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Properties of Plant Analogs of Pharmaceutical Gelatin for Shells of Soft Capsules

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Abstract

It was found that the thickness and the strength characteristics of capsule shells based on gelatin are higher than that of films based on carrageenan. The reduction in the thickness and strength properties of the capsule shell with increasing content of plasticizer in the composition of glycerin was noted. It was shown that the melting point of capsule shells based on carrageenans is higher than that for the capsule shells based on gelatin and starch. According to the results of differential scanning calorimetry, it was found that the capsule shells based on carrageenan are characterized by the maximum value of the melting temperature (70.4°C) and the capsule shells based on corn starch by the minimum (66.4°C). Shells of capsules based on gelatin have the highest value of glass transition temperature (155.8°C), whereas the capsule shells based on carrageenans the lowest (155.8°C). Analysis of the dielectric properties of capsule shells has shown that with increasing frequency there is a reduction of the dielectric constant for all samples being analyzed.

Keywords

Plant analog; Gelatin; Shell; Capsule

Introduction

Natural hydrocolloids (polysaccharides) are promising materials for the production of biodegradable shells of capsules [1]. Thus, R.P.H. Brandelero in his work studied the structural and mechanical properties of biodegradable capsule shells that are starch-based, obtained by blown extrusion [2]. It has been reported [3] about the possibility of obtaining nanocomposite biodegradable capsule shells based on nanocrystalline cellulose, modified by alginate. Barrier properties of biodegradable, composite capsule shells [4] based on the mixture of kappa-carrageenan and pectin were studied in the paper prepared, and it characterized biodegradable capsule shells based on methylcellulose [5,6].

A number of studies of the past decades show the use of flour as a raw material for the production of biodegradable packaging capsule shells. Rayas and Hernarndez obtained edible capsule shells from three kinds of serum flour [7,8], and used whole soybean flour and apple pectin [9]. Tapia-Blarcido et al. and Colla et al. used amaranth flour as raw material to produce edible capsule shells [9].

Ghanbarzadeh et al. suggested a method of modifying the mechanical and barrier properties of the edible capsule shell based on starch, comprising the use of citric acid and carboxymethylcellulose [10,11].

The objective of the work is the comparative analysis of physical, mechanical, thermal, and electrical properties of biodegradable capsule shells derived from natural polysaccharides (corn starch, amylase starch, carrageenans), and gelatin protein.

Materials and Methods

Preparation of the capsule shell

The studied capsule shells were conditionally divided into three groups depending on the composition; the composition of capsule shells is shown in Table 1. Shells of capsules No. 1 relate to group I, capsule shells No. 2-5 to group II, and capsule shells No. 6-10 to group III.

Capsule shells No. 1 were dried at 80°C, and capsule shells No. 2-10 were dried at room temperature.

Characteristics of capsule shells

Thickness of capsules shells

The capsule shell thickness was measured using a micrometer. At least 10 parallel measurements on 5 different sites of capsule shells and the average value were carried out and calculated.

Mechanical properties

Load at break, elongation at break, elastic modulus, and tensile yield strength of the capsule shells were determined using a desktop electromechanical test machine, Instron 3369. Measurement parameters are as follows: effort of 1 kN (225 lbf), vertical test space of 1,067 mm (42 inches), load measurement error of ±0.5%, crosshead speed of 100 mm/min, distance between grips of 15 mm, specimen width of 10 mm.

Thermal properties

Melting point determination of the capsule shells was performed by differential scanning calorimetry (DSC) on the device 401 Netzsch Poemix DSC at a heating rate of 10°C/min in an air atmosphere. Crystallization temperature and glass transition were determined using the combination of simultaneous thermal analysis TG-DTA and DC quadrupole mass spectrometer QMC 403 C under argon.

Dielectric properties

The measurement of dielectric properties of the capsule shell samples were performed by standard capacitor method using the precision-measuring instrument LCR Agilent E4980A.

