Melt-Processing of Biodegradable Polybutylene Adipate Terephthalate (PBAT)/ Polyvinyl Alcohol (PVOH) Multilayer Films with Excellent Mechanical and Barrier Properties for Packaging Applications

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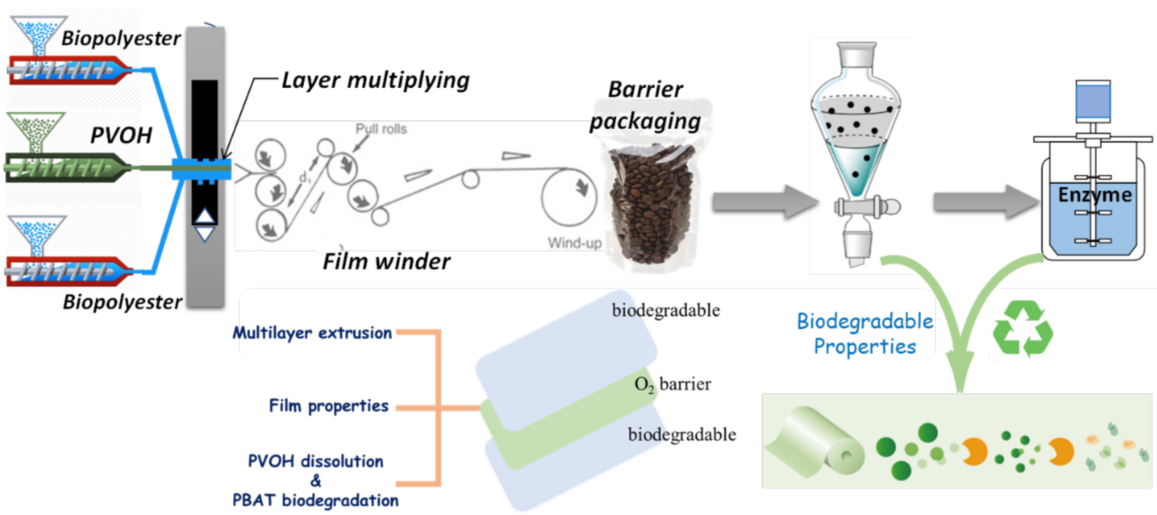
ABSTRACT

Single-use plastic films, made of single or multilayer plastic derived from fossil-based polymers, present serious recycling problems, yet their market share in single-use packaging is on the rise. One major challenge in their recycling is that multilayer film laminations are inseparable, leading to poor properties when recovered and remelted. Multilayer packaging thereby contributes to landfill waste and exacerbates environmental pollution. The combination of water-soluble and biodegradable polymers is presented in this work as a "design for recycling" strategy to obtain high barrier performance and triggered degradable properties in multilayer films, reducing solid-waste problems associated with flexible packaging. Multilayer cast films composed of melt-extrudable poly (vinyl alcohol) (PVOH) as the core layer and poly (butylene adipate terephthalate) (PBAT) as the outer skin layers for packaging applications were produced by the co-extrusion process. Processing conditions were established for the multilayer coextruded cast films that yielded the best combination of properties, performance and potential biodegradability of flexible plastic films. PBAT/PVOH multilayer films displayed excellent adhesion between the two interlayers in the polymer structure. The interlayer microstructure and chemical composition analysis was performed using SEM morphology. It was found that the crystallinity of the PVOH grade plays a crucial role in facile processing and ultimately determined the thermal, mechanical and barrier properties of the co-extruded multilayer films. Moreover, these PBAT/PVOH multilayer cast films exhibited superior barrier properties with an oxygen permeability of 0.89 cc.mil/m^2-day at 0% relative humidity (RH), over monolayer PBAT with an oxygen permeability of 35.0 cc.mil/m^2-day at 0% RH. Finally, warm water dissolution of the PVOH from multilayer films followed by hydrolytic degradation of PBAT layer using 1 M NaOH solution displayed 80 % polymer weight loss after 1 degradation day, demonstrating an ecofriendly way to decompose the material. In the future scope, bioinspired deconstruction of PBAT layers will be studied using enzymes. Given all the components are biodegradable, the final polymer films will also biodegrade in compost conditions, thereby creating another sustainable end-of-life option.



