Effect of Thermoplastic Starch Content on the Properties of Poly(butylene adipate-co-terephthalate) (PBAT) Composites

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Thermoplastic starch (TPS) was evaluated as a filler in a poly(butylene adipate-co-terephthalate) (PBAT) matrix. The effect of different levels of TPS (0%, 10%, 30%, 50%) on the composite was studied. The TPS/PBAT composites were prepared by melt blending modification and high temperature moulding. The mechanical properties, hygroscopicity, water absorption, thermal stability, and micromorphology of the PBAT-based composites were tested. The results showed that the tensile strength of TPS/PBAT composites decreased from 13.7 to 3.83 MPa when the TPS content was increased from 0 wt% to 50 wt%; the flexural and tensile strengths of the composites with the addition of 10 wt% TPS were increased by 14.9% and 16.3%, respectively, compared to those of pure PBAT. The water absorption, moisture absorption balance and contact angle of the composites were improved and the contact angle of the 30 wt% TPS/PBAT composites reached 108 deg. The addition of TPS reduced the coefficient of linear expansion of the composites, which showed better thermal stability. The results are important for the development of new biodegradable composites.

DOI: 10.15376/biores.20.2.3910-3922

Keywords: PBAT; Thermoplastic starch; Mechanical properties; Thermal stability

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INTRODUCTION

Traditional plastics (*e.g.* polyethylene (PE), polypropylene (PP), polystyrene (PS), *etc*.) are becoming increasingly widespread in people's daily lives; however, their recycling and disposal are putting enormous pressure on the ecosystem (Andrady and Neal 2009). Many countries have implemented strategies and policies to reduce single-use plastic films (Kasirajan and Ngouajio 2012). Pollution from traditional plastics can be solved through the use of biodegradable plastics (Kabir and Kaur *et al*. 2020). Poly (butylene adipate-co-terephthalate) (PBAT) is a fully biodegradable polyester that can be decomposed into water and carbon dioxide under natural conditions without polluting the environment. At present, extrusion molding of PBAT-based film materials is the main focus of PBAT research. Research on high-temperature vulcanization molding of PBAT-based composite sheets is still in the early stages. With the increasing market demand for PBAT-based materials, PBAT-based materials have more and more applications in the fields of food packaging, construction, agriculture, automobile interior, *etc*., and the research of PBAT-based composite materials is also of great significance for environmental and ecological protection (Yang *et al.* 2024a). According to the survey, the demand for degradable plastics in China is expected to reach 2.2 million tons in 2025, and its price will drop to a reasonable

level (Li and Wang 2022). At present, the price of PBAT is high, and its wide application is difficult to promote due to market spontaneity. To reduce the cost, PBAT can be blended and modified with other degradable materials.

Starch is one of the most abundant natural polymer materials in nature. It is preferred because of its low cost and wide range of sources. Starch occurs in nature primarily as granules and is widely distributed in plant parts such as seeds, roots, and fruits. Natural starch is a semi-crystalline polyhydroxy polymer with unique physicochemical properties. However, there are significant differences in the crystallinity of starches from different sources, resulting in different properties during processing and application. Due to the strong hydrogen bonding between starch molecular chains, the processing performance of natural starch is relatively poor and its water resistance is weak (Feng *et al.* 2024). To improve the processing performance of natural starch and the mechanical properties of the finished product, it is usually subjected to thermoplastic processing. Through thermoplastic processing, the molecular structure of starch is reorganized and the hydrogen bonds between the molecular chains are partially broken, which improves its fluidity and formability (Rahardiyan *et al.* 2023; Xu *et al.* 2024a). The starch material obtained after thermoplastic processing is called thermoplastic starch (TPS) (Diyana *et al.* 2021). Thermoplastic starch not only has good processing properties, but it also can improve the mechanical properties of products to a certain extent. TPS is widely used in packaging materials, disposable tableware, agricultural films and other fields, and has attracted attention in the field of environmental protection because of its biodegradable properties (Zhang *et al.* 2014). Therefore, the development and application of thermoplastic starch is becoming an important research direction in the field of materials science and engineering.

