facts

CITROFOL® – Citrate Esters as bio-based plasticisers for biodegradable mulch film applications

Mulch films in agriculture

The use of plastic mulch films in agriculture has significantly increased over the past decade, as they offer benefits such as protecting crop from insects, conserving moisture and enabling more efficient use of soil nutrients. Various plastics have been used as mulch films to date, with low-density polyethylene (LDPE) being the most widely used. LDPE is also considered the most effective because of its excellent mechanical strength, low cost, and the protection it offers from sunlight and water.[1,2] However, the main drawback of using LDPE films comes from their disposal after use, which poses a significant environmental pollution problem. This has spurred the development of different kinds of biodegradable mulch films in recent years.[3] Biodegradable films represent a sustainable alternative to LDPE films because, at the end of their life, they can be directly incorporated into the soil, where microflora convert them into carbon dioxide, water and biomass. Biodegradable materials therefore do not produce the damaging waste that traditional mulch films do.[2,4]

The most common bio-based polymers investigated for mulch films are polylactic acid (PLA), starch, cellulose and polyhydroxyalkanoates (PHAs).[4,5] Of these, PLA and PHA blends should be highlighted. Their main advantages when it comes to mulching are improved biodegradability, non-toxicity and commercial availability.[4]

Polyhydroxyalkanoates (PHAs)

Polyhydroxyalkanoates are aliphatic polyesters produced by numerous microorganisms and bacteria. More than 150 different grades of PHAs are currently known, but the most common PHAs are poly poly 3-hydroxybutyrate (PHB) and poly 3-hydroxybutyrate-co-3-hydroxyvalerate (PHBV). Both of them are commercially available short-chain polymers. For mulch film applications, the main drawback of these materials that needs to be overcome are their poor mechanical properties – especially their brittleness – and their relatively high costs. However, by blending PHAs with other polymers, such as PLA, these mechanical properties can be improved and reach values similar to those of polyethylene mulch films. This also reduces the costs associated with the polymer.[4,6]

Polylactic Acid (PLA)

Polylactic acid is an aliphatic polyester made from monomers of lactide, which is synthesised from lactic acid. PLA has good thermoplasticity, biodegradability (under composting conditions) and biocompatibility, which allows it to be used in a wide range of applications. However, as mentioned in the case of PHAs, the high brittleness of PLA can limit its usability as a mulch film material.[4,7]

Several strategies have been developed to overcome these issues. In general, plasticisers are widely used as additives for polymeric materials, as they enhance flexibility, processability and ductility. The addition of plasticisers to fine-tune polymer properties is often preferred over other strategies such as copolymerisation. Additivation is more cost-effective and versatile than copolymerisation, which entails re-synthesis and scale-up for each desired change of the polymer's properties. Different types of plasticisers, such as polyethylene glycol (PEG),[8] citrate esters[9–11] and triacetin,[12] have already been investigated for their ability to improve the flexibility of biopolymers such as PLA and PHAs.

Usability of plasticisers

Nevertheless, there are several important considerations when choosing a plasticiser for a given application. Firstly, good miscibility between the plasticiser and the polymer is essential, as this guarantees a homogeneous blend. Moreover, an efficient plasticiser should reduce the glass transition temperature (T_g) and melting point (T_m) of the plasticised material.[13,14] This enables easier processing at lower processing temperatures and allows expansion of the processing window for polymers with only a small gap between their melting points and thermal degradation. For biomedical and agricultural applications, the plasticisers used should also be non-toxic and show very low levels of migration, as the latter would cause the material to regain the brittleness of biopolymers.[15] Furthermore, plasticisers can also improve the compatibility of hydrophilic fillers; for example, by reinforcing fibres and hydrophobic biopolymers.

For biopolymers in particular, the plasticisers should ideally be bio-based and biodegradable as well. With plasticisers derived from fossil fuels, the overall bio-content of the final product would be diluted and its biodegradability may be hampered. In this regard, CITROFOL® citrate esters are the perfect bio-based and biodegradable choice as plasticisers for biopolymers.

CITROFOL® – benefits as a bio-based plasticiser

CITROFOL® citrate esters are bio-based, safe and environmentally friendly. They are listed in international chemical inventories such as REACH. Citrate esters are produced by the acidic esterification of citric acid and either ethanol or n-butanol. All the required raw materials can be obtained from renewable resources. A further acetylation step can produce additional esters. CITROFOL® types are clear, colourless and odourless liquids and are non-VOCs (volatile organic compounds), with excellent storage stability. Moreover, all CITROFOL® esters are vegan, kosher, halal and non-GMO. Regarding their use as plasticisers, they are versatile and characterised by an excellent human- and eco-toxicological profile. They are therefore the ideal choice for sensitive products such as toys, medical devices and food packaging, as well as for pharmaceutical applications and personal care. They also offer an attractive alternative to petrochemical-based plasticisers such as phthalates, benzoates or adipates. CITROFOL® esters are already used in bio-based polymers such as cellulose acetates and nitrocellulose, where they have a positive impact on processing and the final product properties. Besides their extensive compatibility with various polymers, their ability to be rapidly composted without causing harm to air, soil or water is unique, and is perfectly aligned with the increasing end-of-life requirements for many products manufactured with biopolymers.

