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Effect of glycerol concentration as a biocoating on lotus leaf fracture improvement

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ABSTRACT

This study investigated the fracture improvement of dried lotus leaves using glycerol, beeswax, and chitosan as coating agents. The aim of this study was to investigate the effects of glycerol on lotus leaf fracture improvement. The optimum glycerol content for biocoating was investigated using the recovery percentage of lotus leaves after coating, which indicates the elastic properties of lotus leaves. The 27% recovery rate was increased after treatment with 5 g chitosan, 24% v/v glycerol, and 76% v/v acetic solution for 1 d of immersion. The moisture content and thickness of the coated lotus leaves increased by 38% and 7.1%, respectively. Meanwhile, the contact angle decreased by 24% because of the increase in moisture content. Moreover, the mechanical properties showed that Young's modulus decreased by 76% and the elongation increased by 78% after coating because of the improved fracture of dried lotus leaves. Additionally, thermal stability was increased compared with that of dried lotus leaves according to thermal analysis.

Keywords: lotus leaves; biocoating; glycerol; fracture improvement

1. INTRODUCTION

Currently, in addition to energy issues, environmental concerns are of paramount importance in addressing the challenges faced by humans and living organisms. In recent years, synthetic products and materials have been developed to meet human needs. However, these synthetic materials may have adverse effects on the environment and living organisms, posing risks to the soil, water, and air. To overcome these challenges, Thailand must develop new strategies for environmental conservation. In recent decades, there has been a growing interest in environmental protection driven by the bio-circular-green economy model, which promotes the development and integration of various knowledge sources to enhance the value of resources and biodiversity. Discarded sacred lotus leaves are considered a significant biowaste, and many communities have repurposed them into containers for rice, known as "lotus leaf-wrapped rice." Additionally, efforts have been made to develop various products and decorative items from sacred lotus leaves because of their unique patterns and distinctive characteristics. However, the biological characteristics of lotus leaves pose challenges in terms of durability and color stability when developed into products. Issues such as decay and susceptibility to fungal growth as well as color changes have been observed in lotus leaf-derived products, which are attributed to the biological origin of the raw material.

Considering the ability to extend a product's lifespan and prevent its deterioration, various substances with barrier properties that can effectively block the passage of oxygen and moisture are available. These substances encompass synthetic polymers such as polyethylene, polypropylene, and ethylene vinyl alcohol (Tyagi et al., 2021; Mujtaba et al., 2022). Additionally, biopolymers like chitosan, lignin, cellulose, and modified starch have been explored for their barrier properties (Mujtaba et al., 2022). Furthermore, research in the preservation of fruits and vegetables has investigated the use of compounds such as resin, wax, shellac, and glycerol to enhance the shelf life of agricultural products postharvest (Ncama et al., 2018; Miranda et al., 2021).

Chitosan is a polysaccharide prepared by the deacetylation of chitin, which is a widely abundant natural polymer in living organisms. Chitosan has antimicrobial properties, is nontoxic, biodegradable, and biocompatible (Severino et al., 2015). In addition to its applications in the food industry and agriculture, chitosan has been used to enhance the quality and shelf life of food packaging materials, thereby extending the preservation time of food products (Kanatt et al., 2013). Furthermore, chitosan has been combined with glucoseryl, ketoseryl, and beeswax to improve the water repellency properties of packaging materials made from pineapple leaf fibers, resulting in significantly enhanced water resistance performance (Iewkittayakorn et al., 2020).

Glycerol monolaurate (GML), which is derived from coconut oil, is a widely recognized safe food additive. Studies have revealed that GML has antibacterial properties, making it a popular choice for extending the shelf life of agricultural and food products (Chen et al., 2019). However, because of its limited water solubility, challenges arise in its application, prompting researchers to explore its utility in the form of microemulsions. Research indicates that the use of GML in microemulsion forms effectively retains its antibacterial properties, making it suitable for prolonging the usability of food and fresh produce (Yu et al., 2017). Moreover, GML has been used to enhance the storage stability and barrier properties of natural biofilm packaging. This biofilm not only aids in preservation but also contributes to increased flexibility and reduced gas permeability, particularly against chitosan (Vinod et al., 2020). Furthermore, the incorporation of GML into biofilm packaging enhances its storage capabilities and insulating properties, aligning with the natural characteristics of the biofilm.

