

สมบัติเชิงกลและการย่อยสลายทางชีวภาพของวัสดุคอมพอสิตพอลิบิวทิลีนซัคซิเนตผสมรังไหม

Mechanical properties and biodegradation of poly(butylene succinate)/silkworm cocoon composites

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บทคัดย่อ

งานวิจัยทำการเตรียมวัสดุคอมพอสิตพอลิบิวทิลีนซัคซิเนตผสมรังไหมในอัตราส่วนเท่ากับ 100/0, 90/10, 80/20, 70/30 และ 60/40 เพื่อศึกษาสมบัติเชิงกล (ความต้านทานแรงดึง ความต้านทานแรงกระแทก ความแข็ง) สมบัติการย่อยสลายทางชีวภาพ และลักษณะทางสัณฐานวิทยา ผลการวิจัยพบว่าเมื่อเติมรังไหมในปริมาณมากขึ้น ค่ามอดูลัสของยัง ค่าความต้านทานแรงกระแทก และค่าความแข็งเพิ่มขึ้น ในขณะที่ค่าความต้านทานแรงดึงสูงสุด และค่าการยืดตัว ณ จุดขาด ลดลง การศึกษาการย่อยสลายโดยการฝังกลบในดิน เป็นเวลา 4, 8 และ 10 สัปดาห์ พบว่าการเพิ่มปริมาณรังไหมในการผสมกับพอลิบิวทิลีนซัคซิเนตจะทำให้เกิดการย่อยสลายทางชีวภาพที่ดีขึ้น ในขณะที่การตรวจสอบลักษณะทางสัณฐานวิทยาด้วยเทคนิคกล้องจุลทรรศน์อิเล็กตรอนแบบส่องกราด (SEM) ของชิ้นงานภายหลังทดสอบความต้านทานแรงกระแทก พบว่ามีการกระจายตัวของเส้นใยรังไหมในพอลิเมอร์เมทริกซ์ และมีความไม่เข้ากันระหว่างผิวของรังไหมกับพอลิบิวทิลีนซัคซิเนต ส่งผลให้วัสดุคอมพอสิตที่เตรียมได้มีความต้านทานแรงดึงสูงสุด และค่าการยืดตัว ณ จุดขาด ลดลง

คำสำคัญ: พอลิบิวทิลีนซัคซิเนต, รังไหม, วัสดุผสม, การย่อยสลายทางชีวภาพ, สมบัติเชิงกล

Abstract

In this study, poly(butylene succinate)/silkworm cocoon composites were prepared in the following ratios: 100/0, 90/10, 80/20, 70/30, and 60/40. The mechanical properties (tensile, impact, and hardness tests), biodegradation properties, and morphological characteristics of the composites were examined. The results demonstrated that the Young's modulus, impact strength, and hardness of the composites increased with an increase in the amount of silkworm cocoon, while the tensile strength and elongation at break of the composites decreased. An assessment of the decomposition of poly(butylene succinate)/silkworm cocoon composites buried in soil for 4, 8, and 10 weeks showed that increasing the amount of silkworm cocoons in the composites resulted in increased biodegradation. Moreover, the morphology of the specimen after impact testing was examined using the SEM technique. The SEM images showed that the silkworm cocoon fibers were distributed in the polymer matrix, indicating the incompatibility of the silk and poly(butylene succinate) matrix. This incompatibility resulted in composite materials with decreased tensile strength and elongation at break.

Keywords: Poly(butylene succinate), Silkworm cocoon, Composites, Biodegradation, Mechanical properties

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Introduction

Many countries currently recognize the importance of addressing various environmental problems. The demand for reducing or solving these issues is therefore increasing, especially regarding the problem of plastic waste (Rodriguez-Urbe *et al.*, 2023). This has led to initiatives to tackle the plastic waste problem through various techniques, including reducing and eliminating the use of plastic. However, plastic remains essential to human daily life due to its many advantages, such as durability, lightweight, low cost, and ease of production (Mamatha *et al.*, 2024; Hong *et al.*, 2024). Therefore, researchers are trying to invent and develop plastics that provide convenience in human life while being environmentally friendly and not causing problems after use. The research and development of biodegradable plastics is a promising avenue in this regard (Sena *et al.*, 2023).

