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SYNTHESIS AND CHARACTERIZATION OF THERMORESPONSIVE COMPOSITE PATCHES BASED ON GELLAN GUM, CHITOSAN, AGAR, AND POLY(2-OXAZOLINE) DERIVATIVES

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Abstract: Biodegradable polymer composites were developed using gellan gum, agar–agar, and chitosan as natural matrices, with poly(2-ethyl-2-oxazoline) (PEOZ, 0.15 g) added as a functional modifier. Four film types (GG–Ch, GG–Ch–Ox, A–Ch, A–Ch–Ox) were evaluated for structural and physicochemical performance. The films showed thicknesses of 0.1648–0.3455 mm and masses of 3.70–5.68 g. Incorporation of PEOZ significantly improved folding endurance, increasing it from 68 to 90 cycles in gellan–chitosan and from 42 to 76 cycles in agar–chitosan composites. Swelling and sol–gel analyses revealed higher crosslinking density and lower soluble fraction in modified samples. FTIR spectra confirmed hydrogen bonding and electrostatic interactions, while SEM-EDS of GG–Ch–Ox demonstrated a homogeneous surface with Na^+/Cl^- distribution, indicating efficient ionic crosslinking. The modified systems exhibit improved mechanical stability, water retention, and morphological uniformity, making them promising candidates for biomedical uses such as wound dressings and transdermal patches.

Key words: gellan gum, chitosan, agar-agar, oxazolines, patch, polymer composites, heat-sensitive materials.

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1. Introduction

In modern materials chemistry, creating polymer composites with structural stability and predictable physicochemical characteristics remains a key focus. For applications such as model investigations of diffusion and structural adaptability in polymer matrices, these materials allow tuning of mechanical strength, water-retention capacity, and permeability [1].

This study evaluated several natural polysaccharides as the basis for composite patches, including agar, gellan gum, sodium alginate, and chitosan. Although sodium alginate is biocompatible and gel-forming, it was excluded from further tests due to its low mechanical strength and high water absorption, which limit its suitability for stable polymer networks [2-3].

Gellan gum was selected as a primary structural matrix due to its excellent stability, strength, and ability to form durable gels [4,5]. When combined with other polymers, it provides a robust framework and allows fine-tuning of material properties. Chitosan was incorporated into the composites to enhance moisture retention, strengthen the network, and introduce additional intermolecular interactions [6,7]. Agar was used as a flexible, water-binding component that improves elasticity and influences molecular diffusion within the matrix [8,9].

In this work, two types of composite patches were prepared: gellan gum–chitosan and agar–chitosan, with oxazoline-based polymers incorporated into both formulations to impart thermoresponsive properties. This design allows a direct comparison of how the choice of polysaccharide affects mechanical, sorption, and structural characteristics, while the oxazoline derivatives provide temperature-dependent control over patch performance. Compared to previously reported chitosan-based polysaccharide systems, such as alginate–chitosan or carrageenan–chitosan patches, our approach offers a systematic evaluation of structural stability, water retention, and flexibility under identical preparation conditions, highlighting the novelty and relevance of these formulations.

Oxazoline-based polymers function as thermoresponsive elements, enabling temperature-dependent adjustments in mechanical stability and permeability [10].

The development of polymer composites that combine control over structural and physical properties is relevant to this field, as it provides a foundation for multipurpose materials adaptable to environmental stimuli. The goal of this research is to create and evaluate composite patches composed of agar, gellan gum, chitosan, and oxazoline derivatives, and to examine how formulation and crosslinking conditions influence their structural, sorption, and thermoresponsive properties.

