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Sustainable Labware Solutions: A Study of Biodegradable Modified Polypropylene for Sterilization-Resistance

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REVIEW ARTICLE

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Abstract

This study investigated the impact of incorporating a 1-3% by weight of biodegradable additive into polypropylene (PP) using twin-screw extrusion, followed by injection molding to prepare test specimens. The effects of the additive on the rheological, thermal, mechanical, and optical properties of PP were investigated, along with the impact of two common sterilization methods: autoclave (121°C, 15 min, 0.11 MPa) and electron beam (E-Beam, 50 kGy) irradiation. The results showed that the biodegradable additive minimally affected the flow and melting properties of PP while enhancing the rigidity and heat resistance. Autoclave sterilization, a moist-heat treatment, significantly improved flexural strength and heat distortion temperature (HDT) by acting as an annealing process. E-Beam irradiation notably increased tensile strength due to radiation-induced crosslinking but marginally decreased impact strength. However, irradiation also induced oxidative degradation, impacting color stability and lowering surface energy. Despite these changes after treatments, the biodegradable additive showed potential in maintaining PP's mechanical properties integrity and sustainability, making it a viable option for eco-friendly labware development. This study highlights the promise of the biodegradable

additive for enhancing key properties of PP labware and creating more sustainable plastic labware without compromising product durability and sterilization requirements.

Keywords: Polypropylene, Biodegradable additive, Sterilization, Laboratory plasticware, Surface energy

1. Introduction

The escalating demand for laboratory plasticware products is persistently influenced by urbanization and evolving consumer preferences. The COVID-19 pandemic has intensified consumers' health concerns, requiring hygienic products and services. In addition, to prevent contamination and spreading of infection, the demand for disposable devices has increased, resulting in greater plastic consumption over alternative materials, particularly in microbiology labware and medical devices. Over the past few decades, disposable plastics have become crucial components in research and healthcare laboratories. Traditionally, laboratory glassware has been extensively used due to its durability, transparency, and reusability. Nevertheless, it is heavy, difficult to handle, and prone to breakage during use, cleaning and sanitizing processes, ending in higher overall costs. On the other hand, plasticware is lightweight and less fragile than glassware. When shipped, it necessitates reduced protective wrapping, generates less packaging waste, and may create a lower carbon footprint than glassware. Consequently, glassware has largely been phased out and replaced by single-use plastic labware across a wide application range. It has been reported that the single-use plastic waste equivalents to approximately 70-100 kg per scientist annually (Krause et al. 2020)[8]. Generally, microbiological laboratory wastes are categorized as biohazardous-waste and must be disposed of through specialized waste management systems. In nonclinical laboratories, most plastic wastes comprise thermoplastics such as polypropylene, which are ideally recyclable. Unfortunately, due to the absence of adequate collection and recycling systems, and insufficient infrastructure, a large amount of plastic labware waste ended up in landfills or incinerated, causing pollution and detrimental effects on the environment and overall ecosystem.

There are varieties of sustainable approaches that are possibly implemented, target products and their applications must be considered since each product requires different requirements. The commodity thermoplastics, including polyvinyl chloride (PVC), polyolefins (polyethylene, polypropylene, and their blends), and polystyrene (PS), take more than 75% of all plastics used in labware and medical device applications (Sastri 2013)[16]. They have different properties making them suitable for specific applications. Nevertheless, due to environmental sustainability concerns of single-use plastics, they are considered to be recycled. This leads to the development of PVC and PS alternative materials to make them recyclable (Lloyd 2004; Jiang et al. 2022)[7,9]. The switching to new materials ideally should meet both performance and price effectiveness. On the contrary, polyolefins have been shown to be more acceptable due to their recyclability but they are not biodegradable. Especially when they are carelessly discarded, they are then ended up uncontrolled pollution to the environment. Sustainable needs have driven interest in eco-friendly alternative materials such as biodegradable polymers, making better biodegradability after being disposed (Sin, Rahman & Rahman 2012)[18]. Polylactic acid (PLA) is the most commonly used biodegradable polymers. Many attempts have studied the use of biodegradable polymers as an alternative material for single-use plastic labware (Freeland et al. 2022) [5]. Recently commercial PLA plastic plate for tissue culture has been launched (Nicole Kelesoglu 2024)[13]. However, biodegradable

plastic has been being more expensive than conventional plastic and facing technical challenges such as brittleness and durability, limiting the application of bioplastics in the market.