1. Introduction and Motivation

The market share of flexible, multilayer, and heterogeneous plastic packaging is on the rise despite it being the largest unrecycled fraction of all current post-consumer waste. The major challenge is the inability to separate the different layers and components for recycling and reprocessing.[1] Nevertheless, their vital importance in daily life has contributed to serious environmental issues, directing scientific research towards sustainable, eco-friendly polymer films derived from renewable resources.[2,3] Utilizing biodegradable polymers and disposing of them through composting or enzymatic means can reduce the detrimental impacts of plastics, while maintaining the packaged food's quality, safety, and shelf life.[4,5] Poly (butylene adipate-*co*-terephthalate) (PBAT) is a representative example of a biodegradable polymer with great mechanical and processing qualities; most importantly its aliphatic-aromatic contents give combined flexibility-strength for using in packaging materials, mulch films, and in biomedical applications.[6] Investigation on poly(butylene succinate) (PBS) and poly(butylene succinate-*co*-adipate) (PBSA) as potential biodegradation polymer extends huge interest into food packaging applications due to their eco-friendly attributes and wide processing window, making them suitable for extrusion, cast and blown films.[7]

However, the poor barrier properties of the biobased packaging materials limit their widespread application. Poly (vinyl alcohol), PVOH, is well recognized for its extremely low oxygen permeability with a unique role in the packaging industry. When kept moisture-free, PVOH-based extruded films possess exceptionally low permeability (i.e., high barrier characteristics) towards oxygen, nitrogen, and carbon dioxide, and significantly lower permeability than typical polyolefins.[8] Interestingly, PVOH is inherently biodegradable and has excellent aqueous solubility. It has been investigated as the inner-layer or adhesive tie-layer with polyethylene or polypropylene for multilayer packaging films to enhance their barrier properties.[9-11] PVOH was investigated as an interfacial bonding layer to incorporate polyamide 4 and poly(lactic acid) for multilayer packaging with superior barrier properties.[12] It should be noted that the PVOH must be kept isolated from moisture in the air or in the foods within the package to maintain its high oxygen barrier. Once it absorbs water, it swells and becomes significantly more oxygen permeable.

The combination of PBAT and PVOH is of great promise to enable a multi-layer plastic film because both polymers are biodegradable with complementary barrier and mechanical properties. Compared to conventional non-biodegradable thermoplastics like polyethylene, PET and PVC, a new generation of packaging should seek to be greener, safer and more biodegradable. However, processability and performance are often sacrificed to increase biodegradability, and trade-offs between performance and biodegradation must be managed.

The motivation behind this research lies in the hypothesis that a multilayer film structure combining PBAT (outer skin layers) and PVOH (core layer) can synergistically harness the outstanding features of each thermoplastic polymer while offering more sustainable end-of-life options. The production technology explored was melted extrusion through flat film casting die, with multiple extruders and a feed-block for combining individual polymer layers. By integrating the high flexibility, biodegradability, and water resistance of PBAT with the exceptional gas barrier qualities of PVOH, the resulting film can have superior properties under diverse environmental conditions, addressing the limitations of a monolithic film of either material. In future, the use of layer multiplying elements could also enable a well-developed crystalline morphology which increases the tortuosity of the phases such that both barrier and mechanical properties are increased. This morphology will be helpful not only in increasing the properties during use, but also in facilitating layer separation and enzymatic degradation at end-of-life. The future scope of work also proposes to dissolve the PVOH layer and add the remaining polyester layers to enzyme solutions that can rapidly decompose the remaining material into safe, non-toxic monomer solutions disposable with wastewater. The PBAT outer layer is employed to protect the hydrophilic PVOH inner layer from atmospheric moisture, thereby offering an enhanced barrier structure in situations with relatively high humidity.