Table 1: Properties of citrate esters

	CITROFOL® AI	CITROFOL® AII	CITROFOL® BI	CITROFOL® BII
Chemical name	Triethyl Citrate	Triethyl O-Acetylcitrate	Tributyl Citrate	Tributyl O-Acetylcitrate
Synonym	TEC	ATEC	TBC	ATBC
CAS No.	77-93-0	77-89-4	77-94-1	77-90-7
Molecular formula	$C_{12}H_{20}O_7$	$C_{14}H_{22}O_8$	$C_{18}H_{32}O_7$	$C_{20}H_{34}O_8$
Molecular weight [g/mol]	276.3	318.3	360.4	402.5
Flash point [°C]	150	188	206	218
Boiling Point [°C]	287	326	309	331
Pour point	-40	-43	-63	-57
Colour	Colourless			
Viscosity @ 25°C [mPas]	27	39	24	32

In principle, all CITROFOL® citrate esters should be suitable plasticisers for PHAs and PLA. Their characteristic properties can be found in table 1. Citrate ester plasticisers are readily soluble in PLA and PHAs owing to the polar interactions between the ester groups of the biopolymer and the plasticiser.

1 Granulate properties

1.1. Goal of study

The aim of the study is to generate a basic data set on the processing and the material properties of PHA/PLA blends plasticised with CITROFOL® BII. This citrate ester was chosen due to its perfect fit for technical applications and its low migration behaviour. Different aspects were considered in this study. First, the CITROFOL® BII concentration was varied to analyse how the plasticiser concentration affects the final polymer properties. Secondly, different matrix polymers were tested to show the broad applicability of CITROFOL® BII. Lastly, the properties of CITROFOL® BII were compared with a commonly used benchmark plasticiser (triacetin).

1.2. Materials, compounding and methods

1.2.1. Materials

To investigate the influence of CITROFOL® BII as a plasticiser on PHA/PLA blends, different polymer blend compositions and types of polymers were tested (see table 2). The polyhydroxyalkanoates, PHBV (ENMAT™ Y1000P) and PHB (ENMAT™ Y3000P) were provided by TianAn Biologic Materials (China). In addition, two different PLA types (Ingeo™ 4043D from NatureWorks® LLC (USA, PLA 1), Luminy® L175 from TotalEnergies Corbion (Netherlands, PLA 2)) were tested. All polymer raw materials were dried before use to prevent polymer degradation during processing caused by residual moisture present in the pellets. CITROFOL® BII (tributyl O-acetylcitrate) from Jungbunzlauer Ladenburg GmbH was used at concentrations of 10 and 20 wt.%, and triacetin (Kollisolv® GTA, glycerine triacetate, TA) from BASF was tested at a concentration of 10 wt.% as a benchmark.

Table 2: Composition of PHA/PLA blends with and without plasticiser

Materials	Compound V1	Compound V2	Compound V3	Compound V4	Compound V5	Compound V6
PHBV	75	75	75	75		75
PHB					75	
PLA 1		25	25	25	25	
PLA 2						25
CITROFOL® BII		10	20		10	10
Triacetin				10		

1.2.2. Compounding and processing of plasticised PHA/PLA blends

Using a two-step manufacturing process, the plasticising effect of CITROFOL® BII was investigated with regard to the processability and properties of the polymer blend. In the first step, plasticised granulates of PHA/PLA were produced through melt extrusion compounding. Here, different types of PHAs and PLA were blended in a co-rotating twin-screw extruder and the liquid plasticiser was added using a liquid dosing unit. The plasticised granulate was then melted again in order to prepare injection-moulded test specimens. For this process, an Arburg Allrounder 370S was used, with injection pressures between 400 and 560 bar. Finally, the thermal, mechanical, rheological and migration properties of the test specimens were analysed.

1.2.3. Test methods for characterisation of the compounds and test specimens

Mechanical properties

Mechanical properies like tensile strength, elongation at break and Young`s modulus were determined on a universal testing machine (Zwick 1476), using a cross-head speed of max. 5 mm/min. All tests were conducted according to DIN EN ISO 527-2 with standard shoulder stick type 1A. Five test specimens per sample were used, with dimensions of 4 x 10 x 115 mm (see figure 1).

