Journal of Sustainable Construction Materials and Technologies

Volume 11 | Issue 2 Article 5

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JOURNAL OF SUSTAINABLE CONSTRUCTION MATERIALS AND TECHNOLOGIES 2026;11:278–288

RESEARCH ARTICLE

Moisture-Induced Degradation and Mechanical Durability of Mineral-Filled Polylactide (PLA) Composites

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ABSTRACT

This study investigates the influence of mineral fillers on the hydrolytic stability and mechanical durability of polylactide (PLA) composites under accelerated aqueous ageing conditions. Composites containing halloysite nanotubes (HNT) and bentonite-based fillers (BM, BS, BSP) at 2 wt.% were prepared using a powder-coating and injection moulding approach. Accelerated hydrolytic degradation was conducted at 50 ˚C for up to six weeks. The degradation behaviour was evaluated through mass variation, water absorption, incubation medium analysis (pH and electrical conductivity), mechanical testing, surface observations, and contact angle measurements. The results revealed a clear filler-dependent response. Bentonite-filled composites exhibited increased water absorption and accelerated mass loss, which may be associated with the hydrophilic and layered structure of smectite clays facilitating moisture transport. In contrast, halloysite-filled composites showed comparatively reduced water uptake and improved short-term mechanical performance. Analysis of incubation media indicated decreasing pH and increasing conductivity, reflecting the formation of acidic degradation products and the release of ionic species. A qualitative correlation between moisture uptake and deterioration of mechanical properties was observed, particularly for bentonite-containing systems. Microstructural observations demonstrated the presence of surface roughening, microcracks, and heterogeneous degradation features, which may be linked to non-uniform filler dispersion and localised stress concentrations. Contact angle measurements confirmed ageing-induced surface hydrophilisation, although these results should be treated as indirect indicators of surface physicochemical changes. Overall, the results suggest that filler morphology, in combination with hydrophilicity and dispersion state, influences moisture transport and degradation behaviour in PLA composites. The findings contribute to understanding structure–property–degradation relationships in PLA-based systems and indicate that mineral filler selection may be considered as a strategy for tailoring material performance in moisture-exposed applications.

Keywords: Polylactide (PLA), Mineral-filled composites, Moisture-induced degradation, Halloysite nanotubes (HNT), Bentonite clays, Water absorption, Accelerated ageing, Mechanical durability, Wettability, Sustainable materials

1. Introduction

Polylactide (PLA) is a bio-based, industrially compostable polyester widely applied in sustainable material systems due to its favourable stiffness, processability, and reduced environmental footprint [1, 2]. Despite these advantages, the long-term durability of PLA remains limited by its susceptibility to hydrolytic degradation, particularly under elevated temperature and moisture conditions [2–5]. Exposure to water induces chain scission, molecular weight reduction, and progressive deterioration of mechanical performance, thereby restricting the applicability of PLA in moisture-exposed environments [3]. The hydrolytic degradation behaviour of PLA is governed by multiple factors, including temperature, molecular architecture, and phase morphology. Recent studies emphasise

Received 16 February 2026; revised 5 June 2026; accepted 5 June 2026.
Available online 11 June 2026

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https://doi.org/10.29187/2458-973X.1221

the importance of the three-phase structural model of PLA, consisting of mobile amorphous fractions (MAF), rigid amorphous fractions (RAF), and crystalline domains [6, 7]. Above the glass transition temperature (Tg), water-induced plasticisation and morphological reorganisation may accelerate degradation processes, while below Tg degradation kinetics are strongly influenced by the distribution and stability of RAF regions [6, 7]. Consequently, materials with comparable crystallinity may exhibit markedly different hydrolytic responses depending on their microstructural characteristics. Incorporation of mineral fillers represents an established strategy for modifying the mechanical performance and environmental stability of PLA [4, 8–12]. Clay minerals such as bentonites (smectites) and halloysite nanotubes (HNT) are particularly attractive due to their high surface area and reinforcement potential [8, 9, 13]. However, their influence on hydrolytic degradation is complex and depends on several interrelated filler characteristics, including morphology, hydrophilicity, specific surface area, and dispersion state within the polymer matrix. Layered smectite clays, which are inherently hydrophilic, may facilitate moisture transport through interlayer galleries and contribute to accelerated degradation processes. In contrast, halloysite nanotubes, characterised by a tubular morphology, may influence diffusion pathways and interfacial interactions, potentially modifying moisture transport efficiency. Understanding filler-controlled degradation mechanisms is particularly relevant for sustainable engineering applications, including construction-related polymer components and additively manufactured structures, where materials are frequently exposed to moisture and temperature variations [14–16]. In such systems, resistance to moisture-induced degradation becomes a critical durability factor. However, it should be emphasised that the individual contributions of filler morphology and chemical composition are difficult to fully decouple, as these parameters are inherently interrelated in mineral systems such as bentonite and halloysite. Therefore, conclusions regarding the dominant role of morphology should be interpreted with caution. Therefore, understanding the hydrolytic stability of mineral-filled PLA composites is essential from a materials performance and durability perspective. The present study investigates the influence of selected mineral fillers—halloysite nanotubes and bentonites—on the hydrolytic stability of PLA composites, with particular attention to the role of filler morphology, dispersion behaviour, and moisture interactions. The work focuses on correlating mass variation, water absorption, incubation media evolution, and tensile behaviour to elucidate filler-dependent degradation responses. It should be noted that the scope of this study is limited to a single PLA grade and a fixed filler content of 2 wt.%. Additionally, the applied ageing conditions (50 °C in aqueous environment) represent an accelerated regime, which may not fully reproduce natural exposure conditions. Therefore, the results should be interpreted within the context of the investigated systems.

2. Materials and methods

2.1. Materials

Polylactide (PLA) Ingeo™ 3251D (NatureWorks LLC, USA), a commercial injection-moulding grade widely applied in engineering and sustainable material systems, was employed as the polymer matrix [17].

