

Fabrication and Characterization of Polyvinyl Alcohol-Chitosan Mixed Matrix Membrane

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Abstract

Polyvinyl alcohol (PVA) and chitosan (CS) are biodegradable polymers with promising potential in membrane fabrication. However, a notable gap remains in identifying the optimal PVA-CS composition and additive concentrations that enhance membrane performance. This study seeks to address that gap by building on previous research to advance membrane technology for water treatment applications. In this study, blend membranes were prepared by mixing 1 wt% chitosan (dissolved in 2% v/v acetic acid) with varying concentrations of PVA (6 wt%, 8 wt%, 10 wt%, 12 wt%, and 14 wt%), using 5 wt% sorbitol as a plasticizer. The PVA and CS solutions were mixed in a 1:1 volume ratio and cast using the solution casting method. The membranes were characterized using swelling analysis, Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and tensile strength testing. FTIR results showed a decrease in O-H peak intensity with increasing PVA content, indicating stronger hydrogen bonding between PVA and CS. SEM images revealed smoother and more uniform surfaces at higher PVA concentrations. Swelling tests demonstrated that water uptake decreased with increasing PVA content. Specifically, at 6 g PVA, the swelling index was 4.8056%, whereas at 8 g PVA, it dropped to 3.7548%. Tensile strength analysis showed that increasing the PVA content from 6 g to 8 g significantly enhanced the mechanical properties, with the Young's Modulus rising from 7642.58 MPa to 13438.71 MPa. Overall, the results demonstrate that increasing PVA concentration enhances the membrane's structural and mechanical integrity, suggesting potential for water-related separation applications.

1. Introduction

Water is an essential resource that supports human health, industrial activities, and ecosystem balance. However, rapid industrialization and urbanization have introduced various pollutants into water sources, including heavy metals, organic compounds, and pathogens, which pose significant risks to both public health and the environment. This growing contamination has intensified the demand for efficient and sustainable water treatment technologies to ensure the availability of safe and clean water. Traditional treatment methods often

struggle to address the complexity and diversity of modern pollutants, prompting the exploration of advanced solutions. Membrane-based separation technologies have gained prominence due to their high purification efficiency, reduced chemical usage, and lower energy consumption, making them promising candidates for sustainable water treatment [1], [2], [3]. As Johnson stated, “the challenge of removing emerging contaminants requires technologies that are adaptable, efficient, and environmentally friendly” [2]. Developing and optimizing such technologies is crucial to meet the increasing global demand for clean water and to protect environmental sustainability.

Membrane separation technologies such as microfiltration, ultrafiltration, nanofiltration, and reverse osmosis have become vital in water purification due to their high efficiency, low chemical consumption, compact design, and energy-saving advantages, making them indispensable in municipal and industrial treatment systems [4]. However, the application of membranes in pervaporation systems faces significant challenges, including the trade-off between permeability and selectivity, membrane fouling, concentration polarization, and defect formation in thin membranes, which reduce separation performance and durability [5], [6]. Notably, “the selectivity of membranes decreases with decreasing thickness below a critical limit due to defect formation, which cannot be explained by morphology or sorption differences” [5]. Our research focuses on improving membrane materials and system design to enhance its structure and permeability while mitigating fouling and polarization effects, thereby advancing pervaporation efficiency and operational stability for water treatment applications [7], [8]. As noted, “in the PV process, the membrane plays the most pivotal role and is of paramount importance in governing the overall efficiency” [8].

The performance of membrane systems depends heavily on the materials used in membrane fabrication. Ideal membranes should possess high permeability, mechanical strength, chemical resistance, and fouling resistance to ensure durability and efficiency. Polyvinyl alcohol (PVA) and chitosan (CS) are two polymers of significant interest in membrane development. PVA is a synthetic polymer noted for its excellent film-forming ability, hydrophilicity, chemical stability, and biocompatibility, making it suitable for membrane applications [9]. Chitosan, a natural biopolymer derived from chitin, offers biodegradability, antimicrobial properties, and functional groups that allow chemical modifications [10]. Combining PVA and CS into mixed matrix membranes leverages the strengths of both polymers, potentially enhancing mechanical robustness and environmental sustainability [11], [12]. Hydrophilic membranes, such as those based on PVA and chitosan, are particularly advantageous in water treatment because their affinity for water molecules facilitates higher permeation rates and reduces fouling by minimizing hydrophobic interactions with contaminants [13]. As noted, “hydrophilic membranes exhibit superior antifouling properties due to their ability to form a hydration layer that resists organic foulants” [14]. This hydration layer not only improves flux but also prolongs membrane lifespan, making hydrophilic polymer blends a promising avenue for advancing membrane technology in sustainable water purification.

