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ФИЗИЧЕСКИЕ СВОЙСТВА ПИЩЕВЫХ ПЛЕНОК ИЗ ГИДРОКСИПРОПИЛМЕТИЛЦЕЛЛЮЛОЗЫ, ОБОГАЩЕННЫХ МАСЛОМ ВИНОГРАДНЫХ КОСТОЧЕК

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PHYSICAL PROPERTIES OF GRAPE SEED OIL LOADED HYDROXYPROPYL METHYLCELLULOSE EDIBLE FILMS

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Реферат

Цель исследования: изучение структуры, физико-механических и барьерных свойств пищевых пленок из гидроксипропилметилцеллюлозы (ГПМЦ), обогащенных маслом виноградных косточек.

Материал и методы. В ходе исследования были изучены: структура, физико-механические, газобарьерные и термические свойства пищевых пленок из ГПМЦ, обогащеных маслом виноградных косточек. Пленки были получены путем смешивания водного раствора ГПМЦ с наноэмульсией масла виноградных косточек (GONE) в различных концентрациях и последующего нанесения на стеклянные чашки Петри. Топографию поверхности пленок исследовали с помощью поляризационной микроскопии, которая продемонстрировала наличие кристаллических морфологических единиц разного размера. При более высоких концентрациях масла виноградных косточек стало очевидным неоднородное распределение масляной фазы и наличие полостей, содержащих капли эмульсии. Степень кристализации, которая была оценена с помощью дифференциальной сканирующей калориметрии, увеличивалась с увеличением концентрации масла виноградных косточек. Физико-механические свойства пленок были исследованы в режиме растяжения с использованием универсальной испытательной машины LS1 (Lloyd Instruments).

Результаты. Было обнаружено, что добавление масла виноградных косточек снижает композитную прочность растяжения. Кроме того, газобарьерные свойства композитных пленок (против CO_2 и O_2) улучшались при увеличении концентрации масла виноградных косточек. Основываясь на наших экспериментах, пленки с 5%-ной концентрацией эмульсии показали оптимальные свойства.

Выводы В дальнейшем эти пленки или эмульсионные покрытия будут применяться для улучшения качества фруктов при хранении в холодильнике.

Ключевые слова: барьерная технология, наноэмульсия, топография поверхности, термические свойства, газобарьерные свойства, свойства при растяжении.

Abstract

Objectives: the present work aims to investigate the effect of composition on the structure, physico-mechanical and barrier properties of edible films made from hydroxypropyl methylcellulose (HPMC) with incorporated grape seed oil (GsO).

Material and methods. In this study structure, physico-mechanical, gas barrier and thermal properties of grape seed oil (GsO) loaded hydroxypropyl methylcellulose (HPMC) edible films were investigated. The films were prepared by mixing of HPMC water solution with GsO nanoemulsion (GONE) in different concentrations and further casting in glass Petri dishes. The surface topography of the films was examined by Polarizing Microscopy, which demonstrated a presence of crystal morphological units with different size. At higher GsO concentrations an inhomogeneous distribution of the oil phase and a presence of cavities containing emulsion droplets became evident. The degree of crystallinity, which was estimated by Differential Scanning Calorimetry increased with increasing the GsO concentration. The physico-mechanical properties of the films were examined in tensile mode using an LS1 universal testing machine

(Lloyd Instruments). It was found that the addition of GsO reduced the composite tensile strength.

Results. Furthermore, gas barrier properties of the composite films (against CO_2 and O_2) were improved when the GsO concentration increased. Based on our experiments the films with 5% emulsion concentration showed optimal properties.

Conclusions. In further experiences, these films or emulsion coatings will be applied to enhance the quality of fruits during refrigerated storage.

Key words: hurdle technology, nanoemulsion, surface topography, thermal properties, gas-barrier properties, tensile properties.

