

## DETERMINATION OF INDUCTION OXIDATION TIME OF SECONDARY POLYETHYLENE COMPOSITION

Shakhnoza Mamatova<sup>1</sup>, Matlyuba Safarova<sup>1</sup>, Guzal Davronova<sup>1</sup>,  
Guzal Raxmatova<sup>2</sup>,  Jasur Makhmayorov<sup>3</sup>,  Bakhodir Abdullayev<sup>3</sup>,  
Mingnikul Kurbanov<sup>1\*</sup>

<sup>1</sup>Karshi State University, Karshi, Uzbekistan

<sup>2</sup>Karshi Engineering-Economics Institute, Karshi, Uzbekistan

<sup>3</sup>University of Economics and Pedagogy, Karshi, Uzbekistan

**Abstract.** In this article, based on the results of the research, the oxidation time of composite materials obtained based on secondary polyethene under the influence of temperature and oxygen flow was studied. The obtained results and the properties of oxygen flow and temperature tolerance of the samples were carried out using modern analytical methods. A composite material with the required properties was obtained from a composite made of three components (secondary polyethene, sulfur and melamine). The resulting polymer composite materials can be used in the production of various consumer goods used in the construction industry and other industries.

**Keywords:** Secondary polyethene, melamine, isothermal temperature, stability, thermomechanical.

**Corresponding Author:** Mingnikul Kurbanov, Karshi State University, Karshi, Uzbekistan,  
Tel.: +998901682992, e-mail: [bahodir.abdullayev.92@mail.ru](mailto:bahodir.abdullayev.92@mail.ru)

**Received:** 4 March 2024;

**Accepted:** 17 May 2024;

**Published:** 2 August 2024.

### 1. Introduction

Currently, worldwide demand for polymer composite materials is increasing dramatically. The main reason for this is explained by the increase in the population, production enterprises and the demand for building materials and car tuning equipment. It has been shown that the combination of multi-ton polymer material modification and composite material production methods leads to improvement in the quality of raw materials and composites. The polymer composite materials obtained in this way have shown the expansion of their range and increasing fields of application (Jiang *et al.*, 2024; Pinsuwan *et al.*, 2023; Xiao *et al.*, 2022).

Many scientists have provided information about the increase in thermophysical properties of composites by studying differential scanning calorimetry (DSC) analysis in their scientific works. In some scientific works, the data on the doubling of the spontaneous combustion time compared to the original polythene when fillers are added to polythene has also been mentioned (Jin *et al.*, 2020; Celina, 2013; Zha *et al.*, 2022).

Changes in polymer properties in the recycling processes of polyvinyl chloride (PCH), polyethene (PE) and polypropylene (PP) wastes, depending on the number of

---

#### How to cite (APA):

Mamatova, S., Safarova, M., Davronova, G., Raxmatova, G., Makhmayorov, J., Abdullayev, B. & Kurbanov, M. (2024). Determination of induction oxidation time of secondary polyethylene composition. *New Materials, Compounds and Applications*, 8(2), 223-232 <https://doi.org/10.62476/nmca82223>

recycling and four to five times recycling of polymer materials to a very small amount of changes (much less compared to the primary raw materials) can lead (Xiao *et al.*, 2022; Qin *et al.*, 2024; Xie *et al.*, 2022). Polyethylene terephthalate (PT) is widely used in the chemical industry in the production of various fibers, films and composite materials. It is also reported that the amount of waste during its production and fibre processing reaches 15% and the amount during the production of films reaches 40%. It is reported that polyethylene terephthalate waste can be divided into technological and production waste and composite materials can be obtained from them. Opinions on the widespread use of the extruder method in the disposal of these polyethylene terephthalate wastes are presented (Purohit *et al.*, 2024; Abdullayev *et al.*, 2023a). It is noted that the production of composite materials based on ethylene and propylene copolymers is in the leading position (Battisti *et al.*, 2023). In some scientific works, scientific studies on the decrease in elasticity and increase in density of polymer materials by adding talc to the component have been cited (Fidanovski *et al.*, 2018; Abdullayev *et al.*, 2023b).

