ISSN (electronic): 2302-4399 http://jurnal.fp.unila.ac.id/index.php/jthp/index

Optimization of bioplastic production from nano-crystalline cellulose (NCC) oil palm empty fruit branch with sago starch matrix

[Optimasi pembuatan bioplastik dari nano-crsytalline cellulose (NCC) tandan kosong kelapa sawit (TKKS) dengan matriks pati sagu]

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Received : 17 September 2025, Revised : 30 September 2025, Accepted : 05 March 2026, DOI: 10.23960/jtihp.v31i1.123-133

ABSTRACT

The development of the bioplastics industry addresses critical global challenges, specifically environmental pollution and food safety concerns. Despite their potential, commercial applications of bioplastics remain limited due to suboptimal physical and mechanical characteristics compared to synthetic polymers. However, bioplastics derived from renewable resources offer significant potential as sustainable alternatives. This study investigates the utilization of Nano-Crystalline Cellulose (NCC) derived from Oil Palm Empty Fruit Bunches (OPEFB) as a reinforcing filler to enhance the performance of sago starch-based bioplastics. Additionally, the role of dispersing agents in improving the stability of liquid NCC preparations was evaluated. This study aims to evaluate the optimization of bioplastic formulation using a sago starch matrix reinforced with NCC derived from OPEFB and dispersing agents for potential application in food packaging. Optimization was conducted using the Response Surface Methodology (RSM) with a Central Composite Design (CCD) consisting of two blocks based on the type of dispersing agent (KCl and NaCl) and two factors, namely NCC concentration (1–5%) and dispersing agent (DA) concentration (0.5–3%). The results indicated that the optimal formula was achieved at an NCC concentration of 5% (X_1) and a DA concentration of 0.5% (X_2). This optimized composition yielded a tensile strength of 37.612 MPa and a Water Vapor Transmission Rate (WVTR) of 3.990 g/m^2/day. These findings demonstrate that the mechanical and barrier properties of the resulting bioplastics generally meet the stringent requirements for food packaging applications, positioning OPEFB-based NCC as a viable reinforcing agent for sustainable material development.

Keywords: bioplastics, dispersing agent, NCC, optimization, RSM

ABSTRAK

Pengembangan industri bioplastik merupakan solusi strategis terhadap permasalahan pencemaran lingkungan dan keamanan pangan. Meskipun aplikasinya secara komersial masih terbatas akibat karakteristik fisik dan mekanis yang relatif rendah, bioplastik tetap menjadi alternatif potensial sebagai pengganti plastik sintetis. Salah satu inovasi yang menjanjikan adalah pemanfaatan *Nano-Crystalline Cellulose* (NCC) dari tandan kosong kelapa sawit (TKKS) sebagai filler penguat, serta penggunaan agen pendispersi untuk meningkatkan kualitas sediaan cair NCC dalam proses pembuatan bioplastik. Penelitian ini bertujuan untuk memperoleh komposisi optimal dalam pembuatan bioplastik dengan matriks pati sagu menggunakan filler NCC TKKS dan agen pendispersi. Bioplastik berbasis NCC TKKS dengan matriks pati sagu dioptimasi menggunakan Metode Permukaan Respon (*Response Surface Methodology*/RSM) dengan rancangan *Central Composite Design* yang terdiri atas dua blok, yaitu jenis agen pendispersi (KCl dan NaCl) serta dua faktor, yaitu konsentrasi NCC TKKS (1−5%) dan konsentrasi agen pendispersi (0,5−3%). Hasil optimasi menunjukkan bahwa formula optimal diperoleh pada konsentrasi NCC 5% (X_1) dan konsentrasi agen pendispersi 0,5% (X_2), dengan respon kuat tarik sebesar 37,612 MPa dan laju transmisi uap air (WVTR) sebesar 3,990 g/m^2/hari. Karakteristik bioplastik yang dihasilkan, khususnya nilai kuat tarik dan WVTR, secara umum telah memenuhi persyaratan sebagai bahan kemasan pangan.

