

TECHNICAL SCIENCES

THE TECHNOLOGY FOR EFFICIENT USE OF WASTE TYRES

Shahpalangova B.

lector, Azerbaijan State Oil and Industrial University, Baku, Azerbaijan
<https://orcid.org/0009-0009-4860-6195>

Huseynova L.

lector, Azerbaijan State Oil and Industrial University, Baku, Azerbaijan,
<https://orcid.org/0009-0005-2994-6136>

Huseynova A.

lector, Azerbaijan State Oil and Industrial University, Baku, Azerbaijan,
<https://orcid.org/009-008-3460-8141>
<https://doi.org/10.5281/zenodo.14008135>

Abstract

In recent decades, automobile transportation has become a significant factor affecting the ecology of cities. The increase in technogenic load on the environment occurs not only due to exhaust gases but also because of the large volumes of waste tires produced. It is known that old and worn-out tires, tubes, and other rubber products are classified as waste of the IV hazard category. In global practice, there are various methods to handle such waste, including incineration, regeneration, grinding, recovery, thermal liquefaction, tire use without preliminary preparation, and pyrolysis. End-of-life tires contain valuable recyclable raw materials, comprising 65-70% rubber, 15-25% technical carbon, and 10-15% metal, so their effective recycling can solve ecological problems and ensure high profitability for recyclers. Pyrolysis is a common chemical method for recycling used and end-of-life tires, generating solid carbon residue, liquid, and gaseous phases. From an ecological perspective, pyrolysis offers undeniable advantages as it allows the subsequent reuse of valuable hydrocarbon raw materials. However, the parameters of pyrolysis regimes for used automotive tires and the quality and quantity of the recycled products obtained have not been sufficiently studied to date. Therefore, comprehensive research is essential to develop environmentally safe technologies for processing old and discarded tires using promising pyrolysis methods and to create methods for obtaining high-quality recycled raw materials with enhanced ecological safety.

Keywords: waste tyres, wheels, crumb rubber, recycled raw materials, pyrolysis, destruction, utilization, recycling, thermographic analysis, mathematical modeling, pyrolysis resin, carbon residue.

Introduction

Automobile tires produced in the industry become waste after a certain period—they have a shorter lifespan compared to most rubber products, typically lasting only 3-5 years. However, even after this period, tires retain sufficient technological characteristics for recycling and can be considered as secondary raw materials. The non-recovery of natural burning fossil fuels necessitates the use of secondary fuel resources, especially those with technological potential such as rubber.

Countries with high levels of automobile usage face significant problems with the collection of used tires. In the U.S., the annual amount of used tires is 16-18 million tons, and this number is approaching 2 billion. In the UK, the volume of used tires is 400,000 tons, in Japan it is 600-750,000 tons, in Germany it is 460-510,000 tons, and in Italy it is approximately 250,000 tons. In CIS countries, more than 250-300,000 tons of tires are produced each year, with over 50 million tons of used tires accumulated. The level of recycling and use of outdated tires varies significantly abroad: in Japan, it reaches 87%, while in the U.S. and most Western European countries it is 20-30%, and in Germany, it reaches 50%.

Worn-out tires are generated and accumulated in large and small vehicle fleets, tire service and auto repair facilities, and in the private sector. Most industrialized countries have programs and practices to support the collection and recycling of waste tires. However,

methods such as evacuation, landfilling, and incineration aimed at reducing their harmful environmental impact are considered less effective.

Currently, a large number of used automobile tires are generated worldwide each year, with only 15% of their total volume being recycled and used as secondary products. The remaining tires are stored on the premises of businesses and organizations due to the lack of acceptable processes and technologies. Collecting and storing used automobile tires is virtually impossible due to the large land area required. These types of waste also pose a significant potential environmental threat and are effectively considered a "ticking time bomb." Under the influence of natural climatic factors and rodents, tires are slowly broken down into small particles that disintegrate in the natural environment and are carried over long distances by the wind.

Places where old automobile tires are stored become areas where a large number of different types of living organisms (rodents, birds, microorganisms, etc.) congregate. Therefore, it is necessary to develop effective methods for recycling used tires that address both their recycling and environmental protection. Reducing environmental pollution through the recycling and utilization of old tires is thus a crucial direction for mitigating environmental damage.

Resin formation by heating organic carbonaceous raw materials in the absence of air - pyrolysis process

has been known for a long time, but pyrolysis resin began to attract independent interest much later as a valuable hydrocarbon raw material. It's also known that resin is obtained not only by coal coking process, but also by pyrolysis of any hydrocarbon raw material [1-3]. End-of-life tyre rubber, as a valuable hydrocarbon polymer raw material, has attracted great attention to find ways for recycling. Currently, there are a series of various methods for recycling of waste tyres, but most of the studies are focused specifically on its pyrolysis. There are also many pyrolysis schemes with advantages and disadvantages, so the topic is being studied up to now. Priority methods for recycling organic waste of a wide range of technological solutions offered by local and foreign specialists are the technologies providing:

- high environmental safety of the proposed technological process;
- recycling of waste at the place of direct collection;
- a maximum low energy capacity of utilization process;
- waste-free technological process;
- obtaining final products with commodity value.

