process. The liquid fraction exhibited properties suitable for further refining into fuel or chemical feedstocks, while the solid residue represented a carbon-containing material with potential application as a filler in polymer systems. Thermal analysis revealed that the degradation of tire rubber proceeds in two main stages, corresponding to devulcanization and subsequent decomposition of the carbon structure. These transformations govern the formation and distribution of pyrolysis products. The results confirm that pyrolysis of waste tires is an efficient and environmentally sound approach for waste management, offering a viable pathway for resource

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recovery and the production of value-added materials, including modifiers for bitumen and polymer composites.

Keywords: *waste tire, pyrolysis, thermal degradation, pyrolysis oil, carbon black, gas composition, resource recovery, bitumen modification.*

Annotatsiya. Ushbu tadqiqot chiqindi avtomobil shinalarining termal-oksidlovchi pirolizini ikkilamchi xom ashyoni qimmatbaho mahsulotlarga aylantirish usuli sifatida oʻrganiladi. Jarayon 550 °C da davriy ishlovchi reaktorda amalga oshirildi va gaz, suyuq va qattiq fraksiyalarni hosil qildi. Gaz fazasi yonuvchan komponentlarga boy ekanligi aniqlandi, bu uning jarayon davomida energiyani qayta tiklash potensialini koʻrsatadi. Suyuq fraksiya yoqilgʻi yoki kimyoviy xom ashyolarga keyinchalik qayta ishlash uchun mos xususiyatlarni namoyish etdi, qattiq qoldiq esa polimer tizimlarida toʻldiruvchi sifatida qoʻllanilishi mumkin boʻlgan uglerodli materialni ifodaladi. Termal tahlil shuni koʻrsatdiki, shinalar kauchukining parchalanishi ikki asosiy bosqichda sodir boʻladi, bu devulkanizatsiya va keyinchalik uglerod tuzilishining parchalanishiga mos keladi. Ushbu oʻzgarishlar piroliz mahsulotlarining shakllanishi va taqsimlanishini boshqaradi. Natijalar chiqindi shinalarning pirolizi chiqindilarni boshqarish uchun samarali va ekologik jihatdan xavfsiz yondashuv ekanligini tasdiqlaydi, resurslarni qayta tiklash va bitum va polimer kompozitlari uchun modifikatorlarni oʻz ichiga olgan qoʻshimcha qiymatli materiallarni ishlab chiqarish uchun samarali yoʻlni taklif qiladi.

Kalit soʻzlar: *chiqindi shinalar, piroliz, termal parchalanish, piroliz moyi, texnik uglerod, gaz tarkibi, resurslarni qayta tiklash, bitum modifikatsiyasi.*

Аннотация. В данном исследовании изучается термоокислительный пиролиз отработанных автомобильных шин как способ преобразования вторичного сырья в ценные продукты. Процесс проводился при температуре 550 °C в реакторе периодического действия, в результате чего были получены газовая, жидкая и твердая фракции. Газовая фаза оказалась богатой горючими компонентами, что указывает на ее потенциал для рекуперации энергии в процессе. Жидкая фракция обладала свойствами, подходящими для дальнейшей переработки в топливо или химическое сырье, в то время как твердый остаток представлял собой углеродсодержащий материал с потенциальным применением в качестве наполнителя в полимерных системах. Термический анализ показал, что деградация резины шин происходит в два основных этапа, соответствующих девулканизации и последующему разложению углеродной структуры. Эти превращения определяют образование и распределение продуктов пиролиза. Результаты подтверждают, что пиролиз отработанных шин является эффективным и экологически безопасным подходом к утилизации отходов, предлагая жизнеспособный путь для извлечения ресурсов и производства материалов с добавленной стоимостью, включая модификаторы для битума и полимерных композитов.

