



Features of the pyrolysis process of waste batteries using carbon black as an additive in the construction industry

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Abstract. The paper discusses the technology for recycling used lithium-ion batteries. At the same time, one of the important components in the technology for processing such waste is the recycling of anode material with the extraction of graphite or carbon black, which can be used in the production of fire bricks. It has been shown that materials and compounds contained in lithium-ion batteries are sources of hazardous waste of the second hazard class. At the same time spent accumulators are a source of valuable secondary material resources and contain in their composition up to 16 % wt. % of graphite. The paper proposes to consider the process of processing anode materials of lithium-ion batteries in order to obtain graphite and carbon black from them by pyrolysis. Experimental studies were carried out on the process of decomposition of cathode and anode materials of lithium-ion batteries separately, as well as their mixture by pyrolysis. When studying the kinetics and mechanism of pyrolysis of carbon-containing materials, thermogravimetric analysis of the following materials was carried out: 1) powdered graphite grade GAK-2 (GOST 10273-79); 2) graphite released from the anode during manual disassembly of the LKIT; 3) mechanically activated powders containing cathode material LiNiMnCoO_2 . The characteristics of the pyrolysis process were assessed using thermogravimetric and differential thermogravimetric analyses. Pyrolysis characteristics demonstrate that organic substances contained in batteries can decompose at a pyrolysis temperature of 500 °C for cathode materials and 450 °C for anode materials. This subsequently leads to higher efficiency in the extraction of valuable components with shorter grinding times. It has been shown that the decomposition of a mixture of lithium-ion battery materials removes a larger amount of organic components than the pyrolysis of anode and cathode materials separately. In this case, the rate of decomposition of the mixture of materials occurs more slowly. The activation energy values for lithium-ion battery materials after the pyrolysis stage were determined. The content of components in powder obtained after the pyrolysis stage was determined using the method of atomic emission spectrometry with inductively coupled plasma.

Keywords: lithium-ion batteries, cathode, anode, graphite, pyrolysis, carbon black, activation energy, construction industry

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1. INTRODUCTION

At present, there is an increase in the use of mobile communication devices (MCDs) in the world practice. They use lithium-ion batteries (LIB) as batteries.

Compared to other batteries, lithium batteries have the highest energy density. They store almost twice as much energy as nickel-metal hydride batteries of the same size and weight.

The energy value of Li-ion batteries is mainly determined by the cathode material. Lithium-cobalt oxide (LiCoO₂) with an energy density of up to 180 Wh/kg is the most common option. With lithium-cobalt-manganese-nickel oxide (LiNiMnCoO₂) the energy density increases up to 240 Wh/kg. The advantages of Li-ion batteries are: high energy density, high operating voltage. Despite the long operating time, the life cycle of such batteries is limited [1], and this leads to an increase in hazardous waste generated (hazard class 2). As a result of their storage, the environment is contaminated with hazardous chemicals. These include cobalt (Co) and lithium (Li) compounds, electrolyte including lithium salts (LiClO₄, LiBF₄ and LiPF₆), organic solvent (propylene carbonate), binder polyvinylidene fluoride (PVDF) and graphite [2].

At the same time waste Li-ion batteries are valuable technogenic raw materials containing about 20-25% Co, 3-4% Li, 14.5% Al and Cu in the form of foil, 16% graphite, 14% polymers, 24% steel and 2.5% electrolyte.

If in the field of processing of LIB cathode materials in order to extract valuable components in the form of Co, Ni, Mn, Li and others, a large number of foreign experimental studies are now concentrated [3, 4], the anode material containing copper foil and graphite in its composition remains practically ignored. It should be noted that the technology of obtaining carbon material from graphite and its further use in the production of technical products is practically absent. At the same time, graphite and carbon black are used in the construction industry to produce fire bricks.

Graphite and carbon bricks are widely used. Graphite bricks have high heat resistance and maximum temperature. It has high thermal conductivity, small coefficient of linear expansion and good heat resistance. Special fine-grain graphite and anti-corrosion resins are used in the production of graphite bricks by pressing. Its thermal conductivity and electrical conductivity are reduced by about 30%. Calcined anthracite is used as the main raw material and pitch is used as a binder.

Another type of fire brick is carbon brick, in which the carbon content ranges from 60 to 80%. The raw materials for carbon bricks are mainly anthracite, coke and graphite, with resin, tar and anthracene oil as binders. It is produced by molding from fine particles with a size of 50-80 microns.

