SEM, EDS AND TGA/DTA ANALYSIS OF FLAME-RETARDANT ORGANIC COMPOSITE MATERIALS BASED ON POLYVINYL CHLORIDE

Abdirashidov Durbek
Doctoral Student, Termiz State University, Termiz, Uzbekistan
Tel: (+9989) 686-4321
E-mail: durbek.abdirashidov95@mail.ru
Turaev Khayit
Professor, Termiz State University, Termiz, Uzbekistan
Tel: (+9989) 579-0633
E-mail: khturaev@rambler.ru
Tajiyev Panji
Professor, Termiz State Pedagogical Institute, Termiz, Uzbekistan
Tel: (+9989) 591-6045,
E-mail: panjitajiyev74@gmail.com

Abstract. In this study, the scientific and practical foundations for obtaining flame-retardant organic composite materials based on polyvinyl chloride (PVC) were developed. The synthesized samples were analyzed using Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDS), Thermogravimetric and Differential Thermal Analysis (TGA/DTA/DTG), as well as mechanical testing. These analytical methods were employed to evaluate the dispersion degree of the additives, the integrity of the interfacial structure, the thermal stability of the material, the stress–strain behavior, and smoke emission characteristics.The obtained results demonstrated that the combined use of talc and melamine cyanurate produces a synergistic effect in PVC composites, enhancing flame resistance, heat stability, and overall thermal durability. Moreover, mechanical tests confirmed that the PVC–melamine cyanurate–talc composition exhibited the highest strength and deformation resistance. The findings open new prospects for the practical application of PVC-based composites and provide a pathway for improving the technology of creating efficient flame-retardant organic materials.

Keywords: PVC composites, flame retardancy, melamine cyanurate, talc, SEM/EDS analysis, thermal behavior

Аннотация. В данной работе разработаны научные и практические основы получения огнестойких органических композиционных материалов на основе поливинилхлорида (ПВХ). Синтезированные образцы исследовались с помощью сканирующей электронной микроскопии (СЭМ), энергодисперсионной рентгеновской спектроскопии (ЭДС), термогравиметрического и дифференциально-термического анализа

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(ТГА/ДТА/ДТГ), а также механических испытаний. С помощью этих методов анализа оценены дисперсность добавок, целостность структуры межфазной границы, термическая стабильность материала, его деформационное поведение и характеристики дымовыделения. Полученные результаты показали, что совместное применение талька и цианурата меламина оказывает синергетический эффект в ПВХ-композициях, повышая огнестойкость, термостойкость и общую термостойкость. Более того, механические испытания подтвердили, что композиция ПВХ–цианурат меламина–тальк обладает наибольшей прочностью и деформационной устойчивостью. Полученные результаты открывают новые перспективы практического применения композитов на основе ПВХ и позволяют совершенствовать технологию создания эффективных огнестойких органических материалов.

Ключевые слова: ПВХ-композиты, огнестойкость, цианурат меламина, тальк, анализ SEM/EDS, термические свойства

Annotatsiya. Ushbu tadqiqotda polivinilxlorid (PVX) asosida olovga chidamli organik kompozit materiallarni olishning ilmiy va amaliy asoslari ishlab chiqildi. Sintezlangan namunalar skanerlash elektron mikroskopiyasi (SEM), energiya dispersiyasi rentgen spektroskopiyasi (EDS), termogravimetrik va differentsial termik tahlil (TGA/DTA/DTG), shuningdek, mexanik sinov yordamida tahlil qilindi. Ushbu analitik usullar qo'shimchalarning dispersiya darajasini, sirt tuzilishining yaxlitligini, materialning termal barqarorligini, kuchlanish-deformatsiya xususiyatini va tutun chiqarish xususiyatlarini baholash uchun qo'llanildi. Olingan natijalar shuni ko'rsatdiki, talk va melamin siyanuratni birgalikda qo'llash PVX kompozitlarida sinergetik ta'sir ko'rsatadi, bu esa olovga chidamlilik, issiqlik barqarorligi va umumiy termal chidamlilikni oshiradi. Bundan tashqari, mexanik sinovlar PVX-melamin siyanurat-talk tarkibi eng yuqori mustahkamlik va deformatsiyaga chidamlilikni namoyish etishini tasdiqladi. Tadqiqot natijalari PVX asosidagi kompozitlarni amaliy qo'llash uchun yangi istiqbollarni ochadi va samarali olovga chidamli organik materiallarni yaratish texnologiyasini takomillashtirish yo'lini taqdim etadi.

