

ДИНАМИКА ИЗМЕНЕНИЯ ФИЗИКО-ХИМИЧЕСКИХ СВОЙСТВ Rh(III) И Ir(IV)-СОДЕРЖАЩИХ ПЛЕНОК НА ОСНОВЕ ПРИРОДНЫХ ПОЛИСАХАРИДОВ**А.А. Годзишевская, Е.П. Лопашина, А.А. Снегирева, М.Н. Курасова, Т.С. Трифонова**

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Пленки на основе природных полисахаридов уже сегодня широко используются в координационной химии, медицинской химии, экологической химии, фармацевтике. Широкий спектр областей применения данных материалов связан с их высокой экологичностью, биосовместимостью и биоразлагаемостью. Для определения возможности использования данных материалов необходимо охарактеризовать их физико-химические свойства. Хитозан-агаровые пленки были получены путем отливания из раствора, в качестве пластификатора выступал глицерин. В состав пленок входят катионы Rh^{3+} и Ir^{4+} в различных соотношениях: полимерная матрица/катионы металла. Материалы охарактеризованы с помощью ИК-спектроскопии, дифференциально-термического и термогравиметрического анализа (ДТА и ТГА), а также сканирующей электронной спектроскопии (СЭМ). В данной статье рассмотрена динамика изменения механических свойств (предела кратковременной прочности (H/m^2) на протяжении двух месяцев с момента получения материала). Установлено, что природа металлоцентра влияет на механические свойства, так родийсодержащие образцы характеризуются более высокими показателями прочности, чем соответствующие иридийсодержащие. Поведение материалов в водных растворах также является важной характеристикой для определения возможности использования пленок. Динамика изменения степени набухания была изучена в воде, а также соляном буферном растворе ($pH=7,2$). В буферном растворе пленки подвергаются меньшему набуханию, чем в дистиллированной воде. Степень набухания материалов с включением в состав катионов родия(III) больше, чем у пленок с такими же соотношениями полимерной матрицы и катионов иридия(IV). При хранении материалов в стандартных условиях изменяются не только механические свойства, но и степень набухания образцов. С течением времени показатели падают независимо от состава исследуемых образцов.

Ключевые слова: хитозан, агар-агар, родий, иридий, механические свойства, набухание, термическая стабильность

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Rh(III) AND Ir(IV)-CONTAINING FILMS BASED ON CHITOSAN AND AGAR-AGAR: DYNAMICS OF CHANGES IN PHYSICAL AND CHEMICAL PROPERTIES

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Films based on natural polysaccharides are already widely used in coordination chemistry, medicinal chemistry, environmental chemistry, and pharmaceuticals. The wide range of applications of these materials is due to their high environmental friendliness, biocompatibility and biodegradability. To determine the possibility of using these materials, it is necessary to characterize their physicochemical properties. Chitosan-agar films were obtained by casting from solution with glycerol as plasticiser. The films contain Rh³⁺ and Ir⁴⁺ cations in different ratios of polymer matrix and metal cations. The materials were characterised by IR spectroscopy, differential thermal and thermogravimetric analysis (DTA and TGA), and scanning electron spectroscopy (SEM). In this paper, the dynamics of changes in mechanical properties (short-term strength (N/mm²) over a period of two months from the date of production of the material have been considered. It was proven that the nature of the metal center affects the mechanical properties: rhodium-containing samples are characterized by higher short-term tensile strength than the corresponding iridium-containing ones. The behavior of materials in aqueous solutions is also an important characteristic for determining the possibility of using films. The dynamics of the swelling behavior was studied in water as well as in salt buffer solution (pH=7.2). In buffer solution, the films undergo less swelling than in distilled water. The degree of material swelling with inclusion of rhodium(III) cations is higher than that of films with the same ratio of polymer matrix and iridium(IV) cations. When materials are stored under standard conditions, not only mechanical properties, but also the degree of swelling of the samples change. Over time, the values fall regardless of the composition of the samples under study.

Keywords: chitosan, agar-agar, rhodium, iridium, mechanical properties, swelling, thermal stability

INTRODUCTION

Modern environmental requirements for the production and quality of products of various industries dictate the need to develop new materials and technologies that are environmentally friendly and do not harm living organisms. In this regard, the application of natural biopolymers, especially in the food industry [1, 2] and in medicine [3-5], is attracting a lot of attention. That is why the research of properties and possibilities of polymer films based on natural high molecular weight compounds such as chitosan, agar-agar and gelatin is quite a rapidly developing direction in the chemistry of polymeric materials.

