

Formulation of Film from a Quaternized Starch, Polyvinyl Alcohol and Tetraethylorthosilicate for Food Packaging



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ABSTRACT: This study aimed to formulate a film using quaternized starch (Qs), polyvinyl alcohol (PVA), and with tetraethylorthosilicate (TEOS) for potential food packaging. Corn starch as a renewable resource could ensure sustainability and less negative environmental impact when compared to the use of petrochemical-based materials. The starch was quaternized using hexadecyltrimethylammonium bromide (CTAB) to improve its solubility and solve the retrogradation problem. and the loadings of TEOS were varied at 0-5 % wt./wt. polymer. The formulated films were characterized in terms of functional group, mechanical properties, and water uptake. The FTIR analysis revealed that the quaternization process successfully modified the starch, and the films containing TEOS exhibited new vibrational modes associated with the asymmetric and symmetric Si-O-Si stretching, confirming the effective incorporation of the silica network. Film F2 with 1 wt.% TEOS exhibited good mechanical properties with moderate tensile strength (0.870 MPa), high percentage elongation (301%), and low rigidity represented through Young's modulus (0.814 MPa). The water uptake analysis revealed that increasing the TEOS content significantly improved the water resistance of the hybrid films (10 - 68%) compared to the films without TEOS (99%). The findings of this study contribute to the development of sustainable and eco-friendly packaging materials.

KEYWORDS: Film, Quaternized Starch, Tetraethylorthosilicate, Water Uptake, Mechanical Properties, Food Packaging

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I. INTRODUCTION

Food packaging has a significant role in the quality, safety, and added value of food products. Food packaging materials with good mechanical strength, barrier properties, thermal stability, biodegradability, antioxidant, and antibacterial properties are necessary to extend the lifespan of packaged goods and ensure the safety of food as stated by Wu *et al.* (2017). Up to 2021, the global plastics output was 390 million tons; 40% came from food packaging and less than 10% was recycled (Piñeros-Guerrero *et al.*, 2024; Waring *et al.*, 2018). The food is assured to arrive at clients in perfect condition since it works as a barrier against physical, chemical, and biological hazards. Plastics have been the most often used materials for such applications due to their low cost of manufacture, greater versatility, good barrier capabilities to water and gas permeability, and mechanical and optical features (García-Guzmán *et al.*, 2022). In recent years, there has been an increasing demand for sustainable and eco-friendly packaging materials in the food industry due to their non-biodegradable nature and potential health risks associated with chemical leaching particularly the single-use products derived from petroleum. Nonetheless, several concerns with current food packaging such as the significant negative environmental impact,

including harm to ecosystems and marine life must be addressed (Alamri *et al.*, 2021).

The situation has led to the development of natural biopolymers like cellulose, chitosan, starch, soybean protein isolate, alginate, and pectin for food packaging materials. These biopolymers offer benefits like nontoxicity, biodegradability, global availability, and biocompatibility (Hu and Wang, 2016). Research has focused on developing these polymers for packaging applications to reduce plastic waste. One advantage is their ability to break down in natural environments, creating carbon dioxide, water, and biomass without hazardous byproducts (Samir *et al.*, 2022; Moshood *et al.*, 2022). Certain produced polymers are biodegradable and compostable, like naturally occurring carbon-based polymers such as starch, cellulose, and lignin (Rosenboom *et al.* 2022; Siracusa *et al.* 2008).