2. Review of Related Work

There is a vast body of work on multilayered packaging using traditional polyethylene with poly (ethylene-*co*-vinyl alcohol) (EVOH) copolymers as the barrier.[13,14] The focus of these works is to design and optimize the mechanical properties or barrier performance of such multilayers, thereby lacking the overall importance of developing biodegradable and sustainable plastics, since polyethylene is not degradable, nor EVOH water soluble. Jafari *et al.* processed multilayers of EVOH and PBAT through melt mixing with intended applications in food packaging and agricultural film, rather than polyethylene single or multilayer laminated films.[15] Poly(3-hydroxybutyrate-*co*-3-hydroxyvalerate) (PHBV) has been investigated for use in food packaging and found to be stable and safe.[16] Additives to PHBV have also been used such as ZnO-Ag for enhanced shelf-life,[17] SiO_2 for antibacterial properties,[18] and rubber for toughening.[19] However, the use of PBAT/PVOH blends or laminates for oxygen barrier applications has not been investigated. The closest work is the use of extrusion of PLA/PHBV film (90/10 wt.%) with layer multiplying elements.[20] This work found an optimized blend morphology and crystalline structure provided improved gas barrier properties and increased elongation at break without any loss of tensile modulus. The authors concluded that “such ecofriendly films, obtained from blends of two biopolymers, show a tremendous potential for industrial use.” These examples demonstrate the potential for field-degradable packaging films, but optimization to advance packaging specifications is required.

3. Technology Approach

The current technology to produce multilayer film relies on melt extrusion through a flat film casting die, with multiple extruders and a feed-block for combining 5 or more individual polymer layers. Film thickness can be as low as 50 microns and up to a millimeter depending on the intended application. The cast-film lines use uniaxial or biaxial stretching to orient the polymer chains and thin the film gauge. Excellent control over the process variables is required to produce high quality films at economic rates. In this work, multi-layer melt extruded films were processed at the laboratory-scale using a Dr. Collin GmbH Teach-Line® E20 TH coextruder with five 20-mm screw extruders having a 25:1 L/D ratio. Feed-block technology was used to assemble the extrudate layers into the three or five-layer film by way of a 120 mm adjustable thickness slit die and temperature regulated cast film chill rolls, causing the melt to be quickly quenched. Following that, the extruded film was passed through other rollers such as nip roller and winding roll. This technique features unilateral traction along with rapid solidification.

4. Discussion

Fabrication of the Multilayer Films

PBAT Ecovio F2341 (BASF) and PVOH Hydropol 55218 (Aquapak) with mass-flow rate (MFR) of 5.0-11.0g/10 min@190°C, and 10.9g/10 min@230°C were used as the main polymer materials for fabrication of multilayer films. Prior to mixing, PBAT and PVOH neat resins were initially dried in an air-convection oven at 80 °C for 24 h to eliminate any remaining moisture, therefore avoiding formation of bubbles and hydrolysis during processing. The manufacturing process involved two separate approaches for fabricating monolayer and multilayer films. For monolayer PBAT films, the dried resin was added into a screw extruder programmed with five heating zones set at 140, 160, 180, 190, and 190 °C, with the die temperature at 190 °C at 30 rpm screw speed. For PVOH monolayers, a temperature profile of 180, 190, 200, 200, 200, and 200 °C, from the feed to the die section was set and the screw was run at 30 rpm. In contrast, the multilayer PBAT/PVOH/PBAT films required separate feeding of dried PBAT and PVOH resins from different extruders. Specifically, PBAT was fed into extruder 1 and extruder 5 to form the outer skin layers, whereas PVOH was fed using extruder 3 for the core layer. The extruder heating zones were maintained identical to the monolayer setup, screw-speed were varied to achieve optimum throughput in the die. The "melt temperature" of corresponding plastic resins in each extruder were accurately measured using thermocouple at the extruder exit. This refers to the temperature at which the plastic resin fully melts and turns into a flowing fluid, allowing it to be extruded through the die. The schematic of the co-extrusion trial and process temperature conditions are shown in Figure 1 and Table 1.

