

EXTRUDED FILMS OF BIO-BASED PLASTICS FOR PACKAGING APPLICATIONS

Rostislav Svidler, Tobias Hartmann, Sebastian Buschbeck, Roman Rinberg, Lothar Kroll

Institute of Lightweight Structures, Department of Lightweight Structures and Polymer Technology, Faculty of Mechanical Engineering, TU Chemnitz, Reichenhainer Straße 31/33, 09126 Chemnitz, Germany

ABSTRACT

To contribute to a robust information base, in this work, the chemical composition and processability in the extrusion process of market-relevant bio-based plastics as well as mechanical properties and surface tension of extruded films were investigated.

In the advance, the rheology was determined by measuring melt flow index (MFI). The thermal behavior was analyzed by thermal gravimetric analysis (TGA) and dynamic differential calorimetry (DSC). In the context of structure determination, the infrared spectroscopy (IR) and nuclear magnetic resonance spectroscopy (NMR) are used.

For the purpose of a material-specific characterization, studies about processing parameters were carried out on a sheet extrusion line made by Dr. Collin GmbH. The processing analysis focused on the identification of process determining parameters, such as optimum melt temperature, calander rolls temperature and uniaxial draw ratio of the films. In addition, experiments, concerning the adhesion of the sheet on polished or matted calander rolls in relation to rolling temperature and rotation speed, are performed and measurements, to reduce surface defects, have been validated by parameter optimization.

The films were evaluated qualitatively by elaborated methodology. The evaluation criteria include the processability of bio-plastic, reproducibility of film quality and organoleptic film properties. To characterize the mechanical properties, tensile test DIN ISO 527-3 was selected. The surface free energy, which can be used to evaluate the printability, compatibility with other polymers and the gluability of plastic films, was determined according to DIN ISO 55660-1/2 by means of a video-based optical contact angle measuring instrument.

Index Terms – bio-plastic, bio-plastic blends, bio-plastic films, extruded films, PLA, PHA, PHB, Green PE, Bio PA 11, PBAT, PBS, PBSeT, PEG

1. INTRODUCTION

Owing to the increasing of waste, oil price and general environmental awareness in our society, the industries are leaded to pack their products by a more eco-friendly and sustainable way. In the near future, renewable resources from vegetable, animal elements or microbial production could replace conventional fossil resources in the production of plastics. As well, the bio-plastics may be established as being a modern and environmentally friendly packaging material. For more than ten years, the amount of companies that offer various bio-based plastics for various packaging applications has constantly increased on German and international market. As the notion of bio-plastic is not clearly defined and many

manufacturers offer their products as a "black box", the packaging manufacturers don't often know how these bio-plastics are chemically constructed and classified. As a consequence, the technical aspects, such as the film processing, thermoformability, printability, weldability of the films or compatibility to barrier plastics, are not objectively evaluated and cannot be compared. Furthermore, there is no reliable information about the food-relevant barrier properties and chemical resistance of these new products. Because of the lack of information, it would be difficult for the packaging manufacturer to use these biopolymers and its advantages. As a result, bio-plastics are usually applied for no-sophisticated packaging applications, even though they have the potential to substitute the standard plastics. Without this solid information base, the interest from industry will be not important and the bioplastics would be underestimated.

The term "bioplastic" is not clearly defined. In the following, the notion of bioplastics is considered, in this work, as thermoplastics, which are made from renewable resources and / or have the functionality of biodegradability [1]. These are "Drop-in" Polymers (green polyethylene - GreenPE, polyamide 11 - BioPA 11 und cellulosepropionate - CP), biodegradable bio-based plastics (poly(lactic acid) - PLA, polyhydroxybutyrate - PHB), biodegradable petrochemical based plastics (poly(butylenes adipate-terephthalate) – PBAT, poly(butylenes succinate) – PBS) as well as bio-based and biodegradable plastic blends (a physical blend of two or more compatible polymers). By blending of different plastics, the properties of the compound can be selectively adjusted to requirements. For instance, the PLA is often blended with PBAT and diverse additives and inorganic materials in order to increase the elongation at break and impact strength [2] [3]. A minor addition of other biopolymers (usually less than 5 weight-%), such as PBS, polybutylene succinate adipate - PBSA or PHB (V) enhances the compatibility of PLA and PBAT and biodegradability of blends [4] [7]. These bioplastic blends are most frequently encountered on the international market of bioplastics. They are environmentally friendly, compostable and have a good life cycle assessment.

To contribute to a robust information base, in this work, eleven different market-relevant bioplastics were investigated within the framework of joint project "Processing of bio-based plastics and creation of a competence network" funded by *Fachagentur für nachwachsende Rohstoffe*. Other research partners in the network are Fraunhofer IAP in Potsdam / Golm, SKZ Würzburg and IfBB in Hanover.

