Declaration of Academic Integrity

The participating team declares that the paper submitted is comprised of original research and results obtained under the guidance of the instructor. To the team's best knowledge, the paper does not contain research results, published or not, from a person who is not a team member, except for the content listed in the references and the acknowledgment. If there is any misinformation, we are willing to take all the related responsibilities.

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Date 2025.8.23

S.T. Yau High School Science Award

Research Report

The Team

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Title of Research Report

Construction of Superhydrophobic SiO_2@TiO_2/Chitosan Composite Films in a Pure Water System and Their Green Application in Egg Preservation

Date

23/08/2025

Construction of Superhydrophobic SiO_2@TiO_2/Chitosan Composite Films in a Pure Water System and Their Green Application in Egg Preservation

Felix (Ziyi) Feng

Abstract

With the increasing demand for food safety and green preservation, the development of efficient and environmentally friendly new fresh-keeping materials has become an important direction of food science research. As a nutrient-rich and high-consumption food, eggs are prone to water loss, quality degradation and microbial contamination during storage, thereby shortening the shelf life and affecting food safety. Traditional preservation methods have the problems of limited preservation effect or insufficient safety, so it is urgent to explore safe and green solutions.

In this study, SiO_2@TiO_2 nanomaterials with core-shell structure were synthesized by using titanium dioxide nanoparticles as the core and silica as the coating layer, and a new fresh-keeping coating film was prepared by combining SiO_2@TiO_2 nanomaterials with natural polymer chitosan. Compared with the conventional method, the synthesis process of this study does not use organic solvents and additional metal ions. The process is simple, environmentally friendly, and offers the advantages of green synthesis.The characterization results show that the particle size of SiO_2@TiO_2 nanoparticles is mainly distributed at 5-15 nm, with an average of about 9-10 nm, and the particle size is uniform, which is beneficial to form a stable structure and improve the dispersion. The results of safety test showed that the content of Ti in eggs after different coating treatments was at a very low level, which was far lower than the relevant standard limits, indicating that the coating had high safety in application.

The results of storage experiments showed that SiO_2@TiO_2/chitosan composite coating could effectively delay the increase of egg weight loss rate, maintain high Haugh unit and yolk index, and suppressed the rise in albumen pH while markedly lowering the total microbial count on the eggshell surface. Compared with both the untreated and water-washed controls, the coated eggs exhibited significantly improved storage quality and safety.

In summary, the SiO_2@TiO_2/chitosan composite coating constructed in this study has outstanding performance in extending the shelf life of eggs at room temperature and improving the microbial safety level due to its green synthesis route, excellent biocompatibility, and significant antibacterial and barrier properties, providing new experimental basis and theoretical support for the development of sustainable food preservation materials.

Keywords: **SiO_2@TiO_2 nanomaterials; Chitosan coating; Egg preservation; Antibacterial properties; Green synthesis**

Acknowledgement

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Commitments on Academic Honesty and Integrity

We hereby declare that we

1. are fully committed to the principle of honesty, integrity and fair play throughout the competition.
2. actually perform the research work ourselves and thus truly understand the content of the work.
3. observe the common standard of academic integrity adopted by most journals and degree theses.
4. have declared all the assistance and contribution we have received from any personnel, agency, institution, etc. for the research work.
5. undertake to avoid getting in touch with assessment panel members in a way that may lead to direct or indirect conflict of interest.
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7. observe the safety regulations of the laboratory(ies) where we conduct the experiment(s), if applicable.
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9. agree that the decision of YHSA is final in all matters related to the competition.

We understand and agree that failure to honour the above commitments may lead to disqualification from the competition and/or removal of reward, if applicable; that any unethical deeds, if found, will be disclosed to the school principal of team member(s) and relevant parties if deemed necessary; and that the decision of YHSA is final and no appeal will be accepted.

(Signatures of full team below)

Felix Feng

Name of team member:

Yuanhao Wang

Name of supervising teacher:

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1. Introduction

1.1 The origin of the subject

Eggs are the most common food on the daily table, but they are highly perishable. It is possible to deteriorate in a few days at room temperature, while refrigeration, although effective, consumes energy and takes up space. Based on the question of 'whether it can be safely stored at room temperature', I began to explore scientific research.

Initially, I tried to use the 'nano metal + organic solvent' formula for preservation. The laboratory results showed that the inhibition rate was more than 99 %, the weight loss rate was reduced by about 10 %, and all the indexes met the national standards. However, in the campus, community and supermarket display, still encounter consumers questioned: Nanoparticles will not penetrate into the egg? Are organic solvents really completely volatilized? Is it safe for children to eat for a long time?This realization has driven home the point that research outcomes must not merely 'meet the standard', but also be truly trustworthy.

During the visit to the local egg processing plant, I saw that the preservatives used by the workers emitted a pungent odor. Quality inspectors reported that although the products had passed internal inspection, they were still frequently returned by customers for being 'unqualified'. This makes me understand that the real 'green preservation' is not only chemical safety, but also to achieve 'zero doubt'.

To this end, I propose two 'subtraction problems': removing organic solvents, removing metal antibacterial agents, using only food-grade raw materials, and achieving room temperature preservation in a pure water system. The inspiration comes from the lotus leaf effect in nature: lotus leaves do not need chemical agents, and can prevent the attachment of pollutants only by micro-nano structure and superhydrophobicity. Therefore, I hope to construct a similar structure on the surface of the eggshell to achieve green and no residue preservation.

Although traditional cold storage and chemical coating have been widely used in egg preservation, the former relies on energy, while the latter is based on solvents, metals or antibacterial agents, and the safety and public acceptance are still limited. In recent years, biomimetic superhydrophobic surfaces have become a research hotspot, but the existing methods still rely on organic solvents or complex chemical treatment, and the potential risks are not completely eliminated.

To solve this problem, this study proposes a bionic superhydrophobic coating scheme based on pure water system and chitosan assisted. The specific method is to self-assemble SiO_2@TiO_2 core-shell nanoparticles with chitosan in water to form a micro-nano rough surface to achieve superhydrophobic properties of the eggshell. The SiO_2 shell acts as an inert barrier, effectively isolating the photocatalytic activity of TiO_2 from direct contact with food. Chitosan not only enhances the film-forming properties of the film, but also makes it safe and edible. The process does not use ethanol, acid-base or metal ions, and the by-products are only direct water, which is both green and safe.

1.2 Current Status of Egg Storage and Preservation

Eggs are one of the most common dietary sources of protein, containing up to 14% protein. Their amino-acid profile closely resembles that of human proteins, resulting in high absorption efficiency. They also provide fats, vitamins, lecithin, minerals, and various trace elements[1]. However, eggs are highly susceptible to contamination from animal feces, microorganisms, and moisture after being laid. If eggs are mishandled during distribution or transport, or if the eggs are damaged, economic losses can occur and foodborne illnesses—such as Salmonella and Staphylococcus aureus infections—may be triggered [2]. In the United States, Salmonella causes approximately 1.2 million infections annually, resulting in 23,000 hospitalizations and approximately 450 deaths, making it a significant public health concern. Without effective preservation measures, the shelf life of eggs is significantly shortened, and their key nutrients, such as protein and vitamins, continue to decline with prolonged storage [3].

Currently, most commercially available eggs are sold as fresh eggs, some with simple packaging and others transported in bulk to various markets. During storage, eggs undergo moisture and gas exchange through eggshell pores, and coupled with respiratory processes, this leads to weight loss, enlargement of the air cell, and gradual changes in the physicochemical properties of egg white and yolk [4–6]. Initially, the eggshell and eggshell membrane can block microbial entry into the egg contents, but over time, microorganisms can gradually infiltrate and proliferate, eventually causing egg spoilage characterized by egg white liquefaction, yolk dilution, and rupture of the yolk membrane [7].

Although countries such as the United States, Japan and the Netherlands have successively developed complete sets of egg processing equipment that integrate cleaning, disinfection and grading, they still face significant bottlenecks at key points such as the selection of coating materials, toxicological evaluation of disinfectants and establishment of optimal storage conditions. It is urgent to further optimize the normal temperature storage and transportation system through systematic research to improve the microbial safety and quality stability of eggs[8–10].

1.3 Research Progress on Egg Coating Preservation Materials

Composite coating preservation technology is a food storage method developed based on biomimetic principles. At present, considerable research has been conducted on the application of composite coatings in fruit and vegetable preservation [11] and meat preservation [12]. To date, research on the application of composite coatings in egg preservation is still scarce, and there are few related reports. Egg coating preservation mimics the natural function of the eggshell membrane by applying one or more film-forming materials with good gas barrier properties to form a composite coating. The coating can be applied through dipping, brushing, or spraying onto the eggshell surface to seal pores, prevent microbial contamination from the environment, reduce water evaporation and CO_2 loss, inhibit the rise in internal pH and enzyme activity, and prevent egg white liquefaction, thereby maintaining the freshness, quality, and nutritional value of eggs for an extended period [13–15]. The coating anti-corrosion process is simple and easy, does not require special equipment, has low energy consumption and low cost. It can effectively reduce egg weight

loss, extend shelf life at room temperature, enhance eggshell hardness, and reduce breakage, making it a promising preservation approach.

Eggshell coating preservation technology creates an edible, semipermeable barrier on the eggshell surface, sealing the inherent pores[16]. This synergistically inhibits microbial intrusion and water migration within the egg, effectively preserving egg quality. Existing coatings typically possess selective permeability, inhibiting microorganisms like bacteria and mold while also regulating water vapor and gas exchange [17].

An ideal egg coating should form a dense and strongly adherent film on the eggshell surface, with low hygroscopicity, minimal detachment, and moderate enhancement of eggshell mechanical strength [17]. Coating materials should be low-cost, widely available, and required in small amounts to reduce preservation expenses. From a safety perspective, coatings must be free of carcinogenic, teratogenic, or mutagenic effects and should not interfere with auxiliary antimicrobial agents. In addition, considering consumer habits, coatings should not leave greasy residues or affect the appearance of the eggshell, ensuring good sensory acceptance [18].

Based on material sources and properties, current coating technologies mainly include chemical products, fats and oils, natural edible polymers, and their composites [19]. Chemical coatings are typically represented by paraffin, polyvinylidene chloride, and their composites, which achieve preservation by forming an oil layer on the eggshell surface. Early studies demonstrated that paraffin coatings can reduce egg weight loss and delay the decline of yolk index, but potential safety concerns exist, including residual organic solvents, inedibility, and chemical contamination risks. Animal and vegetable oils (e.g., lard, tallow, peanut oil, palm oil, olive oil) can form moisture-sealing layers on eggshells and slow egg dehydration, but prolonged use may result in greasy surfaces, undesirable odors, and reduced consumer acceptance [20–22].

