# Optimization of Polyurethane Membrane Physical Characteristics of Red Seaweed Biomass Using a Box-Behnken Design

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***Abstract:*** *The polyurethane membrane is used as a separator either by filtration or adsorption, and this process is significantly affected by its strength and physical condition. We synthesized polyurethane membranes using red seaweed with* Gracilaria sp *as a hydroxyl source. The Box-Behnken Design of the Response Surface Methodology (RSM) using Software Design Expert Version 10.0.3.0 with three factors (TRL, TDI, and Glycerin). The F-value of 0.42 suggests that the membrane is less fit, while the P-value of 75.10% indicates that the quadratic design model is suitable for data analysis of physical characteristics. The optimal physical characteristics were obtained at a composition of 0.233 g TRL, 2.675 g TDI, and 0.254 g glycerin with a physical point of 6.5 (strong and elastic). Optimal polyurethane membrane has good thermal and mechanical properties at temperatures of Tg 58 °C, Tm 322 °C, and Td 534 °C, as well as stress and nominal strain values of 69.3 MPa and 5.74%. Polyurethane membrane synthesized from red seaweed has good physical properties. The result of this research is the basis for the development of polyurethane membrane applications from red seaweed.*

***Keywords:*** *physical characteristics; response surface methodology; optimization; polyurethane membranes*

## ■ INTRODUCTION

Membranes are separators used to separate various substances in a gas mixture [1] or liquid [2-3]. This technology continues to develop due to its high selectivity properties [4-5] as well as its ability to be easily synthesized and applied [6-7]. One of the commonly developed membranes is polyurethane [8], which is a type of polymer membrane composed of urethane bonds [9] and formed from the reaction between compounds containing two or more reactive hydroxyl and isocyanate groups [10-12]. Polyurethane membranes can be synthesized using natural materials, such as carrageenan, which has strong and slightly elastic properties with a tensile strength of 340 kgf/mm$^2$, at a 9% elongation percentage [13]. In this study, we used the *Gracilaria sp* type of red seaweed as a hydroxy source for the synthesis of polyurethane membranes due to its abundant availability in coastal areas and the presence of hydroxy groups from carrageenan, alginate, and agar [14].

The polyurethane membrane is also applied as a separator either by filtration [15-16] or adsorption [2,17]. The strength and physical condition of the membrane significantly affect the success of the separation process, using a large pressure driving force and a concentration gradient during filtration. Therefore, a strong and slightly elastic membrane is required [6] to produce a suitable polyurethane membrane, which is strongly influenced by the composition of the materials used to regulate the formation of hard and soft segments [18-19]. The

research used the Box-Behnken Design from Response Surface Methodology (RSM) due to its ability to combine factorial and incomplete group designs to obtain optimal composition [20-21]. This design uses a Design Expert Software Version 10.0.3.0 with three factors and three levels.

Furthermore, RSM is also used to model and analyze the effect of the quantitative variable to obtain optimized results. The relationship between these variables can be described in the form of an equation [22-24]. Furthermore, the response used is the polyurethane membrane's physical or visual characteristics, which assign points to the characteristic level. These points were used as response variables and linked with other factors to produce 3D graphs to determine the optimal composition for polyurethane membrane synthesis. The physical characteristics can be used as a qualitative analysis of polyurethane membranes.

## ■ EXPERIMENTAL SECTION

### Materials

The materials used were 1,4-dioxane as a solvent, glycerin, castor oil as a plasticizer, and toluene diisocyanate (TDI) as a reagent in Pro's quality Analysis (PA) from Merck (Darmstadt, Germany). The sample used was red seaweed (*Gracilaria verucosa* Greville) from the ponds in Lamnga Village, Mesjid Raya District, Aceh Besar District, Aceh Province. The seaweed used was Seaweed Flour (TRL) with a particle size of 777.3 nm.