Because of the challenges encountered in the development of products or fabricated items from lotus leaves, the use of substances with properties that extend lifespan, resist microbial growth, and preserve the distinctive patterns of lotus leaves has become an interesting approach for enhancing the characteristics of lotus leaves in various applications. Notably, prior studies on the combined use of both coating substances on lotus leaves are lacking, making it an intriguing subject for investigation. This paper focuses on studying the preliminary effects of glycerol coating on lotus leaves by investigating the coating time, optimal concentration of glycerol, and physical properties of coated leaves.

2. MATERIALS AND METHODS

2.1 Coating materials

Analytical-grade acetic acid, chitosan, and glycerol were obtained from Chemical Express Co., Ltd. (Thailand). Cosmetic-grade beeswax was supplied by Bangkok Chemical Co., Ltd. (Thailand), and coconut oil was procured from Phiphek (Thailand).

2.2 Lotus leaf preparation

The preparation of dried lotus leaves involved cutting the leaves along the stem to obtain leaves with dimensions of 8 × 8 cm. Subsequently, these cut lotus leaves were dried in an oven at 70°C for 40 min, with the leaves being flipped halfway through the drying process, i.e., at 20-min intervals. Once the allotted time had passed, the dried lotus leaves were left to cool in a desiccator before being stored.

2.3 Glycerol biocoating preparation

Before further study, 5 g of chitosan was added to a 5% acetic acid solution. Subsequently, glycerol was prepared at concentrations of 4%, 8%, 12%, 24%, 36%, and 48% v/v in 5% w/v acetic acid and stirred to achieve a homogeneous solution. Lotus leaf samples were soaked in glycerol solution for 1–7 days. The 40 × 15 mm-coated lotus leaves were folded in half to induce creases to determine the recovery percentage using iTeh Standard, ISO 2313-1. The samples were then subjected to pressure by placing a 500-g weight on top for 5 min. Subsequently, the folded lotus leaf samples were laid on a holding platform to allow them to return to their original state. An indicator board was positioned behind the platform to measure the degree of leaf recovery. The samples were left to recover for 5 min, after which the angle of recovery (A) was measured and recorded. The obtained values were then used to calculate the recovery percentage according to Equation (1).

$$Recovery\ (\%) = \frac{A}{180} \times 100 \qquad (1)$$

2.4 Moisture content analysis

In this study, coated lotus leaves were weighed, and the weights were recorded as the initial weight (A) before drying. Subsequently, the coated lotus leaves were placed in a hot air oven at 105°C for 1 h. Upon completion of the drying period, the dried lotus leaves were reweighed, and the recorded weight was noted as the final weight (B) after drying. The moisture content percentage was then calculated using Equation (2).

$$Moisture\ content\ (\%) = \frac{(A-B)}{A} \times 100 \qquad (2)$$

This procedure was repeated three times for each sample to ensure consistency and reliability of the results.

2.5 Thermal gravimetric analysis

Lotus leaf samples were prepared in alumina crucibles and heated at 10°C/min under a nitrogen (N_2) flow rate of 30 mL/min. The analysis was conducted over a temperature range of 50–600°C, and the weight change of lotus leaves with respect to temperature was examined through graphical analysis.

2.6 Mechanical testing

Investigations of the mechanical properties were conducted using a testometric testing (ISO 527-2) machine equipped with a 50-kN load cell. The lotus leaf samples of dimensions 15 × 80 mm were prepared for testing, and the testing procedure involved a testing speed of 5 mm/min. The experiments were designed to measure Young's modulus, tensile strength, and elongation peak of the samples, with each test conducted in triplicate to ensure statistical reliability.

2.7 Water-repellent property analysis

The water-repellent lotus leaves analysis followed the ASTM-D7334 standard and was evaluated by measuring the contact angle. The contact angle measurement involved placing a lotus leaf on a smooth surface and depositing a single water droplet onto the leaf. Subsequently, the lateral spreading of the water droplet was captured, and the contact angle was measured using an angle meter. This procedure allowed the assessment of the leaf's water repellency with a higher contact angle indicating enhanced water resistance.

3. RESULTS AND DISCUSSION

3.1 Optimal soaking duration

This study explored the appropriate soaking duration for lotus leaves in coating materials to improve the flexibility properties of dried lotus leaves. Various soaking durations, specifically 1, 2, 3, 4, 5, 6, and 7 days, were investigated to assess their effect on the overall flexibility of the coated lotus leaves. A comparison of lotus leaf characteristics at different immersion durations for coating is presented in Table 1. Subsequently, lotus leaf samples were tested under the same conditions as those in the preceding study. As shown in Table 1, all lotus leaves maintained a similar dark brown color and exhibited comparable moisture retention without noticeable drying. The obtained results regarding the performance of elasticity were also analyzed. As analyzed in the recovery percentage, which is shown in Figure 1, the coated lotus leaves displayed closely aligned recovery percentages, even with immersion duration by up to 7 days. No statistically significant differences were observed. However, a 62.59% recovery of coated lotus leaves was achieved compared with only 42.96% for the uncoated lotus leaves. The increase of 19.63% in recovery percentage was attributed to the ability of the lotus leaves to efficiently absorb the coating material within the leaf volume from the first day.