Jiang *et al.* (2024) used sodium-based montmorillonite as a filler to prepare Na-GMMT/TPS pellets, and TPS was blended with PBAT in different proportions. The TPS-PBAT composites were prepared by twin-screw extruder. It was shown that when TPS was used as the matrix, the composites could be substantially increased in tensile strength while maintaining good elongation at break. The elongation at break of TPS-PBAT-4 composites was 233.3%, and the tensile strength was 18.9 MPa. The incorporation of TPS enhanced the thermal stability of corn starch and PBAT. Tian *et al*. (2024) prepared low-cost and shrinkage-resistant PBAT/TPS foams by melt blending PBAT with thermoplastic starch (TPS) using a convenient method and supercritical CO_2 molding and foaming method. Meanwhile, the thermal and rheological behaviors of the PBAT/TPS blending system were investigated, and the effects of different TPS contents on the foaming and shrinkage recovery behaviors of PBAT/TPS foams were analyzed. The results showed that the addition of TPS could improve the shrinkage resistance of PBAT/TPS blended system, which was conducive to the reasonable control of the preparation cost. When the mass fraction of TPS was 20%, the foam shrinkage rate of PBAT/TPS blended system was only 0.017 times-min^{-1}, and the expansion multiplication rate was 4.7 times after stabilization; the raw material cost of the blended system was reduced by about 16% compared with that of pure PBAT. Fourati *et al*. (2021) doped cellulose nanofibers (CNF) into TPS phase using twin screw extrusion. It was found that the tensile strength and modulus increased with the addition of CNF and the optimum performance was achieved at 8 wt% CNF. A narrowing of the distribution of TPS nodules in the PBAT matrix was also observed after the addition of CNF.

Aluminate coupling agent (ACA) is an important surfactant with the advantages of high surface reactivity, wetting and plasticising, environmentally friendly, light coloured

and cheap (Zhao *et al.* 2022). Yang *et al.* (2024b) used an aluminate coupling agent for surface modification of calcium carbonate to carry out calcium carbonate-filled modified PBAT to prepare composites by extrusion moulding. After testing, it was found that the modified calcium carbonate nano-filled PBAT had better tensile properties. Zhang *et al.* (2023) investigated the preparation of new polyethylene (PE) composite panels reinforced with granite sawdust (GN), focusing on the surface modification of GN using aluminium ester coupling agent DL-411. The results showed that the DL-411 coupling agent was firmly attached to the GN surface by chemical bonding. The introduction of the aluminate coupling agent resulted in the formation of a hydrophobic coating on the GN surface. In addition, the modified GN/PE composites exhibited excellent mechanical properties, including higher tensile strength, flexural strength and notched impact strength. The presence of the coupling agent helps to transfer internal stresses within the composite more efficiently.

Thermoplastic starch (TPS) can be used as a modifier to improve the properties of polymer composites. However, most current researchers usually use extrusion and blown film moulding methods (Aversa and Barletta 2022; Xong *et al*. 2022), and there are fewer studies on high-temperature moulding processes. In this study, PBAT/TPS composites were prepared by a high temperature moulding process using PBTA as the matrix and aluminate coupling agent as the melting agent. The effects of different TPS contents on the mechanical properties, moisture and water absorption, surface hydrophobicity and thermal stability of the composites were systematically investigated. Compared to the common extrusion and blown film moulding processes, there have been relatively few studies on high temperature moulding processes, especially in the preparation of PBAT/TPS systems. This study fills the research gap on high-temperature moulding processes in this field, and it provides a new theoretical basis and technical direction for further optimising the properties and practical applications of PBAT/TPS composites.

EXPERIMENTAL

Materials

Polybutylene terephthalate adipate, 200 mesh, was produced by Xinjiang Lanshan Tunhe Chemical Co., Ltd. Thermoplastic starch was produced by Dongguan Zhangmutou Naisi Plastic Raw Material. Aluminate coupling agent was purchased from Quanzhou Kangjin New Material Technology Co.

Material Pre-processing

As the purchased thermoplastic starch (TPS) is in the form of granules, it was necessary to pulverise it to make it more flowable and workable in the production of composites. A Model J-800B multifunctional pulveriser (supplied by Yongkang Minye Industry & Trade Co., Ltd.) was used to perform this step. This converted the granular TPS into a fine powder, which laid the foundation for subsequent TPS preparation, moulding and mixing homogeneity. The pulverized thermoplastic starch and the purchased PBAT raw material were placed in a blast drying oven (DHG-9053A, Shanghai Jiecheng Experimental Instrument Co., Ltd.) and dried at 60 °C for 12 h. After the materials were thoroughly dried to remove any moisture that could affect the integrity of the composite, the PBAT powder, the finely milled thermoplastic starch, and the aluminate coupling agent

were meticulously weighed out according to the precise proportions specified in Table 1. These materials were then introduced into a high-efficiency three-dimensional motion mixer (model BP-5, supplied by Shanghai Zhuo's Instrumentation Co., Ltd.) designed to ensure uniform blending of the components. Inside the mixer, the powder blend was subjected to a continuous mixing process for a duration of 5 min, during which the mixer's three-dimensional motion facilitated comprehensive and homogeneous dispersion of the PBAT powder, thermoplastic starch, and aluminate coupling agent.

