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Improvement of heat-sealing strength of chitosan-based composite films and product costs analysis in the production process

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ABSTRACT

The primary objective of this study was to enhance the heat-sealing strength of composite films made from chitosan and analyze the associated product costs. The approach adopted involved formulating chitosan-based composite films by incorporating gelatin and green seaweed extract. This strategic combination resulted in a notable improvement in heat-sealing strength. The ensuing attributes underwent meticulous examination, encompassing seal strength, FTIR spectroscopy, FE-SEM surface morphology analysis, and DSC thermal properties determination. Data analysis was rigorously conducted using the SPSS program, with outcomes presented as mean values accompanied by standard deviations. Disparities were discerned at a 95% confidence level, ensuring statistical robustness. The findings unveiled that the incorporation of 10% gelatin and 1% green seaweed extract substantially enhanced the seal strength of the chitosan-based composite films. Notably, the introduction of green seaweed extracts disrupted interactions between chitosan's structure and various molecular vibrations. This disruption, coupled with increased ionic interactions and hydrogen bonding, led to improved molecular interdiffusion, ultimately resulting in modified heat sealability. The study identified the optimized conditions as 10% gelatin and 1% green seaweed extract concentrations, which produced the highest seal strength at 19.4 N/m. Further evidence from scanning electron microscopy demonstrated improved interfacial adhesion, attributed to the adjusted surface morphology. The film surface did not contain small scattered particles and presented a smooth phase. This suggests that the chitosan-based composite achieved good interfacial adhesion between the two components in these films. As an essential aspect for practical application, the total production cost of the films was determined to be 606.84 baht. This information renders the data collection from the study valuable for companies seeking to enhance production efficiency and overall profitability.

Keywords: Chitosan, Composite films, Product costs, Seal strength, Thermal analysis

INTRODUCTION

Polymeric biomaterials are one of the cornerstones of biomedical engineering and across various scientific disciplines. Many of these polymers, including chitosan, are employed as viable replacements for synthetic polymers when compared to other biomaterials such as polysaccharides, lipids, and proteins. They are used to produce edible films due to their easy biodegradability, abundance, and sustainability. Chitosan, known for its high potential in terms of non-toxicity, biodegradability, and biocompatibility, possesses excellent characteristics for film formation and stability. These qualities make chitosan an attractive material for food packaging [1]. Chitosan, primarily derived from the exoskeletons of crustaceans, is the second most prevalent polysaccharide in nature and contains inherent bioactive qualities, such as antibacterial and antioxidant activities. These attributes are crucial for developing active food packaging materials [2, 3]. On the other hand, despite chitosan's immense potential, its lack of heat sealability limits its application as a packaging material, requiring its combination with a thermoplastic polymer [4-7]. Creating a chitosan-based polymer by combining a suitable co-polymer with chitosan is an effective choice to improve thermal sealing properties. This process entails bonding two layers of films by pressing them between two heated plates for a specified duration. Several parameters,

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including thickness, composition, molecular structure of the surface and the film's evaporation temperature are crucial factors influencing heat sealability. The ultimate seal strength is also impacted by the seal treatment circumstances, such as jaw pressure and dwell duration, serving as indicators of seal quality [8]. Blending chitosan powder with a copolymer offers a simple and cost-effective method to enhance the heat-sealing capabilities of films produced by solvent casting. However, the impact of different chitosan and copolymer ratios on the heat sealability of blended films remains unknown.

Recently, one possible method for overcoming the limitation of heat sealability is to modify the surface chemistry and molecular structure of chitosan molecules. Prateepchanachai et al. [7], previously employed an approach using glycerol and gelatin as copolymers to modify the surface for adhesive bonding of chitosan-based films. This involved incorporating both glycerol and gelatin into the film-forming solution preparation process. A solution consisting of 25% (w/w) glycerol and 10% (w/v) gelatin was utilized to create a film with superior heat-sealing properties compared to a chitosan solution. This film demonstrated enhanced sealability attributed to increased hydrogen bonding between the chains, facilitating molecular interdiffusion, thereby improving melting and sealing behavior.

