ORIGINAL PAPER

CHEMILUMINESCENCE ASSESSMENT OF OXIDATIVE STABILITY IN BARIUM TITANATE–FILLED STYRENE-BASED COMPOSITES FOR MEDICAL APPLICATIONS

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Abstract. *This study evaluates the oxidation states of formulations comprising styrene-butadiene-styrene (SBS), styrene-ethylene-butadiene-styrene (SEBS), and styrene-isoprene-styrene (SIS). The implementation of two chemiluminescence methods, isothermal and nonisothermal, allows depiction of the evolution of oxidation states as a function of the inorganic filler,* $BaTiO_3$*, added at three concentrations: 1, 3, and 5 wt%. The conversion of hydroperoxides into stable oxygenated products reveals the filler's protective activity. This feature, the improvement of durability, recommends the studied formulation as an appropriate product for medical engineering.*

Keywords: *Styrene-based polymers; barium titanate; degradation; chemiluminescence.*

1. INTRODUCTION

The development of polymer-based materials has expanded significantly due to their ability to provide customized properties for demanding applications, including in the medical field [1]. Polymer composites and copolymers allow the modulation of mechanical behavior, chemical stability, and durability through the controlled interaction between the polymer matrix and secondary phases, such as inorganic oxides [2-4], salts [5,6], clays [7], or metal complexes [8]. These materials have found widespread use across various industrial sectors, including electrical engineering [9,10], aerospace engineering [11], flame-retardant systems [12], radiation shielding [13], food packaging [14], and chemical engineering [15]. However, in medical applications, polymeric materials must meet requirements regarding long-term stability, resistance to oxidative degradation, and preservation of functional properties under sterilization and physiological conditions [16-18].

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Styrene block copolymers, such as SBS, SEBS, and SIS, are increasingly considered for medical devices, tubing, flexible components, and elastomeric elements due to their favourable elasticity, processability, and absence of plasticizer migration. However, their performance in medical environments is strongly influenced by oxidative processes, which can lead to chain scission, crosslinking, embrittlement, and ultimately loss of functionality.

Chemiluminescence (CL) is a highly sensitive analytical technique for investigating early-stage oxidation and degradation in polymers, enabling detection of peroxide formation and radical reactions long before macroscopic changes become apparent. Consequently, chemiluminescence studies provide valuable insights into the oxidative stability and ageing behaviour of styrenic copolymers, essential for assessing their suitability and reliability in medical applications. In this context, the present study focuses on the comparative chemiluminescence behavior of SBS, SEBS, and SIS copolymers, intending to contribute to a better understanding of their degradation mechanisms relevant to medical-grade polymeric materials. Special attention is given to the applications of nanocomposites for drug delivery [19], which enable efficient drug administration, as well as to the preparation of hydrogels for medication [20, 21].

The choice of filler type poses serious problems regarding phase compatibility [22]. This shortcoming is always overcome by integrating a filler into the polymer matrix that strongly interacts with the inorganic material. Silver nanoparticles [23] and nanocarbon systems [24] used in tissue engineering serve as reference examples that demonstrate the versatility of these materials. Among inorganic fillers that transform the basic polymer into high-performance materials, BaTiO3 receives considerable attention due to its favourable compatibility with various polymers [25]. In this context, the crystals of barium titanate become an appropriate additive for the energy storage [26,27], the fabrication of piezoelectrical items [28], the preparation of anticorrosion protections [29], the production of thermistors [30] and solar cells [31], and thermal protection of polymers against oxidation [32].

Due to the tailoring of functional properties with increasing filler concentration and the good dispersibility of barium titanate ($BaTiO_3$) powders in polymer matrices, $BaTiO_3$-filled polymers have attracted considerable interest for medical applications. These include the fabrication of medical scaffolds [33,34], dental materials [35,36], tissue regeneration systems [37], prosthetic components [38,39], and broader biomedical applications [40,41]. In addition, polymer composites containing $BaTiO_3$ in concentrations typically between 5 and 25 wt% have been reported to exhibit antimicrobial activity, further increasing their relevance for medical applications [42]. From an engineering perspective, the functionality of $BaTiO_3$ as a reactive filler in polymer systems is closely associated with its surface chemistry, which allows interactions with the environment. The formation of interfacial bridges between the oxygen atoms on the $BaTiO_3$ surface and the protons in the polymer chain facilitates interfacial coupling, influencing the relaxation behavior and overall stability of $BaTiO_3$-containing materials [43]. This biomimetic feature, which underlies $BaTiO_3$'s suitability for medical engineering applications, also governs its interaction with the host polymer matrix [44].

