CHEMICAL ENGINEERING TRANSACTIONS
VOL. 122, 2025
A publication of AIDIC
The Italian Association of Chemical Engineering
Online at www.cetjournal.it
Guest Editors: Jeng Shiun Lim, Bohong Wang, Guo Ren Mong, Petar S. Varbanov

ISBN 979-12-81206-23-6; **ISSN** 2283-9216
DOI: 10.3303/CET25122029

Optimization of Mechanical Properties in Sago Cellulose-based Biodegradable Films for Sustainable Packaging

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Environmental concerns over plastic waste have accelerated research into biodegradable packaging. This study optimizes the mechanical properties of sago cellulose-based films using Response Surface Methodology (RSM) with a Central Composite Design (CCD). The key formulation factors investigated include glycerol content, pre-treated sago waste (PSW) content, and alkali treatment concentration, which significantly influence the film's elongation at break (EAB), tensile strength (TS), and Young's modulus (YM). The model developed demonstrated a strong correlation with an R^2 value of more than 0.5, confirming the model's adequacy. The optimized biofilm formulation achieved 12.547 MPa in TS, 100.421 % in EAB, and 97.499 MPa in YM, indicating a balance between strength and flexibility. The findings suggest that sago cellulose-based films have strong potential as sustainable packaging materials, offering an eco-friendly alternative to synthetic plastics.

1. Introduction

Plastic pollution has increased interest in biodegradable packaging. Bio-based films from natural polymers are promising due to their sustainability and degradability (Sondari et al., 2019). Among these, sago cellulose, a byproduct of sago palm processing, offers an eco-friendly material for film fabrication, particularly for food packaging (Tan et al., 2021). While sago starch has been commonly studied, the use of cellulose-rich sago waste remains underexplored. As an abundant industrial byproduct, sago waste holds potential for value-added applications like biodegradable films (Tan et al., 2021). However, these biofilms often lack the mechanical strength of synthetic plastics, limiting their use in packaging. Therefore, enhancing mechanical properties such as TS, EAB, and YM is essential.

The mechanical properties of biodegradable films are important for their practical use. This study optimized the mechanical properties of sago cellulose-based films using RSM with CCD. This method helps evaluate multiple factors at once to find the optimal condition (Anggraini et al., 2023). The three main factors studied were alkali treatment concentration, PSW content, and glycerol content. Alkali treatment modifies the cellulose structure, affecting film strength and flexibility (Loganathan et al., 2020). PSW cellulose fibers, enhances film structure and mechanical integrity (Abdullah et al., 2022). Glycerol acts as a plasticizer, to improve film flexibility, but potentially reduces TS (Lai et al., 2016). By adjusting these parameters and analyzing their interactions, an optimal formulation for sago cellulose-based biofilms was determined.

This study aims to develop a biodegradable film with optimized mechanical properties, contributing to sustainable packaging solutions. The findings provide valuable insights into the formulation of biofilms and highlight the potential of sago cellulose as an alternative material to conventional plastics. The use of RSM not only facilitates process optimization but also ensures a systematic approach to enhancing biodegradable film properties for practical applications (Anggraini et al., 2023).

Paper Received: 3 July 2025; Revised: 28 September 2025; Accepted: 22 November 2025
Please cite this article as: Donald J.J., Lai J.C.H., Atan M.F., Saree S., 2025, Optimization of Mechanical Properties in Sago Cellulose-based Biodegradable Films for Sustainable Packaging, Chemical Engineering Transactions, 122, 169-174 DOI:10.3303/CET25122029

2. Materials and methods

2.1 Materials

Sago hampas were collected from Pusa, Sarawak. Standard analytical grade chemicals, including sodium hydroxide (NaOH, Merck & Co.), glycerol ($C_3H_8O_3$, HmBG), and polyvinyl alcohol (PVA, Aldrich Resources Berhad) were purchased from local chemical suppliers and used without modification. The standard solutions were prepared using distilled water.