Kata kunci: agen pendispersi, bioplastik, NCC, optimasi, RSM

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Introduction

Synthetic plastics are one of the largest sources of environmental pollution because they are produced from petroleum-based materials that are not naturally biodegradable. This issue has encouraged the development of alternative materials to reduce plastic waste, one of which is bioplastics. Bioplastics are biodegradable polymers that can be decomposed naturally by microorganisms without causing harmful effects on the environment (Syafri et al., 2021). Various studies on bioplastic production have been conducted using different starch-based materials, including rubber cassava starch (Asngad et al., 2020), taro starch (Bidari et al., 2023), banana corn starch (Devi & Priatmoko, 2024), jicama starch (Syafri et al., 2019; (Taftazani & Dewata, 2022), sago starch (Silviana & Rahayu, 2019; Ayu et al., 2023), cassava starch (Asrofi et al., 2020; Nury et al., 2023), cassava peel starch (Fadhallah et al., 2025), and corn starch (Amalia et al., 2025).

Nanotechnology offers various opportunities for developing innovative and functional materials, including bioplastics. Nano-crystalline cellulose (NCC) is a nanoscale cellulose-based biomaterial with excellent mechanical properties, such as high tensile strength and modulus, and is also biodegradable. NCC can be utilized as a reinforcing material, filler, or carrier in various applications, including medical and pharmaceutical products, packaging materials, composites, wood and paper products, and food systems. Previous studies have reported the development of starch-based bioplastics reinforced with cellulose fibers derived from various sources, such as oil palm empty fruit bunch (OPEFB) fibers (Luthfi et al., 2025; Permadani & Silvia, 2022; Tsaniyah et al., 2026), aloe vera leaf fibers (Dasumiati et al., 2025), dan bamboo fibers (Tang et al., 2025).

Maryam et al. (2021) developed a bioprocess technology for producing NCC derived from OPEFB and applied it as a reinforcing material in polyvinyl alcohol (PVA)-based bioplastic composites. Their findings demonstrated that NCC derived from OPEFB has strong potential as a reinforcing filler. However, several challenges were identified, particularly related to the storage of liquid NCC suspensions, which tend to undergo agglomeration, and the relatively long processing time required for NCC slurry preparation. In addition, the resulting bioplastics showed imperfections, such as the presence of blank spots on the surface due to insufficient mixing and poor homogeneity of the solution. According to Silviana & Rahayu (2019), the addition of a dispersing agent in cellulose slurry preparation can improve mixture homogeneity and produce smoother bioplastic surfaces.

The addition of dispersing agents also plays an important role in the preparation of cellulose suspensions such as microfibrillated cellulose (MFC) and nano-crystalline cellulose (NCC). The stability of MFC and NCC suspensions is influenced by several factors, including ionic concentration (Silviana & Rahayu, 2019) which affects the formation and dispersion of cellulose structures used as fillers in bioplastics. Several studies have applied dispersing agents in bioplastic production, for example in bioplastics based on sago starch and bamboo-derived MFC using KCl as a dispersing agent (Silviana & Rahayu, 2019), bioplastics from avocado seed starch with NCC fiber fillers (Silviana & Rahayu, 2019), bioplastics from avocado seed starch with NCC fiber fillers (Fath et al., 2023), bioplastics produced from sago starch and OPEFB-derived MFC with 1% KCl salt (Rahayu et al., 2023), and OPEFB-based bioplastics plasticized with sorbitol and glycerol in the presence of KCl (Luthfi et al., 2025). The addition of KCl as a dispersing agent has been reported to stabilize bioplastic solutions and improve the dispersion of NCC suspensions (Ayu, 2021).

KCl and NaCl are ionic dispersing agents containing soluble salts that can influence intermolecular interactions within the bioplastic matrix. These salts can form a protective layer that promotes better interaction between ions and polymer chains, thereby modifying intermolecular tension within the material (Sun et al., 2024). The resulting films tend to exhibit smoother and glossier surfaces while providing improved protection for the polymer matrix (Fath et al., 2023; Sinek et al., 2020). Previous research by Rahayu et al. (2023) demonstrated that the addition of KCl in MFC-based bioplastics improved

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film flexibility without altering the bonding structure and significantly increased tensile strength compared with bioplastics without KCl. In general, dispersing agents enhance the uniformity of material distribution and improve the mechanical and thermal properties of the resulting bioplastics (Fath et al., 2023).

Although several studies have investigated the production of bioplastics using combinations of starch matrices, reinforcing fillers, and dispersing agents, most of them employed different materials and processing conditions, resulting in varied bioplastic characteristics (Fath et al., 2023; Luthfi et al., 2025; Rahayu et al., 2023; Silviana & Rahayu, 2019). Therefore, further studies are still required to obtain improved bioplastic properties through different material combinations. This study aims to evaluate the optimization of bioplastic formulation using a sago starch matrix reinforced with NCC derived from OPEFB and dispersing agents for potential application in food packaging.