Table 1. Thermoplastic Starch/PBAT Composite Ratios

Serial number	PBAT (wt.%)	TPS (wt.%)	Aluminate coupling agent
1	100	0	0
2	90	10	5%
3	70	30	5%
4	50	50	5%

Composite Preparation

To prepare the composite materials, the required raw materials for molding were accurately weighed using an electronic balance. The materials were poured into the cavity of the mold and evenly distributed using tools to minimize the formation of voids. The molding process was conducted using a plate vulcanizer, with the upper, middle, and lower plates set to 170, 175, and 170 °C, respectively. A pressure of 4 MPa was applied, and the molding time was maintained for 10 min, with a single degassing step lasting 2 seconds after pre-pressing for 1 minute, followed by continuation of the molding process post-degassing. After the molding time was completed, the mold was transferred to a water-cooled plate for cooling until it reached room temperature. Subsequently, the mold was reheated to approximately 35 °C in the vulcanizer to facilitate demolding. The composite sheets obtained after demolding were then cut into appropriate strips to prepare specimens for subsequent performance testing.

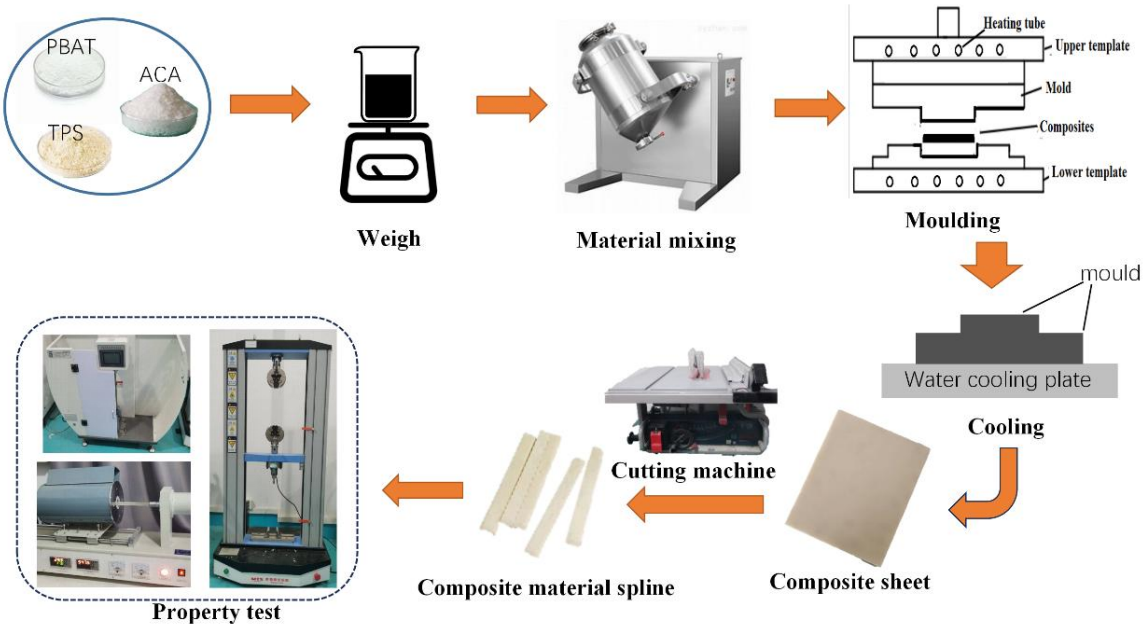

Fig. 1. Schematic diagram of composite material fabrication process

Testing and Characterization

Tensile property test

The tensile properties of composite materials were tested using a computer-controlled universal testing machine following national standard GBT 1040.4-2006(2006). Specimens were cut into strips (120 mm × 8 mm), and their width and thickness were measured using a vernier caliper. The specimens were clamped in the tensile testing module with 15 mm secured at each end, and the test was conducted at a crosshead speed of 50 mm/min. Three tests were performed.

Flexural property test

The flexural properties were evaluated using the GBT 9341-2008 (2008) standard. Specimens (120 mm × 8 mm) were placed in the flexural module of the testing machine with a span length of 50 mm. After measuring the specimen dimensions, the flexural test was performed at a downward speed of 5 mm/min. Data from three specimens were averaged to determine flexural performance.

