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Hydrophobicity of golden-phase leaves coated with zinc oxide-based nanocomposite for decorative local products

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Abstract

Golden-phase leaves are widely used in decorative crafts and heritage products, yet their susceptibility to moisture limits long-term durability, especially in humid environments. This study investigates the wetting behaviour of zinc oxide (ZnO)-based nanocomposite coatings designed to preserve the hydrophobicity and service life of golden-phase leaves. The uncoated leaf exhibits strong intrinsic water repellency, with a water contact angle of 151°, attributed to hierarchical surface roughness that supports a Cassie–Baxter-like wetting regime. However, probe liquids with lower surface tension show reduced contact angles, indicating partial transitions toward Wenzel-like wetting. To compare three coating formulations, ZnO/chitosan (brush), ZnO/polyvinyl alcohol (PVA) (spray), and ZnO/chitosan/PVA (spray) were prepared and evaluated. The binary coatings decreased water contact angles to below 85° due to the hydrophilicity of chitosan and PVA. In contrast, the ternary ZnO/chitosan/PVA coating produced a high water contact angle of 136° and an exceptionally low surface free energy of 2.4 mN/m, tend to suppress liquid infiltration. The results demonstrate that the ZnO/chitosan/PVA coating applied by a simple spray process, alters the apparent wetting behavior of a naturally hierarchical biological substrate. This experimental observation of wettability enhancement on a natural surface complex morphology-dominated is relevant to preservation of decorative natural materials.

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Keywords: Golden leaf vine, Zinc oxide, Hydrophobic coating, Surface wettability, Contact angle

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1. Introduction

Hydrophobicity of plant leaves is one of nature's most fascinating manifestations, vividly exemplified by the self-cleaning behavior of lotus leaves, where water droplets roll off the surface carrying away dirt particles [1–3]. This phenomenon has long inspired studies on surface wettability, an essential physical property describing the interaction between liquids and solid surfaces [3–5]. Wettability is quantitatively assessed through contact angle measurements, which provide insight into how a liquid spreads or adheres to a surface. The measured angle, formed at the interface between the liquid droplet and the solid, reflects the balance of interfacial energies among the solid, liquid, and surrounding gas phases [6]. Factors such as surface chemistry, micro- and nanoscale roughness, and intrinsic sur-

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face free energy govern this behavior. Typically, the contact angle is measured using the sessile drop method, where a droplet of controlled volume is deposited on the surface. Understanding such surface–liquid interactions underpins both fundamental surface science and the design of functional materials with tailored wetting properties [7].

One of the botanical marvels of southern Thailand is the golden-phase leaf derived from the golden leaf vine [8, 9]. This climbing vine, which can ascend up to 30 meters by twining around large host trees, bears distinctively heart-shaped leaves with deeply concaved lobes. The foliage undergoes a remarkable chromatic transformation over time. Newly emerged leaves appear reddish-copper or pinkish, gradually turning bronze within weeks, then golden after approximately three months, and finally silvery after six to seven months. Mature leaves typically measure 10–18 cm in length, with some exceptional specimens reaching up to 25 cm. This rare species is endemic to the rainforests of Narathiwat Province, particularly in the Bacho Waterfall area and the Hala Bala Wildlife Sanctuary. Because golden-phase leaves are revered as symbols of love, prosperity, and happiness in Thai culture, they have increasingly been utilized in decorative crafts and cultural products, where their natural luster enhances both the aesthetic and economic value of local handmade items [10].