Materials and method

Materials and equipment

The materials used consisted of nano-crystalline cellulose (NCC) derived from oil palm empty fruit bunch (OPEFB), sago starch obtained from PT. Pati Sagu Metro–Bogor, potassium chloride (KCl) (Emsure, Merck), sodium chloride (NaCl) (Emsure, Merck), glycerol (Merck), and distilled water supplied by PT. Novalindo. The equipment used in this study included a hotplate stirrer (Thermo Scientific), sieve set for particle analysis (ABM Test Sieving Analysis), oven (Memmert), ultrasonic bath (Elmasonic), laboratory glassware (Iwaki), and bioplastic molds.

Preparation of bioplastics

The preparation of bioplastics consisted of several stages. The first stage involved the preparation of NCC slurry derived from OPEFB. NCC powder was dispersed in a dispersing agent solution at concentrations ranging from 1–5% (w/w) (Maryam et al., 2021), while the dispersing agent concentration varied between 0.5–3.0% (w/w). Two types of dispersing agents were used as experimental treatments, namely potassium chloride (KCl) and sodium chloride (NaCl). The mixture was then subjected to ultrasonic treatment for 1 h to obtain a homogeneous NCC suspension. In the second stage, the starch solution was prepared by dissolving 4 g of sago starch in 100 mL of distilled water (Rahayu et al., 2023). Subsequently, 30 mL of the starch solution was taken for the bioplastic preparation. In the third stage, bioplastics were produced by mixing the NCC slurry with the starch solution according to the specified treatment conditions. The mixture was heated at 90 °C under continuous stirring. After 15 min of heating, glycerol (0.3% w/w) was added as a plasticizer to the mixture (Rahayu et al., 2023). In the final stage, the resulting bioplastic solution was poured into molds and dried at room temperature for 48 h to form bioplastic films. The produced bioplastics were then subjected to mechanical and barrier property testing, including tensile strength and water vapor transmission rate (WVTR) analyses.

Experimental design

The experimental design employed Response Surface Methodology (RSM) using a Central Composite Design consisting of two blocks representing the type of dispersing agent (KCl and NaCl). Two independent variables were evaluated, namely NCC concentration (1–5%) (Akli et al., 2022) and dispersing agent concentration (0.5–3%) (Rahayu et al., 2023). The responses measured in this study were tensile strength (MPa) and water vapor transmission rate (WVTR) (g/m^2/day). The experimental data were analyzed using Design-Expert (DX) software version 13.0 to determine the optimal formulation of bioplastics derived from nano-crystalline cellulose (NCC) of oil palm empty fruit bunch with a sago starch matrix for food packaging applications. Based on the Design-Expert 13.0 software, a total of 14 experimental runs were generated and subsequently verified according to the optimal solution obtained. The experimental design is presented in Table 1.

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Table 1. Experimental design

Std	Block	Run	NCC(%)	DA(%)
7	KCl	1	3	1.75
5	KCl	2	5	1.75
1	KCl	3	3	0.5
2	KCl	4	1	0.5
4	KCl	5	1	3
3	KCl	6	5	3
6	KCl	7	5	1.75
13	NaCl	8	3	1.75
9	NaCl	9	3	0.5
11	NaCl	10	1	3
14	NaCl	11	5	1.75
12	NaCl	12	5	1.75
8	NaCl	13	3	3
10	NaCl	14	1	0.5

Results and discussion

The results of bioplastic production using nano-crystalline cellulose (NCC) derived from oil palm empty fruit bunch (OPEFB) with a sago starch matrix, combined with variations in dispersing agents and NCC concentration, are presented in Table 2. The responses obtained in this study include tensile strength and water vapor transmission rate (WVTR). The experimental responses were further analyzed using Analysis of Variance (ANOVA) with the assistance of Design-Expert (DX) software version 13.0. The statistical analysis indicated that the developed models for both tensile strength and WVTR responses were significant and valid, as shown in Table 3.