Impact strength test

Impact strength testing followed GBT 1451-2005(2005) standards for fiber-reinforced plastics. A pendulum impact tester was used, and the initial energy loss due to friction was calibrated with an empty swing. Specimens with consistent dimensions were positioned horizontally on the impact platform, and the pendulum was raised to its maximum angle before each test.

Hygroscopic performance

According to GB/T 20312-2006 (2008) standard, the 51 mm specimen was dried at 60 °C for 36 h and then put into a constant temperature and humidity box (temperature 23 °C, humidity 90%) for testing. The mass was measured at intervals until the moisture absorption rate was stabilized.

Water absorption test

GB/T 1462-2005 (2005) was used to determine the water absorption of the composites. Specimens were dried at 60 °C for 36 h and their initial mass recorded. They were then submerged in distilled water at 23 °C for 24 h. After soaking, the excess water was removed and the mass was recorded.

Contact angle measurement

Contact angles were measured using a JC2000D1 contact angle instrument. Liquid droplets were dispensed onto the specimen surface, and after stabilization, images of the droplets were captured. The contact angle was calculated using the five-point fitting method for high accuracy.

Coefficient of thermal expansion (CTE) test

The thermal expansion properties were tested using a ZRPY-1000 thermal expansion tester. Specimens (51 mm) were fixed on the sample stage, and the equipment was programmed with a maximum temperature of 80 °C. The thermal expansion coefficient was determined automatically based on the specimen's dimensional changes during heating.

RESULTS AND DISCUSSION

Mechanical Performance Analysis

As shown in Fig. 2(a), the overall tensile strength of the composites showed a decreasing trend with increasing TPS content. When the contents of TPS were 10 wt%, 30 wt%, and 50 wt%, the tensile strengths of the composites were 13.66 MPa, 11 MPa, and 3.83 MPa, respectively. The tensile strengths of the TPS/PBAT composites depended on the amount of TPS added. With the gradual increase of TPS addition, the mechanical properties of TPS gradually dominated and the tensile properties of the material decreased. The main reason for this trend is that TPS in PBAT cannot achieve a completely uniform distribution. There will be some agglomeration within the mixture. This will lead to stress concentration and brittle fracture of fibers, resulting in a significant reduction in the fracture strength of the composite (Du *et al.* 2024).

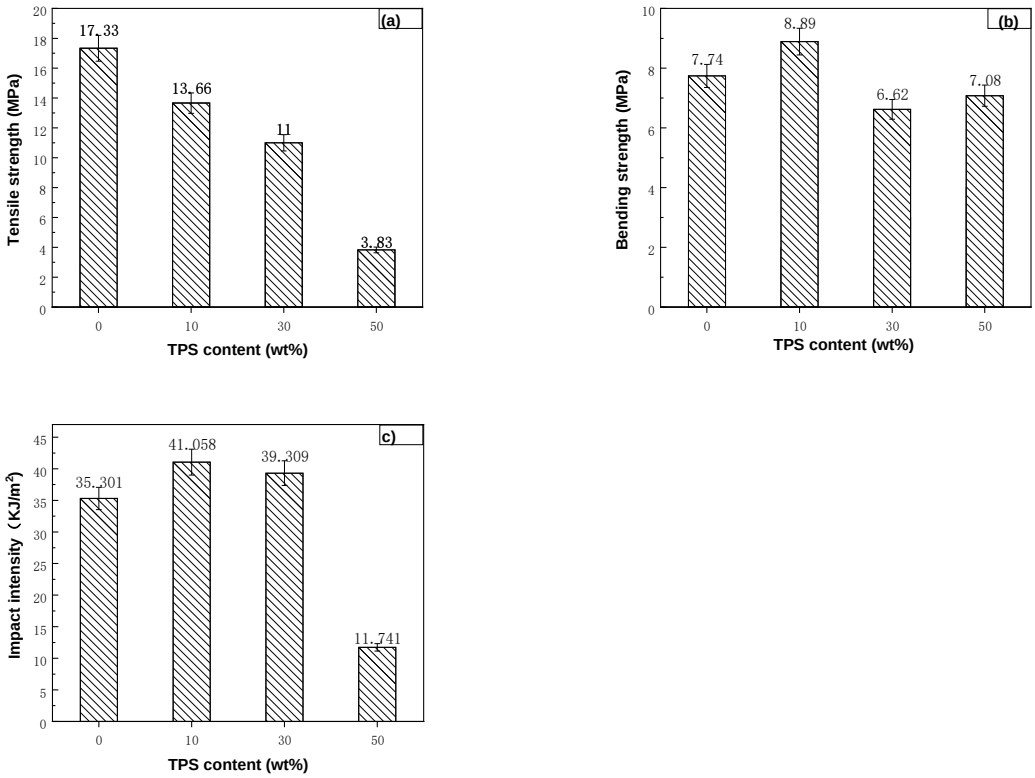

Fig. 2. Mechanical properties of TPS/PBAT composites (a is the tensile strength schematic, b is the bending strength schematic, and c is the impact strength schematic)

