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ANALYSIS OF THE PHYSICAL AND MECHANICAL PROPERTIES OF THERMOPLASTIC GELATIN FILMS OBTAINED BY VARIOUS METHODS AND THEIR COMPUTER-MODELED STRUCTURE

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Abstract

The mixing steps involved in the formation of biodegradable thermoplastic films based on natural polymers limit the ability to achieve the desired properties. The production of thermoplastic gelatin films involves modifying the gelatin matrix with plasticizers to impart thermoplastic properties, allowing it to be processed through traditional plastic manufacturing methods. In this work, water and Glycerol plasticizer were used to modify the bovine gelatin matrix. Plasticizers were added to the gelatin composition in various proportions (from 20 wt% to 100 wt%), and film samples were obtained. Thermoplastic gelatin films were produced by casting a solution onto a defined surface, employing three different methods: by altering the stages of introducing the plasticizer into the gelatin matrix (in 2 methods) and by introducing an additional stirring step during the heating of the solution. The resulting thermoplastic gelatin films were subjected to mechanical tests and compared in terms of relative elongation at break and strength. It was found that the mechanical strength and Young's modulus of gelatin films plasticized with glycerol decreased with increasing glycerol content in gelatin, and the relative elongation at break increased. The computer-modeled structure was analyzed using density functional theory (DFT) to study the electronic structures and interactions of the gelatin biopolymer with plasticizers. The electronic characteristics of the HOMO and LUMO orbitals and the frontier orbitals of the system were calculated.

Keywords: thermoplastic gelatin; glycerol; water; film; mechanical properties; DFT.

АНАЛІЗ ФІЗИКО-МЕХАНІЧНИХ ВЛАСТИВОСТЕЙ ТЕРМОПЛАСТИЧНИХ ЖЕЛАТИНОВИХ ПЛІВОК, ОТРИМАНИХ РІЗНИМИ МЕТОДАМИ, ТА ЇХ КОМП'ЮТЕРНО-МОДЕЛЬОВАНА СТРУКТУРА

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Анотація

Етапи змішування компонентів під час стадійного формування біорозкладних термопластичних плівок на основі природних полімерів обмежують можливість досягнення бажаних властивостей. Виробництво термопластичних желатинових плівок включає модифікацію желатинової матриці пластифікаторами для надання термопластичних властивостей, що дозволяє обробляти її традиційними для виробництва пластмас методами. В цій роботі для модифікації бичачої желатинової матриці використовували воду та пластифікатор гліцерол. Пластифікатори додавали до желатинової композиції в різних пропорціях (від 20 мас.% до 100 мас.%), і отримували зразки плівки. Термопластичні желатинові плівки готували шляхом лиття з розчину на певну поверхню, використовуючи три різні методи: змінюючи етапи включення пластифікатора в желатинову матрицю (в 2 методах) та вводячи додатковий етап перемішування під час нагрівання розчину. Отримані термопластичні желатинові плівки піддавали механічним випробуванням. Плівки порівнювали за відносним подовженням під час розриву та за міцністю. Було виявлено, що механічна міцність та модуль Юнга желатинових плівок, пластифікованих гліцерином, зменшувалися зі збільшенням вмісту гліцерину в желатині, а відносне подовження під час розриву збільшувалося. Комп'ютерно змодельовану структуру проаналізували за допомогою теорії функціоналу густини (DFT) для вивчення електронних структур та взаємодії желатинового біополімеру з пластифікаторами. Були розраховані електронні характеристики орбіталей HOMO та LUMO, а також граничні орбіталі системи.

Ключові слова: термопластичний желатин; гліцерол; вода; плівка; фізико-механічні властивості; DFT.

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Introduction

In recent years, the production of biopolymers based on renewable plant and animal raw materials has been increasingly developing worldwide [1–3]. One of the most significant advantages of using biopolymers instead of synthetic polymers is the possibility of utilizing substances and products that are continuously regenerated in nature as raw materials, unlike oil, coal, and gas, which have limited reserves in nature [4; 5]. The characteristics of biopolymer-based films are mainly determined by the chemical nature of the biopolymer [6]. Protein-based films have good organoleptic and mechanical properties, and also serve as a good barrier for non-condensable gases (O_2, CO_2, and N_2) and aromas [7].

Among natural polymers, gelatin is one of the most protein-rich and rapidly absorbed polymers by microorganisms [8; 9]. Gelatin is a collection of peptides and proteins formed by partial hydrolysis or denaturation of collagen, which contains a disruption of the triple helical structure (Fig. 1) [10–12].