Many studies show that as a result of adding dispersed fillers to polyethene, it is possible to increase all thermophysical properties and reduce flammability.

## 2. Experimental part

To obtain polymer composite materials, sorted pure recycled polyethene was first crushed mechanically. The components were then introduced into the initial mixing process in a ratio of 50:1:1 (recycled PE: sulfur: melamine) for 20 minutes. After that, the composite mixture was stirred in a laboratory extruder at 60 rpm at a temperature of 115-140 °C for 0.5 hours. At a given temperature, the chains of octahedral elemental sulfur are destroyed and free valence bonds of sulfur react with the melamine molecule and the products of thermal destruction secondary polyethene. The composite mixture was passed through the extruder 2-3 times until a homogeneous mixture was formed. The resulting hot mixture was passed through a granulator and granules were prepared from it. After that, studies were carried out to determine the physical and mechanical properties of the resulting polymer composite materials.

The induction oxidation time of secondary polyethene composite samples was determined using a Differential Scanning Calorimeter ASTM D3895 device. Induction Oxidation Measurement is an accelerated thermal ageing test. The induction oxidation time of secondary polyethene composites was studied to determine their stability to temperature and oxygen flow. According to it, the sample crucible and standard crucible were placed in the device at ambient temperature. Before the heating cycle, nitrogen gas was injected into the device for 5 min. It was checked that the device (recorder) that records changes in the sample is connected to the working mode. After that, the samples were heated in the presence of oxygen flow and the programmed temperature mode starting from the ambient temperature. The isothermal temperatures of the samples were monitored at every 20 °C temperature interval. Once the given temperature was sufficient, the heating was stopped according to the programmed temperature. Then, it was kept for 3 minutes until the temperature of the sample returned to equilibrium. After the specified 3 minutes had elapsed, the nitrogen atmosphere in the device was replaced by airflow. The airflow was delivered at the same rate as the nitrogen flow was delivered. The time when the air was supplied instead of this nitrogen atmosphere was considered as time zero of the study. The given temperature was maintained for some time not less

than 2 minutes. At the end of the study, the mode of nitrogen injection was switched and the temperature of the measuring cell was cooled to room temperature.

Thermogravimetric analysis of recycled polyethene composites was carried out on a Shimadzu DTG-60 instrument. To obtain thermogravimetric analysis data for recycled polyethene composite samples, round samples with a diameter of 5 mm were prepared from each sample. Before measurement, samples were taken for 3 hours at a temperature of (23±2) °C and relative humidity (50±5) % according to DavST 12423. Then the scales of the TGA measuring device were calibrated. The system temperature was also calibrated according to the instructions. Temperature calibration is key. The thermocouple device was placed so that it did not touch the samples. The ordinate axis is given at 0. The abscissa axis was adjusted to the required temperature range of 600 °C. The gas flow from the gas cylinder was adjusted to the specified value. The sample was placed using a clamp and its initial mass was recorded. The temperature rise rate control device was set to (10±1) °C/min. The heating program was started and the temperature scan continued until the maximum test temperature was reached. The rate of decrease in the mass of the sample obtained as a result of the experiment was calculated using the following formula:

$$v_m = \Delta m / \Delta \tau,$$

where:  $\Delta m$  – weight reduction, mg;  $\Delta \tau$  – time interval, min.

The study of the IR spectra of the PCMs obtained for the study was carried out using the IR Affinity-1S device. IR spectra were recorded in the range of 4000–400  $\text{cm}^{-1}$ .