2. EXPERIMENTAL SECTION

2.1 Materials and Methods

2.1.1 Investigated polymers

After an in-depth research, some extrudable market relevant bio-plastics were selected for the investigations. Their mechanical properties are approximately equal to that of the conventional petrochemical plastics, such as LDPE, HDPE, PP, PS or amorphous PET. The tested bio-plastics are summarized in Table 1. They can be split into three groups. "Drop-in" solutions (Green PE, Bio-PA and CP), biodegradable "pure" plastics based on renewable raw materials (PLA, PHA) and bio-based and / or biodegradable plastic blends (chemical composition is not known).

Table 1: Investigated bio-polymers

Category	Polymer	Trade Name	Producer / Distributor
based on RRM*, non-biodegradable	Green HDPE	SHC 7260	Braskem/FKuR
based on RRM, non-biodegradable	Bio-PA 11	Rilsan BESN TL	Arkema/Resinex
Based on RRM, non-biodegradable	CP	Cellidor 300-13	Albis Plastic
based on RRM, biodegradable	PLA	Ingeo 4043D	Nature Works / Resinex
based on RRM, biodegradable	unknown	Mirel P4001	Metabolix
based on RRM, biodegradable	unknown	BioFlex A4100CL	FKuR
based on RRM, biodegradable	unknown	BioFlex S6510	FKuR
based on RRM, biodegradable	PLA-PBAT-blend	Ecovio T2308	BASF
petroleum-based, biodegradable	unknown	BioFlex F1110	FKuR
petroleum-based, biodegradable	unknown	MVera B5011	Metabolix
petroleum-based, biodegradable	unknown	MaterBi CF06A	Novamont

* RRM- renewable raw materials

2.1.2 Thermal characterization

The thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) are used to evaluate the thermal properties of the bio-polymers. The thermal decomposition temperature of the materials was determined using TG 209 F1 Iris with mass spectrometer MS 403C Aëolos (NETZSCH) in a He atmosphere. The heating rate was 20 °C/min, and the temperature range was set as: 25 °C to 800 °C. Furthermore, the samples were analyzed using a DSC 204 F1 Phoenix (NETZSCH) under N₂ atmosphere at heating rate at a constant 10 °C/min and heated to 250 °C. The thermogram signal was derived from the temperature difference between the sample and the reference one. The sample of 5-10 mg was placed in a crimped aluminium pan. The glass-transition temperature (T_g), cold-crystallization temperature (T_{cc}), melting temperature (T_m) and crystalline melting enthalpy (H_m) were calculated from the DSC thermogram.

2.1.3 Rheological property analysis

The melt flow of polymer melt was measured according to DIN EN ISO 1133 with capillary rheometer Smart RHEO 2000 (50-450°C, CEAST). The flow rate is determined as a function of applied load. Melt flow index MFI is the mass flow rate expressed in gram per 10 minutes. The sample of 5 g as a powder or a pellet is heated for 5 minutes in the barrel and extruded through the die under a constant load 2.16 kg. The melt flow index of bio-polymers was measured in the specific temperature range for each plastic.

2.1.4 Structural analysis

In order to obtain a better understanding about the chemical composition of the investigated polymers, in this process study, a structural analysis was conducted by infrared spectroscopy (FT-IR) and nuclear magnetic resonance spectroscopy (H-NMR). This background information is required to classify the investigated bioplastics correctly and be able to compare them with each other.

Infrared spectra of the biopolymers were obtained with an FT-IR-Spektrometer Alpha (Bruker) in attenuated total reflectance (ATR) mode with a diamond crystal collecting 32 scans. Each spectrum was obtained within the range of 4000-650 cm^{-1} with a wavelength resolution of 4 cm^{-1} .

NMR analysis was carried out with Varian INOVA 500 NMR System (Varian Inc.) at ^1H measurement frequency of 499.8 MHz, by means of the standard pulse sequence s2pul. The 90 ° pulse width amounted to 10.5 microseconds, the relaxation delay was 30 s and the spectral width was 8 kHz. The 20 mg of the crushed materials were solved in 0.7 ml of deuterated chloroform CDCl_3 (99.8% degree of deuteration) and transferred to a 5 mm NMR sample tube. The measurement was carried at room temperature and 40 of measurements were accumulated. The chemical shifts were referenced to tetramethylsilane TMS (0 ppm). The analysis of the spectra and the quantitative evaluation were performed with the spectrometer analysis software from Varian.