Consequently, the results indicated that the optimal immersion duration for enhancing lotus leaf properties was 1 day. Even when longer immersion durations were used, the outcomes did not significantly differ. Because of the loss of water-repellent properties after lotus leaves undergo drying, leaves with a cellulose-like fiber structure experienced complete absorption of the coating throughout their volume from day 1. This resulted in similar outcomes in subsequent periods. Moreover, prolonged immersion could affect the condition of lotus leaves and lead to unnecessary time consumption. Therefore, the adjustment of lotus leaves through a 1-day immersion period was deemed effective and appropriate for further study.

3.2 Optimal glycerol concentration

The experiment aimed to determine the optimal concentration of glycerol in biocoating for enhancing the flexibility of dried lotus leaves. Various concentrations of glycerol (4%, 8%, 12%, 24%, 36%, and 48% v/v) were investigated for their effectiveness in modifying the quality of lotus leaves by immersion coating. Each glycerol concentration in the coating material had a consistent immersion time of 1 day, as shown in Table 2. The glycerol-free samples retained their greenness, displayed rigidity, and exhibited the lowest recovery percentage. At the initial glycerol concentration, a coating of 4% v/v exhibited a dark brown shade compared with the untreated green leaves. Furthermore, the glycerol-coated lotus leaves showed reduced rigidity, appeared moist, and underwent similar transformations as described earlier.

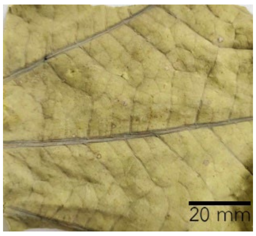
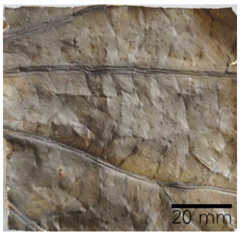
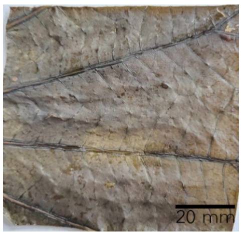
To evaluate flexibility, the recovery percentage was examined according to the ISO 2313 standards. A material with a 180° recovery indicates a high flexibility property, capable of returning to its original state. The results showed an increasing trend in percentage recovery with higher glycerol concentrations. As shown in Figure 2, the lowest recovery percentage was 32.22% for untreated lotus leaves, which gradually increased until it reached a plateau of approximately 36% and 48% v/v glycerol concentrations. This study concluded that the optimal glycerol concentration was 24% v/v because it demonstrated significant improvements compared with lower concentrations and exhibited similar results to higher concentrations. A glyceryl quantity of 24% v/v may be sufficient to facilitate intercalation between the molecular chains of cellulose in lotus leaves. A similar result was found by Jutathip (2019), who studied the effect of glycerol on silk fibrin films. The results show that the incorporation of 20% glycerol achieved optimal conditions because the hydroxyl group of glycerol interfered with the arrangement of silk fibrin, leading to high chain movement. Moreover, further increases in glycerol concentration may result in chemical wastage, affecting cost-effectiveness and diminishing the value of lotus leaf enhancement relative to the marginal improvements observed.

3.3 Biocoating lotus leaf analysis

3.3.1 Moisture content analysis

The moisture content of lotus leaves subjected to modification through immersion in 24% v/v glycerol solution for 1 day was investigated, as depicted in Figure 3. Comparative analysis between modified and uncoated lotus leaves revealed an increase in moisture content by up to 38% in the modified leaf. This enhancement signifies the ability of the leaf to absorb and retain moisture, which is attributed to the glycerol coating. The moisture content, which is governed by the role of glycerol in improving the flexibility of lotus leaves and its excellent adhesive properties (Tarique et al., 2021, 2022), influences various mechanical properties. As discussed in Section 3.2, unmodified dried lotus leaves exhibited significantly lower recovery percentages, indicating that the lower moisture content within these untreated dried lotus leaves plays a crucial role in several mechanical aspects such as strength, flexibility, and resilience to deformation. Consequently, the diminished ability to transmit stress along the fibers made them prone to easy breakage. Despite the glycerol-coated lotus leaves exhibiting desirable properties for further applications, this observation aligns with the findings of Tarique et al. (2021).