### 3. Results and discussion

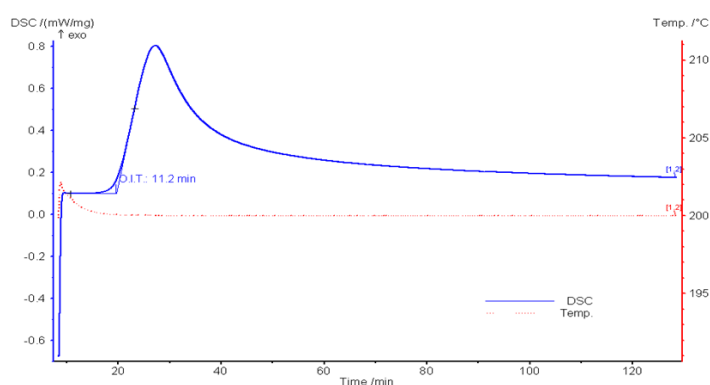
The essence of determining the induction oxidation time of secondary polyethene composition samples is to determine the time of oxidation prevention when the antioxidant stabilizer system present in the test sample is heated at a constant rate in an oxygen or air atmosphere while keeping the sample in an isothermal mode at a certain temperature. Figure 1 shows a schematic representation of differential scanning calorimetry. The time or temperature of the oxidation index is an estimate of the degree of stabilization of the material under study. A high-temperature regime leads to a reduction in the oxidation time of the material. The time or temperature of the oxidation index also depends on the large or small surface area of the material under investigation.

It should be noted that, compared to studies conducted in the presence of air oxygen, the values of the studies conducted in pure oxygen flow decrease with time or temperature.

The time or temperature of the oxidizing induction of the studied sample also depends on the specific properties of the composites added to the sample. In particular, (the DSC) analysis of a composite material consisting of polyethene and sulfur was studied in a DSC F3 differential scanning calorimeter according to the ASTM D 3895 analysis method. An 8.2 mg composite sample was placed in the calorimeter to obtain a differential scanning calorimetry analysis of the secondary polyethene and sulfur composite sample in Figure 2 below. The device automatically opened the flow of nitrogen gas to create an inert environment and at the same time, the heating process of the sample started in the device. The initial temperature of the analysis was set to 25 °C and the sample heating rate was set to 20 °C/minute. The maximum test temperature is 200 °C and its duration is 2 hours. The emergency ignition temperature was set at 240 °C

and the return temperature after the end of the analysis was set at 25 °C. The sample cooling rate was set to 40 °C/minute. The flow rate of nitrogen and oxygen gases injected into the sample was 50 ml/minute. The sample reached the set point temperature at 200 °C for 8.4 minutes. After this elapsed time, the device automatically shuts off the nitrogen flow to the sample and switches to oxygen flow. A change in the peak was observed as oxygen flow entered the sample cell or due to gas exchange.

When the duration of the process reached 9.2 minutes, significant stability or stabilization of the peak began to be observed. The total time of this stabilization process was found to be 16.0 minutes. From this time (after 16.0 min) the stability of the peak began to change and it was observed to move up. This indicates that the destruction processes in the sample are taking place. The stability of the sample peak elevation continued until 25.7 min, after which a downward slope of the sample peak was observed. The highest point of the peak lasted until 26.9 minutes. During the next period (120 minutes - 26.9 minutes = 93.1 minutes), a stable decline of the sample peak was observed. The induction oxidation time of the studied sample was determined - how long it took for the part of the sample to remain in a steady state from the time of temperature stabilization to a sharp change within two hours. To calculate this interval time, the OIT - oxidative induction time (induction oxidation time) calculation button in the computer program connected to the device was used. According to the results of computer analysis, the average induction oxidation period of the sample consisting of secondary polyethene and sulfur was determined to be 11.2 minutes. The results of this above analysis can also be seen in the differential scanning calorimetry analysis graph shown in Figure 1.



