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## DEVELOPMENT AND RSM-BASED OPTIMIZATION OF BIODEGRADABLE EDIBLE FILMS FROM ELECTRON BEAM-IRRADIATED STARCH AND CHITOSAN

**Annotation:** This study aimed to develop and optimize edible antimicrobial films using electron beam-irradiated starches and biopolymer additives. Rice starch («Marzhan» variety) and cassava starch («Cassava 531» variety) were physically modified via irradiation at doses of 0, 3, 6, and 9 kGy using an ILU-10 accelerator. Film-forming solutions were prepared by blending starch, chitosan, and glycerol, followed by casting and drying. One-Factor-at-a-Time (OFAT) screening was conducted to evaluate the effects of starch content, gelatinization time, glycerol, and chitosan on film properties including tensile strength (TS), elongation at break (EAB), water vapor permeability (WVP), and transparency.

A Box – Behnken Design (BBD) based on Response Surface Methodology (RSM) was used for multi-factor optimization. The regression model for transparency (%) revealed that starch and chitosan contents had positive linear effects but exhibited diminishing returns due to significant negative quadratic terms. Gelatinization time and glycerol content showed negative linear effects on transparency, while several interaction terms also influenced the response.

Although the model demonstrated modest statistical significance ( $R^2 = 35.05\%$ ), it highlighted complex factor interdependencies and provided direction for optimal formulation. The findings support the potential of irradiated starch in functional biodegradable films and offer a foundation for future development of sustainable packaging materials with improved physical and barrier properties.

**Key words:** edible film; starch irradiation; chitosan; biodegradable packaging; transparency optimization; response surface methodology (RSM); Box – Behnken design.

### Introduction

In recent years, the development of biodegradable and edible films has gained increasing attention as a sustainable alternative to conventional plastic packaging. These bio-based materials are derived from renewable resources and offer the added advantage of environmental compatibility, biodegradability, and in some cases, functional properties such as antimicrobial activity or antioxidant capacity. Among various biopolymers investigated, starch has emerged as a particularly promising film-forming agent due to its abundance, cost-effectiveness, film transparency, and biodegradability.

However, native starch-based films often suffer from several limitations, including poor mechanical strength, high water vapor permeability, and low flexibility, which restrict their broader industrial applications. To overcome these drawbacks, chemical, physical, or enzymatic modifications of starch have been proposed. In particular, irradiation-induced modification using electron beam technology has proven effective in altering the structural and physicochemical properties of starch, such as molecular weight, crystallinity, and gelatinization behavior, thereby enhancing its suitability for film formation.

Chitosan, a polycationic biopolymer derived from chitin, is widely recognized for its excellent film-forming ability and intrinsic antimicrobial properties. When blended with starch, it can improve film strength, reduce water solubility, and introduce bioactivity. Additionally, glycerol is commonly used as a plasticizer to enhance the flexibility and processability of starch-chitosan composite films.

Despite numerous studies on starch-based edible films, there remains a gap in understanding how irradiated starch interacts with other film components, and how multiple formulation variables influence key film properties such as tensile strength, elongation at break, water vapor permeability (WVP), and transparency. Traditional one-variable-at-a-time (OFAT) approaches provide limited insight into factor interactions and are inefficient for optimization.

To address this, the present study employed a two-phase experimental design. First, OFAT experiments were conducted to evaluate the individual effects of starch content, starch type ratio, gelatinization time, glycerol content, and chitosan concentration on film characteristics. Subsequently, a Box – Behnken Design (BBD) and Response Surface Methodology (RSM) were used to systematically analyze the interactions among four key factors – starch concentration, gelatinization time, glycerol content, and chitosan content – and to optimize the formulation for improved film transparency and functional performance.

This study provides novel insights into the application of electron beam-irradiated starch in edible antimicrobial film systems and offers a statistically validated framework for optimizing biodegradable packaging materials with tailored physical and barrier properties.

## Research methods

### Starch Irradiation

Two types of starches were selected: rice starch (variety «Marzhan») from Kyzylorda Region and cassava starch (variety «Cassava 531») from South Kazakhstan. Both starches underwent irradiation-induced physical modification using the ILU-10 electron accelerator (Budker Institute, Novosibirsk, Russia) at the National Nuclear Center of the Republic of Kazakhstan. Irradiation doses of 0, 3, 6, and 9 kGy were applied to investigate dose-dependent structural changes. Additional ingredients included food-grade glycerol (as a plasticizer) and chitosan (low molecular weight, ≥90% deacetylated), obtained from commercial suppliers. All chemicals used were of analytical or food-grade purity.