Besides, many attempts have been developed to accelerate the degradation of conventional polymers. Such additives claim to retain final product properties with marginal changes to the process. For example, the pro-oxidant additives may comprise metal salts or their complexes (Wiesinger et al. 2020)[21]. Pro-oxidant additive containing (PAC) plastics including oxo-(bio)degradable plastics, are designed to promote plastic degradation through oxidation and other processes (Devalla 2022)[3]. However, they do not entirely degrade under realistic conditions since the additives only promote partial degradation and fragmentation into microplastics. Recently biodegradable additives have been developed to enhance the biodegradation of conventional plastics while avoiding microplastics formation (BioSphere Plastic LLC). [1] Among these, the BioSphere represents a special formulation engineered additive and acts as a catalyst to accelerate the biodegradation rate of plastic. In a microbe-rich environment, microbial colonization, subsequent biofilm formation, and inducing microbial enzyme secretion occur on the substrate surface. The catalyst consists of a unique symbiotic chemistry allowing the naturally occurring enzymes and microbes' microenvironments to break down the physical and molecular structure of the polymer into smaller molecules. The enzyme-additive interaction generated a catalyst complex activating the degradation of polymer chains through sequential biotic pathways: hydrolysis, acetogenesis, and methanogenesis. The microbes then use the smaller molecules for growth and reproduction, which further produce enzymes, utilize the nutrients, and break down those compounds into terminal products like carbon dioxide and methane with small residual quantities of biomass and water. Many studies have been reported about the effect of such additive on the nucleation intensity, growth rate of polypropylene spherulites, mechanical properties (impact strength, yield strength, Young's modulus, and elongation at break) (Mubarak 2018; Mubarak 2022)[11, 12] and degradability under both aerobic and anaerobic conditions (Zafiu et al. 2023)[23]. In addition, there are some commercial plastic labware products using such kind of biodegradable additives such as serological pipets, sample reservoirs, and spatula.

Sterilization is essential for labware in many applications such as microbiology, food industry, healthcare and medical devices. Gamma and electron beam (E-beam) irradiation are commercially proven sterilization technology for single-use plastic labware and medical device products. They are non-thermal sterilization methods using radiation to inactivate microbes with no chemical residual. The use of irradiation sterilization can alter the physical and mechanical properties of polymers due to bond cleavage and cross-linking (Tjong, Li & Li 1998; Makuuchi & Cheng 2011)[10, 20]. For reusable items, autoclaves are the most widely used sterilizers to eradicate microorganisms by using high-pressure steam heated to 121-134˚C with a holding time of at least 15 minutes. The reusable items will most probably be subjected to multiple steam sterilizations before being discarded, plastic labware must have superior toughness and heat resistance. Therefore, the development of plastic labware products ought to focus on both product properties and durability after single or repeated sterilization processes to ensure high-quality products suitable for laboratory use.

To the best of our knowledge, the effect of the BioSphere biodegradable additive on PP polymer after sterilization has not been addressed in scientific literature. Generally, the dosage recommendation by the manufacturer is 1% by weight, as higher concentrations may alter the final products' mechanical and physical properties. For thick products, increasing the additive concentration beyond 1% by weight may accelerate biodegradation (BioSphere Plastic LLC)[1]. Therefore, the present study investigated additive concentrations ranging from 1% to

3% by weight to comprehensively assess their effects on the rheological and thermal properties of the compounded resins. Mechanical and optical properties of PP and PP containing the biodegradable additive before and after treatment with two common sterilization methods were evaluated; autoclave (moist heat sterilization, 121°C, 15 min, 0.11 MPa) and electron beam irradiation (non-heat sterilization, E-Beam, 50 kGy).

2. Experimental

2.1. Materials

Polypropylene homopolymer was used in this study. It has a density of 0.90 g/cm^3 and a melt flow index of 11 g/10 min (2.16 kg/230°C). A biodegradable plastic additive, translucent granule masterbatch, was supplied by BioSphere Plastic LLC-USA (grade BioSphere 201J)[1] . It has a specific gravity of 1.22-1.26 g/cm^3 and a melt flow index of about 45 g/10 min (2.16 kg/230°C).

2.2. Specimen preparation

In this study, melt blending of PP and additive was conducted using a 20 mm co-rotating twin-screw extruder with 40 L/D ratio (Labtech Engineering co., Ltd-Thailand). The loading of the additive was varied from 0, 1%, 2% to 3% by weight, samples were denoted as PP, PP+A1%, PP+A2%, and PP+A3%, respectively. The compounded resins were cooled at room temperature for at least 24 hours to release stress before characterization and preparation in the next step.