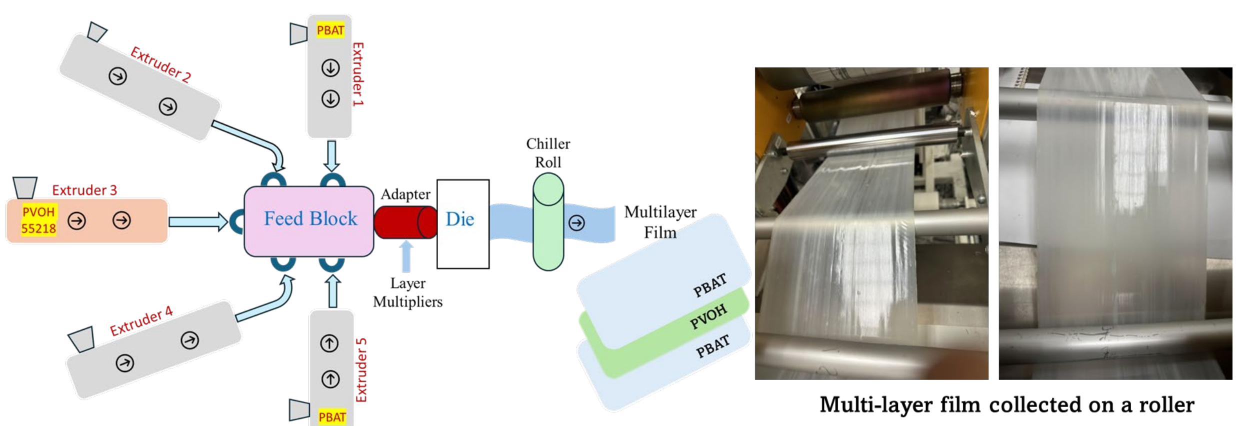


Figure 1. *Schematic representation for the manufacturing process of multilayer films.*

After the temperature profile was set up, the extrusion process produced continuous and stable polymer melts by using a feed block to form and combine each layer forming A-B-A type three-layer structure. This melt was quickly passed through the cooling roller to solidify, forming a cast film. The steel rollers were set at 90-100°C so that continued crystallization during processing would provide enhanced film barrier properties. The resultant film was collected by a take-up device, yielding both single and multilayer coextruded cast films. The desired thickness of the layers was managed by adjusting both the extrusion flow rate of the material and roller take-off speed. The film produced through the co-extrusion cast film die appeared to have no layer-to-layer melt disturbances (melt-fracture or waves) or interfacial instabilities indicating high quality film output.

Table 1. Co-extrusion processing conditions

Polymer		Screw-speed (rpm)	Zone 1 (°C)	Zone 2 (°C)	Zone 3 (°C)	Zone 4 (°C)	Zone 5 (°C)	Zone 6 (°C)	Zone 7 (°C)	Melt-temperature (°C)
PBAT Ecovio	Ext. 3	30	0	140	160	180	180	180	180	162
PVOH 55218	Ext. 3	30	0	180	190	200	200	200	200	173
PBAT-PVOH Multilayer	Ext. 1	20	0	140	160	180	190	190	190	183
	Ext. 3	30	0	180	190	200	200	200	200	173
	Ext. 5	20	0	140	160	180	190	190	190	181

Co-Extrusion

In polymer co-extrusion, it is crucial that the viscosity of the polymers meets the processing temperature, because a difference in melting viscosity might create polymer droplets, encapsulation and interfacial instability. To determine the proper processing temperature profile for the selected PBAT and PVOH resins, complex viscosity and modulus curves were generated using a TA Instruments ARES-G2 parallel-plate rheometer (25 mm parallel plate, gap: 1.5 mm, 5% angular strain). Figure 2a-b illustrates the oscillation frequency sweep curves for the two commercial melt-extrusion grades of PBAT Ecovio and PVOH 55218 at different extrusion processing temperatures. From the results, the optimal polymer viscosity of both PBAT and PVOH matching under typical oscillating shear stress in a rheometer i.e. angular frequency of the oscillation, was achieved at a melt temperature of 180°C. Therefore, the optimal temperature range was determined to be between 180 to 200 °C for co-extrusion processing. By adjusting these polymers' melt temperature or shear rate (screw speed) to more closely resemble viscosity values, film instabilities during processing can be prevented. Also noticeable in the rheological result is the sharp upturn in PVOH complex viscosity and storage modulus at low frequency, particularly at the higher temperature. This is indicative of oxidation of the polymer as it is held at melt temperatures in the air-filled rheometer chamber. Future work will investigate the thermal stability of PVOH at processing conditions and explore the need for inert atmosphere processing.

