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STUDY OF HYBRID HUMIC ACIDS MODIFICATION OF ENVIRONMENTALLY SAFE BIODEGRADABLE FILMS BASED ON HYDROXYPROPYL METHYL CELLULOSE

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Abstract. The possibility of increasing the strength and operational properties of ecologically safe biodegradable polymeric materials based on hydroxypropyl methyl cellulose by using its modification with the different types of humic acids (HAs) from lignite is considered. Hybrid ecologically safe high-strength films with antibacterial properties were obtained for the first time. Physicochemical studies and IR spectroscopy confirmed the development of hybrid structures of hydroxypropyl methyl cellulose, modified with the different types of HAs. Changes in water absorption, tensile strength, relative elongation at break, and time of mold appearance for the environmentally safe biodegradable polymeric materials based on hydroxypropyl methyl cellulose were revealed depending on the content of the different types of humic acids. It was also shown that the hybrid modification of hydroxypropyl methyl cellulose with the different types of HAs allows preserving the biodegradability of the films along with imparting the antibacterial properties. The developed ecologically safe biodegradable films with antibacterial properties based on hydroxypropyl methyl cellulose and HAs, in terms of their operational characteristics, are superior to the known similar biodegradable films based on natural biopolymers.

Keywords: environmentally safe, biodegradable films, hydroxypropyl methyl cellulose, antibacterial properties, humic acids.

1. Introduction

The modern world is a high-tech, comfortable, and dynamic human environment, which is created due to the use of a large number of various polymers and plastics. In fact, modern man cannot fully exist without their use, but, along with uniqueness and irreplaceability, polymers and plastics have a significant drawback - the generation of a large amount of waste that pollutes the environment.[1] Thus, the modern and most significant trend in the field of production, processing, and applications of plastics is the use of biodegradable polymers and polymeric materials, which, being durable during their use, are capable of destruction under the influence of environmental factors.[2-7] Some examples of such materials include biodegradable films based on natural polymers – starch, cellulose, chitosan, *etc.*[8,9] Currently, a wide range of biodegradable packaging films for food and other products are obtained from these materials.[10-13] Nevertheless, packaging biodegradable films with antibacterial properties for long-term storage of food are still not so common due to their rather low operational characteristics.[14,15] To this end, preparation of biodegradable films with antibacterial properties for food packaging is still of great interest. The production of packaging biodegradable films with antibacterial properties has been recently reported in the literature. For example, starch or polylactic acid-based films, which increase the shelf life of green pepper and minimally processed lettuce, have been developed.[16] It has been also found that mixing starch with alginate allows obtaining films that are more resistant to stretching, less permeable to water vapor and less hydrophilic than films containing only starch.[17]

Taking into account the natural antibacterial and antimicrobial properties as well as the relative ease of obtaining humic acids (HAs) – lignite derivatives – they can be an ideal ingredient for the modification of natural polymers in the form of cellulose ethers in order to develop biodegradable biopolymer films with enhanced antibacterial properties.[18,19] In our previous works, the hybrid environmentally safe biodegradable films based on polyvinyl alcohol[20] and hydroxypropyl methyl cellulose[21] modified with HAs from Ukrainian brown coal have been developed and characterized. In these works, the possibility of developing and/or improving the antibacterial properties of the studied polymers has been demonstrated.

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However, both the mechanism of the modification of hydroxypropyl methyl cellulose with HAs and the impact of HAs on the performance characteristics of the hybrid environmentally safe biodegradable films (HESBFs) have not been clarified.

Hence, this study aims at: (i) investigating the physicochemical features of the hybrid humic acids modification of the environmentally safe biodegradable films based on hydroxypropyl methyl cellulose and (ii) determining the effect of humic acids modification on the strength and performance characteristics of hydroxypropyl methyl cellulose-based HESBFs.

2. Experimental

2.1. Materials

Hydroxypropyl methyl cellulose (Walocel™) from Dow Corning (USA) was used as received. Citric acid (99.88 %) was used as a crosslinking catalyst.