An intriguing avenue to achieve the aforementioned goal involves the utilization of phenolic chemicals derived from plant extracts to facilitate uniform cross-linking of gelatin into polymeric chains. This process aids in controlling the functional properties in composite films. During heat sealing, gelatin promotes molecular inter-diffusion, stabilizing hydrogen and covalent bonds within the base polymer and resulting in enhanced sealability of the films. Additionally, gelatin contributes to the formation of thermo-reversible gels, which, in turn, assist in the creation of sealed joints [9]. The green seaweed extract emerges as a promising renewable resource in the marine environment, garnering significant interest and attention in energy, food, and tissue engineering applications. This is attributed to the highly exploitable, easily accessible, and cost-effective source of polysaccharides it provides. In previous studies, Prateepchanachai et al. [8], utilized green seaweed extracts at concentrations of 1% (v/v) or higher to prepare chitosan-based films. Furthermore, studies did not report any improvement in the sealing qualities of the films when red seaweed extract and brown seaweed extract were added at concentrations ranging from 0 to 15% (v/v), in contrast to the results observed with the addition of green seaweed extract. As a result, the properties of a functional group in the gelatin molecule were enhanced through the addition of seaweed. This discovery suggests that green seaweed extract could serve as a novel cross-linker in gelatin molecules for film preparation, providing a viable means to achieve this objective. Despite some research into the combined impacts of gelatin and seaweed on the thermal properties of biopolymers, no studies have been conducted to manufacture and test chitosan-based composite films incorporating gelatin and green seaweed, along with examining the drying process of the resultant film-forming solutions. To date, no study has reported on the influence of modifying chitosan-based composites in the preparation of film solutions on surface microstructure properties. Additionally, there is a lack of research examining the adhesion of sealability, along with the calculation of product costs. This encompasses data collection on material costs, equipment costs, tooling costs, labor costs, energy costs, and overhead costs of the chitosan film production process. These represent the initial research studies on product cost calculation, a crucial metric for identifying and measuring the productivity and profitability of industries.

The objective of this research was to enhance the combined impact of gelatin and green seaweed extract in creating a composite chitosan-based film. The films underwent Fourier transform infrared spectroscopy (FTIR) analysis to examine their surface morphologies, which were further scrutinized by Field Emission Scanning Electron Microscopy (FE-SEM) for excellent resolution. Additionally, the thermal properties of the films were determined using Differential Scanning Calorimetry (DSC), elucidating the mechanisms contributing to their heat-sealing characteristics. The analysis of product costs was also conducted, providing valuable insights for manufacturers to determine the selling price.

MATERIALS AND METHODS

1. Materials

S.K. Profishery Co., Ltd. provided chitosan powder with a molecular weight of 900 kDa and a deacetylation degree of 90.2%. (Samut Sakhon, Thailand). Ruam Chemical 1986 Co., Ltd. provided analytical quality glacial acetic acid and green seaweed extract (Bangkok, Thailand). Top supermarkets sell porcine gelatin powder (Chachoengsao, Thailand). Tropicalife Co., Ltd. supplied the deionized water (Bangkok, Thailand)

2. Creating film-forming solutions

The techniques developed by Prateepchanachai et al. [8], were employed with appropriate modifications, to create a chitosan film-forming solution. In figure 1.

The first step involved dissolving 1.5% (w/v) chitosan powder in a 1% (v/v) acetic acid solution for 6 hours at room temperature, utilizing a magnetic stirrer machine (Framo Gerätetechnik, model M21/1, Eisenbach, Germany). The chemical reaction between chitosan and acetic acid in the aqueous solution, illustrating the film preparation process, is depicted in figure 2.

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Glycerol was then added to the solution at a concentration of 25% (w/w chitosan). Following that, a 10% (w/v) gelatin solution was prepared by dissolving 10 g of gelatin powder in 100 mL of deionized water, achieved by heating at 70 °C for 30 minutes. After preparing the chitosan-glycerol mixture, the gelatin solution was added at concentrations of either 0% or 10% (w/v) to obtain the chitosan-glycerol-gelatin film-forming solutions. Green seaweed extract was then added at either 0 or 1% (v/v), which was the result of preliminary experiments with adding green seaweed extract. Concentrations greater than 1% (v/v) produced films with poorer heat sealing than the target value of chitosan control film. After another hour of stirring at 500 rpm at room temperature, the solution was centrifuged at 19,414 g for 15 minutes to remove undissolved contaminants before hot air drying using a chilled centrifuge equipment.