2.2 Film preparation

PSW biofilms were prepared via solution-casting method with slight modifications from Sondari et al. (2019). PSW was first treated with alkali, then combined with glycerol and PSW content as determined by CCD-RSM. Polyvinyl alcohol (PVA) was used as a matrix, dissolved in distilled water at 90°C with vigorous stirring until fully dissolved. The PSW and glycerol were added to obtain a homogeneous PSW mixture. 20 mL of the mixture was poured into a lubricant-sprayed 8.5 cm petri dish and left at room temperature for 2 h to stabilize and release trapped bubbles. The films were then dried in an oven at 40°C for 7 h, followed by equilibration in a desiccator for further use.

2.3 Experimental design and optimization

RSM-CCD in Design Expert 13 (Stat-Ease, USA) software was used in the data analysis process to identify the optimal composition to produce biodegradable films. The independent parameters were 1 – 3 M NaOH concentrations on PSW, 0.25 – 0.75 g PSW content, and 0.0 – 3.0 mL glycerol content, with coded and actual values presented in Table 1. The responses measured were TS, EAB, and YM, each tested in triplicate, and mean values were used for the analysis (Fathiraja et al., 2022). Regression models were developed to evaluate variable effects, and analysis of variance (ANOVA) was used to assess model significance and fit.

Table 1: Level of coded and uncoded values of variables

Independent variables	Coded levels	-1	0	1
NaOH concentration (M)	A	1.00	2.00	3.00
PSW content (g)	B	0.25	0.50	0.75
Glycerol content (mL)	C	0.00	1.50	3.00

At a 5 % significance level, the ANOVA results indicated that a quadratic model best fit the responses for TS and YM, while a linear model was suitable for EAB. Numerical optimization using a desirability plot was conducted, targeting minimum NaOH concentration, maximum PSW content, and minimum glycerol content. The computed values from the point of prediction served as the basis for the final experimental film development. The optimized film's response data was verified with a predicted response by absolute % error. A significance level of $p < 0.05$ is applied to the probability value.

2.4 Mechanical properties

Tensile testing was done according to ASTM D638 Type V using a Universal Testing Machine (Shimadzu AGS-X) with Trapezium software. TS, EAB, and YM of PSW biofilms were evaluated for 20 sample runs based on the CCD-RSM. The tensile test was performed with load cell of 500 N and test speed of 10 mm/min. Initial slope of stress-strain curve, maximum load, and final extension at break were used to calculate the TS, EAB, and YM. TS is the strength of a material when pulled or stretched, calculated by the amount of force a material can take before it breaks, divided by its original cross-sectional area. EAB measures how much the material stretches before it breaks, calculated by comparing length of material stretched at breaking point to its original length. YM shows how stiff a material is, calculated by dividing stress (how much force is applied) by strain (how much the material is stretched). A higher value means a stiffer material.

3. Results

3.1 Evaluation of mechanical properties

The effect of alkali-treated SW, PSW content, and glycerol content on the biodegradable film's mechanical properties was analyzed using CCD to optimize the independent variables. The measured responses included TS, EAB, and YM. A total of 20 runs were conducted, consisting of 6 central points and 14 true and axial points to estimate repeatability.

The independent variables of NaOH concentration (A), PSW content (B), and glycerol content (C) are indicated by their respective coded terms. The experimental PSW film's data shows that the TS ranges from 0.8372 to

14.6644 MPa. The analysis of the experimental data suited a quadratic model, showing the relationship between independent variables in actual equations as shown in Eq(1).

$$TS = +3.99 - 2.14 \times A + 52.83 \times B - 5.79 \times C + 1.76 \times A \times B - 0.20 \times A \times C + 2.39 \times B \times C + 0.64 \times A^2 - 71.44 \times B^2 + 1.20 \times C^2 \quad (1)$$

Eq(1) predicts the TS of the biofilm based on NaOH concentration, PSW content, and glycerol content. The positive coefficient for PSW content shows it improves TS, while NaOH and glycerol have negative effects. Interaction and squared terms indicate the factors influence TS in combination and in non-linear ways. This model helps explain how these variables affect the mechanical properties of the film. ANOVA results showed that the interaction between NaOH concentration, PSW content, and glycerol content significantly affected TS, best explained by a quadratic model shown in Table 2. The F-value and p-value indicated that the model was significant, with factors B, C, B^2, and C^2 having a notable influence. As shown in Table 3, the R^2 value of 84.96 % indicates a strong fit, although a negative R^2 suggests the overall mean may be a better predictor. The model's coefficient of variance (C.V%) was 26.24, demonstrating good duplicability, precision, and consistency in the experimental data.