### Instrumentation

The equipment used includes glassware, namely measuring cups, beakers, Petri dishes, magnetic stirrers, hotplates, analytical scales, ovens, grinders, and other supporting tools. While the instruments used are FTIR (IR-Prestige-21, Shimadzu), DSC (DSC-60, Shimadzu), TGA (DTG-60, Shimadzu), SEM (JEOL - 6510 LA) and MTS EM tensile test with ASTM D638 Plastics Tension 1229.

### Procedure

#### *Treatment design*

Due to its ability to combine factorial with incomplete blocking design, the Response Surface Methodology with Box-Behnken Design was used to synthesize polyurethane membrane [20]. This research was carried out with the Software Design Expert Version 10.0.3.0 with three factors (TRL, TDI, and Glycerin) and three levels (low, medium, and high). As shown in Table 1, the factors and levels used in this study are based on preliminary investigations. In addition, the combined polyurethane membrane synthesis design using Box-Behnken Design is shown in Table 2.

#### *Polyurethane membrane synthesis*

TRL was weighed according to the combination and put in a beaker containing 5 g of 1,4-dioxane and 0.5 g of castor oil. The solution was homogenized for 10 min, followed by the addition of TDI and glycerin. The

**Table 1.** Design levels of polyurethane membrane synthesis by three factors (x)

| Factor | Parameter | Level | | |
|---|---|---|---|---|
| | | Low (-) | Medium (0) | High (+) |
| $x_1$ | TRL (g) | 0.1 | 0.2 | 0.3 |
| $x_2$ | TDI (g) | 2.0 | 2.5 | 3.0 |
| $x_3$ | Glycerin (g) | 0.2 | 0.3 | 0.4 |

TRL: Seaweed flour
TDI: Toluene diisocyanate

**Table 2.** Combined polyurethane membrane synthesis design using Box-Behnken Design

| Run | TRL (g) | TDI (g) | Glycerin (g) |
|---|---|---|---|
| 1 | 0.2 | 3.0 | 0.4 |
| 2 | 0.2 | 2.5 | 0.3 |
| 3 | 0.3 | 2.5 | 0.4 |
| 4 | 0.2 | 2.0 | 0.2 |
| 5 | 0.2 | 3.0 | 0.2 |
| 6 | 0.1 | 2.5 | 0.4 |
| 7 | 0.2 | 2.5 | 0.3 |
| 8 | 0.1 | 2.0 | 0.3 |
| 9 | 0.3 | 2.0 | 0.3 |
| 10 | 0.1 | 2.5 | 0.2 |
| 11 | 0.3 | 2.5 | 0.2 |
| 12 | 0.2 | 2.5 | 0.3 |
| 13 | 0.2 | 2.0 | 0.4 |
| 14 | 0.3 | 3.0 | 0.3 |
| 15 | 0.2 | 2.5 | 0.3 |
| 16 | 0.2 | 2.5 | 0.3 |
| 17 | 0.1 | 3.0 | 0.3 |

mixture was then polymerized at 60 °C for 2 h while stirring using a magnetic stirrer. The dope solution formed was then molded using a petri dish, then placed in a dust-free room at room temperature for 24 h. After forming the membrane sheet, the membrane was removed from the mold by immersing it in warm distilled water for 1–2 h.

### *Physical characterization*

The visible physical characterization of polyurethane membranes is shown in Table 3. According to points 6 to 7 of the physical characteristics, the membrane is elastic, not easily torn, or strong. This characterization is an initial qualitative analysis used to determine the optimal composition comprising functional group, thermal, and tensile strength analysis.

### *Polyurethane membrane characterization*

The optimal polyurethane membrane characterization of functional groups using Fourier Transform Infra-Red (FTIR), samples were made into KBr pellets (ratio 1:20), the spectrum was recorded in the wavenumber range of 4000–400 $cm^{-1}$. Thermal analysis using the Differential scanning calorimetry (DSC) and Thermogravimetric Analysis (TGA). The observation was carried out under a nitrogen gas flow with a speed of 20 mL/min. The sample with a 10–20 mg weight was heated in an aluminum pan at a temperature of 0–600 °C with a scanning speed of 20 °C/min. For Scanning Electron Microscope (SEM) analysis, the sample was placed on an aluminum plate and coated with palladium gold in a vacuum chamber. The sample was analyzed using Det.BSE and SE at a voltage of 10, 15, and 20 kV. Mechanical analysis using MTS EM tensile test with ASTM D638 Plastics Tension 1229.