2.1.5 Film extrusion

For the purpose of a material-specific characterization, studies about processing parameters were carried out on a sheet extrusion line made by Dr.Collin GmbH. The extrusion line consists of a single-screw extruder type E1.60 of the company ERMAFA and a chill-roll type 168/250/250 from the company Collin GmbH. The extruder has a screw diameter of 60 mm, screw length ratio of 31 (L / D) and 6 heating zones. At the end of the extruder, a sheet die (350 mm) with manual flexlip is mounted via a heatable adapter. This owns a specific designed hanger-manifold with balanced pressure ratio across the width. The chill-roll exhibits a heatable group of rolls, a take-off and wrap device.

The processing analysis focused on the identification of process determining parameters, such as optimum melt temperature, calander rolls temperature and uniaxial stretching ratio of the films. In addition, experiments, concerning the adhesion of the sheet on polished or matted calander rolls in relation to rolling temperature and rotation speed, are performed and measurements to reduce surface defects have been validated by parameter optimization.

2.1.6 Mechanical properties

For tensile measurement, a mechanical testing machine (Zwick/Roell Z010 Proline) was used to provide tensile strength of the samples according to EN ISO 527-3. The specimens corresponded to the shape of Figure 1 and were taken from the extruded films by means of special cutting tool. In the chill-roll extrusion, it occurs, due to the process, the light uniaxial stretching of the films along the machine direction. This leads inevitably to the modification of mechanical properties of the film in the parallel and perpendicular directions. In order to investigate the influence of process-induced stretching, the specimens were taken in both film directions.

2.1.7 Contact angle measurement

In order to calculate the surface tension of the bio-polymers, a video-based optical contact angle measuring system (OCA 20 Dataphysics) was used to measure the contact angle according to DIN ISO 55660-1/2. The measurements were taken at room temperature and at constant humidity. The drop volume was held constant. Three different liquids were used including distilled water, diiodomethane, ethylene glycol. Finally, the dispersive and polar components of surface energy were determined by the method of Owens&Wendt, Rabel and Kälble (OWRK).

3. RESULTS AND DISCUSSION

3.1 Rheological and thermal properties

Table 2 shows the characteristic values of rheological and thermal characterization. The melt flow index was measured at three different temperatures in the processing-relevant area of the respective plastic. The MFI values of 1 to 11 g/10 min are within the rheological range which is necessary for the extrusion of films.

TGA was carried out to investigate the effect of processing on the thermal decomposition of bio-plastics under He atmosphere. Table 2 also summarizes the characteristic temperature of the thermal decomposition, such as the weight loss and the peak of decomposition steps ($T_{\text{peak}1/2}$). These peaks of decomposition occur frequently in binary plastic blends, plastics with high plasticizer content or copolymers. In neat plastics, just one decomposition peak occurs. Two distinct decomposition peaks have Cellidor 300-13 (13 m%. Plasticizer), Bioflex A4100CL / F1110 and MaterBi CF06A, which are probably bioplastic blends. All other bio-plastics decompose in one step. The onset temperature of the decomposition is not listed in the table 2, because TGA measurements are not representative to the real decomposition during processing and cannot give any information about the real mechanism governing this phenomenon under processing conditions (temperature, shear rate, residence time).

The glass-transition temperature (T_g), the cold-crystallization temperature (T_{cc}), the temperature of crystallization peak (T_p) and the crystalline melting enthalpy (H_m) from the second DSC heating cycle (20K/min) are also shown in Table 2. It lacks some characteristic values, as they could not be determined. Owing to the amorphous nature, some T_g and heats of crystallization does not exist. As well, the characteristic values are below the measuring range.

Table 2: Rheological und thermal properties of market relevant biopolymers obtained from the second DSC heating cycle (N_2 , 20K/min) and TGA (He; 10K/min)

Polymer	MFI [g/10 min]	$T_{\text{peak}1}$ [°C]	$T_{\text{peak}2}$ [°C]	Weight loss %	T_g [°C]	T_{cc} [°C]	T_p [°C]	ΔH_m [J/g]
SHC 7260	6,68 (190°C/2,16kg)	469,6	-	99,94	-	-	134,7	183,6
Rilsan BESN TL	1 (235°C/2,16 kg)	447,3	-	99,29	-	-	206,5	35,8
Cellidor 300- 13	10,36 (210°C/2,16kg)	317,7	371,2	97,94	98,1	-	-	-
Ingeo 4043D	3,47 (190°C/2,16 kg)	381,4	-	97,51	62,8	-	156	0,7696