Dog-bone and bar shaped specimens were prepared by injection molding machine (Nissei Plastic Industrial injection machine, model PS40E5ASE-Japan) to obtain samples for mechanical properties testing. The extruder temperature was controlled in the range of 198-210°C, screw speed 150 rpm and injection pressure 80-105 bar. Obtained specimens were kept at condition 23°C, 50% relative humidity at least 24 hours before testing properties.

2.3. Post-treatment sterilization

2.3.1. Autoclave sterilization treatment

The dog-bone and bar specimens were exposed to moist-heat steam sterilization using an autoclave (TOMY SX-700) at 121°C for 15 min under a pressure of 0.11 MPa (15 psi). The specimens were then dried in an oven at 70°C for 18 hours before tests.

2.3.2. E-Beam irradiation

The dog bone and bar specimens were electron beam irradiated at High Dose Dosimetry Laboratory, Irradiation Center, Thailand Institute of Nuclear Technology (Public Organization). All samples were irradiated at 50 kGy as the maximum dose to encompass the potential range of radiation doses that might be encountered for sterilizing labware and medical devices. The minimum and maximum doses were in the range of 50.87-52.93 kGy. The dose uniformity was 1.04 (less than 1.5), considered to be within the good range of radiation quantity distribution.

2.4. Characterization

2.4.1. Compounded resin properties

Differential scanning calorimetry experiments were performed on a Mettler Toledo STARe System DSC3+ in a temperature range of 30–250°C at a rate of 10°C/min. The first heating was performed to remove processing history. The first cooling cycle and second heating cycle were shown. Melt flow index (MFI) measurement was made under a specific load of 2.16 kg at 230°C with Melt Flow Tester Zwick/Roell-Germany, according to ISO 1133. Capillary

rheometer (RH7), Rosand (Malvern Instruments) was performed at the measuring temperature of 210°C with the single bore (16 mm/1 mm flat die). The shear rate range of 100-17,000 1/s was conducted, close to the shear rate generated by the injection molding process. The viscosity vs. shear rate plots were reported.

2.4.2. Mechanical properties

Both tensile and flexural tests were measured by AG-X plus Shimadzu Universal Testing Machine (UTM), Japan with a 10 kN load cell. The tensile properties of PP specimens were examined according to ASTM D638 with a speed of 50 m/min. For flexural properties, the UTM was fitted with a standard three-point bending fixture and samples were flexed until breakage at a rate of 13.65 mm/min using a support span of 51.2 mm.

The heat distortion temperature (HDT) was determined using Instron HDT & VICAT Tester (Model HV3), which refers to ASTM D 648. The three-point bending mode was used. The specimen was heated from ambient to 200°C at a heating rate of 2°C/min and under a constant load of 0.455 MPa. The HDT was determined as the temperature at which the specimens reached a deflection of 0.25 mm. Notched izod impact strength of the sample was performed at 23°C by Instron Pendulum Impact Testing (CEAST 9050) according to ASTM D256 using a 1 J pendulum hammer. At least 10 specimens were tested and averaged for each test.

2.4.3. Optical properties

The Colorimetric Spectrophotometer model 4500L HunterLab-USA was utilized to measure the color of specimens. The result was reported in L*, a*, b* values for each sample. The measurement of transmission was tested by Transmission Densitometer model TBX1000, Tobias, USA. % Transmission of specimens was calculated from the equation: $\%T = 10^{-D} \times 100$;

When %T = % Light transmission through sample specimen; and D = Optical density

2.4.4. Surface energy

The surface energy of PP specimens was determined by measuring the contact angle using four liquids, distilled water, glycerol, formamide, and methylene iodide. All chemicals are analytical grade. The drop shape analysis system (Kruss DSA100, Germany, with an analysis software DSA1) was used to measure the contact angle of each liquid on the surface of the sample at 23°C with 50% relative humidity. The FOWKES method was used for surface-free energy calculation.

3. Results and discussion

3.1. Melted resin properties

The melt flow index (MFI) measurements of neat PP and PP-biodegradable additive composites are presented in Figure 1(a). It was found that the MFI of PP was not notably altered by 1-2% by weight of the additive. However, at 3% loading, a slight increase of the MFI was observed, attributable to the higher MFI of the biodegradable additive (45 g/10 min).