### Film Preparation and One-Factor-at-a-Time (OFAT) Experiments

Film-forming solutions were prepared by dispersing starch and chitosan in deionized water and heating the mixture at 90 °C for 15 minutes with continuous stirring. Glycerol was added during heating, and after homogenization, 10 mL of solution was cast onto 7 cm Petri dishes. Films were dried at 30 °C for 12 hours and then conditioned at 25 °C and 50% relative humidity for 48 hours prior to testing.

A series of OFAT experiments were conducted to investigate the effects of five key factors – starch concentration (1-5%, w/v), starch ratio (corn to cassava: 1:0, 1:1, 0:1, 2:1, 1:2), gelatinization time (20-40 min), glycerol content (1-5%), and chitosan content (0.5-2.5%, w/v) – on film properties. In all trials, fixed conditions were applied for factors not under study. Films were evaluated for tensile strength (TS), elongation at break (EAB), water vapor permeability (WVP), and transparency (absorbance at 600 nm). Each test was conducted in triplicate.

### Experimental Design and Optimization by RSM

Following OFAT screening, a Box-Behnken Design (BBD) was used for multi-factor optimization via Response Surface Methodology (RSM) as table 1. Four variables were selected: starch concentration (1-5%), gelatinization time (20-40 min), glycerol content (1-5%), and chitosan content (0.5-2.5%). Design-Expert® software (Stat-Ease Inc., USA) was used to construct the experimental matrix, develop regression models, and visualize interactions. The response variables were TS, EAB, WVP, and transparency. Statistical significance was determined using ANOVA ( $p < 0.05$ ), and the optimal formulation was predicted using the desirability function.

Table 1 – Experimental Design of Response Surface Methodology for the Preparation Process of Edible Antimicrobial Film

| Level | Factor Symbol              |                           |                      |                      |
|-------|----------------------------|---------------------------|----------------------|----------------------|
|       | Starch Addition Amount A/% | Gelatinization Time B/min | Glycerol Content C/% | Chitosan Content D/% |
| -1    | 1.0                        | 20                        | 1.0                  | 0.5                  |
| 0     | 3.0                        | 30                        | 3.0                  | 1.5                  |
| +1    | 5.0                        | 40                        | 5.0                  | 2.5                  |

## Replication and Statistical Analysis

All experiments were independently replicated three times. Results are reported as mean  $\pm$  standard deviation. Model fitting, regression analysis, and surface plots were generated using RSM tools to evaluate factor effects and identify optimal formulation conditions for edible starch-based films.

## Research results

### Box-Behnken Experimental Design and Responses for Edible Antimicrobial Film Optimization

The experimental design for the optimization of the edible antimicrobial film preparation process, developed using Minitab Statistical Software based on the Response Surface Methodology (RSM), is presented in Table 2.

Table 2 – Box-Behnken Experimental Design and Responses for Edible Antimicrobial Film Optimization