Besides using pure starch, a quaternization process was employed to modify the starch, enhancing its compatibility and functionality within the film matrix. During the quaternization process, quaternary ammonium groups are added to the modified structure where because of quaternization, it frequently dissolves with less viscosity than pure starch, making it simpler to incorporate into a variety of compositions without adversely impacting the texture (Compart *et al.*, 2023). Furthermore, the quaternization process could minimize the retrogradation process of the pure

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starch. Retrogradation is a typical problem where gelatinized starch returns to a more crystalline form upon chilling. Besides the importance of compatibility, other factors to be considered as a good packaging material are mechanical strength, transparency, and barrier properties (Marsh and Bugusu 2007; Fan and Picchioni, 2020). The mechanical properties of the film could be enhanced with the incorporation of nanomaterials and active chemicals (Chawla *et al.* 2021; Gupta *et al.* 2022). Research on PVA/graphene oxide/starch/silver nanocomposite films has demonstrated that the mechanical, thermal, and antibacterial characteristics of starch/PVA films may be further improved by the addition of Tetraethylorthosilicate (TEOS) as the nano precursor (Elgharbawy *et al.*, 2024).

Therefore, the goal of this study is to formulate a film from a blend of quaternized starch (QSs), polyvinyl alcohol (PVA), and tetraethylorthosilicate (TEOS). The formulated film is expected to effectively preserve food quality by enhancing the properties of starch through quaternization and further crosslinked with TEOS. Subsequently, the study investigated the impact of varying concentrations of TEOS on the film's characteristics, specifically examining the functional group, mechanical strength, and water absorption.

III. METHODOLOGY

A. Materials

Corn Starch (*Zea Mays*) was purchased from BT Science Sdn. Bhd. Polyvinyl Alcohol (PVA) with a hydrolysis degree of 87% - 89% (MW: 85000 - 124000) and Tetraethyl orthosilicate (TEOS) with 99% purity were purchased from Sigma-Aldrich (M) Sdn. Bhd, Sodium Hydroxide pellets (NaOH) were purchased from R & M Chemicals, Subang. BT Science Sdn. Bhd. has supplied Hexadecyltrimethylammonium bromide (CTAB), Hydrochloric acid (HCl) with 37% purity as a catalyst, and Ethanol.

B. Methods

1) Preparation of Quaternized Starch (QSs)

3.0 g of corn starch was dispersed and heated in 30 mL of distilled water at 60°C for 1 hour with fixed stirring at 400 rpm. 60 g CTAB was added into the above solution, followed by 120 g sodium hydroxide (NaOH), and the mixture was stirred at 400 rpm with a temperature of 60°C for 5 hours. It was then washed with 150 mL to precipitate out the quaternized starch. The solution was then filtered using filter paper and the resultant white quaternized starch precipitates (QSs) were collected (Pal *et al.*, 2005).

2) Preparation of Polyvinyl Alcohol (PVA)

To prepare the PVA solution, 4 g PVA powder was dissolved in 96 mL of distilled water at a heating temperature of 95 °C for 2 hours under continuous stirring at 400 rpm. Then, it was let to cool down to room temperature (Kassim Shaari *et al.*, 2019).

3) Preparation of Hybrid Solutions (QSs, PVA and TEOS)

2.4 g QSs were dissolved in 40 mL water at 25 °C under vigorous stirring. A weight ratio of 2:3 QSs solution to PVA solution was used for the film formulation. Therefore, 20 g QSs solution was added to 30 g PVA solution, followed by the addition of TEOS at 1% wt./wt. the polymer in the mixture. 1 mL of hydrochloric acid was added to the mixture to initiate the sol-gel process. The mixture was then heated to 80 °C and stirred at 400 rpm for 6 hours. The same procedure was repeated for different concentrations of TEOS: 0% wt./wt. polymer, 0.5% wt./wt. polymer, 3% wt./wt. Polymer, and 5% wt./wt. polymer. Table 1 shows the summary of the film formulation (Lani and Ngadi, 2014). Afterward, each solution was cast onto a glass plate by using a Baker's Film Applicator and dried at room temperature.

Table 1 The formulation of hybrid film.