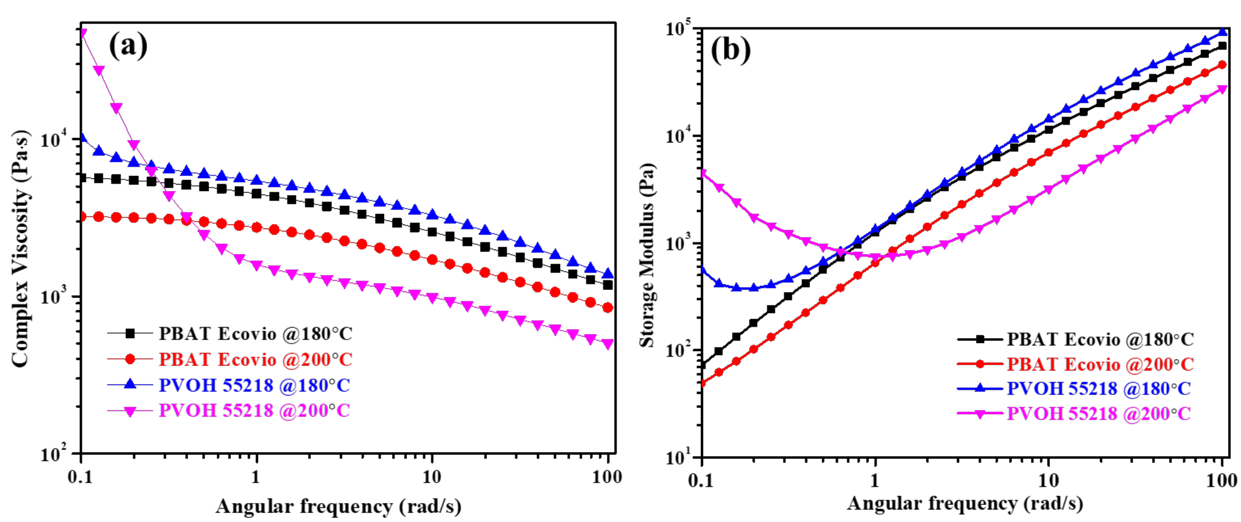


__Figure 2.__ Oscillation frequency curves with (a) viscosity and (b) storage modulus of PBAT and PVOH resins at various processing temperatures.

Multilayer Microstructure

The cross section of the 3-layer multilayer film was examined at the cryofracture surface by JEOL JSM 7401F scanning electron microscopy (SEM) operating at 5 kV accelerating voltage. The samples were adhered to aluminum stubs followed by surface gold sputtering. As shown in Figure 3a, the obtained images show nearly flat and distinct boundaries between the PBAT and PVOH layers. The total average thickness of the multilayer films varies between 100-130 μm. The microstructure of both PBAT and PVOH surface were uniformly smooth without any holes. No delamination was observed between polymer interlayers, even though the cryo-fracture technique exposed them to significant levels of bending stress. This substantiates the excellent bonding or adhesion between the two phases, due to the chemical affinity between ester carbonyls in PBAT and hydroxyl groups in PVOH.[13] While using 50:50 polymer blend as tie-layer to favor better interfacial adhesion, the contents of PVOH core layers increased in the multilayer structure and thicker PVOH layers were observed in some areas of the film (Figure 3b).