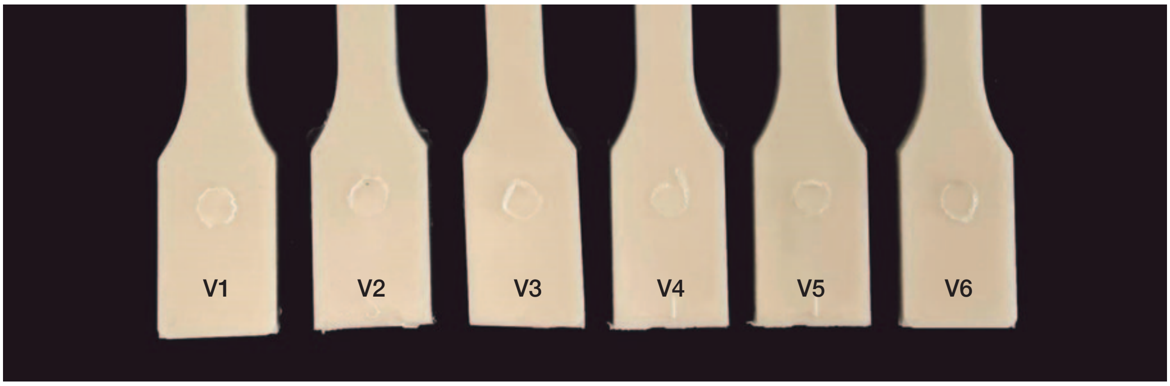

Figure 1: Polymer blend test specimens for mechanical tests

Thermal properties

Glass transition temperature (T_g) and melting point (T_m) were determined using differential scanning calorimetry (DSC) following DIN EN ISO 11357-1. For these tests, a DSC 2/700 from Mettler Toledo with a heating range of -40°C to 200°C and a heating rate of 10 K/min was used. Analysis of thermal properties was always carried out based on the second heating curve.

Rheological measurements
To determine the viscosity of the samples, a plate-plate rheometer (Discovery HR20 by TA Instruments) was used. All measurements were conducted according to DIN ISO 51810. A frequency sweep over a range of 0.1 to 100 Hz at a temperature of 185°C was measured.

Storage tests
The test specimens (three samples per plasticiser type and concentration) were stored under controlled conditions (60°C, 30% ventilation and 50% relative humidity) in a convection oven for 28 days. The mass loss of the test specimens was determined on different days during this period.

1.3. Results

Processing properties and advantages

The investigation showed that different PHA/PLA blends can be successfully compounded with CITROFOL® BII at concentrations of 10 and 20 wt.%. The use of CITROFOL® BII delivered significant benefits. Firstly, the lowest injection pressure and post-injection pressure were obtained for the compound with 20 wt.% CITROFOL® BII, while the highest value was found for the unplasticised sample. In addition, the number of shots – i.e. the volume of material that is used to fill the mould cavity – was increased from 5 to 20 by adding CITROFOL® BII at a concentration of 10 wt.%. This improvement over the unplasticised sample could not be achieved for blends with 10 wt.% triacetin. Here, the pressure, residual volume and dosing time fluctuated significantly more in comparison to the sample containing 10 wt.% CITROFOL® BII.

Part A: Variation of CITROFOL® BII concentration

The influence of different CITROFOL® BII concentrations on a blend based on PHBV and PLA 1 was investigated to show how the plasticiser concentration affects the final polymer properties.

Rheological properties

An important property of polymers which significantly affects their processablity is their viscosity as shown in figure 2, adding CITROFOL® BII resulted in a reduction of the viscosity. In this case, the lowest viscosity was achieved with 20 wt.% CITROFOL® BII. This viscosity reduction may promote easier processing of the blend and facilitate the incorporation of fillers or other additives that are essential for improving or modifying the final polymer properties.