To enhance safety and palatability, recent research has focused on natural edible coating materials—such as chitosan, pullulan and its derivatives, aqueous polysaccharide, plant-derived protein and their synergistic composite coating system [23]. These materials are often combined with surfactants or hydrophobic substances to form composite films, enhancing film density and water resistance. Studies have shown that chitosan coatings can effectively delay egg weight loss, maintain yolk index and egg white pH, and extend the high-quality period of eggs to over seven weeks at room temperature [24]. For example, Chen Wenliang et al. applied 1.5% N-methyl chitosan to black-bone eggs; after three coating applications and 30 days of storage at room temperature, eggs still maintained near-fresh quality, showing significant improvement compared with the control [25]. Xie Jing et al. used chitosan coatings combined with oregano and clove essential oils to treat eggs, achieving a 100% freshness rate, only 0.71% weight loss, and superior yolk index and Haugh unit after five weeks of room-temperature storage [26]. Li Ning et al. used water-soluble soybean polysaccharides as the base, supplemented with anhydrous calcium chloride and glycerol to construct a coating liquid system; after 30 days at room temperature, eggs remained grade A, demonstrating the good preservation performance of polysaccharide-based composite coatings [27].

1.4 Current Challenges in Egg Coating Preservation

Although coating preservation technology has demonstrated certain advantages in extending the shelf life of eggs, multiple challenges remain, limiting its feasibility for industrial and large-scale applications. Firstly, the selection of film-forming agents lacks specificity. Existing research and commercial cases have often directly transferred film-forming agents designed for fruits and vegetables to eggs[28,29]. However, this ignores the significant differences in eggshell pore structure, surface chemistry, and barrier properties compared to fruit and vegetable surface. This makes it difficult for the film properties to meet the physiological and storage requirements of eggs [30].Secondly, the controllability of the coating process is still insufficient.Traditional methods such as dipping, spraying or brushing are difficult to obtain a film layer of uniform thickness under large-scale conditions: if the film layer is too thin, the barrier performance is insufficient; if it is too thick, it is easy to form an anaerobic microenvironment locally on the eggshell, prolonging the drying cycle and increasing the risk of microbial invasion, ultimately endangering the intrinsic quality of the egg product[31]. Thirdly, the existing film-forming materials themselves have inherent defects. Although paraffin can reduce water loss, it often makes the eggshell appear greasy and slippery; more importantly, liquid paraffin may penetrate into the eggshell and induce odor. Animal and plant oil-based coatings have weak film-forming properties and a fast oxidation rate. The peroxides in the oxidation products may pose potential health risks[32]. Even if the physical and chemical indicators such as Hoff units, yolk index and albumen height still meet the standards, the flavor and sensory properties may decline, thereby reducing consumer acceptance. Finally, the industrialization path of film preservation is still unclear. Currently, coating operations are mostly semi-mechanized and lack continuous and standardized equipment and process specifications, which seriously restricts its large-scale promotion in production and distribution links[33]. In summary, existing coating materials and processes face significant bottlenecks in terms of safety, uniformity, sensory quality and scalability. It is urgent to develop green, non-toxic, and water-based coatings with stable film-forming properties to provide sustainable technical support for room temperature storage of eggs.

1.5 Application of Nano-Titanium Dioxide in Food Preservation

Nano-titanium dioxide (TiO_2) is an inert, non-toxic, and environmentally friendly material that exhibits potential antimicrobial activity due to its photocatalytic properties [34]. The core of the bactericidal mechanism of nano-titanium dioxide lies in its photocatalytic activity: after being exposed to ultraviolet light, the valence band holes and conduction band electrons drive the conversion of water/oxygen molecules into hydroxyl radicals (•OH) and peroxide species[35]. The generated reactive oxygen causes oxidative damage to the microbial membrane structure, thereby achieving inactivation. Among various metal oxides used to modify the performance of biopolymer-based packaging, TiO_2 possesses unique properties, providing both UV protection and antimicrobial activity [36].

Nano-TiO_2 exists in three different crystalline forms: rutile (the most stable phase), anatase, and brookite. Among the polymorphs of nano-sized TiO_2, anatase exhibits the strongest photocatalytic activity[37]. Existing research has shown that dispersing TiO_2 nanoparticles into a variety of biopolymer

matrices-such as whey protein and soy protein-can significantly improve the performance of the materials, polylactic acid, and chitosan) can significantly alter the physicochemical properties of the resulting packaging materials [38].

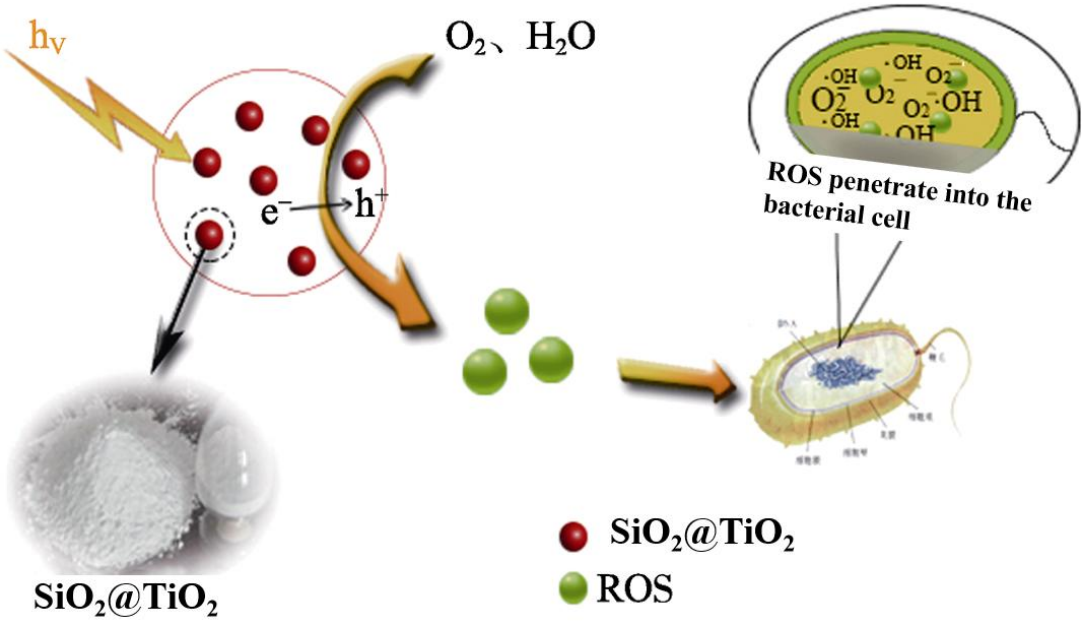

Figure 1. The antibacterial mechanism of SiO_2@TiO_2 composites

In recent years, titanium dioxide (TiO_2) nanoparticles have garnered extensive scholarly attention owing to their low cost, safety profile, and non-toxic nature. One notable feature of nano-TiO_2 is its photocatalytic activity, which has significant implications for fruit and vegetable packaging applications. Yin et al. prepared SPI-MCS-Nano TiO_2 composite films via crosslinking modification and applied them to the preservation of cherry tomatoes during storage. The results showed that the incorporation of TiO_2 nanoparticles effectively mitigated pectin conversion within the fruit and inhibited softening and decay [39]. Qiao et al. investigated the effects of chitosan-nano-TiO_2 coatings on ready-to-eat cantaloupe, and found that the composite film treatment maintained vitamin C content and prevented the reduction of soluble solids compared to uncoated samples [40]. Zhang et al. fabricated biodegradable composite films based on pullulan-carboxymethyl cellulose-nano-TiO_2 via solution casting; the photocatalytic activity of TiO_2 inactivated bacteria, reduced weight loss, and preserved firmness, vitamin C content, and color of strawberries, thereby improving overall quality and extending shelf life [41]. Rokayya et al. developed chitosan-nano-TiO_2-thymol coatings at different concentrations, and reported that the composite films extended the shelf life of blueberries by 8 days [42]. Helal et al. prepared chitosan-nano-TiO_2-sodium tripolyphosphate composite films using sodium tripolyphosphate as a crosslinking agent, which improved the quality of cucumbers during storage, inhibited Salmonella growth, and extended the storage period to 21 days [43].

1.6 Research Significance

This study developed an edible polymer composite coating based on a pure water system for the

preservation and antibacterial properties of eggs at room temperature. Through systematic optimization of the formula and process, the coating has good film-forming properties, barrier properties, and adhesion, significantly reducing the penetration rate of water, oxygen, and microorganisms, thereby extending the shelf life. Organic solvents and heavy metals are abandoned throughout the coating preparation process to avoid the common odors, chemical residues, and safety hazards of traditional technologies. Subsequently, a comprehensive evaluation of water resistance, mechanical strength, antibacterial activity, and sensory quality will be conducted to verify its stability and food safety in actual storage, providing a theoretical and practical basis for the construction of a green and sustainable egg preservation technology.

1.7 Research Process and Technical Route

1.7.1 Preparation and Process Optimization of SiO_2@TiO_2/Chitosan Composite Coating

At the beginning of the study, monodisperse TiO_2 sol was synthesized using the sol-gel process. Subsequently, a silica precursor (e.g., tetraethoxysilane, TEOS) was introduced into the TiO_2 sol, and under controlled pH and reaction conditions, hydrolysis occurred to generate SiO_2, which deposited onto the TiO_2 surface to form core–shell structured SiO_2@TiO_2 composite particles. The obtained SiO_2@TiO_2 composites were then combined with a chitosan solution, and the composite coating was prepared by optimizing parameters such as chitosan concentration, number of coating applications, and drying conditions. The resulting SiO_2@TiO_2/chitosan composite film exhibited a dense structure, excellent hydrophobicity, and high stability. The core–shell design enhances the mechanical strength, structural stability, and hydrophobic performance of the coating, providing an ideal material basis for egg preservation applications.

1.7.2 Structural Characterization of SiO_2@TiO_2/Chitosan Composite Films

To systematically evaluate the composite film's performance, the study employed a contact-angle goniometer to quantify surface hydrophobicity; the microstructure and distribution of SiO_2@TiO_2 were observed using scanning electron microscopy, and the establishment of its core-shell structure was verified using infrared spectroscopy. Particle-size distribution analyses were conducted to assess the nanoscale dispersibility of SiO_2@TiO_2 hybrids, while Brunauer – Emmett – Teller (BET) measurements provided specific surface area and pore-size data, thereby underpinning assessments of film stability. Finally, the migration behavior of TiO_2 within the films was evaluated to ensure material safety.