Introduction. Biodegradable edible packaging can be used as alternative of petroleum-based plastics [1]. The edible packaging are primary food-packaging materials made from natural biopolymers with other edible components on the surface of the foods [7]. The aim of the packaging is to act as a barrier for gas and moisture exchange, to decrease the weight and volatile compound loss, to delay the enzymatic oxidations and the microbiological contaminations [11]. The results of these coatings is satisfactory extended shelf-life or freshness time of fruits (oranges [22], apple cubes [20], melon cubes [17] and sweet cherries [2, 19] etc.), but they also can affect the product quality by modification the internal atmosphere [9], change the appearance and the other sensory properties. To achieve the most optimal properties and to meet the food-grade requirements, the edible films are formed or combined from polysaccharides, proteins and lipids, but can contain essential oils and other low molecular-weight compounds (for e. g. Ca-lactate etc.) as well [6]. The most important aspects in the compound selections are the water-solubility, the barrier, the sensory, the mechanical, the antioxidant and anti-microbiological properties [13]. The combination of polymers with essential oils prevents the degradation of the component of coated food enhance antimicrobial and antioxidant properties, but the application of essential oils is limited because they are not water-soluble, not colorless and flavorless materials and change the mechanical properties as well [8, 12].

Objectives: the present work aims to investigate the effect of composition on the structure, physico-mechanical and barrier properties of edible films made from hydroxypropyl methylcellulose (HPMC) with incorporated grape seed oil (GsO).

Material and methods. Hydroxypropyl methylcellulose (HPMC) and Tween 20, used as a surfactant, were purchased from SIGMA Aldrich, Germany (composition: hydroxylpropoxyl content, ~9%; viscosity: ~15 mPa.s [2% in H_2O, 25°C], plant origin). Food grade Grape seed oil (Ikarov Ltd., Plovdiv, Bulgaria) and Glycerol were purchased from local pharmacy. Distilled water was used in all emulsions and solutions.

Preparation of Grape seed oil nanoemulsion (GONE).

The nanoemulsion was formulated using 10 % w/w grapeseed oil (GsO), 10% w/w Tween 20, and 80% w/w distilled water. The compounds were mixed at room temperature while continuously stirring at 3000 rpm using a PV-1 Vortex Mixer (Grant Instruments, UK) for 5 min. Then the mixture was ultrasonicated (UP100H – Compact Ultrasonic Laboratory Device, Germany) for 3 min.

HPMC/GONE film preparing.

The film was prepared based on the recipe of Lee with small modifications [3]. HPMC powder (1 g) and glycerol (0,15 g) were dissolved in 100 mL of distilled water at 80°C for 1 h by stirring with magnetic stirrer. The solution was then cooled down to room temperature. Subsequently, Grapeseed oil emulsion was added at 0, 2,5, 5 and 7,5% (v/v) and mixed by ultrasonication for 5 min. Finally, the solution was sonicated for 30 min, cast on a glass petri dishes (d=100 mm), and dried in an oven at 30°C for 48 h (figure 1).

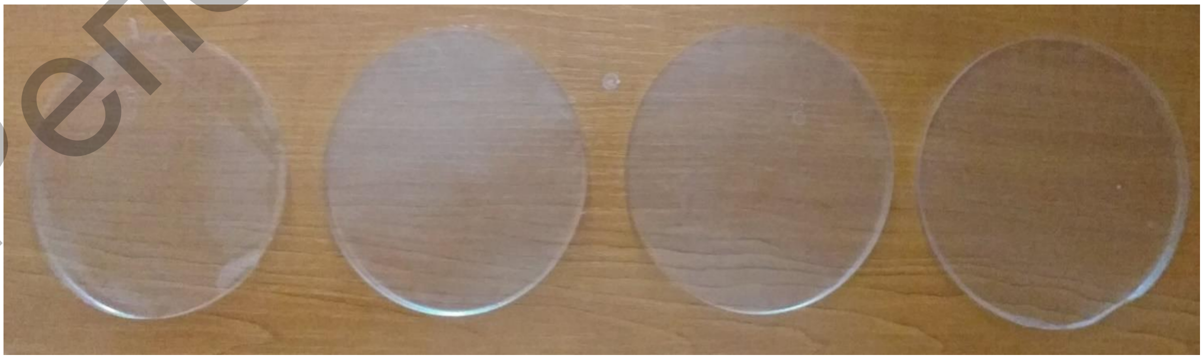


Figure 1 – HPMC/GONE multicomponent films with different GONE concentrations (0%, 2.5%, 5%, 7.5% GONE respectively)

The produced HPMC/GONE multicomponent films were conditioned at 25°C and 50% relative humidity (RH) for 48 h before testing. The prepared films are subsequently called as follows: control (0% GONE), HG-2,5 (2,5% GONE), HG-5,0 (5% GONE), and HG-7,5 (7,5% GONE).