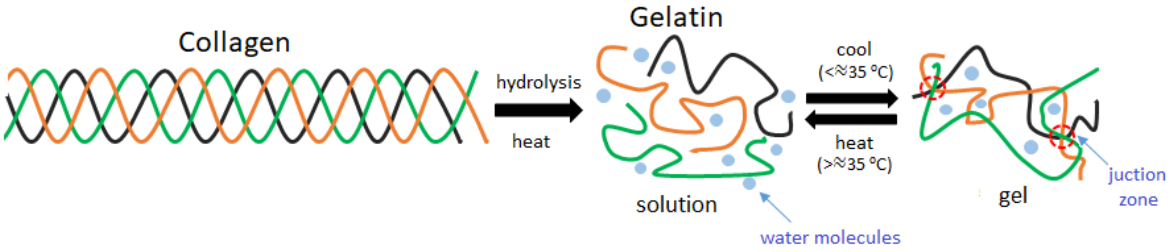


Fig. 1. Physical interaction during the formation of the molecular and supramolecular structure of gelatin as a result of collagen hydrolysis

The physicochemical properties of collagen can be appropriately modulated; moreover, it is relatively cheap and easy to obtain in concentrated solutions [13; 14]. Gelatin is known to be completely biodegradable [15] and has good film-forming properties [16]. Studies on the preparation of gelatin films have shown that native gelatin films exhibit low flexibility and are brittle [17–19]. To increase the stretchability, flexibility, and toughness of the films, plasticizers or nanofillers should be added [20–22]. Processing of natural polymers, polysaccharides, polypeptides, and other polymers requires their conversion to a thermoplastic state [23–26]. To convert gelatin and other natural polymers into thermoplastics, water [27], Glycerol [28–30], oil [31], sorbitol, urea, formamide [32; 33], and many other polar substances are used as plasticizers. The study by Rezaei and Motamedzadegan [34] was consistent with the results of the study by Al-Hassan and Norziah [35], which indicated that glycerol had a better plasticizing effect than sorbitol on the mechanical properties of bovine and fish gelatin films. The plasticizer replaces the hydrogen bonds between gelatin chains with hydrogen bonds between gelatin and glycerol, which promotes conformational changes that lead to increased deformation. The inverse relationship between the elongation and hardness of films causes a decrease in hardness. Gelatin films have been reported to exhibit high stiffness with very low elongation in samples containing <10 % glycerol [36]. At least 20 % Glycerol or other suitable plasticizer is required for successful plasticization of gelatin film [37]. Studies have shown that the addition of plasticizers in various percentages improved the mechanical properties of gelatin films [38]. Different concentrations of glycerol used in film formation have different effects on the mechanical strength values of gelatin films, with glycerol concentrations at 10 % (108.28 ± 6.38 MPa) [39], 20% (1.75 MPa), 25% (1.67 ± 0.12) [35], and 30% (2.91 ± 0.43 MPa) [40]. Good results have been obtained in studies devoted to the preparation of gelatin films and the investigation of their mechanical and other properties [41; 42].

Over the past decade, computer modeling has gained significant attention from researchers for analyzing the electronic structure and interactions of synthetic and natural polymers to develop special-purpose materials [43; 44]. This approach provides valuable insights into drug loading processes, their distribution, complex formation, and release from nanoparticles (nanocarriers) [45; 46]. Theoretical studies have led to an improved understanding of the structural and thermodynamic properties of polymer solutions [47–49]. Significant theoretical progress has also been made in elucidating the structure of polymer solutions across various concentration ranges, based on the analysis of scaling laws [50].

Therefore, this study aims to investigate the effect of different glycerol incorporation routes on the mechanical properties and structural behavior of thermoplastic gelatin films. Particular attention is paid to the influence of the plasticizer addition sequence and content on tensile strength, Young's modulus, and elongation at break. The study seeks to clarify how processing methodology governs plasticization efficiency and structure–property relationships in gelatin-based films.

Material and methods

Materials

Gelatin (Gel) (ISO HACCP) Bloom 200 bovine gelatin produced by "HENAN E-KING GELATIN", ρ=1.3 g/cm^3. Glycerol $C_3H_8O_3$, colorless liquid, M=92.094 g/mol, T_q=290 °C, ρ=1260 kg/m^3. Water distilled H_2O, ρ=1000 kg/m^3.

Methods for obtaining thermoplastic gelatin films

Gelatin-based films were prepared using three different methods, varying the steps of introducing plasticizers into the gelatin composition (in 2 ways) and adding a stirring step during heating of the solution, and thermoplastic gelatin films were formed by pouring the solution onto a specified surface. The gelatin powder was first dried at 50 °C for 24 hours to remove residual moisture. Glycerin was used as a plasticizer in an amount ranging from 20 to 100 % based on the dry mass of 3 g of gelatin. The experiments were repeated twice and 5 samples were tested from each experiment. (Temperature stirring was performed using an "MSE PRO 4 Channel LCD Digital Hotplate Magnetic Stirrer" and a Laboratory oven).

Method 1. 3 g of dry gelatin was first completely dissolved in 100 ml of distilled water at 85 °C, then glycerin was added dropwise, then kept in an oven at 80 °C for 2 hours, and the solution was taken and poured into a 20×30 cm plastic container, and film samples were formed at room temperature (25±2 °C) in 2.5 days.