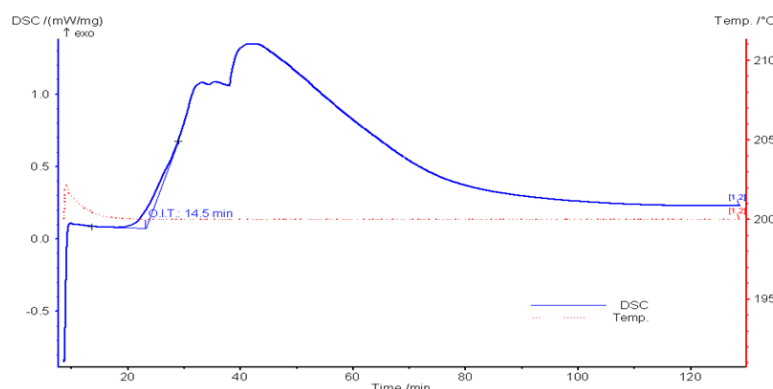
**Figure 1.** Differential scanning calorimetry analysis of secondary polyethylene

Also, the induction oxidation time analysis of the composite material made of secondary polyethene, melamine and modified sulfur was studied. The DSC analysis graph obtained by the induction oxidation time of this sample is presented in Figure 2.

9.6 mg of secondary polyethene, melamine and modified sulfur were taken from the sample to obtain the analysis results. The initial temperature of the analysis was set to 25 °C and the sample heating rate was set to 20 °C/minute. The maximum temperature of the test is 220 °C and its duration is 2 hours.

The emergency ignition temperature was set at 240 °C and the return temperature after the end of the analysis was set at 25 °C. The sample cooling rate was 40 °C/min. The flow rate of nitrogen and oxygen gases injected into the sample was set at 50 ml/minute. The sample reached the set point temperature when it reached 200 °C for 9.0 minutes. After this elapsed time, the nitrogen flow in the device was replaced by an

oxygen flow. A change in the peak of the sample occurred as oxygen flow entered the sample cell.



**Figure 2.** Differential scanning calorimetry analysis of secondary polyethene, melamine and modified sulfur composite material

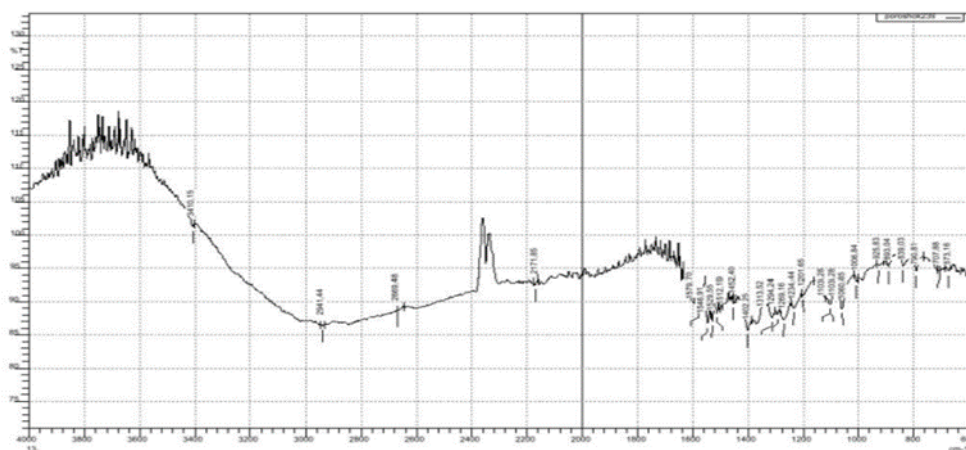
A significant stabilization of the peak was observed when the process duration reached 13.5 minutes. The total time of this first stabilization process was found to be 20 minutes. From this time (after 20 minutes) the stability of the peak began to rise. The exo-rise stability of the sample peak lasted until 32 min. The next exo rise was followed by a period of stability of the sample starting from 34 minutes and lasting until 39 minutes. From this time, another exo-rise peak of the sample began and lasted until 41 minutes.

It was found that the peak oxidation period of the sample at 211 °C lasted up to 43 minutes. During the next period (120 minutes - 43.0 minutes = 77.0 minutes), a stable decline of the sample peak was observed. It was found that the first average oxidation period in these time intervals was 14.5 minutes for the investigated composite sample, while the second oxidation time of the sample was 25.5 minutes.

When comparing the induction oxidation period of the composite samples discussed above, the oxidation time of the sample made of secondary polyethene and sulfur in the flow of oxygen at a temperature of 200 °C lasted from 8.4 to 9.2 minutes, while the oxidation time of the composite sample made of secondary polyethene, melamine and sulfur in the flow of oxygen at this temperature.