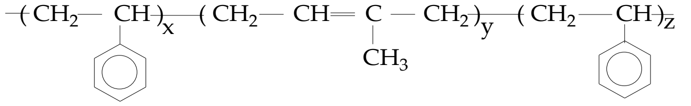
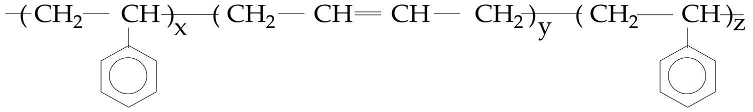
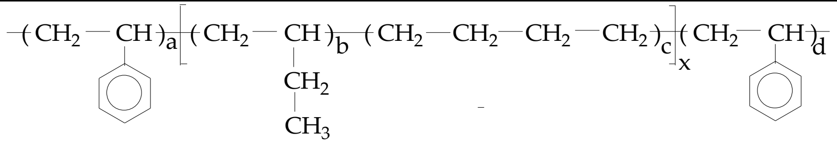
In this context, the present study aims to highlight the role of the $BaTiO_3$ structure in improving the oxidative stability of polymeric materials. By contributing to oxidative protection, $BaTiO_3$ can significantly improve the durability of materials and support the expansion of polymeric applications in environments where long-term stability and effective stabilization are critical requirements.

2. MATERIALS AND METHODS

2.1. MATERIALS

The studied polymers (Table 1) are characterized by the presence of benzene nuclei, which confer a certain radiation stability because of their delocalization of π electrons. SBS was purchased as two types of copolymers with different butadiene/styrene ratios (S3B7 and S4B6, which reflect the different ratios in the material composition), Globalprene™ 3546 and 3501, from LCY Grit Corporation, Taiwan, with different styrene contents. SEBS was manufactured by Globalprene (China) as the 7550-type GLOBALPRENE™, with a linear backbone. Styrene-isoprene-styrene triblock copolymer (SIS) was provided by KRATON, USA, as Kraton D1119 PT, a dusted dense pellet material. All their structures are similar.

Table 1. Structures of the investigated styrene block copolymers

Styrene-isoprene-styrene (SIS)	
Styrene-butadiene-styrene (SBS)	
Styrene-ethylene-butadiene-styrene (SEBS)	

The filler, $BaTiO_3$ powder, and chloroform were bought from Thermo Fisher Scientific (Massachusetts, USA) as a pro analysis grade. Before adding polymers, it was dried for 1 h at 105°C to remove water. The raw materials were not subjected to any modifications or purification before their use as study materials.

2.2. METHODS

The samples were prepared by vigorous shaking of polymer/filler suspension in chloroform containing the corresponding particle loadings (1 wt%, 3 wt%, and 5 wt%), followed by purging 20 μL in aluminium trays. After drying at room temperature to remove the solvent, the resulting films were subjected to CL measurements.

The chemiluminescence measurements were carried out using the Lumipol 3 spectrometer produced by the Polymer Institute of the Slovak Academy of Sciences (Bratislava). For the nonisothermal determinations, a moderate heating rate of 10°C·min^{-1} was applied, while the isothermal investigation was conducted within the temperature range of 140°C to 210°C to correlate polymer structure with the oxidation strength of each material. The measurements were performed immediately after irradiation to avoid decay of short-lived radicals.

3. RESULTS AND DISCUSSION

3.1. RESULTS

The polymer degradation occurs due to the attack of diffused molecular oxygen in the inner zones of the samples. The most vulnerable sites in the studied molecules are tertiary carbon atoms, double bonds, and chain-end protons [45]. As previously reported, the surface and lattice defects act as traps that generate radicals [46]. The formation of bridges between titanate particles and polymer chains contributes to the delay of oxidation.