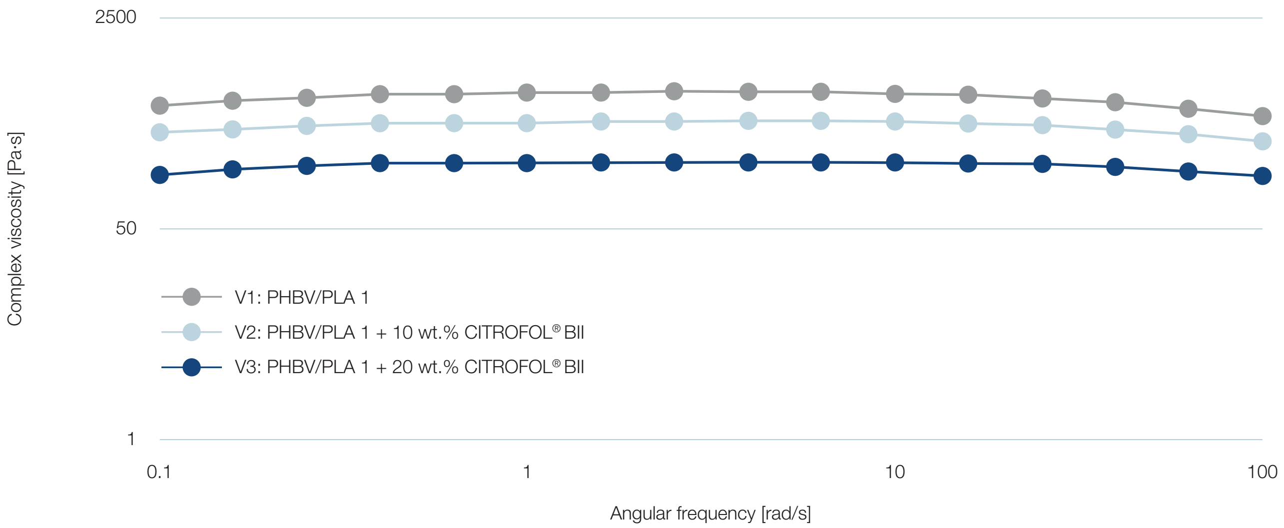

Figure 2: Complex viscosity of unplasticised and plasticised PHBV/PLA blends with 10 and 20 wt.% CITROFOL® BII

Mechanical properties

Mechanical properties such as elongation at break and tensile strength are important characteristics for a polymer, as they determine the polymer's potential applications. Adding plasticisers allows these values to be modified to create tailored polymers with desired characteristic profiles. As illustrated in figure 3 below, adding 10 and 20 wt.% CITROFOL® BII resulted in an acceptable reduction in tensile strength and an improvement of elongation at break compared to the reference sample.

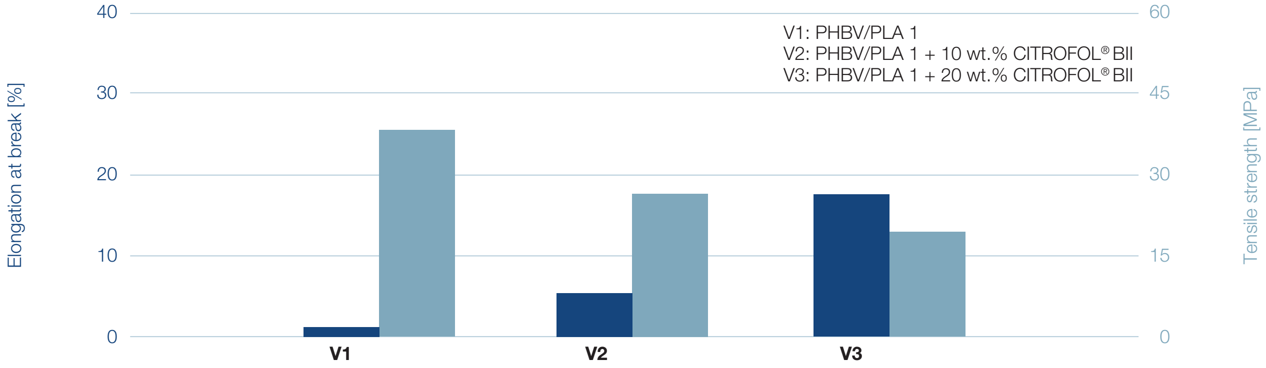

Figure 3: Mechanical properties of unplasticised and plasticised PHBV/PLA blends with 10 and 20 wt.% CITROFOL® BII

Thermal properties

Thermal properties such as glass transition temperature and melting point are also critical parameters, as they give an indication of whether the processing of the polymer can be improved by adding plasticisers. Figure 4 shows the influence of adding CITROFOL® BII, at concentrations of 10 and 20 wt.%, on the melting temperature of PHBV/PLA 1 blends. Overall, it can be seen that the melting point decreases with the addition of CITROFOL® BII. The highest reduction was recorded for CITROFOL® BII at a concentration of 20 wt.%, where a decrease of approximately 10°C was achieved. This leads to a larger gap between degradation temperature and melting point, and therefore to a larger processing window. It is conceivable that a lower processing temperature could lead to a reduction of the energy costs incurred during the production of biodegradable mulch films.

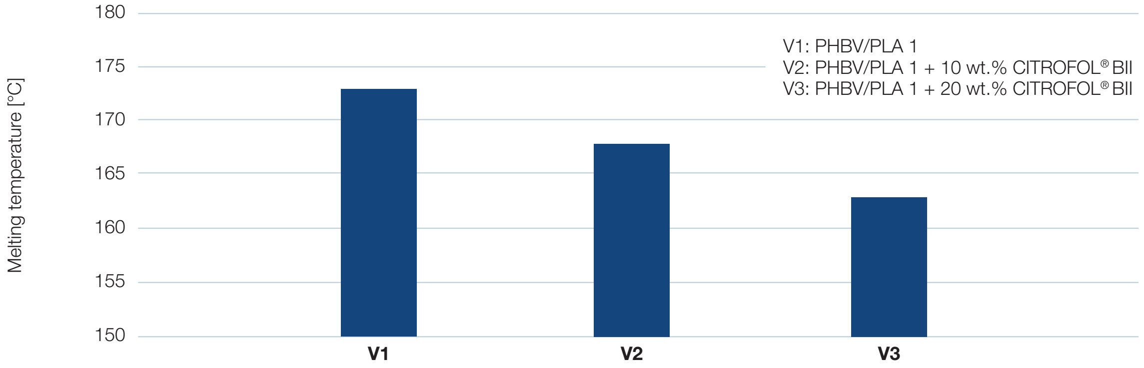

Figure 4: Thermal properties of unplasticised and plasticised PHBV/PLA blends with 10 and 20 wt.% CITROFOL® BII

