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| cetlogo ***CHEMICAL ENGINEERING TRANSACTIONS*** ***VOL. xxx, 2024*** | A publication ofaidiclogo_grande |
| The Italian Associationof Chemical EngineeringOnline at www.cetjournal.it |
| Guest Editors: Valerio Cozzani, Bruno FabianoCopyright © 2024, AIDIC Servizi S.r.l.**ISBN** 979-12-81206-11-3; **ISSN** 2283-9216 |

A Comprehensive Approach to the Fire Safety of Biopolymers Within Industrial Processes

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Although the use of polymers is almost ubiquitous in several industrial and civil applications, their inherent flammability poses a significant concern in terms of safety, requiring a dedicated analysis for the characterisation of fire properties. In addition, the current trend toward more sustainable processes has promoted the replacement of fossil-based traditional polymers with biopolymers. As a way of example, the use of polyhydroxybutyrate (PHB) and poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) is gaining momentum, especially for pharmaceutical and food packaging applications. Nevertheless, most of the current research is focused on the evaluation of mechanical and barrier properties at operative conditions relevant for the final use, as well as on quantifying the shelf life and optimised conditions for their production. The current literature presents a lack of knowledge on the safety parameters and fire behaviour of biopolymers. In this framework, this work presents an overview of the classification, current understanding, and experimental and numerical procedures for the characterization of biopolymers together with an experimental campaign employing small-scale thermogravimetric analysis (i.e., differential scanning calorimetry and thermogravimetric analysis) and bench-scale facilities (e.g., cone calorimeter). The collected data allow for a more robust and informed evaluation of safety aspects related to the production, storage, and transportation of biopolymers, enabling the identification of the most sustainable alternative among possible processes and components.

* 1. Introduction

Over the coming few decades, innovative materials are expected to complement and gradually replace fossil oil-based non-biodegradable materials, especially in the polymer sector (Nanda et al., 2022), to guarantee a more sustainable life cycle. In this sense, it is essential to distinguish the bio-based and biodegradable classifications prior to proceeding any further. According to the current definition, bio-based refers to raw materials replenished by biological processes at rates comparable to or faster than their rate of consumption. These concepts are further detailed in international standards and norms (e.g., ASTM D6866). The term *biodegradability*, i.e., the possibility that the polymer degrades under the action of microorganisms such as moulds, fungi, and bacteria within a specific time and environment is not directly related to the bio-based classification. Indeed, bio-based polymers can be either biodegradable or not, even if they must be in general *degradable*. Indeed, degradable polymers or plastics are intended to decompose by several processes, including physical (mechanical) disintegration, chemical degradation, and the above-cited bio-degradation. Recently, bio-based polymers have been largely introduced in packaging applications, in the construction industry, as pharmaceutical or biomedical ingredients, and in many other industrial applications. However, some potential inherent limitations continue to pose a challenge to their widespread application. Among the others, poor processability, brittleness, hydrophilicity, poor moisture and gas barrier, inferior compatibility, and poor electrical, thermal, and physical properties are worth mentioning (Mekonnen et al., 2013). Deep knowledge of thermal behavior can have beneficial effects on the design of the production plant as well as on safety performances. Due to the recent development of these materials, literature reviews on this topic may lack completeness and clarity. Therefore, it is important to conduct a thorough review of the literature to identify any shortcomings and, consequently, future prospects. Considering the current trends in global markets, it is essential to provide elevated standards and performances for BBP from a techno-economic point of view as well as from safety and environmental perspectives. Quite obviously, several factors can affect the safety parameters of BBP, including the adopted source, the chemical composition, the adopted techniques and methodologies during the production stages, additional post production processes, application fields and even the storage of the materials. For these reasons, the current understanding of the subject has been reviewed and discussed in the following section to highlight the existing gaps.