Ключевые слова: *отработанные шины, пиролиз, термическая деградация, пиролизное масло, технический углерод, состав газа, извлечение ресурсов, модификация битума.*

1.Introduction

The rapid growth of automobile transport has led to a significant accumulation of worn tires, which represent a serious environmental challenge due to their resistance to natural degradation and the risks associated with uncontrolled storage and disposal [1,2]. At the same time, waste tires are a valuable secondary resource containing a high proportion of rubber, carbon black, and metal components, which can be effectively recovered through thermochemical conversion processes [3]. Among various recycling technologies, pyrolysis is considered one of the most promising approaches, as it enables the conversion of waste tires into gaseous, liquid, and solid products with potential industrial applications [4,5]. The gas fraction can be reused as an energy source, the liquid fraction may serve as a precursor for fuels and chemicals, while the solid residue (carbon black) can be utilized in polymer composites and construction materials [6]. In recent years, particular attention has been paid to the use of tire-derived products in bitumen modification. The incorporation of rubber-based additives and carbon materials into bitumen has been shown to improve its mechanical performance, thermal stability, and resistance to low-temperature cracking [7]. Previous studies have demonstrated that waste tire components can enhance the durability of road materials and contribute to the development of sustainable infrastructure [8]. Despite these advances, several challenges remain. In particular, the relationship between pyrolysis conditions and the physicochemical properties of the resulting products is not fully understood. Moreover, the mechanisms of thermal degradation and their influence on product distribution require further clarification. This limits the efficient utilization of pyrolysis products, especially in high-value applications such as bitumen modification and polymer systems [9,10].

The aim of this study is to investigate the thermal-oxidative pyrolysis of waste automobile tires, with a focus on the composition, properties, and potential applications of the resulting products. Special attention is given to the thermal degradation behavior of tire rubber and the characterization of carbon-containing materials as functional additives. The novelty of this work lies in the combined analysis of pyrolysis products and their potential application in resource recovery and bitumen modification systems.

2.Materials and Methods

Waste automobile tires obtained from the Central Material and Technical Base (CMTB) of the Navoi Mining and Metallurgical Combine (NMMC) were used as the raw material in this study. Before processing, the tires were inspected to remove foreign materials such as metal parts and debris, and cleaned from surface contaminants to ensure uniform experimental conditions. Thermal-oxidative pyrolysis was carried out in a batch retort-type reactor under limited oxygen conditions. The reactor was hermetically sealed, and the process temperature was maintained at 550 °C, which corresponds to the range of intensive thermal degradation of tire rubber reported in the literature. During heating, the material decomposed into gaseous, liquid, and solid products. The volatile products were withdrawn from the reactor and directed to a heat exchanger, where condensable fractions were separated as liquid pyrolysis oil, while non-condensable gases were partially reused to sustain the process.

The liquid fraction was collected in a settling tank and characterized separately. To avoid secondary transformations, its physicochemical properties were determined at 450 °C. The analysis included density, viscosity, pH, boiling point, flash point, autoignition temperature, pour point, and molecular weight. The composition of the gas phase was determined using gas chromatography, allowing identification of the main hydrocarbon and non-hydrocarbon components. Thermal behavior of the raw material was investigated using a Labsys Evo derivatograph (Setaram, France) over the temperature range of 50–900 °C. The method enabled simultaneous measurement of mass loss and its rate, providing insight into the thermal degradation stages.

The solid residue obtained after pyrolysis was cooled, mechanically treated, and subjected to magnetic separation to remove metallic inclusions. The resulting carbon-containing material was analyzed in terms of particle size, bulk density, moisture content, ash content, and pH.All experiments were carried out under identical conditions, and the reported results represent average values.

Table 1. Qualitative and quantitative composition of the gas phase of thermal-oxidative pyrolysis of worn car tires

Components	Content of components, %	Components	Content of components, %
Methane	45.0	Hydrogen	17.5
Ethane	14.9	Carbon monoxide	4.2
Propane	5.0	Carbon dioxide	4.9
Butane	3.5		
Propylene	1.5		
Ethylene	2.2		
Butene	1.3		

3.Results and Discussion

The pyrolysis of waste tires generates gas, liquid, and solid products that can potentially be used as modifiers for bitumen. Therefore, the present study focuses on investigating the thermal-oxidative pyrolysis process and analyzing the composition and properties of the obtained fractions.