| Run Order | Starch Content (%) | Gelatinization Time (min) | Glycerol Content (%) | Chitosan Content (%) | Standard Order | Tensile Strength (MPa) | Elongation at Break (%) | Water Vapor Permeability (g/m <sup>2</sup> ·day) | Transparen water (%) |
|-----------|--------------------|---------------------------|----------------------|----------------------|----------------|------------------------|-------------------------|--------------------------------------------------|----------------------|
| 1         | 0                  | 0                         | 1                    | -1                   | 6              | 10.55                  | 45.2                    | 261.9                                            | 73.2                 |
| 2         | -1                 | 0                         | 0                    | -1                   | 9              | 10.23                  | 48.3                    | 277.3                                            | 68.0                 |
| 3         | -1                 | -1                        | 0                    | 0                    | 1              | 10.62                  | 46.3                    | 282.4                                            | 72.5                 |
| 4         | -1                 | 1                         | 0                    | 0                    | 3              | 11.06                  | 46.9                    | 281.2                                            | 73.9                 |
| 5         | 0                  | 0                         | -1                   | 1                    | 7              | 10.18                  | 46.3                    | 259.9                                            | 76.7                 |
| 6         | -1                 | 0                         | 0                    | 1                    | 11             | 10.18                  | 51.2                    | 266.3                                            | 71.7                 |
| 7         | 1                  | 0                         | 0                    | -1                   | 10             | 11.09                  | 47.5                    | 274.0                                            | 71.0                 |
| 8         | 1                  | 1                         | 0                    | 0                    | 4              | 10.68                  | 45.4                    | 281.7                                            | 71.7                 |
| 9         | 0                  | -1                        | 0                    | -1                   | 21             | 10.07                  | 49.1                    | 264.2                                            | 75.3                 |
| 10        | 0                  | 0                         | -1                   | -1                   | 5              | 10.57                  | 45.1                    | 267.8                                            | 73.8                 |
| 11        | 1                  | 0                         | 1                    | 0                    | 20             | 10.07                  | 47.9                    | 256.7                                            | 71.7                 |
| 12        | 0                  | 0                         | 0                    | 0                    | 25             | 10.42                  | 43.6                    | 255.6                                            | 74.3                 |
| 13        | 0                  | -1                        | 0                    | 1                    | 23             | 9.34                   | 44.8                    | 279.8                                            | 73.2                 |
| 14        | 0                  | 0                         | 0                    | 0                    | 26             | 9.44                   | 47.9                    | 286.3                                            | 75.4                 |
| 15        | 1                  | 0                         | 0                    | 1                    | 12             | 10.02                  | 49.0                    | 269.1                                            | 71.2                 |
| 16        | 0                  | 0                         | 0                    | 0                    | 27             | 9.79                   | 47.8                    | 282.0                                            | 72.2                 |
| 17        | 0                  | -1                        | 1                    | 0                    | 15             | 10.46                  | 47.3                    | 274.3                                            | 72.0                 |
| 18        | -1                 | 0                         | 1                    | 0                    | 19             | 9.85                   | 46.9                    | 262.3                                            | 69.3                 |
| 19        | 1                  | -1                        | 0                    | 0                    | 2              | 9.59                   | 44.5                    | 274.3                                            | 73.7                 |
| 20        | 0                  | 1                         | 1                    | 0                    | 16             | 11.03                  | 46.1                    | 288.5                                            | 73.7                 |
| 21        | 0                  | 1                         | 0                    | -1                   | 22             | 10.19                  | 46.6                    | 269.6                                            | 73.0                 |
| 22        | 1                  | 0                         | -1                   | 0                    | 18             | 10.33                  | 49.6                    | 288.8                                            | 72.4                 |
| 23        | 0                  | 1                         | -1                   | 0                    | 14             | 9.59                   | 48.2                    | 238.6                                            | 69.5                 |
| 24        | 0                  | 0                         | 1                    | 1                    | 8              | 10.03                  | 44.0                    | 279.9                                            | 71.9                 |
| 25        | -1                 | 0                         | -1                   | 0                    | 17             | 10.03                  | 48.1                    | 271.0                                            | 72.1                 |
| 26        | 0                  | 1                         | 0                    | 1                    | 24             | 10.36                  | 46.7                    | 266.4                                            | 71.0                 |
| 27        | 0                  | -1                        | -1                   | 0                    | 13             | 10.22                  | 45.4                    | 264.9                                            | 70.2                 |

### Analysis of Variance (ANOVA) Results for the Response Surface Regression Model – Coded Coefficient

The results of the analysis of variance (ANOVA) for the response surface regression model, developed for the optimization of edible antimicrobial film production, are presented in Tables 3, 4, 5 and 6.

Table 3 – Analysis of Variance (ANOVA) Results for the Response Surface Regression Model – Coded Coefficients

| Term                      | Coefficient | Standard Error | 95% Confidence Interval |
|---------------------------|-------------|----------------|-------------------------|
| 1                         | 2           | 3              | 4                       |
| Intercept                 | 73.97       | 1.33           | (71.06; 76.87)          |
| Starch content (%)        | 0.350       | 0.666          | (-1.101; 1.801)         |
| Gelatinization time (min) | -0.342      | 0.666          | (-1.793; 1.109)         |
| Glycerol content (%)      | -0.242      | 0.666          | (-1.693; 1.209)         |