2. Experimental part

Gellan gum, agar, and chitosan were employed as polysaccharide components. Agar powder of microbiological grade (Sigma-Aldrich, Cat. No. 01916, CAS 9002-18-0), gellan gum Gelrite® (Sigma-Aldrich, Cat. No. G1910, CAS 71010-52-1), and low-molecular-weight chitosan (Sigma-Aldrich, Cat. No. 448869, CAS 9012-76-4; MW 150–250 kDa; DDA 75–85%) were employed

without further purification. A further thermoresponsive ingredient, poly(2-ethyl-2-oxazoline), was added to a few chosen samples.

Composite Patches Preparation

Composites of Gellan and Chitosan (Samples GG).

A transparent solution was created by dissolving 1 g of gellan gum in 70 °C distilled water. Chitosan was made as a colloidal dispersion (0.5 g per 50 mL water) and gradually added to the gellan solution under stirring. For 30 minutes, the resulting films (patches) were exposed to ionic crosslinking in solutions of NaCl (0.1 M) and $CaCl_2$ (0.05 M). To get rid of any remaining salts and unreacted species, the samples were thoroughly rinsed with distilled water after crosslinking.

Composites of Agar and Chitosan (Samples A3 and A4)

To create a translucent solution, agar (1 g) was dissolved in hot distilled water. After a gentle addition of a chitosan colloid (0.5 g), glutaraldehyde (2.5 mL) was added as a crosslinker. To let the mixture gel at room temperature, it was transferred into Petri dishes. Because of the patches' adequate structural stability, no further ionic crosslinking was necessary.

Polymer Composites with 2-ethyl-2-oxazoline

Using the same methods as previously mentioned, these samples were made by adding 0.15 g of poly(2-ethyl-2-oxazoline) to the polymer mixture before glutaraldehyde was added. To get rid of any unreacted ingredients and free crosslinking agent, distilled water was used to wash each gel.

Mechanical studies of polymer patches

Measurement of Thickness. A digital caliper was used to measure the thickness of the film at three to five randomly selected locations, such as the margins and center. Each sample's mean and standard deviation were determined.

Endurance Folding. A chosen section of the film was folded repeatedly until obvious cracks or ruptures emerged in order to manually assess mechanical durability. The number of folds maintained before to failure was used to demonstrate folding endurance.

Research on Swelling. To assess how pH affects water absorption, swelling tests were carried out. For up to 72 hours, dried samples (W_0) were submerged in 250 mL of buffer solutions at room temperature. At predefined time intervals, the gels were removed, surface moisture was wiped off, and the samples were weighed (W_t). The swelling ratio was computed once equilibrium was established according to the formula (1):

$$SR = \frac{W_t - W_o}{W_o} \quad 1)$$

Sol–Gel Analysis. Patches were first dried at 40 °C to a constant weight (m_s) and then submerged in 100 mL of distilled water for a week, stirring occasionally, in order to identify the insoluble fraction. Samples were dried once more to a

consistent mass (m_d) following the extraction of soluble components. The following formula (2) was used to determine the gel fraction and soluble fraction:

$$\text{Gel fraction (\%)} = (m_d/m_s)x100,$$

$$\text{Sol fraction (\%)} = 100\text{-Gel fraction} \quad 2)$$

3. Results and discussion

Over the past two decades, polymer systems with tunable properties have become a subject of extensive research due to their ability to control the physicochemical characteristics and functional performance of materials. In particular, hybrid polysaccharide patches exhibit remarkable structural stability, flexibility, and potential applicability across various technological fields [11,12].

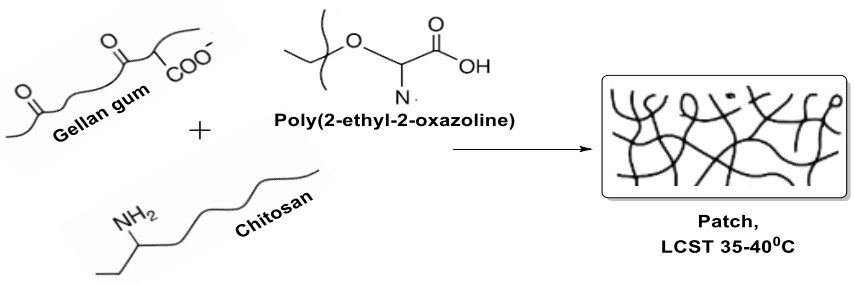

Figure 1 – Illustration of the fabrication and molecular interactions in the thermoresponsive GG–Ch–PEtOx transdermal patch.