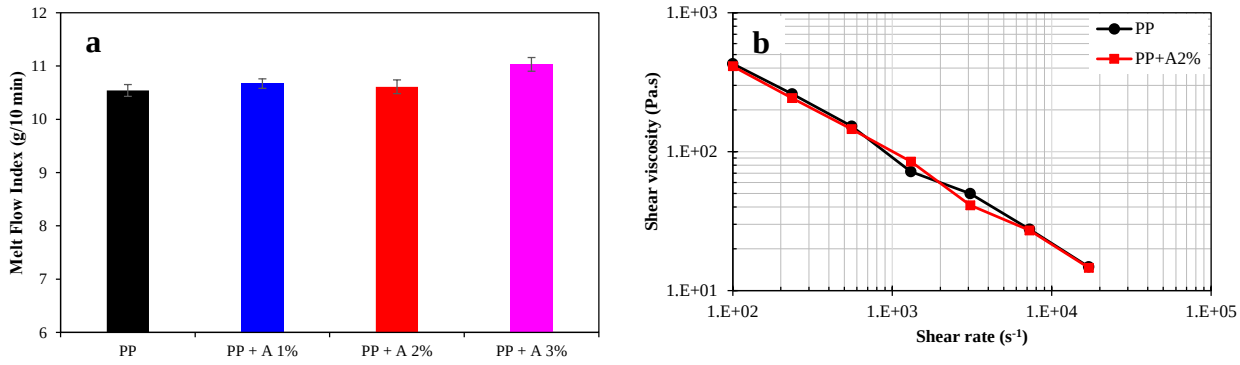

Figure 1 MFI (a) and rheological properties (b) of PP and PP containing biodegradable additives.

Figure 1(b) illustrates the rheological behavior of neat PP and PP containing 2% additive loading (PP+A2%). The flow properties of both materials exhibited nearly identical characteristics across a shear rate range of 100-17,000 1/s. The melted polymers demonstrated entanglement phenomena within the shear rate in the range of 100-1000 1/s, transitioning to local molecular motion at shear rates above 1000 1/s. These results indicate that additive incorporation at 1-2% loading has a negligible impact on rheological behavior and melt flow index of the PP matrix.

Figure 2 displays DSC thermogram of PP and PP containing biodegradable additives. All samples showed a prominent exothermic peak around 120°C. The sharp, well-defined, and nearly identical crystallization peaks for PP and all additive concentrations (1-3%) were observed indicating a uniform crystallization process. The crystallization and melting behaviors of all samples are shown in Table 1. The onset temperature ($T_{c,onset}$) of pure PP was higher (124.5°C) than all PP-containing additives (123.2-123.7°C) suggesting a minimal nucleation effect of the additive. A similar was observed in the crystallization temperature (T_c), additive-containing samples displayed marginally lower T_c (119.4-119.6°C) than the pure PP (120.3°C). This suggests that adding the biodegradable additive (up to 3%) does not significantly affect the crystallization behavior of PP.

An endothermic melting peak of all samples was similarly observed around 165-170°C, representing the crystal structure and melting temperature of PP remains largely unchanged by the additive. All samples showed their melting temperature (T_m) with tiny variations (±1°C) in the range of 165.6-167.4°C indicating that crystal perfection remains largely unchanged as shown by similar melting peaks in the DSC curves. It was found that the heat of fusion (ΔH_m) of the neat PP was higher (96.1 J/g) and slight variations were observed in additive-containing samples (86.8-90.5 J/g). In addition, the incorporation of the additive led to a small reduction in the degree of crystallinity (%X_c) of PP from 46% to 42-43% suggesting that the additive slightly hinders crystallization formation by reducing the free volume (Samper et al. 2018)[15] as increasing the concentration but not significantly.

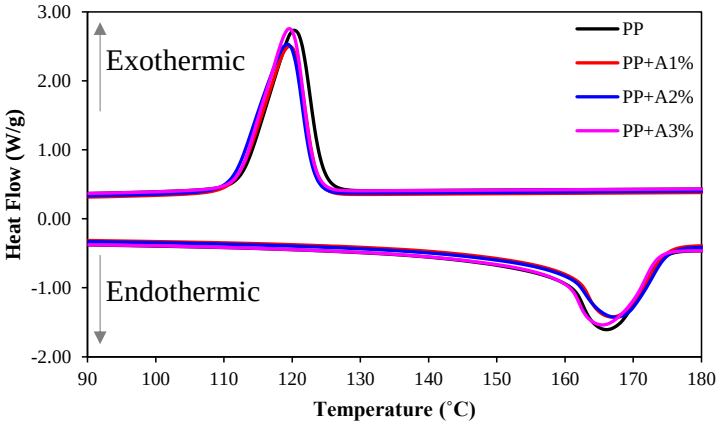

Figure 2 DSC plot showing the first cooling cycle and second heating cycles for the PP, PP+A1%, PP+A2% and PP+A3%.