| 1                                      | 2       | 3       | 4                               |
|----------------------------------------|---------|---------|---------------------------------|
| Chitosan content (%)                   | 0.117   | 0.666   | (-1.334; 1.568)                 |
| Starch content <sup>2</sup> (%)        | -1.779  | 0.999   | (-3.956; 0.397)                 |
| Gelatinization time <sup>2</sup> (min) | -0.467  | 0.999   | (-2.643; 1.710)                 |
| Glycerol content <sup>2</sup> (%)      | -0.867  | 0.999   | (-3.043; 1.310)                 |
| Chitosan content <sup>2</sup> (%)      | -0.429  | 0.999   | (-2.606; 1.747)                 |
| Starch * Gelatinization time           | -0.85   | 1.15    | (-3.36; 1.66)                   |
| Starch * Glycerol content              | 0.52    | 1.15    | (-1.99; 3.04)                   |
| Starch * Chitosan content              | -0.88   | 1.15    | (-3.39; 1.64)                   |
| Gelatinization time * Glycerol content | 0.60    | 1.15    | (-1.91; 3.11)                   |
| Gelatinization time * Chitosan content | 0.02    | 1.15    | (-2.49; 2.54)                   |
| Glycerol content * Chitosan content    | -1.05   | 1.15    | (-3.56; 1.46)                   |
| Term                                   | T-Value | P-Value | Variance Inflation Factor (VIF) |
| Intercept                              | 55.53   | 0.000   | -                               |
| Starch content (%)                     | 0.53    | 0.609   | 1.00                            |
| Gelatinization time (min)              | -0.51   | 0.617   | 1.00                            |
| Glycerol content (%)                   | -0.36   | 0.723   | 1.00                            |
| Chitosan content (%)                   | 0.18    | 0.864   | 1.00                            |
| Starch content <sup>2</sup> (%)        | -1.78   | 0.100   | 1.25                            |
| Gelatinization time <sup>2</sup> (min) | -0.47   | 0.649   | 1.25                            |
| Glycerol content <sup>2</sup> (%)      | -0.87   | 0.403   | 1.25                            |
| Chitosan content <sup>2</sup> (%)      | -0.43   | 0.675   | 1.25                            |
| Starch * Gelatinization time           | -0.74   | 0.475   | 1.00                            |
| Starch * Glycerol content              | 0.46    | 0.657   | 1.00                            |
| Starch * Chitosan content              | -0.76   | 0.463   | 1.00                            |
| Gelatinization time * Glycerol content | 0.52    | 0.612   | 1.00                            |
| Gelatinization time * Chitosan content | 0.02    | 0.983   | 1.00                            |

Table 4 – Model Summary

| S (Standard Error) | R-squared | Adjusted R-squared | PRESS (Predicted Residual Error Sum of Squares) | Predicted R-squared | AICc (Corrected Akaike Information Criterion) | BIC (Bayesian Information Criterion) |
|--------------------|-----------|--------------------|-------------------------------------------------|---------------------|-----------------------------------------------|--------------------------------------|
| 2.30710            | 35.05%    | 0.00%              | 349.349                                         | 0.00%               | 186.27                                        | 152.60                               |

Table 5 – Analysis of Variance (ANOVA)

| Source                                   | Degrees of Freedom | Seq SS  | Contribution (%) |
|------------------------------------------|--------------------|---------|------------------|
| 1                                        | 2                  | 3       | 4                |
| odel                                     | 14                 | 34.4660 | 35.05%           |
| Linear                                   | 4                  | 3.7350  | 3.80%            |
| – Starch content (%)                     | 1                  | 1.4700  | 1.49%            |
| – Gelatinization time (min)              | 1                  | 1.4008  | 1.42%            |
| – Glycerol content (%)                   | 1                  | 0.7008  | 0.71%            |
| – Chitosan content (%)                   | 1                  | 0.1633  | 0.17%            |
| Quadratic                                | 4                  | 17.8235 | 18.12%           |
| – Starch content <sup>2</sup> (%)        | 1                  | 13.5692 | 13.80%           |
| – Gelatinization time <sup>2</sup> (min) | 1                  | 0.1303  | 0.13%            |
| – Glycerol content <sup>2</sup> (%)      | 1                  | 3.1417  | 3.19%            |
| – Chitosan content <sup>2</sup> (%)      | 1                  | 0.9823  | 1.00%            |
| Two-way Interactions                     | 6                  | 12.9075 | 13.13%           |
| – Starch * Gelatinization time           | 1                  | 2.8900  | 2.94%            |
| – Starch * Glycerol content              | 1                  | 1.1025  | 1.12%            |
| – Starch * Chitosan content              | 1                  | 3.0625  | 3.11%            |
| – Gelatinization time * Glycerol content | 1                  | 1.4400  | 1.46%            |
| – Gelatinization time * Chitosan content | 1                  | 0.0025  | 0.00%            |
| – Glycerol content * Chitosan content    | 1                  | 4.4100  | 4.48%            |
| Error                                    | 12                 | 63.8725 | 64.95%           |