In this study, a series of biodegradable composite patches were fabricated from gellan gum, chitosan, and agar–agar, with or without the addition of poly(2-ethyl-2-oxazoline) (PEOZ). Reference patches based on gellan gum–chitosan (GG–Ch) and agar–chitosan (A–Ch) were prepared alongside formulations containing PEOZ (GG–Ch–Ox and A–Ch–Ox) to impart thermoresponsive behavior. Gellan gum was ionically crosslinked by Na^+ and Ca^{2+} ions, providing a mechanically stable network, whereas agar–chitosan films were covalently stabilized with glutaraldehyde, allowing a direct comparison of ionic versus covalent network formation.

The concentration of PEOZ (0.05 g) was selected based on preliminary trials to balance thermoresponsive functionality with mechanical integrity. Its amphiphilic structure and ability to form hydrogen bonds with polysaccharide chains were expected to enhance elasticity, internal cohesion, and overall patch stability. Varying the PEOZ content could modulate the thermoresponsive response, mechanical strength, and swelling behavior, with higher amounts potentially increasing temperature sensitivity but reducing structural robustness, and lower amounts favoring stability but limiting responsiveness.

The incorporation of PEOZ significantly enhances the internal cohesion, elasticity, and overall stability of the composite patches through well-established

intermolecular interactions with the polysaccharide matrix. The amide groups in PEOZ form hydrogen bonds with hydroxyl groups of gellan gum and agar, as well as with amino or hydroxyl groups of chitosan. Such interactions have been demonstrated in previous studies, including hydrogen-bonded complexes of PEOZ with poly(acrylic acid) [13] and in chitosan/PEOZ films, where hydrogen bonding directly contributes to improved network integrity, mechanical performance, and controlled swelling [14]. These hydrogen-bonding interactions increase local network density, thereby supporting mechanical robustness while maintaining thermoresponsive behavior. By adjusting the PEOZ content, the elasticity, cohesion, and thermoresponsive properties of the patches can be systematically tuned, providing a reliable approach to optimize composite performance.

Morphological analysis revealed that the GG–Ch–Ox patches exhibited uniform, smooth surfaces with enhanced internal cohesion, consistent with the stabilizing effect of ionic crosslinking and PEOZ incorporation. The A–Ch–Ox patches maintained flexibility and elasticity due to agar's gel-forming properties, while covalent crosslinking ensured structural stability. In comparison, reference GG–Ch patches were mechanically robust but less responsive to temperature changes, whereas A–Ch patches retained elasticity but exhibited slightly lower cohesion and stability.

These observations indicate that the addition of PEOZ successfully imparted thermoresponsive properties across the series of patches, while the choice of polysaccharide matrix determined the balance between mechanical strength, elasticity, and temperature-dependent behavior. Ionic crosslinking in GG-based patches conferred higher initial mechanical stability, whereas covalent crosslinking in agar-based patches preserved flexibility and smooth morphology. Overall, combining polysaccharide type, crosslinking method, and PEOZ content allows fine-tuning of composite patch properties, providing a versatile platform for future functional applications.

