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BIODEGRADABLE POLYESTERS BASED ON RENEWABLE POLYOLS AND THEIR ANTI FUNGAL PROPERTIES DERIVED FROM SALVIA HISPANICA SEEDS

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ABSTRACT

A new field of study is the use of bio-based materials to create sustainable goods. The use of renewable biomass-derived monomers in the production of green plastics has grown in popularity recently. One of the most important renewable resources that may be obtained in large quantities from a variety of natural and synthetic sources for the production of polyesters is polyols. Renewable polyols for the production of bio-based, biodegradable polyesters are covered in this study. Polyols and diacids, which are very ecologically friendly compounds, are produced as the polyesters break down. Certain elastomeric sustainable polyol-based polyesters may be developed by careful process engineering conditions selection, chemical adjustments, and creative technologies with an industrial focus.

In this review, we have focused on the synthesis of different biodegradable polyesters using renewable polyols, their structure-property relationship, and their potential applications in a range of industrial fields, such as coatings, tissue engineering, etc. And the spectral characteristics and Anti fungal properties of the prepared bio based polyester. Furthermore, we highlighted the significant obstacles in these domains for the use of polyesters and their possible remedies for a safer and more prosperous future.

INTRODUCTION

A growing area of study is the creation of bio-based materials with the goal of replacing polymers derived from renewable resources. Meier et al., (2007) Montero de Espinosa and Meier, (2011), Mutlu and Meier, (2010), Stempfle et al., (2014), Xia et al.,(2013); Belgacem and Gandini, (2008) Biermann et al., (2011) Chernykh et al., (2013); Meier, (2014); Meier et al., (2007). Vegetable oils are incredibly plentiful and inexpensive renewable resources. Among these, soybean oil is the focus of several research projects pertaining to polymer material uses. Soybean oil does find use as a plasticiser, lubricant, or as an ingredient for polymer products. Montero de Espinosa et al. (2014), Chernykh et al. (2013), and Altuna et al. (2011) A few instances also discuss the direct polymerisation of modified oils using vegetable oil to create polymer material. Yang et al. (2014), Winkler and Meier (2014), Tan and Chow (2010), Stemmelen et al. (2011), and Maisonneuve et al. (2013). To far, most efforts for polymerising soybean or linseed oils have comprised a multi-stage triglyceride modification process followed by the crucial polymerisation phase. Stempfle et al. (2014), Ma et al. (2013), and Biermann et al. (2011) Shorter techniques mostly based on anhydride-curing (España et al., (2012), Espinoza-Perez et al., (2011), Ručigaj et al., (2014), Samper et al., (2012) or acid-curing (Shogren et al., (2004), Shogren, (1999) of epoxidised vegetable oil are the most promising approaches. The need for biodegradable polymers in medical applications, like tissue engineering and therapeutic drug administration, has motivated researchers in the field of material science to create novel synthetic polymers. Synthetic biodegradable polymers offer greater versatility than metals and ceramics, allowing for customization to maximize physical qualities, biodegradability, tissue response, and biocompatibility. The design, manufacturing, and application of biodegradable polymers for biomedical uses, including tissue engineering and drug administration, have advanced significantly in the last few decades. Doppalapudi,et.,al.,Shah, T.V.;et. al (2022). Because of their numerous benefits, polyesters are widely used as biodegradable polymers. The existence J.S. Zhang et al (2022) of crystalline regions with both crystalline and amorphous domains is an issue with semicrystalline thermoplastic materials. Non-linear losses in mechanical strength may result from this morphological heterogeneity. (Pavlath, A.E.) (2020). Cross-linking