The incorporation of additives such as sorbitol and acetic acid into PVA-CS membranes plays a pivotal role in enhancing their physical and chemical properties, which directly impact membrane performance in water treatment. Sorbitol functions primarily as a plasticizer, reducing brittleness and improving membrane flexibility and mechanical strength by disrupting intermolecular hydrogen bonds within the polymer matrix [9]. This plasticizing effect facilitates better film formation and durability under operational stresses. Acetic acid, on the other hand, is widely used not only as a solvent but also as a pH modifier during membrane fabrication, promoting improved polymer blending and influencing membrane morphology. Chuang et al. (2000) demonstrated that increasing acetic acid concentration in the casting solution modulates the membrane's skin layer thickness and porosity, enhancing permeability by inducing pore formation: “the addition of acetic acid can change the PVA membrane structure and filtration properties” [15]. Moreover, the acid increases the H_3O^+ ion concentration, accelerating the coagulant influx during phase inversion, which governs membrane asymmetry and performance [15]. Sorbitol's compatibility with PVA also affects surface properties; as the degree of hydrolysis of PVA increases, sorbitol transitions from surface segregation to acting as a homogeneously distributed plasticizer, thereby stabilizing membrane structure [15]. Despite these benefits, the optimal ratios of PVA, CS, and additives like sorbitol and acetic acid remain underexplored, limiting the ability to consistently tailor membrane properties for specific applications. As noted, “the prediction of the segregation is not simple and depends on multiple factors including hydrophobicity, compatibility, blockiness, surface energy, and the mobility of the components” [15]. Addressing this knowledge gap through systematic studies is essential to optimize membrane fabrication processes and scale up PVA-CS membranes for industrial water treatment applications.

Despite the promising effects of additives on PVA-CS membranes, a significant research gap persists in identifying the ideal composition and concentration of polymers and additives to maximize membrane performance. Although various studies have investigated blends of PVA and chitosan with different additive levels, results often remain inconsistent or lack comprehensive characterization, making it difficult to generalize findings or establish fabrication guidelines [15], [16]. For instance, while acetic acid is known to influence membrane morphology and permeability by modulating phase inversion kinetics, the precise mechanisms and

optimal concentrations for different polymer blends require further elucidation [15]. Similarly, sorbitol's role as a plasticizer is established, but its effect on long-term membrane stability and swelling behavior in mixed polymer systems is not fully understood [16]. This lack of systematic characterization hinders the ability to fine-tune membrane properties such as mechanical strength, permeability, selectivity, and antifouling capability, which are critical for practical applications. As highlighted in [16], the membrane composition and crosslinking temperature were optimized in terms of the mechanical and transport properties, indicating the necessity of multi-parameter optimization for performance enhancement [16]. Furthermore, the interplay between polymer interactions, additive effects, and processing conditions complicates the development of standardized fabrication protocols. Bridging this gap through rigorous experimental and analytical studies will enable the design of PVA-CS membranes with tailored properties, facilitating their broader adoption in water purification and other separation technologies. aimed prominence in water purification due to their high efficiency, low chemical use, compact design, and energy efficiency. Techniques such as microfiltration, ultrafiltration, nanofiltration, and reverse osmosis are widely applied to remove specific contaminants based on membrane pore size and material properties. These processes have become integral to municipal and industrial water treatment systems [1].

Addressing the gap in understanding the optimal PVA-CS ratio and additive content is essential for advancing membrane technology in water treatment. Achieving the right balance between permeability, mechanical strength, and fouling resistance is key to developing membranes that are both efficient and durable. This study aims to provide a comprehensive insight into the structure-property relationships of PVA-CS membranes modified with sorbitol and acetic acid, supporting the design of sustainable membranes tailored to meet water treatment demands. The analysis will cover membrane morphology, chemical structure via FTIR, mechanical properties such as tensile strength and flexibility, and swelling behavior. Focus will be placed on understanding how varying PVA composition enhances membrane flexibility and structural stability. Additionally, the study will assess how different compositions influence critical performance factors like water permeability and fouling resistance, which are vital for practical applications. By systematically exploring these variables, the research seeks to identify the most effective membrane formulation that combines functional performance with environmental sustainability. The findings are expected to offer valuable guidelines for optimizing PVA-CS membranes, ultimately contributing to the development of high-performance, hydrophilic membranes capable of addressing the growing challenges in clean and sustainable water treatment technologies.