Table 2: ANOVA for response surface reduced quadratic model for TS

Source	Sum of squares	df	Mean square	F-value	p-value	Remarks
Model	204.46	9	22.72	6.28	0.0041	Significant
A	10.32	1	1032	2.85	0.1222	
B	82.94	1	82.94	22.92	0.0007	
C	43.49	1	43.49	12.02	0.0061	
AB	1.54	1	1.54	0.43	0.5286	
AC	0.71	1	0.71	0.20	0.6677	
BC	6.42	1	6.42	1.77	0.2124	
A^2	1.14	1	1.14	0.31	0.5876	
B^2	54.83	1	54.83	15.15	0.0030	
C^2	20.01	1	20.01	5.53	0.0405	
Residual	36.19	1	3.62			
Lack of fit	36.19	5	7.24			
Pure error	0.00	5	0.00			
Cor total	240.66	19				

Table 3: Fit ANOVA statistical data of TS response

Std. Dev.	Mean	C.V%	R^2	Adjusted R^2	Predicted R^2	Adeq Precision
1.90	7.25	26.24	0.8496	0.7143	-1.1195	9.6310

The PSW films have EAB values ranging from 7.7359 % to 360.683 %. The analysis of experimental data suited a linear mathematical model, which gave the relationship between independent variables in actual equations as shown in Eq(2).

$$EAB = +207.63 + 13.81 \times A - 270.90 \times B + 57.87 \times C \quad (2)$$

Eq(2) predicts the EAB of the biofilm. It shows that increasing NaOH concentration and glycerol content helps improve elongation, while higher PSW content reduces it. This helps explain how each factor affects the film's flexibility. The ANOVA results in Table 4 showed how the linear mathematical model explains the link between the components and elongation responses. The model used in this study includes necessary response data, which explains both its relevance and lack of fit. The ANOVA indicated that the model was significant. In this investigation, factors C and C^2 significantly impacted the EAB response.

Table 5 shows the R^2 value of 57.33 % for EAB, indicating moderate predictive accuracy. This explains that 57.33 % of the variation in EAB is explained by the model, while the remaining 42.67 % is due to unexplained factors or random errors. A good model is shown by the adjusted and predicted R^2 differences being less than 0.2. Despite its limitations, the R^2 confirms a meaningful correlation between the input parameters and elongation. Adequate precision of 9.95, which is more than 4, signifies a strong signal for navigating the design space. The elongation response's C.V% value is 40.54, reflecting noticeable variability due to limitations of the current study and suggests that further experiments with more replicates and controlled conditions may improve the reliability of the EAB. The model still shows acceptable precision and consistency in capturing the experimental data.

Table 4: ANOVA for response surface reduced linear model for EAB

Source	Sum of squares	df	Mean square	F-value	p-value	Remarks
Model	1.23×10^5	3	41030.05	7.16	0.0029	Significant
A	1906.24	1	1906.24	0.33	0.5720	
B	45832.83	1	45832.83	8.00	0.0121	
C	75351.08	1	75351.08	13.16	0.0023	
Residual	91627.47	16	5726.72			
Lack of fit	91627.47	11	8329.77			
Pure error	0.0000	5	0.0000			
Cor total	2.15×10^5	19				

Table 5: Fit ANOVA statistical data of EAB response

Std. Dev.	Mean	C.V%	R^2	Adjusted R^2	Predicted R^2	Adeq Precision
75.69	186.65	40.54	0.5733	0.4933	0.3802	9.9466

YM range of the experimental PSW films was 4.0493 to 54.2587 MPa. The analysis of the experimental data followed a quadratic mathematical model, establishing the relationship between the independent variables in actual equations, as presented in Eq(3).

$$YM = -94.58 + 92.76 \times A + 368.89 \times B - 82.80 \times C + 20.25 \times A \times B - 2.24 \times A \times C - 2.67 \times B \times C - 24.01 \times A^2 - 404.71 \times B^2 + 23.82 \times C^2 \quad (3)$$

Eq(3) predicts the YM of the biofilm. It shows that YM increases with higher NaOH concentration and PSW content, but decreases with more glycerol. The interaction and quadratic terms reflect the influence of these variables on the film's stiffness.