thermoset materials, however, frequently results in completely amorphous or reduced crystallinity materials with predictable mechanical strength loss and linear deterioration rates. Additionally, when combined with better structural integrity, chemical resistance, and thermal stability, thermoset polyesters can provide superior performance. Ye, H.; Zhang, et.,al., (2002) The excessive L.Ojamae et al.(2006) demand for petrochemical products and their detrimental impacts on the environment can be decreased by using polyol polyester polymers derived from renewable resources such vegetable oils, sorbitol, diacids, and cellulose. They support a wide range of applications, including adhesives, sealants, and elastomers, to mention a few. Polyol polyesters hydrolyze to their natural building blocks when they come into prolonged contact with tissues, and they are often environmentally degradable. (Pavlath, A.E.) (2020). Polyesters, a class of polymers with repeating ester groups in their molecular chain, are widely employed in the automotive, marine, aerospace, and apparel sectors. Typically, they are made using petrochemical resources. Interest in creating bio-based polyesters from renewable resources has grown dramatically in recent years. Important monomers obtained from biomass D.Thomasson et al(2006) include isosorbide , (J.S. Zhang et al.)(2018) D-mannitol , 2,5-furandicarboxylic acid , Ye, H.; Zhang, et.,al., (2002) sebacic acid , lactic acid , citric acid, itaconic acid, and glycollic acid. These are used in the production of bio-based polyesters, which are used to replace traditional polyesters based on petrochemicals. Lavilla et al., for example, created bio-based aromatic rigid polyesters using Manx, which is made from D-Mannitol. (Pavlath, A.E.) (2020) Guidotti and colleagues used 2,5-furandicarboxylic acid to create a totally bio-based homo-polyester that has outstanding flexibility and thermal stability. Because they are biodegradable, renewable, and possess special physicochemical properties, these biopolymers have gained popularity as alternatives to synthetic polymers. In 1926, Maurice Lemoigne first identified polyhydroxyalkanoates (PHAs) as a kind of biodegradable polyester. These naturally occurring biopolymers are stored by a range of organisms, mostly prokaryotes, as extra carbon source in the form of intracellular granules, often in contrast to stressful conditions such as high temperatures or environments with elevated salinity or oxygenation Obruca, S.;et.,al(2020) .

MATERIALS AND METHODS

Materials

The polyol used in this study was prepared in our lab using the edible seed of Salvia Hispanica. Citric acid (100%), 1,6-hexanediol (100%), monomers were supplied by Molychem Pvt. Ltd. and used as such.

SAMPLE PREPARATION

Synthesis of aliphatic polyesters was carried out by two stage melt polycondensation technique. At first stage a prepolymer was prepared by carrying equimolar amount of diol and acids were placed in a 250 ml round bottomed flask and the mixture was heated upto 140°C - 145°C for 30 minutes under a constant stream of nitrogen. At second stage the resultant prepolymer was postpolymerized by crosslinking with polyol at different molar ratios, films were cast into glass plate and placed in an air oven maintained at 80°C for 24 h, polyesters were obtained.

POLYMER CHARACTERIZATION

FT-IR SPECTROSCOPY

Fourier Transform Infrared Spectroscopy (FTIR) is a valuable tool for analyzing the chemical structure of bio-based polyesters. Fourier transform infrared (FTIR) spectra of Bio based polyesters were obtained using THERMO NICOLET, AVATAR 370 FTIR SPECTROMETER with KBR crystal in the range of 4000 - 400cm-¹ at 27ºc.

TG/DTA ANALYSIS

TG/DTA thermograms of the polyesters were obtained by heating from 30 °C to 800 °C at a steady pace of 20 °C/min was the goal of the temperature program. The differential thermal analysis (DTA) curve showed endothermic and exothermic transitions, while the thermogravimetric (TG) curve was utilised to determine the percentage mass loss at various phases. The temperatures that corresponded to the highest rates of breakdown were determined using the derivative thermogravimetric (DTG) curve.

SEM ANALYSIS

Scanning Electron Microscopy (SEM) is used to analyse the surface morphology, microstructure, and degradation of bio-based polyesters. Particle dispersion, surface alterations brought on by deterioration (such as erosion or cavities), and the calibre of the interface between the polymer and any reinforcing agents are some of the data it helps disclose. The surface morphology of the bio-based polyester was analyzed using a ZEISS Sigma Field Emission Scanning Electron Microscope (FE-SEM). The analysis was carried out at an electron high tension (EHT) of 3.00 kV with the SE2 secondary electron detector to obtain high-resolution images. A working distance (WD) of 7.0 mm was maintained during imaging, and observations were made at a magnification of 5000× to clearly reveal the microstructural features.

RESULTS AND DISCUSSION

FTIR ABSORPTION BANDS OF BIO-BASED POLYESTER

The FTIR spectrum of the synthesized bio-based polyester is shown in Figure 1, and the major peaks with their assignments are summarized in Table 1. The spectrum confirmed the successful formation of polyester from the bio-based precursor.