2. Methodology

2.1 Materials

Materials used in this experiment include Polyvinyl Alcohol (PVA) and Chitosan powder membrane materials. Sorbitol powder with a molecular weight of 182.17 g/mol was used as a plasticizer, improve the membrane water retention and mechanical properties. Acetic acid is 99% purification and distilled water were used in this experiment. Acetic acid can help enhance the crosslinking between PVA and Chitosan by ensuring proper dispersion and interaction of the components.

2.2 Membrane Preparation

1 wt% of chitosan is dissolved in 2% (v/v) acetic acid with distilled water and stirred for one hour at room temperature until clear. Separately, 5 wt% of Sorbitol is dissolved in distilled water, and once fully dissolved, PVA powder (6wt%, 8wt%, 10wt%, 12wt%, 14wt%) is added and stirred at 90 °C until a clear solution forms. The chitosan and PVA solutions are mixed in a 1:1 volume ratio and stirred until homogeneous. The beaker was closed with aluminum foil to maintain the temperature. The final mixture is poured into a clean petri dish until it reaches 50 g, spread evenly, and dried in an oven at 50 °C for 24 hours before the membrane is peeled off.

2.3 Characterized of Membrane

2.3.1 Functional Group Analysis

Fourier Transform Infrared (FTIR) spectroscopy was used to analyze the functional groups present in the membrane. The five sample membranes were analyzed in the range of 4000 - 600 cm IR spectra, 64 scans using a Cary 630 FTIR (Agilent Technologies Pty Ltd., Mulgrave, Australia), equipped with an ATR device featuring a single diamond crystal reflection. The membranes were placed on top of the diamond crystal and held tight by a clamp.

2.3.2 Morphological Analysis

For morphology analysis, Scanning Electron Microscopy (SEM) images were obtained at 5kV of accelerating voltage with magnification ranging 200. This technique was used to study the cross-sections and thickness of the membranes. The membranes were coated with a thin layer of gold using a Quorum Q150R which sputter coater unit. Before starting morphology analysis, the membranes were dried in oven at 110 °C for 24 hours. This process was carried out to remove moisture and humidity from the membranes.

2.3.3 Swelling Index

A swelling experiment was performed using gravimetric method. This analysis was used to check the hydrophilicity on the membrane. The membranes were cut 2 cm x 2 cm. The membranes were weight in dry conditions accurately (W_d) and ensure the membrane is free from moisture and contaminant. The sample was immersed in the distilled water for 10 minutes. The membrane was wiped gently and weighed the membrane (W_s) until the data stable data were obtained. The degree of swelling, SI (%) was calculated.

2.3.4 Tensile Strength

A tensile strength was performed using Universal Tensile Machine by Lloyd Instruments. The membrane is cut into a dumbbell shape with width and length at the center of the dumbbell shape are 1 cm and 5 cm. The speed machine that is used is 10 mm/min. This technique was used to study the Young Modulus and the maximum capability strength of the membrane.

3. Result and Discussion

PVA-CS Membrane were prepared based on the concentration of Polyvinyl Alcohol between 6wt%, 8wt%, 10wt%, 12wt%, and 14wt%. The analysis that we used in this study is swelling index, morphology, and chemical properties analysis using FTIR and mechanical strength.

3.1 Mechanical Strength

Tensile Strength, Young's Modulus and strain refer to the membrane's ability to resist breaking or deformation when a stretching force is applied. It is a mechanical property that reflects the membrane's durability and flexibility. According to Table 1, the lower Young's Modulus and higher percentage strain is 14wt% of PVA, means 14wt% of PVA are more stiffness than others. More PVA content on the membrane will affect stiffness, because the PVA are crystalline structures. The structure builds more crosslinking whereas high crosslinking will follow high young modulus and lower strain [17]. Other than that, more crosslinking on the membrane may reduce mobility of polymer chains, often lower strain. The membrane becomes stiffer and resists bending. Higher PVA content also leads to higher tensile strength, cause of the stronger crosslinking. Crosslinking creates covalent bonds between chains, increasing the network density due to better load distribution and chain entanglement. In contrast, higher chitosan content on the membrane becomes more stretchable. Chitosan is adding to be flexibility and softness [10].