In method 2, 3 g of dry gelatin powder was premixed with the required amount of glycerin and completely dissolved in 100 ml of distilled water at 85 °C, then kept in an oven at 80 °C for 2 hours, and the solution was taken and poured into a 20×30 cm plastic container, and film samples were formed at room temperature (25±2 °C) in 2.5 days.

In method 3, glycerol gelatin was added to 3 g of dry gelatin, followed by an intensive stirring step at 200 rpm and 80 °C for 1 h on a magnetic stirrer to increase the plasticizer dispersion and chain mobility. The solution was poured into a 20×30 cm plastic container and film samples were formed at room temperature (25±2 °C) for 2.5 days.

The resulting solutions formed flat, flexible thermoplastic gelatin films suitable for mechanical testing. The film sample was transparent in appearance and flexible Fig. 2.

a) b)

Fig. 2. Gelatin with 30 % Glycerol content, a) film (size 20×30 cm) and b) package samples (size 18×25 cm)

Mechanical Testing

Tensile tests were conducted on a Shimadzu AG-X PLUS (Japan) in accordance with the international standard ASTM D 638. Tests were conducted at a speed of 1 mm/min up to 0.3 % deformation to measure Young's modulus (E), followed by a speed of 20 mm/min to measure the tensile strength ($\boldsymbol{\sigma}$) and deformation ($\boldsymbol{\varepsilon}$).

Density Functional Theory (DFT) Analysis

A computational study of the gelatin-glycerol system was performed using density functional theory (DFT) to study the electronic structure, intermolecular interactions, and thermodynamic stability of the composite. The system was modeled by representing glycerol as a small molecule with three hydroxyl groups capable of forming hydrogen bonds. Molecular dynamics (MD) simulations were conducted to investigate the temperature and pressure dependence of the material's structural and mechanical properties. The calculations were performed using software packages, including Quantum ESPRESSO, for large-scale DFT simulations [51–54].

A plane-wave basis set with an energy cutoff of 40 Ry was chosen for the tests. During the geometric optimization, all atoms were completely free to move, and the forces acting on them were minimized to less than 0.01 eV/Å. The optimized structures were then used to estimate geometric parameters and adsorption energies. A vacuum gap of 30 Å was applied to the molecular system to avoid interaction between neighboring systems. The Gaussian program, using the B3LYP/6-31++G(d,p) basis set, was employed to optimize the energy and geometry at the molecular level. These methods enabled the estimation of binding energies, charge distributions, and vibrational spectra, yielding important insights into the flexibility and stability of the composite material.

Results and discussion

The production of thermoplastic gelatin films involves incorporating plasticizers into the gelatin matrix to enhance the thermoplastic behavior of the material, thereby increasing its flexibility, physical and mechanical stability, and thermal stability, and allowing the material to be processed using conventional plastic manufacturing methods. Plasticizers are low-volatility molecules that enable biopolymer materials to modify the functional properties of films by increasing their elongation, flexibility, adaptability, elasticity, stiffness, and mechanical properties [55].

The effect of plasticizers is most clearly evident in the material's mechanical properties. Physically, as the plasticizer ratio increases, the gelatin chains reduce their mutual bonds and interactions, allowing plasticizers to enter between the chains and interact with them, thereby increasing their mobility and decreasing the viscosity (Fig. 3).