Zinc oxide (ZnO)-based nanocomposite coatings have emerged as an effective strategy for preserving and enhancing natural materials, whose aesthetic qualities are otherwise vulnerable to aging and fungal deterioration [11, 12]. The multifunctionality of ZnO as a surface-protective agent maintains the structural integrity of coated materials. When applied as nanoparticles, ZnO enhances ultraviolet (UV) resistance, mechanical durability, and antimicrobial efficacy across various substrates including textiles, polymers, and packaging films [13, 14]. Over the past decade, ZnO has been increasingly used in combination with biopolymers, particularly chitosan [15, 16]. ZnO/chitosan nanocomposites have demonstrated controlled crystallite sizes and enhanced surface area depending on the chitosan concentration [12, 17, 18]. Embedding ZnO nanoparticles within chitosan matrices also improves coating stability and thermal resistance. ZnO/chitosan coatings act as efficient barriers against moisture and microbial contamination, extending the shelf life of packaged foods [19–23]. To obtain lower-viscosity formulations for spray coating, ZnO has been incorporated into polyvinyl alcohol (PVA), producing nanocomposites with good antimicrobial and mechanical performance [24, 25]. Additionally, the combination of chitosan and PVA enhances adhesion, durability, and structural integrity of films [26].

Responding to community needs for water-repellent and longer-lasting decorative applications, this study investigates a ZnO-based nanocomposite formulation for the protective coating of golden-phase leaves. The aim is to develop a coating that preserves their inherent hydrophobicity, offering protection for a complex natural surface. Using different coating methods and formulations, contact angles of the coated and uncoated leaves were compared. Using three probe liquids, the surface free energies were also determined and related to the leaf microstructure.

2. Materials and methods

Golden-phase leaves were obtained from the Golden Leaf Local Enterprise Group in Narathiwat Province, Thailand. The fresh leaves were dried in a plant press at room temperature. After drying for one month, moisture contents were 10–30%. The dried leaves were coated with four different formulations, i.e., Sample A: uncoated control, Sample B: ZnO/chitosan coating applied by brush, Sample C: ZnO/PVA coating applied by spraying, and Sample D: ZnO/chitosan/PVA coating applied by spraying. The synthesis procedures of these ZnO-based nanocomposites were detailed in Pholnak *et al.* [27]. Surface morphology of both coated and uncoated leaves was examined using field-emission scanning electron microscopy (FE-SEM, FEI Quanta 450).

Hydrophobicity was assessed by contact angle measurement and surface free energy analysis using a contact angle meter (Dataphysics OCA-15EC, Germany) equipped with $SCA20_U$ software in sessile drop mode. Water droplets (2 µL) were gently deposited on leaf specimens cut into 1 cm × 3 cm sections mounted on the sample stage. For each sample, three droplets of deionized (DI) water were analyzed and averaged. Surface free energy was determined by using three probe liquids with distinct polar and dispersive components, i.e., DI water, ethylene glycol, and formamide. Three droplets of each liquid were deposited on the sample surface, and the contact angles were recorded.

The surface free energy of the golden-phase leaves was determined using the Wu's method based on contact angle measurements with three probe liquids of known surface tension components [28]. The overall surface tension (γ_L), dispersive component $\left(\gamma_L^d\right)$, and polar component $\left(\gamma_L^p\right)$ of each liquid were as follows.

DI water: $\gamma_L = 72.80\text{mN/m}, \gamma_L^d = 22.13\text{mN/m}, \gamma_L^p = 50.67\text{mN/m}$,

ethylene glycol: $\gamma_L = 48.20\text{mN/m}, \gamma_L^d = 18.91\text{mN/m}, \gamma_L^p = 29.29\text{mN/m}$,

and formamide: $\gamma_L = 58.40\text{mN/m}, \gamma_L^d = 19.81\text{mN/m}, \gamma_L^p = 38.59\text{mN/m}$.

