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Membrane from a blend of quaternised starch/polyvinyl alcohol/polysulfone/TEOS: Formulation and characterisation

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ABSTRACT

This study reported on the formulation and characterisation of hybrid membranes from a blend of quaternised starch (QSs), poly (vinyl alcohol) (PVA), polysulfide (PSF), and crosslinked with tetraethyl orthosilicate (TEOS). Starch quaternisation was conducted to overcome the brittleness and enhance the properties of the starch. TEOS was used to enhance the membrane's structural integrity and mechanical stability. In this study, the effect of varying the loadings of the quaternising agent (hexadecyltrimethylammonium bromide (CTAB)) at 20, 30, and 40 g, together with varying the TEOS concentrations at 1, 3, and 5 wt%, respectively was evaluated for the functional groups, mechanical properties, and water uptake of the membranes. Results show that a successful development of silica networks and strong connections between the polymer matrix and TEOS-derived silica through Si–O–Si and Si–O–C linkages were demonstrated through FTIR. A membrane containing 40 g CTAB with 1 wt% TEOS (M7) was found to be the best formulation, possessing high tensile strength (35 MPa) and good elongation, with a moderate elastic modulus (664 MPa) and the lowest water uptake. This membrane has the potential to be used as a membrane material that requires ductile behaviour with good structural stability.

1. INTRODUCTION

The influence of environmental challenges on public health has become well known. One essential resource for social and economic advancement is water. Water reuse is a way to replace industrial water supplies, which have been critically evaluated due to the growing global demand for water, an exhaustible resource. Water treatment is necessary to improve the quality of the environment and human life and to solve water shortage issues. Membrane technology has taken the lead in wastewater reuse in recent years, and this trend appears to be continuing in the future as it aims to achieve sustainability standards for environmental impacts, usability, flexibility, and adaptability. The use of environmentally friendly materials such as starch for membrane materials is one of the approaches for sustainability measures (Aghazadeh et al., 2018).

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Furthermore, starch as a biomass is an abundant and renewable resource. However, pure starch often lacks the mechanical strength and selectivity that limit its application as a membrane material. Furthermore, pure starch is too brittle to be used as a membrane material. Starch modification can be accomplished by introducing additional functional groups into the starch structure through a variety of chemical changes such as oxidation, phosphorylation, esterification, ozonation, acetylation, carboxymethylation, and quaternisation (Wang et al., 2020). A modification process on the starch through quaternisation could improve its properties and to ensure it can be widely used (Su et al., 2020). With the primary goal of giving the starch biopolymer antibacterial qualities and reducing the brittle structure, the quaternisation of starch using quaternary ammonium salts (QAS) has become a fascinating and potential substitute in this regard. Using sodium hydroxide as a catalyst, the quaternary ammonium reagent performs nucleophilic substitutions on the biopolymer's hydroxyl groups in this chemical process (Cao et al., 2017; Ribeiro de Carvalho et al., 2024).

The incorporation of nanoparticles such as silica can disrupt the matrix's crystalline and molecular network to alter the polymer chain's motion. A good distribution of silica and its interactions with the polymer matrix can improve the mechanical properties of membranes. This situation can be described through the filling of the inorganic nanoparticles into the surface pores, that make the membrane film more difficult to break (H. Wang et al., 2022). Furthermore, the silica acts as a crosslinker, where the increase in crosslinking density in the membrane structure helps to decrease the swelling of the membrane (Sarmah & Karak, 2020). Therefore, in this proposed study, the incorporation of TEOS into the polymer matrix is conducted through a sol-gel method. The sol-gel reaction involves the hydrolysis of nano precursor that is silica and condensation of the resulting hydroxyl groups to form a nanostructure. The process is normally carried out under mild conditions that are at low temperature and atmospheric pressure. In the organic‐inorganic hybrid, nanometre‐ordered inorganic minerals precipitate in situ regularly in the organic polymer matrix to form a covalent bond between the organic phase and the inorganic phase, where this strong interaction may result in a hybrid with markedly improved mechanical properties. The use of TEOS as a precursor has become widely used as it could provide high porosity and large surface area to the systems (Sarmah & Karak, 2020). Another advantage of the sol-gel method is that the concentrations of organic and inorganic components are easy to control in the solution, and both components are dispersed at the molecular level in the membranes so that no phase separation occurs and the resultant membranes are homogeneous (Xu et al., 2009).