Based on literature analysis, significant thermal degradation of rubber products based on used tires occurs in the temperature range of 500-550 °C. Therefore, the thermal-oxidative pyrolysis of used tires was carried out at 550 °C. The liquid fraction, however, was characterized at 450 °C to evaluate its physicochemical properties under milder conditions. The pyrolysis products primarily consist of gas, liquid, and solid phases. The composition of the gas phase was analyzed using a

Table 2. Physicochemical properties of the liquid fraction of thermal-oxidative degradation products of worn car tires at a temperature of 450 °C

Components	Content of components, %
Appearance	Dark brown
Mass fraction of volatile substances, % n/b	2
pH	6.5
Boiling point, K	450
Flash point, K	570
Autoignition temperature, K	725
Pour point, K	243
Molecular weight	1000-1200

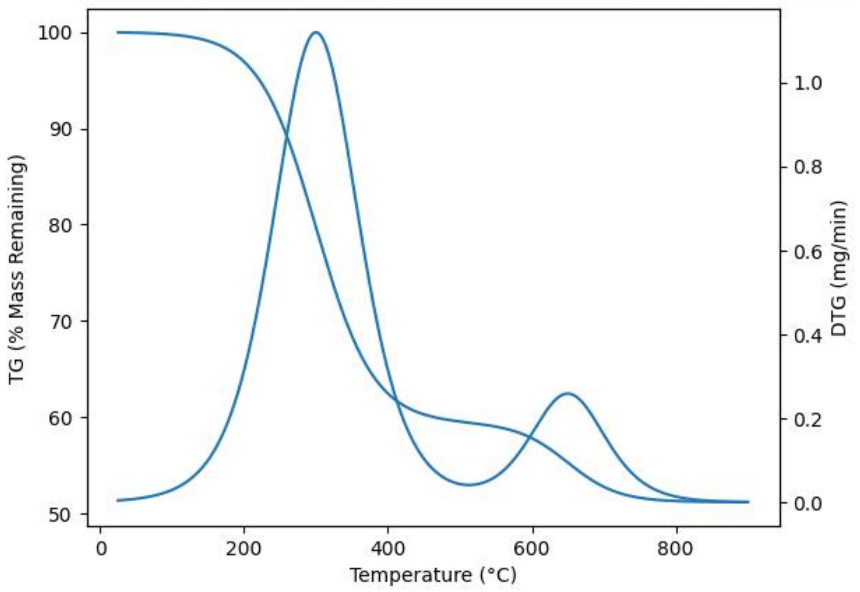

Fig. 1. Derivatogram of worn car tires

gas chromatograph, the results of which are presented in Table 1. The results indicate that the gas phase consists primarily of 45% methane, 14.9% ethane, and 17.5% hydrogen. Therefore, the gas mixture can be reused for burning tires in reactors and pyrolysis units. The physicochemical properties of the pyrolysis product fraction of worn tires were also studied, the results of which are presented in Table 2.

Derivatographic analysis is a modern physicochemical method that simultaneously determines the change in sample mass, the rate of mass loss, and the thermal properties of materials over a wide temperature range. We examined worn car tires using a Labsys derivatograph evo Setaram. The resulting derivatogram is presented in Fig. 1.

The analysis of the derivatogram indicates that the thermal degradation of worn tire rubber occurs in two main stages. The first stage (50–500 °C) is associated with

Table 3. Characteristics of the products of thermal-oxidative pyrolysis of worn car tires

Name of pyrolysis products	%	Physicochemical properties	Recommended area of application
Pyrolysis gas	3,0	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 plants, for firing in pyrolysis reactors.
Liquid fraction	1.0	Density=948 kg/m^3 Viscosity =1.7957	For the production of aromatic hydrocarbons and petroleum products
Carbon-containing materials	9.0	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 filler for rubber compounds
Metal cord	7,0	Wires with a partially shiny surface, dark gray color. Exhibits good compressibility.	As scrap metal

the devulcanization and depolymerization of rubber components. In this temperature range, the cleavage of sulfur cross-links (S–S and C–S bonds) takes place, accompanied by the release of low-molecular-weight volatile compounds. This stage is characterized by an intensive mass loss (40.6%) and a high degradation rate (2.508 mg/min). The second stage (500–800 °C) corresponds to the decomposition of the carbonaceous structure and secondary reactions such as aromatization and carbonization. In this region, the degradation rate decreases (1.837 mg/min), indicating the formation of more thermally stable structures, including carbon-rich residues. At temperatures up to 900 °C, the residual mass (approximately 48.8%) is mainly composed of inorganic components and metal cord, confirming the completion of the thermal decomposition process.