Today chitosan films are already widely used as food packaging [6, 7], antifungal coatings [8, 9], catalysts in various organic syntheses [10, 11], but these

applications do not exhaust the possibilities of this aminopolysaccharide. Chitosan and its derivatives exhibit antiproliferative, antibacterial and antioxidant activity, especially in tandem with various metal ions (Fe³⁺, Ag⁺, Zn²⁺, platinum group metals, etc.) [12-14]. In addition to the biological characteristics of chitosan-based compounds, the mechanical characteristics of these materials (thickness, degree of swelling, thermal stability, mechanical strength and elasticity, oxygen transmission rate, vapour permeability (barrier properties) also play an important role. It is known that as a result of the inclusion of various metal cations in the chitosan matrix, the mechanical properties of the resulting films are significantly improved [15, 16]. Platinum metals (in particular Rh(III) and Ir(IV)) have high antiproliferative, antimicrobial and catalytic activities

[17-20], therefore the study of the effect of metal cations on the mechanical properties of polymer films is relevant concerning their application. Moreover, the addition of natural polysaccharides (agar-agar) to the chitosan matrix should increase the short-term strength of the material, as well as its elasticity and adhesion [21].

A comprehensive study of the physicochemical characteristics of films based on natural polysaccharides of chitosan and agar-agar with the inclusion of platinum metal cations (Rh(III) and Ir(IV)) into their composition allows to determine the area of use of the resulting materials, as well as their shelf life. This work presents the results of investigation of thermal stability, mechanical properties and behavior of materials in aqueous solutions, and considers the influence of the composition of materials on the morphology of the film surface.

EXPERIMENTAL

Preparation of chitosan-agar films

0.1 g of agar-agar was poured into 10 ml of distilled water and left under constant stirring at 150 °C until complete dissolution. 0.4 g of chitosan was dissolved in 15 ml of 1% acetic acid solution with addition of 0.09 g of glycerol under constant stirring and heating to 60 °C for 20 min. 1, 2, 5 or 10 ml of 0.01 M metal salt solution (RhCl₃ and Na₂IrCl₆) was added to the resulting chitosan solutions. The chitosan-salt solutions were degassed in a centrifuge for 10 min at 3200 rpm. The chitosan mixture was added to the agar-agar solution and molded into Petri dishes. The molds were left for a week until constant mass of films.

Table 1

Ratio of starting reagents
Таблица 1. Соотношение исходных реагентов

Encryption	m (Ch), g	m (A), g	m (Gly), g	V(Rh ³⁺)/ (Ir ⁴⁺), ml
ChAIr1	0.4	0.1	0.09	1.0
ChAIr2				2.0
ChAIr5				5.0
ChAIr10				10.0
ChARh1				1.0
ChARh2				2.0
ChARh5				5.0
ChARh10				10.0

Swelling ratio estimation and water solubility analysis

Pre-weighed film (1 cm × 1 cm) was immersed into 30 ml of distilled water and salt buffer solution (pH = 7.2) at room temperature for certain time intervals [22-23]. The water was then removed from the film surface with filter paper and the swollen film was weighed. The degree of film swelling was calculated as follows:

$$A (\%) = \frac{W_2 - W_1}{W_1} \cdot 100$$

where W₁ and W₂ are the initial and swollen weights of the film, respectively.

Methods of analysis

IR spectroscopy

The infrared absorption spectra of the initial substances and obtained complexes were determined using IR Spirit FTIR spectrophotometer on an ATR (diamond) accessory in the range from 600 to 4000 cm⁻¹.

Differential thermal and thermogravimetric analysis

DTA and TGA of obtained compounds were recorded with SDT Q600 thermal analyser (T.A. Instruments, USA). Ceramic crucibles, platinum-platinum-rhodium thermocouples were used for analysis. The heating was performed from 20 °C to 800 °C. The heating rate was 10 deg/min.

Scanning electron spectroscopy (SEM)

SEM images were acquired on a Quattro S electron microscope, 15kV voltage, SEM detector, electron beam 30, in deep vacuum. The tested samples were deposited on a 20 nm thick platinum substrate. The imaging angle was 30°.

Mechanical properties

Mechanical properties were evaluated using a REM-I electronic tensile testing machine (Russia), strain rate of 5 mm/min at 25 °C. The measurements were carried out in three steps.