Biodegradable plastic refers to plastic that undergoes decomposition, ultimately being digested by microorganisms until it becomes water, carbon dioxide, and biomass under appropriate temperature and humidity conditions. After decomposition, no plastic parts or toxins remain (Royer *et al.*, 2023). Examples of biodegradable plastics include polycaprolactone (PCL), polyhydroxybutyrate (PHB), polybutylene succinate (PBS), polybutylene adipate co-terephthalate (PBAT), polylactic acid (PLA), and polyhydroxyalkanoates (PHAs) (Nachod *et al.*, 2021; Xie *et al.*, 2023). Among these biopolymers, polybutylene succinate is considered particularly important due to its straight chain structure, high melting point, crystallinity, heat resistance of up to 200°C, ease of formation, and similar mechanical properties to polyethylene (Zhou *et al.*, 2023). However, polybutylene succinate is still constrained by its high price. Therefore, efforts are underway to enhance and develop it through various methods to reduce costs while maintaining appropriate physical and mechanical properties, as well as biodegradability. For instance, a previous study focused on the preparation of composite materials from polylactic acid and polybutylene succinate mixed with corn starch and wheat straw for biodegradable packaging (Abdollahi Moghaddam *et al.*, 2023). Other research

includes the preparation of composites of polybutylene succinate mixed with natural rubber or activated carbon to increase mechanical properties (Prasertsopha *et al.*, 2020; Singsang *et al.*, 2021) and using various natural fibers as reinforcements for polybutylene succinate (Calabia *et al.*, 2013; Huang *et al.*, 2018; Rodriguez-Urbe *et al.*, 2023).

Silk and other natural fibers are known to enhance mechanical properties, including tensile strength, modulus of elasticity, and impact resistance, of biodegradable composites due to their strong and resilient nature (Han *et al.*, 2010). In addition, natural fibers, such as silk, can promote the biodegradation of biodegradable composites by providing a substrate for microbial activity, thereby speeding up the breakdown of the composite materials into simpler, environmentally benign substances.

Based on the information above, silk fiber can be used as a reinforcement for polybutylene succinate, a biodegradable plastic, to improve mechanical properties and degradation rate. However, the silk production process results in a large number of discarded silkworm cocoons. Therefore, this research aims to use silkworm cocoons as a reinforcing material instead of silk fibers. The mechanical and biodegradation properties of composite materials obtained by mixing polybutylene succinate with silkworm cocoons were studied.

Experimental Procedure

Raw materials

Poly(butylene succinate) (PBS, FZ71PM, BioPBS™, melt flow rate (MFR) of 24.2 g/10 min, PTT MCC Biochem Company Limited) was used as the polymer matrix. The silkworm cocoons (Tubtim Siam) were sourced from a local producer in Yasothon, Thailand. After boiling the silkworm cocoons in water to remove the silk sericin and bring out the silk fibers, the remaining yellow silkworm cocoons were dried and cut into small pieces measuring 1 cm x 1 cm before being used as reinforcement materials. The average diameter of the silkworm cocoon fibers was about $16.03 \pm 2.57 \mu\text{m}$, as shown in Figure 1.

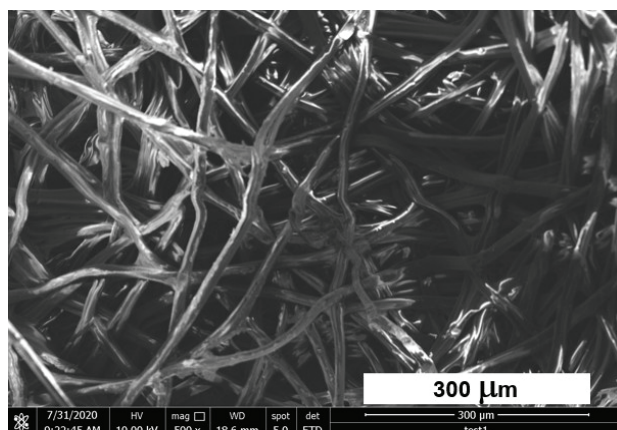


Figure 1 SEM images of silk cocoon.

Preparation of poly(butylene succinate)/silk cocoon composites

To prepare poly(butylene succinate)/silk cocoon composites, both the poly(butylene succinate) and silk cocoons were first dried at 60°C for 24 hours in an oven to remove moisture before mixing. Subsequently, they were mixed using an internal mixer (MX500-D75L90, Chareon Tut Co., Ltd., Thailand) at 145°C with a rotor speed of 50 rpm for 20 minutes. The mixture ratios by weight of poly(butylene succinate) to silk cocoon were 100/0, 90/10, 80/20, 70/30, and 60/40.