The pyrolysis method meets all of the requirements [4-5]. Although periodicals report new researches based on this method every year, there is still a need to continue the studies in this area. Because the main missing aspect of these studies is the impossibility of predicting the quality and quantity of gas, liquid and solid pyrolysis products, that is the result of the lack of in-depth laboratory studies of the process.

The study deals with the comprehensive study of the pyrolysis process of rubber waste under laboratory conditions that allow to find the main arguments for controlling the reaction and obtaining predictable results. Therefore, a laboratory reactor with a cylindrical tube was prepared 50 mm in diameter and 130 mm in height, installed vertically in an electric furnace. Oxygen-free pyrolysis process of tyres and crumb rubbers was carried out on this laboratory facility. Inert gas argon was used for blowing oxygen from the reactor. The resulting pyrolysis gases were sent from the reactor to the cooler and condensate tank. The temperature was controlled by a chromel-alumel thermocouple. Argon consumption was measured by a rheometer. At first, the reactor was heated to process temperature, then argon was released from it for a few minutes. After stopping the supply of argon, the reactor was supplied by water vapor heated to 500...600°C. After adding scrap tyres to the reactor, the temperature was decreased to 380°C, but after 3...10 min, the raw material was heated to the optimum mode temperature and the temperature was kept constant until the end of the pyrolysis. At the end of the reaction, the supply of water vapor was stopped and carbon residue was cooled with argon before discharge. Argon blowing was used to eliminate the effect of oxygen on the pyrolysis process. A steam generator was an additional element of the pyrolysis process and included in the laboratory setup to maintain the reaction temperature and save expensive argon. In addition, water vapor was a weak oxidizing agent, and gasification

of the reaction mixture with its help caused erosion of the carbon surface - although it was a weak oxidizing agent, it had a very active effect. It significantly caused an increase in specific surface area and adsorption capacity of the solid carbon residue - the future sorbent.

Thus, under optimal conditions, it's possible to use partially activated carbon residue from the pyrolysis reactor as a sorbent without further processing. Steam treatment also helps to remove excess ash content from residue caused by inorganic additives in rubber compounds [6-12]. The process temperature, occurrence time and reaction surface are the main parameters of the heterogeneous reactions that the car tyre pyrolysis process belongs. Regarding the latter, it's known that the larger the contact surface of the reagents, the higher the reaction rate [139]. Therefore, the study is carried out with crushed raw materials, the use of which makes possible to determine the temperature effect and reaction time on the yield of the main products. The fraction of scrap tyres with dimensions of 1...3 mm was used for the experiment. The literature proves a different temperature range (from 380°C to 650°C) for conducting low-temperature pyrolysis process of tyre rubber [88, 140].

None of the found sources justified selection of the process temperature, but rubber is a high molecular organic substance consisting of certain monomer units - residues of organic molecules. They are connected to each other by chemical bonds with a certain energy. These bonds are broken when exposed to heat, and the higher the binding energy, the higher the temperature required to break it, since depolymerization process is endothermic [13]. Tyre rubber has a complex multi-component composition, the most rational method for determining the optimal temperature range for the endothermic reaction of its decomposition is the thermographic research method. This method was successfully used in the study of the pyrolysis process of gray coal and carbonaceous clays [14-15]. The temperature range for tyre decomposition was determined using thermogravimetry in an air-free crucible. Thermal decomposition starts at 250-280°C and part of the sample mass is lost. The maximum mass loss occurs in the range of 380...410°C and indicates the maximum destruction of the substance. The process is completed at a temperature of 535±10°C, it's similar to the process of thermal decomposition of gray coal waste and carbonaceous clays [16].

Temperature range was selected for the reaction in laboratory studies on the basis of thermogravimetric data, where the effect of temperature on the yield of pyrolysis products was studied at constant values of the other two reaction parameters (reaction time and state of the raw material, i.e., its size). Three temperature options were used: 380, 460 and 535°C. Due to the very similar peak values of 380 and 400°C, as well as the simplicity of temperature control in the apparatus, 450°C was chosen instead of 400°C in the thermogram. The state of the raw material was maintained stable, and 100 g of crumb rubber sample with a fraction of 1-3 mm was used in the reaction. The reaction time was freely chosen as 1 hour. The yield of products at three different pyrolysis temperatures is shown in Table 1.

Table 1.

The yield of crumb rubber pyrolysis products at various temperatures

Pyrolysis products, % mas.	Temperature, °C		
	380	460	535
Gas (η_Q)	4.8-5.0	5.4-5.9	7.6-7.8
Resin + water (η_M)	30.1- 32.3	48.9-50.1	58.3-60.1
Solid residue (η_B)	60.1-61.0	39.2-40.0	31-31.6
Loss	5-1.7	6.5-4.0	3.1-0.5

Three types of products are obtained in the pyrolysis process of tyre rubber: gas, pyrolysis liquid (resin) and solid carbonaceous residue. The data in Table 3.1 reveal that an increase in the process temperature causes a significant increase in the yield of liquid products, while the yield of solid residues decreases sharply. At this time, a certain increase, although not a significantly was observed in the yield of gaseous products. Two more temperature options - 600 and 640°C were studied despite thermogravimetric analysis data. However, the experiment allowed determining that the subsequent increase in temperature to 600-650°C has a very slight effect on the yield of pyrolysis products.