Kalit so'zlar: PVX kompozitlari, olovga chidamlilik, melamin siyanurat, talk, SEM/EDS tahlili, issiqlik xususiyatlari

Introduction

Polymer materials used in modern construction and industrial applications are distinguished by their high mechanical strength, low weight, chemical stability, and ease of processing. However, most polymers, particularly polyvinyl chloride (PVC), exhibit low resistance to heat and fire, which limits their practical

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applications. Therefore, enhancing the flame resistance of polymers has become one of the key scientific and technical challenges in modern materials research.

The main strategies for improving the fire resistance of polymer materials include the use of flame-retardant additives (antipyrenes), optimization of stabilizer and plasticizer ratios, and the creation of synergistic mixtures with mineral fillers. In particular, incorporating halogen-free flame retardants such as melamine and melamine cyanurate, along with lamellar mineral fillers like talc, significantly increases the fire-retardant efficiency of PVC-based composites.

The relevance of this research lies in the need to develop PVC-based flame-retardant materials that are not only resistant to fire but also environmentally safe, mechanically stable, and suitable for long-term operation. Therefore, it is essential to thoroughly study the interaction mechanisms among plasticizers, stabilizers, and fillers in polymer-composite systems.

In this work, the synergistic effects of the plasticizer DOF, stabilizers melamine and melamine cyanurate, and the mineral filler talc in PVC-based composite materials were investigated. Based on the obtained results, new scientific approaches were developed to enhance the flame-retardant properties of polymer-composite materials.

Materials and Methods

In this study, the experimental analyses were carried out using several advanced instruments. The microstructural characteristics of the obtained samples were examined by a scanning electron microscope (SEM, JEOL JSM-6460LA, Japan), while the elemental composition of the composite surfaces was determined using energy-dispersive X-ray spectroscopy (EDS). Thermal stability and decomposition behavior were investigated by thermogravimetric and differential thermal analysis (TGA/DTA/DTG). These instrumental methods provided comprehensive information on the dispersion of additives, interfacial integrity, and the thermal behavior of the synthesized PVC-based flame-retardant composite materials.

Results and Discussion

In addition to the fire-resistant characteristics of the developed composites, it is important to evaluate how the physical and mechanical properties of intumescent flame-retardant polymer composite materials change over time. It is well known that in construction applications, when intumescent flame-retardant polymer composites are applied to metal structures, various issues may arise due to reduced adhesion over time. These observations indicate that, beyond proper application techniques and adherence to operating conditions, it is also essential to assess the long-term changes in the physicomechanical and chemical properties of such flame-retardant polymer composites. In this research, the interrelation

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between the components enhancing flame resistance—namely, the plasticizer dioctyl phthalate (DOF), the stabilizers melamine and melamine cyanurate, and the local mineral filler talc—was systematically investigated. Initially, the behavior of polyvinyl chloride (PVC) with the addition of DOF plasticizer was analyzed, revealing that an increase in flame resistance can be achieved not only through the use of plasticizers but also by incorporating stabilizers and fillers to create a synergistic effect. Specifically, Figure 3.1 presents the SEM and EDS analyses of the PVC + DOF composite. In the EDS spectrum, the "Energy [kV]" axis represents the radiation energy emitted by the detected elements, while the "Counts/eV" axis reflects the number of detected signals, corresponding to intensity. The higher the peak, the greater the concentration of that element. According to the analysis, the sample primarily contains carbon (C), oxygen (O), and chlorine (Cl). The pronounced chlorine peaks confirm that PVC constitutes the main component of the material, whereas the presence of carbon and oxygen originates from the DOF plasticizer incorporated into the matrix.