Sample preparation and testing

The composites were prepared in each ratio and ground with a plastic grinder to reduce the size, making them suitable for forming the test specimens. The test specimens for mechanical properties and decomposition in soil were formed using hot-compression molding at a pressure of 1500 psi and a temperature of 145°C for 11 minutes.

The mechanical property testing in terms of tensile testing were conducted according to ASTM D638 using a universal testing machine (LS Plus Series, Lloyd) with a load cell of 10 kN and a test speed of 50 mm/min. Hardness testing was conducted according to ASTM D2240 using a Durometer Shore D (GS-612, TECLOCK). Impact testing was performed using an INSTRON model CEAST 9050 according to ASTM D256.

For the biodegradability test, samples with a size of 1 cm x 1 cm x 0.3 cm were dried in an oven at 50°C for 24 hours, then weighed, and the weight recorded as w_0 . The samples were then buried in soil with a pH of

about 6-7.6 and moisture content of about 50–70% of dry soil weight, at a depth of 5 centimeters for 4, 8, and 10 weeks. After degradation, the samples were cleaned and weighed, and the weight recorded as w_f . The percentage weight loss was calculated using the equation: weight loss (%) = $(w_0 - w_f) \times 100/w_0$ (Peñas *et al.*, 2023). Photos of the samples were taken after decomposition durations of 4, 8, and 10 weeks.

The morphology of poly(butylene succinate)/silk cocoon composites was investigated using a scanning electron microscope (SEM, Quanta 250 W7, Philips). The surface of the impact-tested samples was examined to analyze the characteristics of the silk cocoons within the poly(butylene succinate).

Results and Discussion

Mechanical properties of poly(butylene succinate)/silk cocoon composites

The stress-strain curves of poly(butylene succinate)/silk cocoon composites, as shown in Figure 2, indicates that neat poly(butylene succinate) exhibits ductile fracture behavior. However, the addition of silk cocoons increased brittle fracture behavior. This is due to the natural fibers creating stress concentration points and poor interfacial adhesion with the polymer matrix, leading to brittle fracture behavior. These fibers restrict the plastic deformation of the matrix, or, in other words, the silk fibers obstruct the molecular chain movement of the polymers, resulting in reduced ductility.

The average Young's modulus of the poly(butylene succinate)/silk cocoon composites increased with an increase in the silk cocoon content, as shown in Figure 3. The composite with 60/40 weight ratio of poly(butylene succinate) to silk cocoon exhibited the highest Young's modulus, accounting for a 348% increase compared to neat poly(butylene succinate). This indicates that silk cocoons can effectively reinforce poly(butylene succinate) by increasing its modulus due to the enhanced stiffness of the composite. Natural fibers, such as silk cocoon, are typically stiffer than the polymer matrix, and their inclusion restricts the deformation of the composite under load, leading to a higher modulus (Clarizio & Tatara, 2012).

In contrast, tensile strength and elongation at break decreased with an increase in the silkworm cocoon content, as shown in Figures 4 and 5, respectively. This can be attributed to the low bonding force between the silkworm cocoon and poly(butylene succinate), resulting in poor interfacial adhesion (Botev *et al.*, 1999; Sing-sang *et al.*, 2021). The addition of rigid fibers reduces the composite's ability to deform plastically because the fibers do not stretch as much as the polymer matrix. This results in a brittle composite that fractures at lower strains compared to the pure matrix. Essentially, the fibers limit the capacity of the matrix to elongate, thus reducing the overall elongation of the composite material (Seculi *et al.*, 2022).

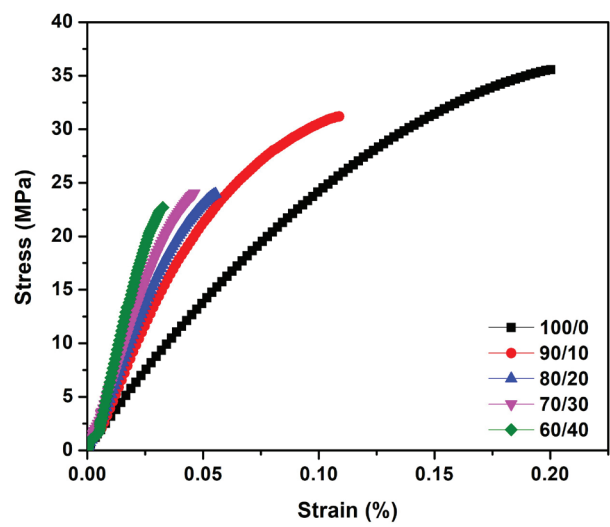


Figure 2 Stress-Strain curves of poly(butylene succinate)/silkworm cocoon composites