Table 2. Response value of tensile strength and WVTR

Std	Block	Run	NCC (%)	DA (%)	Tensile strength (MPa)	WVTR (g/m^2/day)
7	KCl	1	3	1.75	18.594	6.274
5	KCl	2	5	1.75	34.062	4.384
1	KCl	3	3	0.5	29.137	5.148
2	KCl	4	1	0.5	14.828	6.462
4	KCl	5	1	3.0	12.218	6.645
3	KCl	6	5	3.0	33.322	4.482
6	KCl	7	5	1.75	29.995	5.076
13	NaCl	8	3	1.75	37.564	4.234
9	NaCl	9	3	0.5	33.363	4.436
11	NaCl	10	1	3.0	23.576	5.593
14	NaCl	11	5	1.75	39.541	3.471
12	NaCl	12	5	1.75	39.541	3.374
8	NaCl	13	3	3.0	31.806	4.682
10	NaCl	14	1	0.5	19.088	6.183

Table 3. ANOVA response result of tensile strength and WVTR

Response	Model type	F-value	P-value	R^2	Adj. R^2 Model	Pred. R^2 Model	Adeq. Precision	Lack of fit
Tensile strength	Linier	32.02	<0.0001	0.865	0.838	0.763	15.212	0.249
WVTR	Linier	35.73	<0.0001	0.877	0.853	0.775	16.132	0.520

Table 3 presents the results of the ANOVA for the regression models of tensile strength and WVTR responses. The F-value indicates that the generated regression models are statistically significant. A P-value of less than 0.05 suggests that the developed equations can adequately represent the

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experimental data at a confidence level of 95%. The coefficient of determination (R^2) shows that the model explains 86.49% of the variability in the tensile strength response and 87.72% of the variability in the WVTR response. These results indicate that the developed models meet the adequacy criteria for response surface methodology. The model validity is supported by the difference between the adjusted R^2 and predicted R^2 values, which is less than 0.2, and the Adeq Precision values, which are greater than 4. These criteria demonstrate that the models generated using Design-Expert version 13 are capable of adequately predicting the responses. Furthermore, the ANOVA results indicate that the regression models for both tensile strength and WVTR responses follow a linear model. The lack-of-fit values for tensile strength and WVTR were 0.249 and 0.520, respectively, indicating non-significant lack-of-fit. This result suggests that the models are suitable for describing the experimental data and meet the requirements of a good statistical model (Safira & Purbasari, 2022).

Tensile strength response analysis

The tensile strength value is an important parameter commonly used in bioplastic industry standards to evaluate the mechanical properties of bioplastic materials. The role and quantitative effects of each variable on the tensile strength response (Y_1) were evaluated using Design-Expert (DX) version 13 software. In this model, Y_1 represents the tensile strength (MPa), while the independent variables consist of NCC concentration (X_1, %) and dispersing agent concentration (X_2, %). The regression equation describing the relationship between these variables and the tensile strength response is presented in Equation (1).