Table 1 Lotus leaf characteristics at various coating times

Time required for coating (day)	Characteristics of coated lotus leaves	Time required for coating (day)	Characteristics of coated lotus leaves
Uncoated		1	
2		3	
4		5	
6		7	

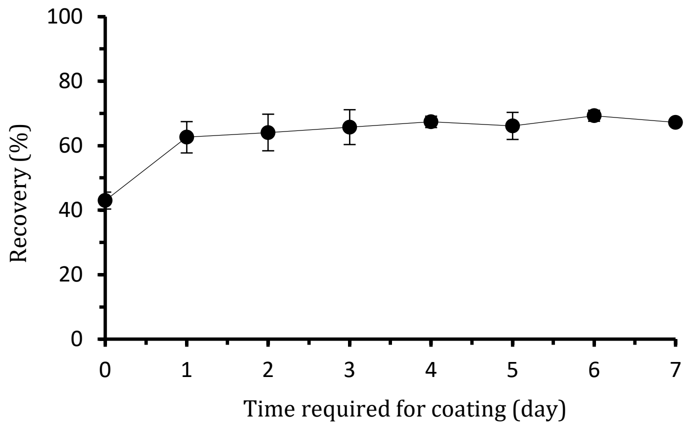


Figure 1. Recovery percentage of lotus leaves using different soaking times at 24% v/v glycerol

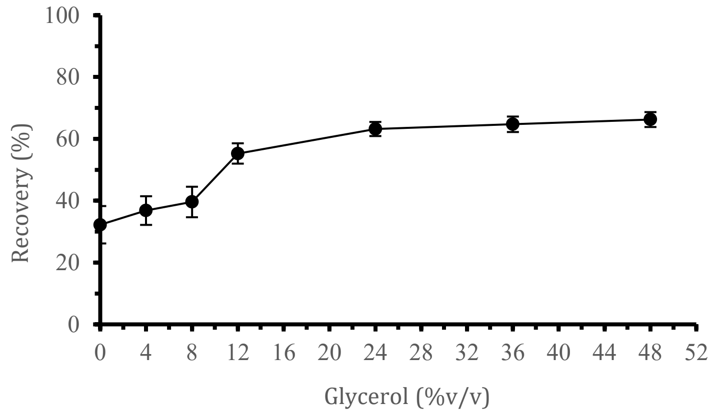
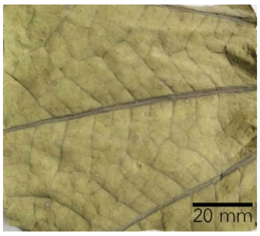
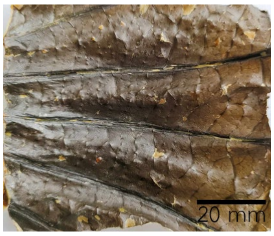
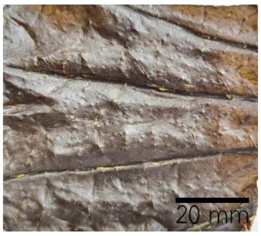
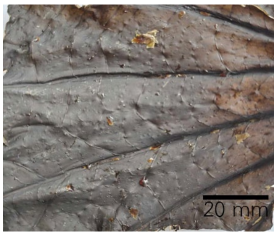


Figure 2. Recovery percentage of lotus leaves using various glycerol concentrations after 1-day soaking

Table 2 Lotus leaf characteristics at various glycerol concentrations

Glycerol content in coating (% v/v)	Characteristics of coated lotus leaves	Glycerol content in coating (% v/v)	Characteristics of coated lotus leaves
Uncoated	 20 mm	4	 20 mm
8	 20 mm	12	 20 mm
24	 20 mm	36	 20 mm
48	 20 mm		