Currently, over 24,000 tons of used tires are stored at the Central Material and Technical Base (CMTB) landfill of the Central Mining Department (CMD) of Navoi Mining and Metallurgical Combine (NMMC). Additionally, 1,800 tons of used tires are written off annually at NMMC divisions, which use them as valuable raw materials in pyrolysis recycling technology. The accumulation and storage of such large volumes of used tires, given the region's hot climate, has a negative impact on the already stressed environment. At the same time, tires at the end of their service life are considered valuable raw materials for various recycling technologies worldwide. Navoi Mining and Metallurgical Combine began searching for an optimal tire recycling technology in 2013. Taking into account the volumes of raw materials, based on a forecast of the plant's capacity to sell rubber crumb, as well as the plant's need for heating oil, specialists from the Navoi State Mining and Metallurgical Institute (NGGI), NGMK, the Central Research Institute (CRI), and the Tashkent Institute of Chemical Technology (TICT) decided to conduct experimental work to develop a technology for the pyrolysis of waste rubber products.

3.1. Description of the technological process and diagram. The feedstock is collected and transported by truck to a waste tire warehouse (Figure 2). For processing, the feedstock is inspected for metal disks and rings, and is cleaned of foreign objects and dirt from the pyrolysis unit. After cleaning, the feedstock is