1.7.3 Effect of SiO_2@TiO_2/Chitosan-Based Composite Films on Egg Storage, Preservation, and Antimicrobial Performance

The obtained SiO_2@TiO_2/chitosan composite film was directly used as an edible coating and evenly coated on the eggshell surface. Their preservative efficacy was evaluated against an uncoated control group. During storage, egg weight, Haugh unit, albumen pH, yolk index, and total microbial counts in the egg contents were monitored to quantify the impact of the coating on egg quality and shelf life. Additionally, total microbial counts on eggshell surfaces before and after coating were measured to assess the antimicrobial efficacy of the composite films. Combined with sensory evaluation, a comprehensive comparison between coated and control eggs over different storage periods was conducted, providing a

systematic assessment of the application performance of SiO_2@TiO_2/chitosan composite films in egg preservation.

1.7.4 Technical Route

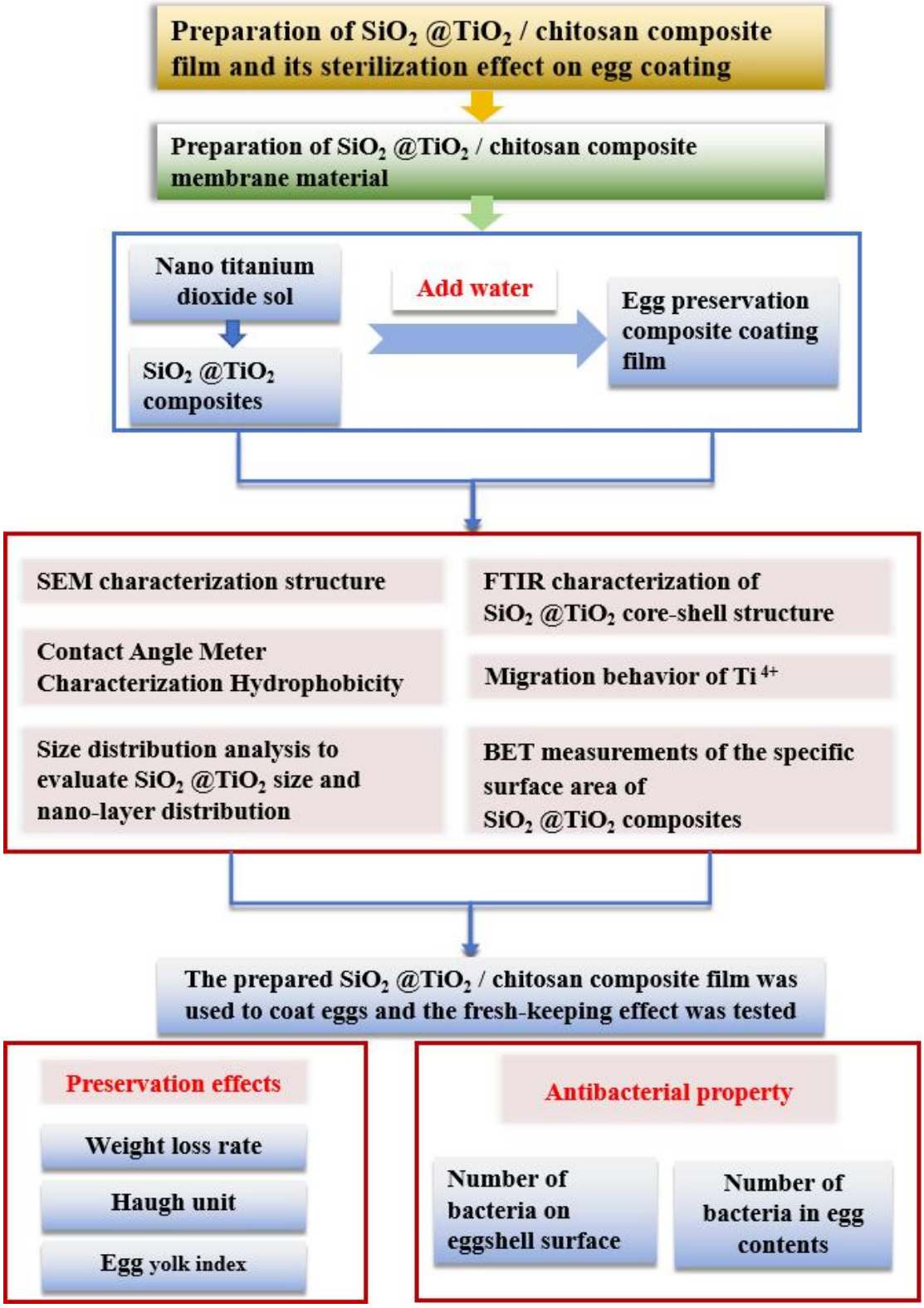

Figure 2. Technical Route of This Study

2. Experimental Section

2.1 Materials and Reagents

Silica precursor (tetraethyl orthosilicate, TEOS), titanium butoxide (TBOT), chitosan (CS), anhydrous ethanol (EtOH), acetic acid, sodium hydroxide, fresh eggs, deionized water.

2.2 Experimental Materials

Glassware: Beakers, volumetric flasks, graduated cylinders, glass rods, Erlenmeyer flasks.

Instruments and Equipment: Analytical balance; Centrifuge, ultrasonic cleaner; Constant temperature magnetic stirrer; Forced air drying oven; Contact angle meter; Field emission scanning electron microscope (SEM); Fourier transform infrared spectrometer (FTIR); Dynamic light scattering particle size analyzer, Specific surface area and porosity analyzer (BET).

3. Experimental Methods

3.1 Preparation of SiO_2@TiO_2/Chitosan Composite Coating

(1) Preparation of TiO_2 Nanocolloid

Prepare the precursor solution by dissolving 3 mL of tetrabutyl titanate (TBOT) in 20 mL of anhydrous ethanol. Slowly add the TBOT solution into 30 mL of deionized water and stir at 70°C for 45 min to obtain a TiO_2 nanocolloid.

(2) Loading SiO_2 onto TiO_2 to Form a Core–Shell Structure

Tetraethyl orthosilicate (TEOS) was used as the silicon source, and controlled hydrolysis was achieved in an ethanol-water co-solvent system. Add the TiO_2 nanocolloid prepared in step (1) and stir under acidic conditions (pH ≈ 4-5, adjusted with acetic acid) for 4 h. Age the mixture at room temperature for 48 h, then dry under vacuum at 60°C for 12 h to obtain SiO_2@TiO_2 composite particles with a core–shell structure.

(3) Preparation of Chitosan Coating Solution

Take 0.4 g of chitosan with a viscosity of approximately 200 mPa • s and add 50 mL of 1% acetic acid solution to fully dissolve it to prepare a chitosan stock solution. Stir thoroughly to obtain a 0.8 g/100 mL chitosan solution.

(4) Preparation of Composite Coating Solution

Add the SiO_2@TiO_2 composite particles from step (2) into the chitosan solution from step (3). Stir well and let stand for 30 min to allow the composite particles to disperse fully, obtaining the SiO_2@TiO_2/chitosan composite coating solution.

(5) Coating Application

Uniformly spray the composite coating solution onto the surface of fresh eggs using a spray method.

The coating thickness can be precisely controlled by means of the number of spraying cycles. After spraying, allow the eggs to air-dry naturally or place them in a drying oven at 30°C to form a dense composite protective coating, completing the egg coating and preservation process.

3.2 Structural Characterization of SiO_2@TiO_2/Chitosan Composite Film

3.2.1 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

The composite was dried, ground into powder, and its infrared spectra were recorded using ATR. Particular attention was paid to the characteristic peaks of Si–O and Ti–O–Ti, in order to verify the successful construction of the SiO_2@TiO_2 core–shell structure within the composite material.

3.2.2 Particle Size Distribution and BET Surface Area Characterization

The particle size distribution of SiO_2 @TiO_2 composite particles was evaluated using a dynamic light scattering particle size analyzer to confirm their nanoscale dispersion state. The BET method was employed to measure the specific surface area and pore size distribution of the composite film, in order to evaluate its stability and barrier performance.

3.2.3 Structural Characterization of SiO_2@TiO_2/Chitosan Composite Film

The water contact angle (θ) characterizes the wetting affinity between a liquid droplet and a solid film surface: low θ (< 90°) indicates hydrophilicity and good wettability, whereas high θ (> 90°) denotes hydrophobicity and poor wettability [44]. This behavior originates from interfacial energetics; at equilibrium, the contact angle θ is determined by Young's equation, which relates the three interfacial tensions [45].

$$\gamma_{SV} = \gamma_{SL} + \gamma_{LV}\cos\theta_e$$

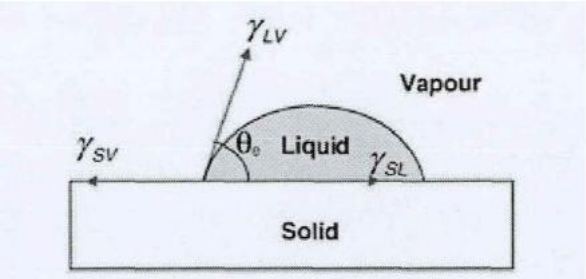

Figure 3. Schematic diagram of contact angle

The contact angles of two coating systems were determined by static sessile drop analysis: (i) EtOH group—SiO_2@TiO_2/chitosan composite coatings prepared with anhydrous ethanol as solvent, evenly applied on a clean glass slide and dried at room temperature; (ii) H_2O group—similar coatings prepared with water as solvent and treated in the same manner. The measurements were performed using an OCA20 video optical contact angle meter.

3.2.4 Scanning Electron Microscopy (SEM) Characterization

The dried SiO_2@TiO_2/chitosan composite films were cut into small pieces, sputter-coated with gold, and observed under a scanning electron microscope to examine the surface morphology, particle distribution, and integrity of the core–shell structure. By comparing the microstructures of the pure chitosan film and the composite film, the compactness and uniformity of the composite film were evaluated.

3.2.5 Ti^{4+} Migration and Safety Evaluation

The migration of titanium ions in untreated eggs and eggs treated with ethanol or water-based systems was quantitatively detected by inductively coupled plasma mass spectrometry (ICP-MS). After immersing the composite film in the simulated solution for a certain period, samples were collected to measure the Ti^{4+} concentration in the solution, thereby evaluating the titanium ion release behavior and safety of the composite film.