Table 1 DSC results of PP blends with biodegradable additive.

	$T_{c,onset}$ (˚C)	T_c (˚C)	T_m (˚C)	ΔH_m (J/g)	$\%X_c$
PP	124.5	120.3	166.2	96.1	46%
PP + A 1%	123.7	119.5	166.9	86.8	42%
PP + A 2%	123.2	119.4	167.4	88.7	42%
PP + A 3%	123.3	119.6	165.6	90.5	43%

These results illustrate that the biodegradable additive has minimal impact on PP's thermal properties indicating good compatibility between PP and the additive, incorporating of the additive causes only minor changes in thermal properties and no additional thermal transitions or significant shifts in peak temperatures. The preserved thermal properties indicating the processing and end-use performance should remain largely unaffected.

3.2. Mechanical properties

Figure 3 shows the mechanical properties of PP and PP containing biodegradable additive before treatment (Origin, black bars) and after post-treatment with two common sterilization methods; autoclave (Autoclave, blue bars) and E-Beam irradiation (E-Beam, red bars).

The flexural strength elaborates the material's ability to resist deformation and the Young's modulus represents the stiffness or rigidity of materials. In non-sterilization series (Origin), it was observed that the additive boosted up 11% flexural strength from 55 to 62 MPa for PP and PP with 3% additive, respectively. A slight increase in Young's modulus was observed as additive concentration increased. This indicates that additive makes PP more difficult to deform and stiffer than neat PP. In addition, the HDT refers to the ability of the polymer to remain stiff under a constant load and elevated temperature, which is used to assess the heat resistance of the polymer (Wong 2003)[22]. The HDT non-linearly improved 6% from 99˚C to 105˚C at 3% loading suggesting that the thermal properties of PP can be improved by incorporation of the additive. In theory, the higher crystallinity leads to higher HDT. However, the crystallinity seems to not play a vital role in HDT improvement in this study (according to the DSC results in Table 1). A slight reduction in crystallinity was affected by the incorporation of the additive but the HDT became higher as increasing the additive contents. Alternatively, it can be explained by the flexural strength and Young's modulus results. The additive boosts rigidity, and thermal stability, further improving HDT and making PP better heat resistant.

Flexural strain, tensile strength, and impact strength were marginally changed across all loadings.

After post-autoclave sterilization, all samples became higher in flexural strength and HDT than their origin samples. Tensile strength, flexural strain, and impact strength were marginally increased across all samples. After autoclaving, the flexural strength of PP was notably enhanced from 55 MPa to 61 MPa, showing a 10% increment. At 2-3% additive loading, the flexural strength showed a 15% improvement to 64 MPa. The flexural strain was partially increased after treatment but declined with increasing additive loading. Particularly, the HDT of neat PP was improved from 99˚C to 104˚C after autoclaving. It became more pronounced at higher additive loadings. The higher additive concentration, the higher HDT was yielded; achieving at 115˚C with 3% loading representing 16% HDT improvement. This indicates that the autoclave treatment does not distort the additive's behavior. The autoclave treatment is a kind of thermal treatment and acts as an annealing effect. This allows polymer molecular chains to rearrange, thus achieving better mechanical properties (Han et al. 2010)[6]. It indicates that the moist-heat treatment increases the samples' rigidity.