Creating a membrane that combines improved functional performance, and biodegradability is the toughest part. To overcome these issues, blending starch as a renewable resource with polysulfone to provide integral stability is a good approach (Chen et al., 2016; Fahmy et al., 2023). Few studies have evaluated the crosslinking process of the quaternised starch-based membranes. The performance of such a hybrid membrane in terms of long‐term stability, antifouling qualities, and structural integrity remains unsolved. There is a knowledge gap in the creation of optimal, blended membranes employing crosslinkers such as TEOS and renewable resources such as starch.

This study evaluates the functional groups, the mechanical properties, and water uptake of the membrane from a blend of quaternised starch/polyvinyl alcohol/polysulfone and crosslinked with TEOS by varying the loadings of the CTAB as the quaternising agent and TEOS concentrations, respectively.

2. MATERIALS AND METHOD

2.1 Materials

In this experiment, potato starch was purchased from Progressive Scientific Sdn. Bhd., Polyvinyl Alcohol (PVA) with a hydrolysis degree of 87–89 wt% (MW: 85000–240000), tetraethyl orthosilicate (TEOS) with 99 wt% purity, and *N*-methyl-2-pyrrolidone (NMP) were purchased from Sigma-Aldrich (M) Sdn. Bhd. Chitosan with a deacetylation degree of 84.4 wt% was acquired from Aman Semesta Enterprises in Shah Alam, Malaysia. Sodium hydroxide pellets (NaOH) and dimethyl sulfoxide (DMSO) were purchased from R&M Chemicals, Subang. BT Science Sdn. Bhd. supplied hexadecyltrimethylammonium bromide (CTAB) and hydrochloric acid (HCl) with 37 wt% purity as a catalyst and ethanol.

2.2 Preparation of quaternised starch

In the first step, 30 mL of distilled water and 3 g of potato starch were mixed and heated to 60 °C for 1 h while being stirred continuously at 400 rpm. Next, 20 g of 1 M sodium hydroxide (NaOH) solution and 20 g of hexadecyltrimethylammonium bromide (CTAB) were added to the mixture. After that, the solution was stirred for 5 h at 400 rpm. The solution was then cooled to room temperature. Then the solution was washed with 150 mL of ethanol. The precipitates were then filtered through filter paper to produce quaternised starch precipitates. The procedures were repeated using 30 and 40 g of CTAB.

2.3 Preparation of polyvinyl alcohol solution

Ten grams of PVA pellets were dissolved in 90 mL of dimethyl sulphoxide (DMSO). The solution was then stirred at 400 rpm and heated at 90 °C for 3 h. Then, the solution was left to cool down to room temperature.

2.4 Preparation of hybrid solution

A solution was prepared by dissolving 2.4 g of quaternised starch in 40 mL of dimethyl sulphoxide at room temperature with vigorous stirring. Subsequently, 20 g of the quaternised starch solution was combined with 30 g of PVA solution for a 2:3 weight ratio 1 wt% TEOS was introduced into the mixture, followed by the addition of 1 mL of hydrochloric acid at a concentration of 37 wt% to initiate the sol-gel process at room temperature. The resulting solution was heated to 80 °C and stirred at 400 rpm for 6 h. This process was repeated for 3 and 5 wt% TEOS, respectively. The formulations are shown in Table 1.

2.5 Preparation of polysulfone (PSF) solution

In the preparation, 11 g of PSF resin pellets were dissolved with 4.25 g of PEG 400 in 69.7 g of NMP solution. The mixture was continuously stirred for 6 h at 400 rpm and 80 °C. Then the mixture was left to cool down to room temperature before preparation of the membrane film.