The dispersed phase incorporated into the PLA matrix consisted of the following mineral fillers:

- **Halloysite nanotubes (HNT)** – tubular aluminosilicate nanofiller with typical dimensions of 30–70 nm in diameter and 1–3 μm in length (Sigma-Aldrich). Halloysite is commonly used as a reinforcing nanofiller and is characterised by low electrical and thermal conductivity [8, 11].
- **Modified bentonite (BM)** – hydrophobically modified smectite clay treated with a quaternary ammonium salt and a hydrophobic polymer, affecting interfacial interactions and moisture-related behaviour [9, 10].
- **Bentonite (BS)** – Wyoming-type bentonite (BOREGEL S130, Polroc I), consisting predominantly of montmorillonite (75–85 wt.%), representing a layered smectite structure [8, 9].
- **Bentonite–polymer system (BSP)** – modified Wyoming-type bentonite (BOREGEL S130 PLUS), based on activated sodium bentonite with high montmorillonite content [8, 9].

The selected fillers differ not only in morphology (tubular vs layered), but also in hydrophilicity, surface characteristics, and dispersion behaviour, which may influence moisture transport and degradation processes. The filler content was maintained at 2 wt.% for all composite formulations. Prior to processing, PLA granules and mineral fillers were dried at 60–80 °C for a minimum of 12 h to minimise residual moisture, as recommended for hydrolysis-sensitive polyesters [2, 3].

2.2. Composite preparation

PLA composites were prepared using a two-step powder-coating and melt-processing approach. In the first stage, PLA granules were mechanically dry-coated with mineral filler powders (a “dusting” or “powdering” process), resulting in the formation of a thin layer of filler particles on the surface of polymer granules. This preliminary treatment enabled initial distribution of the filler phase but did not ensure homogeneous dispersion within the polymer matrix. In the second stage, the dry-coated granules were processed via injection moulding. Homogenisation of the composite was achieved within the screw-based plastification unit. The relatively long screw geometry and associated shear forces promoted effective mixing, dispersion of filler particles, and their incorporation into the molten PLA matrix. Therefore, although initial attachment of filler occurred during dry mixing, the key dispersion and development of interfacial interactions took place under melt-processing conditions inside the injection moulding system. All materials were processed via injection moulding using a MULTIPLAS V4 S 15N vertical injection moulding machine. Processing temperatures were maintained within the range 165–167 ˚C, while the mould temperature was controlled between 25 and 40 ˚C. Standard tensile specimens were produced in accordance with ISO 527. Preliminary microstructural observations indicated that the dispersion state depended on filler type. Systems containing halloysite and modified bentonite exhibited a tendency toward agglomeration, whereas more uniform dispersion was observed for selected bentonite systems. These differences were considered in the interpretation of degradation behaviour.

2.3. Accelerated hydrolytic ageing

Hydrolytic degradation was evaluated under accelerated aqueous conditions. Specimens were fully immersed in distilled water and incubated at 50 ± 1 ˚C for up to six weeks. The selected temperature (50 ˚C) represents an accelerated ageing condition, chosen to intensify hydrolytic processes and enable observation of degradation effects within a reasonable experimental timeframe. Ageing was conducted over a period of up to six weeks, with intermediate evaluation points after one and two weeks. The incubation medium was periodically replaced to maintain stable degradation conditions. It should be noted that ageing at elevated temperature may induce degradation mechanisms that differ from those occurring under natural environmental conditions, particularly due to increased molecular mobility and enhanced water diffusion above or near the glass transition temperature of PLA. Therefore, the results obtained in this study should be interpreted as indicative of accelerated trends rather than a direct representation of long-term behaviour under service conditions. The applied conditions are commonly used in studies on hydrolytic degradation of aliphatic polyesters to accelerate water uptake and chain scission processes.

2.4. Mass variation and water absorption

Mass variation was determined gravimetrically at each ageing interval. Specimens were weighed immediately after removal from the incubation medium (wet mass), followed by drying to constant mass (dry mass). Water absorption was calculated in accordance with ISO 62 [19], based on wet and dry mass differences.

2.5. Incubation media analysis

The evolution of degradation processes was monitored via measurements of pH and electrical conductivity of the incubation medium. pH measurements were performed using an Elmetron pH meter equipped with a glass electrode (calibrated at pH 4.00 and 7.00). Electrical conductivity was measured using an Elmetron conductometer. These parameters were used as indirect indicators of hydrolytic degradation and ion release during ageing.

2.6. Mechanical testing

Tensile properties were determined according to ISO 527 using a Zwick 1435 universal testing machine. The crosshead speed was set to 2 mm/min. The following parameters were evaluated: Young’s modulus (E), tensile strength (Rm), and elongation at break (ϵb). A minimum of five specimens was tested for each material condition. Mechanical testing after longer ageing intervals was not feasible. Specimens subjected to extended hydrolytic incubation exhibited pronounced deformation and dimensional instability, preventing reliable mounting in the testing grips. These observations indicate significant hydrolytic degradation and plasticisation effects occurring at later ageing stages. Consequently, tensile properties are reported for the initial state and after one week of ageing.

2.7. Microstructural and surface observations

Macroscopic surface changes following hydrolytic ageing were examined using a SteREO Discovery.