Film thickness.

The film thickness was determined with a digital micrometer (No. 293-5, Mitutoyo, Japan). Ten thickness measurements were randomly taken on each testing sample. The mean values were used to calculate O2 permeability and CO2 permeability and tensile strength.

Surface topography.

A polarizing light microscope (Leica DM1000) equipped with Leica DFC295 Digital Camera was used to observe the morphologies of the films.

Thermal properties.

The crystallinity of the immobilized GsO was examined by applying the DSC method. For this purpose, DSC 204F1 Phoenix (Netzsch Gerätebau GmbH, Germany) was applied. An indium standard (Tm =156,6℃, ΔHm =28,5 J/g) was used for the temperature and heat flow calibration. The measurements are conducted under argon atmosphere at a heating rate of 10℃/min. Since the phase transitions melting and crystallization of the GsO are in the temperature range from 0°C to -75°C [21], initially the sample films were cooled from room temperature to -75 °C, and then heated to room temperature.

Oxygen and carbon dioxide permeability (O_2P and CO_2P).

The gas permeability properties of films towards oxygen (O2) and carbon dioxide (CO2) were determined as reported by Sánchez-Tamayo [18] and according to ASTM D1434 (2009) [4]. Samples were tested using VAC-VBS Gas Permeability Tester, Labthink, China.

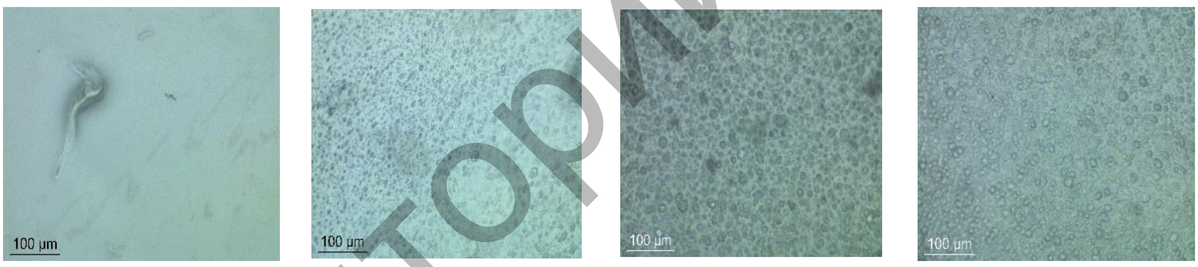
Mechanical properties.

The mechanical tensile properties of the HPMC/GsO films were analyzed by LS 1 (Lloyd Instruments) Universal Testing Machine, according to ASTM D882-18 standard [5]. The films were cut to stripes (wideness: 10 mm, length: 100 mm gap: 50 mm) and fasten the rubber sealed pneumatic clumps. The sample deformation rate was 0,1 mm/s. Seven repetitions were used from each concentrations for the statistics. The tensile curves show the values of modulus of Young’s as the slope of the first linear section, strain and stress at the break point and the work from preload to break.

Result and discussion.

Results of the surface topography.

The surface of the control films (pure HPMC) is smooth and uniform (figure 2) [16]. All other pictures demonstrate a presence of crystal morphological units with different size. The hydrophobic GsO droplets were suspended in a hydrophilic polymer matrix. As the concentration of GsO increases, the films exhibited more irregular appearance and the presence of cavities containing emulsion droplets becomes evident. The surface of the films incorporated with GsO show semi-crystalline structure [10]. The presence of various phases among polymer matrix may be caused the non-uniform film matrix with delaminate structure [15].