$$Y_1 = 14.49 + 4.67X_1 - 0.48X_2 \quad (1)$$

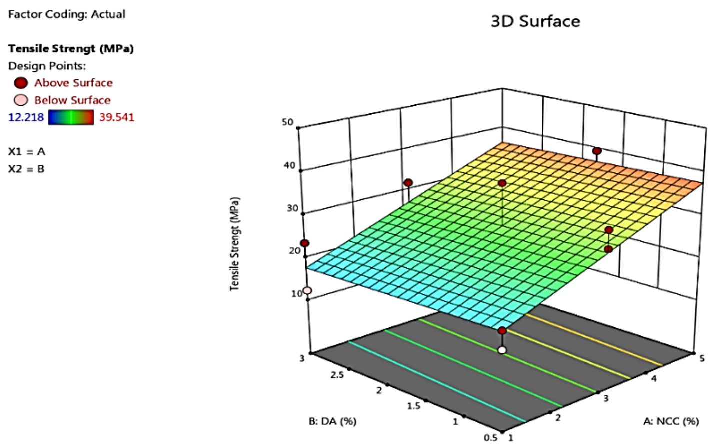

Figure 1. 3D surface plot of tensile strength response

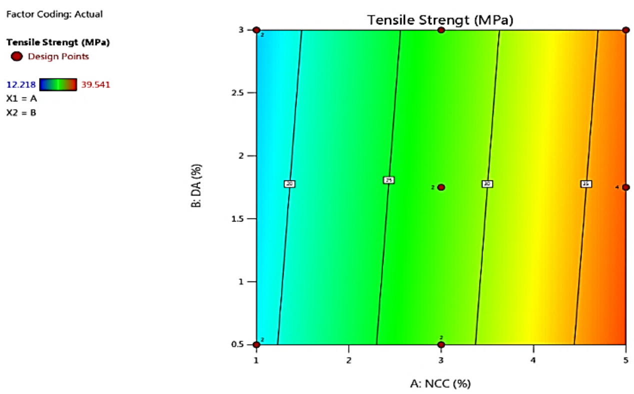

Figure 2. Contour plot of tensile strength response

The effect of NCC concentration and dispersing agent variation on the tensile strength response can be observed in Figure 1, which presents the three-dimensional surface plot, and Figure 2, which illustrates the

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contour plot. The color gradient ranging from blue to red indicates an increase in tensile strength values. The highest tensile strength obtained in this study was 39.541 MPa, while the lowest value was 12.218 MPa. Based on the regression equation for Y_1 (tensile strength), NCC concentration shows a positive effect on the response, indicating that increasing NCC concentration tends to enhance the tensile strength of the bioplastic films (Kasrawati & Sihotang, 2024) In contrast, the dispersing agent (DA) concentration exhibits an antagonistic effect, where lower DA concentrations tend to increase the tensile strength.

The increase in tensile strength with increasing NCC concentration is attributed to the strong intermolecular hydrogen bonding within NCC and its high interfacial surface area, which promotes stronger interactions within the polymer matrix (Maryam et al., 2021). However, excessively high concentrations of both NCC and dispersing agents do not necessarily improve tensile strength. This observation is consistent with the findings reported by Fath et al. (2023), who obtained the highest tensile strength of 1.79 MPa at an NCC concentration of 2% and a dispersing agent concentration of 2%.

WVTR response analysis

The water vapor transmission rate (WVTR) is an important parameter used to evaluate the barrier properties of food packaging materials and is included in the Japanese Industrial Standard (JIS) Z 1707 for food packaging. The regression model describing the WVTR response was developed using the experimental data obtained in this study. In this model, Y_2 represents the water vapor transmission rate ($g/m^2/day$), while the independent variables consist of NCC concentration (X_1, %) and dispersing agent concentration (X_2, %). The mathematical relationship between these variables and the WVTR response is expressed in Equation (2).