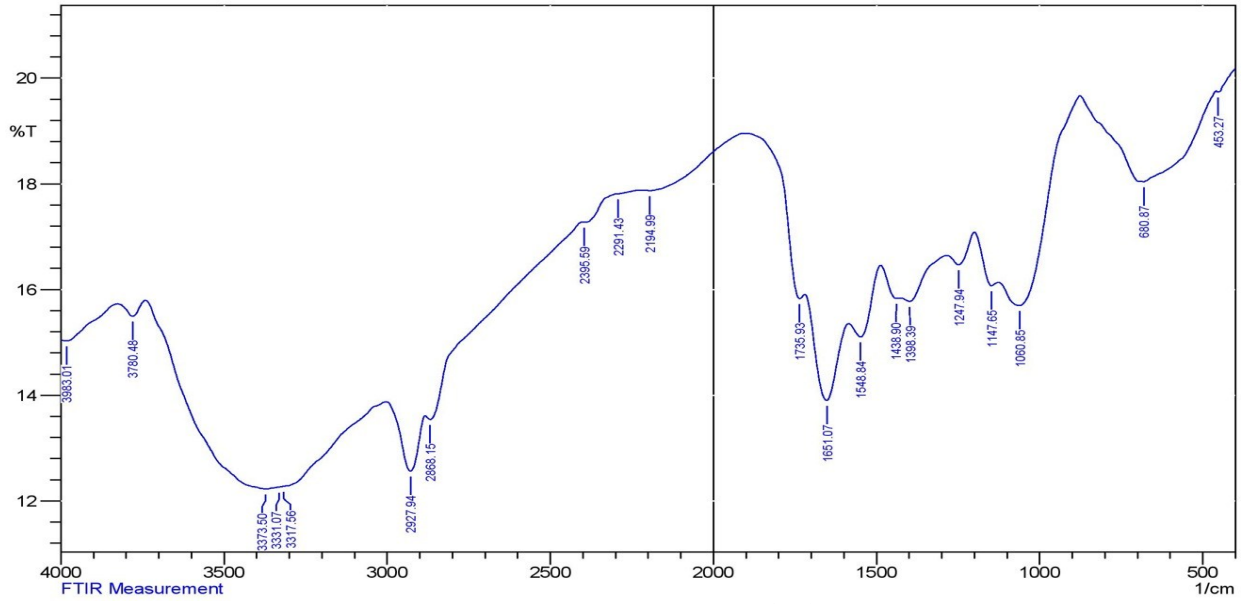


Fig:1 FT-IR spectra of Bio-Based Polyester

A broad absorption band was observed at 3337 cm^{-1}, corresponding to the stretching vibrations of hydroxyl groups (-OH). The presence of this band indicates unreacted hydroxyl end groups in the polymer backbone, which is a common feature in

bio-based polyesters. Characteristic peaks of aliphatic –CH_2 stretching vibrations appeared at 2927 cm^{-1} and 2866 cm^{-1}, representing asymmetric and symmetric stretching modes, respectively. A strong and sharp band was detected at 1735 cm^{-1}, which is attributed to ester carbonyl (C=O) stretching. This peak is considered the most prominent evidence of successful polyesterification, and the absorptions at 1247 cm^{-1} and 1175 cm^{-1} correspond to C–O–C stretching vibrations of ester groups, further confirming polyester bond formation. Comparable peaks were observed in FTIR spectra of bio-based polyester resins synthesized from plant oils based polyol.(Patel et al., 2016).

In addition, peaks around 1450 cm^{-1} were assigned to –CH_2 bending vibrations, while multiple bands in the range of 1100–1000 cm^{-1} indicate the presence of C–O stretching of ester linkages. These results are in good agreement with the spectral features reported for bio-based polyesters by Verma et al. (2020). The fingerprint region (below 800 cm^{-1}) displayed several minor peaks, which can be attributed to out-of-plane bending of C–H bonds in substituted hydrocarbons, reflecting the complex structure of the synthesized polyester matrix. Overall, the FTIR analysis confirmed the presence of hydroxyl, ester, and aliphatic groups, thereby validating the successful synthesis of bio-based polyester.

The major FTIR bands observed in the synthesized bio-based polyester and their corresponding functional group assignments are presented below. These values were compared with reported literature to confirm the successful formation of polyester linkages.