Polymer	MFI [g/10 min]	T _{peak1} [°C]	T _{peak2} [°C]	Weight loss %	T _g [°C]	T _{cc} [°C]	T _p [°C]	ΔH _m [J/g]
Mirel P4001 (160°C/2,16kg)	9,61	302,8	-	75,33	-	-	167,2	31,65
Bio-Flex A4100CL (190°C/2,16 kg)	2,73	292,1	357,5	99,62	53,5	99,4	159,8	23,44
BioFlex S6510 (190°C/2,16 kg)	4,32	375,6	396,1	98,3	60,3	-	150,1	19
Ecovio T2308 (190°C/2,16 kg)	7,85	370,7	-	97,65	59,2	118	151,4	19,07
BioFlex F1110 (190°C/2,16 kg)	6,7	343,3	400,9	89,58	-	53,8	128,1	4,308
MVera B5011 (190°C/2,16kg)	7,94	416,7	-	99,11	-	-	125,4	9,648
MaterBi CF06A (160°C/2,16kg)	2,37	320,2	419,2	96,45	-	-	134,2	8,622

3.2 Structural analysis

As there are no information about the chemical compound and the bio-based content of many commercial available biopolymers (such as the products from FKUR, Metabolix and Novamont), high-resolution ¹H-NMR-Spectrum has been collected, in order to determine their chemical structure. From these spectra, the chemical compounds of all investigated samples could be exactly identified, which are not allowed to become public yet. The background for this decision is the potential economic damage, that the companies could be inflicted by the divulgation of the all contained substances of their compounds. Nevertheless, selected results of the spectroscopic investigations are discussed in the following.

Table 3 shows the chemical composition of investigated samples. The sample Mirel P4001 shows the typical spectrum of a commercial PHA, more specifically a copolymer of P-3-HB and P-4-HB in a composition of 90/10 mol%. Furthermore, in the material, a small amount of unidentified aliphatic additives and inorganic materials is included. Pure PHB is completely linear, isotactic and stereoisomer. This means that PHB has a great tendency to crystallize. By addition of external nucleating agents, the density and size of the spherulites can be specifically controlled. Thus, it is possible to influence the mechanical properties, in terms of stiffness and ductility [2]. This fact expounds the presence of inorganic supplements in the material, which act as extra nucleation points.

BioFlex A 4100CL proves to be polyester blend based on linear aliphatic polyester PLA. As the second and third component, the approximately equal amounts of PHB and PEG are identified. Although PLA and PHB are brittle polyester, a small amount of PHB (in a range of 10 wt%) causes significantly ductile behavior of the PLA/PHB blend [20]. Chemically considered, PEG is a polyether of dihydroxy alcohol ethanediol. Due to the hygroscopicity and exceptionally low toxicity values, PEG is often used as plasticizer and/or as binder for moisture in food packaging or as reactive component within the production of polyesters [2] [3] [21].

In the spectrum of Bioflex F1110 and Mvera B5011 can be seen the typical spectrum of aliphatic-aromatic copolyester of the type Ecoflex (PBAT) from adipic acid, terephthalic acid and butanediol. As a second component is identified a small percentage of PLA (under 10 wt%). The PBAT has a composition of 50 / 25.2 / 24.8 mol%. Due to the relatively low thermal and mechanical properties of neat PBAT, PLA is often used as a blending partner to increase the tensile strength, to reduce elongation at break as well as to improve thermal

stability of PBAT [4] [12] [15]. The mechanical properties and the appearance of such polyester blends are similar to LDPE.

The spectrum of Bioflex S6510 shows PLA blended with a relative large amount of aliphatic polyester PBS (more than 20 wt%) and the presence of inorganic additives. Addition of PBS decreases the stiffness of PLA and improves elongation and impact strength behavior [9] [11] [16]. Literature [10] [16] [17] specifies that 20 wt% of PBS in PLA/PBS blend improves positive mechanical properties and provides a good compatibility between PLA and PBS.

Mater Bi CF06A is also an aliphatic-aromatic copolyester based on terephthalic acid and butanediol. The aliphatic acid component is sebacic acid. The composition of the PBSeT is 49.8 / 22.3 / 27.9 mol%. The PLA content in the blend is only about 3 wt%.

