

## ORIGINAL RESEARCH ARTICLE

# Obtaining a new type of polymer materials based on recycled polyethylene waste, sulfur, and melamine

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## ABSTRACT

This study investigates the valorization of polyethylene household waste through chemical modification to develop environmentally sustainable composite materials. Secondary polyethylene (PE) was treated with elemental sulfur and melamine as chemical modifiers to enhance its properties. The modified PE was characterized using advanced spectroscopic techniques, including Fourier-transform infrared spectroscopy (FTIR) and other analytical methods, in accordance with ISO standards. Physical and mechanical properties, such as tensile strength, elongation at break, and hardness, were systematically evaluated to assess the performance of the resulting composites. The integration of melamine and chemically modified sulfur into secondary PE yielded materials with improved mechanical robustness and functional characteristics suitable for various industrial and consumer applications. This approach demonstrates an effective strategy for reducing polyethylene waste by converting it into value-added, functional composites, thereby promoting ecological sustainability and waste minimization. The findings underscore the potential of chemically modified secondary polyethylene as a viable resource for sustainable material development, contributing to environmental conservation and waste management efforts. Overall, this methodology offers a promising route for the reutilization of polyethylene household waste, aligning with principles of circular economy and ecological responsibility.

**Keywords:** Secondary polyethene; melamine; isothermal temperature; stability; thermomechanical

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## 1. Introduction

Today, on account of the increasing population of the globe, the demand to satisfy the human needs of this generation is also increasing in parallel. Every year, the rise of population worldwide requires the expansion of production and industry. As a result, the globalization of big cities and the development of production and industry leads to the generation of industrial exhaust gases, solid and liquid waste, and household and other waste. In addition, the volume of industrial and production waste is multiple times more than household waste. This indicates that humanity and the environment will face major ecological unbalance in the future<sup>[1-3]</sup>. Every year, the

volume of different wastes rises by 6-7%. Polyethylene bags, which can have a thickness of up to 40 microns, are one of these wastes that have received less attention and are not recyclable<sup>[4-6]</sup>.

Currently, the main goal is to turn polyethylene bags and other types of plastic waste into other consumer goods<sup>[7]</sup>. There are several problems related to the disposal of waste polymer products. Disposal processes have their own specificity, but it is incorrect to think that it is impossible to solve these problems. The most convenient and correct way to dispose of waste from polymer products is their secondary processing<sup>[8,9]</sup>.

A number of scientific research works and practical experiments have been carried out on the secondary processing of polymers and their utilization and improvement of the ecological environment<sup>[10]</sup>. It was also noted that one of the main methods of disposal of polymer product waste is incineration<sup>[11]</sup>. Some articles also mention methods of depolymerization of polymer products by neutral hydrolysis in secondary processing<sup>[12]</sup>. In addition, ways of using polymer waste as textile materials for water and air purification are shown<sup>[13]</sup>. Methods of preliminary preparation of polymer waste recycling in actual devices are explained. Scientific approaches to the increase in the cost of the secondary product obtained in this stage of recycling are presented<sup>[14]</sup>.

Polyethylene Terephthalate (PT) is one of the items that is widely used in the chemical industry in order to produce various fibers, films, and composite materials. Reports witness the fact 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 at least two types such as technological waste and production-related waste. 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<sup>[15]</sup>.

Further observations are evident that the production of composite materials based on ethylene and propylene copolymers is comparatively in the leading position. In some scientific works, certain studies on the decrease in elasticity and increase in density of polymer materials by incorporating talc into the component have concluded that by adding dispersed fillers to polyethylene, it is possible to increase all thermophysical properties and reduce flammability<sup>[16]</sup>.