According to Table 6, the results of ANOVA show how factors relate to one another and how the quadratic mathematical model defines YM. Due to the noise, the model's F-value of 2.32 indicates that it is not significant. The only important model terms that affected YM in this instance were C and C^2.

Table 7 shows an R^2 value of 67.61 %, indicating a fair ability of the independent variables to explain the variation in YM. A negative predicted R^2 suggested that the overall mean might be a more accurate predictor of the response. Adequate precision of 5.5830 confirms a sufficient signal for navigating the design space. The C.V% value in this study was high at 127.54, which typically indicates high variability. This may be due to the structural inconsistency of the PSW biofilm and sensitivity to changes in processing conditions. In this case, the model still demonstrated acceptable precision and duplicability based on the experimental results. Although the quadratic model for YM was statistically accepted based on the model summary, the p-value exceeded 0.05, and the predicted R^2 was negative. This indicates limited predictive accuracy and suggests that the current experimental data may not fully capture the variability in YM. As such, additional experimental runs and refinement of model factors may be necessary in future studies to improve prediction reliability for this response.

Table 6: ANOVA for response surface reduced quadratic model for YM

Source	Sum of squares	df	Mean square	F-value	p-value	Remarks
Model	15271.33	9	1696.81	2.3200	0.131	Not significant
A	119.34	1	119.34	0.1631	0.6948	
B	0.29	1	0.29	0.0004	0.9846	
C	6614.40	1	6614.40	9.0400	0.0132	
AB	205.01	1	205.01	0.2802	0.6081	
AC	90.35	1	90.35	0.1235	0.7326	
BC	8.00	1	8.00	0.0109	0.9188	
A^2	1586.27	1	1589.27	2.1700	0.1717	
B^2	1759.48	1	1759.48	2.4000	0.1520	
C^2	7900.12	1	7900.12	10.8000	0.0082	
Residual	7317.10	10	731.71			
Lack of fit	7317.10	5	1463.42			
Pure error	0.00	5	0.00			
Cor total	22588.42	19				

Table 7: Fit ANOVA statistical data of YM response

Std. Dev.	Mean	C.V%	R^2	Adjusted R^2	Predicted R^2	Adeq Precision
27.05	21.21	127.54	0.6761	0.3845	-1.3926	5.5830

3.2 Optimization analysis

Each response has its own objective function, and all are optimized simultaneously using a multiple-response optimization method. The criteria for optimizing all factors studied in correspondence with the responses are set within the range to achieve maximum TS, YM, and a target of 95 % EAB value at constraints of 1 - 3 M NaOH concentration, 0.25 – 0.75 g PSW content, and 0.01 – 3.00 glycerol content. These objectives were converted into individual desirability functions subject to the constraints, each ranging from 0 (undesirable) and 1 (highly desirable). The desirability function is used for the multiple-response optimization method, with the general formula of the function as shown in Eq(7).

$$Maximize\ D = [d_1(TS) \cdot d_2(EAB) \cdot d_3(YM)]^{1/3} \quad (7)$$

Where d_i is the desirability function of each response. The constraints defined the practical range for each experimental factor, and optimization was conducted using Design-Expert software to determine the ideal combination of A, B, and C for maximum overall desirability (D). Figure 1 illustrates the individual desirability values for each independent variable, which are NaOH concentration, PSW content, and glycerol content, and mechanical property, which are TS, EAB, and YM, contributes to the overall optimization outcome. The combined desirability score reflects the effectiveness of the selected formulation. The desirability values for the independent variables of NaOH concentration is 1.0000, PSW content is 0.9999, and glycerol content is 1.0000. For the responses, the desirability achieved in TS is 0.8469, EAB is 0.9796, and YM is 0.6071. The overall desirability index was 0.9037, indicating a strong compromise among all mechanical properties and validating the effectiveness of the optimized model.