It can be concluded that thermal decomposition of tyre rubber practically ends at 520-535°C, and pyrolysis occurs at different temperatures, but with different proportions of reaction products - with increasing temperature, the yield of liquid products increases, and 535±10°C takes the highest value. At this temperature limit, liquid semi-products have maximum yield. It's the most convenient way to obtain recycled raw materials during the further production of high-quality

engine or furnace fuel.

The process thermogram represents the completion of the raw materials destruction, and this gives reason to consider that the pyrolysis temperature of tyre rubber is optimal at 535±10°C. This value is used by keeping unchanged the main properties of the pyrolysis process - the condition of the raw materials and the reaction time.

The use of raw materials with a high degree of dispersion causes occurrence of a heterogeneous process on a highly developed heat exchange surface. As a result, reaction time and energy consumption for heating the heat carrier are reduced. For the purpose of determining the time required for heating raw materials of various degrees, the problem of non-stationary heat exchange was solved by two-way heat transfer to the surface of tyre samples with a thickness of 10 to 320 mm (Table 2). Three additional temperature conditions - 600, 700 and 800°C were used simultaneously with the determined optimal temperature - 535±10°C.

Table 2.

The time required to reach the heating temperature of scrap tyres depending on its thickness

Scrap tyre thicknesses, mm	Heat temperature, °C			
	535	600	700	800
	Heating time for rubber, min			
10	2	2	2	2
20	5	5	5	5
40	18	17	16	15
80	65	57	54	51
160	245	235	228	204
320	921	912	905	895

Experimental evidence revealed that the tyre heating time varies from 2 minutes to 15 and a half hours. Moreover, it depends primarily on the scrap thickness and not on the final heating temperature. Dependence of the heating time of scrap tyres on its thickness is

clearly shown in Fig. 1. All four curves practically overlap, thus confirming the thermographic analysis data.

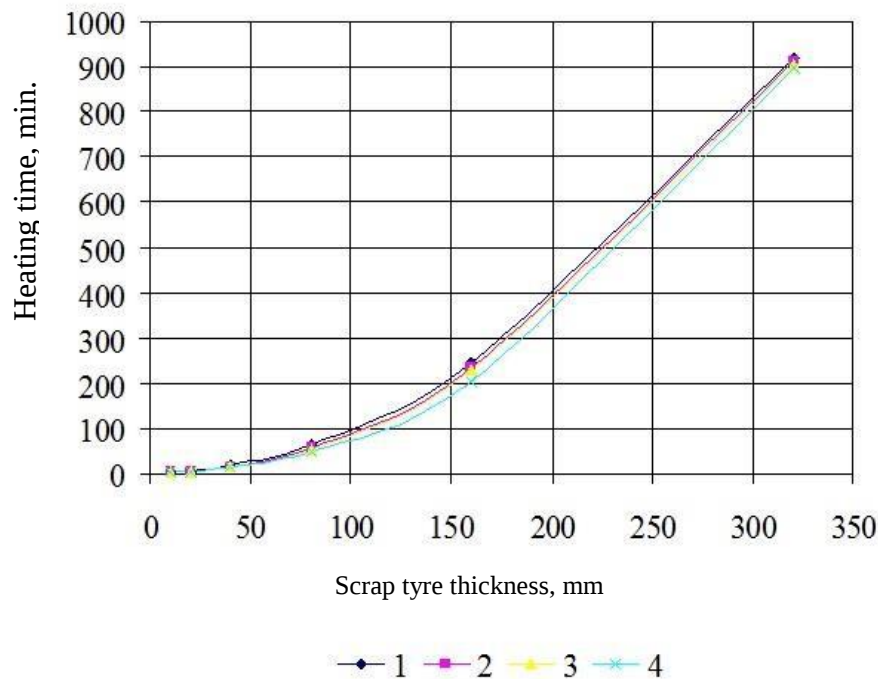


Fig. 1. Dependence of the heating temperature of scrap tyres on the thickness
1 – 535 °C; 2 – 600 °C; 3 – 700 °C; 4 – 800 °C.

Over time, the temperature changes unevenly along the central axis of the plate. The greater the temperature difference between the surface and the plate's central axis, the higher the rate of temperature rise. In order to determine which parameter - the thickness of the crumb rubber or its specific surface area - plays a more important role for heating different grades of

shredded raw materials, spherical samples of raw materials with the appropriate radius were prepared and the specific surface area was calculated for each of them. The dependence of the time required to reach the heating temperature of the central axis of the rubber plate on its specific surface is shown in Fig. 2.