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Ingredients	Quantity, wt. %									
	1	2	3	4	5	6	7	8	9	10
Corn starch	33.5	–	–	–	–	–	–	–	–	–
Glycerol	10.0	10.0	5.0	10.0	5.0	10.0	11.5	12.0	11.5	11.463
Water	66.5	66.5	50.0	40.0	35.0	66.5	70.0	65.67	55.0	65.0
Gelatin	–	33.5	45.0	50.0	60.0	–	–	–	–	–
Kappa-carrageenan	–	–	–	–	–	3.0	3.0	2.0	3.0	3.0
Iota-carrageenan	–	–	–	–	–	0.5	0.5	0.33	0.5	0.5
Gelamil 308	–	–	–	–	–	20.0	–	–	30.0	20.0
Amylase starch	–	–	–	–	–	–	15.0	20.0	–	–
Potassium chloride	–	–	–	–	–	–	–	–	–	0.02
Propyl parahydroxybenzoate	–	–	–	–	–	–	–	–	–	0.0035
Methyl parahydroxybenzoate	–	–	–	–	–	–	–	–	–	0.014

Table 1: Composition of the investigated capsule shells

The contact areas are measuring capacitor plates of rectangular area of 19 × 19 mm size. They are made with a conductive, gel-type Kontactol. The measured quantity is the capacitance and loss tangent. The value of the dielectric constant was calculated by the formula for a flat capacitor.

Statistical processing

All experiments were performed at least in triplicate. Statistical analysis was carried out by methods of mathematical statistics. Confidence level for the statistical analysis was taken to be 0.95.

Results and Discussion

Physical and mechanical properties

Measurement results of physical and mechanical properties of the investigated capsule shells are shown in Table 2.

From Table 2, it follows that the maximum thickness is characterized by capsule shells No. 2 (1.24 mm in the transverse direction, 1.50 mm in the longitudinal direction), 6 (1.30 mm), 5 (1.20 mm), and 4 (1.10 mm); the minimum thickness is for samples No. 7 (0.60 mm) and 8 (0.61 mm). It can be concluded that with increasing content of plasticizer glycerol in the composition of capsule shells, their thickness decreases. Furthermore, it is seen that the thickness of capsule shells based on gelatin is more than the thickness of capsule shells based on carrageenan.

Maximum values of load at break are characterized by samples of capsule shells No. 2 (116.4 N in the longitudinal direction, 95.6 N in the transverse direction), 5 (91.2 N), and 9 (82.5 N in the longitudinal direction, 68.4 N in the transverse direction). The minimum size of the load at break was observed for the samples of capsule shells No. 7 (46.3 N), 3 (42.9 N), and 8 (40.3 N). Thus, the load at break is more for gelatin-based capsule shells than for capsule shells based on carrageenan. With increasing concentration of plasticizer glycerol in the capsule shells' composition, the load at break decreases. In general, the data on strength included in the measurement results correlate with the thickness of capsule shell: the thinner the film is, the lower the load must be applied to rupture.

Maximum elongation at break is observed for the capsule shell samples No. 2 (42.0 mm in the transverse direction, 37.5 mm in the longitudinal direction), 5 (40.2 mm), and 4 (38.7 mm); the minimum is observed for samples 6 (21.9 mm) and 7 (21.9 mm). Consequently, the greater magnitude of elongation at break is characterized by gelatin capsule shells in comparison with capsule shells based on carrageenan. The results obtained are correlated with the thickness measurement data, and load at break: the thicker the capsule shell is, the larger the load at break and elongation at break.