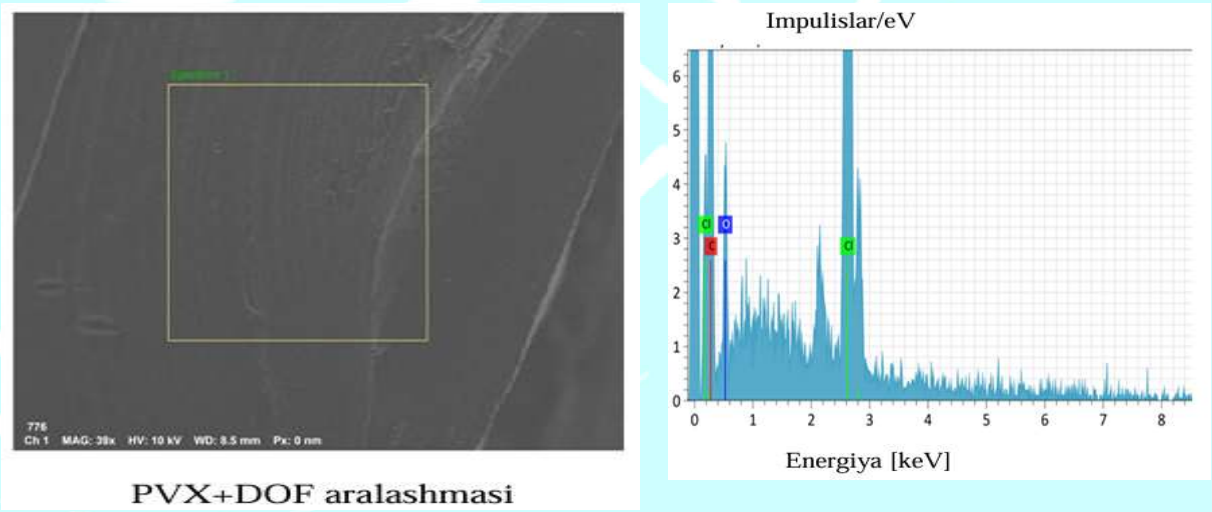


Figure 1. SEM and EDS analysis of the material obtained from the PVC + DOF composite mixture.

According to the data presented in Table 3.1, carbon (C) constitutes about 62 % of the total composition, confirming the presence of organic polymeric material in the sample.

Table 1.

SEM microstructural analysis of the material sample obtained from the polyvinyl chloride–dioctyl phthalate (PVC–DOF) composite mixture.

Element	Atomic Number	Mass [%]	Absolute Error [%] (1 sigma)
Carbon (C)	6	62,32	3,9
Chlorine (Cl)	17	32,23	3,48
Oxygen (O)	8	5,45	0,51
Total		100.00	

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According to the purpose of this research, it is considered appropriate to enrich the composition of polyvinyl chloride (PVC) with fillers, plasticizers, and various stabilizers in order to obtain flame-retardant organic materials. In this study, samples were prepared using halogen-free flame retardant melamine and dioctyl phthalate (DOF) to develop fire-resistant organic materials based on PVC. The relationship between the stabilizer melamine and its contribution to flame resistance was examined. The process was conducted using a twin-screw extruder-granulator, where measured quantities of materials (3,187.5 g of PVC, 750 g of melamine, and 1,062.5 g of DOF) were gradually added in stages. In the first stage, the mixture was maintained at 70 °C under continuous stirring to achieve a homogeneous phase. In the second stage, the extruder screw was heated to 180 °C, and the obtained mixture was converted into a composite form and granulated. The analysis of the resulting samples was performed using a scanning electron microscope (JEOL JSM-6460LA, JEOL Interactive Corporation, Japan). Through electron microscopy and elemental analysis, the morphology and dispersion of melamine particles in the PVC matrix were examined, as well as the synergistic effects that improve flame retardancy. The resulting flame-retardant polymer materials consisted primarily of polyvinyl chloride, dioctyl phthalate, and melamine. By introducing 5–30 wt % of the selected halogen-free flame retardant melamine, the flame resistance of the material was significantly improved. Furthermore, SEM and EDS analyses confirmed the homogeneous distribution of melamine particles over the polymer surface and their contribution to enhanced fire performance.