3.3 Experiment of the preservation and antibacterial effects of SiO2@TiO2/chitosan composite film on eggs

3.3.1 Experimental Design

A single-factor experimental design was adopted under room temperature conditions, with four different treatment groups: a control group (eggs without any treatment), a water-washed group (eggs washed thoroughly with running tap water and air-dried), an ethanol-based preparation group (EtOH-CS/SiO_2@TiO_2) (preservative prepared using absolute ethanol as the solvent, with eggs were immersed in the preservative solution for 30s, then removed and allowed to dry naturally in a clean air flow), and a water-based preparation group (H_2O-CS/SiO_2@TiO_2) (preservative prepared using pure water as the solvent, with eggs were immersed in the preservative solution for 30s, then removed and allowed to dry naturally in a clean air flow). All eggs were arranged in sterile egg trays, with each treatment comprising three biological replicates of 48 eggs apiece. An additional 10 indicator eggs per group were used to measure egg loss, making 154 eggs per group and a total of 616 eggs.

Six sample eggs were randomly selected from each replicate group on storage days 0, 7, 14, 21, 28, 35, 42, and 49, of which three were used to determine egg quality and three were used to measure the total bacterial count on the eggshell and in the egg contents.

Since egg quality at day 0 is unrelated to treatment, 21 fresh eggs were tested for quality indicators on day 0 as baseline data.

3.3.2 Measurement Methods

(1) Weight Loss Rate

The mass of 10 labeled eggs in each group was weighed using an analytical balance every 7 days. The weight loss rate of each egg at different storage periods was calculated using the following formula, and the average weight loss rate for each group was determined:

$$\text{Weight loss rate (\%)} = \frac{M_1 - M_2}{M_1} \times 100$$

M_1, M_2 denote egg mass at day 0 and after storage, respectively.

(2) Haugh Unit

The Haugh unit of each egg was measured according to the instructions of the SONOVA Egg Quality Analyzer (EggAnalyzer™ , Orka Technology Ltd.).

(3) Yolk Index

After separating the egg white from the yolk, the yolk height and equatorial diameter were measured using a digital caliper, and the yolk index (YI) was calculated based on the results:

$$\text{Yolk Index (YI)} = \frac{\text{Yolk Height}}{\text{Yolk Diameter}}$$

(4) Egg White pH

The separated albumen was homogenized and its pH determined with a calibrated pH meter.

(5) Total Bacterial Count

Assess the total aerobic colony counts in the eggshell and egg contents under a sterile laminar flow hood; make an initial dilution by dispensing 7 mL of sterile saline into a 10 mL tube. Three sterile swabs were moistened with the saline and each used to wipe the egg surface once to ensure thorough collection of surface bacteria. After wiping, the swab tips were broken off into the test tube, immersed, and gently shaken to release all bacteria into the saline, forming a bacterial suspension of the eggshell surface. An appropriate volume of this suspension was then suitably diluted, evenly spread onto agar plates, incubated, and the colonies counted to determine the total bacterial count on the eggshell.

Subsequently, the same egg is cracked and the contents are thoroughly homogenized for later use. A certain volume of the egg contents was taken, diluted, spread onto agar plates, incubated, and colonies counted to determine the total bacterial count in the egg contents. The entire process is carried out under sterile conditions to ensure the accuracy of sampling and counting. The colony forming unit (CFU) count per egg was used as the result indicator [46].

4. Results and Discussion

4.1 Structural Characterization of SiO_2@TiO_2/Chitosan Composite Films

4.1.1 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

To confirm the successful coating of TiO_2 nanoparticles with SiO_2, FTIR spectra were compared for TiO_2, SiO_2, and SiO_2@TiO_2 samples by analyzing their characteristic absorption peaks (Figure 4). The results showed that the SiO_2@TiO_2 sample exhibited a typical Si-O-Si stretching vibration peak in the 1080–1100 cm^{-1} region, with the Ti-O-Ti lattice modes at 600-800 cm^{-1} preserved. Compared with pure

TiO_2, the Ti-O-Ti peak is slightly shifted, while the Si-O-Si peak only appears in the composite, demonstrating a uniform SiO_2 shell over TiO_2 cores, yielding a core-shell architecture

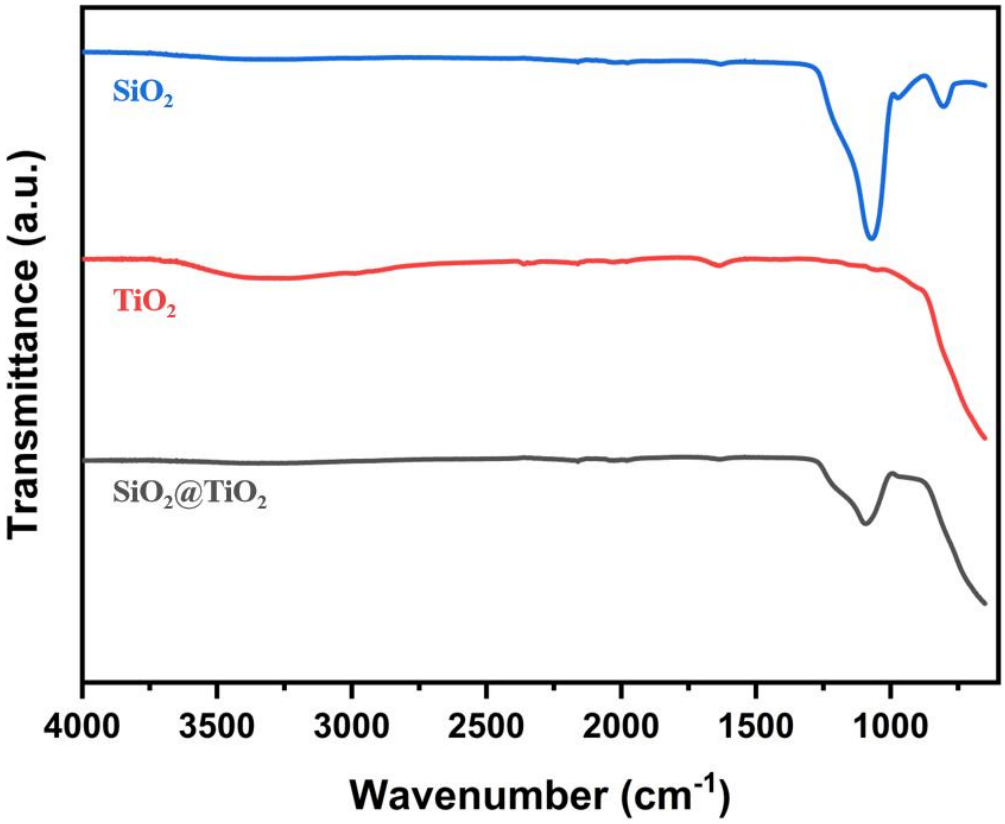

Figure 4. FTIR spectra of TiO_2, SiO_2, and SiO_2@TiO_2 samples

4.1.2 Particle Size Distribution and BET Surface Area Characterization

To evaluate the uniformity of the SiO_2 coating on the TiO_2 nanoparticles and the structural properties of the composite, particle size distribution analysis (Figure 5) showed that the prepared SiO2@TiO2 nanoparticles were mainly concentrated in the 5–15 nm range. The average particle size falls between 9-10 nm, as measured by dynamic light scattering. The distribution was clearly monomodal with a slight shift toward larger sizes. This indicates that most particles were relatively uniform in size, with only a few exhibiting local aggregation or larger diameters. A narrow size distribution and small particle size favor the uniform encapsulation of SiO_2 on the TiO_2 core, densify the core-shell interface, and thus enhance the specific surface area and structural stability of the composite particles.

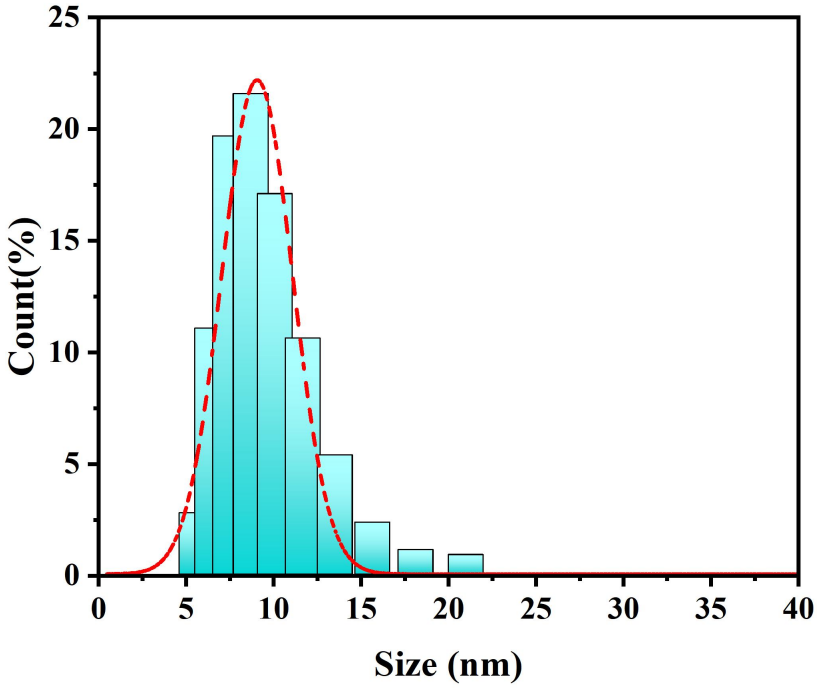

Figure 5. Particle size distribution of SiO_2@TiO_2 nanoparticles

N_2 adsorption–desorption measurements (Figure 6) indicated that the SiO_2@TiO_2 composite material exhibits typical mesoporous characteristics. The multipoint BET specific surface area was 41.5 $m^2 \cdot g^{-1}$, within the common range for mesoporous materials, providing abundant surface active sites. N_2 isotherms were of type IV, exhibiting H2 hysteresis between P/P_0=0.4-0.8, suggesting the presence of an interconnected network of ink-bottle-shaped or slit-shaped mesopores within the material. The BJH (branched joint-joint-joint-half-maximum) pore size distribution reveals a narrow and uniform distribution (full width at half maximum < 5 nm) pores centered at 3-4 nm, none exceeding ~50 nm. This indicates that the SiO_2 shell uniformly coats the TiO_2 surface, effectively suppressing nanoparticle aggregation and the formation of secondary large pores. Overall, the narrow 3–4 nm mesopores combined with a specific surface area of 41.5 $m^2 \cdot g^{-1}$ contribute to enhanced adsorption and barrier performance for water vapor, gases, and organics while retaining structural integrity and mechanical robustness.