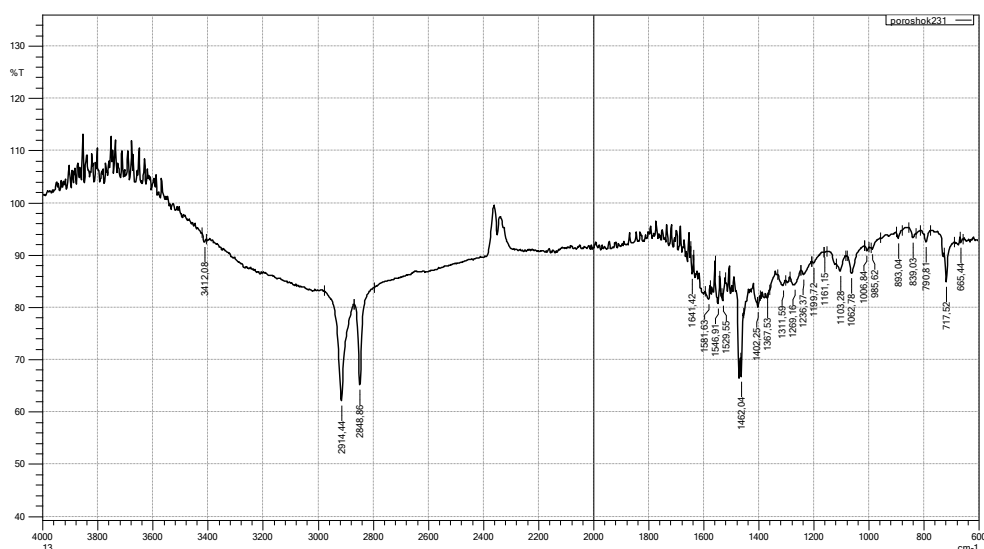
It turned out that it started at 9 minutes and lasted until 13.5 minutes. It was found that the stability period of the sample made of secondary polyethene and sulfur at a given temperature in oxygen flow was 16 minutes, while the stability period of the sample made of secondary polyethene, melamine and sulfur was 20 minutes. According to the obtained results, the average induction oxidation period of the sample consisting of secondary polyethene and sulfur was 11.2 minutes, while the average induction oxidation period of the sample made of secondary polyethene, melamine and sulfur was 14.5 minutes.

According to the analysis of the IR spectrum in secondary polyethene, weak intense absorption bands in the region of  $2941.44\text{ cm}^{-1}$  correspond to asymmetric stretching vibrations of  $\text{CH}_2$  groups in the secondary polyethene molecule and the absorption band of stretching vibrations of the  $\text{C}=\text{C}$  group is observed in the region of  $1579.70\text{ cm}^{-1}$  –  $1512.19\text{ cm}^{-1}$  (Figure 3).



**Figure 3.** IR spectroscopy of recycled polyethene

To compare the structure of the original recycled polyethene and PCM, consisting of recycled polyethene, melamine and sulfur, IR spectroscopy was obtained.