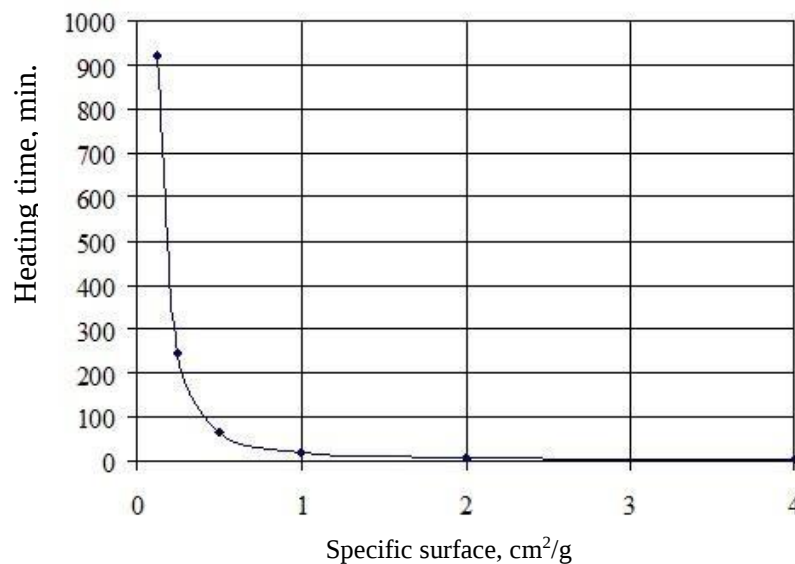


Fig. 2. Dependence of time required for obtaining the heating temperature of crumb rubber on the specific surface

Statistical processing of experimental data was carried out for determining the main parameter of the dependence of the reaction time on the degree of material crushing. The analysis of obtained polynomial

curves (Fig. 3; Fig. 4.) shows that the dependence shown in Fig. 4. has no physical meaning.

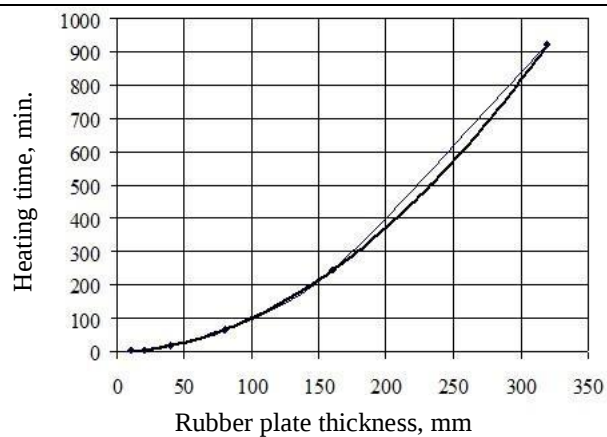


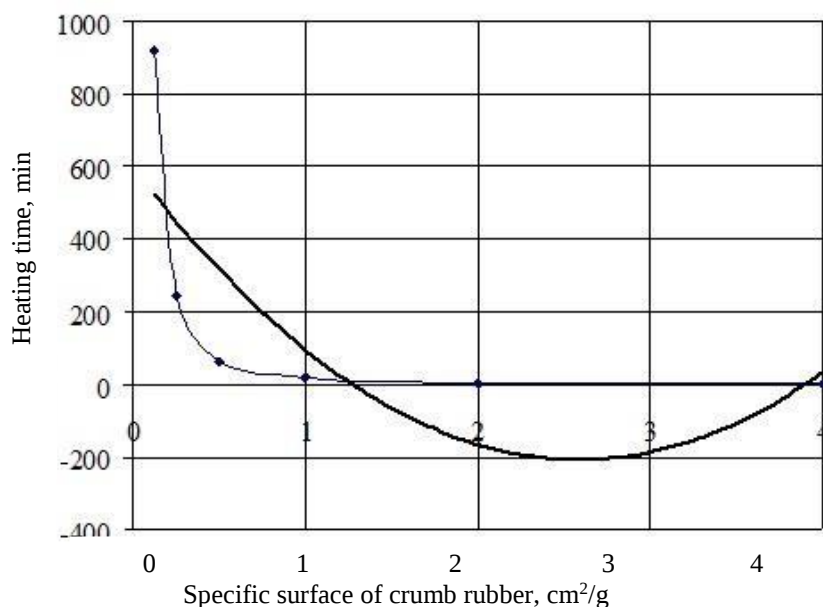
Fig. 3. Dependence of crumb rubber heating time on the thickness

$$(y = 0.0084 \cdot x^2 + 0.1838 \cdot x - 2.0229).$$

Thus, summarization of experimental studies using the method of mathematical modeling allows drawing the following conclusions:

1) the main indicator on which the heating time of the material depends is not its specific surface area, but the thickness of the raw material sample;

2) the time required for the material heating and simultaneously the pyrolysis time practically does not depend on the heating temperature in the temperature range after 535-550°C. The determined parameters do not give an idea about the dependence of the yield of pyrolysis products on the reaction time and the degree of raw materials crushing.