The surface free energy of the coated and uncoated leaves was calculated using Wu's Harmonic Mean method [28]. Contact angles of three probe liquids (water, ethylene glycol, and formamide) were substituted into the working equation:

$$(1 + \cos\theta_i)\,\gamma_{L,i} = 4\left(\frac{\gamma_{L,i}^d \gamma_S^d}{\gamma_{L,i}^d + \gamma_S^d} + \frac{\gamma_{L,i}^p \gamma_S^p}{\gamma_{L,i}^p + \gamma_S^p}\right), \tag{1}$$

where γ_S^d and γ_S^p represent the dispersive and polar components of the total surface free energy, respectively, and i refers to each of the three probe liquids. To facilitate numerical calculation, the following substitutions were made:

$$x = \sqrt{\gamma_s^d}, \qquad y = \sqrt{\gamma_s^p}, \tag{2}$$

and

$$R_i = \frac{(1+\cos\theta_i)\,\gamma_{L,i}}{2}, \tag{3}$$

which transforms the above equation into a linear form:

$$R_i = x\sqrt{\gamma_{L,i}^d} + y\sqrt{\gamma_{L,i}^p}. \tag{4}$$

This yields three linear equations corresponding to the three test liquids. The variables x and y were obtained by solving the system of equations using the least-squares method. The surface free energy components of the solid were then calculated as:

$$\gamma_s^d = x^2, \qquad \gamma_s^p = y^2, \qquad \gamma_s = \gamma_s^d + \gamma_s^p. \tag{5}$$

The resulting surface free energy (γ_S) represents the apparent surface free energy of the leaf surface, influenced by its micro/nanostructure and coating characteristics.

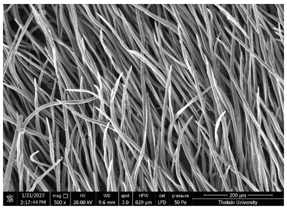
3. Results and discussion

Water droplets in Figure 1a were observed to roll off the surface of the golden-phase leaf, confirming its inherent hydrophobic behavior. This property complements its aesthetic value in handicraft applications, such as the decorated handbag shown in Figure 1b. Similar to lotus leaves, rose petals, and surfaces coated with low-surface-energy materials, the golden-phase leaf exhibits strong water repellency due to its microtextured surface. The FESEM image in Figure 2a reveals dense, hair-like protrusions that create micro-roughness essential for hydrophobic performance. In Figure 2b, a highly magnified fiber shows a diameter of approximately 4700 nm with a tapered tip of about 270 nm. This hierarchical morphology promotes a Cassie–Baxter-like wetting state in which trapped air pockets reduce solid–liquid contact [29]. The average water contact angle of 151° confirms the leaf's superhydrophobicity. Typically defined by contact angles exceeding 150°, such a characteristic suggests potential applications in self-cleaning and anti-wetting surfaces. Figure 3 and Table 1 compare the water contact angles of the uncoated leaf with those coated by three ZnO-based nanocomposites. The ZnO/chitosan (Samples B) and ZnO/PVA (Samples C) coatings yield contact angles of roughly 72° and 87°, respectively. These reductions are consistent with the hydrophilic nature of chitosan and PVA, as well as the presence of hydroxyl groups on ZnO surfaces. When these polar components form the outermost surface, they facilitate hydrogen bonding with water, thereby lowering the contact angle. In contrast, the ternary ZnO/chitosan/PVA (Samples D) coating exhibits a much higher contact angle of approximately 137°. This enhancement is likely due to a morphology-driven shift toward a Cassie–Baxter-like wetting regime. During film formation, the ternary mixture may generate hierarchical roughness, nanoparticle aggregates, or partial phase segregation, which increases air entrapment beneath the droplet.

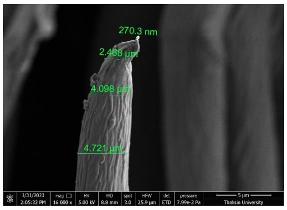
(a)

(b)

Figure 1: Photographs of (a) rolling water drops on golden-phase leaves and (b) a handbag decorated with golden-phase leaves.