$$Y_2 = 6.69 - 0.54X_1 + 0.02X_2 \qquad (2)$$

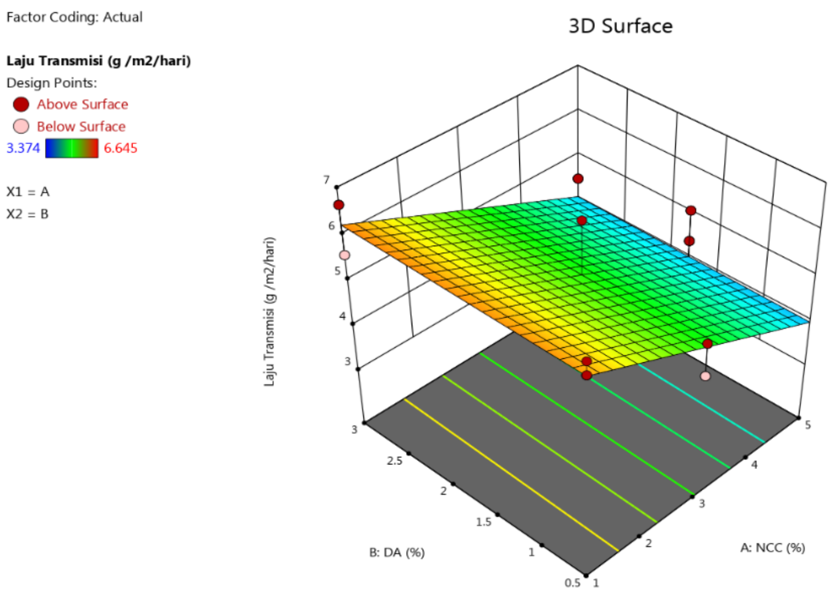

Figure 3. 3D surface plot of WVTR response

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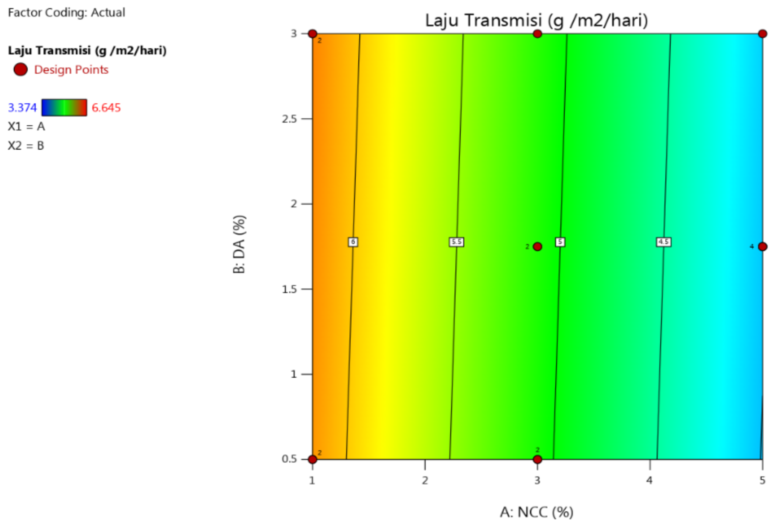

Figure 4. Contour plot of WVTR response

The results of the WVTR response analysis are presented as a three-dimensional surface plot in Figure 3 and a contour plot in Figure 4. The color gradient from red to blue indicates a decrease in WVTR values as the NCC concentration increases. The lowest WVTR value obtained in this study was 3.374 g/m^2/day, while the highest value was 6.645 g/m^2/day. Based on the regression equation for Y_2 (WVTR), NCC concentration shows a positive influence on the response, indicating that increasing NCC concentration tends to reduce the WVTR value (minimization) (Arifin et al., 2025; Maryam et al., 2021). Meanwhile, the dispersing agent (DA) concentration exhibits an antagonistic effect on the WVTR response.

These results indicate that achieving lower WVTR values for bioplastic food packaging requires a higher NCC concentration compared with higher concentrations of dispersing agents. Lower WVTR values are desirable in food packaging materials because they can prevent moisture loss or excessive water absorption, which may accelerate food deterioration (Mualimin et al., 2025; Santoso et al., 2021).

Optimization of bioplastic formulation

The optimal formulation was determined based on the solution options generated by the Design-Expert (DX) version 13.0 software using predefined optimization criteria, as presented in Table 4. The optimization process provides information regarding the selected formulation predicted by the software to achieve the desired response values. Table 4 presents the independent and dependent variables, optimization criteria, variable ranges, and the importance level assigned to each variable.

Table 4. Optimization criteria for bioplastic formulation

Independent and dependent variables	Criteria	Lower limit	Upper limit	Importance
NCC concentration (X_1) (%)	in range	1	5	3 (+++)
DA concentration (X_2) (%)	in range	0.5	3	3 (+++)
Tensile strength (Y_1) (MPa)	Maximize	12.218	39.541	5 (+++++)
WVTR (Y_2) (g/m^2/day)	Minimize	3.374	6.645	4 (++++)

Optimization is a process used to obtain response values with the highest possible desirability (Nury et al., 2024). The objective of optimization is to minimize operational costs while maximizing the desired response values. In this study, NCC concentration was optimized within a range of 1–5% with an importance level of 3 (+++), while the dispersing agent concentration was optimized within a range of 0.5–3% with the same importance level. Both independent variables influence the responses of tensile strength and WVTR. The tensile strength response was optimized with a maximization target and an importance level of 5 (+++++), while the WVTR response was optimized with a minimization target and an importance level of 4 (++++).

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Based on the optimization analysis, the Design-Expert 13.0 software generated 17 predicted solution points representing different combinations of the two independent variables and the response parameters. The availability of multiple predicted solutions demonstrates that response surface methodology using Design-Expert software can serve as an effective predictive tool for reducing the number of costly experimental trials. Therefore, the optimal experimental formulation can be selected from one of the predicted solutions with the highest desirability value.

The optimal solution was obtained at 5% NCC concentration (X_1) and 0.5% dispersing agent concentration (X_2), which produced predicted response values of 37.612 MPa for tensile strength and 3.990 g/m^2/day for WVTR. This formulation was considered optimal because it produced the highest desirability value of 0.905. The desirability value is used to evaluate the accuracy of the optimization results; values closer to 1 indicate higher optimization accuracy. However, optimization does not necessarily aim to achieve a desirability value of exactly 1.0 but rather to determine an optimal process that satisfies all objective functions between process parameters and response variables (Chelladurai et al., 2020). The desirability profiles are illustrated in Figure 5 and Figure 6 as three-dimensional surface plots and contour plots, respectively.