Destruction process of rubber samples with a thickness of 80 mm and 160 mm was carried out at a temperature of $535 \pm 10^\circ\text{C}$ in order to solve the problem. In the first and second cases, the duration of the process was selected according to the previously determined value (Tab. 2). The results of the experiment are set into Tab. 4, and the corresponding graphical dependences are shown in Fig.5.

Fig. 4. Dependence of crumb rubber heating time on the specific surface ($y = 120.31 \cdot x^2 + 621.72 \cdot x + 597.18$).

Table 4.

Dependence of the yield of pyrolysis products (η_i) on the duration of the destruction process and dimensions (S_i) of raw materials

Time, τ , min.	Product yield, % mas.		
	Gas, (η_g)	Liquid, (η_m)	Solid residue, (η_{bq})
<i>For the scraps with the thickness $S = 80$ mm</i>			
15	4.6	28.1	66.2
30	6.4	36.1	55.4
60	7.6	58.2	31.4
90	8.1	60.0	30.4
120	8.9	60.3	30.1
<i>For the scraps with the thickness $S = 160$ mm</i>			
60	4.2	27.9	66.9
120	6.0	35.5	57.6
240	7.8	59.8	32.1
480	8.0	61.1	30.2

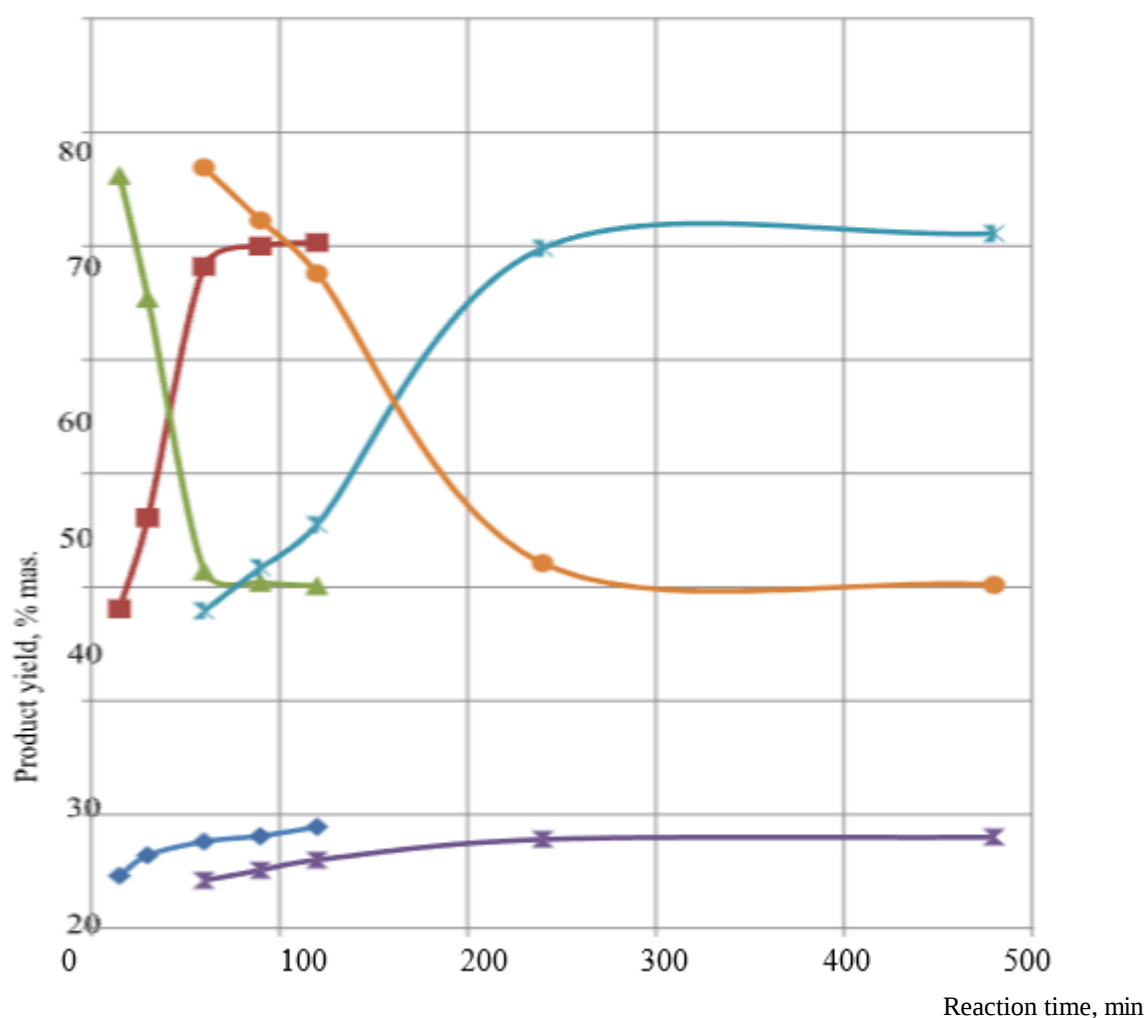


Fig. 5. Dependence of scrap tyre pyrolysis products on the dimensions and reaction time