## ■ RESULTS AND DISCUSSION

### Physical Characteristics of Polyurethane Membranes

Polyurethane membranes are formed by the presence of urethane bonds that occur between two main groups, namely hydroxyl (–OH) and isocyanates (NCO) [18,25], as shown in Fig. 1. The urethane bonds can affect the physical properties of the polyurethane membrane. Points 5 to 8 in Table 3 show that the resulting polyurethane membrane has good physical properties, namely elastic, and does not tear easily. The results of the physical characterization are then entered into the Box-Behnken design, as shown in Table 4.

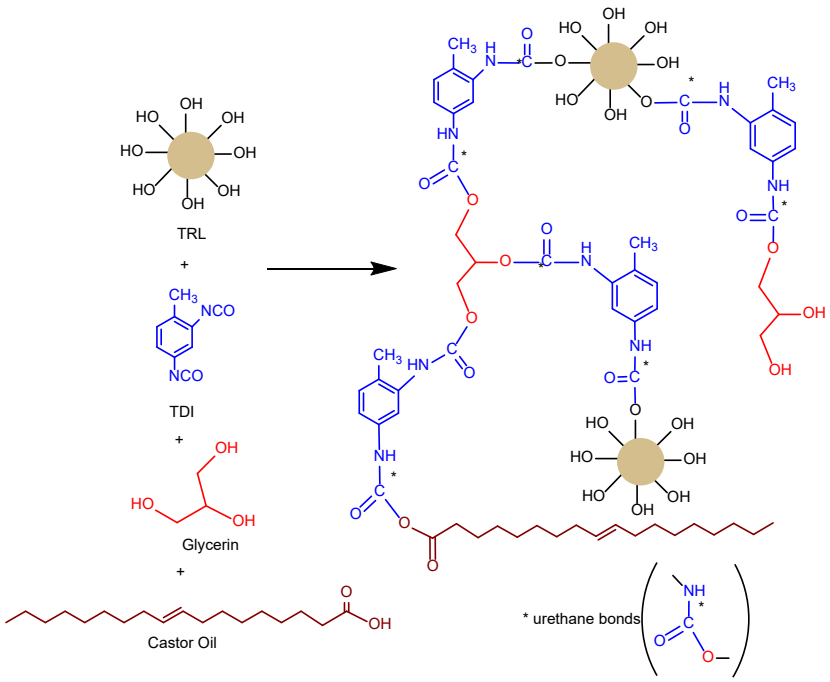


**Fig 1.** Reaction of polyurethane membrane synthesis

## Statistical Design Model

The optimum conditions for synthesizing polyurethane membranes were determined using Box-Behnken Design in Software Design Expert Version 10.0.3.0 with three factors (x) in three levels. This led to 17 runs, as shown in Table 2. The quadratic model design was chosen in this study because it produced high $R^2$ values and low PRESS values for the response to physical characteristics compared to the 2FI, linear, and cubic models. The greater the $R^2$ value, the higher the contribution or role of the factor (x) to the response (y). With the value of $R^2$ above 70%, it is considered to be good enough [20]. Although the cubic model has a high $R^2$ value, it does not have a Pred-$R^2$ and a PRESS value. Therefore, the effect of each variable with a different signal is not controlled. The statistical design and variance analysis of the polyurethane membrane synthesis model are shown in Tables 5 and 6, respectively.