2.6 Preparation of integral membrane

For membrane preparation, one gram of hybrid solution and 40 g of PSF solution were mixed for three hours at 750 rpm and 80 °C. The membrane film was cast using a film applicator. The solution was poured onto a glass plate to form a 200 µm-thick film. After that, it was submerged in a large basin of water for 24 h before the membrane film was dried for one day at room temperature.

Table 1. The formulation of hybrid membrane

Sample	Quaternised Starch Solution (g)	PVA Solution (g)	CTAB (g)	TEOS (wt%)	TEOS (g)
M1	20	30	20	1	0.5
M2	20	30	20	3	1.5
M3	20	30	20	5	2.5
M4	20	30	30	1	0.5
M5	20	30	30	3	1.5
M6	20	30	30	5	2.5
M7	20	30	40	1	0.5
M8	20	30	40	3	1.5
M9	20	30	40	5	2.5

Source: Authors' data

2.7 Characterisation of film

Fourier transform infrared spectroscopy (FTIR)

Fourier transform infrared spectroscopy analysis was performed using FTIR spectrum 2000 from Perkin-Elmer. It was used to determine which functional groups are present in the membrane. The evaluations were done at the wavelengths of 400 cm^{-1} and 4000 cm^{-1}. Three samples were evaluated, and the average was derived.

Tensile strength

The mechanical characteristics of each membrane were measured by using a Universal Testing Machine (Model MDX, Instron Engineering Corporation, Canton, MA, USA). A 2.5 kN load cell was used by following the ASTM D882-88 (American Society for Testing and Materials, 1988) standard procedure. The dimensions of each sample were 80 mm long and 20 mm wide. The thickness of the sample was measured using a vernier calliper. The test was conducted with an initial grip length of 50 mm and a crosshead speed of 50 mm/min. The elastic modulus, strain, break strain, and tensile strength were evaluated. Three samples were evaluated, and the average values for each parameter were calculated.

Water uptake analysis

The films were cut into a rectangular shape of 2 cm × 2 cm and immersed in ultrapure water. After 24 h, the wet films were removed and weighed immediately (m_{wet}). The films were oven-dried at 50 °C for 24 h. The weight of the film (m_{dry}) was then recorded. The water uptake was calculated based on the average obtained from five (5) samples of each film. The wt% water uptake was calculated using Eq. (1).

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

3. RESULTS AND DISCUSSION

3.1 Functional group analysis

The chemical functional groups and the effectiveness of the cross-linking process in the hybrid membranes were assessed using FTIR spectroscopy. Fig. 1(a), 1(b), and 1(c) showed FTIR spectra of the membranes with 20, 30, and 40 g of CTAB, respectively.