In Fig. 1, the contribution of filles in different concentrations is presented. The differences that are revealed characterize the improvement of material stability due to the interaction with the filler. The greatest amelioration of stability is illustrated by the extension of the onset oxidation temperature of SEBS specimens. The other materials seem to be slightly protected by barium titanate at lower temperatures up to 200 °C. Among these polymers, the most vulnerable are SBS materials, whose structures lack a methyl screening effect on the double bond in the median component, as occurs in SIS. At temperatures exceeding 200 °C, stability is governed by structural peculiarities. While SEBS consists of saturated chains, the other three materials have a double bond, whose low scission energy induces their degradation temperature.

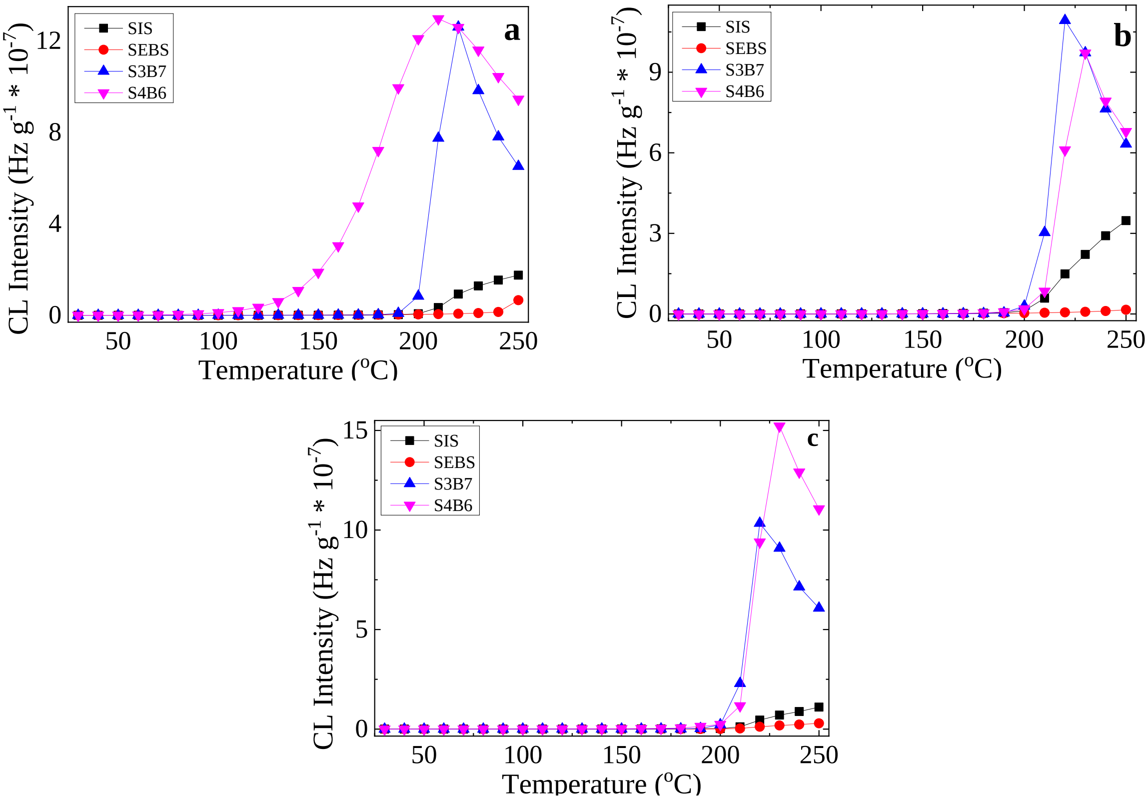


Figure 1. Nonisothermal CL spectra recorded on some polymers containing benzene nuclei (heating rate: 10°C·min^{-1}). Filler loadings: (a) 1 wt%, (b) 3 wt%, (c) 5 wt%.

The dependence of the polymer support on filler concentration, as highlighted by nonisothermal chemiluminescence measurements, is shown in Fig. 2. Evidence of differences in the effects of filler concentration is evident in the main kinetic parameter, the onset oxidation temperature. The first insight into the degradation progress at low loading (1 wt%)

reveals a correlation between OOT values and the oxidation strength of materials. Under these circumstances, the protection efficiency ranking places the investigated polymers in the following order:

SIS < SBS (S3B7) < SBS (S4B6) < SEBS

At temperatures exceeding 200°C, higher filler loadings (3 wt% and 5 wt%) exhibit only minor differences in the measured chemiluminescence (CL) intensities. This suggests that a concentration of 3 wt% is optimal, as it provides comparable oxidative behavior while inducing only minimal changes in the mechanical properties of the composites. A comparison of the intensity scales further shows that SBS-based materials emit more intense CL signals, indicating a lower thermal stability of these polymers.