The highest value of elastic modulus is characterized by the capsule shell sample No. 9 (130.8 MPa in the transverse direction, 144.3 MPa in the longitudinal direction); the low elastic modulus is observed for

Sample no.	Longitudinal/transverse measurement	Sample thickness, mm	Load at break, N	Elongation at break, mm	Elastic modulus, MPa	Tensile yield strength, MPa
1	Transverse	0.92 ± 0.09	61.9 ± 6.2	24.9 ± 2.5	98.4 ± 9.8	8.4 ± 0.8
2	Transverse	1.24 ± 0.12	95.6 ± 9.6	420 ± 4.2	34.6 ± 3.5	7.4 ± 0.7
	Longitudinal	1.50 ± 0.15	116.4 ± 11.6	37.5 ± 3.8	40.3 ± 4.0	8.4 ± 0.8
3	Transverse	0.81 ± 0.08	42.9 ± 4.3	28.9 ± 2.9	41.9 ± 4.2	8.3 ± 0.8
4	Longitudinal	1.10 ± 0.11	70.6 ± 7.4	38.7 ± 3.9	51.4 ± 5.1	7.8 ± 0.8
5	Transverse	1.20 ± 0.12	91.2 ± 9.1	40.2 ± 4.0	33.7 ± 3.4	–
6	Longitudinal	1.30 ± 0.13	54.8 ± 5.5	21.9 ± 2.2	47.6 ± 4.8	6.4 ± 0.6
7	Transverse	0.60 ± 0.06	45.3 ± 4.6	21.9 ± 2.2	26.8 ± 2.6	6.0 ± 0.6
8	Longitudinal	0.61 ± 0.06	40.3 ± 4.0	25.6 ± 2.6	80.1 ± 8.0	5.6 ± 0.6
9	Transverse	0.87 ± 0.09	68.4 ± 6.8	28.0 ± 2.8	130.8 ± 13.1	6.8 ± 0.7
	Longitudinal	0.91 ± 0.09	82.5 ± 8.3	22.7 ± 2.3	143.3 ± 14.4	8.6 ± 0.9
10	Transverse	0.86 ± 0.09	58.9 ± 5.9	21.8 ± 2.2	91.7 ± 9.2	8.1 ± 0.8
	Longitudinal	0.86 ± 0.09	68.3 ± 6.8	33.3 ± 3.3	71.9 ± 7.2	9.6 ± 0.9

Table 2: Physical and mechanical properties of the investigated capsule shells

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Sample no.	Group no.	Tm, °C Single measurement	Tm, °C The mean value for the group	Tg, °C Single measurement	Tg, °C The mean value for the group
1	II	70.4 ± 3.5	69.5 ± 3.5	151.2 ± 7.6	155.8 ± 7.8
2		66.8 ± 3.3		153.3 ± 7.7	
3		70.4 ± 3.5		159.8 ± 8.0	
4		70.3 ± 3.5		158.0 ± 8.0	
5	I	66.4 ± 3.3	66.4 ± 3.3	154.7 ± 7.7	154.7 ± 7.7
6	III	76.0 ± 3.9	70.4 ± 3.5	–	149.0 ± 7.5
7		71.8 ± 3.6		156.9 ± 7.8	
8		66.7 ± 3.3		146.0 ± 7.3	
9		68.9 ± 3.4		149.8 ± 7.5	
10		67.6 ± 3.4		143.4 ± 7.2	

Table 3: Results of measurement of the melting temperature (Tm) and glass transition (Tg) of capsule shells by differential scanning calorimetry

capsule shell sample No. 7 (25.8 MPa). Thus, the presence of Gelamil 308 in the composition of capsule shells increases the elastic modulus. In addition, the high value of elastic modulus (98.4 MPa) is characterized by sample No. 1, containing corn starch.

Yield strength under tension for test capsule shell samples varies in a small range from 5.6 to 9.3 MPa. Thus, the minimum value of the tensile yield strength was observed for samples No. 8 (5.6 MPa) and 7 (6.0 MPa), and the maximum was for sample No. 10 (8.1 MPa in the transverse direction, 9.3 MPa in the longitudinal direction).

Thermal properties

Results of DSC are shown in Table 3.

From Table 3, it follows that the maximum value of the melting temperature (70.4°C) is characterized by the capsules belonging to group III (based on carrageenans) and minimum (66.4°C) by capsule shells of group I (based on corn starch). Capsule shells of test group II (gelatin-based) is in intermediate position; the average melting point of them was 69.5°C.