Table 1 Mechanical strength analysis

Concentration of PVA (wt%)	Young's Modulus (MPa)	Percentage Strain at Limit	Tensile Strength
6	7642.58	84.31	0.0014
8	13438.71	34.72	0.000654
10	85.376	42.18	0.3342
12	1715.36	73.63	0.0181
14	93.501	117.34	0.6619

Table 1 shows data are fluctuated, non-linear. This fluctuation may be attributed to uneven crosslinking efficiency, water retention and strong polymer interaction and hydrogen bonding and variability in membrane casting conditions which thickness of each membrane during the analysis testing.

3.2 Swelling Index Analysis as a Function of PVA Composition in PVA-CS Membranes

The swelling index is referred to the membrane's ability to absorb water and expand in size. It is to measure the degree of swelling for PVA-CS, different PVA weighing were used to compare the capability of each membrane to

resist swelling. The comparisons were made for all sample membrane based in triple times samples being immersed in distilled water for 10 minutes, and the mass after immersed were recorded. This means all the values for PVA-CS membrane are summarized in Table 2.

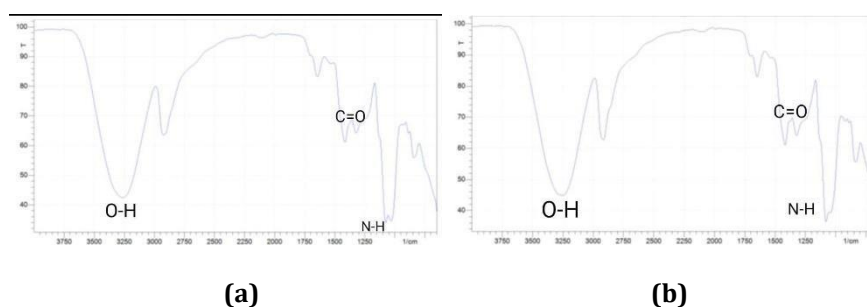
Table 2 Swelling index

Concentration of PVA (wt%)	Initial (g)	Final (g)	Swelling Index (%)
6	0.084	0.4876	4.8056
8	0.1033	0.4913	3.7548
10	0.079	0.469	4.9367
12	0.0976	0.3813	2.9044
14	0.1422	0.4723	2.32

According to Table 2, the higher swelling Index percentage is 10 wt% of PVA which is 4.9367%. the higher swelling index indicates a more hydrophilic and less crosslinked structure. It will be allowing more water to penetrate the membrane. PVA and chitosan can form hydrogen bonds with water molecules because it contains polar functional group (-OH and -NH₂). PVA has a large number of hydroxyl groups, PVA also making a highly hydrophilic, and Chitosan contain both amino and hydroxyl groups. Both materials can attract water and form a water molecule. The higher the number of hydrophilic functional groups, the greater the membrane' water affinity [17]. Next, the concentration of 14 wt% PVA is lower Swelling Index because of the membrane are tightly packed chains. Table 1 shows a 14 wt% are lower young's modulus which is stiffer and dense morphology. The membrane is not permeability [18]. The optimum swelling index is at 8 wt%, there are not too stiff where it is allowing water and at the same not brittle under high pressure.