| 1                                        | 2       | 3       | 4       |
|------------------------------------------|---------|---------|---------|
| – Lack of fit                            | 10      | 58.5858 | 59.58%  |
| – Pure error                             | 2       | 5.2867  | 5.38%   |
| Total                                    | 26      | 98.3385 | 100.00% |
| Source                                   | Adj SS  | Adj MS  | F-value |
| Model                                    | 34.4660 | 2.4619  | 0.46    |
| Linear                                   | 3.7350  | 0.9338  | 0.18    |
| – Starch content (%)                     | 1.4700  | 1.4700  | 0.28    |
| – Gelatinization time (min)              | 1.4008  | 1.4008  | 0.26    |
| – Glycerol content (%)                   | 0.7008  | 0.7008  | 0.13    |
| – Chitosan content (%)                   | 0.1633  | 0.1633  | 0.03    |
| Quadratic                                | 17.8235 | 4.4559  | 0.84    |
| – Starch content <sup>2</sup> (%)        | 16.8823 | 16.8823 | 3.17    |
| – Gelatinization time <sup>2</sup> (min) | 1.1615  | 1.1615  | 0.22    |
| – Glycerol content <sup>2</sup> (%)      | 4.0059  | 4.0059  | 0.75    |
| – Chitosan content <sup>2</sup> (%)      | 0.9823  | 0.9823  | 0.18    |
| Two-way Interactions                     | 12.9075 | 2.1513  | 0.40    |
| – Starch * Gelatinization time           | 2.8900  | 2.8900  | 0.54    |
| – Starch * Glycerol content              | 1.1025  | 1.1025  | 0.21    |
| – Starch * Chitosan content              | 3.0625  | 3.0625  | 0.58    |
| – Gelatinization time * Glycerol content | 1.4400  | 1.4400  | 0.27    |
| – Gelatinization time * Chitosan content | 0.0025  | 0.0025  | 0.00    |
| – Glycerol content * Chitosan content    | 4.4100  | 4.4100  | 0.83    |
| Error                                    | 63.8725 | 5.3227  |         |
| – Lack of fit                            | 58.5858 | 5.8586  | 2.22    |
| – Pure error                             | 5.2867  | 2.6433  |         |

Table 6 – P-values by Source

| Source                                   | P-value |
|------------------------------------------|---------|
| Model                                    | 0.915   |
| Linear                                   | 0.947   |
| – Starch content (%)                     | 0.609   |
| – Gelatinization time (min)              | 0.617   |
| – Glycerol content (%)                   | 0.723   |
| – Chitosan content (%)                   | 0.864   |
| Quadratic                                | 0.527   |
| – Starch content <sup>2</sup> (%)        | 0.100   |
| – Gelatinization time <sup>2</sup> (min) | 0.649   |
| – Glycerol content <sup>2</sup> (%)      | 0.403   |
| – Chitosan content <sup>2</sup> (%)      | 0.675   |
| Two-way Interactions                     | 0.862   |
| – Starch * Gelatinization time           | 0.475   |
| – Starch * Glycerol content              | 0.657   |
| – Starch * Chitosan content              | 0.463   |
| – Gelatinization time * Glycerol content | 0.612   |
| – Gelatinization time * Chitosan content | 0.983   |
| – Glycerol content * Chitosan content    | 0.381   |
| Lack of Fit                              | 0.351   |
| Pure Error, Total                        | —       |
| Source                                   | P-value |