Figure 2(b) shows that the flexural strength of 10% TPS was 8.89 MPa. In comparison to that of PBAT composites without added organic fillers, it was increased by 14.9%. When the TPS content was increased to 30% and 50%, the flexural strengths of TPS/PBAT composites were 6.62 MPa and 7.08 MPa, respectively, which were lower than those of 10% TPS. Compared to PBAT alone, the flexural strength decreased by 14.5% and 8.5%, respectively. The main reason for the above situation is that a small amount of TPS plays the role of physical linker in PABT, which is sufficient to connect the PBAT molecular chains (Liu *et al.* 2023). From Fig. 2(b) it can be seen that the flexural strength

of 10% TPS was 8.89 MPa, compared to that of PBAT composites without added organic fillers, indicating an increase by 14.9%. When the TPS content was increased to 30% and 50%, the flexural strengths of TPS/PBAT composites were 6.62 MPa and 7.08 MPa, respectively, which were lower than those of 10% TPS. Compared to PBAT alone, the flexural strength decreased by 14.5% and 8.5%, respectively. The main reason for the above situation is that a small amount of TPS plays the role of physical linker in PABT, which is sufficient to connect the PBAT molecular chains(Liu *et al.* 2023), so that the composites are characterized by high flexural strength. If the content of TPS is increased, TPS will be unevenly dispersed in PBAT, and some defects will be formed in the composite molding process, resulting in easier bending under stress (Zheng *et al.* 2024).

As shown in Fig. 2(c), the overall trend of the composites showed an increase followed by a decrease. The impact strength of the composites with the addition of 10 wt.% and 30 wt.% TPS was improved over the impact strength of the pure PBAT composite, reaching 41.1 and 39.3 KJ/m^2, which corresponds to increases by 16.3% and 11.4%, respectively. This indicates that a small amount of organic fillers can enhance the impact strength of PBAT-based composites and improve the stiffness of the composites. It is also possible that the ACA may physically entangle or chemically react with the molecular chain segments of the polymer, improving the compatibility of the blend system and thus the toughness of the blend system (Zhao *et al.* 2022). When the content of TPS reached 50 wt.%, the impact strength of the composites decreased to 11.7 KJ/m. The reason for the rapid decrease in 50 wt% impact strength is due to agglomeration of TPS in PBAT due to uneven dispersion of TPS in PBAT, which leads to an increase in the number of defects within the material, thus reducing the impact toughness of the material.

Moisture and Water Absorption Performance Analysis

Figure 3(a) shows the 96-h moisture absorption amounts of TPS/PBAT composites, from which it can be seen that the moisture absorption of the composites increased rapidly from 6 h to 48 h. After 48 h of moisture absorption, except for 30% of TPS/PBAT composites, other composites reach the equilibrium of moisture absorption. This is due to the fact that hygroscopicity testing of dried composites results in the rapid absorption of a large number of water molecules, which causes the hygroscopicity of the material to grow faster during the initial phase of the test (Wang *et al.* 2023).