Pred R-Squared and Adj R-Squared values are 0.6694 and 0.8489, with a reasonable difference of 0.18, which is less than the expected difference of 0.2. The Adeq Precision value is the signal to noise ratio, which is greater than 4, with a ratio of 10.520, in the quadratic model, which indicates an adequate signal. Therefore, this model can be used to navigate spatial design [24,26-27]. The Model F value is 10.99, and Prob > F less than 0.0500 indicates that the model is significant. The significant factors to the polyurethane membrane characteristics are B, C, BC, and B2, with a p-value less than 0.0500 and greater than 0.100, thereby indicating that the model is insignificant. Furthermore, the insignificance of the F-value of lack of fit at 0.42 and a p-value of 75.10% indicates that the quadratic design model is suitable for

**Table 3.** Point physical characteristics of polyurethane membranes

| No | Physical characteristics | Point |
|---|---|---|
| 1 | Crushed or shapeless | 1 |
| 2 | Brittle or crumbles easily | 2 |
| 3 | Not elastic and breaks easily | 3 |
| 4 | Not elastic and does not break easily | 4 |
| 5 | Elastic and easy to tear | 5 |
| 6 | Elastic does not tear easily | 6 |
| 7 | Elastic and strong | 7 |
| 8 | Stiff and easy to tear | 8 |
| 9 | Stiff and not easily tear | 9 |
| 10 | Rigid and hard | 10 |

**Table 4.** The physical characteristics results of the polyurethane membrane

| Run | Factor 1 A:TRL (g) | Factor 2 B: TDI (g) | Factor 3 C: Glycerin (g) | Response Physical (Point) |
|---|---|---|---|---|
| 1 | 0.2 | 3.0 | 0.4 | 8 |
| 2 | 0.2 | 2.5 | 0.3 | 7 |
| 3 | 0.3 | 2.5 | 0.4 | 8 |
| 4 | 0.2 | 2.0 | 0.2 | 5 |
| 5 | 0.2 | 3.0 | 0.2 | 5 |
| 6 | 0.1 | 2.5 | 0.4 | 7 |
| 7 | 0.2 | 2.5 | 0.3 | 7 |
| 8 | 0.1 | 2.0 | 0.3 | 5 |
| 9 | 0.3 | 2.0 | 0.3 | 5 |
| 10 | 0.1 | 2.5 | 0.2 | 6 |
| 11 | 0.3 | 2.5 | 0.2 | 6 |
| 12 | 0.2 | 2.5 | 0.3 | 7 |
| 13 | 0.2 | 2.0 | 0.4 | 6 |
| 14 | 0.3 | 3.0 | 0.3 | 7 |
| 15 | 0.2 | 2.5 | 0.3 | 6 |
| 16 | 0.2 | 2.5 | 0.3 | 7 |
| 17 | 0.1 | 3.0 | 0.3 | 6 |

**Table 5.** Statistical design model of polyurethane membrane synthesis

| Source | Linear | 2FI | Quadratic | Cubic |
|---|---|---|---|---|
| Std.Dev | 0.69 | 0.68 | 0.39 | 0.45 |
| R-Square | 0.6139 | 0.7083 | 0.9339 | 0.9496 |
| Adj R-Square | 0.5248 | 0.5333 | 0.8489 | 0.7985 |
| Pred R-Square | 0.3010 | -0.0444 | 0.6694 | N/A |
| Adeq Precision | 9.005 | 6.869 | 10.520 | 7.671 |
| PRESS | 11.10 | 16.59 | 5.25 | N/A |