* 1. Classification of Bio-Based Polymers

Commonly adopted raw materials for BBP are starch, oils, cellulose, and fermentation products from sugar cane or sugar beet. Depending on the adopted sources and production methods, three major types of bio-based polymers can be identified, as reported below:

Polymers obtained directly from biomass are mainly polysaccharides, including starch, cellulose, chitosan, and alginates. The most important BBP is based on soy protein concentrate (SPC). Both SPC and starch-based biopolymers are produced by relatively low-cost fermentation techniques or microbiological methods and are biodegradable. Major drawbacks such as low mechanical properties, water, and thermal barrier properties have restricted their application in packaging industries. These biopolymers are often blended with other BBPs to improve properties, extend their industrial applications and decrease their production cost.

Polymers from bio-sourced monomers include materials having the same structures as fossil fuel-derived polymers, thus offering a combination of performance and renewability even if they are typically non-biodegradable. The monomers are produced via biomass fermentation followed by chemical reactions, and major advancements in fermentation efficiency, biowaste utilization, catalysis and/or purification techniques are still needed to make their manufacturing economically viable (Yang et al., 2022).

Bio-synthesized polymers include Polylactic acid or polylactide (PLA) and polyhydroxyalkanoates (PHA), more specifically poly(3-hydroxybutyrate (P3HB) and poly(3-hydroxybutyrate-co-3-hydroxyvalerate (PHBHV). Both PLA and PHA are produced from starches. PLA and PHAs naturally degrade and are often blended with starch-derived polymers to improve mechanical, biodegradation and morphological properties. Currently, the performances are typically lower if compared to the oil-based matrices. Nevertheless, due to their biocompatibility and biodegradability, the development of new solutions may replace soon traditional, petroleum-based polymers (Atiwesh et al., 2021). Hence, only bio-synthesized polymer will be analysed in the following.

* 1. Characterization of Bio-Based Polymers

Given the required chemical and mechanical properties, the choice of the specific biopolymer depends on several characteristics, including moisture absorption and thermodegradation stability. A complete understanding of the chemical stability can have beneficial effects in terms of process design as well as safety analysis (Andriani et al., 2024).Considering the organic nature of these polymers and their composites, the main concern derives from their vulnerability to flames and high-temperature conditions. Indeed, the vast majority of unwanted fires are fueled by organic polymers and their fire resistance is lower than fossil-based polymers (T. R. Hull and B. Kandola, 2009). Hence, the knowledge of thermal behavior can help in the identification of critical circumstances during the manufacturing, storage and utilization phases. From a phenomenological point of view, at high temperatures bio-polymers gradually break down, potentially producing highly flammable gases once mixed with the atmosphere. A self-sustaining combustion cycle can be established if there is sufficient heat to maintain the polymer decomposition rate high and the concentration of volatiles flammable (Kim et al., 2021). The fire behavior and flammable properties of materials depend on intrinsic chemical composition and molecular structures. Considering the complex structure of the material at a microscopic scale and the implications of it on the phenomena under investigation (e.g., molecular scission and changes in the distribution of molecular weight) (Kumar Vangala et al., 2017), the experimental approach is largely preferred for this aim. Typical systems suitable for laboratory-scale analysis are *Differential Scanning Calorimetry* (DSC) and *Thermogravimetric Analysis* (TGA) both in academic and industrial fields. The main outcomes of an experimental campaign devoted to the characterization of thermal behavior at a small scale can be intended as the glass transition temperature (Tg), melting temperature (Tm), autoignition temperature (TAIT), flashpoint temperature (Tf), degradation temperature (Td) and enthalpy of combustion (∆Hc). More recently, the Cone Calorimeter has been introduced to describe the fire behavior and degradation mechanisms of solids at bench-scale, to integrate results related to conventional calorimetry techniques at the laboratory scale (De Liso et al., 2024a)*.* In addition, an alternative protocol employing the same experimental set-up has been proposed for the quantification of the ignitability of condensed phase species at atmospheric conditions, once exposed to an external heat flux (De Liso et al., 2024b). During each test, a controlled and programmed heat program is implemented. Simultaneously, the amount of oxygen consumed, the evolution of the mass of the sample with time, and the exhaust properties can be monitored. Quantifying the amount of thermal energy released by calorimetry is pivotal and enables the identification of appropriate fire suppression methods, and the categorization of materials according to their flammability (Das et al., 2018).