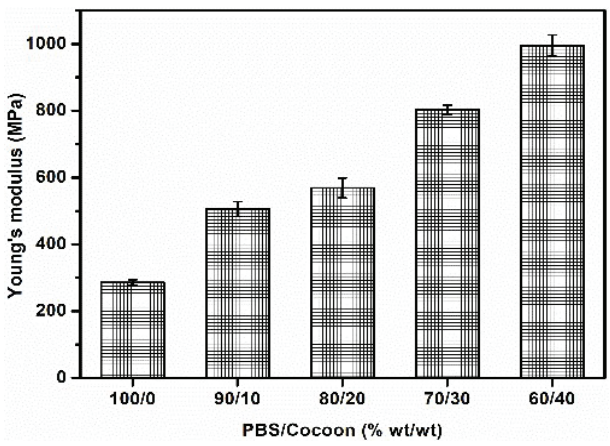


Figure 3 Young's modulus of poly(butylene succinate)/silkworm cocoon composites

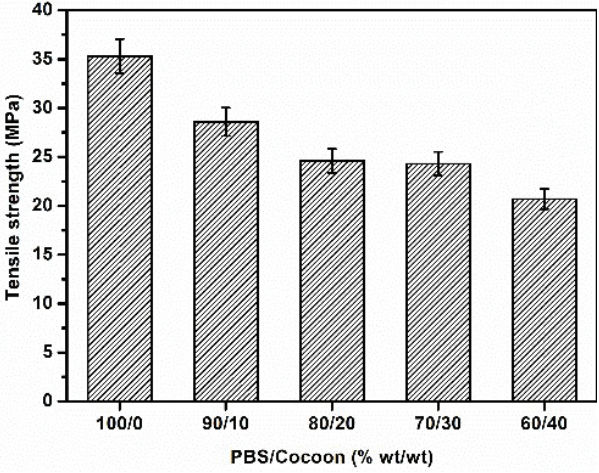


Figure 4 Tensile strength of poly(butylene succinate)/silkworm cocoon composites

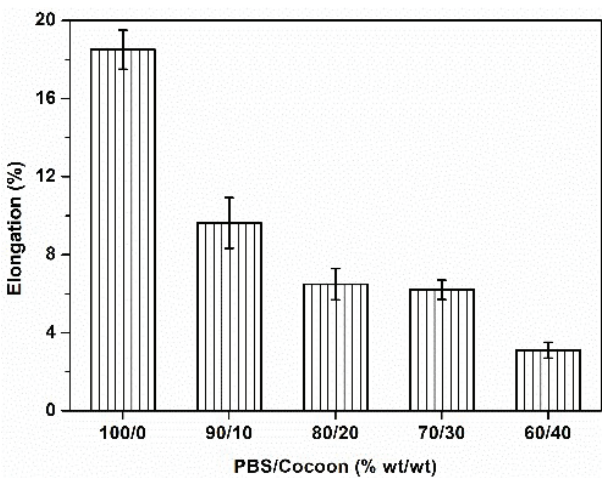


Figure 5 elongation at break of poly(butylene succinate)/silkworm cocoon composites

Figure 6 illustrates the hardness of the poly(butylene succinate)/silkworm cocoon composites. It was found that the hardness slightly increased with an increase in the silkworm cocoon content. This might be because poly(butylene succinate) is a biodegradable polymer that is often noted for its brittleness and lower mechanical strength, despite being flexible and processable. When reinforced with natural fibers, including silk fibers, PBS composites tend to show improved mechanical properties such as increased hardness and tensile strength (Rajgond *et al.*, 2024). This improvement is attributed to the effective distribution of stress and the interaction between the polymer matrix and the reinforcing fibers (Chen *et al.*, 2017). According to a previous study, natural fibers can increase the hardness of biocomposite materials (Nithikarnjanatharn & Samsalee, 2022).