Table 1 – Composition of synthesized polymer patches

Simple	**Gellan gum (g)**	**Agar-agar (g)**	**Chitosan (g)**	**glutaraldehyde (mL)**	**poly(2-ethyl-2-oxazoline) (g)**
GG-Ch **(A)**	1	-	0.5		
A-Ch **(C)**	-	1	0.5	2.5	
GG-Ch-Ox **(B)**	1	-	0.5		0.05
A-Ch-Ox **(D)**	-	1	0.5	2.5	0.05
* Note. Gellan gum samples are crosslinked with cations Na^+ Agar-agar samples are crosslinked with glutaraldehyde					

The obtained compositions were homogeneous and mechanically stable, which made them suitable for further evaluation (Figure 2). The next stage of the

study included measurements of film thickness and folding endurance, tests for swelling and sol–gel ratio, and instrumental analyses SEM imaging.

Thickness, Weight Variation, and Folding Endurance of the Synthesized Patches. Table 2 summarizes the basic physical and mechanical parameters of the obtained polymer patches, including weight variation, mean thickness, and folding endurance. Two polymer systems were investigated: gellan–chitosan (GG–Ch) and agar–chitosan (A–Ch), as well as their modified forms containing a functional additive (GG–Ch–Ox and A–Ch–Ox).

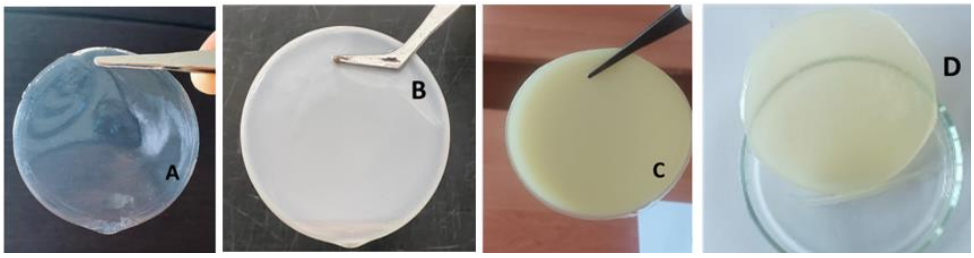

Figure 2 – Transdermal patches based on polysaccharides: A- based on gellan gum without poly-2-ethyl-2-oxazoline; B – based on gellan gum with poly-2-ethyl-2-oxazoline; C - based on agar-agar without poly-2-ethyl-2-oxazoline; D – based on agar-agar with poly-2-ethyl-2-oxazoline.

For the gellan–chitosan system, the control GG–Ch sample exhibited a mean weight of 4.34 ± 1.23 g, an average thickness of 0.1648 mm, and a folding endurance of 68 ± 0.3 cycles. Upon modification (GG–Ch–Ox), the sample mass slightly decreased to 3.70 ± 1.12 g, while the thickness increased marginally to 0.1732 mm. Notably, folding endurance improved significantly, reaching 90 ± 0.7 cycles. This enhancement suggests that the modifier contributes to better structural flexibility and cohesion, likely by reinforcing interpolymer interactions and reducing brittleness within the gellan-based matrix.

For the agar–chitosan composites, a similar tendency was observed but with a more pronounced effect. The unmodified A–Ch patch showed a higher mass (5.68 ± 1.48 g) and thickness (0.3455 mm), but relatively poor flexibility (42 ± 0.8 cycles). After modification (A–Ch–Ox), both weight and thickness decreased (4.95 ± 1.40 g and 0.3047 mm, respectively), while folding endurance nearly doubled to 76 ± 0.4 cycles. Such improvement indicates enhanced mechanical resilience and flexibility of the polymer matrix, which can be attributed to better chain mobility and uniform distribution of the functional additive throughout the agar–chitosan network.

The standard deviation values for weight variation were relatively high (SD ≈ 1.1–1.5 g), possibly due to manual casting and material heterogeneity, whereas deviations in folding endurance were minimal (< 1 cycle), confirming reproducibility of the mechanical behavior within each series. Overall, agar-based patches were thicker and heavier than gellan-based ones, which corresponds to

the naturally denser structure of agar networks. However, both systems exhibited a distinct improvement in mechanical endurance after modification.