Fig. 3 presents the isothermal chemiluminescence spectra, which describe the progression of oxidative degradation under steady environmental conditions. Unlike non-isothermal measurements, isothermal CL data reflect oxidation kinetics rather than the availability of thermal energy, enabling the identification of degradation trends relevant to specific application ranges. For medical applications, these materials demonstrate adequate thermal stability for safe use in devices and equipment operating at temperatures below 150°C.

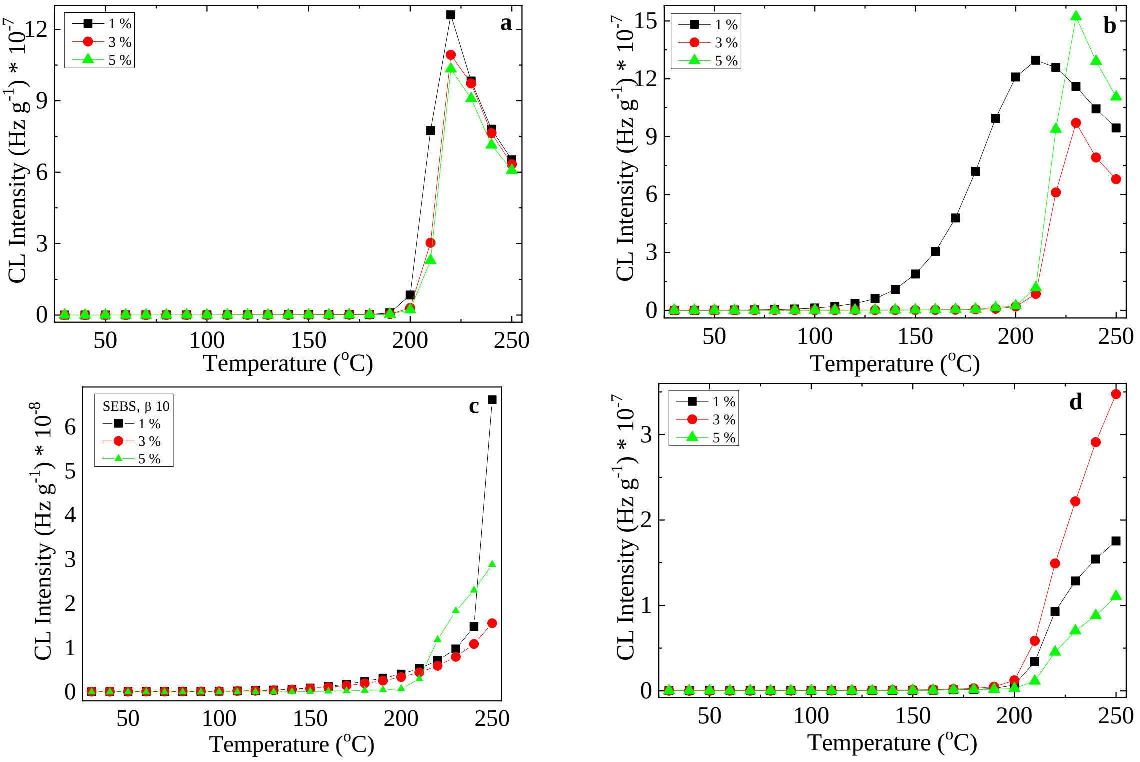


Figure 2. Nonisothermal CL spectra recorded on each studied composite with polymers containing benzene nuclei (heating rate: 5 °C min^{-1}). Polymer material: (a)SBS (S3B7), (b)SBS (S4B8), (c)SEBS, (d) SIS.

The distinct behavior of SIS indicates that this polymer may undergo partial chain scission during storage; however, upon short-term heating, the filler acts as an effective stabilizing agent, and the degradation process proceeds in a controlled manner. Overall, the CL results highlight the significant effectiveness of the filler in delaying oxidative degradation in the investigated polymers.

