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Investigation of the 3-hydroxyvalerate content and degree of crystallinity of P3HB-co-3HV cast films using Raman spectroscopy

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Abstract

The structural properties of extruded cast films of poly-3-hydroxybutyrate (P3HB) and poly-3-hydroxybutyrate-co-3-hydroxyvalerate (P3HB-co-3HV) were analysed by Raman spectroscopy. A sensitive band for the side chain of the 3HV was found at 844 cm^{-1} (ν_{CC} , $\nu_{\text{C-COO}}$). Using an internal standard at 1101 cm^{-1} ($\nu_{\text{C-O-C}}$, ρ_{CH_3}), a model for calculation of the 3HV content was developed. This was based on analysis of samples with a 3HV content of 0-12 mol%. Additionally, P3HB-co-3HV samples were tempered in order to create different degrees of crystallinity. The induced changes in the Raman spectra were analysed. The Raman model to determine the degree of crystallinity considers the sensitive band at 2999 cm^{-1} (ν_{asCH_2}) and the internal standard at 1059 cm^{-1} ($\nu_{\text{C-O}}$, $\nu_{\text{C-CH}_3}$, ρ_{CH_2}). The investigated crystallinity was in a range of 0.69-0.86.

1 Introduction

Biopolymers are currently the subject of ongoing research to develop sustainable packaging solutions. Thermoplastic biopolymers are of particular interest, and their properties are continually being improved. Besides thermoplastic biopolymers synthesised from lactic acid, other materials such as aliphatic polyesters and polysaccharides generated by plants or microorganisms¹ can be used. Among these, polyhydroxyalkanoates (PHAs) are particularly promising and are the focus of this work. PHAs are extracted from renewable resources and are biodegradable, water-insoluble and exhibit thermoplastic properties.² They can be produced by microorganisms³, recombinant bacteria, genetically engineered plants or by anaerobic digestion of biological waste⁴ using agricultural raw materials as resources.⁵ They have the advantage of being fully degradable by bacteria, fungi or algae, with the degradation products being water and carbon dioxide.⁶ PHAs are produced from various hydroxy-alkanoates; the molecular mass ranges from 50-1,000 kDa.⁴ PHAs have a high degree of polymerisation and crystallinity (55-80%¹) and are optically active, piezoelectric and isotactic.⁴ Challenging, however, is their limited availability on a large scale and also the inadequate structural and mechanical properties of the final films.

Depending on the monomer units, PHA films with a wide variety of mechanical properties can be produced - from hard crystalline to elastic. The incorporation of a long side-chain, such as 3-hydroxyhexanoate (3HH), into an otherwise highly crystalline poly(3-hydroxybutyrate) (P3HB) leads to reduced crystallinity and increased flexibility.⁷ An increasing amount of a longer side chain (shown for 3-hydroxyvalerate (3HV)) lowers the melting temperature.⁸ In order to alter the morphology and thereby improve the final properties of PHA films, longer side chains can be incorporated.

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