For the E-Beam irradiated series, flexural strength, tensile strength, and HDT were found to rise after irradiation and became more pronounced with higher additive concentrations. After E-Beam irradiation, a distinct improvement in flexural strength of the neat PP and PP-containing additives. For pure PP, the flexural strength was increased from 55 MPa to 62 MPa showing 13% improvement after irradiation. Similar trends were found in PP-containing additives. The highest flexural strength was achieved at 65 MPa (18% improvement) at 3% loading. Irradiation led to a decline of the flexural strain with increasing additive loading. Tensile strength improvement was solely impacted by the radiation. All irradiated samples displayed up to 50% tensile strength improvement (28-29 MPa) as compared to the untreated PP (PP origin, 19 MPa), suggesting that the radiation solely contributes a positive benefit on tensile strength and maintains its advantage across all additive loadings. The impact of the additive is almost negligible in this case. It could be explained that the radiation may influence the cross-linking nature of PP and contribute positively to the tensile strength (Chaudhari et al. 2007). [2] The influence of both sterilization treatments on Young's Modulus of PP was not observed as shown by relatively similar Young's Modulus values across all samples. A slight increase was observed with higher additive concentration. The radiation raised the HDT of the neat PP by 11% (from 99˚C to 110˚C), reflecting the restriction of the chain mobility and lower deformability as a consequence. However, only marginal HDT improvement appeared in PP-containing additives after irradiation. Their HDT values still increased as increasing the additive amount from 105˚C to 109˚C for PP containing 1% and 3% additive, respectively. Radiation caused consistently lower impact strength in all samples, especially in PP-containing additives. Impact strength seems to be partially compensated by increasing additive concentration. This indicates that the radiation may not only interfere with the PP but also interrupt the additive such as breaking of bonds in the polymer chains of the additive's components. Unfortunately, the composition of the additive is unrevealed. The real mechanisms and behaviors of such additives are still unclear. It can be noticed that the additive itself can be disturbed by the radiation leading to impact strength loss which can be compensated by increasing the amount of additive.