Thus graphite and carbon bricks are expensive products and are widely used in the construction, metallurgical and chemical industries [5].

It is known that carbon-containing components, for example, in carbon bricks, can be partially replaced by carbon black (pyrocarbon), which can be obtained by pyrolysis. It is known to obtain carbon black from used car tires [6]. At the same time, the technical literature does not cover the production of carbon black by pyrolysis from waste LIB.

This paper deals with the technology of battery recycling by pyrolysis method to obtain carbon-containing components from various LIB cells.

2. METHODS AND MATERIALS

The pyrolysis process uses mechanically activated powder containing cathode (LiCoO₂, LiNiMnCoO₂ or LiMnO₂) and anode (graphite) materials with copper and aluminum impurities as a starting material. In addition, the powder contains a certain amount of electrolyte and binder component.

The pyrolysis process was studied in a special laboratory pyrolyzer installed in a muffle furnace. The design of the muffle furnace for the pyrolysis process is shown in Fig. 1.



Fig. 1. Design of the muffle furnace: 1 – body; 2 – furnace muffle; 3 – door; 4 – heating elements; 5 – support plate; 6 – front control panel; 7 – thermocouple; 8 – door muffle.

The electric chamber furnace is lined from inside with muffle 2 made of mullite-silica fiber. A support plate 5 is placed inside the furnace. The furnace is heated by means of heating elements 4. A muffle 8 is installed on the door 3. The furnace chamber is placed in the casing 1. The temperature in the furnace is controlled by thermocouple 7. The current furnace temperature is displayed on the control panel 6.

The main characteristics of the furnace include: 1) furnace temperature control range from 200 to 1250 °C; 2) time of furnace output to the temperature mode; 3) power consumption - not more than 2.8 kW.

The unit can be operated with forced extraction by a fan with discharge of generated gases through a lined pipe. The electric furnace is equipped with software and can be connected to a computer with the ability to display the parameters of the electric furnace and pyrolysis modes on the screen and record them in a text file.

The electric furnace has the possibility of setting an adjustable heating rate in the range from 1 to 15 °C /min. The heating rate does not exceed the programmed value, but cannot be lower than the set value, in particular when the working volume of the chamber is loaded and, in particular, when the temperature in the electric furnace exceeds 700 °C. The heating speed “0” corresponds to the maximum possible speed.

All entered programs are stored in volatile memory, so even when the power is turned off, all information is saved.

The pyrolysis process is carried out in a corundum pyrolyzer. The pyrolyzer consists of a corundum crucible with a sealing lid, connected by means of a corundum tube to a lined chimney located in the outer part of the muffle furnace. The material to be processed is placed in a rectangular container (boat).

A number of studies have shown [7-11] that the main factors affecting the quality of product decomposition and material balance are temperature and rate of heating medium supply. Thus, slow pyrolysis (heating rate 5-80 °C /min) with a long time interval at low temperature (300-600 °C) seems preferable for carbonization of graphite (obtaining carbon black ≈ 35 % (wt.)). However, fast pyrolysis (at a heating rate of more than 200 °C/s, a temperature of 500 °C with a short time exposure) is more oriented to obtain liquid hydrocarbons (≈ 75 % (wt.)) after cooling of pyrolysis gases.

With further increase of the process speed and temperature (800 °C) of the supplied agent, the productivity of combustible gases increases, the composition of which also strongly depends on the technological modes.

In this study, pyrolysis technology is used to remove the organic liquid phase and convert graphite into powdered carbon black. Thermogravimetry-gas, chromatography-mass spectrometry (TG-GC-MS) was used to analyze the pyrolysis characteristics of organic matter in electrode materials. The

kinetics of organic pyrolysis was analyzed on the basis of the mode parameters of the process. Also on this basis, a high-resolution three-dimensional X-ray microanalyzer (3D-XRM) in combination with a scanning electron microscope (SEM) was used to reveal the enhanced release mechanism of cathode materials.

Thermogravimetric analysis of materials was used to study the kinetics and mechanism of pyrolysis of carbon-containing materials. Powdered graphite of GAK-2 grade (GOST 10273-79), graphite separated from the anode during manual disassembly of LKIT, mechanically activated powders containing cathode and anode material were used. In the experiment, the process of removing organic binder and electrolyte of different grades from battery components was investigated. Their compositions used in cathode and anode materials are given in Tables 1 and 2 [12].