3.3 Chemical Properties Analysis

FTIR spectroscopy was used to verify changes in the chemical structure of PVA and CS when concentration of PVA was changed and incorporated into the formulation. As the FTIR spectra examined, the broad peak of the hydroxyl (O-H) group stretching was observed at 3275 cm. As we can see from all the graphs, the only thing that makes them different from each other is their hydroxyl peak intensity, for example from the 6 g of PVA graph their hydroxyl peak is more intense than 8 g of PVA graph. The difference in peaks intensity between the graph indicated an increase in the O-H group as the concentration of PVA was increased for each membrane. As the PVA concentration was increased for each membrane, the O-H functional group that present in the membrane will also be increased. However, with higher PVA content, the excess hydroxyl groups tend to form stronger hydrogen bonds thus leading to tighter molecular packing. This tighter molecular packing will restrict the free vibration of O-H groups, then resulting in a decrease in peak intensity and shortening the O-H peak in the FTIR spectra [19]. This tighter molecular packing will help hydroxyl groups tend higher PVA content. The functional of amides can be observed at 1325 cm and 1625 cm, corresponding to conjugated carbonyl (C=O) group stretching and amide (N-H) group bending [16]. The presence of these functional groups in the spectra confirms the successful incorporation of chitosan within the membrane matrix. The presence of these functional groups in the spectra confirms the successful incorporation of chitosan within the membrane matrix. Therefore, the characterizations of the membrane were confirmed by the appearance of all functional groups in the FTIR spectra. Fig. 1 presents the FTIR spectra of PVA-CS membranes with varying PVA concentrations highlighting changes in the intensity of the hydroxyl (O-H) stretching peak.



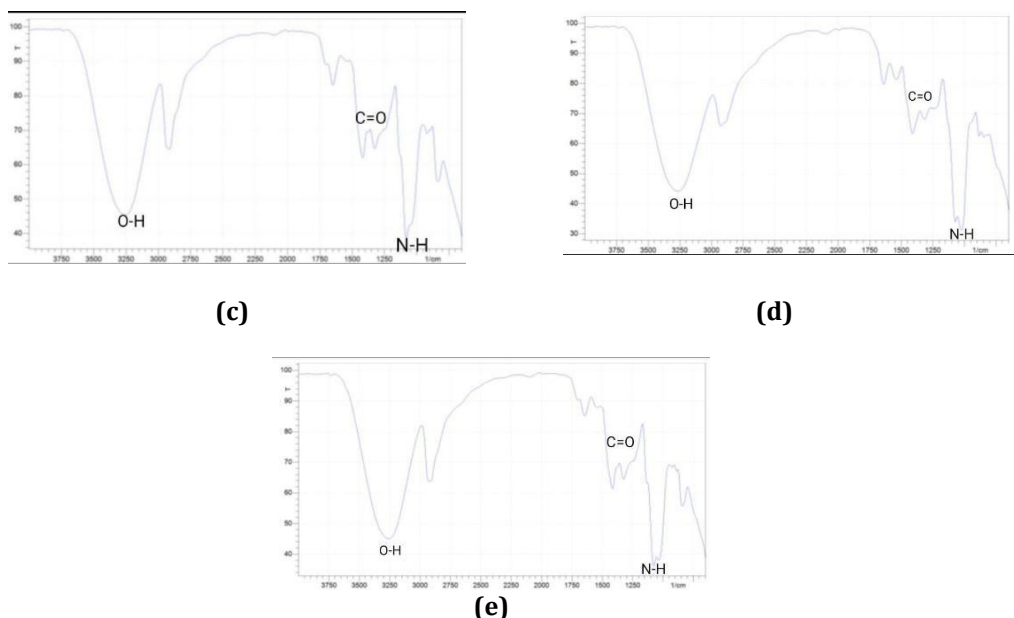


Fig. 1 FTIR spectra of PVA-CS membranes with different PVA concentrations (a) 6 g of PVA; (b) 8 g of PVA; (c) 10 g of PVA; (d) 12 g of PVA; (e) 14 g of PVA

3.4 Membrane Morphology and Its Influence on Separation Performance

The morphology refers to the surface structure and internal features of a material. This study was to check the thickness and cross-linking of the membrane. The observation on the membrane showed the difference between Fig. 2 (a) and Fig. 2 (b). Following Fig. 2 (b), the membrane are dense where it is a compact, tightly packed structure with no visible pores. Their surface is smooth and uniform. The membrane is having strong crosslinking, which limits the free volume inside the membrane [20]. The membrane is good for separation process because the structure provides high selectivity, whereas the membrane can effectively separate molecules. A dense membrane can selectively allow water to pass through due to its hydrophilic nature, while blocking ethanol molecules. Dense membrane structure is good for separation because it provides a tight, selective barrier that controls. It helps achieve high purity, better rejection, and stable performance in separation process. 14wt% is the good membrane because of the dense structure. In contrast, Fig. 2 (a) have visible pores within their structure, appear rough and uneven with open spaces. These membranes are allowed for higher permeability whereas it be less effective because the molecules water- ethanol to pass through more easily, reducing the separation efficiency [21].