## 2. Materials and methods

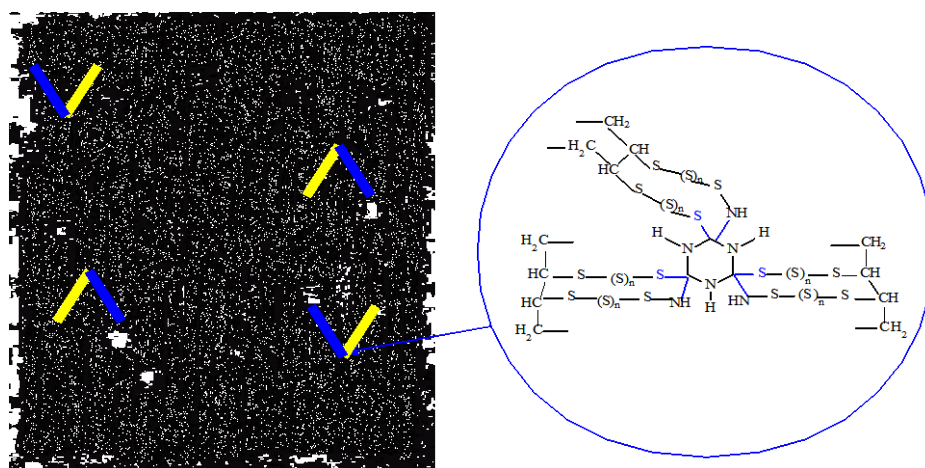
The study focused on modifying recycled low-pressure polyethylene ( $\rho = 0.9246 \text{ g/cm}^3$ , liquefaction temperature 402 K) with elemental sulfur (Sigma-Aldrich (Merck, Germany), 99.5 -100% purity, reagent grade, yellow crystalline powder, molecular mass of 256.48 g/mol.) and melamine (Sigma-Aldrich (Merck, Germany), 99%, purity, analytical grade, white crystalline powder, molecular weight 126.12 g/mol.) in a 50:1:1 ratio. After cleaning, drying, and mechanical crushing, the polyethylene mixture was intensively stirred at 120-135 °C for 0.5 hours, then pressed into 2-2.5 mm thick sheets. Thermogravimetric analysis (TGA) was performed using a Shimadzu DTG-60, with 5 mm round samples conditioned according to international standards. The system temperature was calibrated, and the heating rate was set at  $(10 \pm 1) \text{ }^\circ\text{C/min}$  to 600°C under controlled gas flow. Infrared (IR) spectroscopic analysis was performed using an IR Affinity-1S device within the 4000-400  $\text{cm}^{-1}$  range. The softening temperature was determined by the VICAT method (IC6+N) based on international standards. Three 10 mm<sup>2</sup>, 4 mm thick specimens were tested by dipping a 1 mm<sup>2</sup> cylindrical tool under a load of 1019 g. The temperature at 1 mm penetration was recorded, and the tests were repeated three times for each specimen. Induction oxidation time was measured using a differential scanning calorimeter (ASTM D3895-DSC). The samples were heated under a nitrogen flow, then exposed to oxygen, and the onset of oxidation was recorded. The test provided an assessment of the stability of recycled polyethylene composites<sup>[17]</sup>. The recycled polyethylene utilized in this study was

gathered, sorted, cleaned, and pelletized from post-consumer waste, mostly HDPE bottles and LDPE bags. This source was chosen because of its abundance and relevance to the problems surrounding the management of plastic trash.

### 3. Results and discussion

Several practical studies have been conducted to obtain various industrial consumables and construction materials through the secondary processing of waste polyethylene films, composed of polyethylene and expired materials. The research utilized the method of chemical modification of recycled polyethylene, aiming primarily to dispose of industrial and household polymer material waste and contribute to environmental protection. The processed polymer composite material also aims to enhance resistance to temperature, natural elements, and other aggressive environments, while achieving improvements in strength, fluidity, softening temperatures, and other physical and mechanical properties.

The modification of polymer materials is generally categorized into two major groups: structural modification and chemical modification. Chemical modification entails the addition of substances that form chemical bonds with the macromolecules of the initial polymer product, establishing a network of covalent bonds between the macrochains. Specifically, in the thermal processing of recycled polyethylene, cross-links are generated between macromolecules through either macrochain breaks or structuring processes. The hydrogen molecule of the amino group in the melamine molecule acquire the imine structural state due to the internal rearrangement of the adjacent nitrogen atom. This process is made easier by temperature. During composite formation, the double bond between nitrogen and carbon atoms in the melamine molecule is disrupted. Observations reveal that modified sulfur atoms attach to the valence bonds resulting from the rupture of the double bond between nitrogen and carbon atoms in the melamine molecule. Additionally, these modified sulfur atoms attach to the second empty valence bonds, forming cross-chemical bonds with thermo-destructed recycled polyethylene macromolecules. The double bonds between carbon and nitrogen in the three-imine structures of the melamine molecule are broken, and they form chemical bonds with the double bonds of thermo-destructed recycled polyethylene macromolecules. In this process, the chemical bonding of modified sulfur and melamine to the linear recycled polyethylene macromolecule is illustrated in Figure 1:

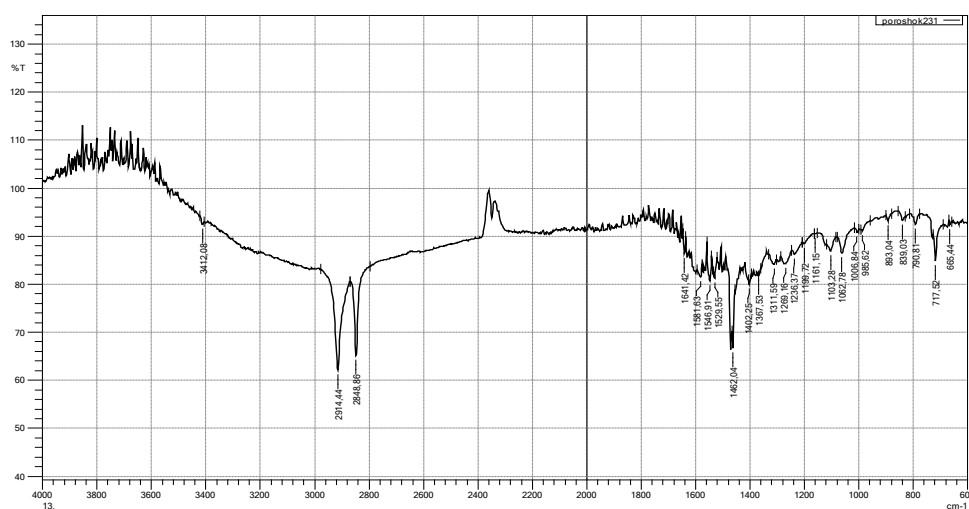


**Figure 1.** Modified sulfur and melamine bonded banded and linear recycled polyethylene

In this process, in addition to the aforementioned connection, it is also possible for modified sulfur to bond with one of the nitrogen-carbon bonds in the compound formed by breaking the double bond of the melamine molecule, and with the remaining two nitrogen-carbon bonds of the product resulting from the bond breakage of the term destruction product of recycled polyethylene. Furthermore, it is possible for

modified sulfur atoms from the breakage of double bonds due to two imine groups of melamine to join one recycled polyethylene macromolecule chain, forming a chemical bond with the remaining nitrogen-carbon bond. Additionally, the modification of sulfur can occur due to the breakage of carbon-nitrogen bonds characteristic of the three imine groups in the molecule of modified sulfur melamine.

The modified sulfur and melamine molecules integrated into recycled polyethylene establish chemical bonds between the macromolecules of recycled polyethylene, creating a stitching effect. Subsequently, this process leads to the vulcanization of recycled polyethylene products through the formation of sulfide bridges between modified sulfur and recycled polyethylene macromolecules. Furthermore, the inclusion of polymerized sulfur and melamine in recycled polyethylene alters the substantial and mechanical properties of the resulting composite materials. In these polymer composite materials, heat resistance increases, and flammability decreases. Oxidation stability is enhanced, and the coefficients of resistance to external air, aggressive environments, and light are also elevated compared to the original polyethylene products. Bonds in the structure of a composite material based on melamine, modified sulfur, and recycled polyethylene were examined using infrared spectroscopy<sup>[18,19]</sup>.



**Figure 2.** IR-spectrum of recycled polyethylene composite material

Absorption lines at  $3412.08\text{ cm}^{-1}$  in the IR-spectrum of the recycled polyethylene, melamine, and sulfur-based composite material presented in Figure 2 above represent the N-H collective characteristics of the melamine particles in the combination. Meanwhile, the scissor vibration absorption lines characteristic of the  $\text{NH}_2$  group were determined in the area of  $1641.42\text{ cm}^{-1}$ . The valence atmospheres, characteristic of asymmetric vibrations of the  $\text{CH}_2$  group in the polyethylene molecule, were observed in the region of  $2914.44\text{ cm}^{-1}$ . It was found that the absorption lines representing the symmetric valence vibrations of the  $\text{CH}_2$  group in this composite material are observed in the region of  $2848.86\text{ cm}^{-1}$ , and the absorption lines characteristic of the deformation vibrations of this group are observed in the region of  $1402.25\text{ cm}^{-1}$ . Deformation vibrations characteristic of  $\text{C}=\text{C}$  bonds of this composite material are observed in the region of  $1641.42\text{ cm}^{-1}$ . Distortion vibrations characteristic of  $\text{CH}_2$  groups of similar polymer combinations were observed in the array of  $893.04\text{ cm}^{-1}$ . Absorption lines at  $1311.59\text{--}1199.72\text{ cm}^{-1}$ , represent the valence vibrations of the C-N bond, the basis of the carbon-nitrogen bond in the formation of recycled polyethylene, melamine, and sulfur composite, N-S, representing the formation of molecule bonds between the nitrogen atom and sulfur in the melamine molecule, showed absorption lines in the region of  $1649\text{ cm}^{-1}$ . It was also observed that the absorption lines corresponding to the modified S-S bond are observed in the region of  $665.44\text{ cm}^{-1}$ , and the valence vibrations characteristic of the C-S-C bond are spotted in the region of  $717\text{ cm}^{-1}$ .