* + 1. Small-scale experimental systems

For the sake of comparison, the available data on the thermal and safety-related parameters of the most relevant bio-synthesized polymers are reported in Table 1.

Table 1: Safety and thermal properties of bio-synthesized polymers. Data from (Seggiani et al., 2021), (Wang et al., 2019), (Turco et al., 2021), (Zhang et al., 2022), (Carrasco et al., 2021), and (Garlotta, 2019).

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Polymer** | **Tg [K]** | **Tm [K]** | **TAIT [K]** | **Tf [K]** | **Td [K]** | **∆Hc [J/kg]** |
| Polylactic Acid, PLA | 333  | 423\*  | NA | NA | 523  | 1.7 ∙ 107  |
| Polyhydroxybutyrate, PHB | 280  | 453\*  | NA | NA | 493  | 1.01 ∙ 105  |
| Poly(3-hydroxybutyrate-co-3-hydroxy valerate), PHBV | 276  | 426\*  | NA | NA | 443  | - 3.42 ∙ 104  |
| Poly(butylene succinate-co-butylene adipate), PBSA | 245  | 357\*  | NA | NA | 723  | 1.32 ∙ 105  |

Tg = glass transition temperature; Tm = melting temperature; Tf = flash point temperature; Td = degradation temperature; ∆Hc= enthalpy of combustion.

Although most of the studies analysing bio-synthesized polymers are focused on PLA, some general conclusions can be drawn based on the observations reported for PLA. For this material, the experimental DSC data retrieved in the current literature refer to the operating conditions and nature of the samples, in terms of crystallinity and processing (Mngomezulu et al., 2019), showing a peak of the melting process (433 – 438 K) and the peak which identifies the glass transition of the material (333 – 340 K). Based on the reported works, the melting enthalpy is between 10 and 22 J/g. The thermal degradation of PLA can be attributed to several factors (Lim et al., 2022) such as: hydrolysis by trace amounts of water; zipper-like [depolymerization](https://www.sciencedirect.com/topics/biochemistry-genetics-and-molecular-biology/depolymerization); oxidative random main-chain scission; intermolecular [transesterification](https://www.sciencedirect.com/topics/biochemistry-genetics-and-molecular-biology/transesterification) to [monomer](https://www.sciencedirect.com/topics/pharmacology-toxicology-and-pharmaceutical-science/monomer) and oligomeric esters; intramolecular transesterification resulting in the formation of monomer and [oligomer](https://www.sciencedirect.com/topics/pharmacology-toxicology-and-pharmaceutical-science/oligomer) lactides of low molecular weight. From the TG and DTG curves (Carrasco et al., 2021), it can be observed that different samples are affected by the influence of the oxidizing environment. The result is a shift of the thermal decomposition curve and also of the maximum degradation rate of the material. The influence of composition turns out to be relevant because of the addition of plasticizers and fillers, whose volatility and the bond established with the polymer matrix can influence the shift in the degradation curve. In addition, also the morphology influences the degradation rate. Since most commercial polymers are produced as pellets, the surface-volume ratio and porosity of the material affect the diffusive and retro-diffusive mechanisms of the gas phase in the particle pores, consequently displacing the degradation profile. Starting from the experimental data, many studies proposed a decomposition mechanism including a random scission, unzipping depolymerization and intermolecular transesterification during the pyrolysis phase (Abe et al., 2004). However, McNeill and Leiper (McNeill and Leiper, 1985) demonstrated that a peak in acetaldehyde concentration can be reached at 503 K during degradation, with a significant production also observed up to 713 K. The fall in proportion can be attributed to the thermal decomposition of acetaldehyde, which produces CH4 and CO in the process. The presence of metal impurities from process catalysts is a factor that also significantly accelerates the thermal deterioration of bio-synthesized polymers.