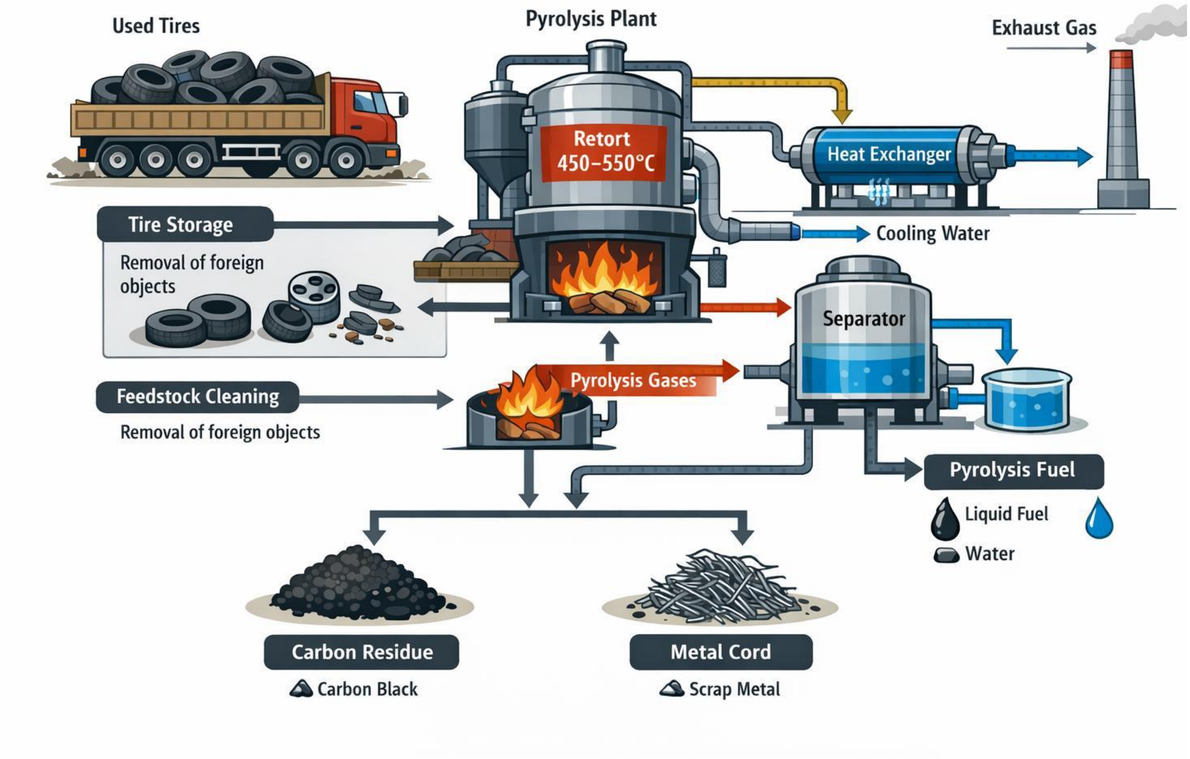

Fig. 2. Technological process diagram

loaded into a retort (crucible). In the retort, the feedstock decomposes at temperatures ranging from 450-550 °C, producing semi-finished products: gas, pyrolysis liquid, carbon-containing material, and metal cord (scrap metal). During the pyrolysis process, the retort is hermetically sealed. All rubber is heated but does not burn (except for the lower portion, which smolders). After the gases are released, approximately 800 kg of wood waste is burned through the nozzle and the pyrolysis unit, and gas is supplied with air at a controlled ratio to ensure stable combustion. Dilution with air at a specific ratio allows the operating temperature to be maintained (the temperature in the crucible must not exceed 550 °C). Pyrolysis gases (gases from tire decomposition) are removed through a feedstock heating pipe (through the crucible). The thermal decomposition of the feedstock (worn tires) occurs without air. When the feedstock is heated to operating temperature, it undergoes thermal decomposition, releasing a large amount of gas. This gas is diverted to a heat exchanger via a connecting pipe, where it condenses. The resulting hydrocarbon fractions, released during rubber heating, are directed from the retort to a heat exchanger (condensation of pyrolysis gases), through which the hydrocarbon fractions are cooled and condensed, turning into liquid pyrolysis fuel. The fractions that do not condense are sent as gas, partly to the retort and partly to the exhaust. The cooling water is recycled and reused. Liquid fuel, metal cord and carbon-containing residue are sent to a warehouse.

The liquid collects in a product settling tank. As the pyrolysis liquid accumulates, it drains into a storage tank, and the resulting water is drained from the bottom of the settling tank through installed pipes. The heat exchanger is cooled with process water (recycled water, pumped from a cooled water pool, is used). After the process is complete, the furnaces are cooled, the crucible is removed from the furnace, and after natural cooling, the crucible lid is removed, and the solid residue is discharged. The next crucible is then installed in the furnace and loaded with the processed raw material. To ensure a continuous production cycle, six crucibles are used (three working crucibles and three cooling crucibles). Gas is returned to the furnace firebox to maintain the process; if necessary, the remaining gas is released through a stack. The carbon-containing residue, after quenching and cooling, is sieved to separate the wire from the metal cord.

3.2. The technology is efficient and energy-saving. All processed products are free of highly toxic substances. The possibility of decomposition products being released into the atmosphere is eliminated. The most important positive features of the technology are:

- low energy consumption (compared to other technologies), closed cycle and cost-effectiveness, the possibility of using various types of fuel for technological needs: liquid, solid, gaseous;
- zero waste - there is no production waste that requires subsequent disposal;
- The production of highly marketable products, including energy resources. A tire recycling facility not only addresses the environmental issue of tire recycling but also enables the transformation of tire recycling into a cost-effective, highly efficient process, something no other technology currently offers;
- Environmentally safe: processed products do not contain highly toxic substances. The possibility of decomposition products escaping into the atmosphere is eliminated; the equipment is environmentally friendly, fire- and explosion-proof.

Using discarded tire waste as a raw material is important from both an economic and environmental perspective. Tires are valuable secondary raw materials, containing 65-70% rubber, 15-25% carbon black, and 10-15% metal cord. In today's increasingly stringent waste management regulations, pyrolysis is the most