Film	Quaternized Starch (g)	PVA (g)	TEOS	
			% wt./wt. polymer	g
F0	20	30	0	0
F1	20	30	0.5	0.25
F2	20	30	1	0.5
F3	20	30	3	1.5
F4	20	30	5	2.5

C. Characterization of the Film

1) Fourier Transform Infrared (FTIR) Analysis

Fourier-transform infrared spectroscopy (FTIR) was employed in this study to determine the chemical composition and structure of the formulated films. The information on the molecular functional groups in the film can be known. The Fourier Transform Infrared Spectroscopy (FTIR) Perkin-Elmer Spectrum 2000 was used and the analysis was done at a wavelength between 400 cm⁻¹ and 4000 cm⁻¹ (Birkner and Wang, 2013).

2) Tensile Strength Analysis

The mechanical properties of each film were measured using an Instron Universal Testing Machine (Model MDX, Instron Engineering Corporation, Canton, MA, USA) equipped with a 2.5 kN load cell according to the standard method ASTM D 882-88. Each film was cut into a 3 cm x 8 cm rectangular piece. The machine was set to a tensile mode, with a 50 mm starting grasp separation and a crosshead speed of 50 mm/min. The tensile strength (TS) was calculated in MPa by dividing the highest load by the initial cross-sectional area (m²) of the films. The Strain, *E* (%) was determined by dividing the total extension of the films (at rupture) by the initial length of the film multiplied by 100, and Young's Modulus was obtained from the slope of the initial straight-lined portion of the graph.

3) Water Uptake Analysis

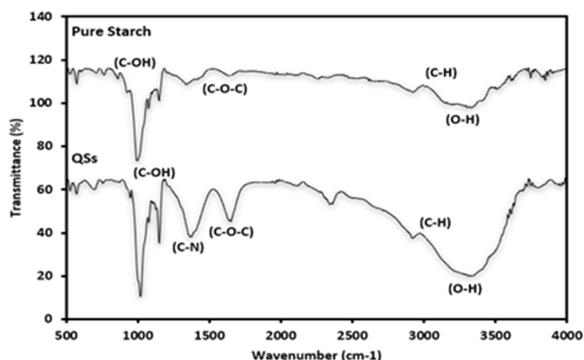
In the analysis of water uptake, the films were cut into a rectangular shape with area of 1 cm² and immersed in ultra-pure water. After 24 hours, the wet films were removed and weighed immediately (m_{wet}). Next, the films were oven-dried at 50°C for 24 hours. The weight of the film (m_{dry}) was then recorded. The water uptake was calculated based on the average obtained from 5 samples of each film. The % water uptake was calculated using Equation 1.