- ◆ Gas ($S=80$ mm) ■ Liquid ($S=80$ mm)
- ▲ Solid residue ($S=80$ mm) ✕ Gas ($S=160$ mm)
- ✕ Liquid ($S=160$ mm) ● Solid residue ($S=160$ mm)

The following conclusions can be drawn on the basis of a set of experimental data, including the pyrolysis of crumb rubbers:

1) increase in the reaction time causes a corresponding increase in the yield of the vapor-gas mixture and a decrease in the yield of the solid residue, and only after reaching the previously determined time (Tab. 3), the yield of the products practically does not change that proves a full completion of the tyre destruction process during the reaction;

2) the change in the size of the tyre samples

doesn't effect on the yield of recycled products, it only causes an increase in the process time due to the increase in the size of the particles.

Increasing interest in liquid pyrolysis products as an alternative to fuel provides conduction of a detailed study of experimental data for analyzing the yield of liquid products at different heating temperatures (Tab. 5). The yield of liquid pyrolysis products at different temperatures was analyzed and the dependence of liquid pyrolysis products on the destruction temperature of tyre rubber was obtained (Fig. 6).

Table 5.

Analysis of yield of liquid products of pyrolysis process of crumb rubber at different temperatures

$T, ^\circ\text{C}$	Initial destruction temperature, $T_b, ^\circ\text{C}$	$T - T_b, ^\circ\text{C}$	$z_m, \% \text{ mas.}$	$\frac{1}{z}$	$\frac{1}{T - T_b} \cdot 10^3$
375	$\ln\left(\frac{1}{z_m}\right)$	125	0.312	3.23	8
450		200	0.459	2.18	5
535		275	0.592	1.7	3.64

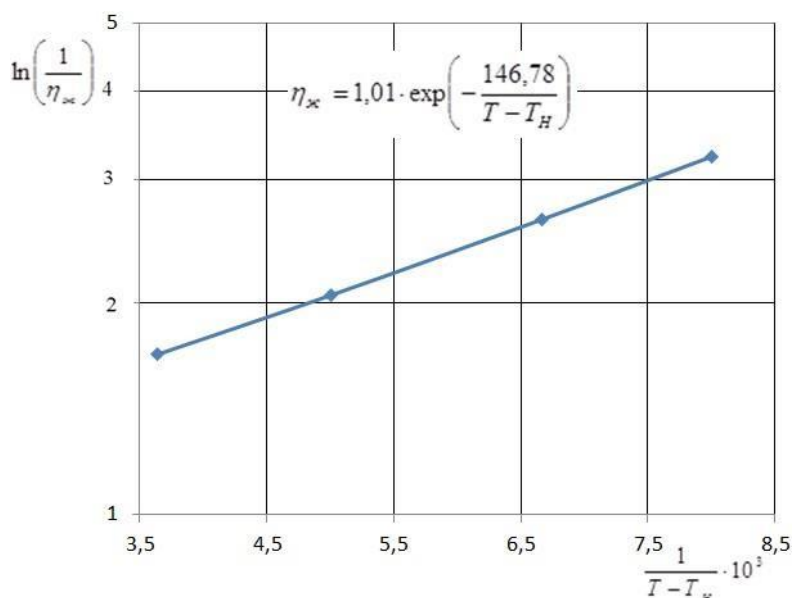


Fig. 6. The temperature of the rubber destruction process ($\frac{1}{T - T_b} \cdot 10^3$) effect on yield of liquid pyrolysis products ($1/\eta_m$)

Mathematical form of the effect of the destruction temperature of tyre rubber on the yield of liquid pyrolysis products is as follows:

$$\eta_m = 1.01 \cdot \exp\left[-\frac{146.78}{T - T_b}\right] \quad (1)$$

where η_m is the yield of liquid pyrolysis products (% mas.);

T – decomposition temperature ($^\circ\text{C}$);

T_b – the starting temperature of intensive destruction ($T_b = 250^\circ\text{C}$).

(3.1.) dependence was obtained on the basis of the use of the process model characterizing the temperature dependence of the reaction rate:

$$\ln \frac{1}{\eta_m} = a \cdot \left[\frac{1}{T - T_b} \right] + b \quad (2)$$

here $a \cdot \left[\frac{1}{T - T_b} \right]$ – polynomial characterizing the activation energy of the process; b – is a pre-exponential polynomial.

The yield of liquid pyrolysis products for samples with a thickness of $S = 80 \text{ mm}$ and $S = 160 \text{ mm}$ the effect of the duration of the tyre destruction process was studied on the basis of the analysis of the experimental data presented in Table 4, using the progressive model of "S-shaped curves" to show the mechanism of complex technical systems and processes (Fig. 7.). The mathematical formula of the dependence of the yield of liquid pyrolysis products of tyre rubber on the destruction time for two samples (thickness $S = 80 \text{ mm}$ and $S = 160 \text{ mm}$),:

$$\eta_{m80} = \frac{\eta_{m80}^{max}}{1 + \frac{2,994}{e^{7,507 \cdot \tau}}} \quad (3)$$

$$\eta_{m160} = \frac{\eta_{m160}^{max}}{1 + \frac{4,52}{e^{10,675 \cdot \tau}}} \quad (4)$$

here η_{m80}^{max} and η_{m160}^{max} maximum yields of liquid pyrolysis products for samples with thicknesses $S=80$ mm and $S=160$ mm, respectively; τ is the destruction time, min.