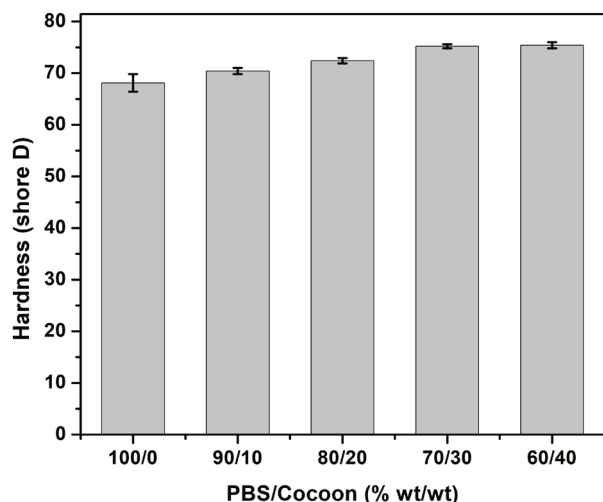


Figure 6 Hardness of poly(butylene succinate)/silkworm cocoon composites

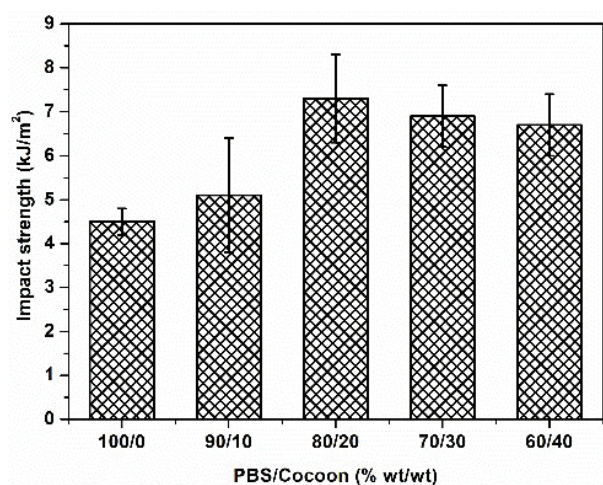


Figure 7 Impact strength of poly(butylene succinate)/silkworm cocoon composites

The impact resistance of the poly(butylene succinate)/silkworm cocoon composites at weight ratios of 100/0, 90/10, 80/20, 70/30, and 60/40 was 4.51 ± 0.26 kJ/m², 5.08 ± 1.27 kJ/m², 7.28 ± 1.01 kJ/m², 6.93 ± 0.67 kJ/m², and 6.65 ± 0.71 kJ/m², respectively (Figure 7). It can be observed that the impact resistance increased with an increase in the silkworm cocoon content. This improvement is attributed to the silkworm cocoon fibers, which help delay crack initiation and control propagation within the polymer matrix, thereby distributing stress and absorbing energy (de Albuquerque *et al.*, 2000). The composite with the ratio of 80/20 exhibited the highest impact resistance, with slight decreases observed at 70/30 and 60/40, respectively, indicating that 80/20 was the optimal ratio. The decrease in impact strength at higher ratios may be attributed to the agglomeration of silkworm cocoons, causing them to act as stress concentrators when their content exceeds the optimum value (Johansson *et al.*, 2023). However, even with this slight decrease, the impact resistance remained higher than that of pure poly(butylene succinate).