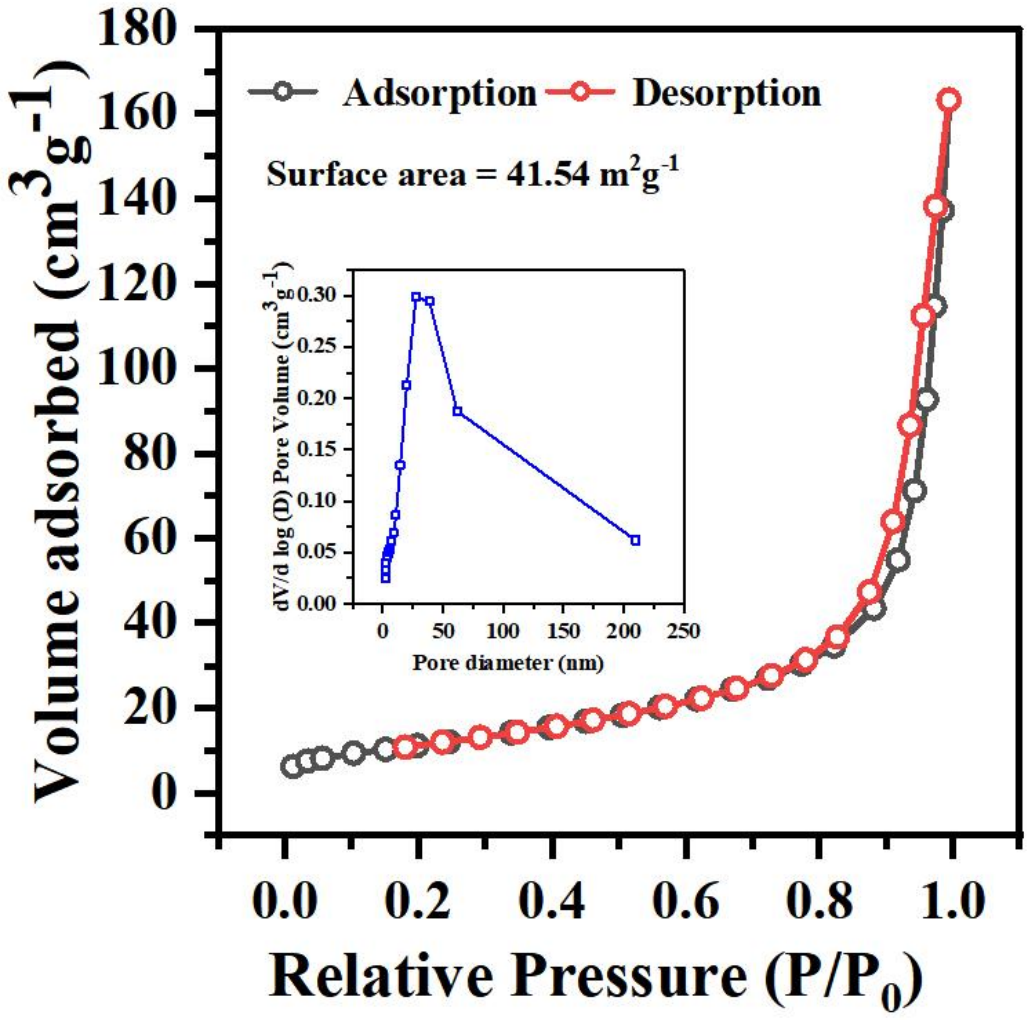

Figure 6. SiO_2@TiO_2 N_2 isotherms and BJH pore-size profile

4.1.3 Water Contact Angle Measurement Results

To investigate the surface wettability and functional performance of SiO_2@TiO_2/chitosan composite films under different solvent conditions, this study compared films prepared with absolute ethanol as the solvent (EtOH-CS/SiO_2@TiO_2 group) and those prepared in a pure water system (H_2O-CS/SiO_2@TiO_2 group). Contact angle measurements (Figure 7) showed that the water contact angle of the EtOH-CS/SiO_2@TiO_2 group group was 138°, indicating strong hydrophobicity, while the H_2O-CS/SiO_2@TiO_2 group exhibited a contact angle as high as 175°, reaching the superhydrophobic level. According to calculations based on the Young equation, the adhesion work of droplets on the EtOH group surface was approximately 18.7 $mN \cdot m^{-1}$, whereas it was only 0.28 $mN \cdot m^{-1}$ on the H_2O group surface, indicating a reduction in droplet adhesion by two orders of magnitude. These results suggest that droplets on the H_2O group surface can hardly stay in place and easily roll off under gravity, demonstrating significant self-cleaning potential.

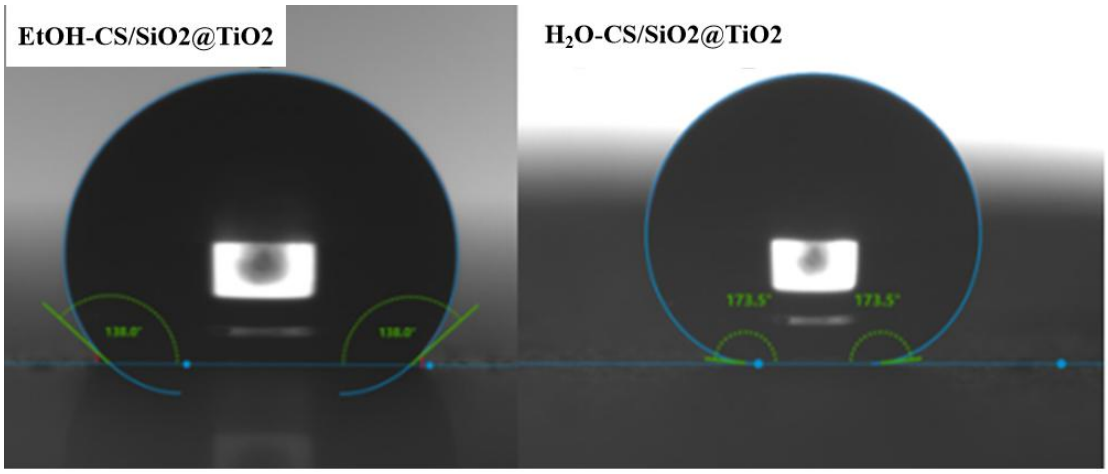

Figure 7. Water contact angles of the EtOH-CS/SiO2@TiO2 group (left) and H_2O-CS/SiO_2@TiO_2 group (right)

4.1.4 Scanning Electron Microscopy (SEM)

The SEM results in Figure 8 show that the surface of the pure chitosan coating is dense and smooth, with no identifiable pores. In contrast, the SiO_2@TiO_2/chitosan composite coating exhibits a loose and irregular microtexture, with uniformly dispersed nanoparticles and some localized aggregation. This structure prevents droplets from staying on the surface, allowing them to roll off quickly under the force of gravity, which thereby endows the material with outstanding self-cleaning and antifouling capabilities. These results indicate that the microstructural design of the composite film and the choice of preparation solvent play a critical role in its functional performance.

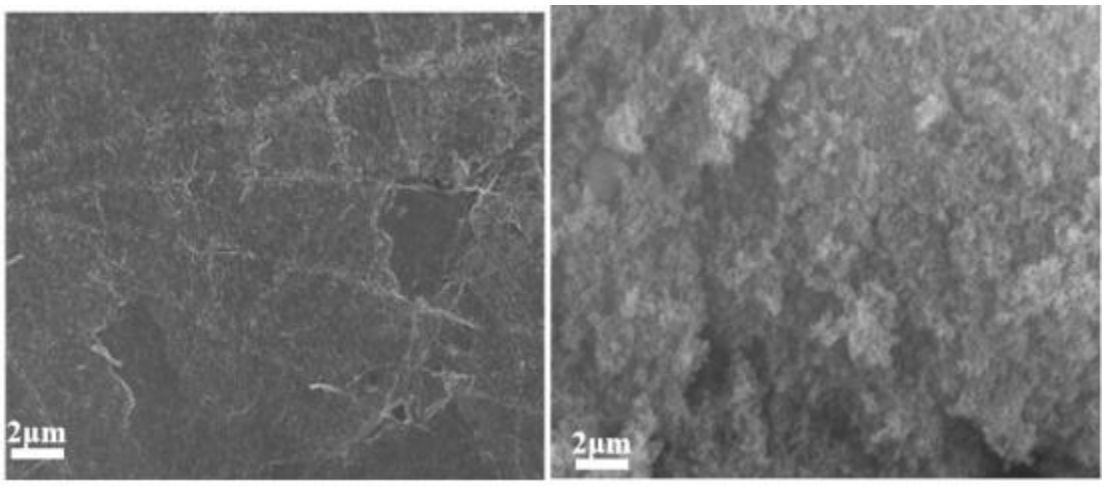

Figure 8. Scanning electron microscope photos: The left picture shows a single chitosan coating, and the right picture shows a composite coating formed by blending SiO_2@TiO_2 and chitosan.

4.1.5 Ti^{4+} migration and safety assessment

To evaluate the effect of the preservative coatings on Ti^{4+} migration in eggs, Ti^{4+} content was measured in untreated eggs and eggs coated with different systems, as shown in Table 1. The data indicated that the Ti^{4+} residue in untreated eggs was 0.827 mg/kg. After coating with the ethanol-based preservative film, Ti^{4+} content slightly increased to 1.00 mg/kg, while eggs coated with the water-based film showed a

Ti^{4+} content of 0.939 mg/kg. Overall, the migration levels were extremely low, indicating that Ti^{4+} migration during the coating process was well-controlled and negligible. The slightly higher migration in the ethanol system may be related to minor effects of the solvent on the surface activity and solubility of Ti^{4+} particles in the coating, whereas the water-based system, with a denser and better-dispersed film, showed slightly lower migration. These results demonstrate that, regardless of the coating system used, the prepared preservative films exhibit good safety in terms of Ti^{4+} residues in eggs, meeting the requirements for food contact materials [47].