Thermogravimetric analysis was carried out on the equipment of Analytical Testing Laboratory (ATL). The temperature range was varied from 20 to 800 °C at heating rates of 5, 15, 25 °C /min. Thermogravimetric test was performed in nitrogen atmosphere at a flow rate of 50 ml/min. The heating rate and temperature range used during the tests were consistent with the conditions used for slow low-temperature pyrolysis of solids during actual reaction operations [13, 14]. The mass of the sample used for thermogravimetric testing was from 10 to 15 mg. The same amount was used in the pyrolyzer experiments. In addition, during the experiment we determine: 1 – pyrolysis start temperature t_i ; 2 – process end temperature t_f ; 3 – maximum reaction speed w_{max} ; 4 – maximum temperature t_{max} ; 5 – time before decomposition begins T_e ; 6 – total thermal conversion time T_f ; 7 – time to reach the maximum reaction speed T_{max} . These characteristics are calculated using the graphical analysis method [13].

Table 1. Main products of pyrolysis of cathode materials in the gas phase at a temperature of 500 °C.

№	Pyrolysis (decomposition) products	Chemical formula	Battery components
1	Vinylidene fluoride	$C_2H_2F_2$	Organic binding agent (polyvinyliden fluoride (PVDF))
2	1,3,5-trifluorobenzene	$C_6H_3F_3$	
3	1,4-difluoro-benzene	$C_6H_4F_2$	
4	1,1,1,3,3,3-hexafluoropropane	$C_3H_2F_6$	
5	Ethylene carbonate	$C_3H_4O_3$	Electrolyte
6	Propylenecarbonate	$C_4H_6O_3$	
7	1,2,4-trifluorobenzene	$C_6H_3F_3$	Organic binding agent (PVDF)

Table 2. Main products of pyrolysis of anode materials in the gas phase at a temperature of 450 °C.

№	Pyrolysis (decomposition) products	Chemical formula	Battery components
1	Heptene-3-ol-1 (Heptene-3-yl alcohol)	$C_7H_{14}O$	Organic binding agent/electrolyte
2	1,3-vinyl ethylene	C_4H_6	Organic binding agent (butadiene-styrene rubber, (BSR))
3	Styrene	C_8H_8	
4	2,3,6,7-tetramethyl-4-octyne	$C_{12}H_{26}$	
5	Enanthic acid	$C_7H_{14}O_2$	Organic binding agent/electrolyte
6	5,5-dimethyl-2-hexene	C_8H_{16}	Organic binding agent (BSR)
7	4,4-dimethyl-1-hexene	C_8H_{16}	
8	Caprylic acid	$C_8H_{16}O_2$	Organic binding agent/electrolyte

The use of graphical dependences obtained from TG and DTG data allows to determine the stages of the pyrolysis process: 1 – by loss of external moisture and insignificant mass loss during inert heating; 2 – intensive mass loss with yield of volatile compounds during pyrolysis; 3 – inert decomposition of heavy hydrocarbons and carbonization of carbon residue. Argon, CO_2 and steam are used as inert environment in pyrolysis of various materials.

3. RESULTS AND DISCUSSION

Studies of the decomposition process of organic components, cathode and anode materials during pyrolysis were carried out in a thermogravimetric analyzer using thermogravimetric (TG) and differential thermogravimetric (DTG) analysis curves. The pyrolysis temperature was varied from 25 to 700 °C and at heating rates of 5, 15 and 25 °C/min.

The results of investigation of the pyrolysis process at different heating rates separately for cathode and anode materials by TG and DTG methods are shown in Fig. 2 a, b and Fig. 3 a, b, respectively.