$$\text{Water uptake} = \frac{m_{wet} - m_{dry}}{m_{wet}} \times 100 \quad (1)$$

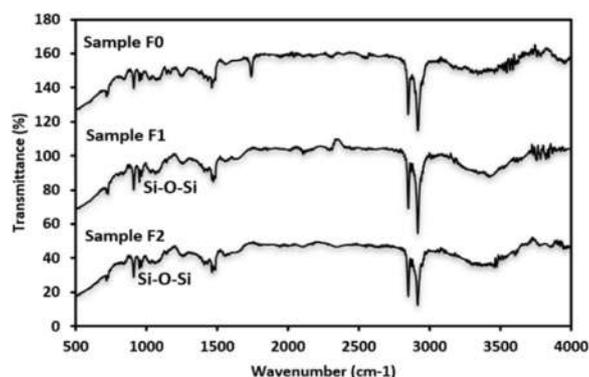
IV. RESULTS AND DISCUSSION

A. FTIR Analysis

Figure 1(a) displays the FTIR spectra of pure starch and quaternized starch (QSs) respectively. The FTIR spectra showed similar peaks at 3200-3300 cm⁻¹ corresponding to the O-H groups of the starch precursor, 3000-2900 cm⁻¹ corresponding to C-H stretching vibrations, and 1700-1500 cm⁻¹ from C-O-C stretching vibrations. Following the quaternization process, the band at 995.76 cm⁻¹ for pure starch, which is a peak for C-OH bonds, moved somewhat higher to 1020.79 cm⁻¹ which represents the C-N bond. The emergence of new bonds due to the quaternization process was similar to that obtained by Wang *et al.* (2022). Figure 1(b) shows the FTIR spectra of hybrid films with different TEOS concentrations. In comparison to film F0, the spectra of the films containing the inorganic precursor (Fig. 1b) show an extension of the bands between 1180 and 850 cm⁻¹, especially in film F2. This implies the existence of novel vibrational modes, asymmetric and symmetric Si-O-Si stretching modes of silica, resulting from the hydrolysis and condensation of TEOS where research by Gupta *et al.* (2022) found similar findings. More particularly, 1100-1000 and 950-900 cm⁻¹ were attributed to $\nu(\text{Si-O-Si})$ and $\nu(\text{Si-OH})$.



(a)



(b)

Figure 1 FTIR spectra (a) Pure Starch and Quaternized Starch (QSs) (b) Hybrid Film with Different TEOS Concentrations.

B. Tensile Strength Analysis

The results from mechanical testing on all films are shown in Table 2. Based on the table, for the F0 film, which does not contain TEOS, the tensile strength approaches 1.69 MPa with a strain of 124% before it breaks at 0.685 MPa and 143% strain. For film F1, even though it has the highest tensile strength, it breaks much faster than F2 thus showing film F1 is more rigid and less flexible than F2. Film F2 has a lower tensile strength at 0.870 MPa than F0 and F1 but has the longest elongation at 301% break strain. For films F3 and F4, both films have low tensile strength and break at considerably lower strains. It shows that both films are brittle and less flexible. This situation is due to the excessive inclusion of silica that resulted in the agglomeration of silica. This situation shows that the correct distribution and interaction of inorganic particles with organic components such as quaternized starch and PVA polymer is beneficial to improving mechanical characteristics. Film F2 performs extremely well in comparison to the other samples, demonstrating that the crosslinking process particularly the hydrolysis and condensation process allows the silica sol to react with the hydroxyl group from quaternized starch and polyvinyl alcohol to provide adequate interfacial interaction in the matrix of the polymer film (Jansson and Thuvander, 2004; Priyanka *et al.*, 2024). Meanwhile, the large aggregation of nanoparticles in the polymer matrix in the formulation of films F3 and F4 has reduced the tensile strength and flexibility of the films as reported by Lin *et al.* (2023).

Table 2 Mechanical Properties of Hybrid Film with Different TEOS Content (%).

Film	Ultimate Stress (MPa)	Ultimate Strain (%)	Break Stress (MPa)	Break Strain (%)	Modulus (MPa)	Break Energy (MJ/m ³)
F0	1.69	124	0.685	143	1.86	895000
F1	2.78	192	2.11	209	4.47	3910000
F2	0.870	290	0.864	301	0.814	1560000
F3	0.185	33.7	0.01	73.8	0.935	63500
F4	0.148	25	0.02	74.8	0.1	39100

Young's modulus, also known as the modulus of elasticity, is a measure of a material's stiffness and its ability to resist deformation under stress. In the context of the QSs/PVA/TEOS hybrid films, Young's modulus provides insights into how the addition of TEOS affects the stiffness and flexibility of the films. As shown in Table 2, film F2 has Young's modulus of just 0.814 MPa due to its ease of deformation during elongation. This can be attributed to the optimal distribution and interaction of silica particles with the quaternized starch and PVA polymer in film F2. However, Young's modulus for films F0 and F2 is higher than F2 showing stiffer films. The large silica nanoparticles in the polymer matrix of films F3 and F4 do not contribute to an increase in tensile strength but rather lead to a decrease in Young's modulus (Pingan *et al.*, 2017). As the TEOS content increases, the silica network becomes more dominant, leading to a reduction in the overall flexibility and extensibility of the film. This results in a decrease in the film's rigidity. The silica network formed by the TEOS provides additional reinforcement and interactions with the polymer chains, leading to an increase in the film's tensile strength. However, as the TEOS content continues to increase, the film may become more brittle and prone to fracture, leading to a reduction in the overall tensile strength. High silica concentrations can cause silica aggregation in the film matrix, which serves as a stress concentration point and compromises the structural integrity of the film matrix network. This reduces cross-linking between matrix components and causes a collapse in mechanical properties (Zhang *et al.*, 2023).