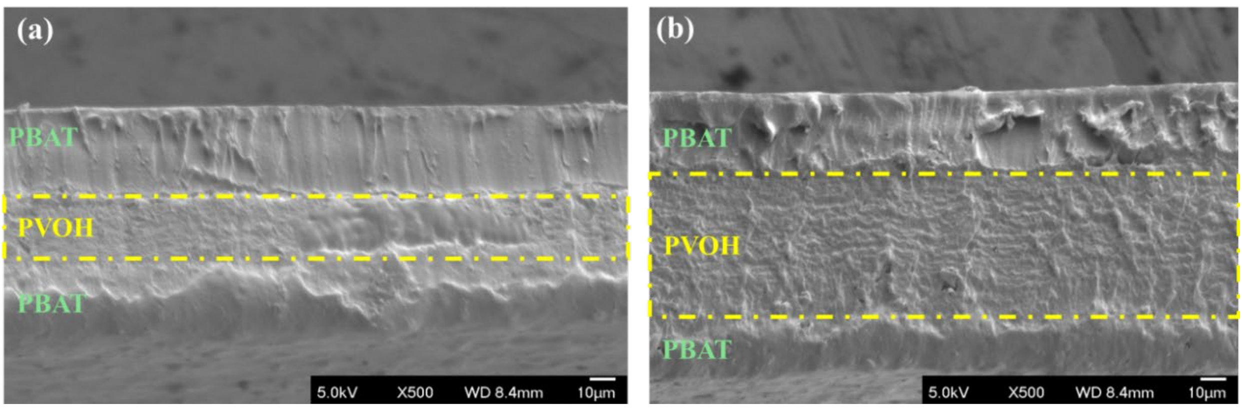


__Figure 3.__ Cross-sectional micrographs of the multilayered films at different film locations captured using SEM: (a) 3-layer (edge), and (b) 3-layer (middle).

Mechanical Properties of the Multilayer Films

To preserve the food's integrity inside the container and withstand shipping and handling, packaging materials must have sufficient mechanical strength and ductility. Figure 4 shows the mechanical properties of PBAT and PVOH monolayers and multilayer films. The specimens were tested using the Instron 5900 series universal tensile tester following ASTM D882-18 with 50 mm gauge length and a test speed of 50 mm/min. Both PBAT and PVOH monolayer films exhibited plastic deformation with a necking and strain softening phenomenon. PVOH tensile strength (TS) is much higher than PBAT films, while its elongation at break (EAB) is lower. In contrast, PBAT-PVOH multilayers experienced a brittle fracture under external forces with no strain softening. The tensile strength of the multilayer was retained but elongation at break drastically dropped. PBAT-PVOH films exhibited the TS and EAB values of 43.3 ± 3.4 MPa and 78.0 ± 3.5 %, respectively.

The comparison of calculated modulus clearly illustrates that the combination of PBAT and PVOH resulted in good interfacial interaction between the skin and core layers, mediated by the dipolar functional groups. While PVOH offered high modulus and low ductility, PBAT offers better flexibility in the multilayer. Nevertheless, the PBAT-PVOH multilayer film still exhibits higher mechanical strength and toughness than other reported biodegradable multilayer films.[12]