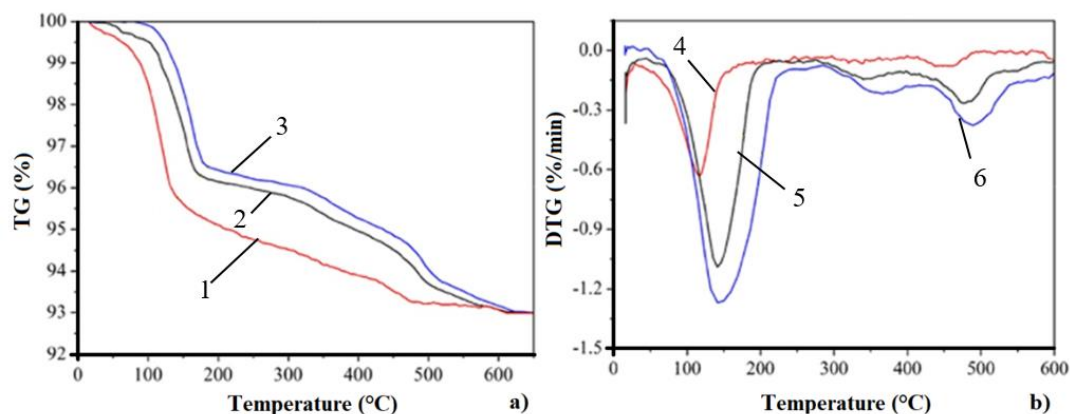


Fig. 2. Results of studies of the process of decomposition of organic substances in cathode materials using TG and DTG methods depending on temperature: a) – loss of sample mass; 1 – 5 °C/min; 2 – 15 °C/min; 3 – 25 °C/min b) – change in the rate of decomposition; 4 – 5 °C/min; 5 – 15 °C/min; 6 – 25 °C/min.

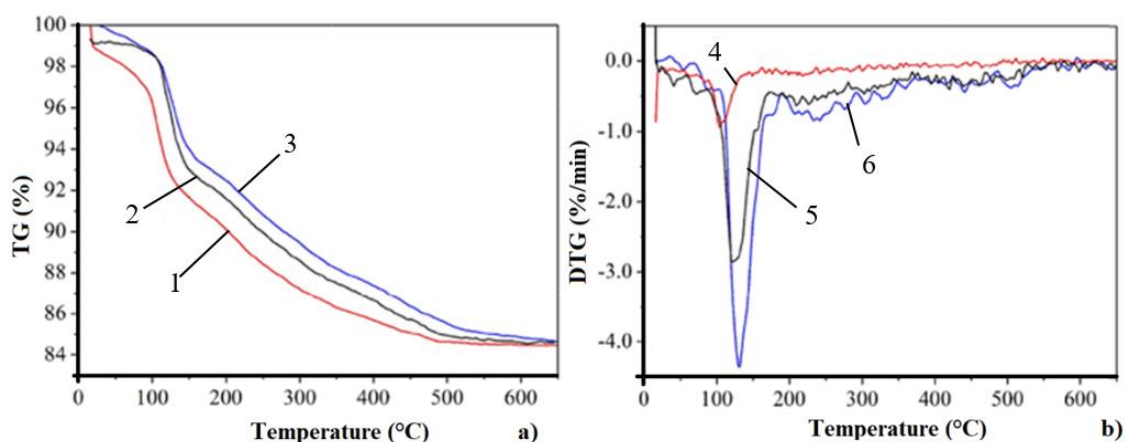


Fig. 3. Results of studies of the process of decomposition of organic substances in anode materials using TG and DTG methods depending on temperature: a) – loss of sample mass; 1 – 5 °C/min; 2 – 15 °C/min; 3 – 25 °C/min; b) – change in the rate of decomposition; 4 – 5 °C/min; 5 – 15 °C/min; 6 – 25 °C/min.

From the presented graphical dependences it is evident that the decomposition efficiency of TG-based LIB electrode materials can be described by three stages. The first stage for both cathode and anode occurs at a temperature of 120 °C. The second stage is at 250 °C. The third stage for cathode materials proceeds at 500 °C and for anode materials at 450 °C. It can be seen from the data obtained that the total mass loss of cathode materials is about 7 %, while that of anode materials is 15 %. Compared to cathode materials, anode materials have more organics. At the same time, the same products are formed as a result of pyrolysis, presented in Table 3. Products formed during decomposition of cathode and anode materials at 500 °C and 450 °C, respectively, are presented earlier in Tables 1 and 2.

Table 3. The main products of pyrolysis of LIB electrode materials at temperatures of 120 °C and 250 °C.