Storage tests

The results of the storage tests are presented in figure 5. In the storage tests, the blend with 10 wt.% CITROFOL® BII showed a mass loss of 0.5% after 28 days of storage, while a mass loss of around 0.7% was observed for the blend with 20 wt.% CITROFOL® BII. The mass loss thus slightly increases with higher plasticiser concentration. In general, the overall mass loss is comparatively low, as the sample without plasticiser showed also a mass loss of 0.2%. This indicates a very low volatility behaviour of the plasticiser.

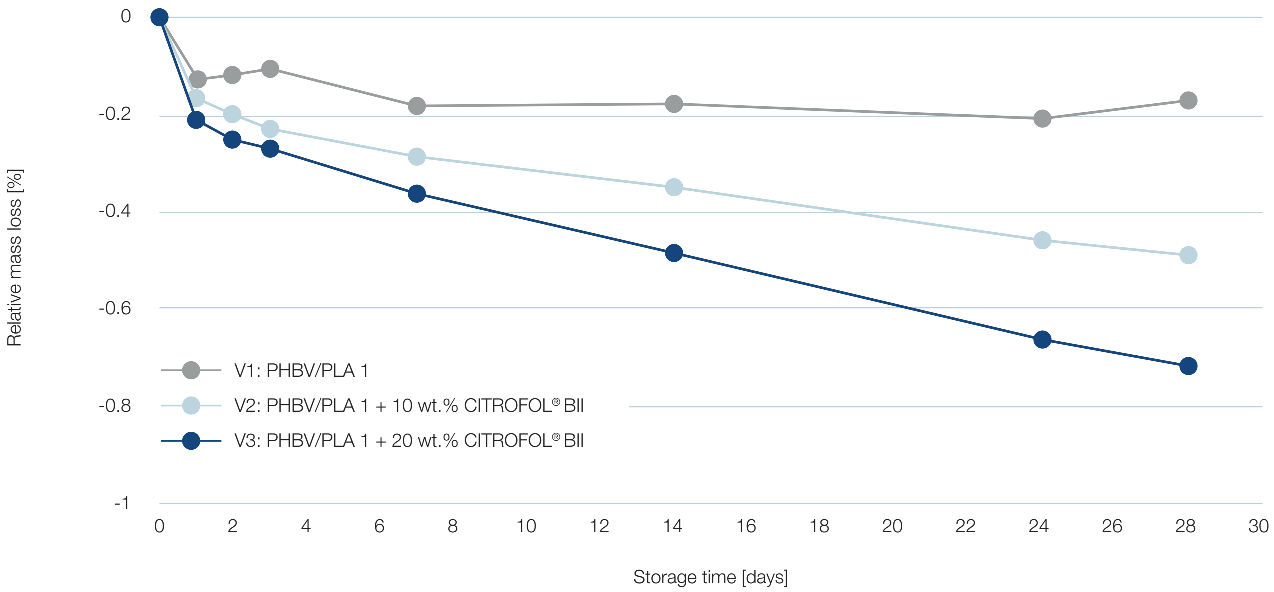

Figure 5: **Storage tests of unplasticised and plasticised PHBV/PLA blends with 10 and 20 wt.% CITROFOL® BII**

Part B: Compatibility with different PLA and PHAs grades

The influence of CITROFOL® BII on different types of PHAs (PHB and PHBV) and two grades of PLA was investigated to show the broad applicability of this citrate ester.

Similar results for the rheological measurements were observed for all samples. A slightly higher viscosity was achieved only in the case of the polymer blend with PHB, since PHB has a higher molecular weight than PHBV. With regard to the thermal properties, melting points between 167 and 169°C were determined. This indicates that CITROFOL® BII's efficacy as a plasticiser was nearly identical for all tested polymer blends.

The mechanical properties are shown in figure 6. Again, nearly identical results for tensile strength and elongation at break were obtained for all samples. The small difference between V2, V5 and V6 is probably due to the different molecular weight distribution of the polymer grades used, as previously stated.