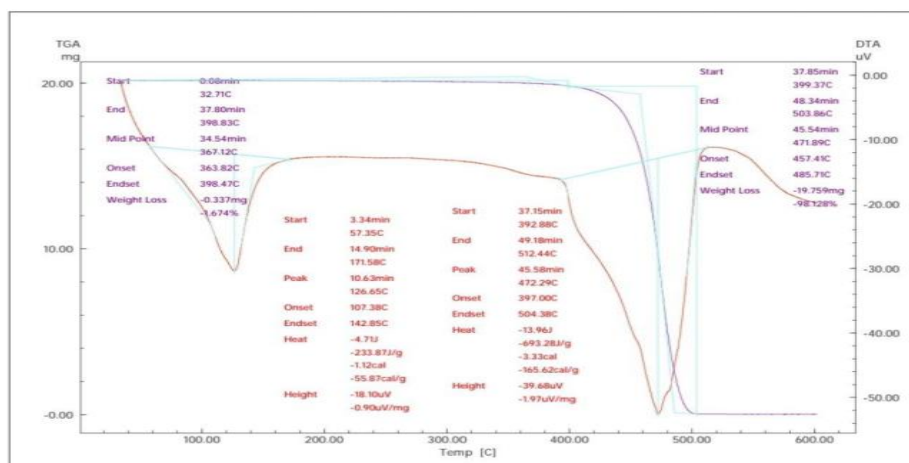


**Figure 4.** IR spectroscopy of PCM (recycled PE + sulfur + melamine)

In the IR spectrum of a polymer composite material obtained based on recycled polyethene, melamine and sulfur, the absorption band at  $3412.08\text{ cm}^{-1}$  represents the presence of N-H bonds, a group characteristic of the melamine molecule in the composite and the absorption band of scissor vibrations, characteristic of the  $\text{NH}_2$  group, is observed in the region  $1641.42\text{ cm}^{-1}$ . The absorption band of stretching vibrations, characteristic of asymmetric vibrations of the  $\text{CH}_2$  group in the secondary polyethene molecule, was observed in the region of  $2914.44\text{ cm}^{-1}$ . It was found that in this composite material, the absorption band representing symmetrical stretching vibrations of the  $\text{CH}_2$  group is observed in the region of  $2848.86\text{ cm}^{-1}$  and the absorption band characteristic of the bending vibrations of this group is observed in the region of  $1402.25\text{ cm}^{-1}$ . Deformation vibrations of a composite material consisting of recycled polyethene, melamine and sulfur, a characteristic absorption band for C=C bonds, were observed in the region of

1641.42  $\text{cm}^{-1}$ . Deformation vibrations characteristic of  $\text{C}=\text{CH}_2$  groups of the same polymer composite appear in the region of 893.04  $\text{cm}^{-1}$  and characteristic of the carbon-nitrogen bond in this composite are observed in the region of 1311.59 – 1199.72  $\text{cm}^{-1}$ , as well as a band absorption indicating the formation of a bond between the nitrogen atom and sulfur in the composite is observed in the region of 1649  $\text{cm}^{-1}$ . In the region of 1462.04  $\text{cm}^{-1}$ , an intense absorption band was observed, representing the stretching vibration characteristic of the  $\text{C}=\text{N}$  group, representing the aromaticity of the melamine molecule included in the composite. It was also found that the absorption band representing the modified S-S bond is observed in the region of 665.44  $\text{cm}^{-1}$  and the absorption band of stretching vibrations characteristic of the C-S-C bond is observed in the region of 717  $\text{cm}^{-1}$ .

The results of thermogravimetric analysis of composite materials based on recycled polyethene, sulfur and melamine were compared with the results of thermogravimetric analysis of the original recycled polyethene.



**Figure 5.** Derivatogram of secondary PE: TGA-thermogravimetric analysis curve; DTA – differential thermal analysis curve

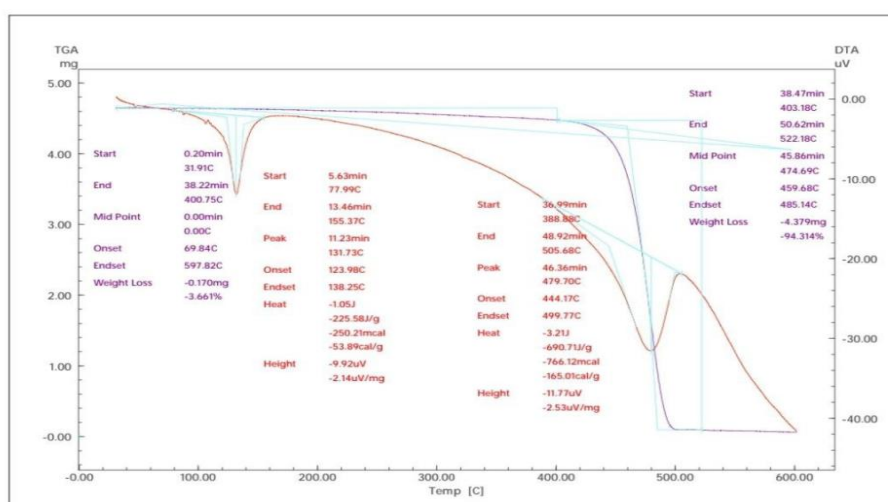
Based on the results of the thermal analysis of samples of the original recycled polyethene presented in Figure 3, important identification thermal properties were determined. For the original recycled polyethene, two decomposition periods can be observed: the first initial decomposition temperature is 32.71 °C and the final decomposition temperature is 398.83 °C. During the process, the mass of the sample decreases by 1.674 %. During the time between 0.08-37.80 minutes, the mass loss of the original recycled polyethene in the initial periods of decomposition is 0.337 mg.