These results demonstrate that the incorporation of the functional additive effectively enhances flexibility and durability under repeated deformation, particularly in agar–chitosan matrices. Further mechanical testing (e.g., tensile strength, DMA) and statistical analysis (t-test or ANOVA) would provide deeper insight into the significance and mechanism of these improvements (Table 2).

Table 2 – Results of a mechanical study of polymer patches

Formulation Code	Weight Variation (g)	Mean Thickness (mm)	Folding Endurance
GG-Ch	4.34 ± 1.23	0.1648	68 ± 0.3
GG-Ch-Ox	3.70 ± 1.12	0.1732	90 ± 0.7
A-Ch	5.68 ± 1.48	0.3455	42 ± 0.8
A-Ch-Ox	4.95 ± 1.40	0.3047	76 ± 0.4

The observed differences in weight and flexibility among the synthesized patches indicate that the introduction of the functional additive not only improves mechanical stability but also alters the internal structure and water-binding capacity of the polymer network. Since both gellan and agar matrices possess hydrophilic functional groups capable of interacting with water molecules, it was essential to investigate how the modification affects the swelling behavior of the patches. The swelling properties provide valuable information about the crosslinking density, porosity, and diffusion characteristics of the material, which are critical parameters for its potential use in controlled drug delivery and biomedical applications [15-17].

The illustration depicts the swelling kinetics of GG-Ch, A-Ch, and A-Ch-Ox samples at a pH of 4.01 (Figura 3a.). All samples demonstrate a fast mass rise within the initial 10–20 minutes, thereafter followed by a steady stabilization. The A-Ch-Ox sample has the greatest swelling capacity, due to the presence of oxazoline groups that augment hydrophilicity and water retention. The GG-Ch and A-Ch samples exhibit mild swelling attributable to denser crosslinked networks and ionic interactions within the polysaccharide matrix.

The diagram depicts the swelling characteristics of GG-Ch, A-Ch, and A-Ch-Ox samples at pH 9.18 (Figura 3b.). All samples exhibit a fast mass augmentation over the initial 10–20 minutes, subsequently followed by stabilization at equilibrium. The A-Ch-Ox sample has the greatest degree of swelling, owing to increased hydrophilicity and a more porous network structure resulting from oxazoline integration. In alkaline settings, GG-Ch has a superior swelling capacity relative to A-Ch, presumably due to the ionization of gellan functional groups and enhanced hydration of the polysaccharide matrix.

Swelling studies demonstrated that the A-Ch-Ox sample displayed the greatest hydrophilicity and water absorption in both acidic and alkaline environments, due to the presence of oxazoline moieties that enhance network

flexibility and hydrophilic connections. The GG-Ch and A-Ch samples exhibited considerable swelling attributable to denser crosslinked structures and robust ionic interactions among polysaccharide chains.

Sol–Gel analysis was conducted to determine the proportion of crosslinked (gel) and soluble (sol) fractions in the polymer samples. Dried patch films were immersed in distilled water for one week to extract the soluble components, then re-dried to constant weight. The gel fraction indicates the degree of network crosslinking, while the sol fraction corresponds to the unbound polymer chains. An increase in the gel fraction after incorporating poly(2-ethyl-2-oxazoline) demonstrated enhanced structural integrity and a higher degree of polymer crosslinking [18].