C. Water Uptake Analysis

Quaternized starch (QSs) and polyvinyl alcohol (PVA) are hydrophilic polymers because their backbones include several -OH groups. As a result, films made with these materials are susceptible to moisture and humidity which is undesirable for the application as food packaging material (Arayaphan *et al.*, 2020). This shortcoming restricts the usefulness of these films as it creates a challenge during the film processing, and handling as it is easily disintegrated (Bumbudsanpharoke *et al.*, 2024; Deng *et al.*, 2024). Figures 3, 4, 5, 6, and 7 show pictures of the films after they were soaked in ultra-pure water for 24 hours. The F0 and F1 films exhibit signs of breakdown, however, the F2, F3, and particularly F4 films maintained their structural integrity. Table 3 summarizes the percentage of water uptake for each film. Film F4 exhibited the lowest water uptake of around 10% after 24 hours of

immersion, compared to film F3 which had a water uptake of around 40%.

The film F0 without the TEOS had the highest water uptake of around 99%, which was attributed to the high water solubility of PVA and the QSs. The formation of a more crosslinked and compact structure of the films with the increment in TEOS content has reduced the water uptake. Similar findings were reported by Zhang *et al.* (2023) where the addition of 9% (w/w) of silica to the sodium alginate/PVA films resulted in a water solubility reduction to 80% from 100%. The tetraethylorthosilicate (TEOS) as a silica nanoprecursor, acts as a crosslinking agent, reacting with the hydroxyl groups of the quaternized starch and PVA to form a siloxane network. This siloxane network possesses strong hydrogen bonds that restrict the movement of the polymer chain, and the reduction of available hydroxyl groups has led to low swelling and penetration of water molecules into the film structure. The higher the TEOS content, the more extensive the crosslinking, resulting in a more hydrophobic and less water-permeable hybrid film (Deng *et al.*, 2024).

Table 3 Water Uptake Analysis on Hybrid Film.

Film	Weight of the films		Water Uptake (%)
	m_{wet} (g)	m_{dry} (g)	
F0	0.151	0.001	99.3
F1	0.032	0.006	81.3
F2	0.022	0.007	68.1
F3	0.027	0.016	40.7
F4	0.030	0.027	10

**Figure 3 Film F0 with 0% TEOS.**



Figure 4 Film F1 with 0.5% TEOS.



Figure 5 Film F2 with 1% TEOS.



Figure 6 Film F3 with 3% TEOS.



Figure 7 Film F4 with 5% TEOS.

V. CONCLUSION

Based on the results, a successful quaternization of corn starch has been carried out using CTAB as a quaternizing agent that was proven through the FTIR spectra where the change in the distinctive C-OH band confirmed the effective quaternization. Results also show the effective inclusion of the inorganic TEOS component into the hybrid film structure, where the cross-linking of the film with TEOS improved its mechanical characteristics and hydrophilicity. Film F2 with TEOS 1% wt./wt. polymer was the best formulation in terms of moderate tensile strength, high percentage elongation, and low rigidity represented through Young's modulus. Films with TEOS have better water resistance reflecting its good integral stability. These findings contribute to the development of sustainable and eco-friendly packaging materials that can address the growing concerns about the environmental impact of conventional plastic-based packaging. More research and optimization on the formulation will be conducted in the future.

AUTHOR CONTRIBUTIONS

N. Z. Kassim Shaari: Supervision, Writing – review & editing. **N. I. Roslan:** Methodology, Validation, Writing – original draft.

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