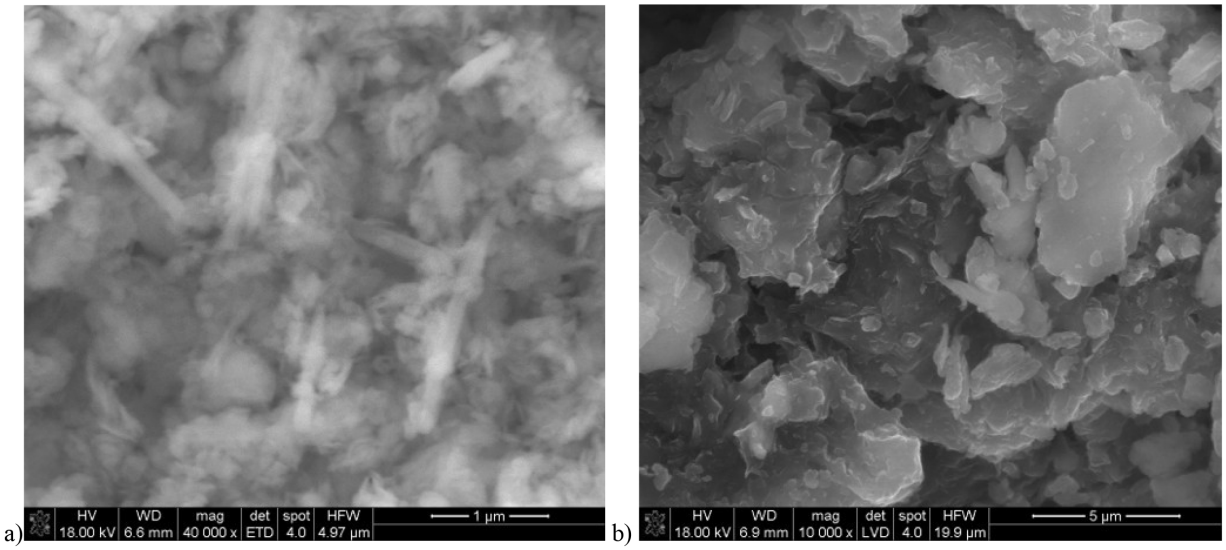

Fig. 1. SEM images of mineral fillers: (a) halloysite nanotubes (HNT), showing tubular morphology and partial agglomeration; (b) bentonite particles exhibiting layered, plate-like structure.

V8 stereomicroscope (ZEISS, Germany). The observations were conducted to identify degradation-induced surface defects, cracking phenomena, and morphological irregularities associated with moisture exposure. Additionally, the morphology of mineral fillers and composite surfaces was analysed using scanning electron microscopy (SEM). The observations were carried out using a FEI Nova NanoSEM 200 scanning electron microscope operated at an accelerating voltage of 18 kV. Both secondary electron (ETD) and low-vacuum detectors were used depending on the analysed sample and magnification conditions. The SEM investigations were performed to evaluate particle morphology, size, dispersion behaviour, and the presence of agglomerates, as well as to assess microstructural changes induced by hydrolytic degradation.

2.8. Surface wettability

Surface wettability was evaluated by contact angle measurements using the sessile drop method with a DSA10 Mk2 system (Krüss GmbH, Germany). A distilled water droplet (0.2 μL) was deposited on the specimen surface, and the contact angle (θ) was determined by optical image analysis. Measurements were performed at room temperature. For each material condition, ten independent measurements were conducted to ensure statistical reliability. The contact angle measurements provide indirect information on surface physicochemical changes induced by hydrolytic ageing.

3. Results and discussion

3.1. Morphology of mineral fillers

Representative SEM images of the applied mineral fillers are presented in Fig. 1.

Halloysite nanotubes (HNT) exhibit a characteristic tubular morphology, with elongated structures in the micrometre length range and nanometre-scale diameters. The observed morphology is consistent with literature descriptions of halloysite as a naturally occurring aluminosilicate with anisotropic geometry. Partial agglomeration of nanotubes is also visible, which may influence dispersion behaviour within the polymer matrix. In contrast, bentonite particles display a predominantly layered, plate-like morphology typical of smectite clays. The particles form stacked and irregular aggregates composed of thin lamellae, creating a hierarchical structure with a high specific surface area. Such morphology is associated with the presence of interlayer galleries, which can act as pathways for moisture transport. The pronounced differences in particle geometry between the tubular halloysite and layered bentonite are expected to influence both dispersion behaviour and transport phenomena in PLA composites. In particular, the layered structure of bentonite may facilitate moisture diffusion, whereas the tubular morphology of halloysite may increase diffusion tortuosity and disrupt continuous transport pathways. Additionally, the degree of agglomeration observed for both filler types suggests that dispersion in the polymer matrix may not be fully uniform, which may further affect local

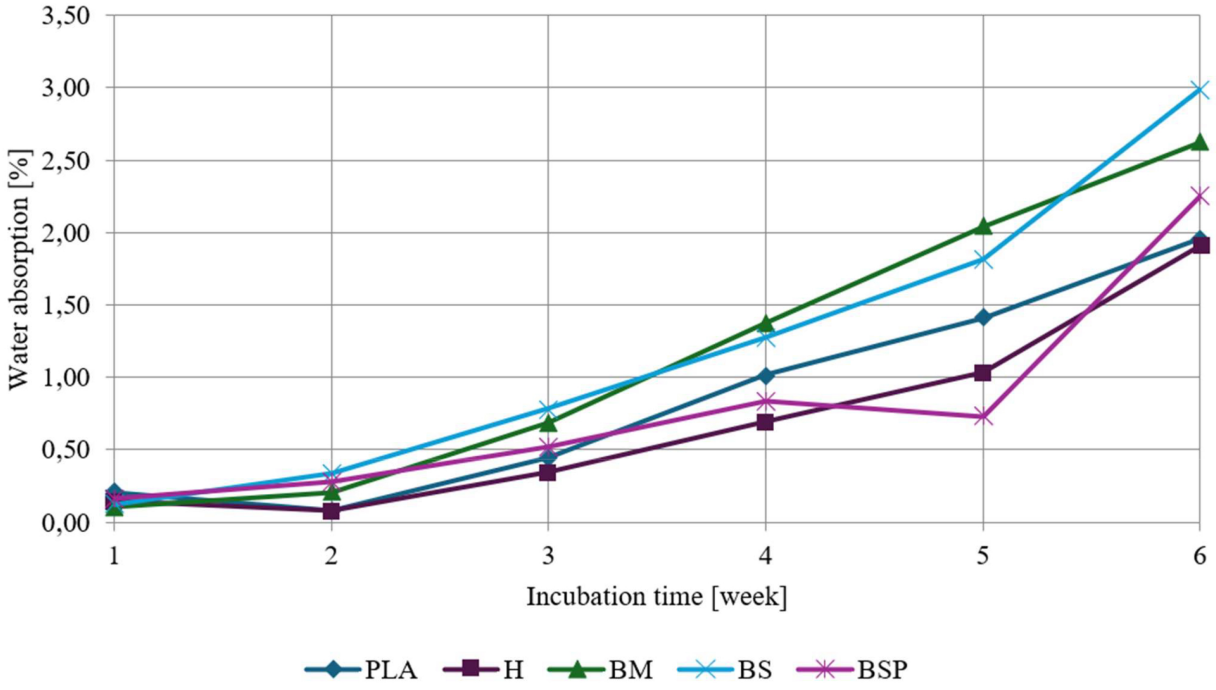

Fig. 2. Water absorption behaviour of neat PLA and mineral-filled PLA composites during hydrolytic ageing at 50 °C.

transport processes and degradation behaviour. These aspects are discussed in relation to hydrolytic degradation in the following sections.