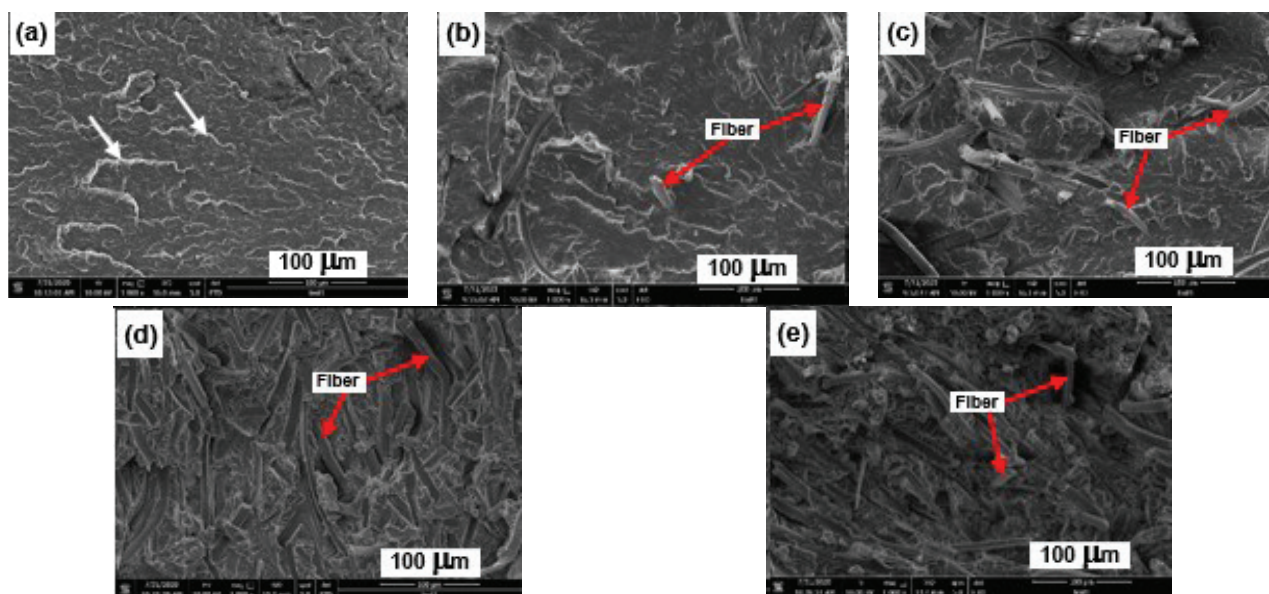


Figure 8 SEM images of poly(butylene succinate)/silkworm cocoon composites in ratios of (a) 100/0, (b) 90/10, (c) 80/20, (d) 70/30, and (e) 60/40.

The surface characteristics of the impact- tested samples of the poly(butylene succinate)/silkworm cocoon composites are shown in Figure 8. It was observed that the surface of pure poly(butylene succinate) exhibited regular tear lines resulting from impact testing, as indicated by the white arrows in Figure 8(a). When silkworm cocoon fibers were added, tear lines and silk fibers could be seen scattered throughout the poly(butylene succinate), with

some fibers protruding and detaching from the matrix, as indicated by the red arrows in Figures 8(b)-(e). It was also found that the silkworm cocoons were distributed evenly. As the concentration of silkworm cocoons increased, the silk fibers became more widely distributed across the surface of the impact test samples, and the density of the fibers increased.