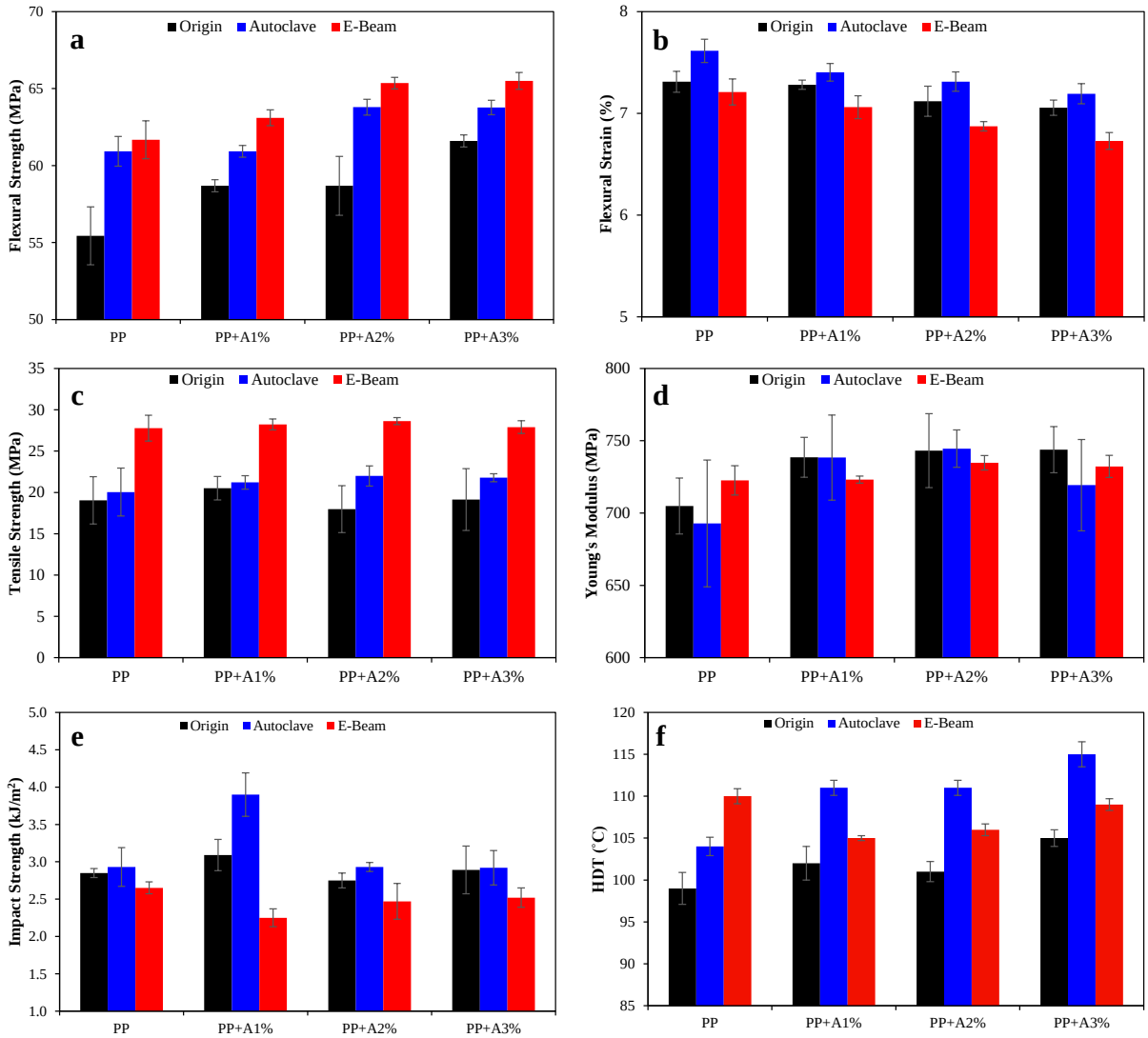

Figure 3 Mechanical properties of PP, PP+A1%, PP+A2%, and PP+A3% injection specimens: (a) flexural strength, (b) flexural strain, (c) tensile strength, (d) Young's modulus, (e) impact strength, and (f) HDT.

3.3. Optical properties

The appearance of all specimens was presented by measurement of %transmission and color parameters (L*, a*, and b* values) represent lightness (+L*)/darkness (-L*), red (+a*)/green (-a*), and yellow (+b*)/blue (-b*), respectively, as shown in Figure 4 (a)-(d). The transmittance of samples decreased after both treatments with increasing biodegradable additives. The presence of the additive and autoclave treatment caused a lighter color (higher L* value) and a small shift to green (more negative a*) and blue (more negative b*). A significant color shift was observed in all E-Beam radiated samples by shifting to green and yellow revealing that the irradiation process gave rise to oxidative degradation of materials (Seguchi et al. 1983)[17]. In addition, it was reported that radiation-induced oxidative degradation of the stabilizing additives and generated palmitic acid and stearic acid leading to the yellowish color and contributing to color changes of the PP (Fintzou et al. 2007)[4].

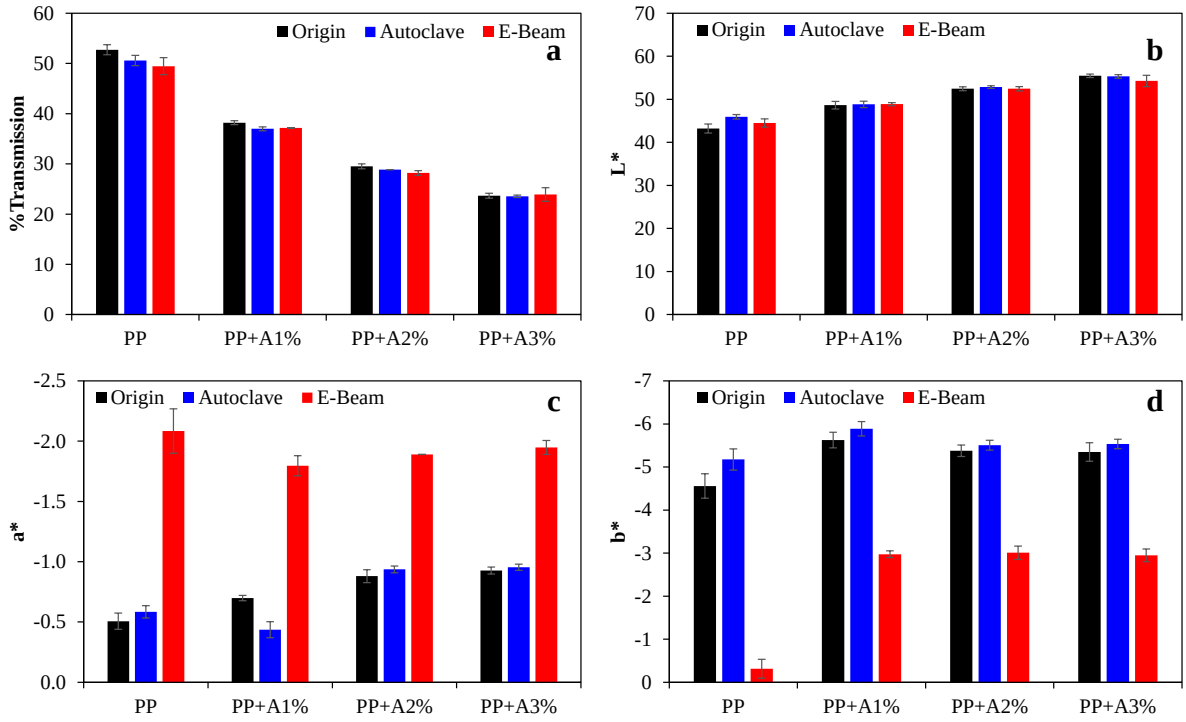

Figure 4 Optical properties of PP, PP+A1%, PP+A2%, and PP+A3% injection specimens: (a) %Transmission, (b) L, (c) a* and (d) b*.*