DISCUSSION OF THE RESULTS

IR spectroscopy

The spectra of chitosan-agar films show a broad absorption band in the range from 3500 to 3100 cm⁻¹, in which the valence vibration bands of -O-H (chitosan, agar-agar) and -N-H (chitosan) bonds are detected, which confirms the presence of intermolecular hydrogen bonds between the functional groups of the polymer, as well as the presence of water molecules in the polymer structure. The shift of the band corresponding to valence vibrations of C-H bonds (2858 cm⁻¹ – chitosan-agar matrix, 2287 cm⁻¹ – metal-containing films) confirms the interaction of the polymer matrix with metal cations.

Differential thermal and thermogravimetric analysis

Analysis of TG and DTA curves of chitosan-agar film confirmed the presence of weakly-bound water in the polymer film, which is removed at temperatures up to 152 °C. This process is accompanied by a mass loss of 10.55%. The exothermic effect at 522.65 °C refers to the burnout of the organic part of the film and is accompanied by a mass loss of 95.76%.

Table 2

The results of DTA and TGA
Таблица 2. Результаты ТГ и ДТА

Encryption	T _(dehydration) , °C	Weight change, %	T _(combustion of molecule's organic part) , °C	Weight change, %	Residual weight, %
ChA	152.32	10.55	522.65	95.76	4.24
ChAIr10	148.18	12.09	525.57; 568.52	91.93	8.07
ChARh10	139.23	11.39	624.43	95.32	4.68

The addition of metal cations does not significantly change the nature of thermal degradation of the film. The content of coordinated and weakly-bound water slightly increases (11-12% mass loss, water elimination occurs in the range of 140-150 °C). A shift of the exothermic effect corresponding to the destruction of the organic content of the film to 525.57-568.52 °C and its broadening is observed. Rhodium-containing samples are characterized by an even higher temperature (624.43 °C) narrow exothermic effect accompanied by almost complete mass loss (95.32%).

Thus, it can be concluded that the thermal stability of polymer films depends on the nature of the cation introduced. Rhodium-containing films are characterized by a greater thermal stability than the corresponding iridium-containing films.

Scanning electron spectroscopy

The surface morphology of chitosan-agar films qualitatively depends on the presence and concentration of Rh³⁺ and Ir⁴⁺ cations in the material composition. The metal-containing films are characterized by a smoother surface (smaller number and larger size of ridges) than the chitosan-agar matrix without the presence of metal cations. With an increase of metal concentration in the films, regardless of the nature of the metal center, the number of cristae increases.

Mechanical properties

With increasing metal concentration in the films, the mechanical properties increase and depend on the nature of the metal center. Thus, rhodium-containing films are characterized by higher values of maximum film load before rupture and short-term tensile strength.

The short-term tensile strength of metal-containing samples grows for a month and then gradually decreases, which may be related to the film drying and increase of crystallinity of the obtained materials, i.e. aging of the samples. Rhodium-containing films are characterized by higher mechanical properties than the corresponding iridium-containing ones.

The degree of swelling

The degree of swelling of chitosan-agar metal-containing films was determined in water and salt buffer solution (pH = 7.2). Swelling of chitosan-agar films is limited in contrast to pure chitosan films without metal introduction, which swells unlimitedly until the sample is destroyed. The introduction of metal into the investigated films reduces the degree of swelling of the corresponding sample compared to the pure matrix sample. Rhodium-containing samples are characterized by lower swelling degree than the corresponding iridium-containing samples.

The medium of the experiment affects the results obtained. The degree of swelling of chitosan matrix in salt buffer solution containing Na⁺, K⁺, Cl⁻, Ca²⁺, Mg²⁺, HPO₄²⁻, H₂PO₄⁻ ions is by 2.4 times less than in distilled water. The influence of the presence of the above-mentioned ions in the buffer solution on the degree of swelling of metal-containing films is insignificant, but the parameters in the salt buffer solution are also 1.2 times lower than for the corresponding samples in the experiment conducted in distilled water.