Table 3: Chemical composition of investigated bio-plastics determined by ¹H-NMR spectroscopy

Trade Name	Polymer
Mirel P4001	Copolymer P-3-HB/P-4-HB
BioFlex A4100CL	PLA-PHB-PEG-Blend
BioFlex F1110	PBAT-PLA-Blend
MVera B5011	PBAT-PLA-Blend
BioFlex S6510	PLA-PBS-Blend
MaterBi CF06A	PBSeT

3.3 Mechanical properties

Selected results of mechanical testing are summarized in Figures 1, 2 and 3. The thickness of the tested films was 100 µm. To investigate the influence of process-induced stretching, film samples in parallel and perpendicular to the extrusion film direction were taken. The investigated bio-plastic films can be divided into two groups. The first group includes bio-based polyesters such as PLA (Ingeo 4043 D), PHB (Mirel P4001) and PLA-based blends (Bioflex A4100CL, Ecovio T2308) and BioPA 11 (Rilsan BESNO TL) and CP (Cellidor 300-13), which have relatively high elastic moduli, high strength and low elongation of break. According to the bioplastic type, the mechanical properties can be similar to the standard plastics, such as PP, PS or amorphous PET and they have the potential to substitute them in specific applications. From these materials, packaging films, cups for cold drinks, plastic bags or catering products are primarily manufactured. Some of these bio-plastics, such as Ingeo PLA or Ecovio T2308, have food regulatory approval and can be used as a single or multi-layer film for thermoformed food packaging depending on the required permeability values.

The second group includes products with low elastic modulus, high elongation of break and high toughness at low temperatures, such as Bioflex F1110, B5011 and Mvera MaterBi CF06A. This flexibly bio-blends are generally biodegradable in an industrial composting facility and can substitute conventional low density polyethylene - LDPE in numerous short-lived packaging applications. The main market for these plastics are currently thin fruit and vegetable bags, compostable garbage bags, carrier bags or bubble wrap for packing shock-sensitive items. Due to the favorable characteristics and in combination with other polymers, the use of these low-temperature ductile bio-plastics as packaging for frozen foods is conceivable. Another potential field of application can be breathable and waterproof "backsheets" films, which are used for example in diapers or feminine hygiene application.

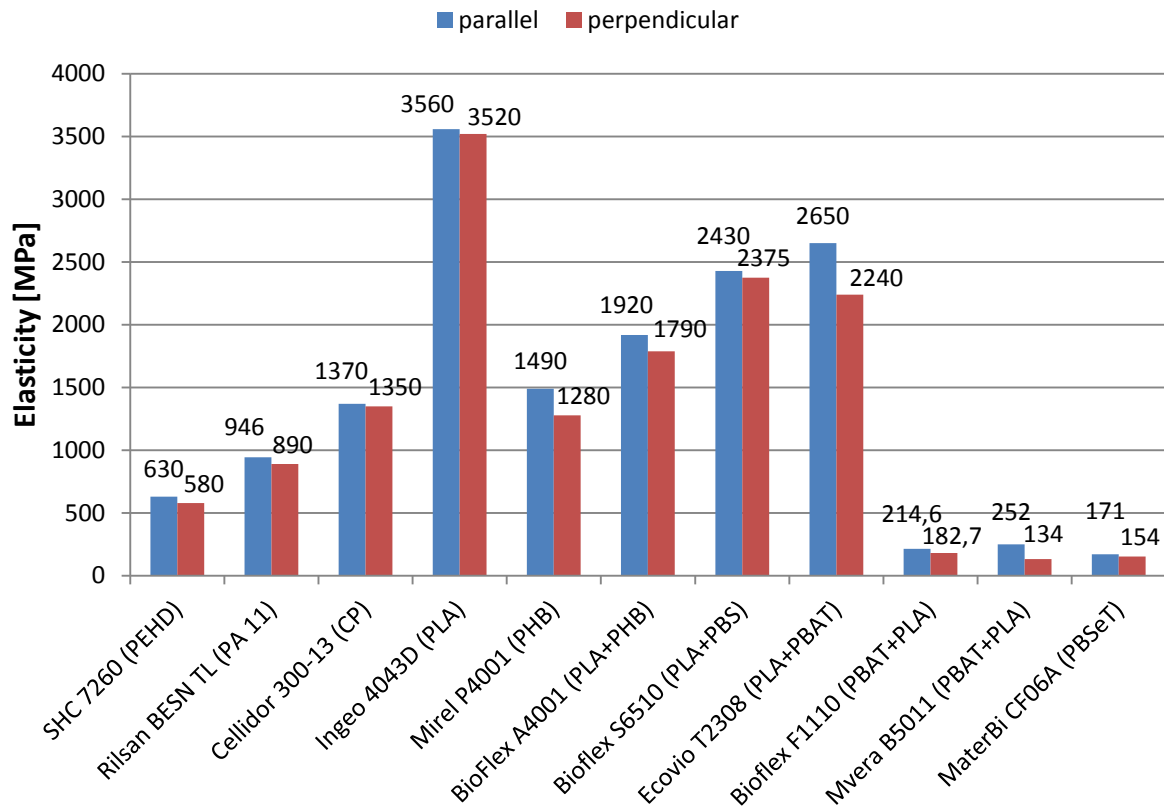


Figure 1: Elasticity of the investigated bio-polymers determined according to DIN 527-3 parallel und perpendicular to the direction of extrusion. Thickness of the tested films is 100 μm .