The direct comparison of the recorded CL spectra allows us to remark on the unusual behavior of SIS. It means this material impedes oxidation. The close vicinity of two neighbouring benzene nuclei that frame a methyl moiety and a double bond belonging to the isoprene fragment promotes a certain degree of stability. However, the most resistant material from the studied group of polymers is SEBS. It shows the oxidation progress at the higher temperature of 210°C. Its structure is characterized by the high content of saturation as a linear sequence of six carbon atoms. On the other hand, the separation between the curves depicting the contributions of various filler concentrations is greater for SBS (S3B7) and SEBS, indicating that interactions between the two phases are more pronounced. The most recommended system, based on the delayed oxidative degradation achieved with $BaTiO_3$ powder, is SEBS containing 5% filler. As shown in Fig. 3D, the propagation stage takes the longest time, and the 5 wt% concentration offers the best protection against material ageing.

Because the essential contribution to the preservation of material integrity by the efficient delay of oxidation, the addition of this barium titanate makes possible to extend the application ranges of studied polymers from the medical purposes (scaffolds, prostheses, drug delivery materials, dental supports, patches) over the special conditions of nuclear engineering, aeronautics, geothermal protection during seismic events and many complex situations.

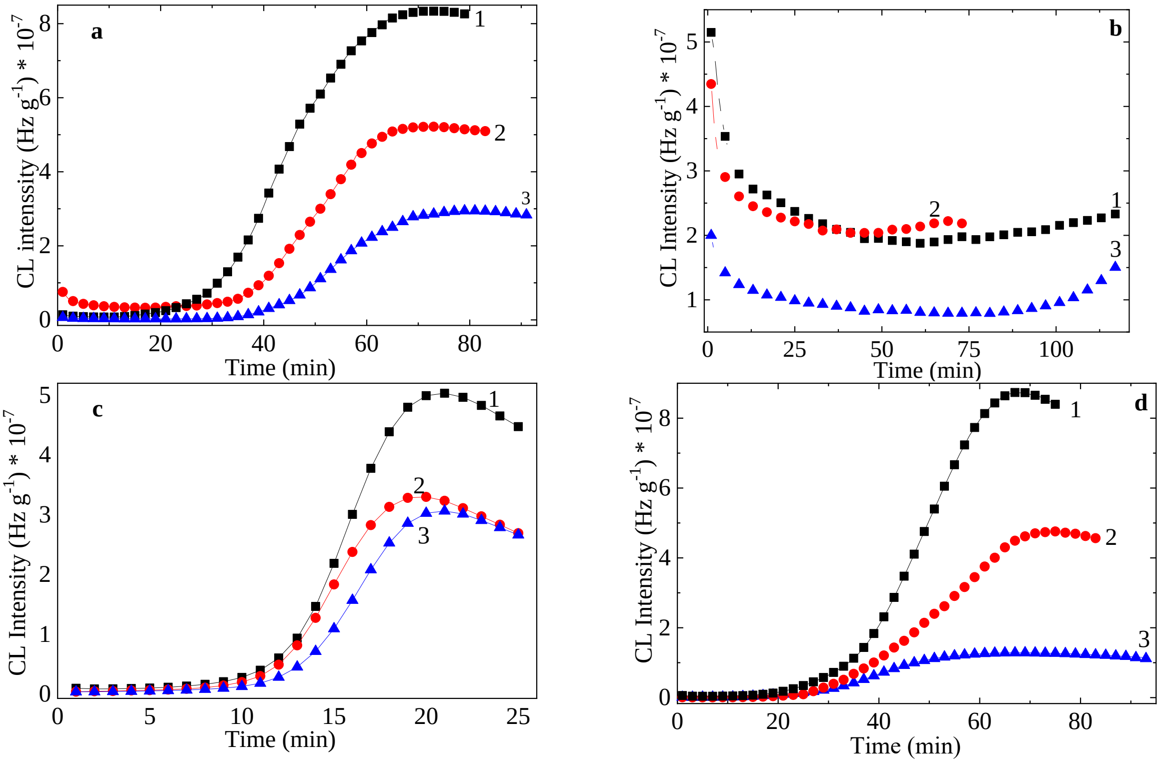


Figure 3. Isothermal CL spectra recorded on some polymers containing benzene nuclei. Testing temperatures: (a) 140°C, (b) 170°C, (c) 150°C, (d) 210°C. Polymer material: (a) SBS (S3B7), (b) SIS, (c) SBS (S4B8), (d) SEBS. Filler concentrations: (1) 1 wt %, (2) 3 wt%, (3) 5 wt%.