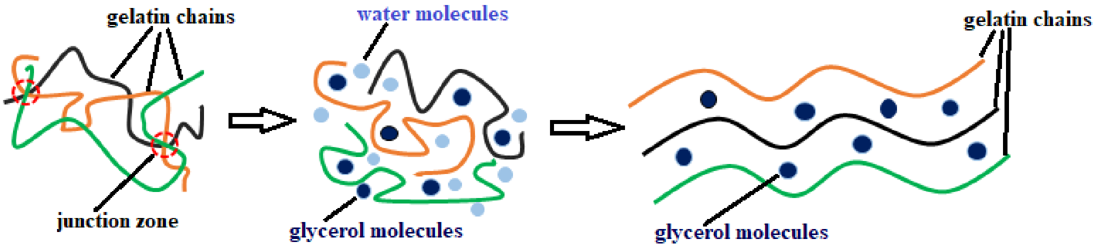
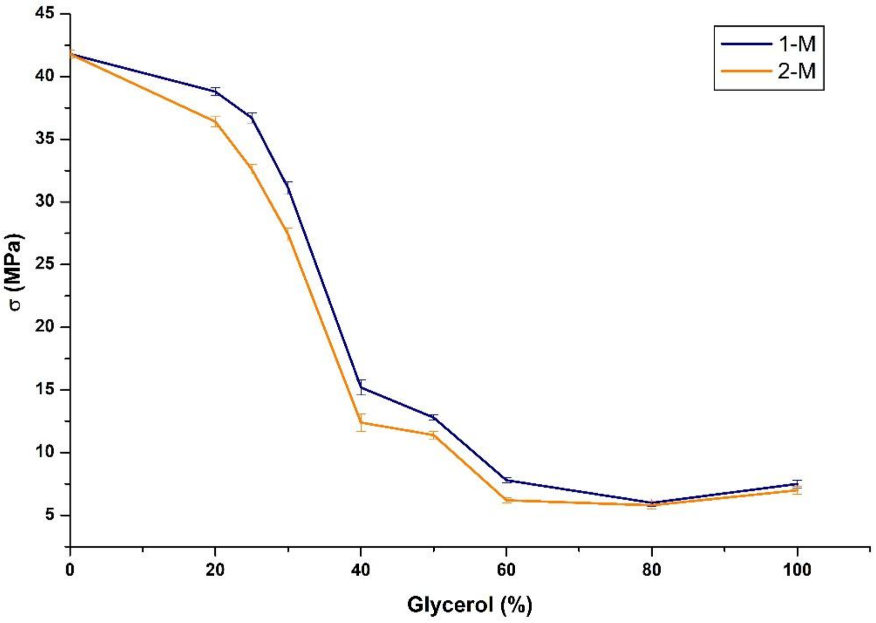
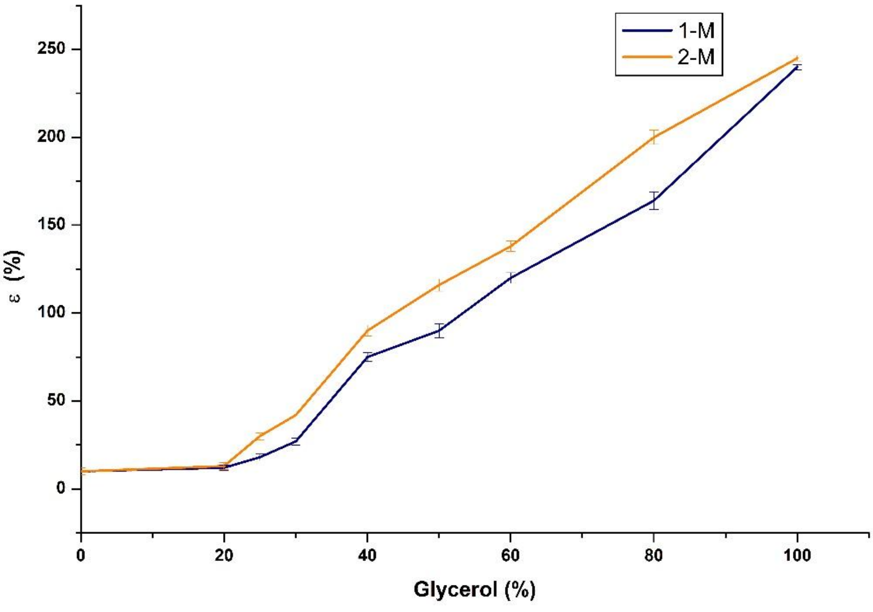


Fig. 3. Physical interaction of gelatin with plasticizers

In a research study aimed at producing thermoplastic films from gelatin and achieving mechanical properties comparable to those of conventionally used films by investigating the mixing stages, the mechanical properties of film samples obtained using three different methods were analyzed. The following results were obtained from the mechanical properties of thermoplastic gelatin films obtained by method 1: Fig. 4.1-M.



a)



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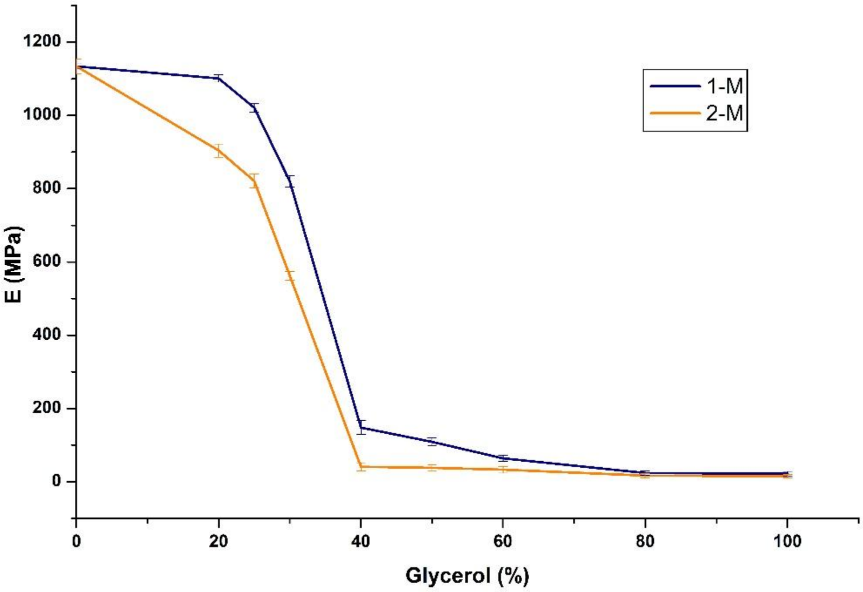


c)

Fig. 4. Method 1 (1-M) and Method 2 (2-M). Changes in mechanical properties, tensile strength (a), relative elongation (b) and Young's modulus (c), with increasing glycerol content in gelatin