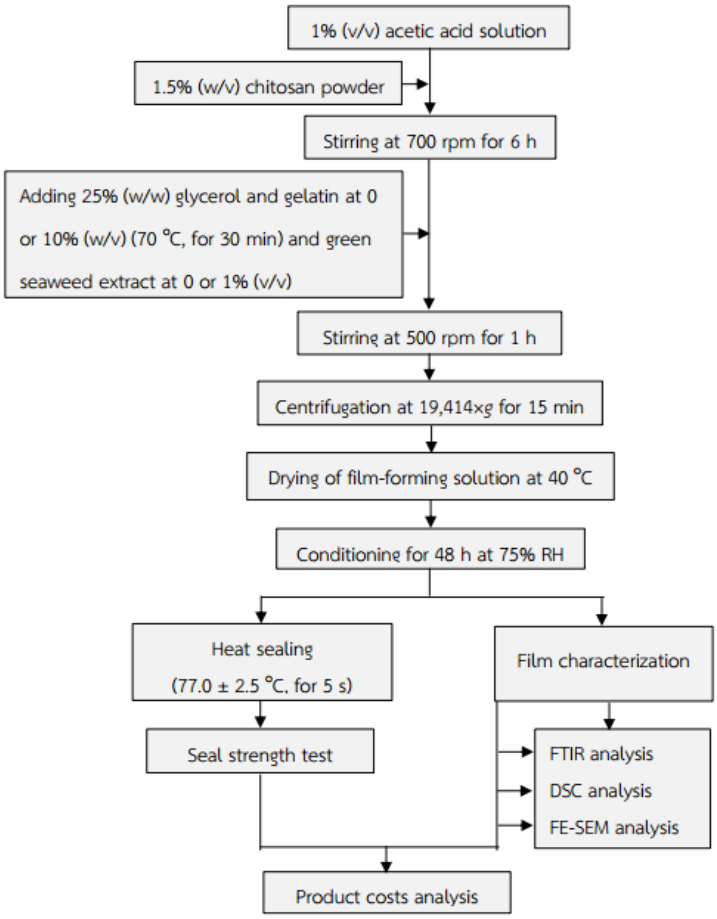

Figure 1 Designing overall experimental steps.

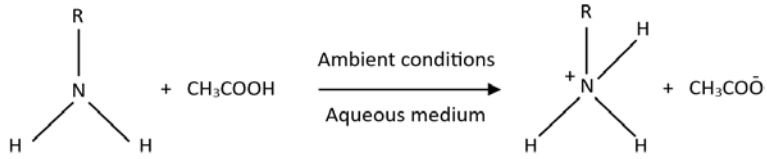

Figure 2 Illustrates the chemical reaction between chitosan and acetic acid in an aqueous solution, showcasing the film preparation process.

3. Preparation of chitosan films

To cast a film, each prepared film-forming solution (16 g) was applied onto an acrylic plate with dimensions of 13 × 10 cm. Drying was conducted at a hot air temperature of 40 °C and an air velocity of 0.25 m/s until the film reached a moisture content of around 14% (dry basis). Following this, the film was stored in a desiccator containing a saturated solution of sodium chloride, creating a relative humidity of 75%, for at least 48 hours before undergoing further characterization (This relative humidity level represents the average conditions in Thailand).

4. Characterization of chitosan film

4.1 Seal strength evaluation

As depicted in figure 3, the film sample was cut into a 10 × 2.5 cm strip, placed on top of another

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10 × 2.5 cm film strip, and heat-sealed with a 1 cm width at 77.0 ± 2.5 °C. This temperature represents the upper limit of the heat-sealing capability of the automatic sealing machine, which does not allow for adjustments to different temperatures. The sealing was performed for 5 minutes using an automatic heat sealer (AUTOMATIC EXTERNAL PACKING MACHINE, DZ-400T, Bangkok, Thailand). The heat-sealed film's sealing strength was assessed using an analysis comparable to a texture analyzer and the ASTM Standard Test Method F88 [9], with minor adjustments. The texture analyzer clamped each leg of the sealed film and suspended it perpendicular to the test direction. The seal strength in N/m is indicated as the force required for sealing damage, measured as the needed maximum forces:

$$\text{Seal strength} = \text{Peak force}/\text{Film width} \quad (1)$$

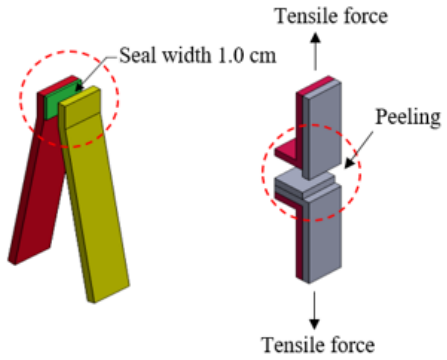

Figure 3 Simplified depiction of test specimen measurements and directions for peel test (a) film sample with bar sealing with 1.0 cm width (b) direction of pull is 90° to the seal in peeling test.