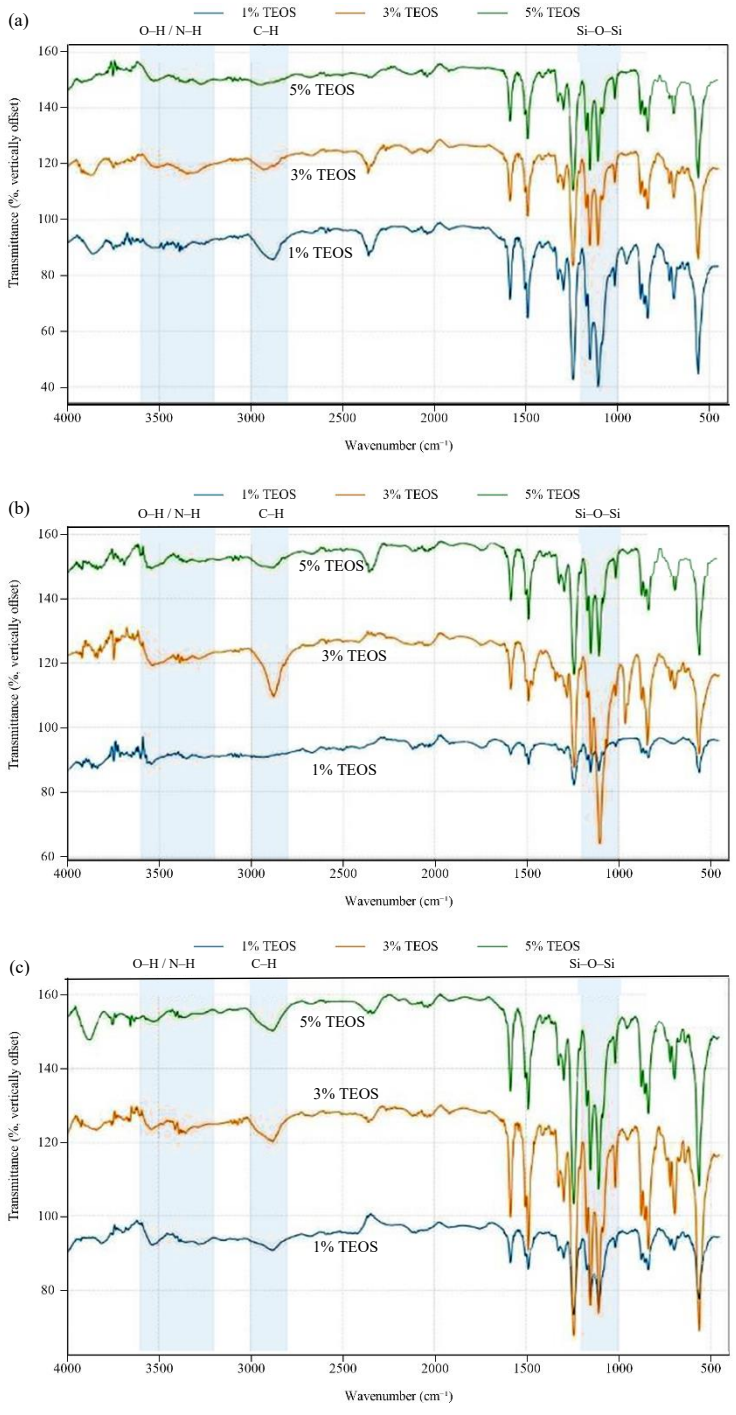


Fig.1. FTIR spectra (a) 20 g CTAB (b) 30 g CTAB (c) 40 g CTAB

Source: Authors' data

TEOS concentrations were 1, 3, and 5 wt% in Fig. 1. All samples showed broad absorption bands between 3600 and 3200 cm^{-1}, attributed to O–H stretching vibrations of the PVA and starch backbones, as well as N–H groups from the quaternised components. Sharp peaks in the 3000–2800 cm^{-1} region indicate aliphatic C–H stretching vibrations, which correspond to polymer hydrocarbon chains from the quaternised starch (Santinon et al., 2023). Increasing CTAB loading increased this peak's intensity due to a higher degree of quaternisation. Membranes with 40 g CTAB resulted in distinct and sharp peaks for the quaternary ammonium groups and aliphatic (C–H) groups at 1475 cm^{-1} as compared to other loadings of CTAB (Rukmanikrishnan et al., 2021).

For each figure, the spectra showed notable changes in the fingerprint region, particularly between 1200 cm^{-1} and 850 cm^{-1}, as the TEOS content rose from 1 to 5 wt%. The asymmetric Si–O–Si stretching modes are indicated by the band's appearance and intensity at about 900 cm^{-1}, which reflects the inorganic silica network formed by the hydrolysis and condensation of TEOS. Additionally, Si–OH and C– N stretching is responsible for the peaks seen at 1050 – 950 cm^{-1}, which show that the silica sol and the organic hydroxyl groups of the quaternised starch and PVA successfully interacted at the interface (Cruz-Quesada et al., 2021). From Fig. 1(c), the membrane with 40 g CTAB and 5 wt% TEOS shows the best results in terms of chemical integration and crosslinking density among the nine samples examined. A stronger inorganic network formation was evidenced by the sample's highest peak intensity in the 1000–1100 cm^{-1} region, which corresponds to the asymmetric stretching of siloxane bonds (–Si–O–Si) and (Si–O–C) linkages that increases with higher concentrations of TEOS. By comparing different loadings of CTAB, the 40 g series exhibit a more notable reduction and broadening of the hydroxyl (OH) stretching band (3200–3500 cm^{-1}), suggesting a higher level of interaction between the polymer matrix and the silica precursor (Wu et al., 2024).