It was found that the addition of gelatin and a plasticizer (glycerin) to the gelatin composition in amounts ranging from 20 % to 100 % had a significant effect on its relative elongation: as the glycerin content in the gelatin composition increased, the elongation at break increased, while the tensile strength and Young's modulus decreased. The film thickness values obtained by casting using water-based plasticization without adding Glycerol to the gelatin composition are given on the ordinate axis in the figures above (4-a, b, c, figures 1-M). According to its mechanical properties, the tensile strength was 41.8 MPa, the relative elongation at break was 8% and the Young's modulus was 1134 MPa. It was found that the changes in mechanical properties depended on the amount of glycerol in the gelatin, i.e., when glycerol was added to the gelatin in amounts ranging from 20 % to 100 %, the tensile strength decreased from 38.8 MPa to 6.5 MPa, the Young's modulus decreased from 1101 MPa to 22.4 MPa, and the relative elongation increased from 9 % to 240 %. From the above physical interaction diagrams (Figure 3), it can be observed that this process weakens the interactions between the gelatin chains. Glycerol establishes hydrogen bonds between the interacting chains, which is facilitated by glycerol, thereby increasing their plasticity and transforming them into a flexible thermoplastic. The excess amount of glycerol between the chains acts as a lubricant in the material, increasing the relative sliding of the chains, which in turn increases the relative elongation and decreases the strength.

The mechanical properties of thermoplastic gelatin films obtained by method 2 differed from those of method 1 (Fig. 4 .2-M). According to the results of the mechanical property tests on the thermoplastic films obtained using method 2, as in method 1, the relative elongation at break increased with increasing glycerin content, while the tensile strength and Young's modulus decreased. The difference in method 2 was that when comparing the glycerin content in the composition by 10 % and 20 %, it was found that the maximum difference in elongation at break of samples with the same percentage of glycerin added increased by up to 40 %. In method 2, the tensile strength decreased from 36.4 MPa to 6 MPa, and the Young's modulus decreased from 904.4 MPa to 15.8 MPa compared to method 1. The tensile strength values in samples with a glycerol content of up to 50 % were close to those of conventional polymer (polyethylene) films, allowing for their application in this field. Plasticizers, such as glycerol and water, included in the gelatin composition, compete for interaction with gelatin molecules. The samples prepared according to Method 1 contained a combination of water, Glycerol, and gelatin. The high water content dominates the interaction with gelatin, and this process continues until the water evaporates. As a result of the loss of water, the interaction between gelatin chains increases again. In method 2, initially, Glycerol completely interacts with gelatin, ensuring effective plasticization. At the next stage, the effect of water occurs, allowing it to freely settle between gelatin chains and not prevent their interaction. As a result, the samples obtained by this method are better plasticized, which leads to an increase in the ductility of the material and a decrease in strength.

Method 3 of obtaining a thermoplastic gelatin film is the same as method 2 in terms of adding the components to each other, but differs in that they are mixed with a mixer at a certain temperature for a certain time. The mechanical properties of the TPJ film containing 40% glycerol obtained by this method were compared with the samples containing 40 % glycerol obtained using the two methods mentioned above (Table 1 and Fig. 5).

Table 1

Mechanical properties of TPJ films containing 40% glycerol obtained using three different methods

№	Water/gelatin/glycerol ml/gr/gr	Method	Thickness (μm)	σ [MPa]	ε [%]	E [MPa]
1.	100/3/1.2	1	65	15.02±1.9	75±3.9	148.9±19
2.	100/3/1.2	2	65	12.4±2.6	90.6±4.7	41.6±11
3.	100/3/1.2	3	65	8.1±2	105.4±6.8	35±7