3.4. Surface energy

Low-retention plastic labware and consumables are specially designed to minimize sample loss due to adhesion and ensure a clean release of liquids. To minimize the surface adhesion of plasticware, using special additives or surface-modification agents are incorporated into the matrix formulation. Therefore, the effect of additive and post-treatment sterilization on surface adhesion of the PP specimen was considered to ensure the potential use of the additive in plastic labware and consumable applications. In this study, four different liquids (water, glycerol, formamide and methyl iodide) were used to determine and compare the surface energy of PP and PP containing of 2% additive along with two sterilization methods; autoclave and E-Beam. The total surface energy is the sum of the dispersive and polar parts (Owens & Wendt 1969)[14]. According to non-polar type of PP in nature, the contribution of the dispersive components (London, Casimir, and van-der-Waals forces) to the surface energy of the samples is bigger than the polar components (polar interactions, hydrogen bonding, acid-base interactions) (Song et al. 2019)[19]. In Figure 5, it was shown that the additive can lower surface energy from 36.9 to 33.9 mN/m, for PP and PP with 2% loading, respectively. Moreover, the reduction of the surface energy of all samples was profound after both treatments, mainly reducing the dispersive part. E-Beam irradiation pronouncedly lowered surface energy to similar values in the range of 26.1-26.5 mN/m for both PP and PP containing 2% additive, indicating that the influence of irradiation overwhelmed the impact of the additive.

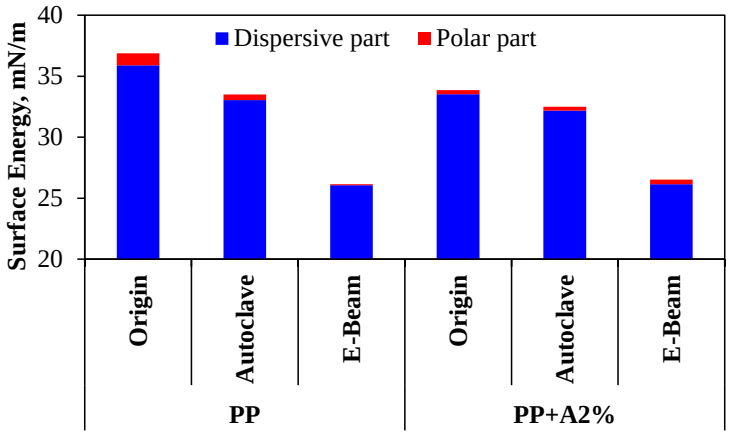

Figure 5 Surface energy of PP and PP+A2% before and after post-treatment (autoclave and E-Beam).

4. Conclusions

This study investigated the influences of incorporating a biodegradable additive and two common sterilization methods: autoclaving and electron beam (E-Beam) irradiation on the properties of polypropylene (PP) intended for plastic labware applications. The incorporation of the additive illustrated minimal impact on the rheological behavior and melt flow index, demonstrating good matrix compatibility. Notably, the additive improved rigidity and thermal stability showing 11% and 6% improvement in flexural strength and HDT at 3% loading, respectively. Moist-heat sterilization via autoclaving resulted in a substantial enhancement in flexural strength (by 15%) and HDT with 3% incorporation of additive suggesting the thermal stability of the additive. In contrast, E-Beam irradiation displayed distinct outcomes, particularly a 50% tensile strength and 18% flexural strength improvement attributed to radiation-induced crosslinking. However, the additive's stability diminished after irradiation, shown by a reduction in impact strength and HDT as compared to their untreated samples. Therefore, there are clear trade-offs between different materials' properties, the biodegradable additive content, and the sterilization methods. Regardless of the changes after treatments, this study suggests that the biodegradable additive possesses a promising attribute in enhancing both the durability and environmental sustainability of PP-based laboratory plasticware and disposable items. The choice of use should depend on the specific application requirements and target critical properties of the finished products. Future research directions should encompass a comprehensive study of multiple autoclave cycle effects and expansion to diverse polymer grades to optimize property enhancement and broaden potential applications.

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