3.2. Mass variation and water absorption

All investigated materials exhibited a characteristic two-stage response during hydrolytic ageing. An initial increase in specimen mass was observed, corresponding to progressive water absorption (Fig. 2), followed by a reduction in mass at longer ageing intervals (Fig. 3). This behaviour is consistent with hydrolytic degradation mechanisms reported for PLA, where water uptake precedes chain scission and the subsequent leaching of soluble oligomeric degradation products [2, 3].

A pronounced filler-dependent effect was evident. Bentonite-filled composites displayed significantly higher water absorption compared to neat PLA and halloysite-filled systems (Fig. 2). This behaviour may be related to the hydrophilic nature and layered morphology of smectite clays, which can facilitate moisture transport through interlayer galleries. Additionally, differences in filler dispersion and the presence of particle agglomerates may further influence local water diffusion pathways and moisture uptake. Enhanced water ingress **may promote** hydrolytic cleavage of ester bonds, resulting in accelerated mass loss at extended ageing times (Fig. 3). Similar behaviour has been widely reported for PLA/clay systems. Paul et al. [11] demonstrated that montmorillonite-containing PLA nanocomposites exhibit accelerated hydrolytic degradation due to facilitated moisture diffusion through clay galleries. Comparable observations were reported by Rapacz-Kmita et al. [6, 9], who showed that smectite fillers significantly modify degradation kinetics depending on clay chemistry and interfacial interactions. In contrast, halloysite-filled composites showed comparatively reduced water uptake (Fig. 2). The tubular morphology of halloysite nanotubes may contribute to moderated moisture transport by increasing diffusion tortuosity and disrupting continuous diffusion pathways. However, this effect may also depend on the degree of dispersion and potential agglomeration of the nanotubes within the PLA matrix. Overall, the results suggest that moisture transport and degradation behaviour are influenced by a combination of filler morphology, hydrophilicity, and dispersion state, rather than by a single dominant factor.

3.3. Evolution of incubation media (pH and conductivity)

Changes in the incubation medium provided indirect insight into degradation kinetics. A decrease in pH was detected during the early ageing stages (Fig. 4), indicating the formation of acidic degradation products, primarily lactic acid and low-molecular-weight oligomers. This trend is

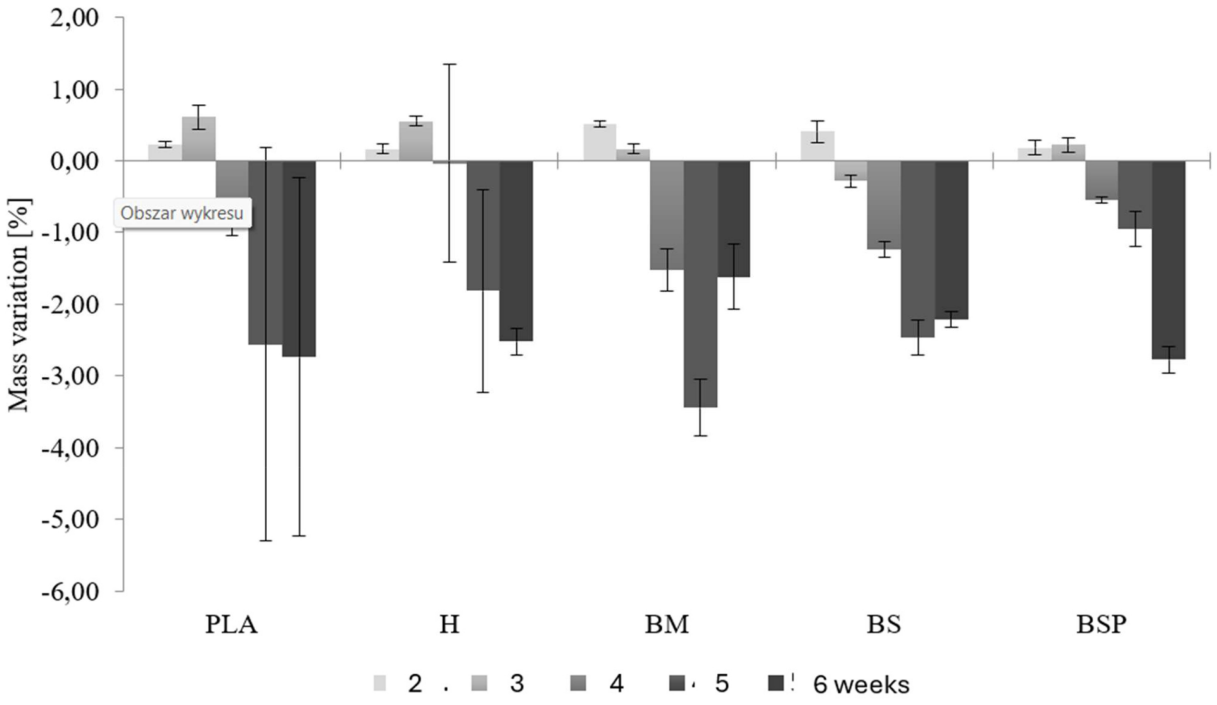

Fig. 3. Mass variation of PLA and PLA composites as a function of hydrolytic ageing time.