**Table 6.** ANOVA analysis for a quadratic model of the physical characteristics of a polyurethane membrane

| Source | Sum of Squares | df | Mean Square | F Value | p-value Prob > F | Characterization |
|---|---|---|---|---|---|---|
| Model | 14.83 | 9 | 1.65 | 10.99 | 0.0023 | significant |
| A-TRL | 0.50 | 1 | 0.50 | 3.33 | 0.1106 | |
| B-TDI | 3.13 | 1 | 3.13 | 20.83 | 0.0026 | |
| C-Glycerin | 6.13 | 1 | 6.13 | 40.83 | 0.0004 | |
| AB | 0.25 | 1 | 0.25 | 1.67 | 0.2377 | |
| AC | 0.25 | 1 | 0.25 | 1.67 | 0.2377 | |
| BC | 1.00 | 1 | 1.00 | 6.67 | 0.0364 | |
| $A^2$ | 0.095 | 1 | 0.095 | 0.63 | 0.4529 | |
| $B^2$ | 3.41 | 1 | 3.41 | 22.74 | 0.0020 | |
| $C^2$ | 0.042 | 1 | 0.042 | 0.28 | 0.6126 | |
| Residual | 1.05 | 7 | 0.15 | | | |
| *Lack of Fit* | 0.25 | 3 | 0.083 | 0.42 | 0.7510 | not significant |
| *Pure Error* | 0.80 | 4 | 0.20 | | | |
| Cor Total | 15.88 | 16 | | | | |

data analysis of physical characteristics [28]. The relationship between the polyurethane membrane's physical characteristics and the factor (x) based on the coefficient value is seen in Eq. (1) and the 3D plot in Fig. 2.

$$y = 6.80 + 0.25(A) + 0.63(B) + 0.88(C) + 0.25(AB) + 0.25(AC) + 0.50(BC) - 0.15(A)^2 - 0.90(B)^2 + 0.100(C)^2 \quad (1)$$

## Polyurethane Membrane Optimization

The optimization results using the Response Surface Methodology with Box-Behnken Design provide five solutions of polyurethane membrane composition from seaweed flour, as shown in Table 7. These five solutions can be used as a reference for the production of polyurethane membranes because they have a Desirability value of 1.0 [29]. The optimization results using the Response Surface Methodology resulted in a predictive physical value of 6.5 with strong and elastic physical characteristics. The experimental results of the optimal composition show the physical values 6 and 7, the results of the predictions and experiments show the appropriate results.

## Optimal Polyurethane Membrane Characterization

The FTIR spectrum results in Fig. 3 show the formation of the urethane bonds on the polyurethane membrane, indicated by the absorption attributable to the –NH bond at the wavenumber of1583 and 3348 $cm^{-1}$. It is also supported by the vibration of the –C=O bond at the wavenumber of 1726 $cm^{-1}$ with an intensity of 63.56%, –CN amine at the wavenumber of 1249 $cm^{-1}$ with an intensity of 63.38%, as well as –CH alkane the wavenumber 2931 $cm^{-1}$ with an intensity of 66.29%. In addition, the weakening of the –NCO uptake of toluene diisocyanate was also observed at the wavenumber 2265 $cm^{-1}$ [30].

Thermal analysis is an important parameter used to determine the membrane's stability against temperature