3.2 Tensile Strength

Fig. 2 and Table 2 show the tensile characteristics and stress - strain behaviour of the membrane formulated with 20 g CTAB at TEOS concentrations of 1 wt%, 3 wt%, and 5 wt%. The findings showed that as TEOS concentration rises, tensile strength also rises. This improvement is due to the formation of the silica network that strengthens the polymer matrix and raises crosslinking density (Bapat et al., 2022). Similarly, the elastic modulus increases significantly with TEOS concentration, that portrays greater stiffness. The increase in stiffness and reduced strain resulted from the reduction of polymer chain mobility. The ultimate strain falls from 11 % at 1 wt% TEOS to 10.5% at 3 wt% TEOS, then declines further to 6.19 % at 5 wt% TEOS. This reduced elongation represents higher brittleness due to the inflexible and naturally brittle character of silica, which limits chain extensibility and promotes earlier fracture. A more continuous silica - polymer network generated at higher TEOS concentration could slow crack propagation and increase the strength (Z. Wang et al., 2022). As a result of higher stiffness, the 5 wt% TEOS membrane has the maximum strength with less flexibility. The membrane with 3 wt% TEOS provides the most balanced mechanical performance, combining high tensile strength, moderate stiffness, and acceptable elongation, making it the optimal composition for applications requiring both strength and flexibility.

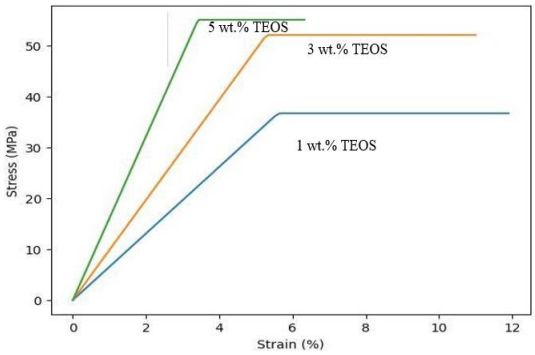


Fig. 2. Stress-strain curve of QSs/PVA/TEOS of 20 g CTAB membrane

Source: Authors' data

Table 2. Tensile properties of integral membranes 20 g CTAB

Sample	Ultimate Stress (MPa)	Ultimate Strain (wt%)	Break Stress (MPa)	Break Strain (wt%)	Modulus of Elasticity (MPa)
M1	37.1	11	36.7	11.9	685
M2	52.1	10.5	52.1	11	985
M3	55.8	6.19	55.1	6.32	1610

Source: Authors' data

Fig. 3 and Table 3 depict the stress‐strain and tensile parameters of membrane films formulated from 30 g CTAB with various TEOS concentrations. From Table 3, it was obvious that for each concentration of TEOS, the ultimate stress and break strain are lower than those for the membrane with 20 g CTAB. This phenomenon shows that excessive content of CTAB might cause agglomeration or structural heterogeneity, which disrupts the strength of the membrane matrix. The elastic modulus values are like those from 20 g CTAB. In terms of ductility, M5 has the lowest ultimate strain (2.35%), indicating the most brittle behaviour.

