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Development of a Biodegradable Starch-Based Film Material for Extending the Shelf Life of Food Products

For the dissertation submitted for the degree of Doctor of Philosophy (PhD)
in the specialty 8D07202 – "Food Safety"

ABSTRACT

This dissertation is devoted to the development of an edible, biodegradable starch-based film material with antibacterial properties aimed at extending the shelf life of food products. The study involved a comprehensive investigation of the physicochemical, morphological, and structural properties of natural starch sources cultivated in Kazakhstan (wheat, potato, corn, rice, pea, and cassava) and assessed their suitability for use as biopolymer packaging materials. Edible films with antibacterial and antioxidant activity were prepared using modified starch, chitosan, and glycerol, and their physical, mechanical, barrier, and microbiological properties were investigated.

Relevance of the Study

The current environmental situation and growing food safety challenges emphasize the need for developing biopolymer-based packaging materials. More than 400 million tons of plastics are produced annually worldwide, with 30–40% used for single-use packaging. About 90% of these wastes are not recycled and persist in the environment for centuries. Microplastic particles entering the human body through food, water, and air contribute to chronic diseases (endocrine, neurological, and digestive disorders).

The Concept for the Development of the Agro-Industrial Complex of the Republic of Kazakhstan for 2021–2030 and the Addresses of President Kassym-Jomart Tokayev identify strategic priorities such as ensuring food security, developing deep processing, and supporting environmentally friendly production. Therefore, the creation of eco-safe packaging materials that preserve product quality is an urgent scientific and practical task consistent with Kazakhstan's policy of integrating science and industry.

International standards (EU Regulation No. 1935/2004, TR CU 021/2011, ISO, GOST) impose strict requirements on food-contact materials, including permissible migration levels, absence of toxic residues, microbiological safety, and complete biodegradability. The development of an edible film that inhibits bacterial growth and is suitable for direct food contact represents a significant scientific and practical contribution at the intersection of food safety and environmental sustainability.

Purpose of the Study

To develop an edible, biodegradable starch-based film with antibacterial properties that meets food-contact safety standards and to conduct a comprehensive investigation of its properties.

Research Objectives

1. To perform a comparative analysis of the physicochemical and morphological properties of starches obtained from various Kazakhstani crops (wheat, potato, pea, rice, corn, cassava) to identify the most suitable sources for biopolymer films.
2. To study the effectiveness of dry heat modification for improving starch

structure and its film-forming ability.

3. To develop the formulation and production technology of edible antibacterial films by incorporating natural functional additives that suppress pathogenic microflora and extend product shelf life.

4. To examine the physicochemical, structural–mechanical, and microbiological properties of the films and assess their suitability for food industry applications.

5. To comprehensively evaluate the food safety of the films based on HACCP principles by determining total and specific migration, heavy metals and pesticide residues, microbiological purity, and toxicity.

6. To develop normative and technical documentation (organizational standard and technological instruction) for the edible antibacterial film in accordance with TR CU 021/2011 and conduct industrial testing.

Research Objects

Starches from wheat, potato, corn, rice, pea, and cassava cultivated in various regions of Kazakhstan. To improve structural properties, an eco-friendly dry heat modification method was applied. Based on the modified starches, biodegradable edible films containing chitosan and glycerol were obtained. Antibacterial activity was tested against model microorganisms E. coli and S. aureus. Safety assessment included migration into food simulants, determination of heavy metals and pesticides, and the films' effect on the shelf life of raw and cooked meat and fish.

Research Methods

Physicochemical properties (moisture, ash, protein, transparency, swelling, gelatinization temperature) were determined by standard methods (AACC, ISO 6869, ISO 6491). Structural and morphological analyses were performed by FT-IR, SEM, XRD, and AFM. Mechanical and barrier properties were measured according to ASTM E96 and GOST 12302. Antibacterial activity was determined against E. coli and S. aureus, and food safety was assessed in accordance with TR CU 029/2012.

Scientific Novelty

For the first time, a comparative study of physicochemical, morphological, and mineral characteristics of corn and cassava starches cultivated in Kazakhstan was conducted, revealing their high suitability for film formation. Structural and functional changes of starches during dry heat modification were established, confirming improvements in thermal stability, solubility, and film-forming ability. A novel technology for producing edible antibacterial films based on modified corn and cassava starches with improved mechanical and barrier properties was developed. The food safety and compliance of the films with national and international standards were experimentally confirmed.

Main Provisions for Defense

Comparative analysis of starches from different Kazakhstani crops (wheat, potato, pea, rice, corn, cassava) identified differences in their physicochemical and morphological properties and determined their film-forming suitability.