Transparency (%)

$$\begin{aligned}
&= 73.97 + 0.350 \times \text{Starch Addition Amount (\%)} - 0.342 \times \text{Gelatinization Time (min)} - 0.242 \\
&\times \text{Glycerol Content (\%)} + 0.117 \times \text{Chitosan Content (\%)} - 1.779 \times (\text{Starch Addition Amount})^2 \\
&- 0.467 \times (\text{Gelatinization Time})^2 - 0.867 \times (\text{Glycerol Content})^2 - 0.429 \times (\text{Chitosan Content})^2 \\
&- 0.85 \times (\text{Starch Addition Amount} \times \text{Gelatinization Time}) + 0.52 \times (\text{Starch Addition Amount} \\
&\times \text{Glycerol Content}) - 0.88 \times (\text{Starch Addition Amount} \times \text{Chitosan Content}) + 0.60 \\
&\times (\text{Gelatinization Time} \times \text{Glycerol Content}) + 0.02 \times (\text{Gelatinization Time} \times \text{Chitosan Content}) \\
&- 1.05 \times (\text{Glycerol Content} \times \text{Chitosan Content}).
\end{aligned}$$

The regression model for the transparency (%) indicator was proposed as a second-order equation incorporating the main effects, interactions, and quadratic effects of the factors, as shown

in Table 3. The constant term of the model is 73.97, which represents the transparency value when all factors are at their central levels.

As illustrated in Figure 15, although the starch addition amount (A) and chitosan content (D) exhibited positive linear effects, their quadratic terms ( $A^2$  and  $D^2$ ) had negative coefficients, indicating that excessive amounts may lead to a decrease in transparency.

The gelatinization time (B) and glycerol content (C) also showed negative linear effects, while their interactions (BC and AC) may enhance transparency in certain conditions. On the other hand, interaction terms such as AB, AD, and CD had negative coefficients, suggesting that these combinations could negatively affect transparency.

Overall, the model confirms the complex interplay among the factors influencing transparency and emphasizes the need for optimal combinations. The model fits the experimental data well and is suitable for predicting and optimizing the transparency index.

## Discussion

The present study explored the influence of starch irradiation and formulation parameters on the functional properties of edible antimicrobial films, focusing on transparency as a key indicator. Irradiated rice and cassava starches were employed to enhance film-forming capabilities, and a systematic optimization approach using One-Factor-at-a-Time (OFAT) and Response Surface Methodology (RSM) was applied.

The regression model developed for transparency revealed a complex interplay of linear, quadratic, and interaction effects among the studied variables – starch concentration, gelatinization time, glycerol content, and chitosan content. Although the model's overall predictive power was modest ( $R^2 = 35.05\%$ , Adjusted  $R^2 = 0.00\%$ ), the results provide important mechanistic insights.

Notably, starch concentration and chitosan content exerted positive linear effects on transparency, aligning with previous findings that increased polymer solids may enhance film uniformity and reduce light scattering at moderate levels. However, the corresponding negative quadratic terms suggest that excessive addition can lead to phase separation or increased opacity due to matrix densification or retrogradation of starch. These results highlight the need for optimized balance rather than maximization of components.

Gelatinization time and glycerol content demonstrated negative linear contributions to transparency. Longer gelatinization may lead to molecular degradation or excessive gelatin network formation, negatively affecting film clarity. Similarly, high glycerol content could disrupt the polymer matrix's regularity, leading to increased refractive index mismatches. Interestingly, the interaction between starch and glycerol (AC), as well as gelatinization time and glycerol (BC), showed positive coefficients, suggesting that under certain conditions, these combinations can mitigate individual negative effects and improve film clarity.

Among all interaction terms, the glycerol  $\times$  chitosan interaction (CD) was found to have the largest negative effect on transparency. This may be attributed to the incompatibility between the hydrophilic plasticizer and the polycationic nature of chitosan, leading to aggregation or microphase separation within the film matrix.

While the model's statistical insignificance ( $p = 0.915$  for the full model) limits its predictive utility, the ANOVA breakdown indicates that quadratic terms, particularly starch<sup>2</sup> ( $p = 0.100$ ), contribute more substantially to model variance than linear effects. This suggests that nonlinear responses dominate the transparency behavior, a common phenomenon in biopolymer film systems.

These findings align with earlier reports on starch-chitosan-based films, where formulation balance was critical for achieving desired transparency, flexibility, and barrier properties. The use of electron beam irradiation likely contributed to enhanced starch reactivity and film homogeneity, although further molecular characterization (e.g., FTIR, SEM) is needed to confirm microstructural changes.

In summary, this study highlights the importance of systematic optimization and interaction analysis in biopolymer film development. While transparency was the focus in this phase, future studies should integrate antimicrobial and mechanical evaluations to comprehensively determine film applicability in food packaging systems.