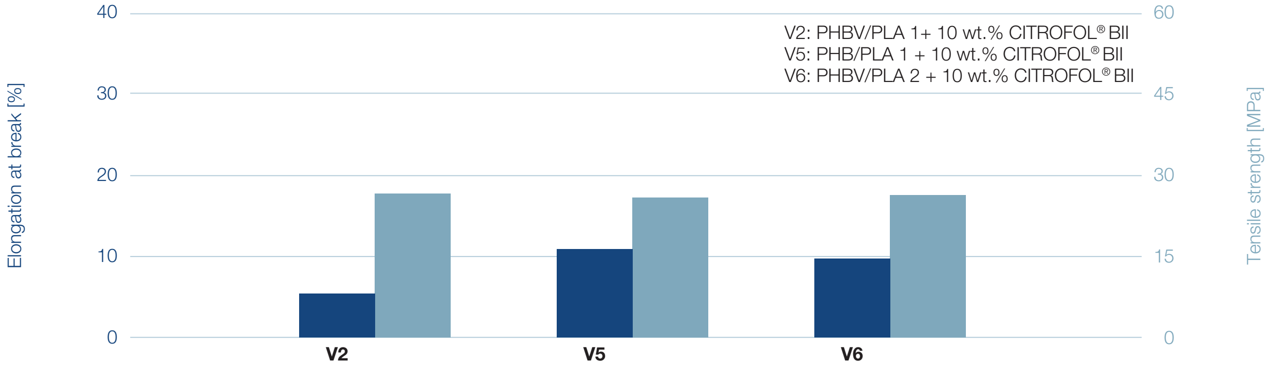

Figure 6: Mechanical properties of different types of PHA/PLA blends with 10 wt.% CITROFOL® BII

Furthermore, all tested blends with 10 wt.% CITROFOL® BII showed the same low mass loss after a storage time of 28 days, with values of around 0.5%.
In summary, these results give rise to the conclusion that CITROFOL® BII has a broad applicability with reproducible plasticising properties for different polymer systems.

Part C: Comparison to a market benchmark

In the third set of tests, the performance of citrate esters was compared to that of triacetin, a market benchmark. Triacetin was chosen as it has a certain structural similarity to CITROFOL® BII.

The rheological measurements revealed that the blends with 10 wt.% CITROFOL® BII and 10 wt.% triacetin had nearly identical viscosity profiles. In addition, the melting points are also in the same range – 168°C for CITROFOL® BII and 167°C for triacetin – showing that the efficacy of both substances as plasticisers is nearly identical.

The results of the storage tests are shown in figure 7. The sample with triacetin had a significantly higher mass loss than the samples plasticised with CITROFOL® BII. Therefore, CITROFOL® BII seems to be the better choice for long-term applications.

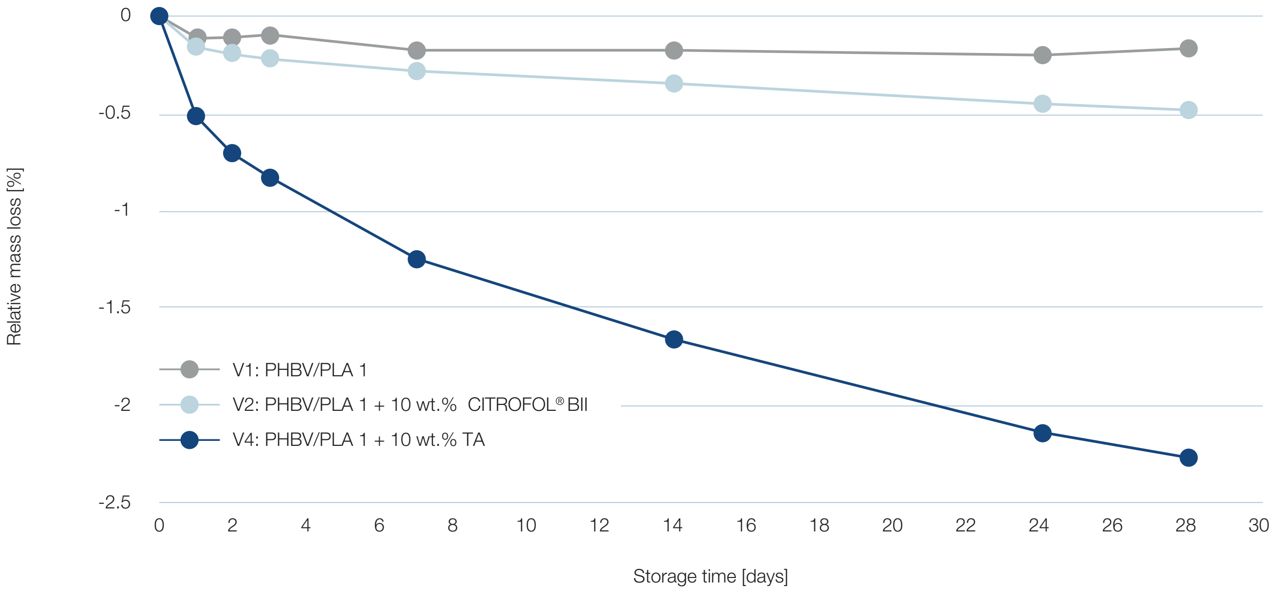

Figure 7: Storage tests of unplasticised and plasticised PHBV/PLA blends with 10 wt.% CITROFOL® BII and triacetin (TA)

2 Film properties

2.1. Goal of study

The second section of the study involved conducting proof-of-concept film blowing experiments to determine the processability of PHBV/PLA blends with CITROFOL® BII. For this purpose, PHBV/PLA films with 5 and 10 wt.% CITROFOL® BII were prepared.