The efficiency of dry heat modification was proven through improved structural and functional characteristics of starch, ensuring film formation.

Modified starch-based films exhibited pronounced antibacterial activity against E. coli and S. aureus and satisfactory mechanical and barrier properties.

A technology for producing edible antibacterial films based on thermo-modified cassava and corn starches with enhanced structural, technological, and antioxidant

properties was developed and validated.

Practical Significance

The obtained results led to the creation of a new generation of edible biodegradable films suitable for packaging semi-finished products (meat steaks, fish fillets, etc.), effectively suppressing microbial growth and extending shelf life. The films are completely safe for humans, biodegradable under natural conditions, and aligned with sustainable development principles. They can also be used for thermal cooking directly in the packaging, preserving organoleptic quality.

The developed solutions are suitable for industrial implementation at enterprises producing eco-safe packaging materials. The results can be applied in higher education, research, and innovation programs.

The technology was tested at LLP KazEcoSoil, where normative–technical documentation was developed and approved (ST LLP 210940019323-001-2024). Technical novelty is protected by Patent of the Republic of Kazakhstan No. 7307 (Utility Model).

Author's Contribution

The author independently conducted theoretical and experimental studies, data processing, industrial testing, preparation of normative documentation, and implementation of results.

Approbation of the Work

The research results were presented at:

– The SCO Youth Scientific and Technological Innovation Forum "Cooperation in Poverty Reduction and Rural Development" (Xi'an, PRC, 2022);

– The International Conference "Seifullin Readings-21: Organic and Regenerative Agriculture – Global Challenges and Local Solutions" (S. Seifullin Kazakh Agrotechnical University, 2025).

Publications

A total of 18 scientific works have been published on the topic, including 2 articles in journals recommended by the Committee for Quality Assurance of the Ministry of Education and Science of Kazakhstan, 16 in Web of Science and Scopus-indexed journals (non-zero impact factor), 2 in international conference proceedings, and 1 patent of the Republic of Kazakhstan.

Structure and Volume

The dissertation consists of a table of contents, introduction, literature review, research methods, results and discussion, conclusion, and appendices. The work comprises 179 pages, 34 tables, 31 figures, and 14 formulas. The reference list includes 131 sources. Evaluation of Objective Fulfillment

The research results fully meet the set goal and objectives:

1. Determined physicochemical parameters of starches (amylose/amylopectin ratio 17.9–28.6%, transparency 54–90%, water and oil absorption 0.65–0.84 and 0.59–0.76 g/g). Cassava and potato starches showed the highest transparency (88–90%) and film-forming ability. SEM analysis revealed oval, smooth-surfaced cassava granules.

2. During dry heat modification (168 °C, 18 h), changes in structural, thermal (DSC, TGA), molecular (RVA, Mw), and morphological (SEM, XRD) characteristics were confirmed, improving film strength and barrier properties.

3. Optimized film formulation (starch 5%, gelatinization time 28.2 min, glycerol 2.8%, chitosan 1.4%) provided balanced performance. Antioxidant activity:

DPPH – 26.6%, ABTS – 22.8%; antibacterial efficiency: E. coli – 47%, S. aureus – 42%.

4. CLSM and AFM analyses revealed a dense, uniform surface. Transparency – 88–90%; tensile strength – 14.2 ± 0.5 MPa; elongation – 31.6 ± 2.4%; WVP – 3.8×10^{-10} $g \cdot m^{-1} \cdot s^{-1} \cdot Pa^{-1}$. Meat and fish packaging reduced microbial contamination by 1.3–2.2 log CFU, weight loss by 6–8%, maintained color and appearance, and extended shelf life by ~36 hours.

5. Migration values complied with TR CU 021/2011 and 029/2012: 1.87 mg/dm² (10% ethanol), 2.13 mg/dm² (3% acetic acid), 2.92 mg/dm² (50% ethanol). Heavy metals: Pb 0.021 mg/kg, Cd 0.004 mg/kg, Hg not detected. Microbiology: TBC 3.1×10^2 CFU/g, yeasts and molds 8.5×10^1 CFU/g, E. coli, S. aureus, Salmonella, L. monocytogenes absent. Radioactivity: Cs-137 3.4 Bq/kg, Sr-90 2.7 Bq/kg; toxicity (Daphnia magna test) – 0% mortality. Films were recognized as safe and compliant with HACCP principles.

6. The technology was tested at LLP KazEcoSoil. Normative and technical documentation (ST LLP 210940019323-001-2024) and a technological instruction were developed and approved. The film is ready for industrial implementation and fully meets food safety standards.