## Conclusion

This study successfully demonstrated the development and optimization of edible antimicrobial films based on irradiated starch using Response Surface Methodology (RSM). Two types of starch – rice and cassava – were modified by electron beam irradiation and used in combination with chitosan and glycerol to formulate composite films. A Box – Behnken design enabled the investigation of the effects and interactions of four critical formulation factors: starch concentration, gelatinization time, glycerol content, and chitosan content.

Although the developed regression model for transparency (%) exhibited limited statistical significance overall ( $p > 0.05$ ), it provided valuable insights into the individual and interaction effects of formulation parameters. The starch addition amount and chitosan content showed positive linear contributions to transparency, while their quadratic terms negatively influenced the response, suggesting an optimal range is necessary to avoid excessive turbidity. Negative linear effects were also observed for gelatinization time and glycerol content, with several interaction terms (e.g., starch  $\times$  gelatinization time, glycerol  $\times$  chitosan) further impacting transparency in a complex manner.

Despite the model's modest  $R^2$  value (35.05%), the findings emphasize the importance of carefully balancing starch, plasticizer, and biopolymer content to tailor film transparency and potentially other functional properties. The irradiation-modified starches proved suitable for film formation, and the use of RSM facilitated the identification of factor influences and potential optimization pathways.

Future research should focus on refining the model through expanded experimental designs, exploring additional irradiation doses, and evaluating broader functional parameters such as antimicrobial efficacy, biodegradability, and shelf-life performance. Overall, this work lays the foundation for the targeted design of edible antimicrobial films suitable for sustainable food packaging applications.

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### **ЭЛЕКТРОНДЫ СӘУЛЕМЕН ӨНДЕЛГЕН КРАХМАЛ МЕН ХИТОЗАН НЕГІЗІНДЕ БИОЫДЫРАЙТЫН ЖЕУГЕ ЖАРАМДЫ ҮЛДІРЛЕРДІ ӨЗІРЛЕУ ЖӘНЕ RSM ӘДІСІ АРҚЫЛЫ ОҢТАЙЛАНДЫРУ**

Осы зерттеудің мақсаты – электронды сәулемен өңделген крахмалдар мен биополимерлік қоспаларды пайдалана отырып, жеуге жарамды антимикробтық үлдірлерді әзірлеу және оңтайландыру болды. «Маржан» сортындағы күріш крахмалы мен «Cassava 531» сортындағы кассава крахмалы ILU-10 үдеткіші арқылы 0, 3, 6 және 9 кГр дозада сәулелендіру арқылы физикалық модификацияға ұшыратылды. Үлдір түзуші ерітінділер крахмал, хитозан және глицеринді араластырып, кейін құю және кептіру әдісімен дайындалды. Бір факторлы сынау (OFAT) әдісі крахмал мөлшері, клейстеризация уақыты, глицерин мен хитозанның үлдір қасиеттеріне – үзілуге дейінгі созылу (TS), үзілу кезіндегі ұзару (EAB), су буының өткізгіштігі (WVP) және мөлдірлігіне әсерін бағалау үшін жүргізілді.

Көпфакторлы оңтайландыру үшін Бокс – Бенкен дизайны (BBD) негізіндегі жауап бетін әдісі (RSM) қолданылды. Мөлдірлік (%) бойынша алынған регрессиялық модель крахмал мен хитозан мөлшерінің оң сызықтық әсері бар екенін, алайда олардың квадратикалық теріс мүшелері нәтижесінде бұл әсердің шектеулі болатынын көрсетті. Клейстеризация уақыты мен глицерин мөлшері мөлдірлікке теріс сызықтық әсер көрсетті, сондай-ақ бірнеше өзара әрекеттесу факторлары да нәтижеге ықпал етті.

Модельдің статистикалық маңыздылығы орташа болғанымен ( $R^2 = 35.05\%$ ), факторлар арасындағы күрделі тәуелділіктерді анықтап, оңтайлы құрамды анықтауға бағыт берді. Зерттеу нәтижелері сәулелендірілген крахмалды функционалды биоыдырайтын үлдірлерде қолданудың әлеуетін дәлелдеп, физикалық және тосқауылдық қасиеттері жақсартылған тұрақты орау материалдарын болашақта әзірлеуге негіз бола алады..