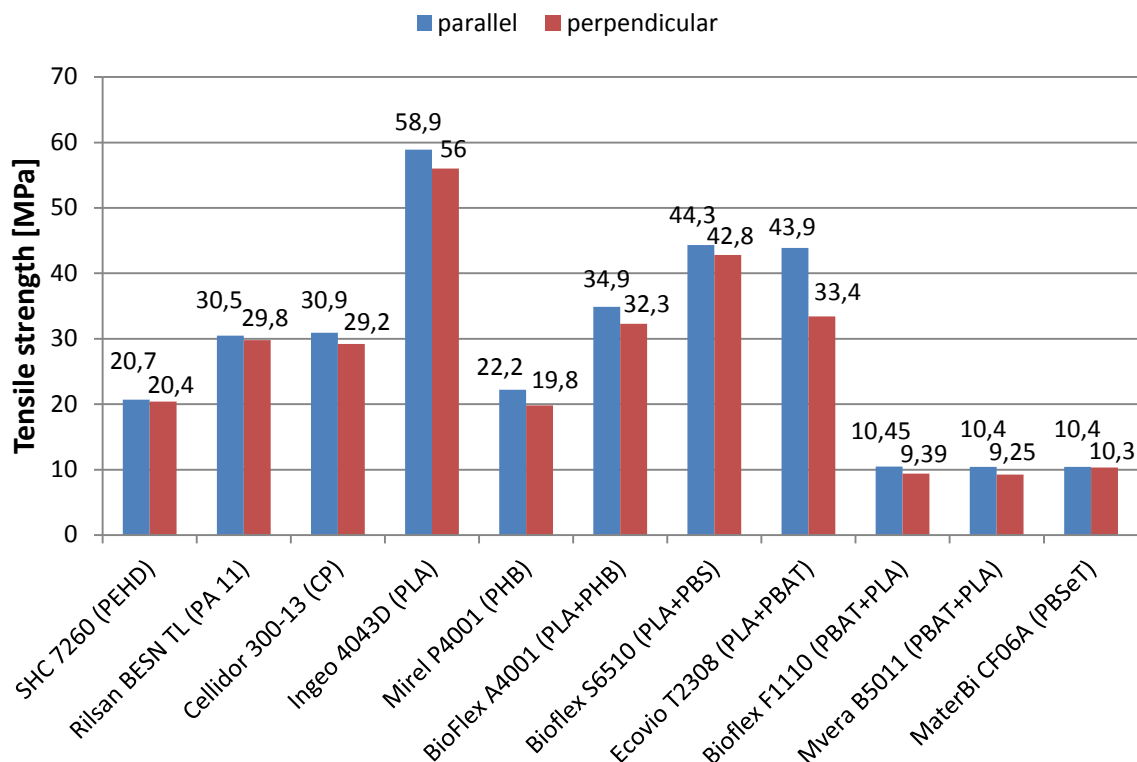


Figure 2: Tensile strength of the investigated bio-polymers determined according to DIN 527-3 parallel und perpendicular to the direction of extrusion. Thickness of the tested films is 100 μm .