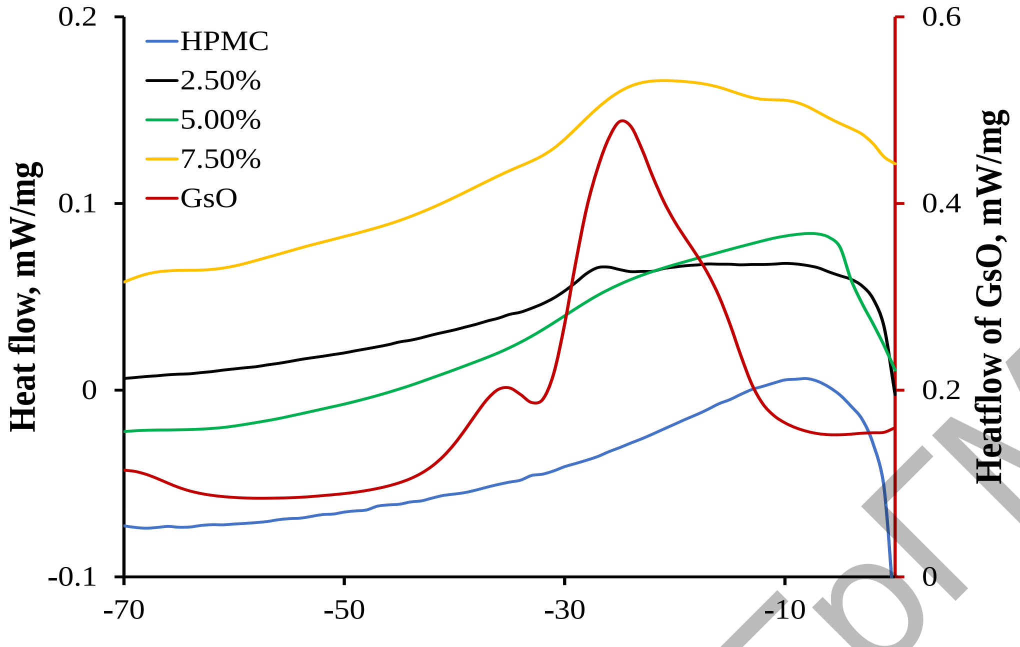


HPMC 2,5% GsO 5% GsO 7,5% GsO

Figure 2 – Surface morphology of HPMC/GONE films

Thermal properties.

The DSC thermograms of pure GsO, HPMC film and HPMC/GONE multicomponent films are presented in figure 3. The DSC curve of GsO is characterized with broad endothermic peak in the range from -50°C to -10°C, which is associated with melting transition. Much smaller endothermic peaks were observed in the thermograms of HPMC/GONE films, which could be interpreted as partly crystal phase of the immobilized GsO.



Temperature, °C

Figure 3 – DSC diagrams of the HPMC/GONE films

Taking in mind the specific enthalpy of fusion of different film that is calculated as the area of the melting peak, one could estimate that the crystallinity of the immobilized GsO varied between 38% for the lowest concentration and 58 % for the highest concentration (table 1). The partial crystallization is due to the presence of a polymer network, which prevents the formation and growth of crystals. These interactions between the HPMC and GsO molecules could be similar like the interactions HPMC and curcumin molecules reported by da Silva [16].

Table 1 – Thermal properties of HPMC/GONE films

Edible coating	GsO concentration in the dry film, %	Experimental enthalpy of fusion, J/g	Enthalpy of fusion, based on the included GsO, J/g	Crystallinity of GsO, %
HPMC (0% GsO)	0	0,00	0,00	0
2,5% GsO	17	3,98	10,54	38
5,0% GsO	25	7,22	15,50	47
7,5% GsO	31	10,87	18,60	58

Gas permeability.

The addition of GsO leads to increase in the O_2 and CO_2 permeability, which is due to interruptions in the polymer network (figure 4.). The crosslinks in the polymer matrix between the HPMC and GsO molecules may increase the relative humidity of the films and make a softer matrix (see later) and increase the gas transition [10].