If the Damkeler criterion is taken into account, the dependencies (3) and (4) are as following:

$$D_{a80} = 0,334 \exp[-7,507 \tau] \quad (5)$$

$$D_{a160} = 0,221 \exp[-10,675 \tau] \quad (6)$$

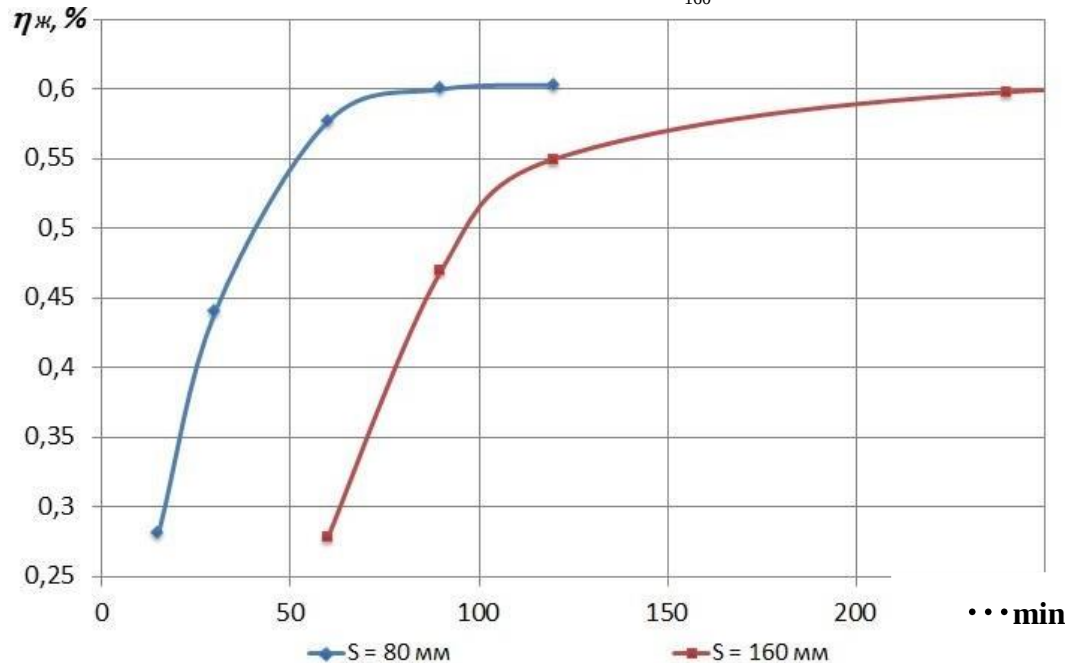


Fig. 7. Effect of tyre destruction time (τ , min.) on the yield of pyrolysis liquid products (η_m , % wt.) for samples with thicknesses $S = 80$ mm and $S = 160$ mm

Table 6.

Experimental evidence of dependence of liquid pyrolysis products yields (η_m) on destruction time (τ ,) for the samples with thicknesses $S=80$ mm and $S=160$ mm

T , min	τ , min	$S = 80$ mm			$S = 160$ mm		
		$\ln\left(\frac{\eta_{max}}{\eta_i} - 1\right)$	$\eta = \frac{\eta_{max}}{\eta_i} - 1$	% mas.	$\ln\left(\frac{\eta_{max}}{\eta_i} - 1\right)$	$\eta = \frac{\eta_{max}}{\eta_i} - 1$	% mas.
15	0.125	0.137	1.146	0.281	0.174	1.19	0.279
30	0.25	-0.994	0.37	0.44	-0.64	0.528	0.4
60	0.5	-3.1	0.045	0.577	-2.2	0.111	0.55
90	0.75	-5.299	0.005	0.6	-3.829	0.022	0.598
120	1	-	-	0.603	-	-	0.611

The established regularities allow to argue that pyrolysis process is a complex reaction, and the yield of pyrolysis products depends on many factors, mainly the temperature, size of the raw material samples, and the reaction time. Change in the degree of crushing of raw

materials doesn't cause a change in the yield of products, only the reaction completion time increases. Optimal pyrolysis temperature, corresponding to the maximum yield of liquid products and complete destruction of tyre rubber, is $535 \pm 10^\circ\text{C}$.