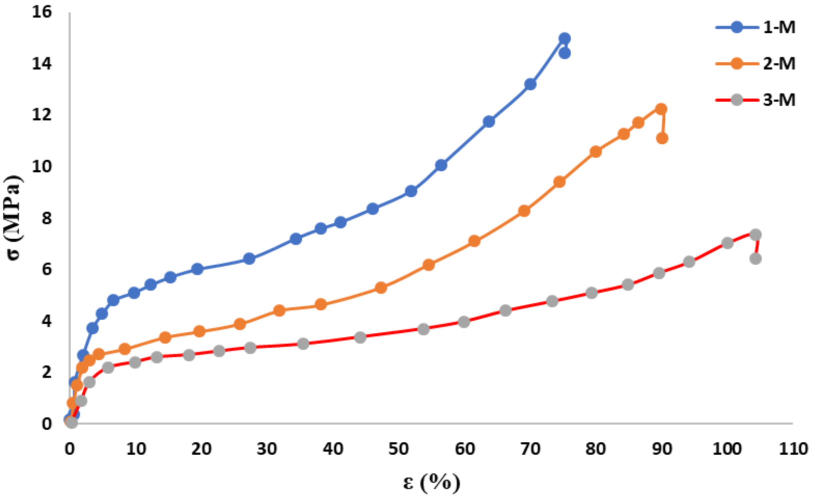


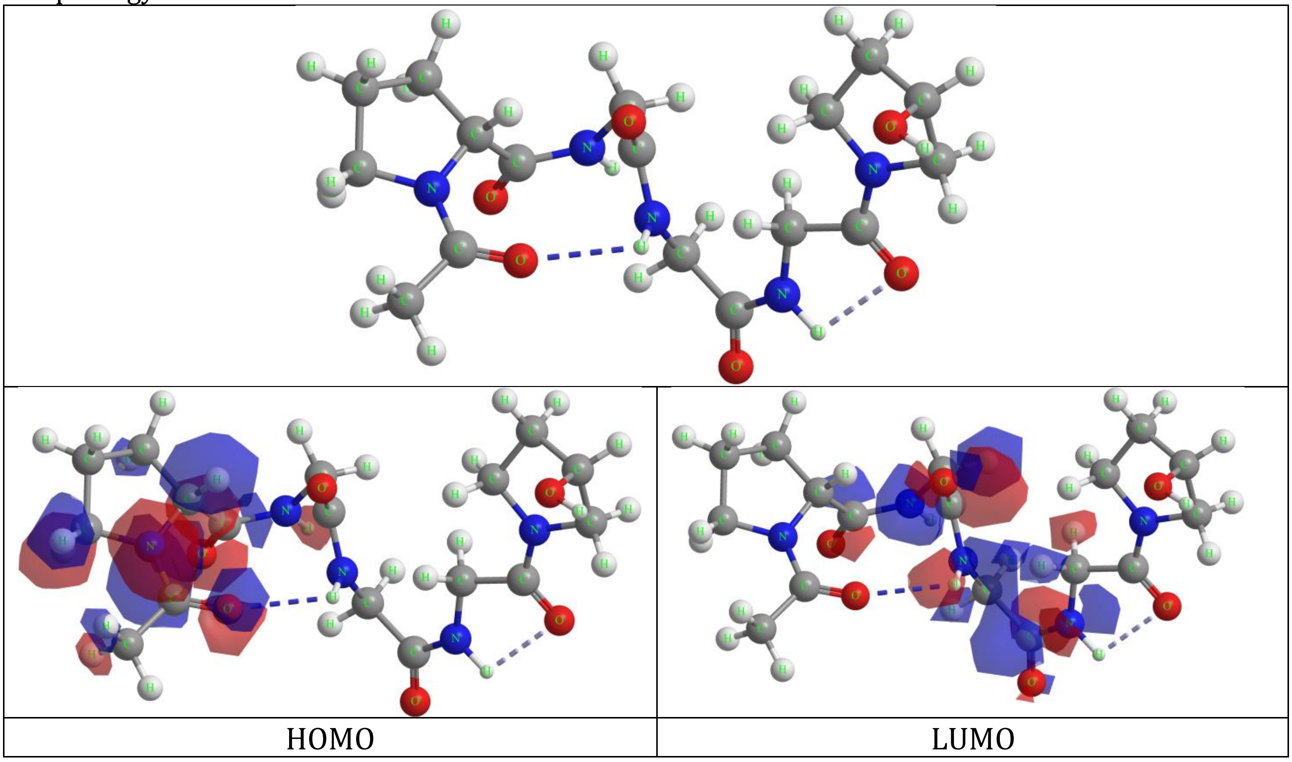
Fig. 5. Mechanical properties of samples with 40% glycerol added to the gelatin composition in 3 different ways (method 1-M, method 2-M, method 3-M)

The effect of the methods used to obtain gelatin-based thermoplastic films on their mechanical properties is shown in Table 1. The films prepared by method 1 had the highest strength, with a maximum tensile strength of 15.2 ± 1.9 MPa. At the same time, it was found that the Young's modulus of these samples was 148.9 ± 19 MPa, which is the highest Young's modulus index.

However, the relative elongation at break was 75 ± 3.9 %, and the elasticity was relatively low. In method 2, the plasticization of the material improved, and the maximum tensile strength decreased to 12.4 ± 2.6 MPa, but the relative elongation at break increased to 90.6 ± 4.7 %. The Young's modulus was 41.6 ± 11 MPa, indicating that the strength decreased due to the increased elasticity of the material. In method 3, plasticization was carried out to a higher extent due to the movement of the chains. As a result, the tensile strength decreased by 8.1 ± 2 MPa, and the elongation at break increased to 105.4 ± 6.8 %, which indicates that the material was effectively plasticized. The film had a Young's modulus value of 35 ± 7 MPa, indicating its strength, which confirms the elasticity of the film. Based on these results, it can be concluded that the stages of forming gelatin films are also important. Proper plasticization of gelatin helps to improve the distribution of polypeptide chains and helps achieve improved structural uniformity morphology.

The addition of glycerol to gelatin can cause phase separation or excessive plasticization. Excessive glycerol can disrupt the polymer matrix excessively, resulting in a decrease in viscosity as the material loses its structural integrity and cohesive properties. The point at which additional glycerol cannot be effectively incorporated into the polymer matrix, or the addition of excess glycerol, acts as a lubricant rather than a plasticizer, resulting in minimal change in mechanical properties.