* + 1. Bench scale experimental systems

Table 2 summarizes the operative conditions adopted and main results obtained in reference studies employing bench-scale systems, such as sample thickness (σ), time to ignition (TTi), peak of heat release rate (pHRR), total heat released (THR) and residual mass (wr) for PLA.

Table 2. Operative condition and main results of experimental data from cone calorimeters.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Source | σ [mm] | TTi [s] | pHRR [kW/m2] | THR [MJ/m2] | wr [%] |
| X. Wang et al. (2019) | 3 | 56 | 466 | 745 | 0.7 |
| Zhang et al. (2021) | 4 | 74 | 436 | 86 | 2.3 |
| Liu et al. (2011) | 6 | 85 | 436 | 146 | 0 |

Cone calorimeter, offer the possibility to compare the heat release rate and the total heat release versus the time of the samples in different conditions. Most of the cone calorimeter tests in the literature have been carried out following the procedures described in ISO 5660. In this case, square specimens of different thicknesses are irradiated at a heat flux of 35 kW/m2, simulating a mild fire scenario in the proximity of the analysed sample (Zhang et al., 2021). An almost linear increase in the HRR until pHRR can be observed after ignition. However, the slope recorded during the growing phase is affected by the sample thickness. Indeed, experimental data highlight the influence of sample thickness also on the time to ignition, namely the greater the thickness, the higher the time to ignition. This trend can be attributed to the larger heat capacity of deeper samples. Indeed, provided that the same heat flux and exposed surfaces have been tested, reaching a given temperature and activating the decomposition requires a longer time. Similarly, THR increases with specimen thickness because of the melt-dripping behavior. Remarkably, the amount of residual material can vary significantly. However, this aspect can be mostly attributed to the presence of impurities within the starting sample since low residue remained after combustion.

* 1. Methodology

Based on the discussed elements, the use of cone calorimeter appears to be a self-standing strategy for the characterization of bio-polymers. In this work, a cone calorimeter was employed to evaluate the combustion behavior of the most common market grade biopolymeric materials: polyhydroxybutyrate (PHB), poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) and polylactic acid (PLA). The instrument operates by measuring the oxygen consumption during combustion, which is directly related to the heat released. To ensure accurate and reliable measurements, the cone calorimeter system is calibrated using a methane burner. Additional elements such as IR and paramagnetic analyzer are respectively devoted to the CO/CO2 and O2 measurements. On the other hand, the soot production was monitored by a laser photometer beam through the detection of the optical density. Further details of the equipment can be found elsewhere (De Liso et al., 2024c). The samples, with a thickness of 1 cm, were placed on aluminum foil supported by an insulating material inside a square-shaped sample holder. The materials were exposed to a controlled radiant heat flux, simulating fire conditions, while parameters such as heat release rate (HRR) and mass loss rate (MLR) were recorded. The effects of boundary conditions, including the external heat flux (7, 15, 25, 35, 50 kW/m2), were evaluated experimentally to mimic credible conditions in the case of accidental scenarios in the proximity of the investigated mixtures. The collected data were considered for the evaluation of the estimation quality of the existing mechanisms and the identification of the rate-determining steps either for pyrolysis or flame-related scenarios. The composition of exhaust gas was also monitored during the tests to provide further insights into the toxicity and visibility of the produced flames.