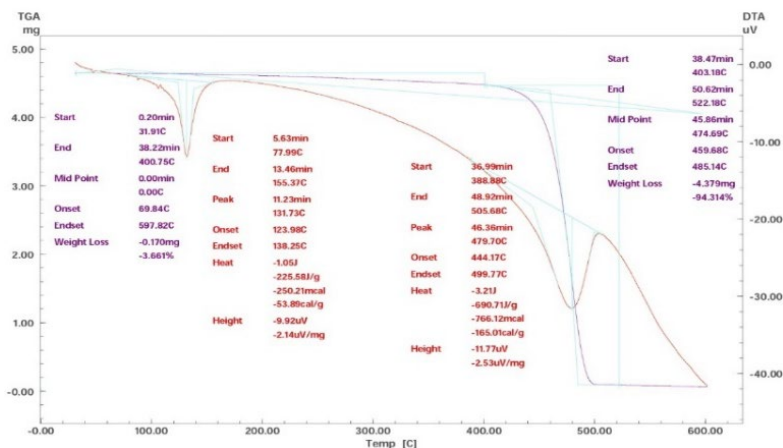
Research on the thermal stability of combined materials obtained based on polyethylene that is recycled reveals that plastics change their primary properties during their service life under the influence of heat, moisture, tension, light, and air oxygen. As a result, they harden, become brittle, and experience a deterioration in their electrical properties. Temperature has a particularly negative effect on most polymer material properties. The FTIR spectra show the characteristic bands of polyethylene (PE): The CH<sub>2</sub> rocking (730-720 cm<sup>-1</sup> ~2915 cm<sup>-1</sup> ~2848 cm<sup>-1</sup>), the CH<sub>2</sub> bending (scissoring) at ~1465 cm<sup>-1</sup>, ~2915 cm<sup>-1</sup>, ~2848 cm<sup>-1</sup> asymmetric and symmetric, and the CH<sub>2</sub> asymmetric and symmetric stretching vibrations at ~2915 cm<sup>-1</sup> ~2848 cm<sup>-1</sup> asymmetric and symmetric, respectively. Since PE only shows nonpolar hydrocarbon vibrations, it naturally lacks polar group vibrations and has low reactivity. Melamine, in contrast, exhibits triazine ring out-of-plane units at 800-700 cm<sup>-1</sup>, broad NH peaks at 3500 - 3200 cm<sup>-1</sup>, and a strong CN triazine ring at 1650-1550 cm<sup>-1</sup>, all within the range of CN and ring-breathing units 1400 - 1000 cm<sup>-1</sup>.

For instance, a decrease in temperature increases the brittleness of most polymer materials, whereas heat causes a decrease in properties such as the linear expansion coefficient and impact viscosity. It also leads to a decrease in the mechanical properties of polymer materials and an increase in their properties, such as breaking, bending, compression, and hardness. All research conducted on composite materials made from recycled polyethylene waste meets the requirements of international standards for polymer materials. Thermogravimetric analysis was determined by the difference between the initial temperatures and decomposition rates of the samples. Thermogravimetric results of composite materials based on recycled polyethylene were calculated based on the rate of mass reduction of the samples taken for the experiment:

$$vm = \Delta m / \Delta \tau, \quad (1)$$

where:  $\Delta m$  - mass reduction, mg;  $\Delta \tau$  - time interval, min.

The results of thermogravimetric analysis of compound substantial based on recycled polyethylene, melamine, and modified sulfur (Figure 3) were reviewed.