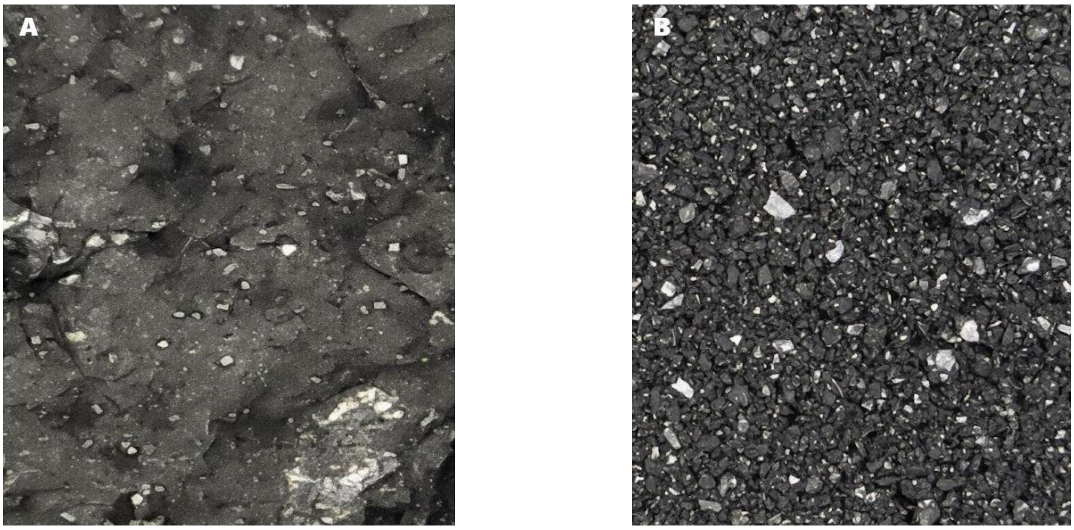

Fig. 3. Microscopic general appearance of carbon-containing material after pyrolysis.

promising and environmentally friendly method for recycling tires, yielding valuable chemical products. Among the pyrolysis products, the most valuable is carbon-containing material. The application of carbon black depends on a range of physical and chemical properties. However, most processors specify minimum quantities in their technical documentation, including ash content, bulk density, moisture content, sulfur content, and pH.

The Navoi Mining and Metallurgical Plant's central mine department operates a pyrolysis unit with a capacity of one ton per day. Therefore, to identify and expand potential applications for carbon-containing material, a detailed study of its physicochemical properties is necessary. Thermal-oxidative pyrolysis produces a carbon-containing material that is a relatively brittle, lumpy, black substance with a grayish tint and an unpleasant odor (Fig. 3a), some of which contain metallic inclusions (Fig. 3b).

Before use, the carbon black was crushed using a BB 600 laboratory jaw crusher. Analysis of the study results shows (Fig. 3) that the particles of carbon-containing material have different sizes. Among the particles of carbon-containing material, those greater than 0.045 mm and less than 0.25 mm (> 0. 045 < 0.25), i.e. 0.062 mm, account for 62.0% of the total particle content. Particles with a size from 0.062 mm to 0.5 mm, i.e. particles of 0.25 mm, account for 24.0% by weight. Also, particles of 0.5 mm in size account for about 10% by weight. The use of carbon-containing material containing crushed metal inclusions as a filler in rubber products negatively affects the quality of rubber products. Magnetic separation is used to extract these metal inclusions. After crushing, the carbon-containing material is a dispersed dark black powder.

Ground carbon-containing material can be recommended as a filler for polymers and polymeric materials. Carbon black is primarily used as a structural filler for polyethylene, polypropylene, polyvinyl chloride, and other polyolefins. The addition

Table 4. Physicochemical characteristics of the carbon-containing material before crushing (CM -1) and crushed (CM -2)

Characteristics	pH, g/cm^3	pH	A^d , %	W^a , %
CM -1	0.32 4 ± 0.02	6, 4	2 1 ,6 6 ± 0.44	0.2 3 ± 0.05
CM -2	0.40 7 ± 0.02	6, 4 -5, 5	2 3 ,7 1 ± 0.44	0.40 ± 0.05

of carbon black improves the durability of polymeric materials and increases their resistance to light aging. The physicochemical characteristics of the carbon-containing material before grinding (CM -1) and after grinding (CM -2) are presented in Table 4.

Analysis of the research results shows that reducing the particle size of carbon-containing material leads to an increase in bulk density, acidity, moisture content and has virtually no effect on ash content.

Conclusions

The results of this study demonstrate that thermal-oxidative pyrolysis of waste tires at 550 °C is an effective approach for converting secondary raw materials into valuable products. The process yields a gas fraction with a high content of combustible components, a liquid fraction with potential for further processing into fuel and chemical feedstocks, and a solid carbon-containing residue suitable for use as a filler in polymer materials. Thermal analysis confirmed that the degradation of tire rubber proceeds in two main stages, corresponding to devulcanization and subsequent decomposition of the carbonaceous structure. This behavior explains the formation of both volatile products and a stable solid residue.

The obtained findings indicate that waste tire pyrolysis can be considered not only as an environmentally sound disposal method, but also as a promising route for resource recovery and material reuse, particularly in the production of functional carbon materials and bitumen-modifying additives.

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