TGA-DTA Results of Bio-based Polyester

Thermal stability of the synthesized bio-based polyester was evaluated using thermogravimetric (TGA) and differential thermal analysis (DTA) under nitrogen atmosphere. The TGA curve revealed a three-step degradation behavior. The initial minor weight loss below 150 °C corresponded to the evaporation of residual moisture and entrapped volatile compounds. The second major degradation step, observed between 250 °C and 450 °C, was attributed to the scission of ester linkages and decomposition of the polyester backbone. Beyond 500 °C, a gradual weight loss was observed, indicating the breakdown of char residue and further thermal decomposition

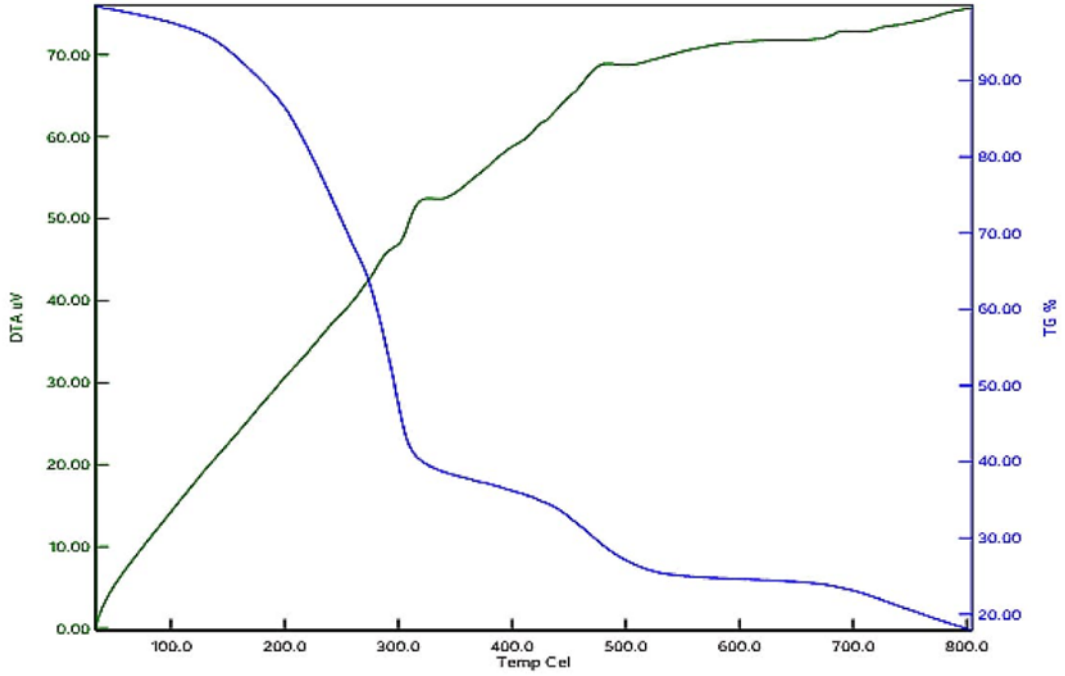


Fig:2 TGA-DTA of Bio-Based Polyester

Parameter	Details
Sample Name	Bio-based Polyester
Instrument Module	TG/DTA
Sample Weight	6.820 mg
Reference	Alumina (10.820 mg)
Gas Atmosphere	Nitrogen
Pan Material	Alumina
Heating Program	30–800 °C, 20 °C/min
Observed Weight Loss Steps	Step I: Moisture/volatiles loss (below ~150 °C) Step II: Major degradation of polymer backbone (~250–450 °C) Step III: Final char decomposition (>500 °C)
DTA Observations	Endothermic peak at low temperature (moisture release) Broad exothermic transitions corresponding to polyester chain degradation

The DTA thermogram supported these findings, with an initial endothermic peak corresponding to moisture loss, followed by broad exothermic transitions that reflected the structural decomposition of the polymer matrix. The overall results confirm that the bio-based polyester exhibits good thermal stability up to ~250 °C, making it suitable for applications requiring moderate thermal resistance.