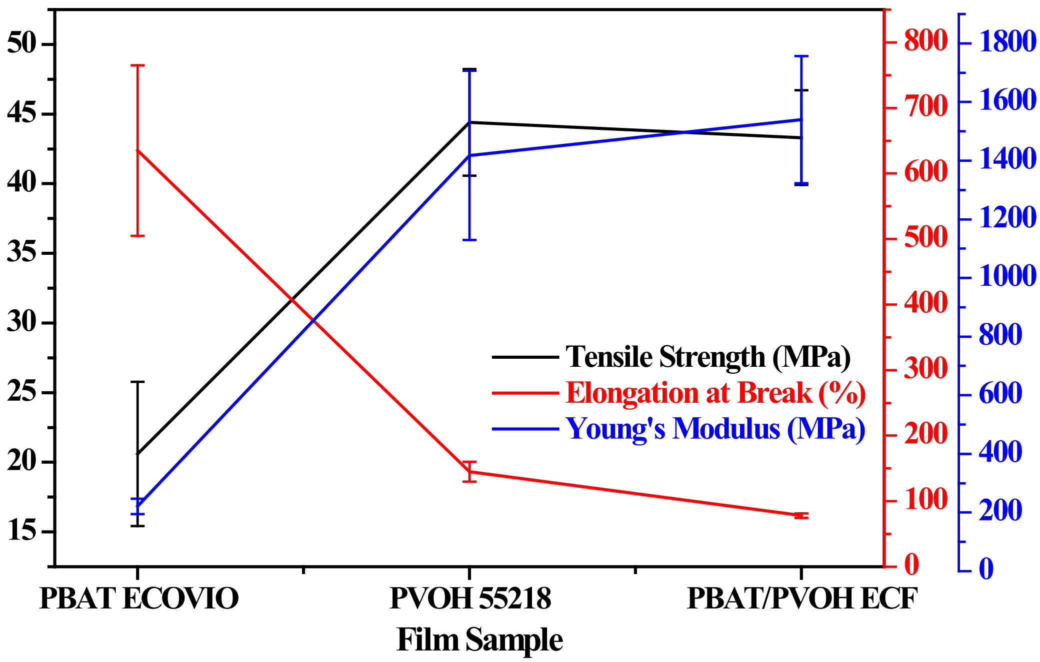


Figure 4. *Tensile properties of monolayer PBAT and PVOH films versus PBAT/PVOH three-layer extruded cast films.*

Barrier Properties of the Coextruded Films

Oxygen permeability is a key factor in packaging applications, since it plays a fundamental role in food preservation and expanding their shelf-life. Table 2 lists the measured barrier properties for extruded polymer films measured using MOCON OX-TRAN® 2/22 series oxygen permeability tester at 23°C and 0% RH. PVOH and PBAT films had oxygen permission rate (OPR) values of 0.25 cc.mil/m^2-day and 35 cc.mil/m^2-day at 0% relative humidity (RH), respectively. As expected, the hydrogen bonds between the polymer chains together with the crystalline structure provide PVOH with a low oxygen transmission rate. PBAT-PVOH multilayer also demonstrate significant barrier performance enhancement over monolayer PBAT (outer skin layer) with an OPR of 0.89 cc.mil/m^2-day at 0% RH. The PBAT skin layers provided protection to the PVOH core layer from atmospheric moisture and the formation of hydrogen bonds at the interface of PVOH and PBAT significantly limited the diffusion of oxygen molecules, resulting in the higher oxygen barrier performance of the multilayer films. Overall, the PBAT-PVOH multilayer films showed excellent resistance to oxygen transmission compared to similar previous reports on biodegradable films and thereby, could be a valid sustainable substitute for common packaging materials.[11,12,15]

Table 2. Barrier properties and parameters of co-extruded PBAT-PVOH polymer films

Polymer Films	Gauge (mil)	Sample Area cm²	OTR (cc/m².day) 0% RH, 23 °C	OPR (cc mil/m²-day) 0% RH, 23 °C
PBAT ECOVIO	1.5	50.0	22.751	35.036
PVOH 55218	2.1	5.0	0.120	0.252
PBAT-PVOH Multilayer	3.5	5.0	0.254	0.890

4. Hydrolytic Degradation and Future Scope

The degradable film was subjected to warm water dissolution (50-55°C for 3-4 hr.) to remove the PVOH from the extruded multilayer films. This was followed by hydrolytic degradation of remaining dried PBAT layer using 1 M NaOH solution (pH 13.5-14.0), which displayed 80 % polymer weight loss after 1 day of degradation, demonstrating the potential for use in field-degradable packaging films (Figure 5).The future scope study will investigate the option of enzymatic degradation via addition of phosphate buffered saline solutions with esterase enzymes such as lipase or cutinase, which have previously shown the ability to decompose polyester and PBAT films.[21] The high surface area of the polyester's layers on exposure to enzyme solutions (different concentrations) will rapidly decompose the remaining material into safe, non-toxic monomer solutions disposable with wastewater, or preferably for recovery of the monomers, thereby eliminating the need for treatment of solid waste.