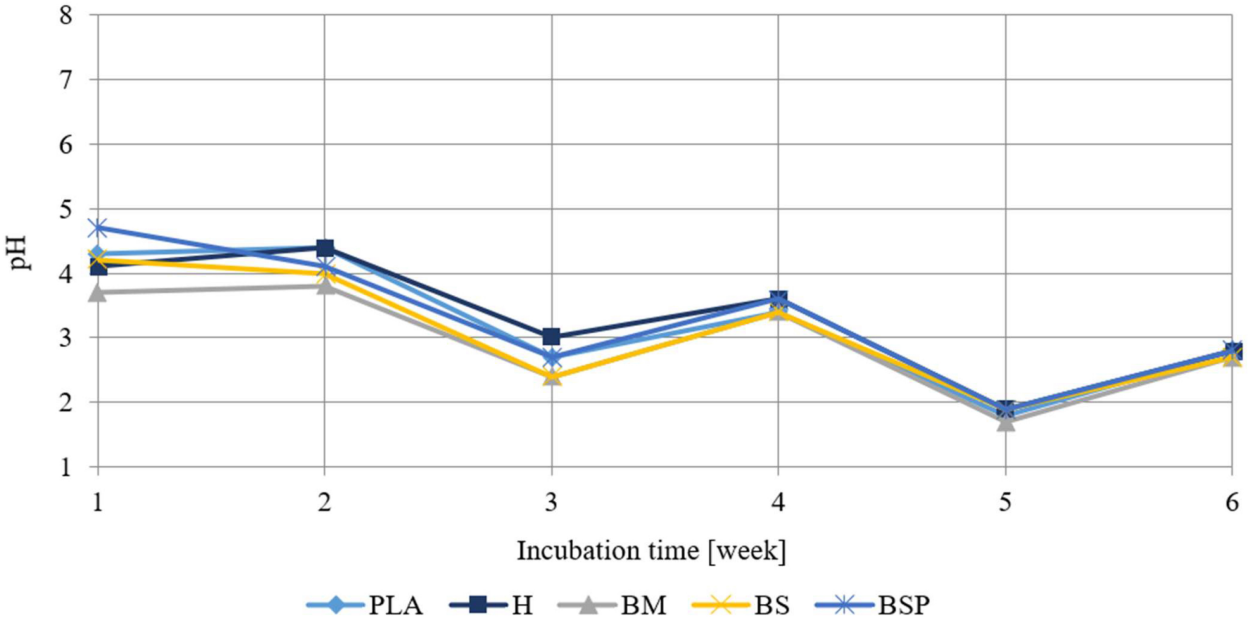

Fig. 4. Evolution of incubation medium pH during accelerated hydrolytic degradation.

characteristic of hydrolytically degrading aliphatic polyesters [2, 16].

Simultaneously, electrical conductivity increased with ageing time (Fig. 5). The conductivity evolution may reflect the combined contribution of soluble degradation products and ionic species released from mineral fillers. Bentonite-containing systems exhibited a more pronounced conductivity rise (Fig. 5), which may suggest intensified degradation and/or increased ion exchange processes associated with smectite clays.

The coupled pH and conductivity responses confirm that mineral fillers influence not only water transport but also the chemical environment governing degradation reactions. The observed pH decrease is consistent with classical autocatalytic hydrolysis mechanisms of PLA, where lactic acid formation accelerates chain scission [2, 3]. Similar incubation

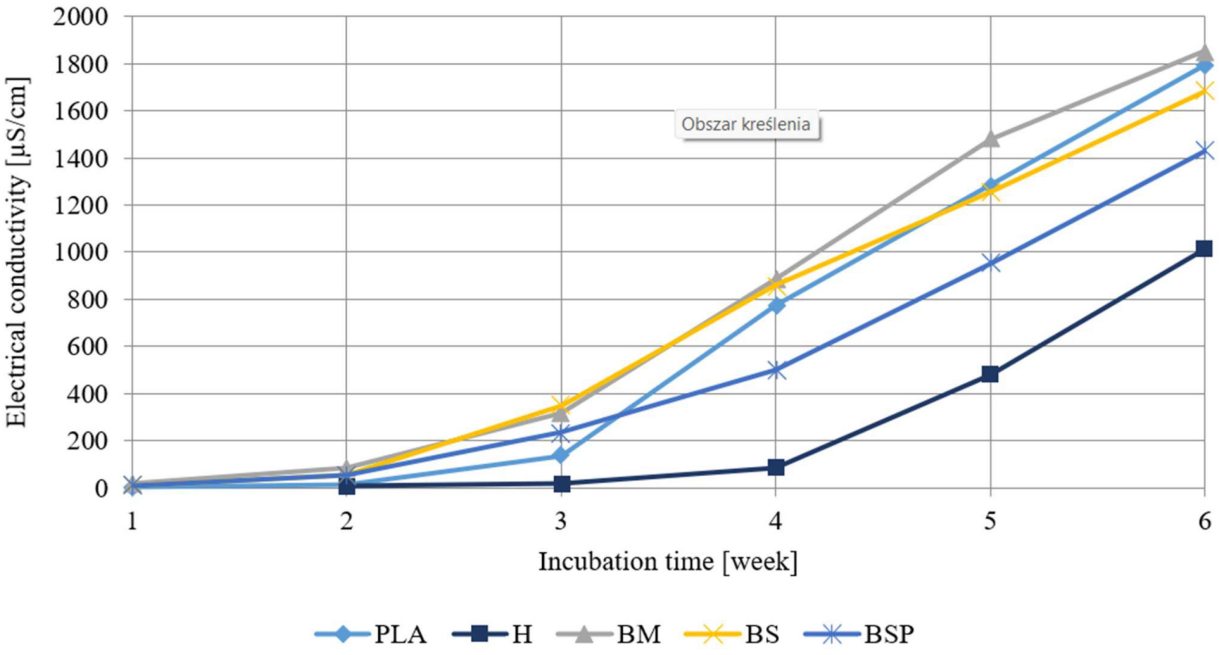

Fig. 5. Electrical conductivity changes of incubation media during hydrolytic ageing.