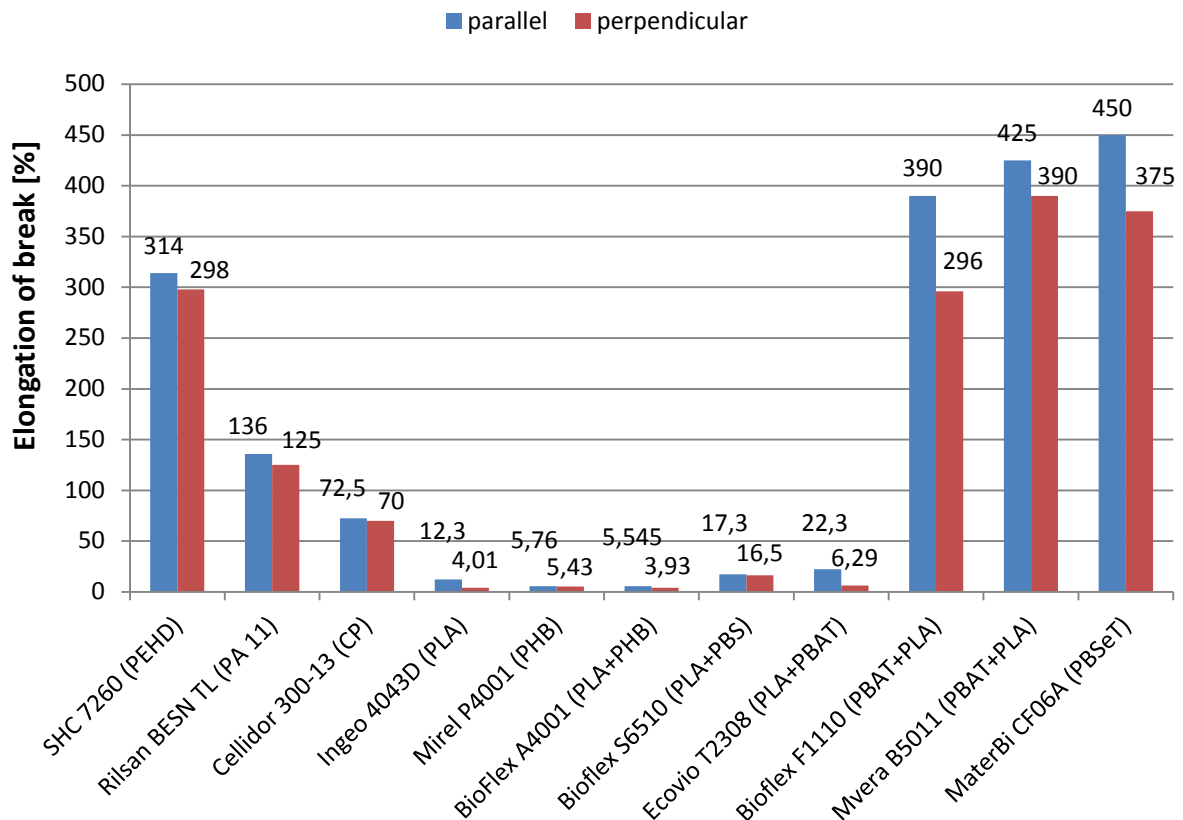


Figure 3: Elongation of break of the investigated bio-polymers determined according to DIN 527-3 parallel und perpendicular to the direction of extrusion. Thickness of the tested films is 100 μm .

3.4 Results of contact angle measurement

Figure 4 shows the calculated total surface tension and the dispersive and polar components by contact angle measurement. The surface tension values vary between 22.52 mN/m (Bioflex F1110) and 51.83 mN/m (Bioflex A4100CL). In general, the greater the surface tension of the plastic surface in relation to the liquid surface tension is, the better the wettability is and the smaller the contact angle is. For a permanent connection between a liquid and a substrate surface, the surface energy of the substrate should exceed about 2-10 mN/m the surface tension of the liquid [23]. The overview of the necessary surface tension for adhesion with usual liquids is shown in Table 4:

Table 4: Absolute values of the surface tension of usual fluids [23]

Colors / Adhesives	Surface tension [mN/m]
UV printing color	48-56
Water-based printing color	50-56
Coating	46-52
UV adhesive	44-50
Water-based adhesive	48-56

The practice shows that the polar component of the surface tension is crucial for the adhesion. Adhesion is higher when the polar components of the surface tension of printing color, coating medium or adhesive and the ones of the substrate surface are equal [22]. The measured values in Figure 4 show that most plastics (except Bioflex A4100CL) will have moderate or even low wettability with UV-/water based printing colors and adhesives due to their relative low total surface tension and less polar components. In case of very low total surface tension, such as in Bioflex F1110, Mvera B5011 and MaterBi CF06A, can even be assumed that the printability and gluability will be weak. With Mirel P4001 and Bioflex A4100, a good printability can be expected with water based printing colors due to the higher polar components of the surface tension. In order to achieve better wettability of the film surfaces, the application of surface modification by corona or plasma treatment can be useful.