Table 1 shows the characteristics of the lignite experimental samples with different metamorphism degrees, from which HAs have been produced. HA obtained during the extraction of lignite with an alkaline solution of sodium pyrophosphate followed by extraction with a 1% solution of sodium hydroxide and precipitation with mineral acid were used as hybrid modifiers. Humic acids are known to be generally safe for use in humans, as these cause no toxicity issues and is suggested as a potential safe natural active substance and largely nontoxic and nonteratogenic.[22,23]

2.2. Methods

The hybrid ecologically safe biodegradable materials in the film form based on hydroxypropyl methyl cellulose were obtained by the watering method from hydroxypropyl methyl cellulose solutions (2 wt. %) by dissolving the polymer in distilled water at a mass ratio of hydroxypropyl methyl cellulose : water as 2:100 at 363–373 K. Afterwards, 0.03 wt.% of citric acid – a crosslinking catalyst – was added to the obtained hydroxypropyl methyl cellulose solutions. To analyze the properties, hydroxypropyl methyl cellulose solutions were obtained at different concentrations of humic acids (5, 10, and 15 wt. %).

FTIR spectra were recorded on an IR spectrophotometer SPECORD 75 UR at 293–298 K in the wavenumber range of 4000–500 cm^{-1} under the following conditions: slit – 3, recording time – 13.2 min., time constant – 1 sec.

Determination of water absorption of HESBFs in cold water was performed according to ISO 62:2008.

The tensile strength properties of the HESBFs were determined according to ISO 527-2:2021. The tests were carried out on an IR 5040-5 tensile machine in the uniaxial tension mode at a temperature of 295 K. The speed of testing samples was 25 mm/min. The relative elongation at break (%) and tensile strength (MPa) were determined.

Table 1. Technical analysis of lignite

Lignite sample (No. HA)	Proximate analysis, wt. %			
	W^a (%)[a]	A^d (%)[b]	S^d_t (S^{daf}_t) (%)[c]	V^{daf} (V^d) (%)[d]
No. 1 (HA 1)	16.8	48.7	2.08 (2.50)	56.7 (29.1)
No. 2 (HA 2)	8.1	8.3	1.72 (1.87)	47.7 (43.7)
No. 3 (HA 3)	30.6	36.7	2.78 (4.00)	63.0 (43.7)

[a] W^a is the moisture in analytical state.
[b] A^d is the ash content in dry state.
[c] S^d_t (S^{daf}_t) is the total sulfur content in dry state (per organic mass).
[d] V^{daf} (V^d) is the yield of volatile substances per organic mass (in dry state).

Degree of biodegradation was evaluated according to DSTU EN 14995:2018 (EN 14995:2006, IDT).[24] Agar-agar, which does not contain food substances, was prepared and poured into Petri dishes. Samples of HESBFs were sterilized by immersion in 70% ethyl alcohol for 1 minute followed by air-drying for 72 hours. Then, HESBFs were placed on the surface of a Petri dish and 100 μL of a suspension of viable fungal spores was applied on the top (a consortium of species *A. niger*, *P. funiculosum*, *P. variotii*, *A. terreus*, *A. pullulans*, *P. ochrochloron* in the amount of 106 spores per 1 mL for each species). These species of microorganisms are specific for growth on biodegradable plastics and live in natural biocenoses. Samples of HESBFs were incubated in a thermostat for 6 months at a temperature of 302 K and relative humidity ≥ 90% and weighed every month.

The differential pressure method and a VAC-V1 vacuum tester were used to measure the oxygen gas permeability of HESBFs. Vacuuming was carried out for 8 hours.

Antibacterial properties were determined by the inhibition time of the active growth zones of *A. niger* molds on the surface of hybrid environmentally safe biodegradable materials in a nutrient medium using an electronic microscope Digital Microscope HD color CMOS Sensor

(China). The time of mold appearance for the samples of HESBFs was measured visually by recording the time of composition preparation and the time of the first visual signs of mold appearance on the surface using a digital clock.

3. Results and Discussion

The FTIR spectra of the initial humic acids, the hydroxypropyl methyl cellulose as well as the systems hydroxypropyl methyl cellulose + 5 wt.% of humic acids and hydroxypropyl methyl cellulose + 10 wt.% of humic acids (**Fig. 1**) have been analyzed.

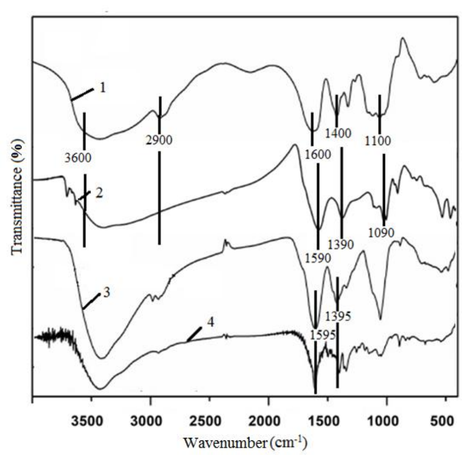
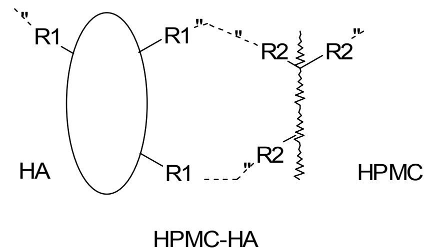