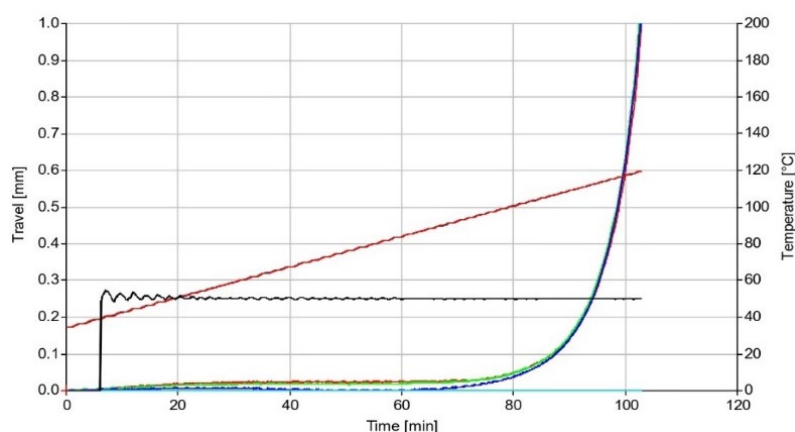


**Figure 3.** Derivatogram represents recycled polyethylene, melamine, and modified sulfur composite, includes TGA (Thermogravimetric Analysis) and DTA (Differential Thermal Analysis) curves, illustrating its thermal stability and decomposition behaviour

According to the thermogravimetric analysis of the recycled polyethylene, melamine, and modified sulfur composite, the DTA curve readings reveal output signal line values for two heat absorption effects at temperatures of 131.73 °C and 479.70 °C. The first decomposition duration of this combined material is observed in the temperature range of 31.91 °C to 400.75 °C, lasting from 0.20 minutes to 38.22 minutes. During this period, a decrease in the composite amount was found to be 0.170 mg, with a percentage of 3.661%. The following level or second decomposition time of the combination started at 38.47 minutes and ended at 50.62 minutes, with an initial temperature value of 403.18 °C and a final temperature value of

522.18 °C. The mass fall of the composite with the total mass during this period was 4.379 mg, with a mass fraction of 94.314%.

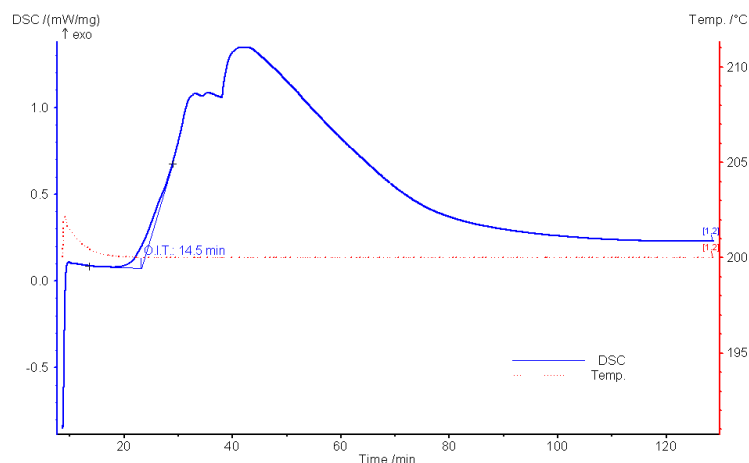
The softening point of the recycled polymer composite samples was determined using the VICAT analyzer (Model: IC6 + N) (method for determining the softening point of thermoplastics according to Vicat). This method aims to determine the heat resistance of plastics according to the Vicat softening temperature methods. During the processing of polymer material, it expands in terms of volume under the influence of temperature. Determining the thermal conductivity coefficient, temperature transfer coefficient, specific heat capacity, and other indicators of any material helps to determine the maximum heating and cooling capabilities of the material<sup>[20,21]</sup>. The heat-softening temperatures of recycled polyethylene, melamine, and composite material samples based on modified sulfur were determined using the Vicat softening temperature methods. According to the method, the initial softening temperature of the recycled polyethylene composite started from 34°C. The results, based on three experiments for each sample, showed an average temperature value of 119.5°C. During the research, it was found that the depth of penetration of the needle into the sample was 0.02 mm at the specified temperature (119.5°C) and given load (1019 g). Figure 4 below illustrates the thermal softening temperature graph of the recycled polyethylene, melamine, and modified sulfur composite using the Vicat softening temperature methods.