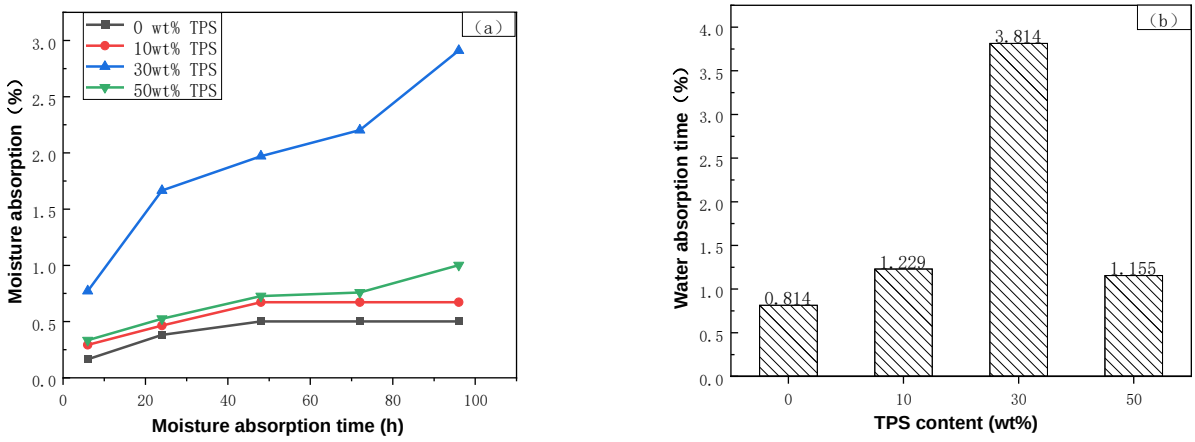

Fig. 3. Moisture and water absorption of TPS/PBAT composites

With the increase of moisture absorption time, the moisture difference between the material and the environment decreases, and the moisture absorption gradually reached an equilibrium. The moisture absorption of 30% TPS/PBAT composites increased with time after 48 h. The reason for this phenomenon is that the TPS is not sufficiently encapsulated by the PBAT, which results in a part of the hydrophilic TPS being located on the surface of the composites. The reason for this phenomenon may be that the PBAT did not sufficiently wrap the TPS, resulting in part of the hydrophilic TPS being exposed, which makes the overall moisture absorption rate of the composite continuously increase. Subsequent studies can be carried out by refining the gradient around 30 wt% TPS.

The 24 h water absorption of TPS/PBAT composites is shown in Fig. 3(b). Pure PBAT had the lowest water absorption of 0.81%. PBAT is a hydrophobic material with fewer internal polar groups, so the water absorption was lower. When TPS was added, the water absorption percentage was increased, and the highest 24 h water absorption of 30 wt.% TPS/PBAT composite was 3.8%. This was confirmed by the moisture absorption percentage, which indicated that the moisture and water absorption were relatively good. TPS is rich in starch and contains a large number of hydrophilic groups in the molecular structure (Xu *et al.* 2024b). These groups tend to form hydrogen bonds with water molecules, thus increasing the water absorption of the composites. Due to the fact that PBAT is a degradable material, increased moisture and water absorption can accelerate the degradation rate and reduce the service life of the composite (Liu 2024).

Surface Hydrophobicity Analysis

As shown in Fig. 4, the hydrophobicity of the composite surface increased with the addition of 10% and 30% TPS, reaching 107.8° and 108.4°, respectively.