In addition to determining the optimal conditions for plasticizers used to obtain gelatin films, theoretical solutions for material formation or polymer composites are available. Today, studies based on receiving the electronic structures of an object and its computer modeling are utilized in many areas [44; 56–62]. Theoretical solutions for determining the optimal conditions for plasticizers are presented in Fig. 6, which shows the optimized geometric representation of the gelatin molecule.


HOMO

LUMO

Fig. 6. Optimized geometry and frontier molecular orbitals of the gelatin molecule

As can be seen from Fig. 6, the gelatin unit is stabilized by one type of intramolecular hydrogen bond, specifically the -N–H----C=O bond. The lengths of the hydrogen bonds are 2.4 and 2.2 Å. In experimental studies, glycerol is used to break the interchain bonds.

In a glycerol solution, the hydration shell consists mainly of amorphous water, and a decrease in the density of glycerol is observed near the biopolymer molecule. In an aqueous solution, a water layer forms around the gelatin molecule. This layer plays a crucial role in the gel formation process, stabilizing the solubility and structure of the biopolymer (Fig. 7).

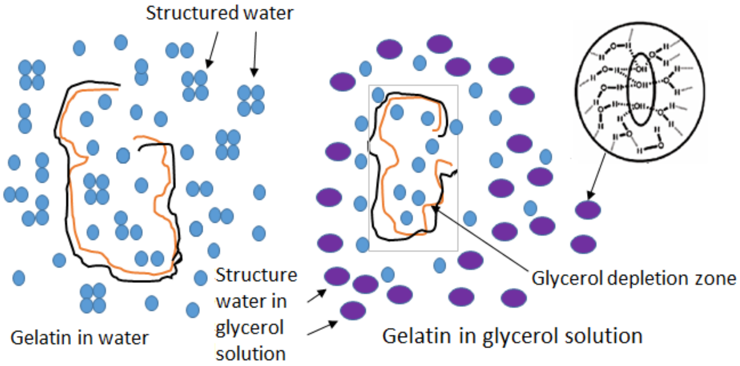


Fig. 7. Interaction of gelatin molecules with solvent molecules in aqueous and glycerol solutions

In a glycerol solution, the distribution of water and glycerol around the gelatin molecule is different. The main difference is that the density of glycerol molecules around the gelatin molecule decreases, that is, the concentration of glycerol near the biopolymer is lower, and more water remains. This means that glycerol does not accumulate around the biopolymer, and water molecules remain as the main hydration layer, which affects the physicochemical properties of the solution, the structure-forming properties of gelatin, and the dissolution process.

The optimized geometric shape of the interaction of gelatin with glycerol molecules is presented in Fig. 8.