**Figure 4.** Thermal softening temperature curve of recycled polyethylene, melamine, and modified sulfur composite by vicat method

Determination of the induction oxidation time of recycled polyethylene composites. The initiation of the oxidation period of recycled polyethylene complex samples was determined using an ASTM D3895-DSC Differential Scanning Calorimetry (Standard Test Method for Determination of Induction Oxidation Time of Polyolefins by *Differential Scanning Calorimetry*). The duration or temperature of the oxidation data serves as an estimate of the material's stabilization degree. It is important to note that a high-temperature regime leads to a reduction in the oxidation time of the material. Additionally, the oxidation results' duration or the level of temperature depends on the surface area of the material under investigation, with research conducted in a pure oxygen stream resulting in a shorter time or temperature compared to research conducted with air oxygen<sup>[22-25]</sup>. Furthermore, the time or temperature of the oxidizing induction of the studied sample is influenced by the specific characteristics of the composites added to the sample. In Figure 5 below, the analysis of induced oxidation time for a composite sample based on recycled polyethylene, melamine, and modified sulfur is presented:





**Figure 5.** DSC analysis of composite material obtained based on recycled polyethylene, melamine, and modified sulfur

Composite sample (9.6 mg) was utilized to obtain the analytical results. The analysis commenced with an initial temperature set at 25°C, employing a sample heating rate of 20°C/minute. The maximum test temperature was 220°C, with a duration of 2 hours. The temperature of emergency ignition was set to 240°C, and the return temperature after the analysis was fixed at 25°C. The cooling rate of the sample was established at 40°C/minute. The flow proportion of nitrogen and oxygen gases injected into the sample was maintained at 50 ml/minute. The sample reached the set point at a temperature of 200°C within the period of 9.0 minutes. Subsequently, the nitrogen flow in the device was replaced by an oxygen flow, resulting a change in the peak of the sample. A notable stabilization of the peak was observed after a period of 13.5 minutes of the process. The total tenure for this initial stabilization process was found to be 20.0 minutes. Beyond this time of the process (after 20.0 minutes), the stability of the peak began to increase. The stability of the first peak of the sample persisted until 32.0 minutes and a second period of stability for the sample was observed from 34.0 minutes. The second stability duration of the sample was determined to be 39.0 minutes. From this point, the sample peak started to rise again and continued until 41 minutes. The highest oxidation duration of the sample at 211°C was observed up to 43 minutes. During the subsequent period (2 hours, i.e., 120 minutes - 43.0 minutes = 77.0 minutes), a stable decline of the sample peak was noted. The average duration of the first oxidation period during this investigation was found to be 14.5 minutes for the composite sample, while the second oxidation period of the sample was 25.5 minutes.

By using inverse vulcanization or reactive blending to combine rPE with molten elemental sulfur and melted melamine (or melamine-formaldehyde resin) as a co-crosslinker or reinforcing reactive additive, you can transform discarded PE into a sulfurous, cross-linked polymer composite. The most practical manufacturing processes to report are solvent-free mechanochemical mixing, reactive melt blending, or reactive extrusion, followed by heating. These methods provide materials with improved thermal/mechanical and innovative properties while using inexpensive sulfur, a byproduct of industrial processes.

Melamine and sulfur were selected because they are complementary to one another. Melamine is a crosslinker that contains nitrogen and is resistant to heat and flame, while sulfur is employed as a reactive crosslinker and source of functionality. When combined, they enable the transformation of low-value and ineffective recycled polyethylene trash into multipurpose and high-performance polymer materials, which is consistent with the circular economy's tenets and the development of superior material designs.

## 4. Conclusion

The production of polymer composite products using recycled polyethylene waste materials, such as melamine and sulfur, has been studied in this research. According to the discussion, the resulting composites have excellent mechanical strength and thermal stability, which supports the findings of TGA, Vicat

softening temperature, and DSC. New structural features that suggested the existence of chemical interactions between polyethylene, sulfur, and melamine could have emerged, according to the results of the IR spectroscopy. These findings imply that these composites can be applied to construction applications such tiles, linoleum, panels, profiles, and decorative materials. To demonstrate the relative benefits of such composites over conventional building polymers and unmodified polyethylene, more research is necessary. The most obvious changes in the work should be the direct comparison with virgin and secondary polyethylene in the future, along with the more comprehensive study of the bonding processes that underlie it.

## Author contributions

Shakhnoza Mamatova: Conceptualization, methodology, investigation, writing- original draft; Bakhodir Abdullayev: Visualization, project administration, writing- reviewing and editing; Mingnikul Kurbanov, Mohamed Rifky: Supervision, visualization, project administration, writing- reviewing and editing, reformatting, grammar editing; Guzal Davronova, Murodjon Samadiy, Abduakhad Kodirov, Matluba Safarova, Lobar Bozorova, Guzal Rakhmatova, Umida Shabarova, Anora Jumayeva, Rano Toshkodiroya, Yakibova Dilafuz, Dilbar Atakulova, Jasur Farmonov: Investigation and resource.

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## Conflict of interest

The authors declare no conflict of interest.

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