2.2. Materials, film blowing process and methods

2.2.1. Materials

To investigate the processability of PHBV/PLA films with CITROFOL® BII as plasticiser, different polymer blend compositions were prepared.

PHBV (ENMAT™ Y1000P) was supplied by TianAn Biologic Materials (China) and PLA (Ingeo™4043D) from Nature-Works® LLC (USA). In addition, CITROFOL® BII from Jungbunzlauer Ladenburg GmbH was added in concentrations of 5 and 10 wt.%. In this case, no benchmark was analysed (see table 3).

Table 3: Composition of PHBV/PLA blends with CITROFOL® BII

Materials	Film V1	Film V2	Film V3
PHBV	75	75	75
PLA	25	25	25
CITROFOL® BII		5	10

2.2.2. Film blowing with plasticised PHBV/PLA blends

After compounding (as described in 1.2.2), films of PHBV/PLA blends were manufactured using a blown film extruder with a blow-up ratio of 1:3 and a die diameter of 63 mm. The processing temperature during the film production was between 175°C and 185°C. These conditions resulted in a 200 mm-diameter film tube.

2.2.3. Test methods for characterisation of the films

Mechanical properties of the films

To obtain data on the mechanical properties of the films, tensile strength and elongation at break were determined. These properties were evaluated using a universal testing machine (Zwick 1476) following DIN EN ISO 527-3.

Disintegration

The disintegration of PHBV/PLA samples under composting conditions was tested based on a slightly adapted version of ISO 20200:2015 standard. Different films were prepared (with dimensions of 25 x 25 x 0.2 mm), placed between an iron mesh and buried 6 cm deep in plastic reactors (30 x 20 x 10 cm) containing solid synthetic wet waste. This solid synthetic wet waste was prepared by mixing 10% compost, 30% rabbit food, 10% starch, 5% sugar, 1% urea, 4% corn oil and 40% sawdust and subsequent adding distilled water at a 45:55 ratio. The reactors were placed in an air circulation oven (Heraeus, type T6) at 58ºC for 50 days. Periodically, water was added to the plastic containers to maintain the relative humidity in the composting environment. Additionally, the solid synthetic wet waste was regularly mixed to guarantee aerobic conditions during the composting process. Films were recovered from the containers several times during this period for photography and visual evaluation of the influence of CITROFOL® BII on the disintegration rate of the samples.

2.3. Results

Processing properties and advantages

It was demonstrated that adding CITROFOL® BII helped to improve the film blowing process. For the unplasticised blend system, production of the film was very difficult, almost impossible, and resulted in a non-homogeneous film thickness. Addition of 10 wt.% CITROFOL® BII significantly improved processing, resulting in a film with a very homogeneous thickness. A more stable process and continuous extrusion blown process was achieved.

Mechanical properties of the films

The results of the mechanical properties are illustrated in figure 8. From these measurements, it can be seen that adding CITROFOL® BII slightly improves the mechanical properties. However, optimisation is desirable to improve these values. Higher CITROFOL® BII concentrations could be an option.

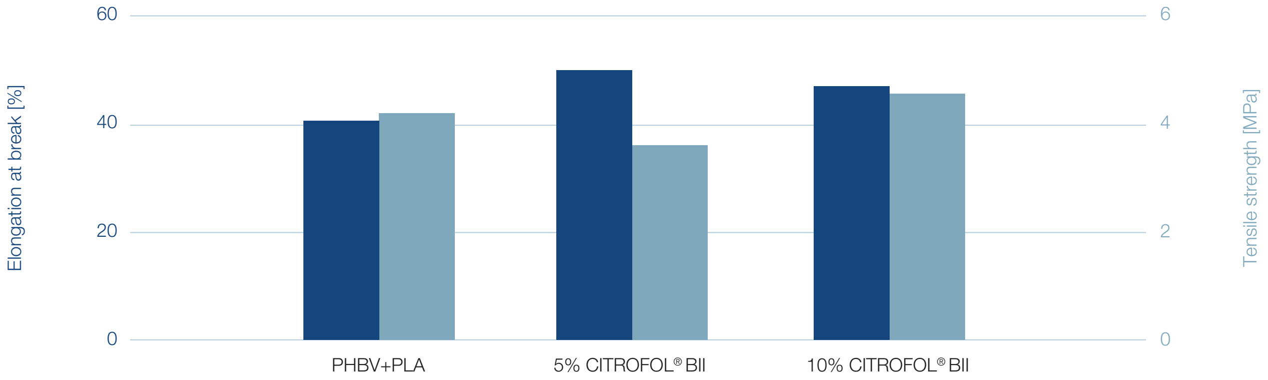

Figure 8: Mechanical properties of unplasticised and plasticised PHBV/PLA blends with 5 and 10 wt.% CITROFOL® BII