Fig. 1. FTIR spectra: 1 – hydroxypropyl methyl cellulose; 2 – humic acids; 3 – hydroxypropyl methyl cellulose + 5 wt.% of humic acids; 4 – hydroxypropyl methyl cellulose + 10 wt.% of humic acids

In the FTIR spectrum of the system hydroxypropyl methyl cellulose + 5 wt.% of humic acids (Fig. 1, curve 3), the absorption bands typical to hydroxypropyl methyl cellulose and humic acids are clearly seen, *e.g.*, a broad band at 3100–3600 cm^{-1} (hydroxyl group); a band at 2750–2900 cm^{-1} (methyl group); bands at 1400 and 1600 cm^{-1} (aromatic C–C bonds); a band at around 1500–1650 cm^{-1} (carboxyl group); and a band at 1000–1150 cm^{-1} (C–O bonds).[25] There is a significant difference between the FTIR spectrum of the system hydroxypropyl methyl cellulose + 5 wt.% of humic acids and the FTIR spectra of hydroxypropyl methyl cellulose and humic acids, *i.e.*, the adsorption band, which belongs to the carboxyl groups of humic acids, shifts from 1595 cm^{-1} to the wavenumbers of 1625-1650 cm^{-1} for hydroxypropyl methyl cellulose + 5 wt.% of humic acids. Such changes in the FTIR spectra indicate that humic acids react with hydroxypropyl methyl cellulose *via* the multipoint interaction between the carboxyl groups and the hydroxyl groups of the hydroxypropyl methyl cellulose with the formation of the structure shown in Fig. 2.[25]



R1 = -OH, -COOH,-PhOH

R2 = -OH

Fig. 2. The structure of the system hydroxypropyl methyl cellulose-humic acids, which is formed by the mechanism of a matrix synthesis (HA – humic acids, HPMC – hydroxypropylmethylcellulose)

In fact, the given structure of the hydroxypropyl methyl cellulose-humic acids system implies that it is formed by the mechanism of a matrix synthesis within the polymer hybrid modification.

Afterwards, it has been conducted a study on determining the effect of hybrid humic acids modification on the most important characteristics of HESBFs with antibacterial properties, *i.e.*, water absorption, tensile strength, relative elongation at break, time of mold appearance, and degree of biodegradation. Figs. 3–6 demonstrate how the above-mentioned parameters of the films prepared from hydroxypropyl methyl cellulose and HAs depend on the content of different type HAs.

Thus, it has been revealed that the hybrid modification of hydroxypropyl methyl cellulose with humic acids by the mechanism of a matrix synthesis for the development of biodegradable films allows reducing their water absorption, increasing the major strength characteristics along with imparting the antibacterial properties as evidenced by the findings on mold appearance time for the films. The effect of the hybrid modification on the enhancement of the HESBFs characteristics for the different types of humic acids increases in the range HA1 < HA2 < HA3. This is due to the higher content of volatile substances in initial lignite 3 and the higher content of the polar C–O, O–H, C=O, and $-NH_2$ groups in the HA3 sample of humic acids, as reported in our previous works.[18-21] It should be also noticed that the optimal content of humic acids in HESBFs is 10 wt. %. That enables enhancement of the important performance characteristics of HESBFs, *i.e.*, the maximum strength at break of 17 MPa, the relative elongation at break of 19%, and the minimum water absorption of 360%. The reason for this is the multipoint interaction between the carboxyl groups of the humic acids and the hydroxyl groups of the polymer as well as the general increase in the number of hydrogen bonds.