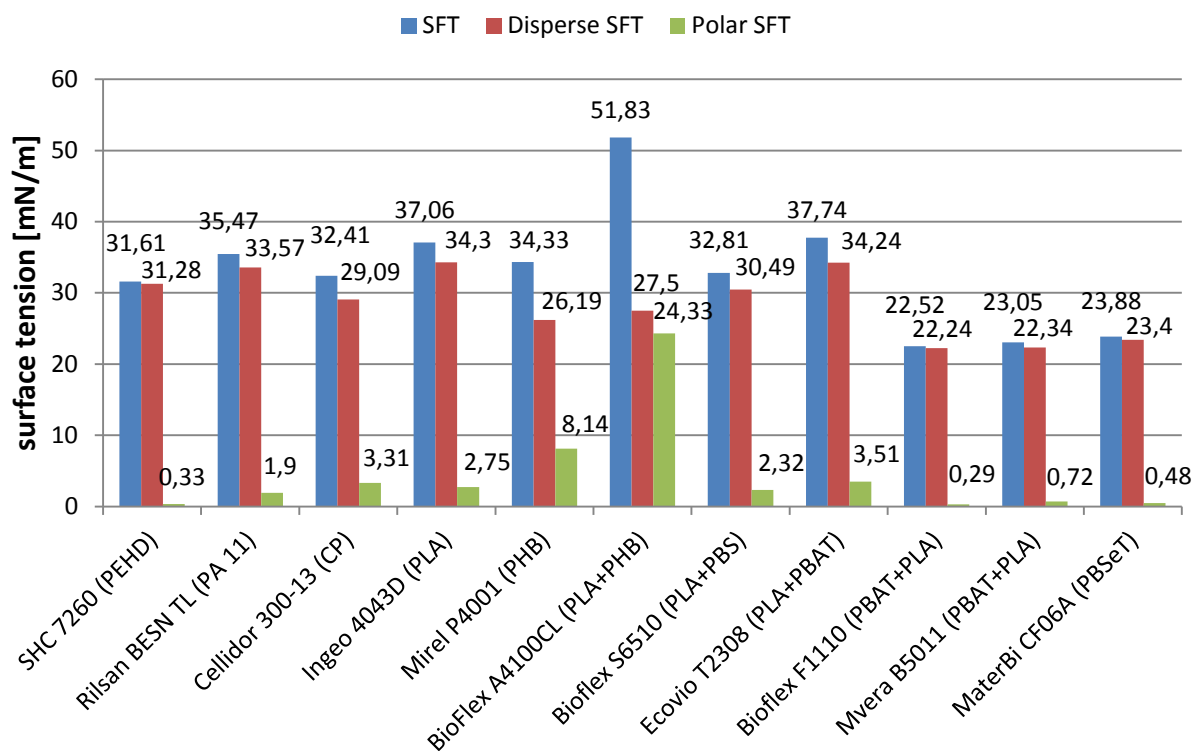


Figure 4: Total, polar and disperse surface tension (SFT) of investigated bio-polymers determined according to the method of Owens&Wendt, Rabel und Kälble.

The result of BioFlex A4001 is noticeable. Both the total surface energy and the polar component of the surface energy differ significantly from other bio-plastics. One possible reason for this finding is a high PEG content in the material (approx. 10 m%), which was detected by NMR analysis. A high proportion of PEG can cause an increased affinity to distilled water, which was used as the test fluid. Due to this, the water drop spreads out on the surface and the determination of the contact angle is erroneous [14]. This can have negative consequences for the printability of this bio-plastics.

4. CONCLUSION

In this study, eleven market-relevant bio-plastics were investigated in terms of their thermal and rheological properties, chemical composition and processability in the extrusion process. Furthermore, the mechanical properties were determined by a tensile test and the potential printability and gluability of the films were evaluated qualitatively on the basis of measured surface tension. The structure determination by FT-IR and ¹H-NMR spectroscopy showed that a lot of offered bio-plastic products are biodegradable polyester and bio-based polyester blends based on PLA or PBAT. Overall, Green HDPE, bio PA 11, CAP, PLA, PLA (-PHB, -PBAT, -PBS) blends, PHB and PBSeT could be investigated and could be processed as films in good quality without chemical additives or modifications. The analysis of mechanical properties showed, that there are stiff (PLA, PHB, PLA-PBAT blends) and flexible (PBSeT, PBAT-PLA blends) bio-plastics. As a result, they could replace the conventional plastics, such as LDPE, HDPE, PP, amorphous PET or PS. The measurement of the surface tension and its dispersive and polar components made apparent the potential printability and gluability of the films. Based on the available knowledge, it can be predicted that the printing, adhesive bonding or coating of the bio-plastic films could be problematic without plasma or corona treatment process. In summary, it can be stated that, by means of this extensive study, a solid knowledge base, regarding extrusion processing of bioplastics, could be created. In the near future, this information will successfully help to answer several questions about the processing industry.

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CONTACTS

Dipl.-Ing. Rostislav Svidler
Dipl.-Chem. Tobias Hartmann
Dipl.-Wi.-Ing. Sebastian Buschbeck
Dr.-Ing. Roman Rinberg
Univ.-Prof. Dr.-Ing. habil. Lothar Kroll

rostislav.svidler@mb.tu-chemnitz.de
tobias.hartmann@mb.tu-chemnitz.de
sebastian.buschbeck@mb.tu-chemnitz.de
roman.rinberg@mb.tu-chemnitz.de
slk@mb.tu-chemnitz.de