4.2 Determination of film FTIR Spectra

A film sample was spectroscopically examined using FTIR (ATR-FTIR) with modifications based on the methods of Prateepchanachai et al. [8], employing an FTIR spectrometer (Nicolet iS50, Thermo Scientific, Waltham, USA). The FTIR spectra were studied using a DTGS detector with a resolution of 4 cm^{-1} and a wavelength range of 4,000 to 400 cm^{-1}. The FTIR spectra had a wavelength accuracy of 0.1 cm^{-1} at 1,600 cm^{-1}.

4.3 Field emission scanning electron microscope (FE-SEM) analysis

The surface morphology of the microstructure films was assessed using a field emission scanning electron microscope (FE-SEM) (JEOL, JSM7800F model, Japan) in conjunction with the PCSEM software, employing an accelerating voltage of 15 kV. The samples were coated using a sputtering technique (QUORUM, Q150R ES model, UK) with a layer of gold (Au). The sputter conditions included a sputter current of 23 mA and a sputter time of 45 seconds.

4.4 Determination of thermal properties by differential scanning calorimetry (DSC)

Differential Scanning Calorimetry (DSC) (DSC 3+, Mettler Toledo model, Switzerland) was utilized to assess the thermal characteristics of composite chitosan films. Films (1 mg) were placed in an aluminum pan containing 20 µL of light. The nitrogen flux was maintained at 50 milliliters per minute. The analysis involved heating the films from 20 to 130 °C at a rate of 10 °C/min, followed by a 5-minute isotherm. Subsequently, the films were cooled from 130 to 5 °C at a rate of 20 °C/min, followed by another 5-minute isotherm in nitrogen. Finally, the glass transition temperatures were determined by heating the films to 300 °C at a rate of 10 °C/min (T_g) [8].

5. Statistical investigation

All data were analyzed using the SPSS® program (version 17; SPSS Inc., Chicago, IL) and are presented as mean values with standard deviations. To identify differences between mean values at a 95% confidence level, Duncan's novel multiple range tests were employed. Every experiment was conducted in triplicate.

RESULTS AND DISCUSSIONS

1. FTIR spectra

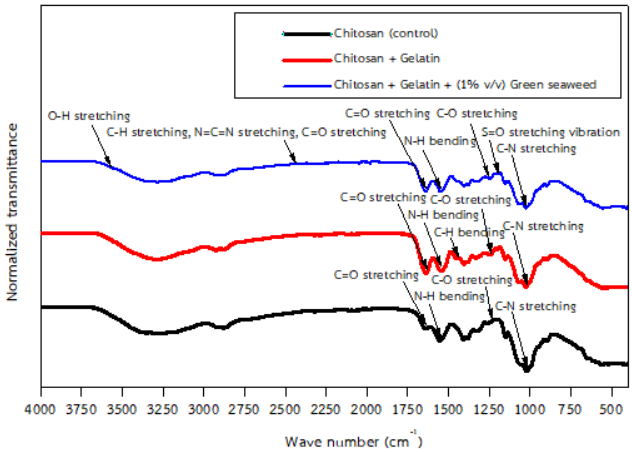

Figure 4 FTIR spectra of film samples prepared at various concentrations of 10% (w/v) gelatin and 1% (v/v) green seaweed.

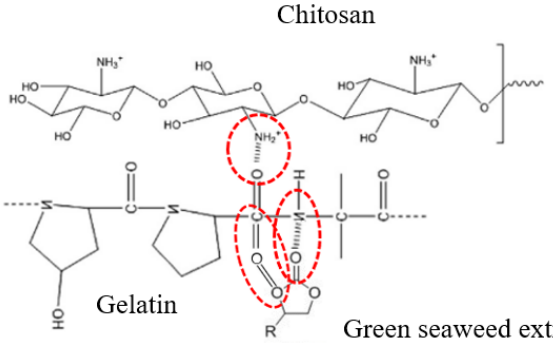

Figure 5 A tendency diagrammatic representation of intermolecular between chitosan, gelatin, and green seaweed molecule.