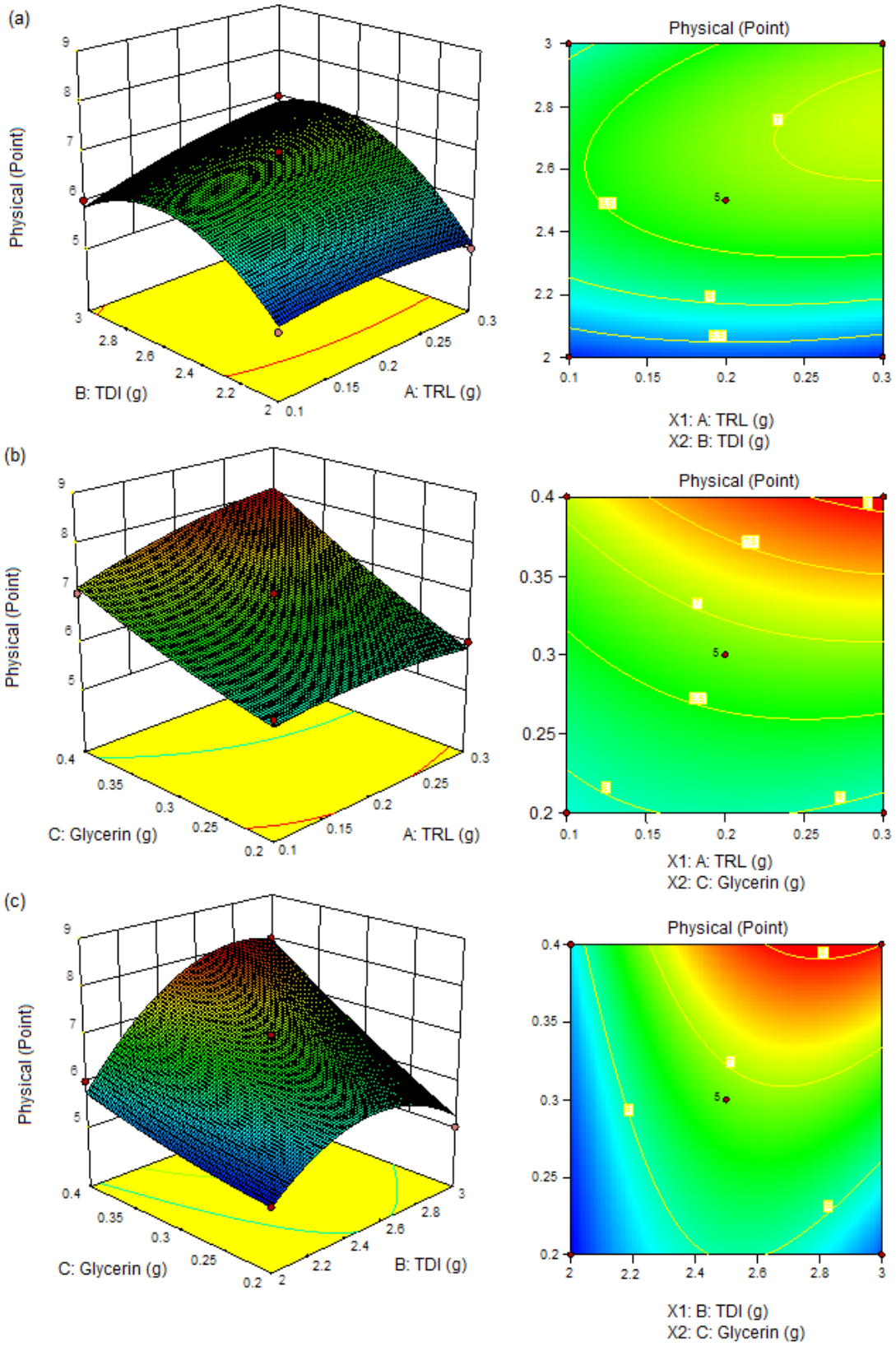


**Fig 2.** 3D plot and surface plot of the relationship between (a) TRL and TDI to Physical, (b) TRL and Glycerin to Physical (c) TDI and Glycerin to Physical

**Table 7.** Optimal compositional solution for polyurethane membrane synthesis

| No | TRL (g) | TDI (g) | Glycerin (g) | Physical (point) Predictions | Physical (point) Experiment | Desirability |
|---|---|---|---|---|---|---|
| 1 | 0.233 | 2.675 | 0.254 | 6.5 | 7 | 1.0 |
| 2 | 0.218 | 2.546 | 0.257 | 6.5 | 6 | 1.0 |
| 3 | 0.136 | 2.849 | 0.304 | 6.5 | - | 1.0 |
| 4 | 0.281 | 2.197 | 0.352 | 6.5 | - | 1.0 |
| 5 | 0.192 | 2.156 | 0.394 | 6.5 | - | 1.0 |