(a)

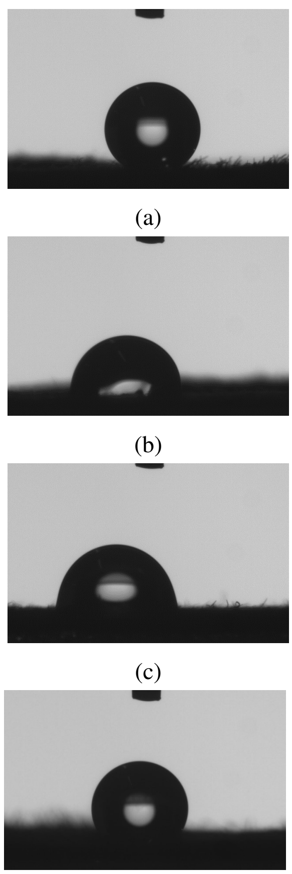


(b)

Figure 2: (a) lowly and (b) highly magnified SEM images of hair-like structure on the surface of a coated leaf.

Table 1: Contact angle and surface free energy of coated golden-phase leaves and the uncoated control using three liquids.

Sample	Coating formulation and method	Average contact angle (degree)			Surface free Energy (mN/m)
		DI water	Ethylene glycol	Formamide	
A	Uncoated control	151±3	135±2	132±2	4.8±0.8
B	ZnO/chitosan brush-coating	72±2	44±6	50±3	38.6±0.7
C	ZnO/PVA spray-coating	87±1	85±2	85±4	20.6±0.8
D	ZnO/chitosan/PVA spray-coating	137±3	130±2	126±3	2.4±0.5



(a)

(b)

(c)

(d)

Figure 3: Images from contact angle measurement showing water drops on (a) sample A (Uncoated control), (b) sample B (ZnO/chitosan coating by brush), (c) sample C (ZnO/PVA coating by spray), and (d) sample D (ZnO/chitosan/PVA coating by spray).

Such microstructural features can reduce effective surface polarity despite the inherently hydrophilic chemistry of the constituents. These findings align with previous reports showing that ZnO enhances water resistance, while the addition of chitosan and PVA improves coating adhesion, durability, and overall barrier performance [30]. Similar hydrophobic improvements have been observed in other ZnO-based systems, such as ZnO combined with titanium dioxide/chitosan [31] or incorporated into acrylic resin matrices [32]. However, the effect of ZnO is not universally hydrophobic. For instance, when ZnO nanoparticles are added to a chitosan–silk fibroin blend, the water contact angle significantly decreases corresponding to more hydrophilic surface [33].

Contact angles measured using ethylene glycol and formamide further illustrate differences in wetting behavior. Ethylene glycol, which has a lower surface tension than water and is moderately polar, produced consistently lower contact angles across all samples. While the ZnO/chitosan coating exhibited the lowest contact angle of 44.24°, the slight decrease to approximately 130° on the highly hydrophobic ZnO/chitosan/PVA coating reflects the retaining of strong spreading. Formamide, with strong polarity and surface tension higher than ethylene glycol but lower than water, displayed decreased contact angles, comparable to those using ethylene glycol. These contact angle values are discussed as average trends rather than absolute surface constants dure to the limited number of measurements and the inherent heterogeneity of natural leaf surfaces. While the observed reduction in contact angles is consistent with a transition from Cassie–Baxter-like wetting to a Wenzel-like behaviour as the liquid penetrates surface asperities [34, 35], a rigorous identification of wetting states of coated leaves requires quantitative roughness parameters and dynamic measurements such as contact-angle hysteresis or sliding angles. The wetting-state discussed in this study is therefore interpretive rather than conclusive, consistent with the data available.

Contact angle data from the three probe liquids were used to estimate the surface free energy via Wu's method [28]. Water served as a polar probe, ethylene glycol as a predominantly dispersive probe, and formamide as an intermediate polarity liquid. The resulting apparent surface free energies, calculated using Wu's harmonic mean method, are summarized in Table 1. The ZnO/chitosan brush-coated surface exhibited the highest apparent surface free energy (38.6 ± 0.7 mN/m), consistent with the intrinsic hydrophilicity of chitosan. The ZnO/PVA spray-coated sample showed a moderate value of 20.6 ± 0.8 mN/m. In contrast, the ZnO/chitosan/PVA spray-coated surface yielded a remarkably low apparent surface free energy of 2.4 ± 0.5 mN/m, corresponding to the strongest observed water-repellent behavior among all samples.