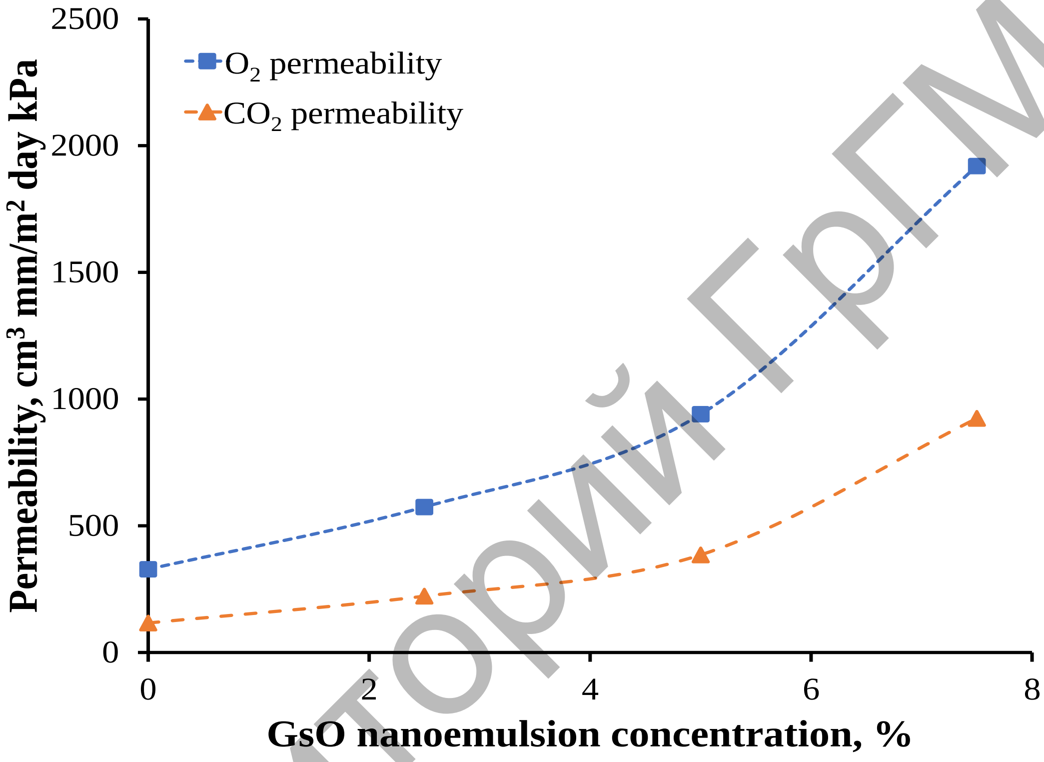


Figure 4 – Gas permeability of the HPMC/GONE films

Mechanical properties.

The mechanical properties of edible films has key importance in their food coating applications [15]. The pure HPMC film is rigid and has high mechanical resistance [5]. An increase in the GsO concentration resulted in a decrease in tensile stress and Young's modulus (figure 5).