The second decomposition temperature of the original recycled polyethene starts from 399.37 °C and ends at 503.86 °C. The time spent on this decomposition ranges from 37.85 to 48.34 minutes. The weight loss during this period is 19.759 mg or 98.128 %. In the DTA curve readings of the original recycled polyethene, two thermal absorptions can be seen at 126.65 °C and 472.29 °C.

A thermogravimetric analysis of the composite material based on recycled polyethene, melamine and modified sulfur was also carried out. According to the thermogravimetric analysis of the recycled polyethene, melamine and modified sulfur composite, two thermal absorptions can be seen in the DTA curve reading at 131.73 °C

and 479.70°C. The first period of decomposition of this composite material occurs in the temperature range from 31.91 °C to 400.75 °C. The duration of this decomposition ranges from 0.20 to 38.22 minutes. It was found that the quantitative decrease in the mass of the composite was 0.170 mg or 3.661%.

It was also found that the duration of the second decomposition of the same composite was 38.47 – 50.62 minutes, the temperature at the beginning of decomposition was 403.18 °C and the end temperature was 522.18 °C. It was determined that the weight loss of the composite relative to the total weight during this period was 4.379 mg or 94.314%.



**Figure 6.** Derivatogram of the recycled PE + melamine + sulfur composite. TGA curve of thermogravimetric analysis; DTA curve of differential thermal analysis

#### 4. Conclusion

In conclusion, it can be said that, according to the results obtained by the method of differential scanning calorimetry, the oxidation period of composite materials made based on secondary polyethylene in the oxygen flow depends firstly on the type, nature, quantity and chemical properties of the components included in the composite. In particular, when obtaining a composite compound consisting of secondary polyethylene and sulfur, due to the breaking of double bonds formed by the term destruction of polyethylene, the combination of modified sulfur occurs under the influence of temperature. This leads to an increase in the subsequent thermal destruction time of the composite material and an increase in the stability period. Similarly, in the sample of the composite material made of secondary polyethylene, melamine and modified sulfur, it is observed that connections between carbon atoms of secondary polyethylene and nitrogen atoms of sulfur, sulfur and melamine molecules and chemical bonds between carbon atoms of secondary polyethylene and nitrogen atoms of melamine molecule occur. Such bonds increase the stability of the formed composite materials to oxidation in temperature and oxygen flow. This is confirmed by the results of differential scanning calorimetry of the above-mentioned samples.

Based on the conducted research, we can say that the physical and mechanical properties of the studied PCMs primarily depend on the structure, quantity, chemical



nature and properties of the added components. The introduction of compounds such as sulfur and melamine into PCM helps to increase the high heat resistance of the resulting polymer composite materials. When PCM burns, the added composites formed on its surface primarily form coke, which blocks the interphase boundaries between the flame and the polymer material. Sulfur, which is part of the PCM, forms a chemical bond with the polymer matrix and vulcanizes it. Also, during combustion, sulfur in PCM forms a foam layer, which prevents the diffusion of air oxygen into the polymer material. At the same time, sulfur oxides formed during the ignition process cause a decrease in the flame temperature due to the formation of a gas phase between the flame and the composite material.