It should be emphasized that this value represents an effective or apparent surface free energy, rather than an intrinsic thermodynamic property of the coating material. When applied to highly rough, heterogeneous, and porous natural substrates such as leaves, Wu's method can significantly underestimate the intrinsic surface energy due to morphological effects. Trapped air pockets, hierarchical roughness, and droplet pinning at surface asperities reduce the real liquid–solid contact area and thus lower the apparent interfacial interaction measured by contact angle analysis. Consequently, the exceptionally low value obtained here reflects the combined influence of surface chemistry and micro–nano topography, rather than purely chemical surface energetics. Despite these limitations, the pronounced reduction in apparent surface free energy provides strong evidence of a highly water-repellent interface, which is advantageous for limiting moisture ingress in natural fibers, papers, and polymeric substrates [36].

In addition to modifying surface wettability, the long-term preservation of golden-phase leaves requires improvements in environmental and physical stability. Using the same ZnO-based coating formulation, our previous work has already demonstrated enhanced color retention of coated golden-phase leaves under QUV accelerated aging tests [27], indicating promising resistance to moisture and UV degradation. While comprehensive durability metrics such as abrasion resistance and adhesion strength remain to be investigated, these initial results suggest that the coating may contribute to improved longevity under environmental exposure.

Future work should extend beyond wetting characterization to include detailed surface chemical analysis, which would significantly strengthen the mechanistic interpretation of the observed wetting behavior. Techniques such as Fourier-Transform Infrared Spectroscopy and X-ray Photoelectron Spectroscopy would enable direct verification of surface functional groups and confirm the proposed synergistic interactions among ZnO nanoparticles, chitosan, and PVA, which in the present study are inferred primarily from morphology and supported by prior literature. Furthermore, ongoing collaboration with local artisan communities in Phatthalung Province aims to explore the practical implementation of coated golden-phase leaves in environmentally responsible decorative products, thereby building on the findings from this work to link surface engineering with sustainable cultural craftsmanship.

4. Conclusion

The golden-phase leaf exhibits excellent intrinsic water repellency, as evidenced by a water contact angle reaching 151°. This high degree of hydrophobicity indicates a morphology-induced wetting transition, most likely arising from hierarchical surface roughness that entrap air pockets. Such features promote a Cassie–Baxter-like interfacial state, a mechanism further supported by the observed microscale texture in the SEM images. As expected for rough hydrophobic surfaces, probe liquids with lower surface tension, i.e., ethylene glycol and formamide, partially penetrate the surface asperities and thereby yield lower contact angles than water, reflecting a localized transition toward the Wenzel-like wetting behaviour.

The introduction of protective ZnO/chitosan and ZnO/PVA films significantly alters this wetting behavior. Owing to the intrinsic hydrophilicity of chitosan and PVA, these binary coatings reduce the water contact angle to below 85°, indicating that the coating chemistry dominates the surface behavior and suppresses the natural hydrophobicity of the leaf substrate. In contrast, the ternary ZnO/chitosan/PVA composite achieves a markedly higher water contact angle of approximately 136°. This enhancement suggests synergistic interactions among ZnO nanoparticles and the two polymer matrices, which promote the development of a favorable micro–nano surface architecture and a reduction in apparent surface free energy. The estimated value of 2.4 mN/m, while exceptionally low, is consistent with the observed high water repellency of the ternary-coated surface. Overall, this work demonstrates that ternary ZnO/chitosan/PVA coatings provide an effective strategy for modifying wetting behavior while maintaining the aesthetic qualities of natural substrates.

Data availability

The dataset used in this study is available upon request to the corresponding author.

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