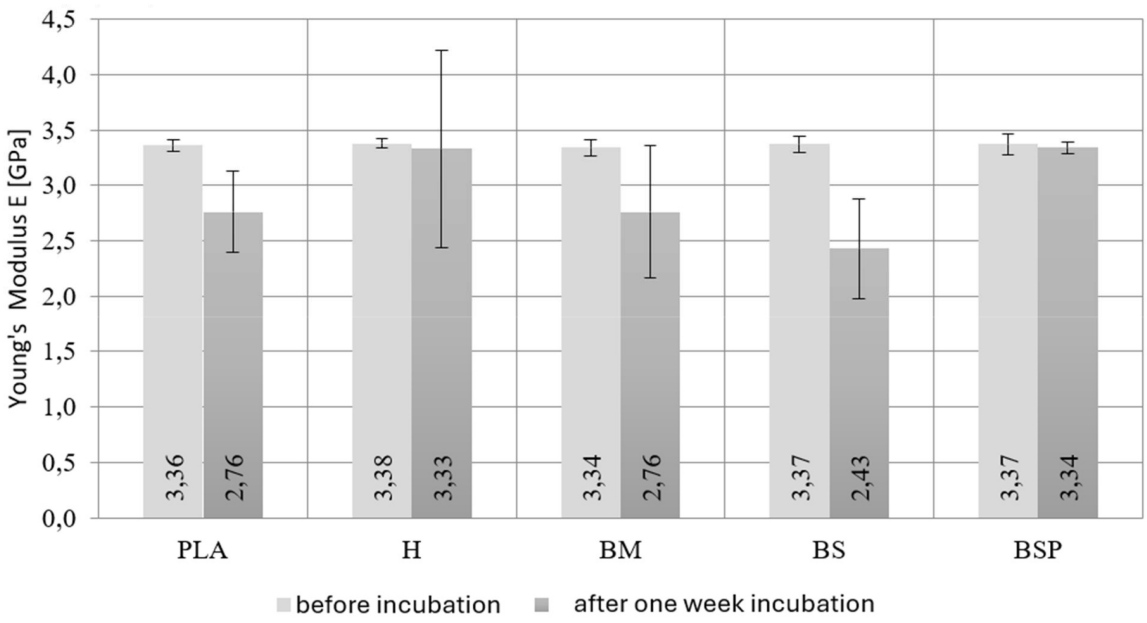

Fig. 6. Young's modulus of PLA and mineral-filled composites before and after one week of hydrolytic ageing.

media evolution has been described by Klimczuk et al. [16] under thermally accelerated ageing conditions. The pronounced conductivity increase observed in bentonite-filled systems may also indicate the contribution of ion exchange processes characteristic of smectite clays. Comparable effects have been previously reported in polymer/clay degradation studies, where mineral phases are suggested to participate in interfacial reactions and modify degradation media chemistry [6, 11].

3.4. Tensile behaviour

Hydrolytic ageing resulted in a progressive reduction of tensile properties across all material systems. The decline in Young's modulus (Fig. 6) and tensile strength (Fig. 7) is consistent with molecular weight reduction and microstructural damage induced by hydrolytic chain scission [2, 3].

Halloysite-filled composites demonstrated improved short-term mechanical performance

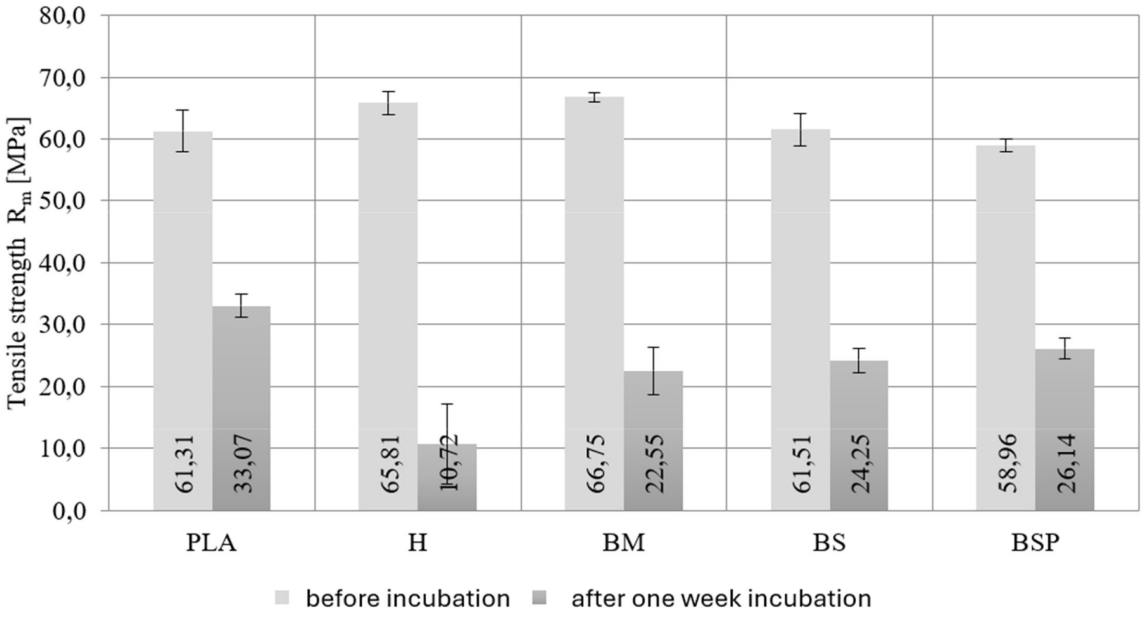

Fig. 7. Tensile strength of investigated materials before and after one week of hydrolytic ageing.