Table 1: Ti^{4+} Content in Eggs after Different Coating Treatments (mg/kg)

Type	Ti^{4+} Content (mg/kg)
Control group	0.827±0.05
EtOH-CS/SiO2@TiO2 group	1.000±0.05
H_2O-CS/SiO_2@TiO_2 group	0.939±0.05

4.2 Evaluation of egg storage, preservation and antibacterial properties based on SiO_2@TiO_2/chitosan composite membrane

4.2.1 Weight Loss Rate

As storage time increases, the microporous structure of the eggshell allows internal moisture and metabolic gases to gradually escape through diffusion or evaporation, resulting in a continuous increase in weight loss rate [48]. As shown in Figure 9, the weight loss rate of eggs in each treatment group showed a monotonically increasing trend during storage. Among them, the H_2O-CS/SiO_2@TiO_2 group exhibited the smallest increase in weight loss, remaining lower than the other three groups from days 7 to 49. By the 49th day of storage, the weight loss rates of the three treated samples were reduced by 26.70%, 30.04% and 24.84% respectively compared with the control, water-washed, and EtOH-CS/SiO_2@TiO_2 groups, respectively.

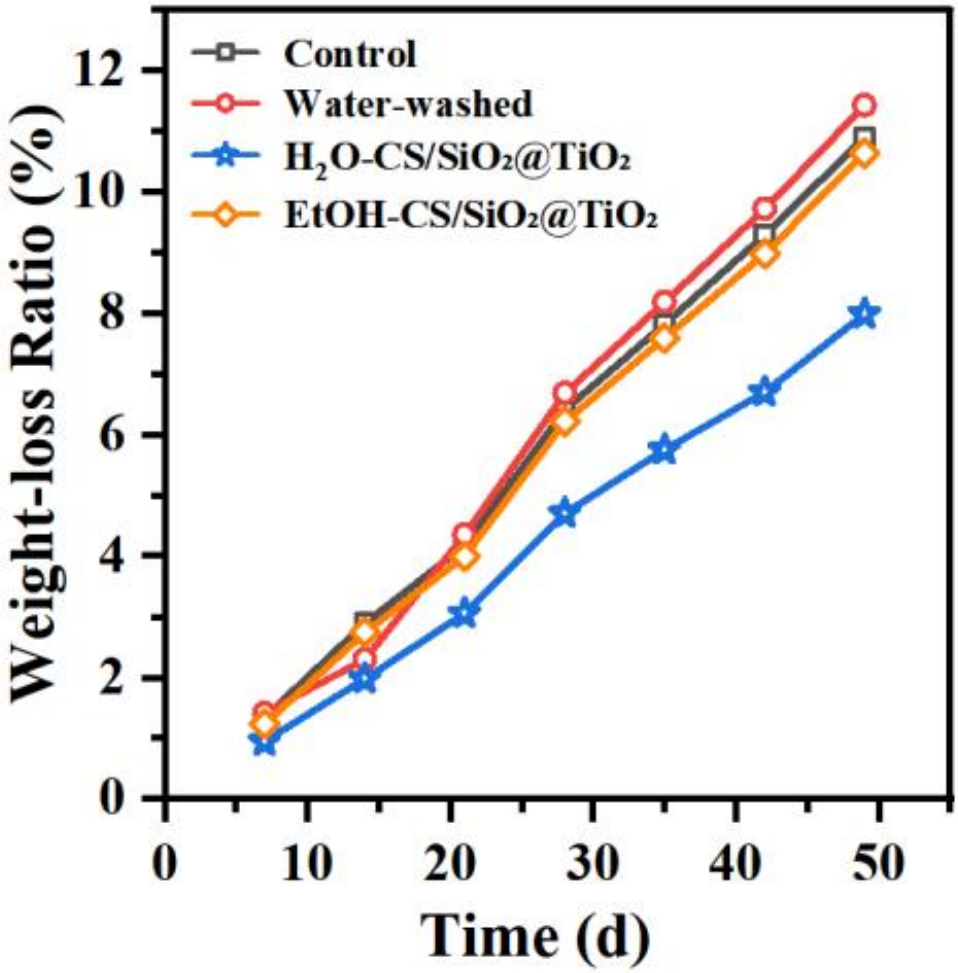

Figure 9. Weight loss rate of eggs under different treatments during storage

The differences among the treatment groups indicate that the H_2O-CS/SiO_2@TiO_2 coating was particularly effective in reducing weight loss, as its sealing effect on the eggshell micropores effectively inhibited moisture loss. The EtOH-CS/SiO_2@TiO_2 group showed a moderate effect. In contrast, the control and water-washed groups exhibited higher weight loss rates, especially the water-washed treatment, which damaged the naturally occurring cuticle on the eggshell surface, weakening its barrier function and accelerating moisture loss, thereby reducing the storage stability of the eggs.

4.2.2 Hartz unit

Concentrated egg white content is positively correlated with egg freshness and is directly reflected in higher HU. According to the HU grading standard, the HU value of AA grade eggs is greater than 72, A grade is between 60-72, B grade is less than 60, and generally high-quality fresh eggs are usually classified as AA grade [48].

As shown in Figure 10, the Haugh unit (HU) of eggs in all treatment groups generally decreased over the storage period, which is consistent with the simultaneous decline in albumen height and egg grade. At

day 0, the HU was 78.99, corresponding to AA grade. By day 7, the differences among the treated groups were minimal. By day 14, the HU of the the the H_2O-CS/SiO_2@TiO_2 group was higher than that of the EtOH-CS/SiO_2@TiO_2 group and the water-washed group. On day 21, the H_2O-CS/SiO_2@TiO_2 group maintained a higher HU than the water-washed group, while being similar to the other two groups.Between days 28 and 42, no significant differences were detected among the groups; however, on day 49, the the H_2O-CS/SiO_2@TiO_2 group exhibited higher HU values than the water-washed and EtOH-CS/SiO_2@TiO_2 groups. Overall, the HU decreased rapidly during the early storage period (days 7–14) and then declined more gradually, with occasional slight increases, possibly due to individual variation among different batches of eggs. These results indicate that the the H_2O-CS/SiO_2@TiO_2 treatment maintained relatively higher HU values at certain time points, demonstrating a notable preservation effect.

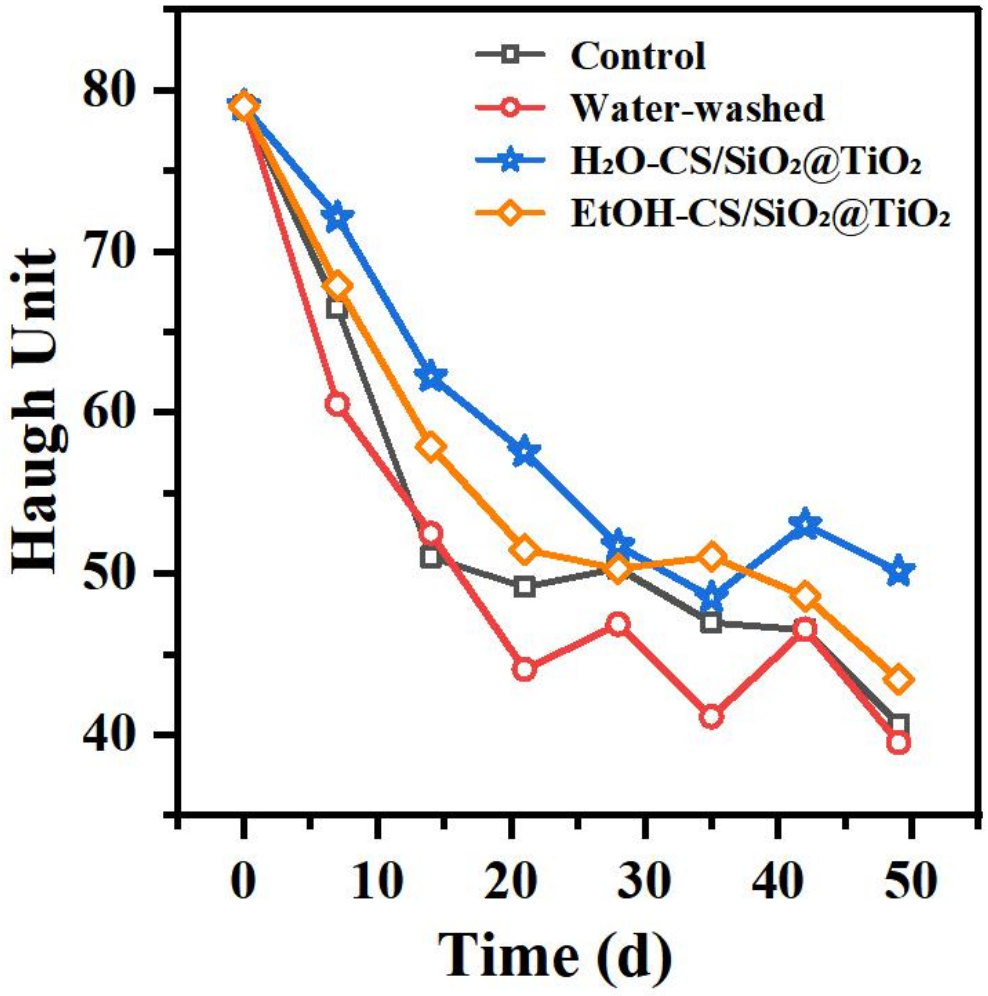

Figure 10. Changes in yolk index of eggs under different treatments during storage

4.2.3 Yolk Index

During storage, protein degradation and water migration into the yolk cause yolk volume expansion and a decrease in height, leading to a reduction in YI, which may eventually result in yolk membrane

rupture and the occurrence of broken yolks [48].

As shown in Figure 11, the YI of eggs in all treatment groups decreased over the storage period. At day 0, the YI of all groups was 0.44. On day 7, the H_2O-CS/SiO_2@TiO_2 group exhibited higher YI than the blank and water-washed groups. No appreciable differences were observed among the treatments between days 14 and 28. On day 35, the H_2O-CS/SiO_2@TiO_2 group showed higher YI than the water-washed group, while being comparable to the EtOH-CS/SiO_2@TiO_2 and blank groups. On day 42, the YI of the H_2O-CS/SiO_2@TiO_2 group was similar to that of the EtOH-CS/SiO_2@TiO_2 group; the blank group had nearly all eggs broken and unmeasurable, and only one egg in the water-washed group remained intact. By day 49, only the H_2O-CS/SiO_2@TiO_2 group maintained intact yolks, while the EtOH-CS/SiO_2@TiO_2 group had only one egg remaining intact.