3.2. DISCUSSION

A comparative study of the thermal resistance of polymers using chemiluminescence analysis was conducted. The performance of block polymers containing benzene nuclei in the backbone units is closely related to the diffusion of oxygen into the inner layers, which also depends on the molecular structure [47]. The achievement of thermal strength depends on the decay rates of the intermediate hydroperoxides [48], which determine the trends in oxidation ageing. All the studied polymers containing a median unit as hydrocarbon moieties model the degradation due to their involvement in the scission process [49]. This mode of depreciation is supported by the participation of molecular fragments derived from centred–carbon radicals resulting from the scission of vicinal bonds in benzene components [50]. The reactivity of styrene block polymers is systematically used to recover polymer waste, owing to the ability of aged materials to reintegrate fragments into rigid structures [51]. However, the failure of long-term stability is determined by the direct formation of microstructural islands in the material morphology, which contribute to stress induced by different environmental factors. The matrices are rather degraded than plasticized, affecting the durability and the functional properties [52].

The mechanism of oxidation for the styrene block copolymers was previously analyzed in detail [53]. The preliminary stage initiating oxidation involves the formation of peroxy units via proton removal at the α-position relative to the double bond in the central hydrocarbon segment. Then, the chain mechanism illustrated by the Bolland and Gee scheme is developed [54]. The bonding strength of these protons determines the lifetime of materials that are thermally driven in the foreseeable kinetic model [55]. The confidence in this assumption is based on the trends observed in the present chemiluminescence measurements. The CL approach confirms the stabilization effects of fillers in the studied composites with increasing additive loadings, starting from the ability of some spots on the particle surfaces to act as the traps of radicals. The CL investigations on the SEBS samples subjected to thermal degradation [48] reveal the involvement of peroxy structures resulting from the attachment of oxygen from the diffused stream. This mechanistic view opens the way for stabilization, by which these radicals are trapped in the lattice defects [46].

The comparison of material stabilities, with polystyrene as a basic fraction of block copolymers, allowed us to emphasise the importance of median segments that confer a relative capacity to resist the attack of diffused oxygen during thermal treatment in environmental applications [56, 57]. The availability of SBS for the generation of radicals' presumable intermediates in the binding of asphaltous layers [58], is justified by the possibility of an easier fragmentation of the butadiene moieties that bridge the segments of polystyrene. There have been several studies in which these styrene block copolymers serve as binding components to enhance the functional characteristics of basic materials, including polypropylene [59], EPDM [60], polystyrene [61], polylactic acid [62], silicone rubber [63], and many others. This availability demonstrates that these block copolymers may serve as a radical source for polymer recycling, enhance the functional characteristics of minor polymer components, and be used as a binding agent in the manufacture of high-performance materials for special purposes.

4. CONCLUSIONS

The present study demonstrates the beneficial contribution of barium titanate to improving the thermal stability of styrene block copolymers. The chemiluminescence investigations reveal the involvement of the particle surface as the concentration of $BaTiO_3$ filler increases. The outer layers of the inorganic lattice act as scavengers for free radicals generated by heating. The presence of double bonds and substituted carbon atoms in the structure of the studied polymers explains their susceptibility, especially at temperatures above 150°C. However, the presence of ethylene in the molecular chains of SEBS confers greater stability, while the isoprene configuration induces a slight increase in thermal resistance in composite systems. The applications as binder agents in several polymer blends may be justified by the stabilization effect of benzene nuclei and the scission of median moieties, isoprene or butadiene. The presence of barium titanate enables intimate interaction between the polymer phase and the inorganic content. At the boundary limits created by the outer layers of crystalline filler, the penetration of radicals into the lattice holes removes them from the degradation chain. Accordingly, this method of stability improvement may be extended to other polymers whose oxidative degradation proceeds via a radical mechanism. The assistance of this filler is based on electronic interactions, in which unpaired electrons from the surrounding radicals fill the holes, where the electron deficit provides the opportunity for joining. The studied composites offer versatile routes for the recovery of polymer waste and for the initiation of new compositions comprising an organic component, polymer materials, and an inorganic phase that can engage in the degradation chain. This innovative idea enables the preparation of various polymer composite formulations that help maintain processing and equipment in good condition for extended periods. The presented results are confident for several new medical interventions, such as restorations.

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