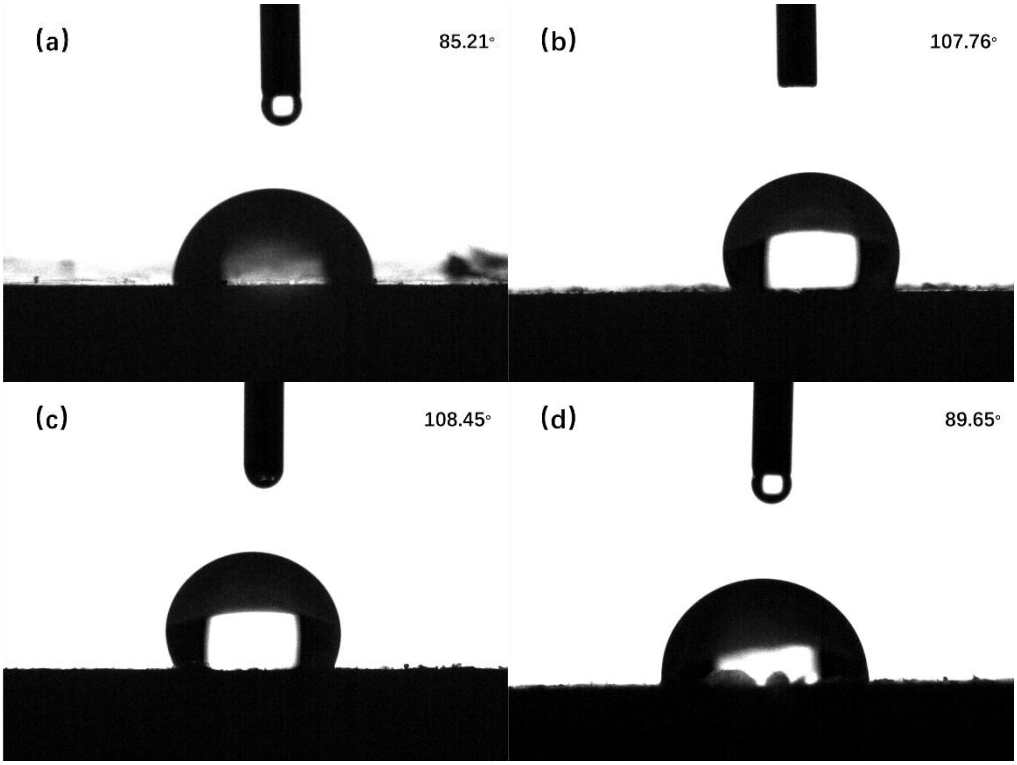

Fig. 4. Contact angle of TPS/PBAT composites (a is pure PBAT, b is 10 wt.% TPS, c is 30 wt.% TPS, d is 50 wt.% TPS)

The contact angle of the composites was 89.6° at 50% TPS content. The contact angles of the composites were increased by the addition of TPS. Although TPS itself is hydrophilic, the introduction of aluminates can improve the hydrophobicity of the composite surface. The organic chain (COR′group) in the aluminate coupling agent will intertwine with the PBAT molecules, binding the TPS and PBAT together and changing them from hydrophilic and oleophilic to lipophilic and hydrophobic (Ji 2024). The contact angle increases and then decreased, probably because the small amount of TPS was mainly distributed in the interior of the polymer composites and is not fully exposed on the surface, making the composite surface more hydrophobic.

Thermal Stability Analysis

Figure 5 shows that the linear expansion coefficient of pure PBAT increased more than that of the TPS/PBAT composites. The pure PBAT had the highest coefficient of linear expansion, and the curve remained stable over a wide temperature range. This indicates that pure PBAT had higher thermal expansion and better molecular chain flexibility. After adding a small amount of TPS, the linear expansion coefficient decreased. This was due to the high stiffness of TPS (Lin *et al.* 2023), which limits the thermal movement of the PBAT molecular chain upon addition. As the TPS content was further increased, the linear expansion coefficient continued to decrease, indicating that increasing the amount of rigid components in the composites had a more significant inhibiting effect on the thermal expansion. The TPS particles acted as physical barriers that restrict the ability of the PBAT chains to move freely when subjected to thermal stress. This constraint is due to the TPS's high stiffness, which hinders the molecular chain's ability to elongate or expand in response to temperature changes. The slight recovery of the linear expansion coefficient of the composites when 50 wt.% TPS was added may be related to the weakening of the phase separation or interfacial interaction due to the high TPS content (Shang *et al.* 2024). The incorporation of TPS generally decreased the linear expansion coefficient of PBAT.

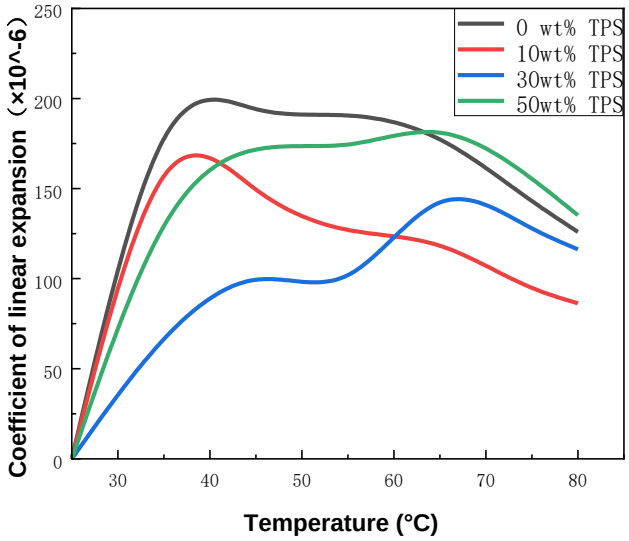

Fig. 5. TPS/PBAT coefficient of linear expansion

SEM Analysis

As the mechanical properties of 10 wt% TPS and 50 wt% TPS were quite different, the microstructure was observed, as shown in Fig. 6. It is apparent that the tensile sections of the composites with both blends show material being pulled out of the matrix and deformed. At the same time, it can be seen that the surface of the filaments stretched when 10 wt% TPS was added was rough, indicating that the two were more tightly bonded. By contrast, the surface of the filaments stretched from the matrix when 50 wt% TPS was added was smooth during the stretching process, and the presence of TPS agglomeration phenomenon can be seen at the same time. This may be the reason for the decrease in mechanical properties of the 50 wt% TPS/PBAT composites.