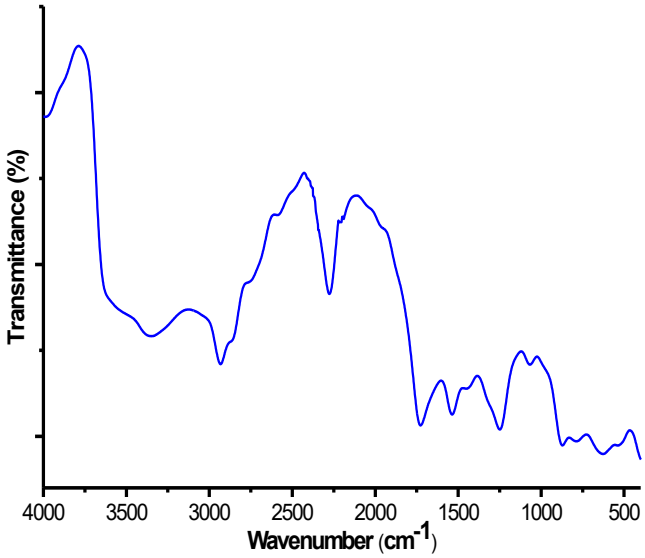


**Fig 3.** Optimal FTIR spectrum of polyurethane membranes

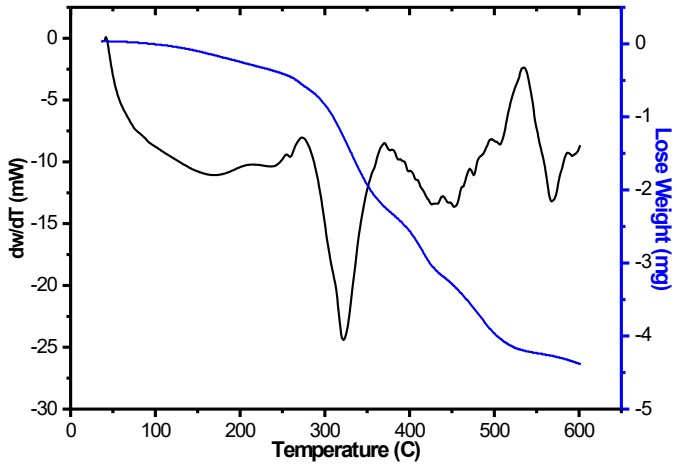


**Fig 4.** Optimal DSC thermogram and TGA polyurethane membrane

during storage and usage. The DSC thermogram (Fig. 4) shows that the optimal polyurethane membrane has a $T_g$ of 58 °C, $T_m$ of 322 °C, and $T_d$ of 534 °C, while the polyurethane membranes have $T_m$ and $T_d$ values. This high level is caused by the strong urethane bonds and cross-links that form crystal segments or regularities in the hard segments [1,18]. The polyurethane membrane from carrageenan has a $T_g$ of 243 °C and $T_m$ of 423 °C. The difference in value is due to the difference in the base material used and the polymerization conditions [13]. The TGA thermogram shows the change in sample weight to the optimal temperature rise of the polyurethane membrane at an initial degradation temperature ranging from 105.24 to 267.48 °C for water evaporation and cellulose determination. The weakest units in the macromolecular structure of the next degradation temperature are in the range of 267.48–423.17 °C, which is the degradation of urea and urethane bonds from hard segments and other aliphatic bonds. The final degradation value above 423.17 °C is the remaining structures [25,31-33] with a residue of 11.7%.

The SEM results of the optimal polyurethane membrane can be seen in Fig. 5. There is a gap on the inside of the membrane formed from the urethane bonds. The polyurethane membrane produced in this study is