These results indicate that the H_2O-CS/SiO_2@TiO_2 treatment effectively preserved yolk structure during storage, with a relatively smaller decline in YI compared to the water-washed group, which showed the greatest reduction. Overall, the coating demonstrated a clear preservation effect by inhibiting albumen degradation and preventing yolk rupture.

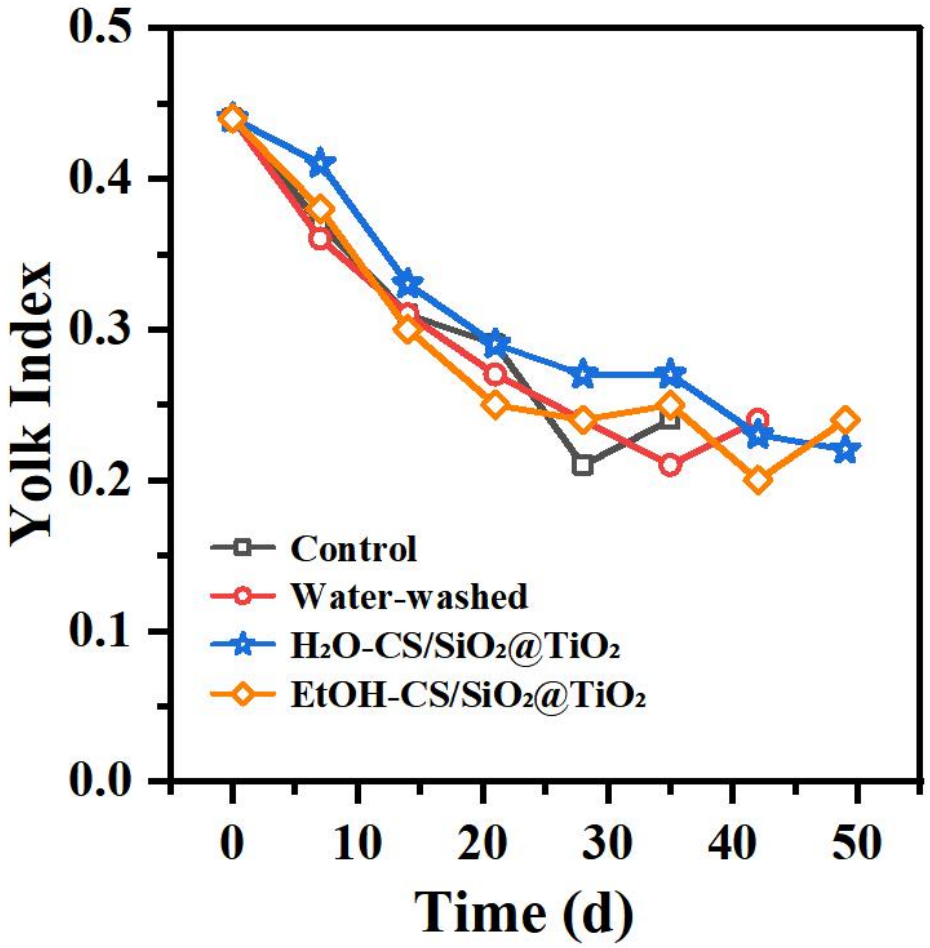

Figure 11. Changes in albumen pH of eggs under different treatments during storage

4.2.4 Albumen pH

Previous studies have shown that the pH of fresh egg albumen is approximately 8.73 [48], which is consistent with the measured pH of 8.74 in this study at day 0. As shown in Figure 12, the albumen pH of all treatment groups generally exhibited a fluctuating upward trend during storage, with the H_2O-CS/SiO_2@TiO_2 group showing the smallest increase, followed by the EtOH-CS/SiO_2@TiO_2 group. At day 7, all groups exhibited a notable increase in albumen pH, followed by a gradual and fluctuating rise. Specifically, on days 7 and 14, the H_2O-CS/SiO_2@TiO_2 group maintained lower albumen pH than the other treatment groups; on day 21, it was lower than the EtOH-CS/SiO_2@TiO_2 group; from days 28 to 42, differences among groups were minimal; by day 49, the H_2O-CS/SiO_2@TiO_2 and EtOH-CS/SiO_2@TiO_2 groups had notably lower pH than the blank and water-washed groups.

This change is primarily attributed to CO_2 produced by proteolytic enzymes and microbial activity during storage, which can escape through eggshell pores, resulting in increased albumen pH [48]. Conversely, CO_2 that does not escape can dissolve back into the albumen, creating a repeated dissolution and escape cycle, leading to pH fluctuations. These results indicate that the H_2O-CS/SiO_2@TiO_2 coating effectively mitigates the increase in albumen pH during storage.

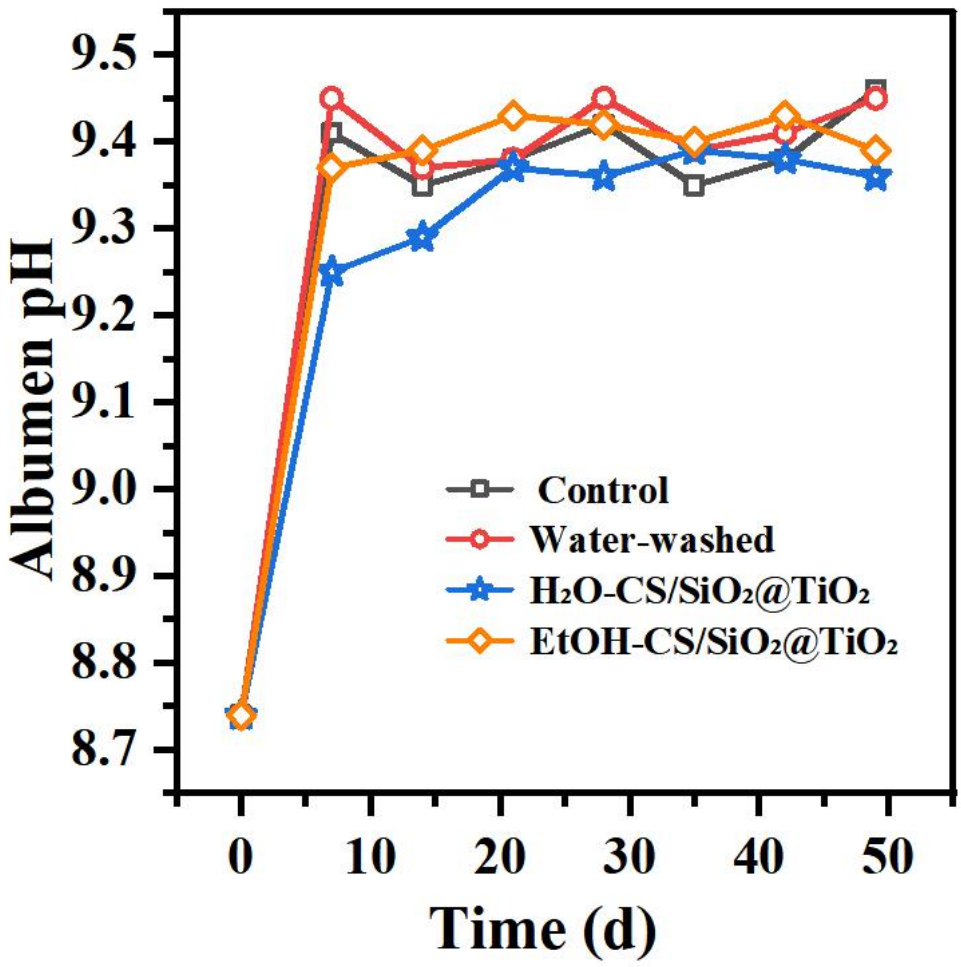

Figure 12. Shell microbial load dynamics across treatments over storage

4.2.5 Total Bacterial Count

(1) Total Bacterial Count on Eggshell Surface

As shown in Figure 13, the total bacterial count on the eggshell surface fluctuated greatly in the blank group and the water-washed group, whereas both the H_2O-CS/SiO_2@TiO_2 group and the EtOH-CS/SiO_2@TiO_2 group consistently remained within the range of 0–1000 CFU/egg. Specifically, at day 0, the total bacterial count on the eggshell surface of the blank group was 4812 CFU/egg, while the other three groups were all 0. At days 7, 28, and 35, the counts in the H_2O-CS/SiO_2@TiO_2 and EtOH-CS/SiO_2@TiO_2 groups all values lay below those recorded for the control and water-washed groups. At days 14, 21, and 42, the order of bacterial counts was H_2O-CS/SiO_2@TiO_2 group = EtOH-CS/SiO_2@TiO_2 group < water-washed group < blank group. By day 49, the counts in the H_2O-CS/SiO_2@TiO_2 and EtOH-CS/SiO_2@TiO_2 groups all values lay below those recorded for the control and water-washed groups. These results indicate that treatment with H_2O-CS/SiO_2@TiO_2 and EtOH-CS/SiO_2@TiO_2 can effectively inhibit bacterial growth on the eggshell surface, maintaining a relatively low bacterial count, thereby contributing to egg preservation.