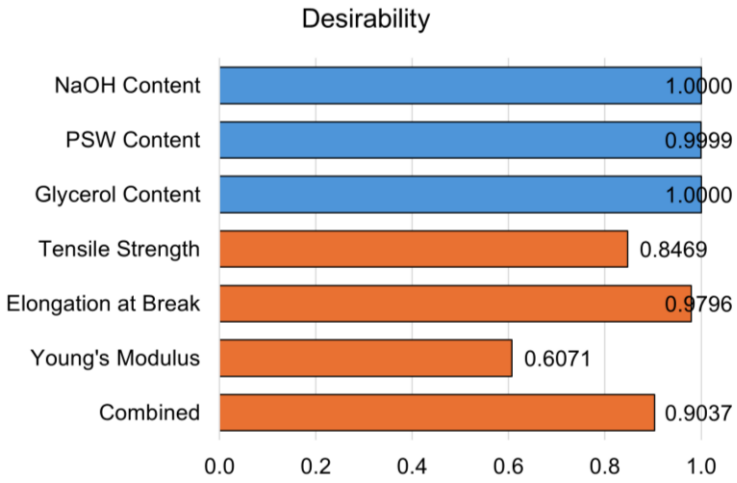

Figure 1: Bar graph representing the individual desirability of all responses in correspondence with the combined desirability

Table 8: Response value measured experimentally and predicted by software

Analysis	Unit	Predicted	Measured	95 % PI low	95 % PI high
TS	MPa	12.547	10.390	5.422	10.205
EAB	%	100.421	119.572	106.435	266.859
YM	MPa	97.499	36.281	-14.932	53.064

The optimal formulation was determined to be 2 M NaOH concentration, 0.5 g PSW content, and 0.01 mL glycerol content. Based on RSM model, the predicted mechanical properties were TS of 12.547 MPa, EAB of 100.421 %, and YM of 97.499 MPa. To verify the reliability of the model, experimental trials were performed under these optimized conditions using the Design-Expert software. Table 8 presents a side-by-side comparison of the predicted and measured results, along with the corresponding 95 % prediction intervals (PI).

For TS, the experimental value was 10.390 MPa, which was lower than the predicted value and slightly outside the PI upper bound of 10.205 MPa. This deviation suggests that while the RSM model captures the general trend, it may slightly overestimate TS under certain conditions. Possible causes for this difference include small but impactful variations in film preparation—such as non-uniform NaOH penetration or PSW dispersion—and inherent inconsistencies in the raw materials. Such factors can weaken molecular bonding and reduce TS, indicating that further refinement in process control or increased data points could improve prediction accuracy. In contrast, the EAB results showed excellent alignment between the model and experimental data. The experimental EAB of 119.572 % was well within the 95 % PI range of 106.435 % to 266.859 %, suggesting the model reliably predicts the flexibility and ductility of the films. This strong agreement also implies that the model effectively accounts for how glycerol and NaOH concentrations influence chain mobility in the polymer matrix.
For YM, the experimental value of 36.281 MPa fell within the wide PI range of -14.932 to 53.064 MPa but was considerably lower than the predicted 97.499 MPa. This large difference highlights a higher degree of uncertainty in the model's YM predictions, likely due to greater variability in stiffness-related responses across the experimental dataset. YM is particularly sensitive to microstructural changes—such as residual stresses, phase separation, or incomplete crosslinking—which may not be fully captured by the model. This suggests that while the current model is useful for qualitative trends, YM predictions may require additional experimental data or more targeted modelling approaches to improve precision.

4. Conclusion

This study successfully optimized the mechanical properties of sago cellulose-based biodegradable films using RSM-CCD via Design Expert 13 software. The results indicate that glycerol content, PSW content, and alkali treatment concentration significantly influence the film's TS, EAB, and YM. The optimized formulation yielded a TS of 10.390 MPa, an EAB of 119.572 %, and YM of 36.281 MPa, demonstrating an ideal balance between mechanical strength and flexibility. The statistical model used was found to be reliable, with an R^2 value of more than 0.5, validating the experimental results. These findings highlight the potential of sago cellulose-based films as a viable, sustainable alternative to conventional plastic packaging, contributing to the advancement of eco-friendly materials in industrial applications.

Acknowledgments

This research was supported by a grant from the Ministry of Higher Education with Grant No. NAT/I08/UMS-KPT/86361/2024.

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