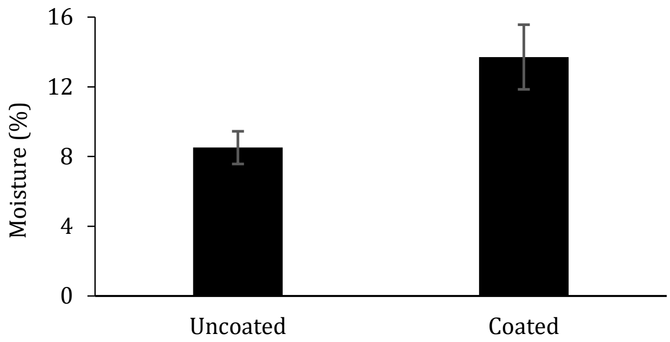


Figure 3. Moisture content between uncoated and coated lotus leaves at 24% v/v glycerol after 1-day soaking

3.3.2 Mechanical testing

The lotus leaves underwent a modification process to enhance flexibility through immersion and coating with glycerol at a concentration of 24% v/v for 1 day. The results revealed a significant change in Young's modulus, tensile strength, and elongation compared with those of dried lotus leaves. Table 3 shows that Young's modulus of coated lotus leaves decreased by 75.61 N/mm^2, or 76%, compared with that of dried lotus leaves. This reduction was attributed to the glycerol coating acting as a plasticizer that infiltrated the fiber matrix, decreasing the interfiber attractive forces. This finding is consistent with that of Wang and Jing (2017), who studied the mechanical properties of biocoated materials modified for flexibility with glycerol, indicating a similar reduction in Young's modulus.

The tensile strength of dried lotus leaves was measured at 1.16 N/mm^2, indicating good interfiber attraction and a certain level of fiber strength. Conversely, lotus leaves coated with glycerol exhibited a tensile strength of 1.23 N/mm^2, indicating a 5.7% increase compared with the uncoated counterpart. This result is consistent with Young's modulus value decreasing after coating lotus leaves with glycerol. Additionally, the results suggest a structural development in the fiber network due to the interaction with glycerol. Effective transmission of mechanical forces within the fiber network resulted in improved tensile strength. In contrast, an increase in the quantity of glycerol beyond a certain threshold decreases the tensile strength, as indicated by Tarique et al. (2021). This study observed a decrease in the maximum tensile strength due to the development of a material through the incorporation of high glycerol concentration, resulting in a predominant composition of glycerol and a reduction in polycellulose components responsible for strength. Consequently, this led to a decrease in the maximum tensile strength.

For the elongation study, lotus leaves coated with glycerol showed an increase of 1.69 mm, representing a 78% increase compared with the uncoated lotus leaves. Glycerol plays a role in reducing the stiffness of cellulose chain fibers, facilitating the elongation of lotus leaf fibers under tension. This enhanced ability to absorb mechanical forces led to increased elongation, making lotus leaves more resistant to tearing (Tarique et al., 2021).

Additionally, because of its chemical properties, chitosan in this experiment could form strong hydrogen bonds with the hydroxyl groups of cellulose, resulting in a reinforced structure and improved water resistance when used as a component in coating materials. Incorporating chitosan into the coating formulation enhances the strength and durability of lotus leaves, thereby reinforcing their mechanical properties.

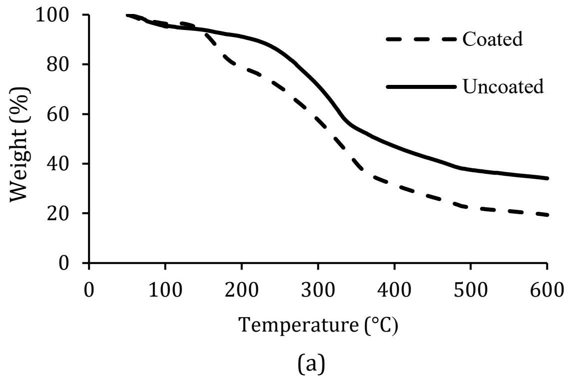
Table 3 Mechanical properties of lotus leaves coated with 24% v/v glycerol and soaked for 1 day

	Values (mean±SD)	
	Uncoated	**Coated**
Young's modulus (N/mm^2)	98.25±0.95	22.64±0.93
Tensile strength (N/mm^2)	1.16±0.12	1.23±0.10
Elongation (mm)	0.46±0.14	2.15±0.42