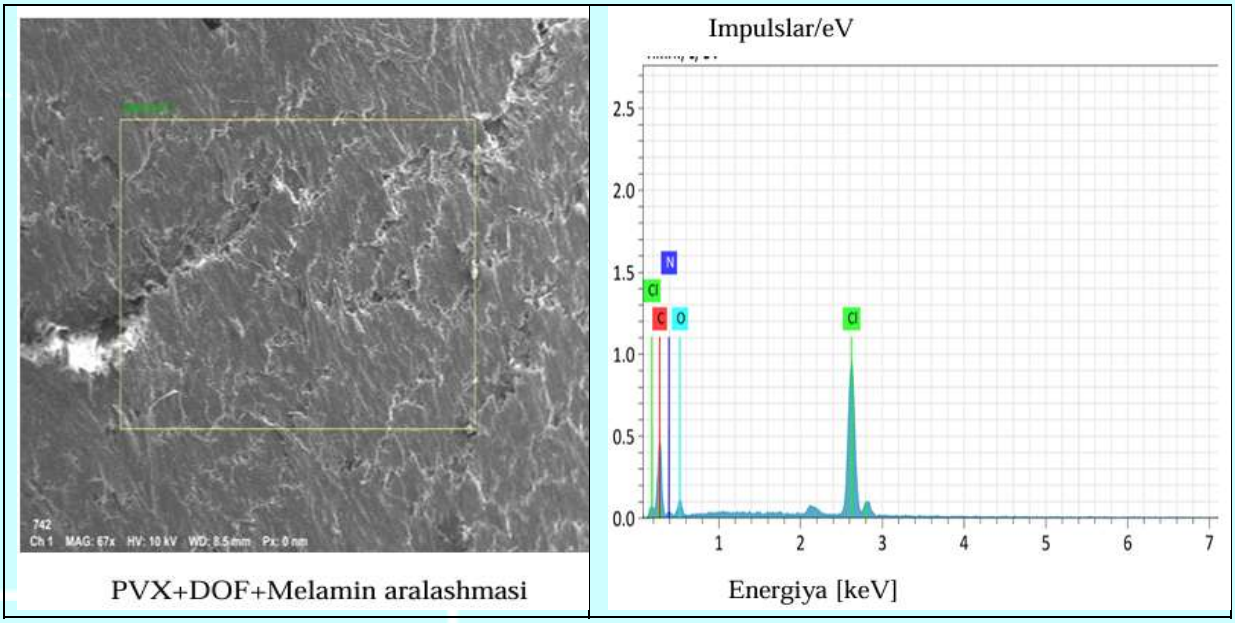


Figure 2. (a) SEM microstructure of the surface of the material sample obtained from the PVC–melamine composite (67×, 10 kV, 200 µm). (b) EDS spectrum of the surface of the PVC–melamine composite sample.

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According to Figure 2, an EDS analysis was performed on the selected "Spectrum 1" region. The results revealed that the surface of the material exhibited a lamellar structure with localized microvoids and fine cracks formed as a result of internal stresses. This morphology reflects the interaction between the polymer matrix and the flame-retardant additive melamine. Analysis of the SEM images and EDS spectra indicated that the melamine particles incorporated into the PVC-SG3-based composites were well dispersed within the matrix, although some regions contained minor microvoids. Overall, the combined SEM and EDS results confirmed the successful incorporation of the additives into the composite material; however, they also suggest that additional dispersion methods—such as high-shear mixing or the use of coupling agents—may be required to improve interfacial integrity. The compositional analysis of the obtained sample is presented in Table 2. The spectrum shown in the figure was obtained using the Energy Dispersive X-ray Spectroscopy (EDS) method, which enables the identification of chemical elements present in the sample. The high peak intensity of chlorine (Cl) indicates its significant content in the material. The presence of carbon (C), nitrogen (N), and oxygen (O) further confirms the organic nature of the composite. Thus, the analyzed sample represents a flame-retardant organic material that demonstrates superior fire resistance compared with the PVC + DOF composition.

Table 2.

SEM microstructural analysis of the material sample obtained from the polyvinyl chloride–melamine composite.

Element	Atomic Number	Mass Normal [%]	Absolute Error [%] (1 σ)
Carbon (C)	6	56.22	2.64
Chlorine (Cl)	17	33.39	1.82
Oxygen (O)	8	6.43	0.35
Nitrogen (N)	7	3.96	0.44
Total		100.00	

Specifically, Figure 2 presents the SEM and EDS analyses of the material obtained from the polyvinyl chloride–di(2-ethylhexyl) phthalate–melamine mixture. The elemental composition of the sample consists of 56.22% carbon (C), 33.39% chlorine (Cl), 6.43% oxygen (O), and 3.96% nitrogen (N).