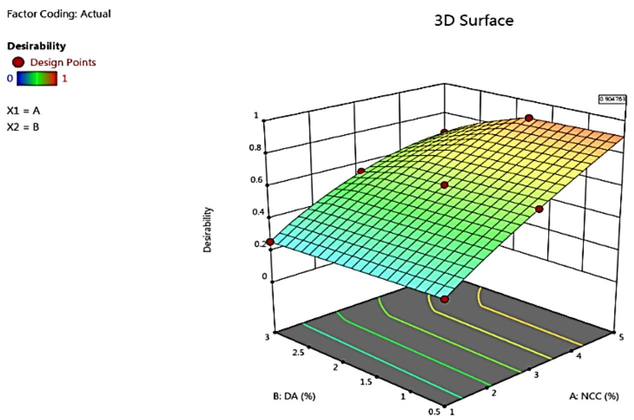

Figure 5. Three-dimensional desirability plot of the optimized bioplastic formulation

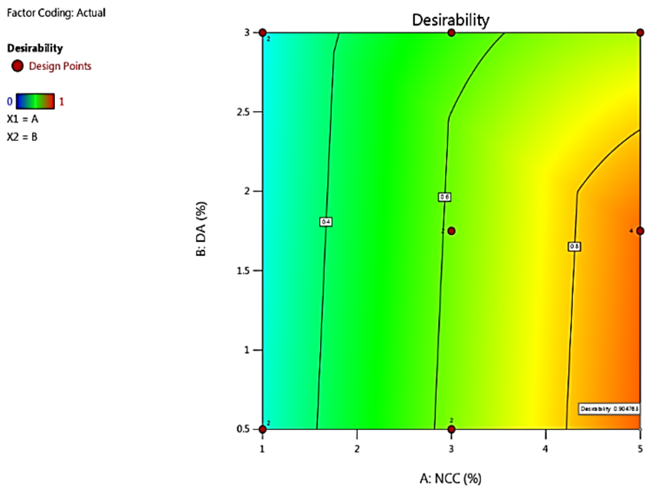

Figure 6. Contour plot of desirability for the optimized bioplastic formulation

Verification of the optimal formulation

Based on the verification of the optimal solution recommended by the Design-Expert 13.0 program, experimental validation produced a tensile strength of 26.67 MPa and a WVTR value of 4.98 g/m^2/day at an NCC concentration of 3% and a dispersing agent concentration of 1.75%. When compared with the predicted values presented in Table 5, which were 27.66 MPa for tensile strength and 5.10 g/m^2/day for WVTR, the verification results fall within the 95% prediction interval (PI) range between the lower and upper limits. This indicates that the predicted model provides reliable results and that the optimized formulation for achieving maximum tensile strength and minimum WVTR is sufficiently consistent (Shapi'i et al., 2025). The optimization results indicate that the incorporation of NCC significantly improves mechanical strength while simultaneously reducing water vapor permeability of the bioplastic films.

Table 5. Verification of optimal formulation

Response	Prediction	Verified	95% PI low	95% PI high
Tensile strength (MPa)	27.66	26.67	19.69	35.63
WVTR (g/m^2/day)	5.10	4.98	4.46	5.75

Conclusion

The optimal formulation in this study was achieved at an NCC concentration of 5% (X_1) and a dispersing agent concentration of 0.5% (X_2), resulting in a tensile strength of 37.612 MPa and a water vapor transmission rate (WVTR) of 3.990 g/m^2/day. Based on the results of this study, future research may focus on optimizing dispersing agents and NCC slurry, as these factors influence the physical characteristics and WVTR of bioplastics during the production process. In addition, further studies may investigate the potential application of this bioplastic for large-scale industrial use in the food packaging sector.

Acknowledgements

The authors would like to express their gratitude to the Human Resources Development Agency for Industry (BPSDMI), Ministry of Industry of the Republic of Indonesia, for the financial support through the SPIRIT Grant Program 2023. The authors also acknowledge Politeknik ATI Padang for supporting the completion of this research.

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