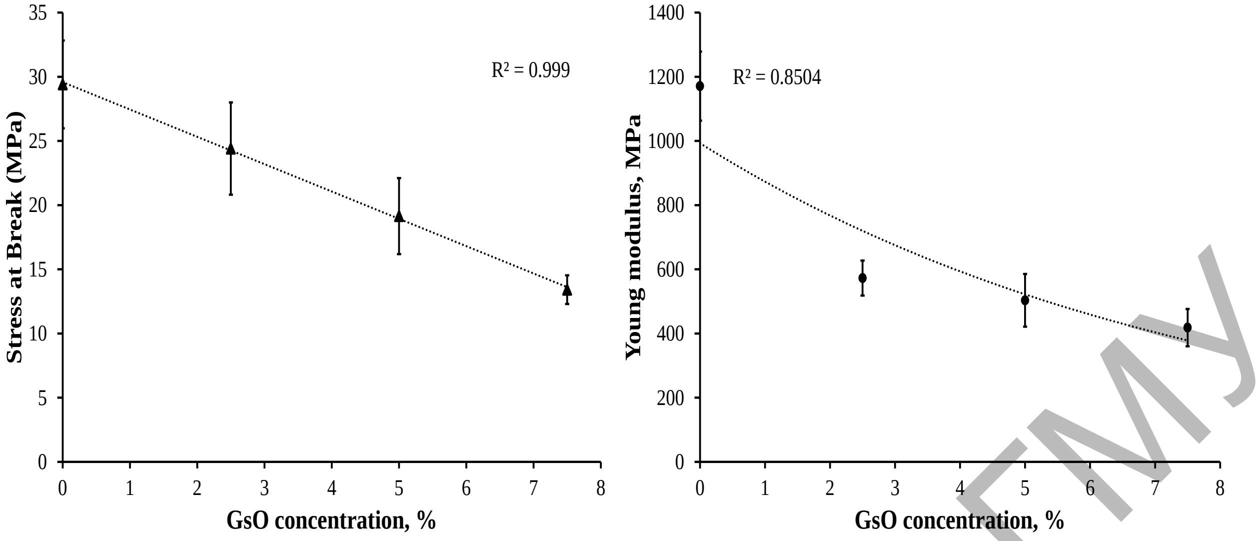


Figure 5 – Tensile stress and Young's modulus of the HPMC/GONE films

Strain at break increases between less than 10% and 30% indicating higher flexibility. It is similar to reported by Patil et al for glycerol nanocellulose films [15]. At the same time the rupture work increases, hence the film toughness increases (Fig 6). The reduction of mechanical strength is probably due to the aggregation of GsO particles which induce phase separation and poor particle distribution, consequently resulting in poor mechanical strength [14].

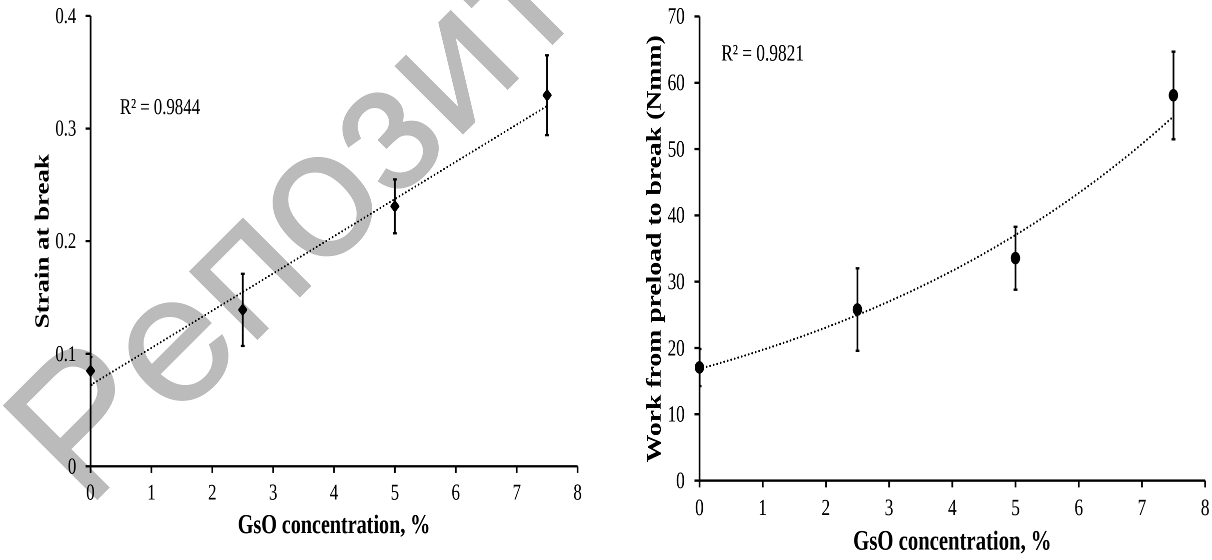


Figure 6 – Strain and rupture work of the HPMC/GONE films

Conclusions.

At higher GsO concentrations irregular appearance of the film surface and the presence of cavities containing emulsion droplets became evident.

Furthermore, gas barrier properties of the composite films (against CO_2 and O_2) were improved when the GsO concentration increased.

The addition of GsO reduced the composite tensile strength.

Based on our experiments the films with 5% emulsion concentration showed optimal properties.

In further experiences, these films or emulsion coatings will be applied to enhance the quality of fruits during refrigerated storage.

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