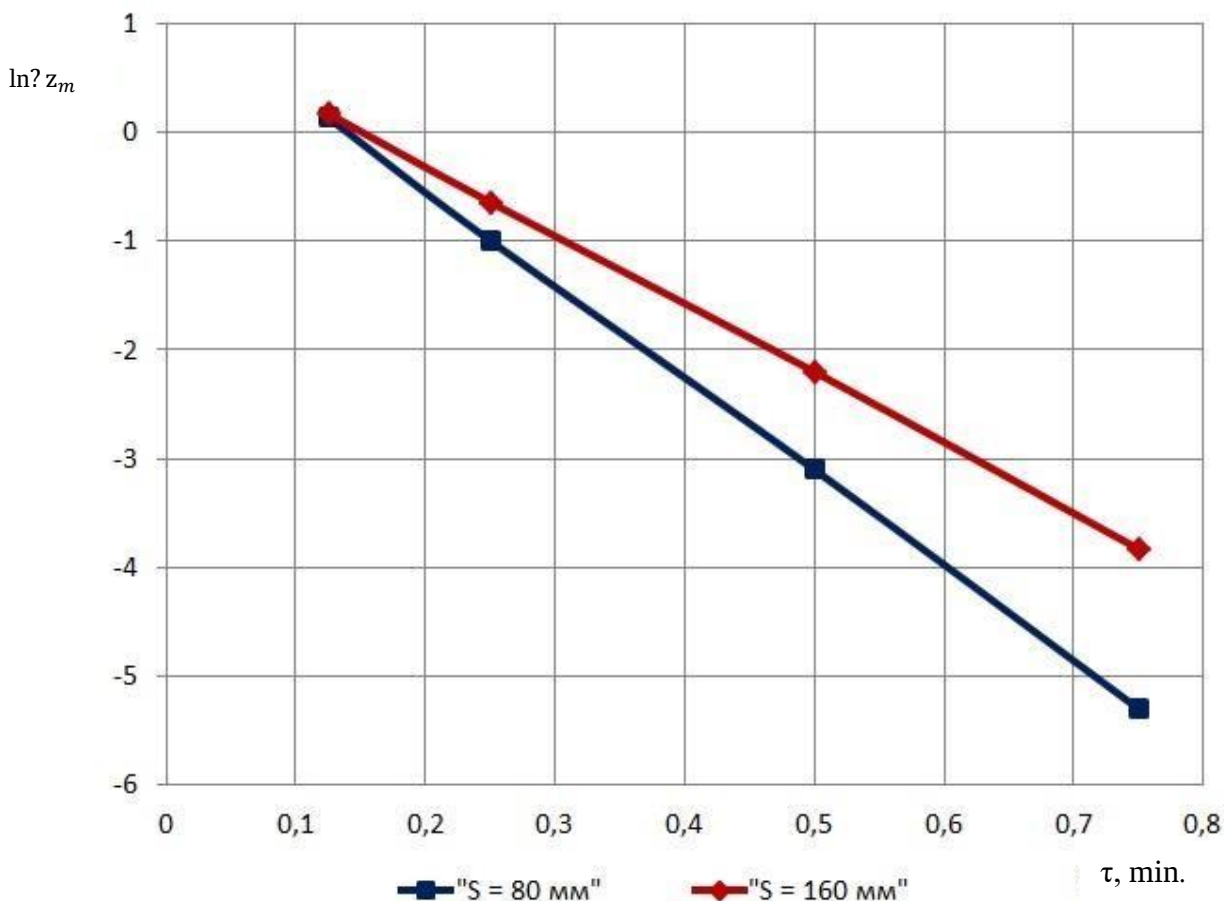


Fig. 8. Time dependence of pyrolysis liquid product yield ($\ln z_m$, % mas.) tyre destruction (τ^1 , min) for the samples with thicknesses $S = 80$ mm and $S = 160$ mm

Analytical dependences of the yield of liquid pyrolysis products on tyre destruction temperature and time of destruction of liquid pyrolysis products make it possible to determine the important parameters of the technology for recycling rubber technical products and ensure the creation of prerequisites for a fundamental approach to the design of eco-friendly pyrolysis units for recycling waste car tyres.

Conclusions

1. The environmental safety, feasibility and expediency of using tyre pyrolysis technology was substantiated as an efficient method for recycling large-tonnage rubber waste containing valuable hydrocarbon raw materials;
2. The optimal parameters of tyre pyrolysis process were found experimentally and confirmed by mathematical modeling;
3. Liquid semi-products had maximum yield at $535 \pm 10^\circ\text{C}$ - the optimal temperature of tyre destruction process. It's very convenient for obtaining recycled raw materials for further production of high-quality engine or furnace fuels.
4. The dependence of liquid pyrolysis product yield on decomposition temperature of tyre and destruction time was determined;
5. Physico-chemical properties and chemical composition of pyrolysis products were determined.
6. Analytical dependences determine the important parameters of the technology for recycling of

rubber technical products allowing a reasonable approach to the design of eco-friendly devices for the pyrolysis of waste car tyres.

7. The yield of pyrolysis products and the composition don't depend on the degrees of crushing of raw materials.

8. Chemical composition and physico-chemical properties of pyrolysis products of tyres - gas, liquid and solid substances were determined by independent methods.

9. Expediency of pyrolysis method was justified for recycling of waste tyres.

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