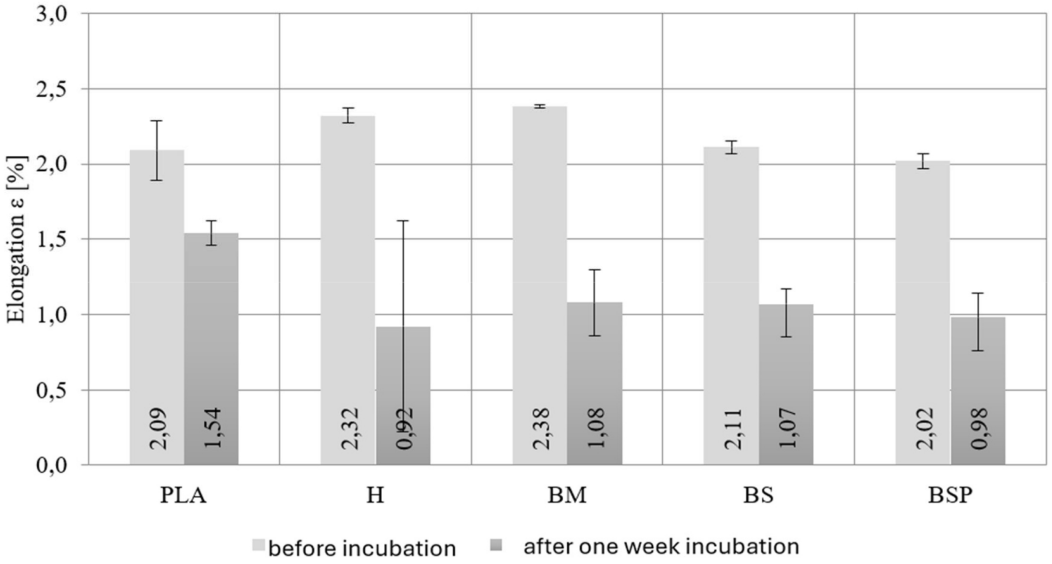

Fig. 8. Elongation at break of investigated materials before and after one week of hydrolytic ageing.

relative to neat PLA, characterised by increased stiffness (Fig. 6) and tensile strength (Fig. 7) prior to ageing. This reinforcement effect is commonly associated with the high aspect ratio and favourable stress-transfer characteristics of halloysite nanotubes [6, 8, 19]. However, hydrolytic exposure led to mechanical deterioration in all composites. Bentonite-filled materials exhibited a more pronounced degradation of tensile properties (Figs. 6 to 8), which may correlate with their higher water absorption behaviour. Increased moisture uptake may accelerate hydrolytic degradation processes and promote the formation of microstructural defects, such as microcracks and interfacial weakening.

Elongation at break values (Fig. 8) further indicate progressive embrittlement, consistent with hydrolytic chain scission and reduced molecular mobility [2]. Due to pronounced specimen deformation observed at extended ageing intervals, reliable tensile testing beyond one week was not feasible. This dimensional instability itself may be considered indirect evidence of significant hydrolytic plasticisation and structural deterioration.The observed mechanical degradation aligns with findings by Limsukon et al. [2], who demonstrated that hydrolysis-induced chain scission

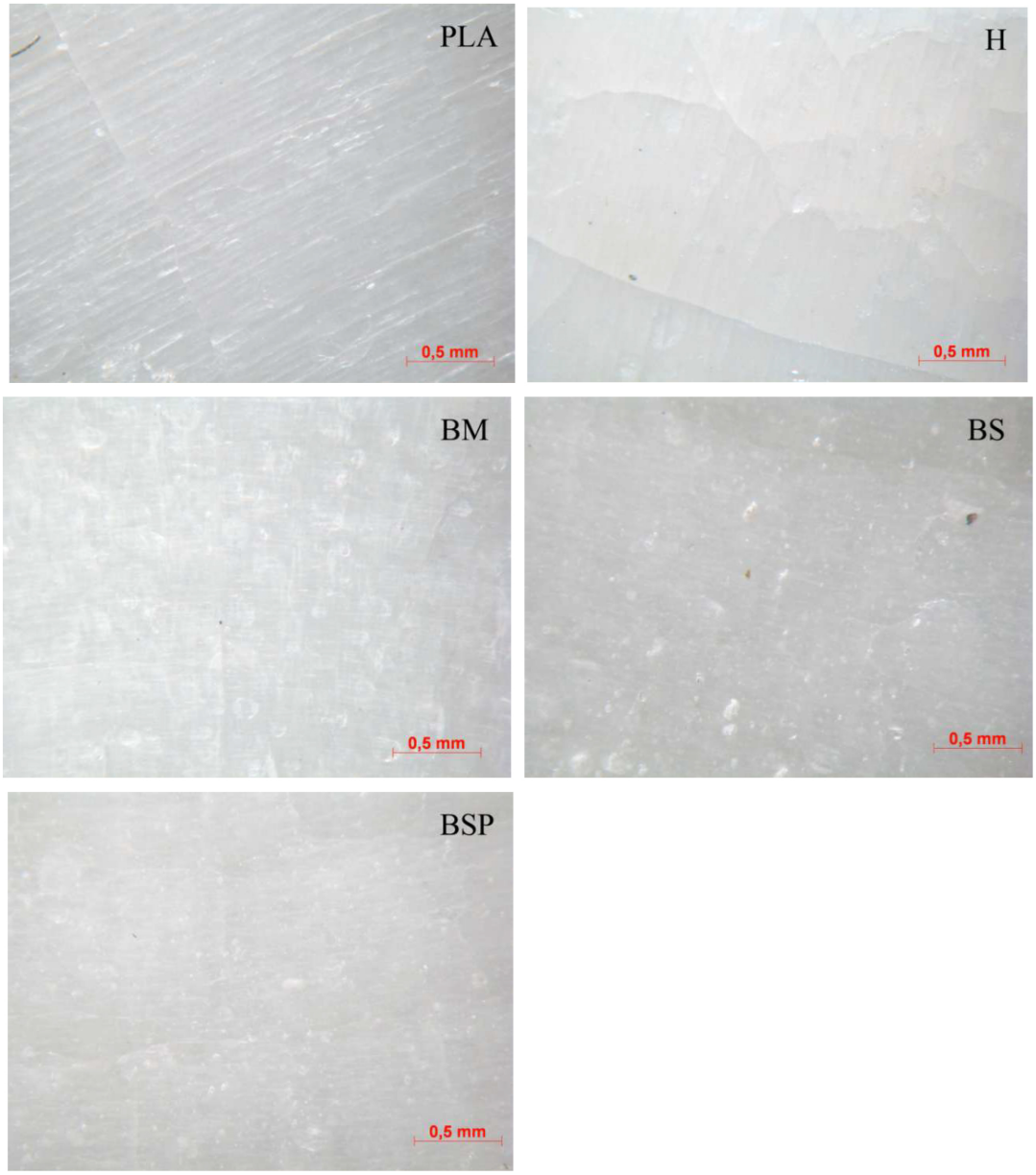

Fig. 9. Representative macroscopic surface morphology of PLA composites following hydrolytic ageing (6 weeks), magnification 48×.

leads to rapid loss of mechanical integrity, particularly near or above Tg. Similar reductions in tensile performance of PLA/smectite nanocomposites were reported by Rapacz-Kmita et al. [6, 9]. A qualitative correlation between increased water absorption and reduction of mechanical properties was observed, particularly for bentonite-filled systems.