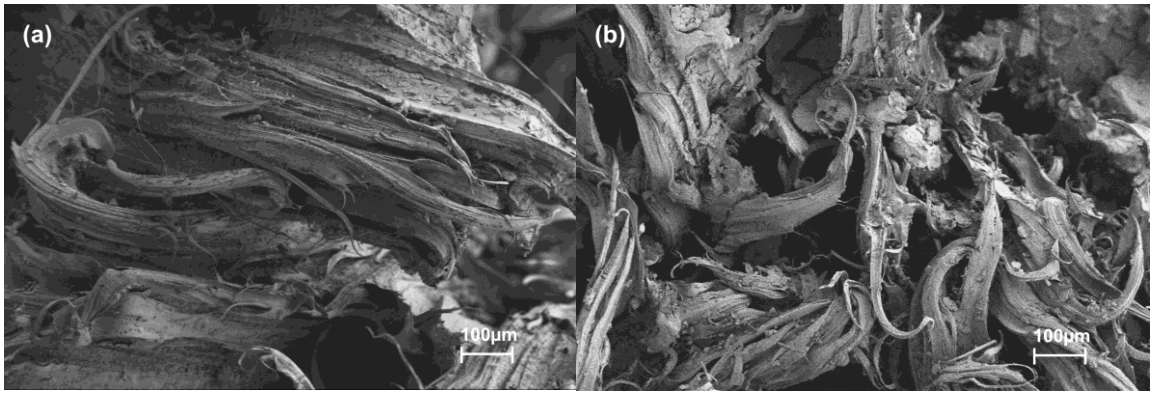

Fig. 6. Scanning electron microscopy of TPS/PBAT composites (a is 10 wt.% TPS, b is 50 wt.% TPS)

CONCLUSIONS

1. The mechanical properties of thermoplastic starch/poly (butylene adipate-co-terephthalate) (TPS/PBAT) composites were significantly affected by the addition of TPS. The overall tensile strength of the composites showed a decreasing trend with the increase in TPS content from 13.7 to 3.83 MPa. The flexural strength of the composites was improved by the addition of 10% TPS, but the flexural strength tended to decrease with the further increase in TPS content. In terms of impact strength, the composites showed some improvement with the addition of 10 wt.% and 30 wt.% TPS, but the impact strength decreased with 50 wt.% TPS content. Agglomeration was found to be present within 50 wt% of the composites by SEM, which affected the mechanical properties of the composites. Thus, a moderate amount of TPS enhanced the flexural and impact strength of PBAT composites.
2. The 24 h water absorption and hygroscopic equilibrium of the composites were higher than those of pure PBAT with the addition of organic fillers, which was attributed to the fact that the organics themselves had more hydroxyl groups that can easily bind with water molecules. The contact angle of the 30% TPS and PBAT blends was also relatively high, with a contact angle of 108.4°. The contact angle of the composites with the addition of organics and aluminate coupling agent was 85.2. The contact angle of the pure PBAT composites was the lowest at 85.2°. The increase in surface contact angle of the composites with the addition of both organics and aluminate coupling

agent suggests that the introduction of aluminate increased the hydrophobicity of the material surface.

3. Adding TPS reduced the linear expansion coefficient of pure PBAT and improved the thermal stability of the composites. The linear expansion coefficient of the 30 wt.% TPS/PBAT composites was generally low, with a peak value of 65 to 70 °C, which was not easily changed by changing the ambient temperature, and had a high thermal stability. Therefore, the moderate addition of TPS improved the thermal expansion properties of PBAT.

ACKNOWLEDGMENTS

This work was financially supported by National Scientific Research Program Cultivation Fund of Chengxian College of Southeast University (Grant No. 2022NCF003); the Natural Science Foundation of the Jiangsu Higher Education Institutions of China (Grant No. 24KJB430008); Anhui Provincial Department of Education Natural Science Major Project (Grant No. 2022AH040238); Anhui Provincial Natural Science Foundation Face Project (Grant No. 2308085ME178); Anhui Highly Efficient Collaborative Innovation Project (Grant No. GXXT-2023-058); Anhui Agricultural Substance Unveiling and Hanging Project (Grant No. 311152350024); Anhui Province Synergy Innovation Program of 2022 (Grant No. GXXT-2022-077); Anhui University Synergy Innovation Program (Grant No. GXXT-2023-022);

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Article submitted: December 23, 2024; Peer review completed: February 8, 2025; Revised version received: February 18, 2025; Accepted: March 28, 2025; Published: April 8, 2025.
DOI: 10.15376/biores.20.2.3910-3922