## References

- Abdullayev, B., Makhmayorov, J., Ro'ziyeva, Z., Shabarova, U., Deng, T. & Samadiy, M. (2023a). Study of the mutual influence of components in the lithium nitrate–ammonium chloride–water system. *New Materials, Compounds and Applications*, 7(3), 188-193.
- Abdullayev, B., Rifky, M., Makhmayorov, J., Usmanov, I., Deng, T. & Samadiy, M. (2023b). Adsorption method and adsorbents for the recovery of lithium compounds from water sources. *International Journal of Engineering Trends and Technology*, 71(9), 212-226.
- Battisti, M., Andreoli, S., Bacile, R., Oldani, C., Ortelli, S., Costa, A.L. & Albonetti, S. (2023). Aquivion® PFSA-based spray-freeze dried composite materials with SiO<sub>2</sub> and TiO<sub>2</sub> as hybrid catalysts for the gas phase dehydration of ethanol to ethylene in mild conditions. *Applied Catalysis A: General*, 654, 119065.
- Celina, M.C. (2013). Review of polymer oxidation and its relationship with materials performance and lifetime prediction. *Polymer Degradation and Stability*, 98(12), 2419-2429.
- Fidanovski, B.Z., Popovic, I.G., Radojevic, V.J., Radisavljevic, I.Z., Perisic, S.D. & Spasojevic, P.M. (2018). Composite materials from fully bio-based thermosetting resins and recycled waste poly (ethylene terephthalate). *Composites Part B: Engineering*, 153, 117-123.
- Jiang, J., Cai, X., Ren, H., Cao, G., Meng, J., Xing, D. & Liu, B. (2024). Effects of polyethylene terephthalate microplastics on cell growth, intracellular products and oxidative stress of *Scenedesmus* sp. *Chemosphere*, 348, 140760.
- Jin, K., Vozka, P., Kilaz, G., Chen, W.T. & Wang, N.H.L. (2020). Conversion of polyethylene waste into clean fuels and waxes via hydrothermal processing (HTP). *Fuel*, 273, 117726.
- Pinsuwan, K., Opaprakasit, P., Petchsuk, A., Dubas, L. & Opaprakasit, M. (2023). Chemical recycling of high-density polyethylene (HDPE) wastes by oxidative degradation to dicarboxylic acids and their use as value-added curing agents for acrylate-based materials. *Polymer Degradation and Stability*, 210, 110306.
- Purohit, V.B., Pięta, M., Pietrasik, J. & Plummer, C.M. (2024). Towards sustainability and a circular Economy: ROMP for the goal of fully degradable and chemically recyclable polymers. *European Polymer Journal*, 112847.
- Qin, H., Lu, H.H., Shen, X.Q., Xin, Z.J. & Yang, B. (2024). Design, preparation and electromechanical characteristics analysis of piezoelectric 1-3-type composites with sandwich polymer structures. *Sensors and Actuators A: Physical*, 366, 115024.
- Xiao, J., Wang, J., Yuan, F., Pan, X., Samadiy, M., Deng, T. & Guo, Y. (2022). Volumetric properties of the binary aqueous system lithium metaborate from 283.15 to 363.15 K and 101 kPa: Experimental and thermodynamic model. *Journal of Molecular Liquids*, 366, 120174.
- Xie, Y., Zhang, Y., Qin, J., Samadiy, M. & Deng, T. (2022). Synthesis of spherical composite CMC-LTO-EGDE-ME for lithium recovery from geothermal water. *Journal of Chemistry*, 2022.

- Zha, S., Lan, H.Q. & Huang, H. (2022). Review on lifetime predictions of polyethylene pipes: Limitations and trends. *International Journal of Pressure Vessels and Piping*, 198, 104663.
- Zhu, X., Wang, Y., Miljković, M., Li, R. & Hao, G. (2024). Effects of polymer structure on the physicochemical and performance-related properties of SBS-modified asphalt binders subjected to short-term aging. *Construction and Building Materials*, 411, 134446.