3.3.3 Thermal gravimetric analysis

Analysis of the heat behavior of the lotus leaves was conducted to identify various components and the composition of the structure of lotus leaves. This was achieved by observing the onset of decomposition, which occurred because of a decreasing trend in the weight change of the sample. The data confirm that there was glycerol within the lotus leaves modified with a 24% v/v glycerol coating after 1 day of immersion. As presented in Figure 4a, uncoated lotus leaf analysis revealed the main decomposition stages in three steps. In the initial stage, within the temperature range of 70–140°C, moisture evaporation occurred. The second stage, between 140°C and 340°C, involved the decomposition of cellulose and hemicellulose polymers. The final stage, within the temperature range of 340–460°C, was associated with the decomposition of lignin components. At this point, the weight stabilized until it reached 600°C. The major carbonaceous components, such as char, cellulose fibers, and lignin, remained unchanged at elevated temperatures. Using the TGA technique, it was evident that the modification of lotus leaves with 24% v/v glycerol coating lead to a reduction in weight during four distinct stages. In the first stage, between 70°C and 140°C, moisture evaporated along with the decomposition of the coating wax, and acetic acid was used in the coating process. The second stage, occurring between 140°C and 240°C, involved the decomposition of glycerol in the presence of hemicellulose. The third stage, within the range of 240–340°C, involved the simultaneous decomposition of cellulose fibers, hemicellulose, and chitosan. The final stage, between 340°C and 460°C, involved the decomposition of lignin. Additionally, chitosan underwent decomposition at temperatures ranging from 340°C to 460°C, which are relatively high temperature. When incorporated into the coating, this property enhanced the thermal properties of lotus leaves, thereby increasing their heat resistance.

The TGA results align with the DTG analysis presented in Figure 4b, where a similar second peak was observed, resembling the study conducted by Yaradoddi et al. (2022) on the thermal stability of biocomposite materials derived from orange peel. The addition of glycerol to facilitate reshaping and enhance mechanical properties resulted in a significant weight loss at four main stages, similar to that observed in uncoated lotus leaves. The graph further illustrates the different thermal stability of coated and uncoated lotus leaves. In their natural state, uncoated lotus leaves exhibit molecular cellulose chains connected by van der Waals forces, imparting low thermal stability, i.e., flexibility in response to heat. This is advantageous for use as a fuel, yielding high thermal energy. However, in glycerol-coated lotus leaves, the decrease in van der Waals forces results in a more flexible structure, leading to enhanced thermal stability. This is a notable advantage in heat dissipation, as indicated by the TGA and DTG analysis. Therefore, glycerol-coated lotus leaves exhibit superior thermal stability than uncoated leaves at the same temperature. This is evidenced by the data presented in the graphs, which emphasize the role of glycerol as a plasticizer within lotus leaves.



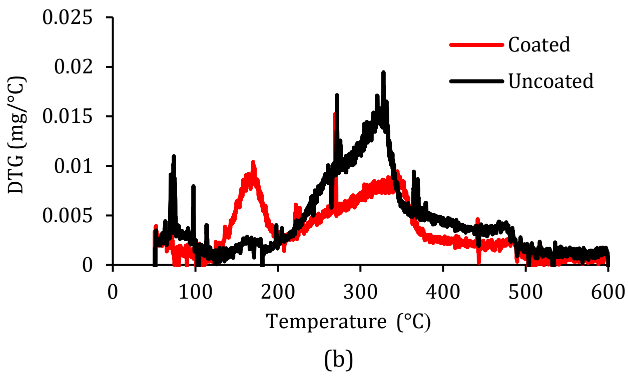


Figure 4. Thermal analysis of lotus leaves at 24% v/v glycerol, soaked for 1day (a) TGA and (b) DTG

4. CONCLUSION

This study investigated methods for enhancing the flexibility of lotus leaves by applying a coating. Glyceryl was selected as the coating component, and the coating process involved immersion. Subsequently, this study explored the optimal conditions that influenced the flexibility of lotus leaves, along with an examination of the improved physical properties after treatment. The experimental findings indicate that using a coating with glyceryl as a component through immersion significantly improves the flexibility of lotus leaves. This suggests that the use of glyceryl in the coating yields favorable results for enhancing lotus leaf flexibility. Furthermore, immersion coating promotes better adjustment of lotus leaves under optimal conditions, which include a solution of 5 g chitosan, 24% v/v glyceryl, and 76% v/v acetic acid, with an immersion time of 1 day. Furthermore, the lotus leaves treated with the coating exhibited distinct characteristics compared with untreated dried lotus leaves, such as an increase in moisture content and elongation due to the absorption of the glyceryl coating. Moreover, glycerol enhanced the fibers of lotus leaves, resulting in improved mechanical properties. Thermal stability analysis using TGA revealed a decrease, consistent with the property of glyceryl, which aids in lowering the material's decomposition temperature. Confirming the presence of glyceryl in lotus leaves, the peak of glyceryl decomposition was identified in the second stage of decomposition.

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