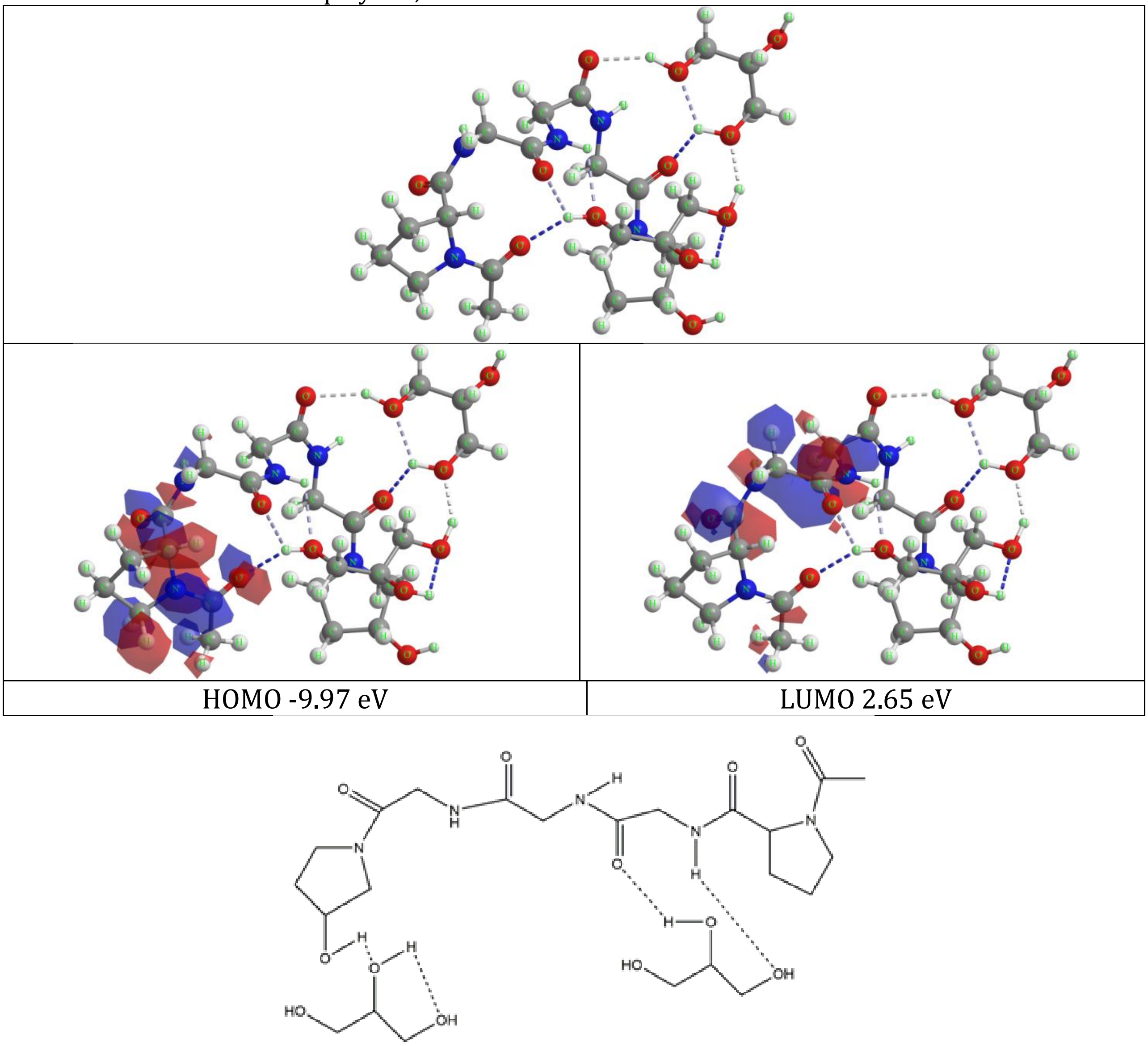


Fig. 8. Optimized interaction geometry of a glycerol molecule with a gelatin structural unit

Fig. 8 shows the optimized interaction geometry of a gelatin structural unit with two glycerol molecules. This figure reflects the optimized geometry obtained as a result of quantum-chemical calculations, i.e., the most stable state. The system is stabilized by the formation of three intermolecular hydrogen bonds. Gelatin (-C=O) – Glycerol (-OH), Gelatin (-NH) – Glycerol (-OH) and Gelatin (-OH) – Gelatin (-OH) (single intermolecular bond) these bonds strengthen the interaction between gelatin and glycerol, stabilizing the structure. The bond formed between the carbonyl group (-C=O) and the hydroxyl group (-OH) of glycerol increases the rigidity and stability of the gelatin molecule. The bonds formed between the amine group (-NH) of gelatin and the hydroxyl group (-OH) of glycerol make the polymer matrix more flexible, contributing to plasticization. Interactions are formed between the hydroxyl (-OH) groups of gelatin molecules. Such interactions strengthen the internal bonding of the molecules and affect the mechanical properties of the structure.

The lengths of hydrogen bonds formed between gelatin and glycerol molecules are given in Table 2.

Table 2

Interaction parameters of the gelatin-glycerol system

Gelatin	Glycerol	Hydrogen Bond Lengths, Å
O (28)	H(76)	2.23
O(16)	H(76)	2.05
O(12)	H(68)	2.07
O(8)	H(74)	2.27
H(54)	O(35)	2.22

It can be seen that the bond lengths range from 2.05 Å to 2.27 Å, indicating that the hydrogen bonds are moderately strong. The shortest bond (2.05 Å) is the O(16)-H(76) pair, which has a relatively strong effect. The longest bond (2.27 Å) is the O(8)-H(74) pair, which is a relatively weak hydrogen bond. These results confirm the interaction of glycerol molecules with the gelatin structure, helping to understand the stability that occurs during plasticization. These interactions, formed through hydrogen bonds, have a significant impact on the mechanical properties of gelatin, including its elasticity and strength.

The electronic orbital structure of gelatin and glycerol, as determined through theoretical studies, provides valuable insights into film formation, changes in mechanical properties, and physicochemical properties. This information is important for the creation of gelatin-glycerol-based biomaterials.

Conclusion

Thermoplastic gelatin films plasticized with glycerol were successfully prepared using three different processing methods that differed in the sequence and conditions of plasticizer incorporation. The results demonstrated that both glycerol content and processing route significantly influence the mechanical performance of the obtained films. An increase in glycerol content from 20 to 100 wt.% led to a systematic decrease in tensile strength and Young's modulus, accompanied by a pronounced increase in elongation at break, confirming the effective plasticizing action of glycerol. The magnitude of these changes depended on the method of glycerol introduction, indicating that processing history plays a key role in governing structure–property relationships in thermoplastic gelatin films. Comparative analysis of the three methods showed that films prepared with additional intensive mixing exhibited more stable mechanical behavior at higher glycerol contents, suggesting improved plasticizer distribution and enhanced chain mobility. These trends support the hypothesis that the mechanism of glycerol action changes beyond a certain concentration range. Importantly, this study establishes a clear relationship between glycerol concentration, processing methodology, and mechanical properties, thereby addressing the research aim of clarifying how plasticizer incorporation routes affect the mechanical behavior of thermoplastic gelatin films.

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