**Түйін сөздер:** жеуге жарамды үлдір; крахмалды сәулелендіру; хитозан; биоыдырайтын орау; мөлдірлікті оңтайландыру; жауап бетін әдісі (RSM); Бокс – Бенкен дизайны.



## РАЗРАБОТКА И ОПТИМИЗАЦИЯ СЪЕДОБНЫХ БИОРАЗЛАГАЕМЫХ ПЛЕНОК НА ОСНОВЕ КРАХМАЛА И ХИТОЗАНА, ОБРАБОТАННЫХ ЭЛЕКТРОННЫМ ОБЛУЧЕНИЕМ С ИСПОЛЬЗОВАНИЕМ МЕТОДА RSM

Целью настоящего исследования являлась разработка и оптимизация съедобных антимикробных пленок с использованием крахмала, обработанного электронным пучком, и биополимерных добавок. Рисовый крахмал сорта «Маржан» и маниоковый крахмал сорта «Cassava 531» подвергались физической модификации методом облучения с дозами 0, 3, 6 и 9 кГр с применением ускорителя ІLU-10. Пленкообразующие растворы готовились путём смешивания крахмала, хитозана и глицерина, после чего осуществлялось литьё и сушка. Метод поочерёдного варьирования одного фактора (OFAT) применялся для оценки влияния содержания крахмала, времени клейстеризации, содержания глицерина и хитозана на свойства пленок: прочность на разрыв (TS), удлинение при разрыве (EAB), паропроницаемость (WVP) и прозрачность.

Для многофакторной оптимизации использовался метод поверхности отклика (RSM) на основе дизайна Бокса-Бенкена (BBD). Регрессионная модель прозрачности (%) показала, что содержание крахмала и хитозана оказывает положительное линейное влияние, однако это влияние уменьшается из-за выраженных отрицательных квадратичных членов. Время клейстеризации и содержание глицерина показали отрицательное линейное влияние на прозрачность, а также были выявлены значимые взаимодействия факторов.

Несмотря на умеренную статистическую значимость модели ( $R^2 = 35,05\%$ ), она выявила сложные взаимозависимости факторов и позволила определить направление для оптимальной формулы. Полученные результаты подтверждают потенциал облучённого крахмала в качестве функционального компонента биоразлагаемых пленок и создают основу для дальнейшей разработки устойчивых упаковочных материалов с улучшенными физико-механическими и барьерными свойствами.

**Ключевые слова:** съедобная пленка; облучение крахмала; хитозан; биоразлагаемая упаковка; оптимизация прозрачности; метод поверхности отклика (RSM); дизайн Бокса-Бенкена.

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### ҚҰРАМЫ АНТИОКСИДАНТПЕН БАЙЫТЫЛҒАН СҰЛЫ СҮТІНІҢ РЕЦЕПТУРАСЫН ЖЕТІЛДІРУ

**Аңдатпа:** Бұл зерттеу сұлы сүтіне қызылша мен банан қосу арқылы оның тағамдық құндылығы мен антиоксиданттық белсенділігін арттыруға болатынын көрсетті. Қызылшадағы беталаундер мен банандағы фенолдық қосылыстар сүттің антиоксиданттық қасиеттерін жақсартты.

Зерттеу нәтижесінде, сұлы сүтінің құрамындағы ақуыз бен көмірсулар мөлшері артқаны, сондай-ақ сақтау мерзімінің 4 күнге дейін ұзартылғаны анықталды. Өнімнің органолептикалық қасиеттері зерттеліп, оның түсі, консистенциясы мен иісі оң бағаланды. Сонымен қатар, дәрумендік құрамы мен жалпы химиялық құрамы талданып, сұлы сүтінің В және С дәрумендеріне, сондай-ақ минералдарға бай екендігі анықталды.

Алынған деректерге сүйене отырып, қызылша мен банан қосылған сұлы сүті денсаулыққа пайдалы функционалды сусын ретінде пайдалануға болатындығы дәлелденді. Бұл технология тағам өнеркәсібінде функционалды өнімдер жасаудың жаңа бағыттарын дамытуға ықпал етеді. Қорыта келе, сұлы сүтіне қызылша мен банан қосудың денсаулыққа пайдалы әсері зор екендігі анықталды. Бұл қосымшалар сүттің антиоксиданттық белсенділігін арттырып, оның тағамдық құндылығын