The differential thermogravimetric (DTG) analysis of the obtained samples was further examined. As is well known, DTG is one of the thermal analysis

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methods that measures the rate of change in a substance's mass as a function of time or temperature. In other words, this technique records how the sample's mass changes as its temperature increases. The DTG curve expresses the rate of mass loss (dm/dt) as a function of temperature.

This method is commonly used for the following purposes:

– studying the thermal degradation processes of polymers,

– determining the thermal stability of materials,

– quantifying volatile components within a sample, and

– investigating the kinetics of chemical reactions.

DTG analysis is often performed in conjunction with Thermogravimetric Analysis (TGA). While the TGA curve displays the dependence of a sample's mass on temperature, the Differential Thermal Analysis (DTA) curve determines the rate of mass change and more clearly identifies the peaks corresponding to decomposition processes (Figure 3).

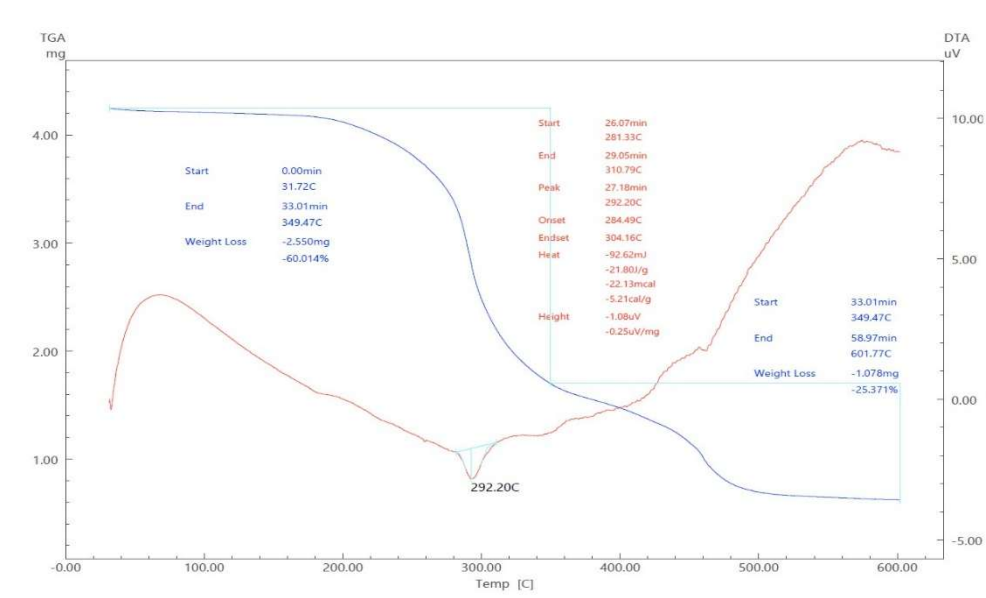


Figure 3. DTG and TGA analysis of the sample obtained from the polyvinyl chloride–melamine composite.

From the graph, it can be observed that the sample undergoes a two-stage thermal decomposition process. The first major weight loss occurs in the temperature range of 31.72–349.47 °C (−60.01%), with an endothermic peak recorded at 292.20 °C. The second stage takes place between 349.47 °C and 601.77 °C, resulting in an additional weight loss of −25.37%. The remaining mass fraction (~15%) represents the thermally stable residue.

Thus, the TGA/DTA results confirm the presence of phases that maintain a certain degree of thermal stability even at elevated temperatures, and indicate that the main decomposition process occurs around 292 °C. However, in our experiments, the thermal analysis of the sample containing polyvinyl chloride

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combined with the mineral filler talc and the flame retardant melamine cyanurate demonstrated significantly improved performance (Figure 4).