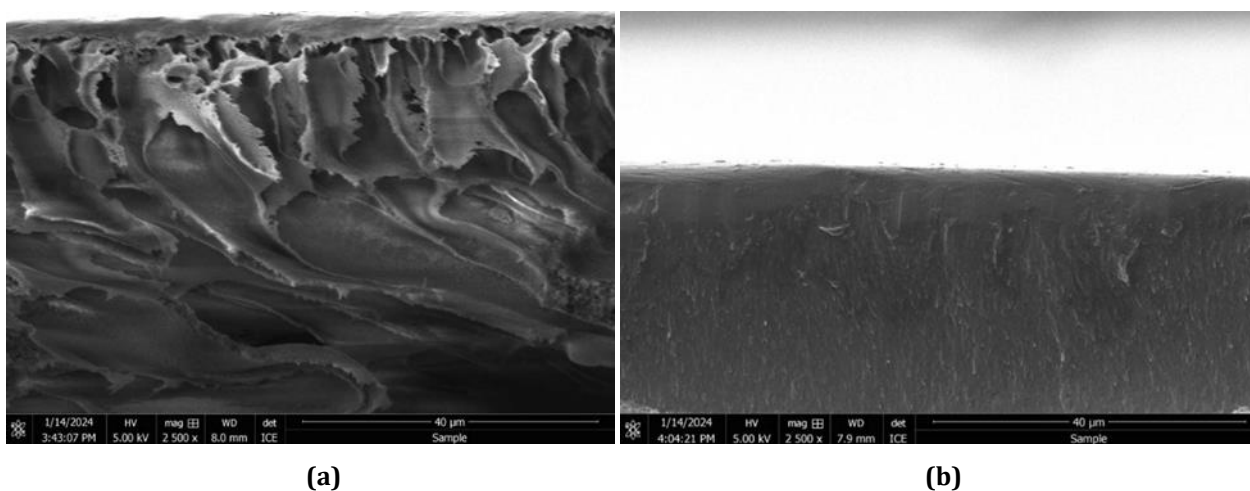


Fig. 2 Morphology of PVA-CS mixed matrix membrane of (a) porous membrane; (b) dense membrane

4. Conclusion

To conclude, this study aimed to develop a mixed matrix membrane using PVA and CS for application separation between water (H₂O) and ethanol. Based on the results obtained, it was found that the concentration of PVA

played a crucial role in producing an ideal membrane. The membranes were analyzed using four types of analysis which are morphology, chemical properties, tensile strength and swelling degree. From the results, it shows that the best concentration that can separate ethanol from water is 8wt% of PVA. This is because at the concentration of 8wt% of PVA the membranes have the optimum swelling index not too low and not too high, thus can give us higher selectivity, better mechanical stability and less structural distortion during separation. It is because if the swelling index is too low the membranes will swell too little, causing the permeation of the membrane to drop too much, thus resulting in very slow separation process [22]. Then, if the swelling index is too high the membrane will easily become loose and less durable over time because both ethanol and water can pass through easily cause of the lower selectivity [23]. After that, it is because the tensile strength for concentration of 8g PVA is moderately high, higher tensile strength will make the membrane more durable, mechanically stable under pressure and high temperature and less likely to tear or deform during separation process. However, if the tensile strength is very high the membrane will be too thick and the membrane will become too rigid thus reducing its swelling and diffusivity.

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Conflict of Interest

Authors declare that there is no conflict of interest regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: **study conception and design:** Zainul Zariff Zainul Hisham, Muhammad Ihsan Zulkifli, Aqil Naufal Mahdani; **data collection:** Zainul Zariff Zainul Hisham, Muhammad Ihsan Zulkifli, Aqil Naufal Mahdani; **analysis and interpretation of results:** Zainul Zariff Zainul Hisham, Muhammad Ihsan Zulkifli, Aqil Naufal Mahdani, Basirah Fauzi; **draft manuscript preparation** Zainul Zariff Zainul Hisham, Muhammad Ihsan Zulkifli, Aqil Naufal Mahdani, Raudah Mohd Adnan, Basirah Fauzi. All authors reviewed the results and approved the final version of the manuscript.

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