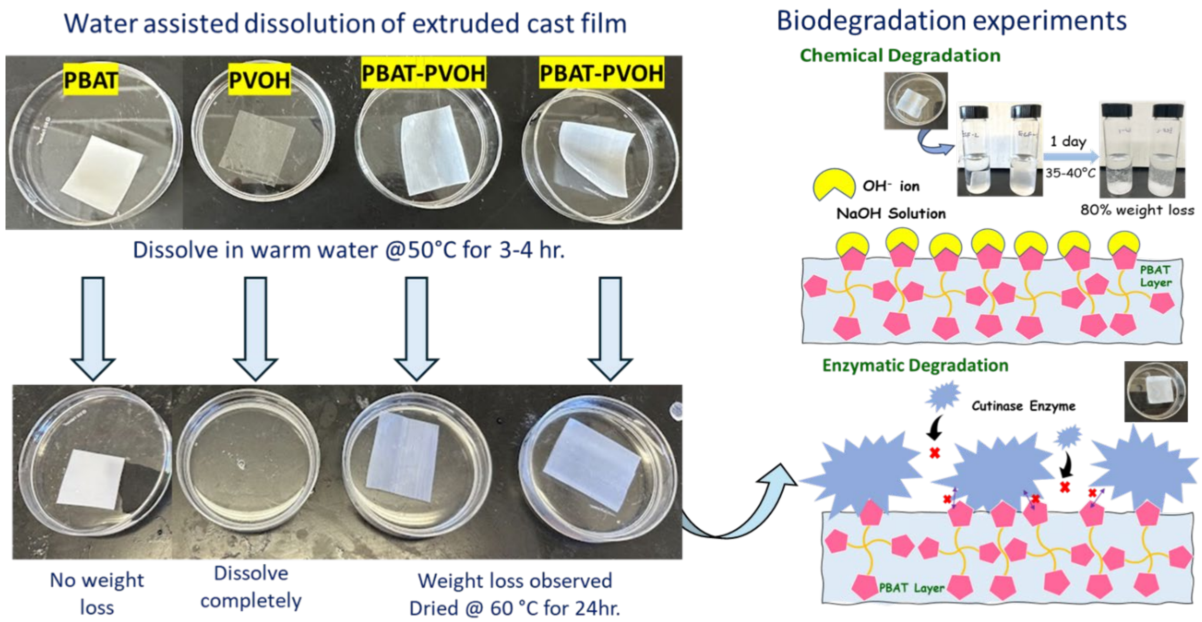


__Figure 5.__ Multilayer aqueous dissolution and biodegradation as the recycling options.

5. Conclusions & Recommendations

Multilayer films consisting of a melt-extrudable poly (butylene adipate terephthalate) as the outer skin layers and poly (vinyl alcohol) as the core layers were successfully produced through the multilayer co-extrusion process on a laboratory-scale extrusion line. Rheological characteristics of PBAT and PVOH resins exhibited similar viscosity and melt flow profile at the chosen processing temperature. The cross-sectional microstructure morphology demonstrated good interlayer adhesion, likely due to strong hydrogen bonding between the polymers. The mechanical characteristics of PBAT-PVOH multilayer films include a tensile strength of 43.3 MPa, a modulus of elasticity of 1540 MPa and an extension at break of 78%. Additionally, the oxygen barrier properties of the films (OTR of 0.25 cc/m^2-day) showed noteworthy enhancement over monolayer PBAT and PVOH with OTR of 22.7 cc/m^2-day and 0.12 cc/m^2-day at 0% RH, respectively. Additionally, aqueous dissolution of the PVOH from multilayer films followed by hydrolytic degradation of PBAT layer using 1 M NaOH solution showed 80 % polymer weight loss after 1 day of degradation. Future research scope will investigate the enzyme deconstruction of the PBAT layer from multilayer plastic films, creating another sustainable end-of-life option.

This research described the design of novel packaging barrier films using PBAT/PVOH with lower environmental footprints and competitive properties to incumbent materials, as well as state-of-the-art film extrusion processing technologies and water-assisted separation of polymer layers. These developments have the potential to offset the use of virgin polyolefins in the $30 billion market for flexible films. This research and development work will produce a multilayer film standard to meet needs in the food and commodities industries, provide a path for recovery of material value, and reduce demand for primary polyolefins through advanced technologies for circular packaging aligning with global sustainability goals.

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