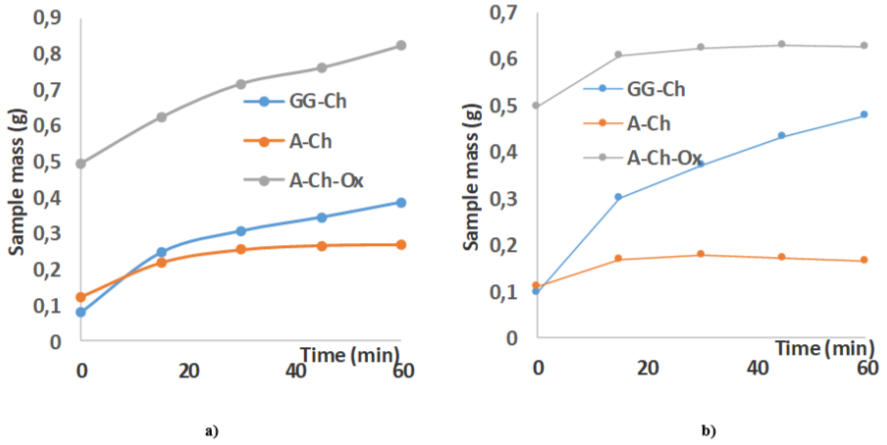

Figure 3 – Swelling behavior of GG-Ch, A-Ch, and A-Ch-Ox composite samples at different pH values: (a) 4.01 and (b) 9.18.

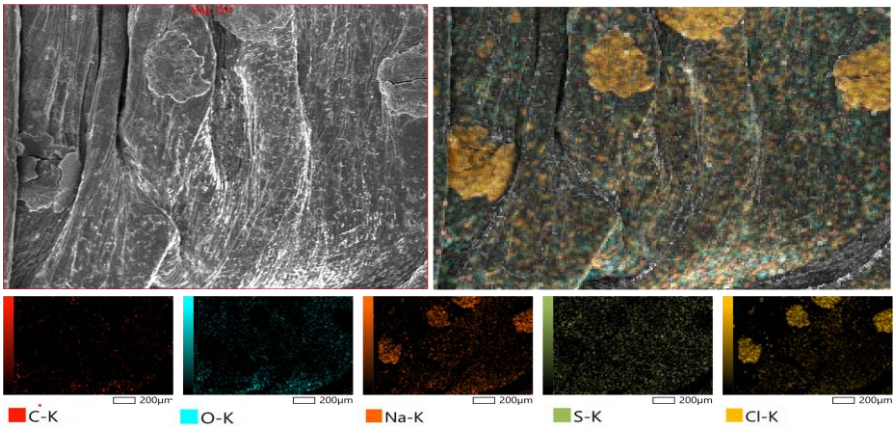

Figure 4 – SEM-EDS analysis of the GG-Ch-Ox sample shows a homogeneous surface morphology with localized Na^+ and Cl^- enrichment, indicating effective crosslinking in the presence of NaCl.

However, while spectral analysis provides indirect evidence of structural reorganization, it does not reveal the morphological characteristics of the obtained composites. Therefore, scanning electron microscopy (SEM) was employed to visualize the surface architecture and assess the distribution uniformity of the components within the polymer matrix (Figura 4).

Scanning Electron Microscopy (SEM) combined with Energy-Dispersive X-ray Spectroscopy (EDS) analysis of the GG-Ch-Ox sample, composed of gellan gum, chitosan, and oxazoline, revealed a homogeneous surface morphology with localized dense regions corresponding to sodium and chloride ion accumulation [19]. This indicates efficient crosslinking of the polymeric matrix in the presence of NaCl.

The uniform distribution of carbon (C) and oxygen (O) reflects the polysaccharide backbone, while sulfur (S) traces originate from dimethyl sulfoxide (DMSO) used during sample preparation. These findings confirm the successful formation of a composite GG-Ch-Ox polymer network with evenly distributed elements.

4. Conclusion

Biodegradable composite patches based on gellan gum, chitosan, and agar–agar were successfully obtained, and the targeted modification with poly(2-ethyl-2-oxazoline) (PEOZ) significantly improved their functional properties. PEOZ enhanced mechanical strength, folding resistance, and cohesion of the polymer network, while also providing more controlled swelling and higher structural stability, as confirmed by sol–gel analysis. SEM data supported the formation of a more uniform and integrated matrix in the modified samples. These findings indicate that PEOZ is an effective enhancer for polysaccharide-based composites and that the developed patches hold strong potential for biomedical use, including wound care and transdermal delivery.