№	Pyrolysis products	Source
Pyrolysis products of cathode and anode materials at 120 °C		
1	Dimethyl carbonate	Electrolyte
2	Ethylene carbonate	Electrolyte
3	Propylene carbonate	Electrolyte
Pyrolysis products of cathode and anode materials at 250 °C		
4	Dimethyl peroxide	Electrolyte
5	Dimethyl carbonate	Electrolyte
6	Ethylene carbonate	Electrolyte
7	Propylene carbonate	Electrolyte
8	Methylacetic acid	Electrolyte

Thus, the study of the thermal decomposition process is carried out using a thermogravimetric analyzer, which allows to determine the temperature, time and rate of oxidation and pyrolysis.

The activation of carbon material undergoing thermal decomposition in an inert environment can be described using activation energy [14]. The activation energy of decomposition of samples is determined using the Friedman, Ozawa-Flynn-Wall (OFW) and Kissinger-Akahira-Sunose (KAS) methods. According to thermogravimetric analysis, temperatures are extracted for several heating rates, different graphs are plotted for the same degrees of conversion, and then the energy value from the degree of conversion is determined [14].

Friedman's method uses the Arrhenius equation, and for the kinetics of the thermal decomposition process, the equation is used:

$$\frac{d\alpha}{dt} = kf(a), \quad (1)$$

there: $f(a)$ – reaction mechanism function; α – conversion rate (vaporization) of organic substances at time t , given in equation (2), k – reaction rate constant.

$$\alpha = \frac{\Delta m}{\Delta m_f} = \frac{m_0 - m_t}{m_0 - m_f}, \quad (2)$$

there: m_0 – initial mass of the sample; m_t – sample mass at the moment of time t ; m_f – the final mass of the sample.

Using the Arrhenius equation in the form

$$E_0 = -RT \ln\left(\frac{k}{A}\right), \quad (3)$$

we may obtain the value of k .

In equation (3), the following notations are used: k – reaction rate constant; A – pre-exponential multiplier; $R = 8.314$ (J/mol·K) – universal gas constant; T – temperature, K; E_0 – activation energy.

Here, in pyrolysis, the activation energy determines the rate of reaction (thermal decomposition). From Eq. (3) after transformations we obtain the expression of the reaction rate constant in the form:

$$k = A \exp\left(-\frac{E}{RT}\right). \quad (4)$$

Then the basic reaction rate equation of the decomposition process in pyrolysis can be obtained:

$$\beta \frac{d\alpha}{d\tau} = Af(\alpha) \exp\left(-\frac{E}{RT}\right). \quad (5)$$

Friedman proposed to use the logarithm of the reaction rate $\frac{d\alpha}{d\tau}$ as a function of temperature at a given degree of conversion. The obtained Friedman kinetic model for the n-th order of reaction is written in the form:

$$\ln\left(\frac{\beta d\alpha}{d\tau}\right) = \ln[Af(\alpha)] - \frac{E}{RT} \quad (6)$$

In this case, the reaction rate at a constant degree of transformation x is only a function of temperature. The function $f(\alpha)$ is written in the form:

$$f(\alpha) = (1 - \alpha)^n, \quad (7)$$

there n – a numerical parameter determining the reaction order. The parameter n has a physical meaning in the range from 0 to 3. At higher values of x , this parameter allows a more accurate description of the thermogravimetric curve.

Taking this into account, equation (6) is written in the form:

$$\ln\left(\frac{\beta d\alpha}{d\tau}\right) = \ln A + n \ln(1 - \alpha) - \frac{E}{RT} \quad (8)$$

Using equation (8), the value of the slope of the linear approximation $\ln\left(\frac{\beta d\alpha}{d\tau}\right)$ versus $\frac{1000}{T} = -\frac{E}{R}$ allows us to determine the apparent activation energy with a given conversion value.

The solution of the presented equations and determination of the main kinetic parameters is carried out using the results of TG and DTG analyses. By plotting the graphical dependence of $\ln\left(\frac{\beta d\alpha}{d\tau}\right)$ on $1/T$ we obtain a straight line (Fig. 4).