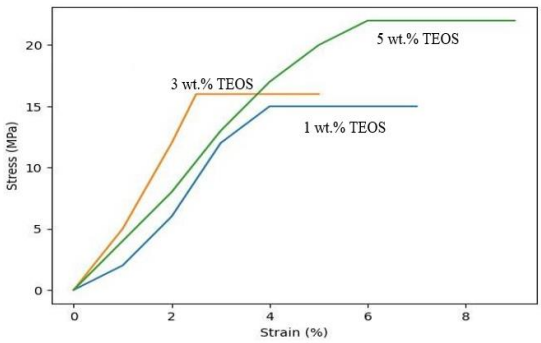


Fig. 3. Stress-strain curve of QSs/PVA/TEOS of 30 g CTAB membrane

Source: Authors' data

Table 3. Tensile properties of integral membranes 30 g CTAB

Sample	Ultimate Stress (MPa)	Ultimate Strain (wt%)	Break Stress (MPa)	Break Strain (wt%)	Modulus of Elasticity (MPa)
M4	15.4	4.43	15.4	4.43	610
M5	15.8	2.35	15.5	2.41	800
M6	22.9	6.39	19.2	6.7	968

Source: Authors' data

Fig. 4 and Table 4 show the stress - strain behaviour of the membranes formulated from 40 g CTAB with different concentrations of TEOS. The same trend in the ultimate stress, break strain, and the elastic modulus as those of membranes from 20 g CTAB was observed. The ultimate stress is 35 MPa for 1 wt% TEOS, reduces to 22.1 MPa at 3 wt% TEOS, and then increases back to 36.7 MPa at 5 wt% TEOS. The reduced strength and strain for 3 wt% might be due to the excess degree of quaternisation that surpassed the crosslinking degree. The higher strength and elastic modulus at 5 wt% TEOS were due to increased silica network formation by TEOS hydrolysis and condensation, which increases load transfer and structural reinforcement within the polymer matrix. These results demonstrate that increasing TEOS content enhances crosslink density and reduces polymer chain mobility, resulting in enhanced stiffness. In terms of ductility, 1 wt% TEOS membrane has the highest ultimate strain (9.9 wt%), showing more flexibility. Due to the dense and brittle nature of the silica - rich network, increasing TEOS concentration reduces elongation at break, as seen with 3 wt% and 5 wt% TEOS membranes. This demonstrates the common trade-off between tensile strength and elongation in strongly crosslinked polymer systems.

The 40 g CTAB membrane demonstrated the best overall mechanical performance out of the three CTAB loadings. Membranes with 40 g CTAB exhibited tensile strength comparable to that of membranes with 20 g CTAB but with less stiffness as portrayed by the elastic modulus. The increase in strength was attributed to stronger network interaction because of an increase of the electrostatic interaction between quaternary ammonium cations and carboxylic acid anions (Han et al., 2022). Membrane M7 with 1 wt% TEOS exhibited the best performance in terms of good tensile strength (35 MPa) with moderate elastic modulus (664 MPa) and adequate elongation, which demonstrates an ideal balance between strength, stiffness, and strain capacity.

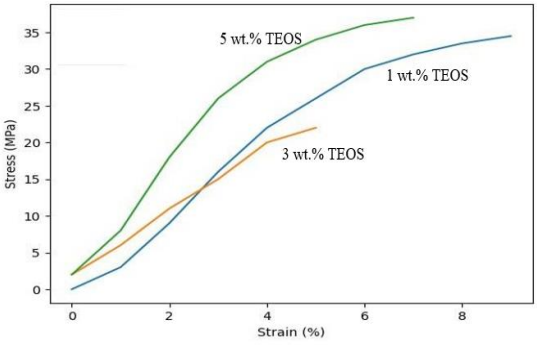


Fig. 4. Stress-strain curve of QSs/PVA/TEOS of 30 g CTAB membrane

Source: Authors' data

Table 4. Tensile properties of integral membranes 40 g CTAB

Sample	Ultimate Stress (MPa)	Ultimate Strain (wt%)	Break Stress (MPa)	Break Strain (wt%)	Modulus of Elasticity (MPa)
M7	35	9.9	35	10.3	664
M8	22.1	4.66	22.1	4.84	569
M9	36.7	6.67	34.3	7.34	968

Source: Authors' data

3.3 Water uptake analysis

Polyvinyl alcohol (PVA) and quaternised starch (QSs) are hydrophilic polymers because their molecular backbones contain many hydroxyl (–OH) groups. As a result, membranes made from these materials tend to absorb large amounts of moisture when exposed to water or humid conditions, which limits their use as membranes or thin films. Since excessive swelling may cause the membrane structure to partially disintegrate, high water affinity can have a negative impact on film handling, dimensional stability, and mechanical integrity.