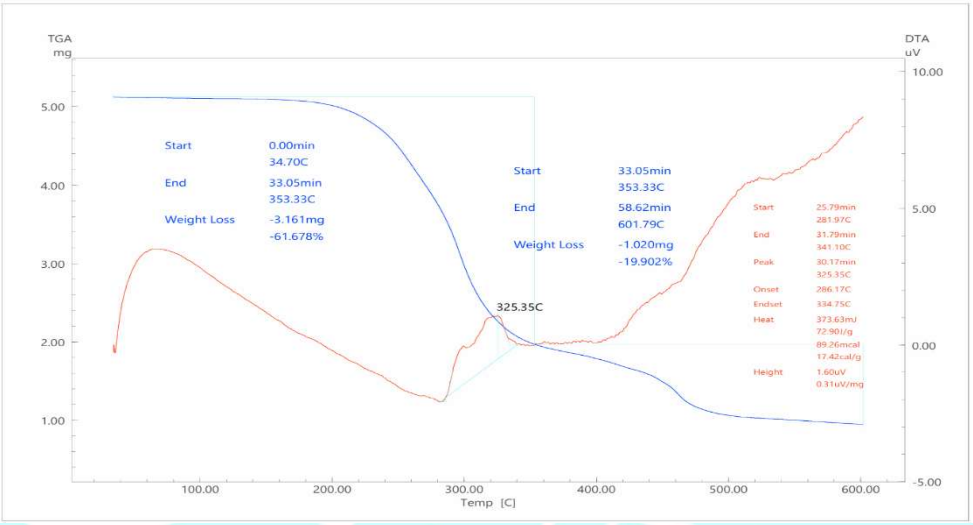


Figure 4. DTA and TGA analysis of the sample obtained from the polyvinyl chloride–melamine cyanurate–talc composite.

In the first stage of analysis, the temperature range of 34.7 °C to 353.3 °C was examined. Within this interval, the sample exhibited a weight loss of 3.161 mg (≈ 61.68%), which corresponds to the release of moisture, adsorbed water, and certain volatile organic components. The DTA curve showed a distinct endothermic peak at 325 °C, indicating the onset of the primary thermal decomposition process.

In the second stage, within the temperature range of 353.3 °C to 601.8 °C, the sample experienced an additional weight loss of 1.020 mg (≈ 19.9%). This phase corresponds to the decomposition of the main polymer backbone or the combustion of the polymer chains. Throughout the overall degradation process, carbonization and oxidation reactions were observed. The total weight loss reached approximately 81.65% (61.68% + 19.97%), while the remaining ~18.35% of the mass represents a thermally stable residue.

A strong endothermic effect was recorded at 323.35 °C on the DTA curve, marking the beginning of the main decomposition event. In the subsequent 400–600 °C range, exothermic processes such as oxidation or combustion may occur. These results confirm that the sample undergoes a two-step thermal degradation: the first around 300 °C with the elimination of unstable polymer segments, and the second at higher temperatures associated with the deeper decomposition and oxidation of carbonaceous residues. The presence of residual mass indicates the existence of inorganic components—such as fillers, catalysts, or oxides—within the material.

The combined TGA/DTA results thus demonstrate that the composite exhibits a two-stage thermal degradation pattern. The first stage (34.7–353.3 °C)

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corresponds to approximately 61.68% weight loss due to volatile release and matrix decomposition, with a pronounced endothermic peak at 353.3 °C indicating the start of major thermal transformation. The second stage (353.3–601.8 °C) accounts for an additional 19.9% loss, reflecting deeper chain degradation and carbonization. Overall, the material lost roughly 81.65% of its total mass, leaving about 18.35% residue that remained stable even at high temperatures, confirming the presence of thermally resistant inorganic phases such as fillers or oxides.

Hence, the TGA/DTA results confirm that certain thermally stable phases persist at elevated temperatures, and that the principal decomposition occurs around 353 °C. The findings further verify the synergistic effect of talc and melamine cyanurate, which enhances the fire-retardant efficiency of the organic composite material.

Conclusion

The synergistic interaction of DOF plasticizer, melamine, melamine cyanurate, and talc within the PVC matrix enabled the development of an efficient fire-retardant composite material. SEM/EDS analyses confirmed the successful incorporation and uniform dispersion of flame-retardant additives in the PVC matrix. Thermal analyses (TGA/DTA) revealed that the PVC–melamine cyanurate–talc composite exhibited the highest thermal stability among all formulations. These composite materials demonstrate strong potential for application in the construction and electrical industries as fire-resistant coatings or insulating materials.

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