* 1. Results

In this study, the combustion behaviour of PHB, PHBV, and PLA was analyzed using a cone calorimeter to assess their thermal degradation and combustion characteristics under controlled heat flux. The heat release rate (HRR) profiles (Figure 1, a) revealed distinct trends. More in detail, PHBV exhibited the highest peak HRR (~581 kW/m²), followed by PHB (~506 kW/m²), while PLA displayed the lowest peak (~268 kW/m²). The distinct HRR profiles observed for PHB, PHBV and PLA reflect the complex interplay between thermal degradation, mass transport, and combustion kinetics. For PHB and PHBV, the sharp rise in HRR during the initial stages of combustion can be attributed to rapid pyrolysis, driven by the thermal decomposition of the polymer chains into volatile degradation products. These materials undergo degradation through random chain scission, which accelerates the release of combustible volatiles such as hydrocarbons, CO, and CO₂ (Kervran et al., 2022). The higher peak HRR observed for PHBV compared to PHB may be attributed to the influence of the valerate groups and the branched structure, which enhance the release of reactive volatiles due to their lower thermal stability (Qu et al., 2000). As the combustion progresses, the HRR decreases sharply for PHB, indicating that the majority of the combustible material is consumed rapidly, leaving minimal residual mass. PHBV, however, exhibits a more gradual decline in HRR, potentially due to the presence of intermediate degradation products or differences in the rate of volatilization associated with its branched structure. PLA shows a delayed and broader HRR peak, likely a result of its semi-crystalline structure and higher thermal stability compared to PHB and PHBV. The ester groups in PLA contribute to slower degradation kinetics, which is reflected in the more prolonged combustion process and the relatively lower intensity of its HRR peak (Chen et al., 2023). Phenomenologically, these differences also influence the flame dynamics. The rapid release of volatiles in PHB and PHBV results in flames with likely higher turbulence and potentially higher soot formation due to incomplete combustion of the volatiles. PLA, however, produce a more stable flame with reduced soot emissions, owing to its controlled and sustained release of combustion products. The slower degradation of PLA showed also a lower intensity of visible flames compared to PHB and PHBV.



Figure 1. Heat Release Rate (HRR) profiles (a.) and Mass Loss Rate (MLR) curves (b.) of PHB, PHBV and PLA at 35 kW/m2 obtained with cone calorimeter.

The mass loss rate (MLR) trends (Figure 1, b) reinforced these observations: PHBV and PHB exhibited the fastest degradation rate, while PLA displayed slower and more gradual mass loss. The delayed degradation of PLA can be attributed to its rigid molecular structure and strong intermolecular forces, which may interfere with the volatilization and the thermal breakdown. More in detail, PHB and PHBV demonstrate sharp MLR peaks, corresponding to the rapid release of volatiles during their respective thermal decomposition processes. However, the broader MLR peak of the PHBV suggests the presence of more complex degradation pathways or a slower release of volatiles due to its molecular composition. PLA, on the other hand, exhibits an extended MLR profile with a longer plateau, indicative of a steady release of volatile products over time, consistent with its slower degradation kinetics. The fire behaviour of the biopolymers can be compared to fossil fuel-based plastics (De Liso et al., 2025), revealing that, despite their higher susceptibility to thermal degradation, biopolymers exhibit near complete combustion with negligible soot production. To achieve similar performance and thermal stability of fossil fuel-based plastics, strategies such as incorporating flame retardants (e.g. phosphorus-based compounds or aluminium hydroxide) or blending with more thermally stable polymers can be employed.

* 1. Conclusions

This work presents an overview of the classification of bio-polymers and the current understanding of the decomposition paths and safety parameters, with specific emphasis on the techniques and current knowledge of the phenomena ruling their thermal, flame, and safety behaviour. Thermogravimetric analysis is largely indicated as a convenient approach for preliminary screening as well as for the selection of operative conditions to be investigated in details. Based on the gathered information, the cone calorimeter was identified as a promising system for the characterization of these materials. Besides, a set of operative conditions relevant to industrial applications was selected and adopted for a dedicated experimental campaign. The degradation of PHB, PHBV, and PLA was analysed at this stage. The acquired knowledge of the overall reaction rate of bench-scale products can be also essential for the development of predictive macrokinetic models accounting for chemical microkinetics, thermodynamics, and transport phenomena. Detailed studies and analyses of biopolymer materials are needed because of their use and production process limitations, especially in areas with fire hazards.

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