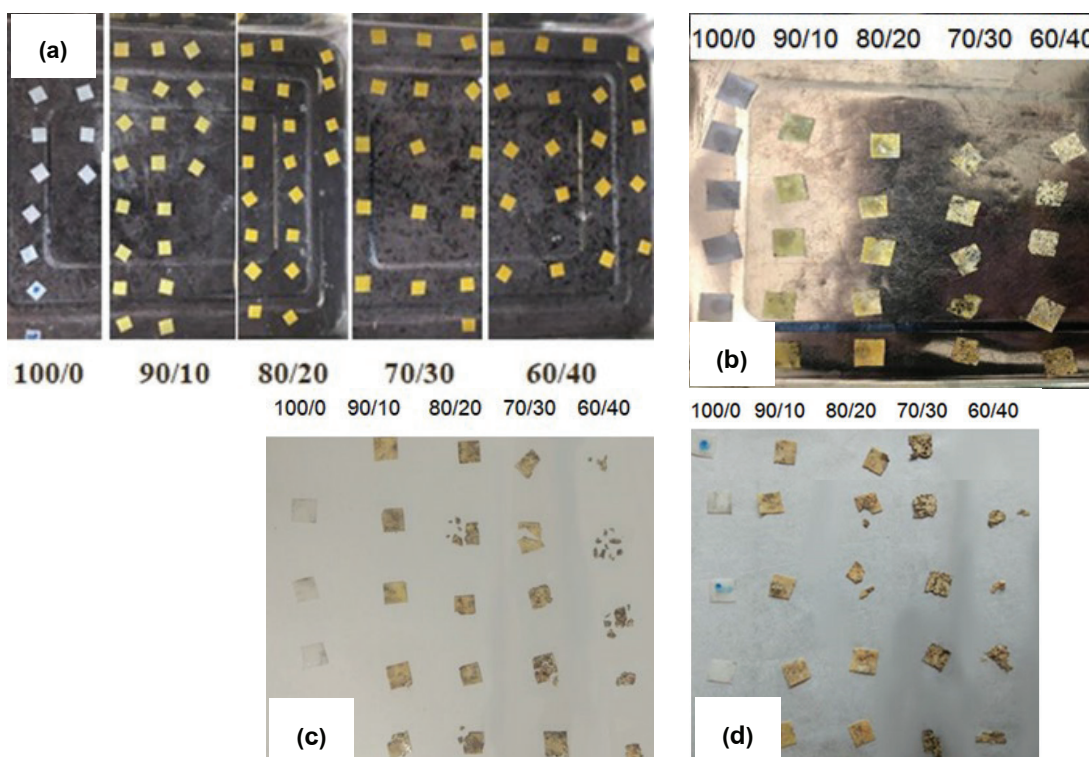


Figure 9 Biodegradation photos of poly(butylene succinate)/silkworm cocoon composites (a) before, (b) after 4 weeks, (c) after 8 weeks, and (d) after 10 weeks

Figure 9 shows the photos of poly(butylene succinate)/silkworm cocoon composites, which were buried in soil to assess their biodegradation behavior. The images revealed that after being buried, the specimens exhibited a roughened surface and evident fractures, indicative of biodegradation. These surface alterations and fractures became more pronounced with longer burial durations, suggesting ongoing surface erosion over time (Wei *et al.*, 2022).

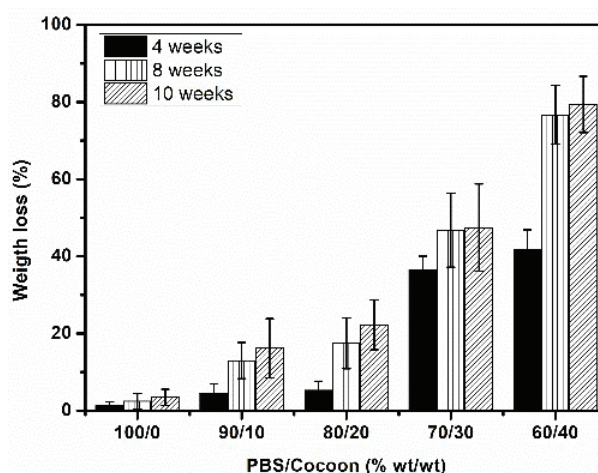


Figure 10 Weight loss of poly(butylene succinate)/silkworm cocoon composites in soil

Table 1 Biodegradation rate of poly(butylene succinate)/silkworm cocoon composites

Composite ratios	Biodegradation rate (mg/day)	
	average	S.D.
100/0	0.016	0.001
90/10	0.082	0.018
80/20	0.106	0.028
70/30	0.361	0.116
60/40	0.533	0.073

The weight loss percentage of poly(butylene succinate)/silkworm cocoon composites over various burial periods is shown in Figure 10. It was found that after 4 weeks of burial, the composites with ratios of 100/0, 90/10, 80/20, 70/30, and 60/40 had average weight loss percentages of $1.3 \pm 1.0\%$, $4.5 \pm 2.4\%$, $5.3 \pm 2.4\%$, $36.5 \pm 3.5\%$, and $41.8 \pm 5.1\%$, respectively. The results indicate that the percentage of weight loss generally increased with an increase in the amount of silkworm cocoon. The composite with the ratio of 60/40 showed the most significant biodegradation at 4 weeks. This trend remained consistent as the burial duration extended to 8 and 10 weeks. After 10 weeks, the 60/40 composite had the highest biodegradation, with a weight loss of $79.1 \pm 7.4\%$, demonstrating that the poly(butylene succinate)/silkworm cocoon composite is naturally biodegradable. This degradation likely resulted from microbial activity under environmental conditions (Sasimowski *et al.*, 2023). Additionally, the 60/40 composite exhibited the highest biodegradation rate, at 0.533 ± 0.073 mg/day, as shown in Table 1.

Conclusions

Poly(butylene succinate)/silkworm cocoon composites were prepared using an internal mixer and hot-compression molding. The ratio of poly(butylene succinate) to silkworm cocoon affected the mechanical properties of the composites. The Young's modulus, impact resistance, and hardness increased with an increase in the amount of silkworm cocoon, while the tensile strength and elongation at break decreased due to poor interfacial

adhesion between the silkworm cocoon and poly(butylene succinate). In addition, the results from the biodegradation test showed that the poly(butylene succinate)/silkworm cocoon composites with a ratio of 60/40 had the highest biodegradation, at $79.1 \pm 7.4\%$, after 10 weeks.

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