The capsule shells of group II (gelatin-based) had the highest value of the glass transition temperature (155.8°C); the lowest value of the glass transition temperature (155.8°C) was typical for the capsule shells of group III.

Based on an analysis of physical and mechanical properties of degradable capsule shells, the greatest interest for further investigation was for the samples of capsule shells No. 2, 5, and 9, as characterized by the maximum values of strength characteristics. In this regard, the further thermogravimetric studies (Figures 1-3) were conducted.

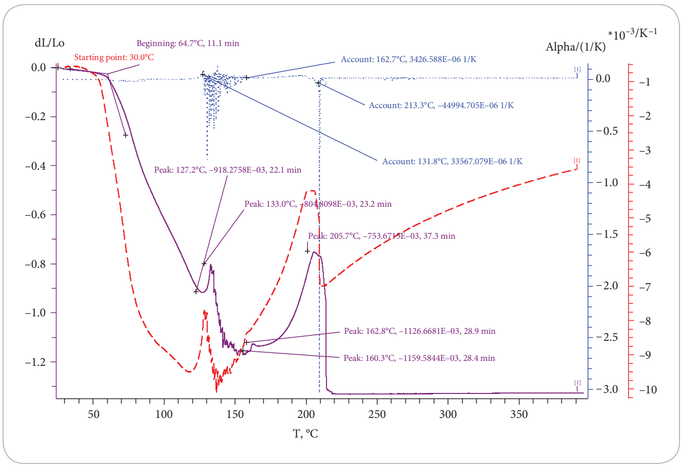

Figure 2: The thermogram for capsule shell sample No. 5

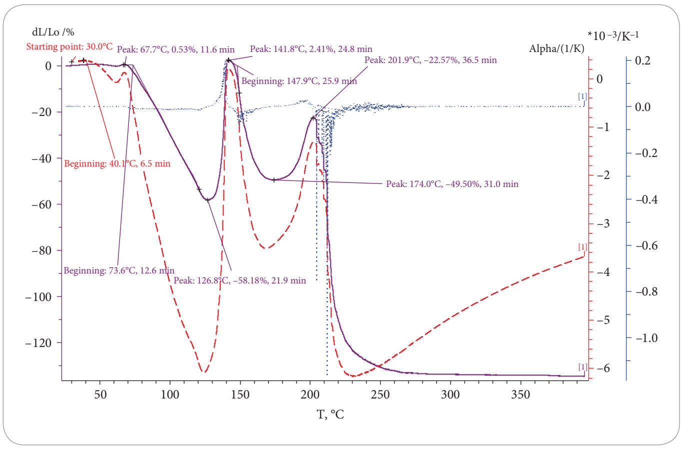

Figure 1: The thermogram for capsule shell sample No. 2

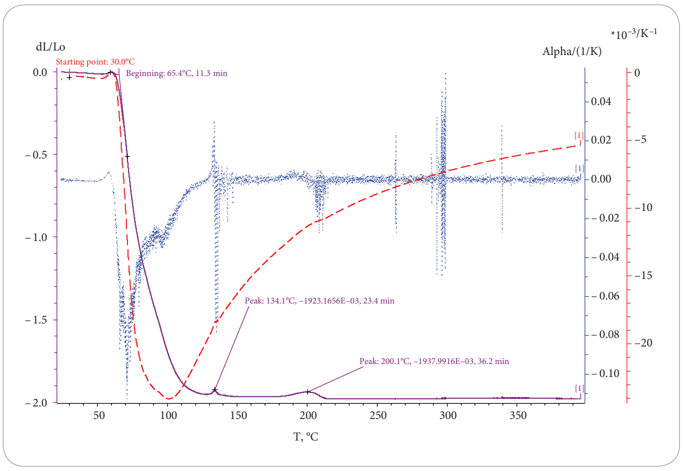

Figure 3: The thermogram for capsule shell sample No. 9

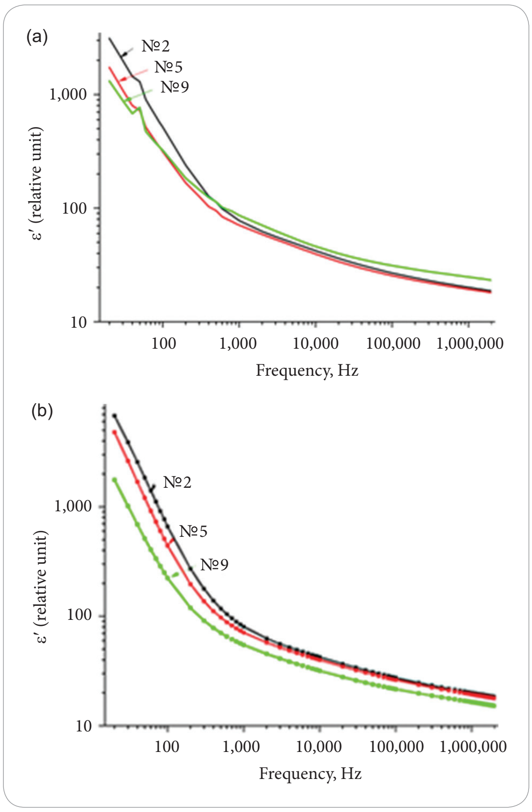

Figure 4: Average values of dependence on the frequency of the dielectric constant without (a) and with (b) a metal coating samples No. 2, 5, and 9

The data of Figures 1-3 show that endothermic melting peak is observed in all the test samples. Initial temperature ranges from 54 to 60°C. For the samples No. 5 and 9 on the DSC curve, inflection, corresponding to the transition into high state, is also presented. The glass transition temperature for these samples was 154-159°C. There were no changes for sample No. 2 on the thermoanalytical curve that can be associated with the transition to high-elastic state.

For all the test samples in the gravimetric curve, the mass loss close to 200°C is observed, followed by several endothermic peaks on the DSC curve. Thermal decomposition of the sample is conducted by several steps that can be seen by a large number of peaks and endothermic change in the slope of the curve gravity. Complete degradation of the polymer occurs after 400°C.

Dielectric properties