SEM ANALYSIS OF BIO-BASED POLYESTER

The bio-based polyester synthesized from chia seed oil-derived polyol, citric acid, and 1,6-hexanediol represents a novel and sustainable approach to polymer development, leveraging renewable resources and multifunctional monomers. . Scanning electron microscopy (SEM) analysis of the synthesized polyester reveals a surface morphology characterized by flake-like, layered structures, indicative of heterogeneous phase distribution and effective crosslinking. These microstructural features suggest

potential applications in biodegradable packaging, biomedical scaffolds, and eco-friendly coatings. The presence of crystalline domains and petal-like formations observed under SEM at 6000× magnification further supports the hypothesis of a well-organized polymer matrix, influenced by the interplay of aliphatic diol and multifunctional acid components. Overall, the combination of chia seed oil-based polyol with citric acid and 1,6-hexanediol yields a promising bio-polyester with desirable structural and environmental attributes.

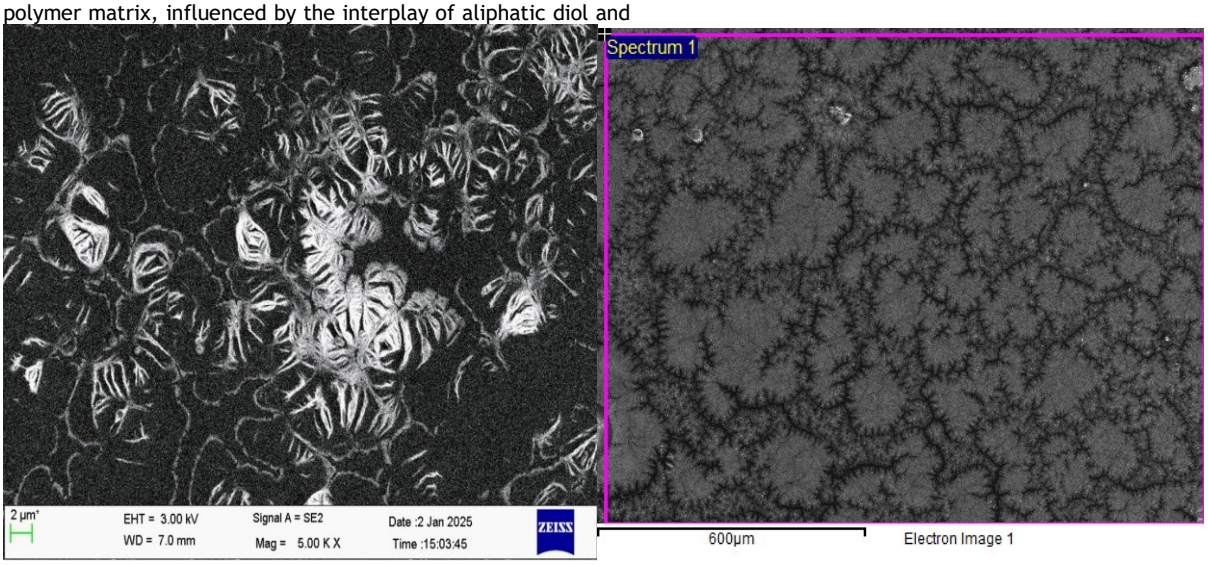


Fig:-3 SEM IMAGE OF BIO BASED POLYESTER

ANTI FUNGAL ACTIVITY

Test organism	Polyester(mm)	Control (nystatin)(mm)
Candida parapsilosis	14.2mm	16
Aspergillus flavus	19.0	15

The agar well diffusion technique was used to assess the synthetic bio-based polyestrs antifungal efficacy against Aspergillus flavus and Candida parapsilosis. The common antifungal medication utilised for comparison was nystatin. Table 2 provides a summary of the zone of inhibition values.

The polyester had strong antifungal action, according to the data. In comparison to Nystatin (16 mm), the bio-based polyester exhibited a modest inhibitory zone (14.2 mm) against Candida parapsilosis. Remarkably, the polyester showed a larger inhibitory zone (19 mm) for Aspergillus flavus than the control (15 mm).