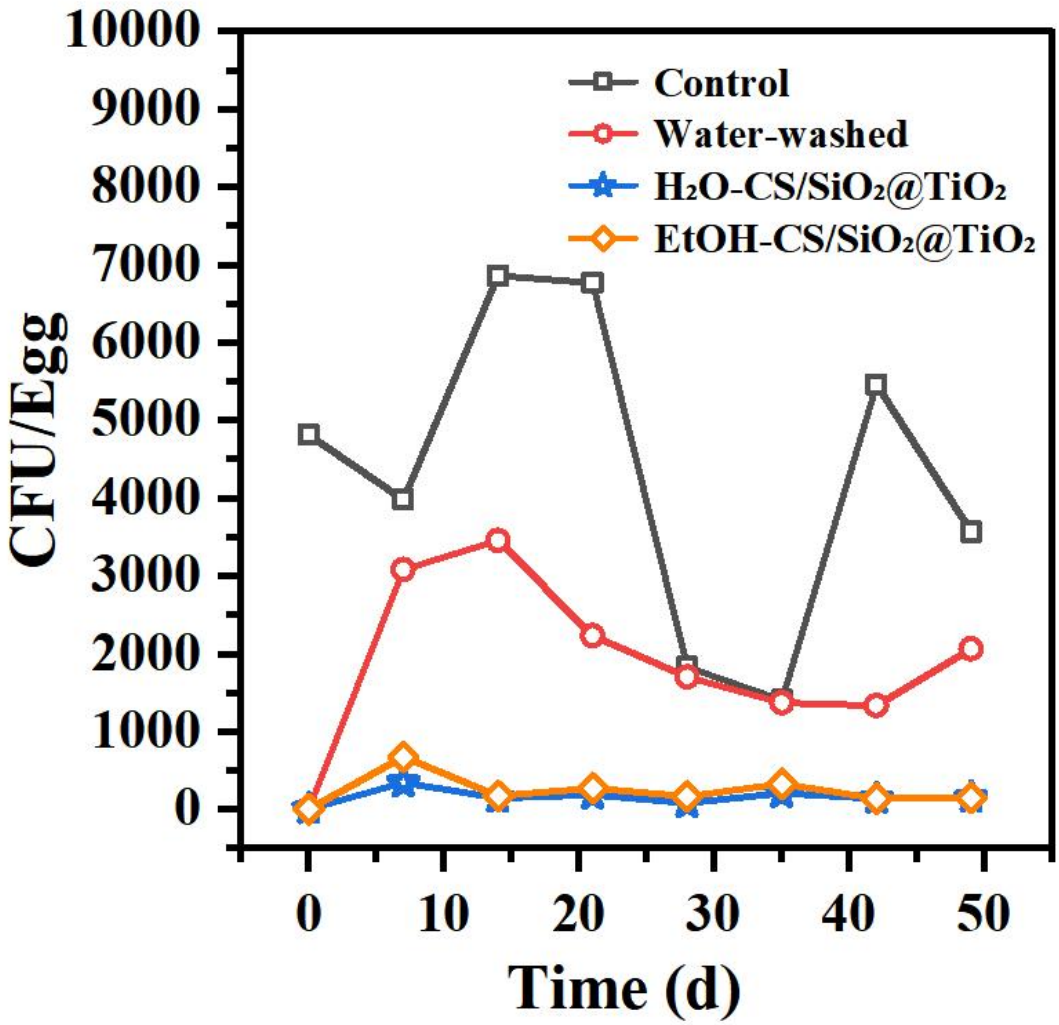

Figure 13. Changes in total microbial counts on eggshells under different treatments during storage

(2) Total Bacterial Count in Egg Contents

As shown in Figure 14, the total bacterial count in the contents of eggs from all treatment groups remained at 0 before 42 days of storage. By day 49, the H_2O-CS/SiO_2@TiO_2 group exhibited a lower bacterial count in the contents compared with the EtOH-CS/SiO_2@TiO_2 group and the blank group, while all three groups showed lower counts than the water-washed group.

Studies have shown that bacterial contamination of eggs is closely related to the laying hen breeding system, feeding practices, and processing and storage environments [49]. Eggs are easily contaminated by bacteria from feces when laid through the cloaca; during storage and distribution, they may also be exposed to environmental microorganisms, resulting in microbial growth on the eggshell surface [50]. Under normal circumstances, the interior of fresh eggs is essentially free of bacteria , mainly because the natural protective cuticle on unwashed eggs provides a certain barrier against microbial invasion. However, as eggs are exposed to the environment for longer periods and under varying temperatures, this protective effect gradually weakens.

Studies have shown that the total bacterial count on the eggshell surface is approximately 10^4 CFU, and its variations closely track the microbial burden in both feed and ambient air[51]. Under the same temperature conditions, washed eggs are more prone to spoilage than unwashed eggs [52]. In this experiment, the bacterial count on the eggshell surface did not continuously increase over time; it fluctuated around the thousands in the blank and water-washed groups, while in the H_2O-CS/SiO_2@TiO_2 and EtOH-CS/SiO_2@TiO_2 groups it remained within the hundreds. The bacterial count in the egg contents of all groups remained 0 for the first 42 days, with bacteria detected only at day 49. At this point, the H_2O-CS/SiO_2@TiO_2 group had lower bacterial counts than the EtOH-CS/SiO_2@TiO_2 and blank groups, and all three lay beneath the water-washed level. These results indicate that the coating treatment exerted an inhibitory effect on bacteria both on the eggshell surface and in the contents. Overall, the bacterial counts in this experiment were relatively low, which may be attributed to the high-quality feed and rearing conditions at the selected farm, and the improved lab conditions toward the end of the trial.

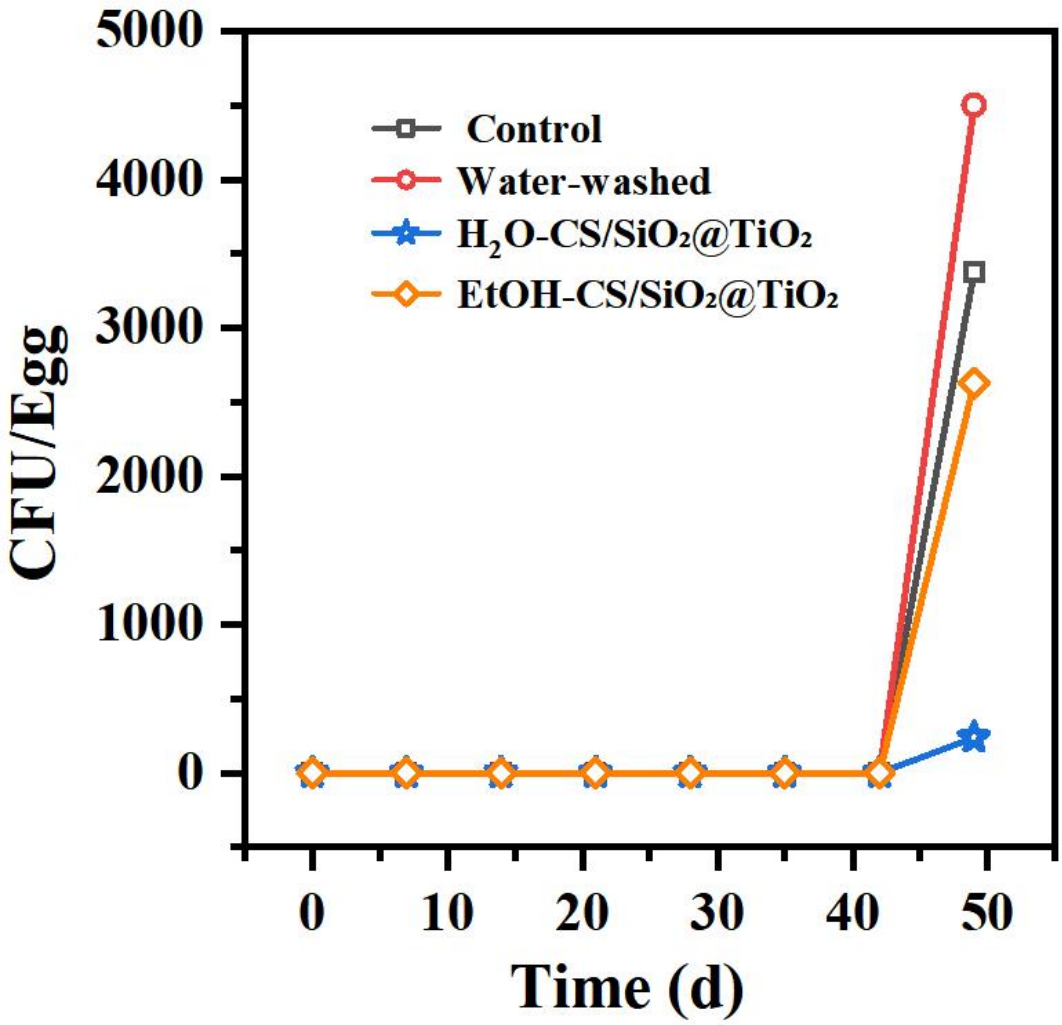

Figure 14. Microbial dynamics in egg contents across treatments throughout storage

5. Conclusions and Perspectives

5.1 Conclusions

In this work, we systematically constructed a superhydrophobic SiO_2@TiO_2/chitosan composite coating using pure water as the medium and verified its effectiveness in both room-temperature preservation and antimicrobial protection of eggs. The main conclusions are summarized below:

(1) Successful synthesis of SiO_2@TiO_2 functional material

Using the sol-gel process, a core-shell composite material with TiO_2 as the core and SiO_2 as the shell has been successfully prepared. This method is simple to operate, green and environmentally friendly, and does not require the introduction of metal ions or organic solvents throughout the process. The obtained material exhibited uniform particle size, structural stability, and controllable functionality, providing an efficient and stable functional core for subsequent coating applications.

(2) Simple coating preparation with excellent performance

By combining SiO_2@TiO_2 functional material with an aqueous chitosan solution, the coating could be directly applied to eggs without ethanol or other organic solvents as diluents. The formed coating exhibited a hydrophobic surface structure, effectively reducing moisture loss and serving as a microbial barrier, thus demonstrating promising preservation and antibacterial potential.

(3) Significant preservation and antibacterial effects

In storage experiments, the H_2O-CS/SiO_2@TiO_2 group showed superior performance: lower weight loss, smaller declines in Haugh unit and yolk index, slower changes in albumen pH, and consistently lower colony-forming units on both eggshells and contents. These findings indicate that the SiO_2@TiO_2 functional material synthesized in an aqueous system exerts remarkable preservation and antibacterial effects while avoiding the potential risks associated with metal ions and organic solvents.

5.2 Future Perspectives

Fine-tuning the Core-Shell Structure

Systematically optimizing the coupling parameters of the TiO2 core particle size and SiO2 shell thickness, combined with surface functionalization strategies (such as fluorosilane grafting and quaternary ammonium salt covalent modification), synergistically improves the composite's surface hydrophobic angle, antibacterial activity (inhibition zone diameter ≥25 mm), and DPPH radical scavenging rate (≥ 90%).

(2) Expanding Application Scenarios

Using egg preservation as a benchmark model, the coating's preservation efficacy on typical fresh produce, such as strawberries, Agaricus bisporus, and chilled pork, was further evaluated. A shelf life prediction model was constructed to provide data support for a diversified green food preservation system.

(3) Lifecycle Safety and Environmental Assessment

Gauge the water‑energy footprint and waste output of the aqueous route; use LC-MS/MS and ICP-MS to track the migration and degradation behavior of the coating under different storage conditions (25℃, 4℃, and exposure to light) to establish migration limit thresholds that comply with FDA/EU standards.

(4) Smart Packaging Integration

Coupling the SiO2@TiO2 functional layer with a pH-responsive anthocyanin film, a thermosensitive cholesteric liquid crystal, or an NFC wireless sensor chip enables real-time visual monitoring of food freshness (TVB-N, total bacterial count), boosting packaging data value.

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