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FTIR spectroscopy was employed for the functional groups analysis of the chitosan-based composite film, as illustrated in figure 4. The addition of gelatin and green seaweed to the film resulted in increased peak intensities at 4,000-3,450 cm^{-1} (O-H stretching), indicating intermolecular interactions between amino acids of gelatin and phenolic compounds in green seaweed. Hydrogen bonds formed to engage with the chitosan structure. Additionally, higher peak absorptions were observed in the wavenumber regions of 3,130-1,700 cm^{-1}, associated with aldehyde (C-H stretching), carbodiimide (N=C=N stretching), and carboxylic acid (C=O stretching). These peaks indicated electrostatic interactions between gelatin peptide hydrogen bonds and green seaweed phenolic compounds, enhancing intermolecular interactions within the chitosan chains. This increased adhesion strength between the films, influencing the seal strength of the films. A schematic representation of the strong intermolecular interaction, mainly between chitosan, gelatin, and green seaweed extract composite film, is depicted in figure 5. Therefore, chitosan-based composite films were selected for this study due to their unique properties, versatility, and ability to interact with other components, aligning with the study's objectives of enhancing film properties and analyzing associated costs.

2. Heat seal strength

When sealing was conducted at a temperature significantly lower than the melting point of the sealant material, all samples failed in the peeling mode, as depicted in figure 3 (b). This outcome is anticipated since a decrease in the sealing strength of the composite film leads to the disentanglement of polymer molecules, resulting in the bond pulling apart.

Table 1 presents the maximum heat seal strength values for the film samples, calculated using equation 1. Films sealed at 77.0 ± 2.5 °C with a dwell length of 5 seconds exhibited peeling mode failure of the seal, indicating optimal seal strength. Sealing the control film proved challenging due to its rigid crystalline structure and the presence of strong intermolecular or intramolecular hydrogen bonding. The limited mobility of chitosan chains during thermal breakdown resulted in poor molecular penetration at the film surface and, consequently, poor sealability.

The addition of gelatin and green seaweed extract to the film-forming solution enhanced the film's seal strength. This enhancement is likely due to the ionic interaction and hydrogen bond between the -COOH groups of gelatin and the $-NH_2$ group of chitosan, leading to electrostatic interactions and intermolecular hydrogen bonds between opposite charges. This, in turn, enhanced molecular interdiffusion between chains. The addition of green seaweed extract resulted in the maximum seal strength at 19.4 ± 1.0 N/m. This could be attributed to the increased repulsion force between gelatin and green seaweed molecules, leading to increased partial denaturation and crystallization. As a result, they transformed into a more orderly structure, reflecting the lower rigid structure of the film network, and increasing thermal stability.

The addition of green seaweed at a concentration of 1% (v/v) resulted in the highest sealing strength compared to the control film. However, further increases in green seaweed concentration (above 5% (v/v)) did not significantly increase the seal strength. This value is lower than the heat-sealed chitosan reported by Prateepchanachai et al. [7] due to the use of an impulse heat-sealing machine with a very high heat-sealing temperature. Therefore, in future research, experimental conditions should be set as close as possible to the previous study.

Table 1 Heat seal strength and glass transition temperature of different films.

Film type	Heat seal strength (N/m)	Glass transition temperature (°C)
Chitosan (control)	$4.5±1.3^{A}$	$110.1±1.9^{C}$
Chitosan + Gelatin	$14.2±1.0^{B}$	$100.5±2.5^{B}$
Chitosan + Gelatin + 1% (v/v) Green seaweed extract	$19.4±1.0^{C}$	$94.8±1.4^{A}$