Disintegration of films

Disintegration of PHBV/PLA film samples under composting conditions was also studied. The appearance of these films after different time intervals during storage under composting conditions is shown in figure 9. PHBV/PLA films without plasticiser started to fragment after 20 days, whereas PHBV/PLA films with CITROFOL® BII (10 wt.%) had already started to disintegrate after 10 days. This suggests that the presence of CITROFOL® BII speeds up the disintegration of PHBV/PLA under composting conditions due to the increased polymer chain mobility caused by the plasticiser. Moreover, the time needed to completely disintegrate the films was significantly shorter for plasticised samples. While disintegration took 30–48 days for unplasticised samples, this period of time was reduced to 25–30 days for plasticised PHBV/PLA samples. A short disintegration time might be especially beneficial for crops with a relatively short cultivation time.

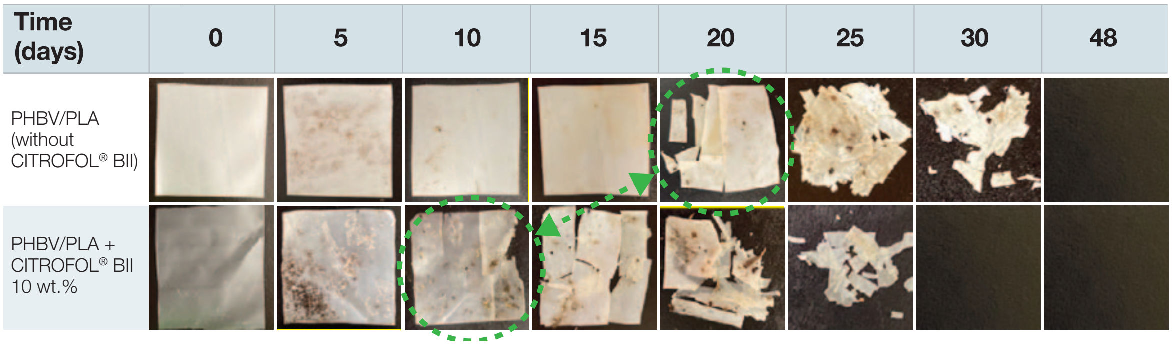

Figure 9: Appearance of PHBV/PLA film samples after various periods under composting conditions

The results of the disintegration trials of this study are an initial indication that CITROFOL® BII may reduce the time required for PHBV/PLA films to disintegrate by composting. It should be noted that such trials do not determine the biodegradability of plastics under composting conditions. To do so, further testing according to EN 17033 "Plastics – Biodegradable mulch films for use in agriculture and horticulture" would be necessary.

Conclusion

This study investigated the influence of CITROFOL® BII as a plasticiser for PHA/PLA blends for mulch films. The efficiency of this citrate ester was evaluated in terms of the thermal, mechanical, processing, migration and disintegration properties of plasticised PHA/PLA samples. Overall, the results of this study demonstrated that the use of CITROFOL® BII improves the processing properties by reducing the glass transition temperature, the melting point and the complex viscosity of PHA/PLA compounds. These facts may result in a lower energy consumption during the compounding process. In addition, broad compatibility of CITROFOL® BII across a variety of biopolymers was successfully demonstrated, and CITROFOL® BII significantly improved the ductility of these blends. The storage results point towards very low migration of CITROFOL® BII when used as a plasticiser. Furthermore, an improved film quality compared to the unplasticised material could be demonstrated for PHBV/PLA. Another promising finding obtained by the present study was an accelerated film disintegration under composting conditions by the addition of CITROFOL® BII (10 wt.%), which was due to the increased polymer chain mobility caused by the plasticiser. This could be particularly advantageous for applications in agriculture and contribute to a more sustainable production process for biodegradable mulch films.

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About Jungbunzlauer

Jungbunzlauer is one of the world's leading producers of biodegradable ingredients of natural origin, which enable its customers to manufacture healthier, safer and more sustainable products. Jungbunzlauer is among the largest global producers of citric acid and citrate esters, which are well-known under the CITROFOL® brand. Product innovation and continuous process improvements in our state-of-the-art plants result in unique, high-quality products. Citrate esters have excellent human- and eco-toxicological profiles and provide good versatility and compatibility with numerous polymers. They are characterised by highly efficient solvation, low migration and non-VOC attributes. CITROFOL® grades offer a sustainable alternative to petrochemical-based plasticisers. They are therefore the preferred choice for sensitive products like toys, medical devices, food packaging, pharmaceutical applications and personal care.
Moreover, all CITROFOL® esters are vegan, kosher, halal and non-GMO.

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