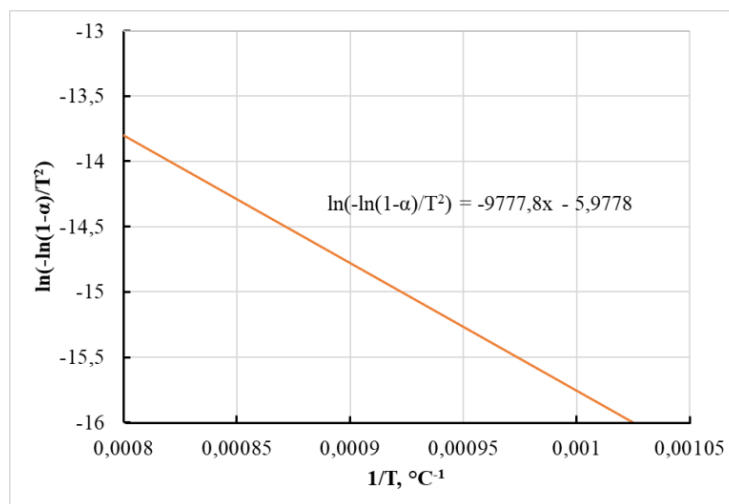


Fig. 4. Dependence of change of parameter $\ln\left(\frac{\beta d\alpha}{d\tau}\right)$ from $1/T$.

The obtained dependence is considered in the range of change of conversion degree $\alpha = 0.1-0.9$ [15] and is approximated by a straight line in the form $\ln\left(\frac{\beta d\alpha}{d\tau}\right)$ or $\ln(-\ln(1 - \alpha))T^2 = C_1 + C_2 \cdot \frac{1}{T}$.

The tangent of the angle of inclination is $m = \frac{E_a}{R}$. In this case, it is assumed $\frac{1000}{T} = -\frac{E}{R}$.

Having obtained the approximation curve, the coefficient C_2 should be multiplied by the universal gas constant R and then we obtain the activation energy:

$$E = -C_2 \cdot R \quad (9)$$

The algorithm for calculating the activation energy using thermal decomposition data of natural graphite TG and DTG (Fig. 5) is presented below.

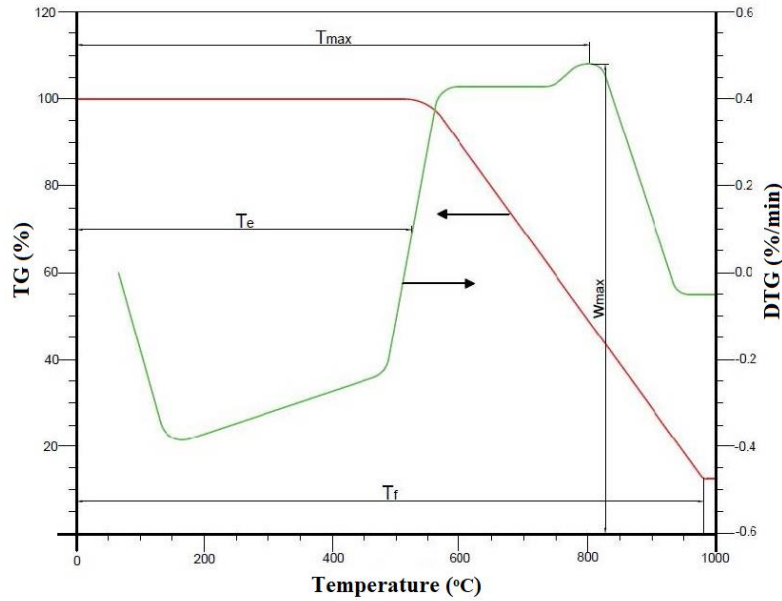


Fig. 5. Profiles of thermal decomposition of TG and DTG of natural graphite (battery GAK-2) in an inert argon environment.

Using the methodology of [14], we find the temperatures T_e (time before the beginning of decomposition); and T_f using the tangent method for TG profiles of the beginning and end of intensive oxidation. After determining the temperatures, we determine the time by the formulas:

$$T_e = \frac{t_i - t_0}{10} = \frac{550 - 45}{15} = 34 \text{ min} \quad (10)$$

$$T_f = \frac{t_f - t_i}{10} = \frac{970 - 550}{15} = 28 \text{ min} \quad (11)$$

there t_i – the temperature of the beginning of intensive oxidation; t_0 – initial temperature; t_f – temperature of the end of intensive oxidation; 15 – heating rate ($^{\circ}\text{C}/\text{min}$).

In our case, we multiply the coefficient C_2 by the universal gas constant R and obtain the activation energy:

$$E = -(-9777.8 \cdot 8.314) = 81293 \frac{\text{J}}{\text{kg}} = 81.3 \text{ kJ/kg}$$

Further, we conducted experiments on thermal decomposition of crushed material of lithium-ion batteries (LIB) containing cathode and anode material. At the same time, metal inclusions were previously removed. The results of the thermal decomposition process by TG and DTG methods are presented in Fig. 6. Using the above described methods, the activation energy was determined: $E_i = 85.4 \text{ kJ/kg}$.