3. Thermal characteristics

The Differential Scanning Calorimetry (DSC) technique investigated the thermal transition temperatures affecting the sealing temperatures of polymers. Table 1 also presents the glass transition temperatures (T_g) of chitosan-based composite films with gelatin and green seaweed extract. The chitosan film (control) exhibited the highest T_g value of 110.1 ± 1.9 °C due to the N-H bending and O-H stretching, leading to the development of intramolecular hydrogen bonding and constrained mobility of the chitosan chains. This behavior was reflected in the difficulty of adhesion and heat seal strength. The addition of gelatin and green seaweed resulted in the lowest T_g value at 94.8 ± 1.4 °C, which is lower than that of the control film. The decrease in T_g value may be attributed to the plasticizing effect of the composite chitosan film with gelatin and green seaweed, increasing the free volume and intermolecular distance in the films due to the repulsion force between the chitosan chains. This led to a reduction in the transition temperature of the resulting film. Therefore, the composite chitosan film with gelatin and green seaweed exhibited different thermal transition characteristics compared to the pure chitosan film.

Several studies assessing biopolymer films have utilized SEM to establish correlations between film attributes and morphological structures, as demonstrated in the research by Souza et al. (2010) [10] and Castello et al. [11]. Figure 6 presents the results of FE-SEM images comparing the chitosan

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(control) film (figure 6a) with the combined addition of gelatin and 1% (v/v) green seaweed extract film (figure 6b). The FE-SEM images of the chitosan film exhibited a characteristic pattern on the film surface, appearing as a smooth phase with a nonporous, homogeneous, and continuous matrix displaying high structural integrity [12]. The surface was flat and compact, with extremely sparsely scattered tiny particles and no phase separation. The combined addition of gelatin with 1% (v/v) green seaweed extract significantly altered the chemical structure of the pure chitosan film, resulting in a more regular structure, which is likely reflected in the observed changes in the film's surface morphology. Notably, the film surface did not contain small scattered particles and presented a smooth phase. This suggests that the chitosan-based composite achieved good interfacial adhesion between the two components in these films.

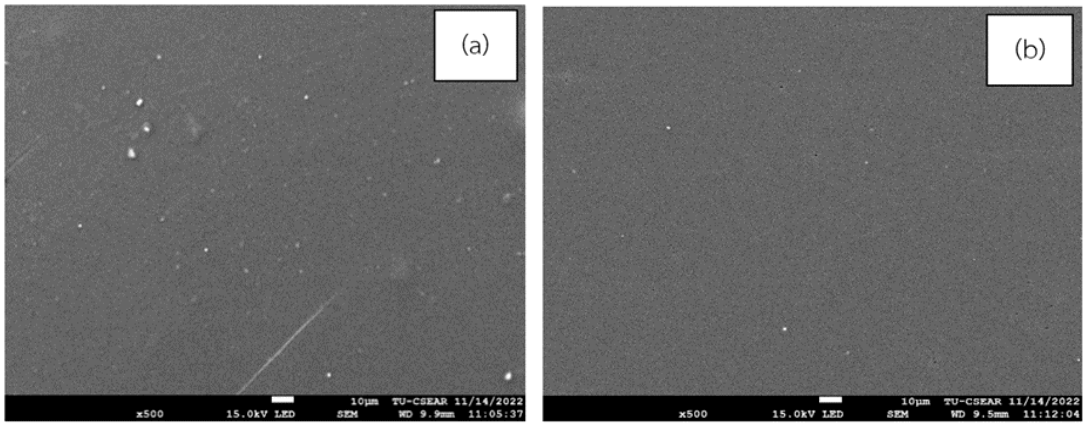

Figure 6 FE-SEM micrographs of difference films condition to (a) pure chitosan (regulate) film, and (b) combined addition of chitosan and gelatin with 1% (v/v) green seaweed extract film.

4. Product costs analysis

The product costs analysis pertains to the expenses incurred in creating packaging films intended for sale to customers. In industrial settings, the scale of production is akin to a system processing inputs to generate outputs, encompassing raw material costs, equipment costs, tooling costs, labor costs, energy costs, and overhead costs. To ascertain whether a firm is making a profit or incurring a loss, a thorough assessment of product costs or production expenses incurred throughout the manufacturing process is necessary, utilizing established accounting standards. The cost of goods becomes a primary reference point for determining the selling price of the final products [13].