Investigation of dielectric properties of capsule shells was performed on samples No. 2, 5, and 9 from the different groups. Spectra of capsule shells' permittivity are shown in Figure 4, which shows an increase in the frequency of a reduction in the dielectric constant for all the analyzed samples. It can be seen that the maximum reduction in permittivity occurs in the frequency range from 5 to 1,000 Hz, while in the frequency interval between 1 kHz and 1 MHz, this magnitude decreases slightly.

Conclusions

The physical and mechanical, thermal, and dielectric properties of the biodegradable capsule shells, which are based on natural polysaccharides (starch, carrageenan) and gelatin, were studied.

It was found that the thickness of the capsule shell based on gelatin is more than the thickness of the films based on carrageenans; the thickness of the capsule shell decreases with increasing content of plasticizer, which performs the function of glycerol.

It has been shown that the higher magnitude of load at break and elongation at break are characterized by gelatin capsule shells as compared with capsule shells based on carrageenan. As the plasticizer concentration increases in the capsule shell composition, the load at break and elongation at break decrease.

According to the results of DSC, it was found that the maximum value of the melting temperature (70.4°C) are characterized by the capsule shell based on carrageenan and minimum (66.4°C) by the capsule shell based on corn starch. Capsule shells based on gelatin have a glass transition temperature of the highest value (155.8°C), whereas the capsule shells based on carrageenans have the lowest (155.8°C).

Analysis of the dielectric properties of the capsule shells has shown that with increasing frequency there is a decrease of the dielectric constant for all samples analyzed; the maximum reduction of this magnitude occurs in the frequency range from 5 to 1,000 Hz.

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