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Conflicts of Interest: The authors declare no conflicts of interest.

ГЕЛЛАН САҒЫЗЫ, ХИТОЗАН, АГАР ЖӘНЕ ПОЛИ(2-ОКСАЗОЛИН) ТУЫНДЫЛАРЫ НЕГІЗІНДЕ ТЕРМОРЕСПОНСИВТІ КОМПОЗИЦИЯЛЫҚ ПАТЧТАРДЫ ӘЗІРЛЕУ

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Түйіндеме: Биологиялық ыдырайтын полимерлі композициялар геллан сағызы, агар-агар және хитозан негізінде әзірленді. Механикалық қасиеттерін жақсарту үшін 0.15 г мөлшерінде поли(2-

этил-2-оказолин) (PEOZ) енгізілді. Төрт үлгі (GG–Ch, GG–Ch–Ox, A–Ch, A–Ch–Ox) құрылымдық және физика-химиялық параметрлері бойынша салыстырылды. Пленкалардың қалыңдығы 0.1648–0.3455 мм, массасы 3.70–5.68 г аралығында болды. PEOZ қосу қатпарлануға төзімділікті айтарлықтай арттырды: геллан-хитозанда 68-ден 90 циклге, агар-хитозанда 42-ден 76 циклге өсті. Ісіну және сол-гель талдауы модификацияланған үлгілерде айқаспалы байланыс тығыздығының жоғарылағанын көрсетті. FTIR деректері полисахаридтер арасындағы сутектік және электростатикалық байланыстарды растады. SEM-EDS (GG–Ch–Ox) біртекті бет пен Na^+/Cl^- иондарының таралуын көрсетіп, тиімді иондық айқаспалануын дәлелдеді. Жаңартылған материалдар механикалық тұрақтылығы, су ұстауы және морфологиялық біркелкілігі арқасында жара таңғыштары мен трансдермальді патчтар сияқты биомедициналық қолдануларға қолайлы.

Түйінді сөздер: геллан сағызы, хитозан, агар-агар, оксазолиндер, патч, полимерлі композиттер, ыстыққа сезімтал материалдар.

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РАЗРАБОТКА ТЕРМОЧУВСТВИТЕЛЬНЫХ КОМПОЗИТНЫХ ПЛАСТЫРЕЙ НА ОСНОВЕ ГЕЛЛАНОВОЙ КАМЕДИ, ХИТОЗАНА, АГАРА И ПРОИЗВОДНЫХ ПОЛИ(2-ОКСАЗОЛИНА)

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Резюме: Биоразлагаемые полимерные композиции получены на основе геллановой камеди, агар-агара и хитозана, с добавлением 0.15 г поли(2-этил-2-оказолина) (PEOZ) для улучшения свойств. Четыре варианта плёнок (GG–Ch, GG–Ch–Ox, A–Ch, A–Ch–Ox) исследованы по структурным и физико-химическим характеристикам. Толщина составляла 0.1648–0.3455 мм, масса — 3.70–5.68 г. Введение PEOZ повысило устойчивость к многократному сгибанию: в системе геллан–хитозан с 68 до 90 циклов, в агар–хитозане — с 42 до 76 циклов. Анализы набухания и сол–гель показали увеличение плотности сшивки и снижение растворимой фракции. FTIR подтвердил наличие водородных и электростатических взаимодействий. SEM-EDS изображения GG–Ch–Ox выявили однородную поверхность и распределение Na^+/Cl^-, указывая на эффективное ионное сшивание. Модифицированные системы демонстрируют повышенную механическую прочность, удержание воды и морфологическую однородность, что делает их перспективными для медицинских применений — перевязочных материалов и трансдермальных патчей.

Ключевые слова: геллановая камедь, хитозан, агар-агар, оксазолины, патч, полимерные композиты, термочувствительные материалы.

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