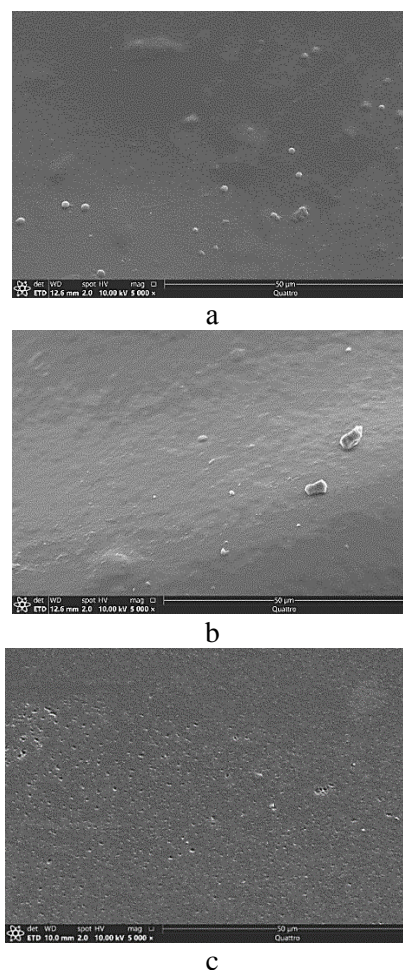


Fig. 1. SEM-imaging: (a) ChARh10, (b) ChAIr10, (c) ChA
Рис. 1. СЭМ-снимки (a) ChARh10, (b) ChAIr10, (c) ChA

Table 3

Mechanical properties of metal-containing chitosan-agar films

Таблица 3. Механические свойства металлосодержащих хитозан-агаровых пленок

Encryption	Short-term tensile strength, Н/мм ²			
	1st day	14 th day	28th day	56th day
<i>ChAlr1</i>	7.86±0.04	6.79±0.03	33.09±0.17	32.82±0.16
<i>ChAlr2</i>	20.57±0.10	23.99±0.12	24.19±0.12	29.99±0.15
<i>ChAlr5</i>	20.95±0.10	24.42±0.12	29.33±0.15	20.50±0.10
<i>ChAlr10</i>	19.35±0.10	26.17±0.13	27.78±0.14	33.94±0.17
<i>ChARh1</i>	33.08±0.17	42.54±0.21	47.37±0.24	32.63±0.16
<i>ChARh2</i>	38.61±0.19	33.89±0.17	38.56±0.19	37.63±0.19
<i>ChARh5</i>	38.80±0.19	15.65±0.08	23.56±0.12	41.92±0.21
<i>ChARh10</i>	34.16±0.17	23.79±0.12	18.29±0.09	27.30±0.14

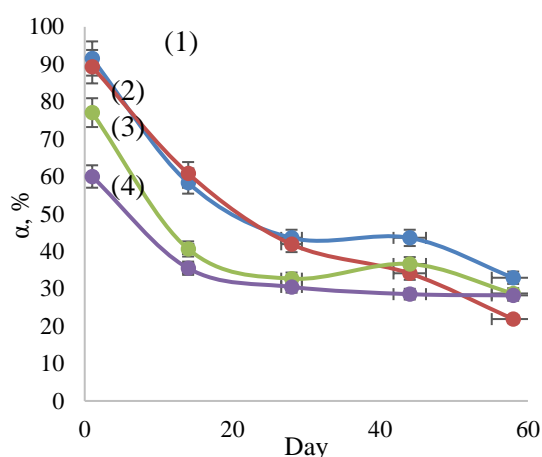


Fig.2. Dependence of the maximum degree of swelling on the shelf life of materials: (1) – ChAlr in water; (2) – ChARh in water; (3) – ChAlr in buffer; (4) – ChARh in buffer

Рис.2. Зависимость максимальной степени набухания от срока хранения материалов: (1) – ChAlr в воде; (2) – ChARh в воде; (3) – ChAlr в буфере; (4) – ChARh в буфере

The dynamics of changes in the degree of swelling of polymer films was being determined for two months, with a 14-day interval.

The obtained materials were stored under standard conditions with access to air. Aging of materials leads to a lower degree of swelling of both chitosan-agar matrix and metal-containing polymer films. However, over time the rate of changes in studied conditions has almost no dependence on nature of the metal center.

Table 4

Maximum degree of swelling of the chitosan-agar film within two months

Таблица 4. Максимальная степень набухания хитозан-агаровой пленки в течение двух месяцев

Day	α_{\max} , % in H ₂ O	α_{\max} , % in buffer
1	376.19	143.48
14	145.83	77.78
28	100.00	70.21
44	75.00	54.17
58	52.94	57.97

CONCLUSION

The results of studies show that rhodium-containing samples are characterized by greater thermal stability, higher indices of mechanical properties and swelling degree than the corresponding iridium-containing samples and the chitosan-agar matrix. However, such dependence is not evident in the research of the behavior of materials in water solutions. Thus, polymer matrix without metal cations in their structure is distinguished by the maximum indices of swelling degree while the rhodium-containing films are distinguished by the minimum ones.

The surface morphology of the films does not depend on the nature of the introduced metal centre. Aging of materials is accompanied by a decrease in the values of mechanical properties, as well as the degree of swelling. However, the changes are insignificant, which confirms the possibility of further use of the obtained materials in various spheres.

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