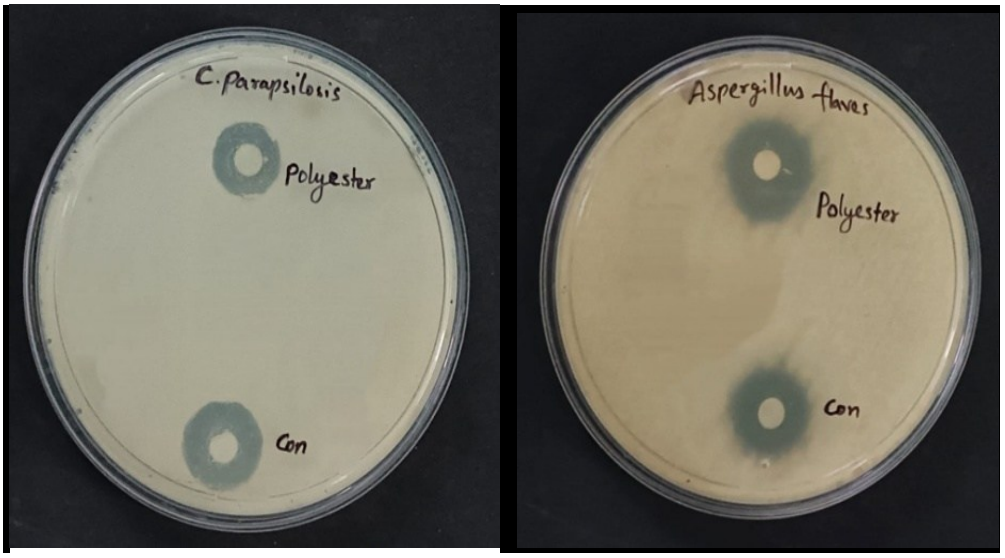
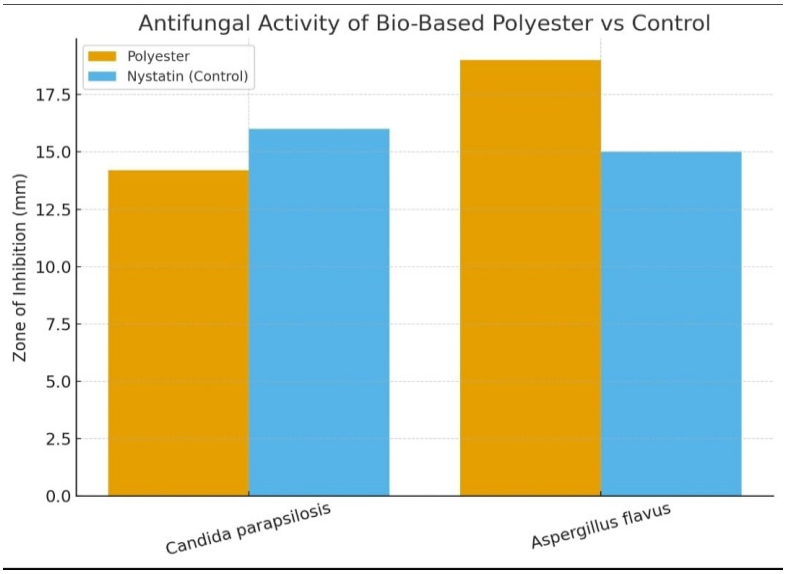


Fig:- 4 Antifungal activity of Bio based polyester

Comparing the inhibition zones of bio based polyester vs nystatin for both fungi



This implies that the bio-based polyester made from seed-based polyol and citric acid/diol has antifungal qualities by nature, perhaps because the polymer matrix contains hydroxyl, ester, and left over phytochemical groups that prevent fungal development.

CONCLUSION

Through a two-stage melt polycondensation process, the current work shows how to successfully synthesise biodegradable polyesters utilising renewable polyols obtained from Salvia hispanica (chia) seed oil, citric acid, and 1,6-hexanediol. Characteristic ester connections were confirmed structurally by FTIR spectroscopy, and multi-step degradation with stability up to about 250 °C was shown by thermal analysis, confirming their suitability for use in moderate-temperature settings. SEM imaging revealed multilayer crystalline morphologies, indicating potential for packaging or biological applications as well as efficient crosslinking. The polyesters had noteworthy antifungal efficacy against Aspergillus flavus, with inhibition zones that were larger than those of nystatin, a common antifungal drug. These results demonstrate that the synthesised materials have both inherent bioactivity and environmental degradability, highlighting their potential as sustainable substitutes for industrial and biomedical applications.

To further confirm their economic viability, future research should concentrate on scalability, long-term biodegradation behaviour, and wider antibacterial effectiveness.

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