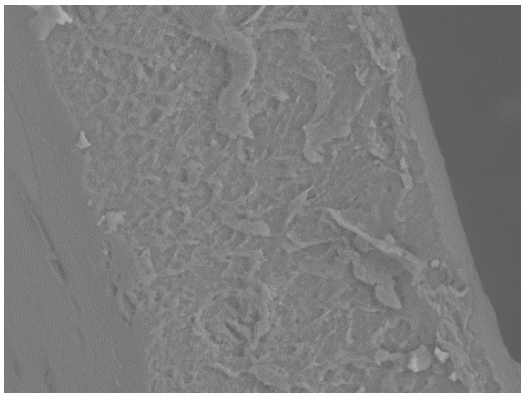

**Fig 5.** Cross-section of optimal polyurethane membrane

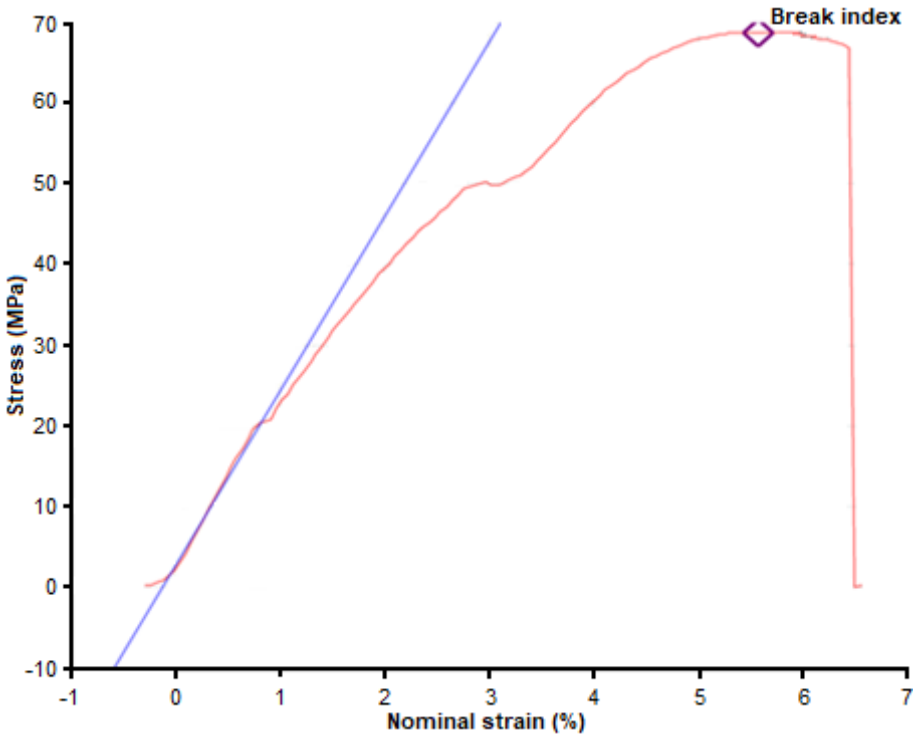


**Fig 6.** Optimal polyurethane membrane tensile strength curve

applied to the ammonia adsorption and filtration processes. In future studies, it needs to have good mechanical properties with the ability to withstand water pressure. The optimal polyurethane membrane's mechanical properties are good, with a stress value of 69.3 MPa and a nominal strain of 5.74%, as shown in Fig. 6. The expected polyurethane membrane has strong mechanical properties, and it is slightly elastic, with the strength of the membrane related to its ability to withstand water pressure. Meanwhile, the elongation or the level of membrane elasticity is related to the strain of pore size, which can affect the adsorption or filtration process of the analyte.

## ■ CONCLUSION

The polyurethane membrane's optimal physical characteristics made from red seaweed were obtained at a composition of 0.233 g TRL, 2.675 g TDI, and 0.254 g glycerin with a physical point of 6.5 (strong and elastic). Optimal polyurethane membrane has good thermal and mechanical properties, with a value of $T_g$ of 58 °C, $T_m$ of 322 °C, $T_d$ of 534 °C, a stress value of 69.3 MPa, and a nominal strain of 5.74%. The polyurethane membrane synthesized from red seaweed has good physical characteristics. The results of this study are the basis for the development of polyurethane membrane applications from red seaweed.

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## ■ AUTHOR CONTRIBUTIONS

SN conducted the experiment, S, BG, R, and M supervised and revised the manuscript. All authors agreed to the final version of this manuscript.

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