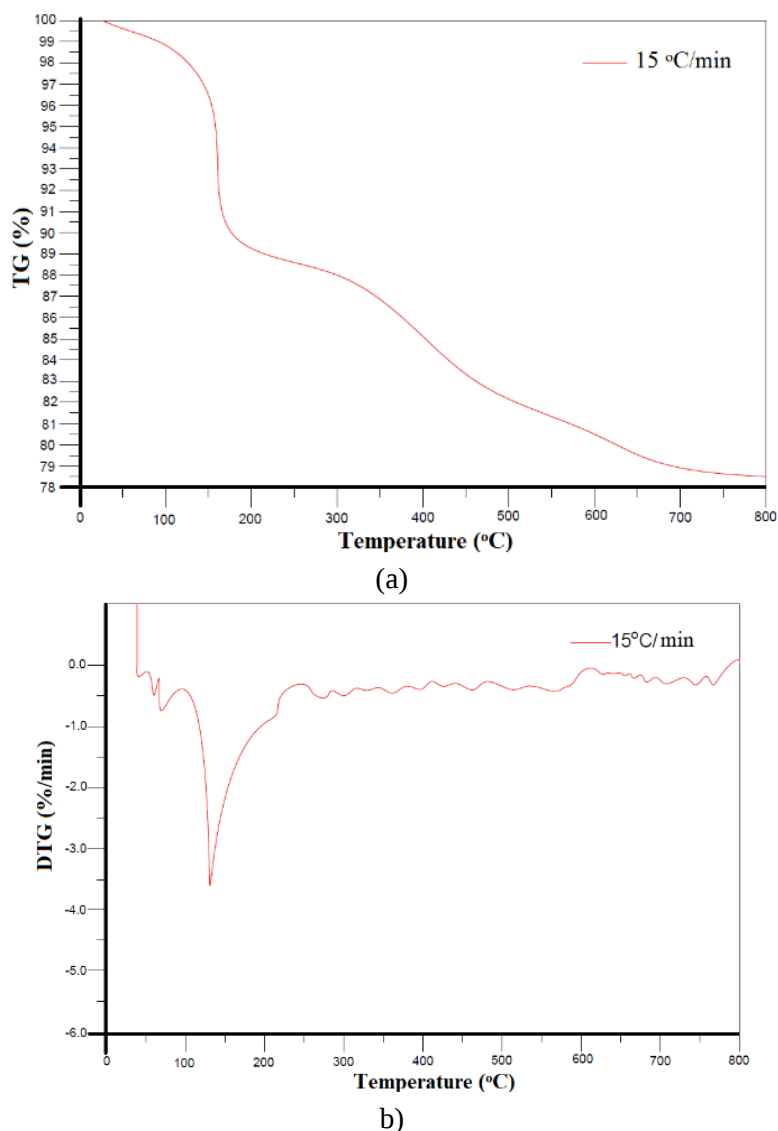


Fig. 6. Results of studies of the process of decomposition of organic substances of crushed LIB material using TG and DTG methods depending on temperature: a) – loss of sample mass; b) – change in the rate of decomposition.

As a result of investigations of the thermal decomposition process by TG and DTG methods, activation energy values were obtained for four types of materials: 1) for cathode LIB material $E_k = 38.0$ kJ/kg; 2) for anode LIB material $E_a = 62$ kJ/kg; 3) for natural graphite $E_{pg} = 81.3$ kJ/kg; 4) for crushed LIB including anode and cathode $E_i = 85.4$ kJ/kg.

4. CONCLUSIONS

1. The issues of technology of utilization of waste lithium-ion batteries were considered.
2. Methods of recycling of anode, cathode materials, natural graphite and shredded battery cells were analyzed. A description of the decomposition kinetics by TG and DTG methods using the Friedman method was proposed.
3. The activation energy values for the above materials were obtained.
4. The results of thermal studies allow recommending regime parameters for the pyrolysis process of various types of batteries.
5. It is shown that in the production of graphite and carbon bricks instead of natural graphite carbon black obtained by pyrolysis can be used.

5. ACKNOWLEDGEMENTS

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