3.5. Role of filler morphology in degradation mechanisms

The results highlight an important role of filler morphology in controlling degradation pathways. Layered bentonite structures may enhance moisture diffusion and facilitate hydrolytic attack, whereas tubular halloysite particles may contribute to moderated water transport. Recent studies emphasise that hydrolytic degradation of PLA must be interpreted within the framework of its three-phase structure (MAF/RAF/crystalline domains) [2, 3]. Variations in filler morphology and dispersion may influence local microstructure, including the distribution of rigid amorphous fractions and chain mobility, which in turn may affect degradation kinetics. Surface observations (Fig. 9) support this interpretation, revealing degradation-induced

Table 1. Average contact angle (θ) values for neat PLA and mineral-filled composites before and after hydrolytic ageing (mean ± standard deviation).

Material	Contact angle (θ) [°]	
	Before incubation	After 6 weeks incubation
PLA	75 ± 2	63 ± 6
H	79 ± 2	61 ± 2
BM	63 ± 4	60 ± 3
BS	78 ± 2	53 ± 3
BSP	76 ± 2	56 ± 4

defects and microstructural damage consistent with diffusion-controlled hydrolytic mechanisms.

Observed features include surface roughening, formation of microcracks, and localised defects, which may be associated with differential moisture penetration and internal stress development during degradation. Additionally, areas of heterogeneous degradation may be linked to non-uniform filler dispersion and the presence of particle agglomerates, which can act as sites of stress concentration and preferential degradation. These observations reinforce the concept that degradation behaviour of PLA composites cannot be explained solely by filler chemistry. Instead, the results suggest that transport phenomena governed by filler morphology, dispersion state, and interfacial structure may play a significant role in determining degradation behaviour [8]. However, it should be noted that the individual contributions of morphology and chemical composition cannot be fully separated within the scope of this study.

3.6. Surface wettability

Contact angle measurements (Table 1) revealed ageing-induced modifications of surface characteristics. Neat PLA exhibited a reduction in contact angle following hydrolytic ageing, indicating increased surface hydrophilicity. This behaviour is consistent with hydrolytic chain scission and the formation of polar functional groups [3]. A pronounced filler-dependent response was observed. Bentonite-filled composites showed more significant decreases in contact angle, which may correlate with their elevated water absorption behaviour (Fig. 2) and accelerated mass loss (Fig. 3). This effect may be associated with degradation-induced surface roughness and the increased exposure of hydrophilic mineral domains. Halloysite-filled composites exhibited comparatively moderated wettability changes, consistent with their reduced water uptake. Comparable wettability evolution has been reported in hydrolytically aged PLA systems. Kobayashi et al. [3] observed analogous surface hydrophilisation effects associated with chain scission and structural reorganisation.

The wettability results provide additional insight into moisture–material interactions. However, it should be emphasised that contact angle measurements represent an indirect assessment of surface physicochemical changes and do not directly reflect bulk degradation processes. Variations in contact angle values may be influenced by both chemical changes and surface morphology, including roughness and heterogeneity induced during degradation. Nevertheless, the observed trends are consistent with differences in water uptake and degradation behaviour, suggesting that surface energy modifications may contribute to diffusion-controlled ageing mechanisms.

4. Conclusion

This study investigates the influence of mineral fillers on the hydrolytic stability and mechanical durability of PLA composites. The results indicate a filler-dependent response, influenced by characteristics such as morphology, hydrophilicity, and dispersion state. Accelerated aqueous ageing at 50 °C revealed distinct degradation responses depending on filler morphology and moisture affinity. Bentonite-filled composites exhibited increased water absorption and accelerated mass loss, which may be associated with the layered structure of smectite clays facilitating moisture transport and promoting hydrolytic degradation processes. The associated reduction in tensile properties may indicate that hydrophilic fillers contribute to moisture-assisted deterioration mechanisms in PLA. Halloysite-filled PLA composites showed improved short-term stiffness and tensile strength, together with comparatively moderated hydrolytic sensitivity. The tubular morphology of halloysite nanotubes may contribute to reduced moisture transport efficiency and delayed degradation effects. Contact angle measurements provided complementary evidence of ageing-induced physicochemical surface modifications, although they should be interpreted as indirect indicators of surface changes. Overall, the results suggest that filler morphology, in combination with other factors such as dispersion and interfacial interactions, plays an important role in controlling moisture diffusion and degradation behaviour. However, it should be noted that the individual contributions of filler morphology and chemical composition cannot be fully separated within the scope of this study. The findings highlight that mineral filler selection may be considered as a strategy for tailoring degradation behaviour of PLA-based materials, particularly in applications exposed to moisture. The presented results contribute to understanding

structure–property–degradation relationships in PLA-based composites under moisture exposure.

Conflict of interest

The authors declare no conflict of interest.

Data availability

The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

Funding statement

This research was supported by AGH University of Krakow under statutory research funding.

Author contribution

Barbara Szaraniec – study concept, sample preparation, execution of experimental tests, data analysis, and manuscript writing. Aleksandra Ptaszkowska – sample preparation, experimental testing, and data analysis. Alicja Rapacz-Kmita – preparation and modification of additives, preparation of the literature section, and assistance in manuscript development.

Ethics

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