Table 5. Water uptake analysis on hybrid membrane

Sample	W_{dry}	W_{wet}	Water uptake (wt%)
M1	0.039	0.067	71.8
M2	0.055	0.146	165.5
M3	0.041	0.089	117.1
M4	0.041	0.073	78
M5	0.087	0.277	218.4
M6	0.038	0.147	286.8
M7	0.042	0.064	52.4
M8	0.039	0.101	159
M9	0.057	0.142	149.1

Source: Authors' data

The water uptake values for all membranes are shown in Table 5. The M6 membrane had the largest water uptake among all samples, which is 286.8 wt%, indicating substantial swelling and a more open polymer network that allows water to infiltrate the matrix. The absorption of water in QS/PVA-based membranes are principally controlled by hydrogen bonding between water molecules and hydroxyl groups found in polymer chains. Based on the table, for each CTAB loading, increasing TEOS concentration significantly increases swelling behaviour but reduces slightly at 5 wt%. When the hydroxyl groups of quaternised starch interact with the siloxane, the quantity of free –OH groups that can absorb water is reduced (Wu et al., 2024). The compact structure of the membrane restricts the mobility of polymer chains and reduces the amount of water molecules that can enter the matrix, resulting in improvement in the dimensional stability and moisture resistance of the membranes (Wu et al., 2024). A higher concentration

of hydrophilic groups enhances the affinity for water, resulting in increased swelling. The percentage swelling of membranes depends on the strong siloxane (Si–O–Si) network created inside the polymer matrix by TEOS. However, 5 wt% of TEOS did not enhance the structural stability, which might be due to the agglomeration of an excess amount of silica. As seen in Table 5, membranes with 1 wt% TEOS at each CTAB loading were found to exhibit good structural stability and less swelling. Membrane M7 had the lowest water uptake, which was 52.4%, indicating a denser structure that limits water diffusion, and this formulation was the optimum.

4. CONCLUSION

This research suggests that good integration of quaternised starch with silica from tetraethyl orthosilicate substantially affects the structure and properties of the hybrid membranes. Although membrane M9 has good intensity in terms of the silica network, membrane M7 from 40 g CTAB and 1 wt% TEOS was determined to be the best overall formulation based on its moderate tensile strength (35 MPa) and low elastic modulus (664 MPa) while retaining adequate strain capacity, showing a good balance of strength, stiffness, and ductility, and exhibited moderate swelling, indicating greater structural stability.

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CONFLICT OF INTEREST STATEMENT

The authors agree that this research was conducted in the absence of any self-benefit, commercial or financial conflicts, and declare the absence of conflicting interests with the funders.

AUTHORS' CONTRIBUTIONS

Hazirah Zaki: Investigation, formal analysis, and writing – original draft; **Norin Zamiah Kassim Shaari:** Supervision, writing – review & editing, and validation.

DECLARATION OF GENERATIVE AI IN THE WRITING PROCESS

During the preparation of this work, the author(s) used Grammarly/improve grammar to write clearly and error-free. After using this tool/service, the author(s) reviewed and edited the content as needed and take(s) full responsibility for the content of the publication.

DATA AVAILABILITY/SUPPLEMENTARY MATERIALS

All data generated or analysed during this study are included in this published article.

ETHICAL COMPLIANCE STATEMENT

The authors confirm that this study was conducted in accordance with established ethical standards and institutional guidelines. All procedures complied with the Safety, Health, and Environmental (HSE) protocols of Universiti Teknologi MARA. Where human participants were involved, participation was voluntary, informed consent was obtained, and data anonymity and confidentiality were strictly maintained. The authors further confirm that the research was conducted with integrity, and all data were reported accurately without fabrication, falsification, or inappropriate manipulation.

ETHICS APPROVAL STATEMENT

The authors declare that this study did not involve human or animal subjects and complies with institutional ethical guidelines.

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