Given that the film prepared from chitosan with gelatin and green seaweed extract exhibits superior heat-sealing properties compared to a pure chitosan solution, this research focuses on calculating costs under these conditions. The calculation of raw materials is defined by the amount of material utilized at the material's cost. Specifics regarding raw material usage during the production process in October 2022 are summarized in Table 2. The total raw material cost for using chitosan-based composite films in the production process is 15.40 baht/ml. Tooling costs amount to 80 baht/L. Direct labor costs involved in the production process are 400 baht/day. Total electricity costs are 92.09 baht/day. Total overhead costs cover all manufacturing expenses not included in direct materials and direct labor, calculated from actual costs incurred during a specific period, amounting to 19.35 baht/pcs. Equipment costs total 51,149 baht.

Table 2 Production cost of chitosan-based composite film.

Cost type	Quality (per unit)	Cost (baht/unit)
Raw material costs	Chitosan 1,800.00 baht/kg	0.027 baht/ml
	Green seaweed 2,369.00 baht /kg	2.37 baht/ml
	Glycerol 120.00 baht/kg	3.00 baht/ml
	Gelatin powder 930.00 baht/kg	1.00 baht/ ml
	acetic acid 89.00 baht/kg	9.00 baht/ml
	Total raw material costs	15.40 baht/ml
Tooling costs	Deionized water 80.00 baht/L	80.00 baht/L
Labor costs	Wage 0.83 baht/min	400.00 baht/day
Energy costs	Hot air oven 3,300.00 watts, 8 hrs.	89.69 baht/day
	Impulse heat sealer PS450 600 watts/hr.	2.40 baht/day
	Total energy costs	92.09 baht/day
Overhead costs	Grove 255.00 baht/100 pcs.	2.55 baht/pcs.
	Mask carbon 373.00 baht/50 pcs.	7.50 baht/pcs.
	Tissue paper 89.00 baht/6 pcs.	7.00 baht/pcs.
	Foil bag 115.00 baht/50 pcs.	2.30 baht/pcs.
	Total overhead costs	19.35 baht/pcs.
Total production cost (a)		606.84 baht
Number of films that produce (b)		7 pcs.
Production cost of films (13×10 cm) per package (a/b)		86.69 baht/pcs.
Equipment costs	Hot air oven 37,900.00 baht	
	Beaker glass 1,000 ml 1,430.00 baht	
	Spatula stainless 220.00 baht	
	Digital weighing 2,874.00 baht	
	Measuring pipette 340.00 baht	
	Pipette rubber 75.00 baht	
	Plate casting 10.00 baht/pcs.	
	Impulse heat sealer PS450 8,000.00 baht	
	Maintenance 300.00 baht/year	
	Total equipment costs	51,149.00 baht

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Table 2 further illustrates that the total production cost during October 2022 is 606.84 baht. This indicates that the film, while demonstrating excellent heat-sealing properties, tends to be relatively expensive for packaging polymer use. Assuming the manufacturing is completed in one day, disregarding the presence of commodities in the process, and considering that 7 pcs of chitosan-based composite films can be produced, the production cost per film (with dimensions of 13 × 10 cm, suitable for packaging or wrapping) is calculated by dividing the total production cost by the number of films produced, resulting in 86.69 baht/pcs. Based on these findings, companies and researchers can utilize the data collection of total production cost components as a foundation for determining the cost of products sold and assessing profitabily.

CONCLUSION

The combined addition of 10% (w/v) gelatin and 1% (v/v) green seaweed extract significantly improved the seal strength of composite chitosan-based films. This enhancement was accompanied by a detailed product cost analysis. The introduction of green seaweed extract is believed to disrupt interactions within the chitosan structure. Consequently, the addition of green seaweed extract enhances the film's seal strength by increasing ionic interactions and hydrogen bonding between the -COOH groups of gelatin and the -NH_2 group of chitosan. This reaction leads to electrostatic interactions and intermolecular hydrogen bonds between opposite charges, ultimately improving molecular interdiffusion between the chains and modifying heat sealability. This improvement is further evidenced by a significant reduction in the glass transition temperature, lowered by 94.8 ± 1.4 °C compared to films made from pure chitosan. Optimal conditions were identified at a concentration of 10% (w/v) gelatin and 1% (v/v) green seaweed extract, resulting in the highest seal strength of 19.4 N/m. Surface inspection using FE-SEM revealed changes in the smooth surface morphology, indicating reasonably high interfacial adhesion between the two components. While the total production cost of the films amounted to 606.84 baht, this dataset offers valuable insights for companies engaged in the production of goods, potentially influencing overall profitability.

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