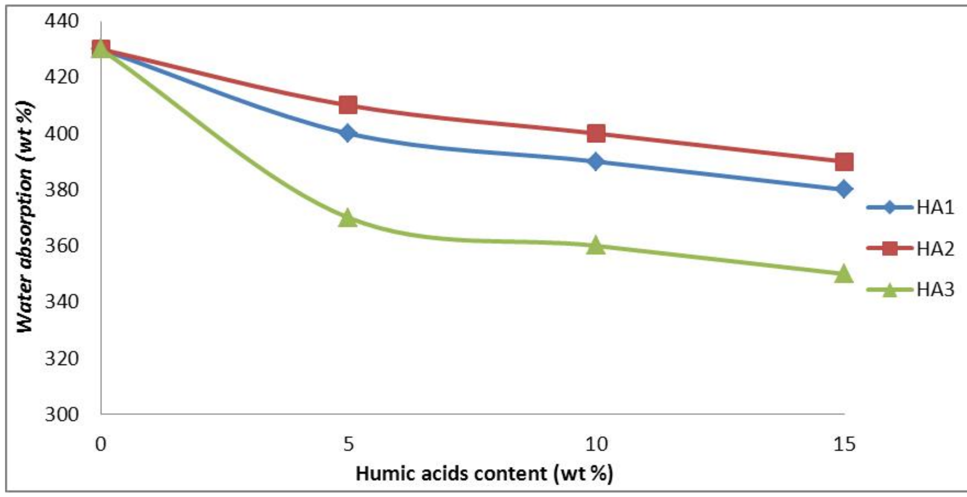


Fig. 3. Water absorption of HESBFs with antibacterial properties based on hydroxypropyl methyl cellulose *vs.* the content of humic acids of different types

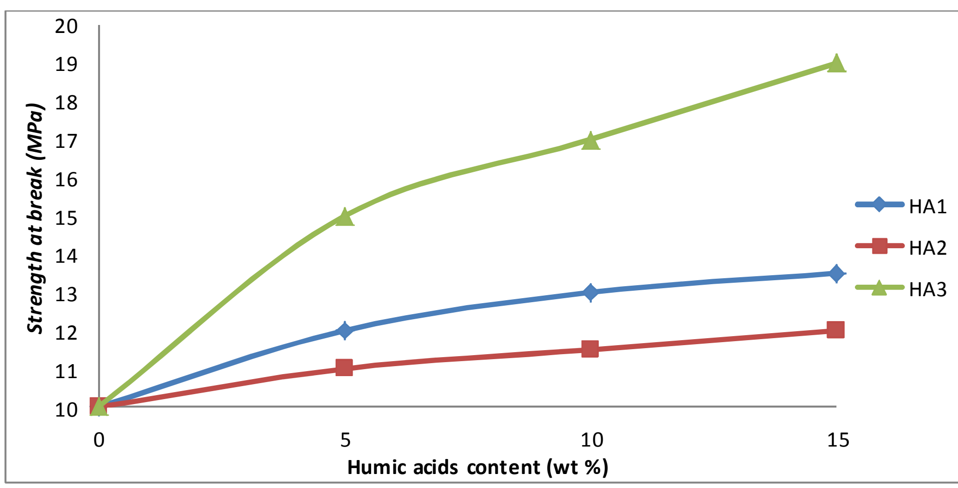


Fig. 4. Strength at break of HESBFs with antibacterial properties based on hydroxypropyl methyl cellulose *vs.* the content of humic acids of different types

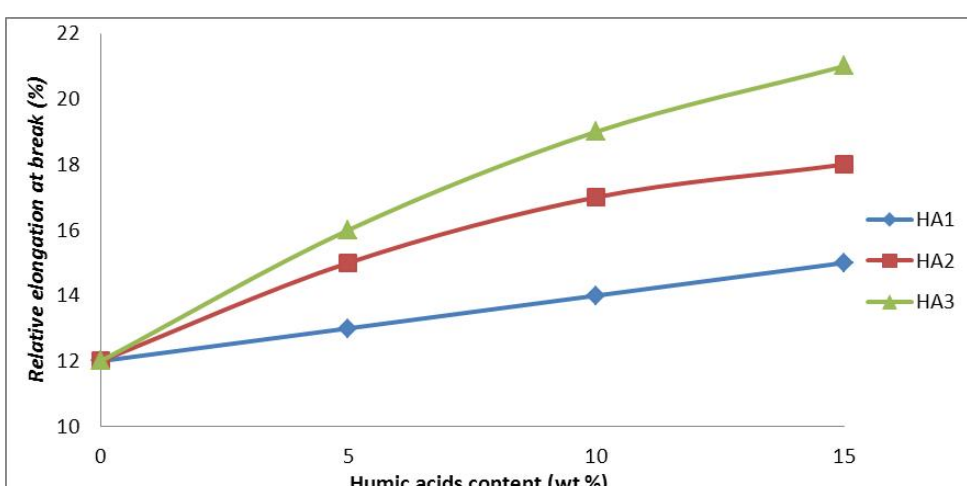


Fig. 5. Relative elongation at break of HESBFs with antibacterial properties based on hydroxypropyl methyl cellulose *vs.* the content of humic acids of different types

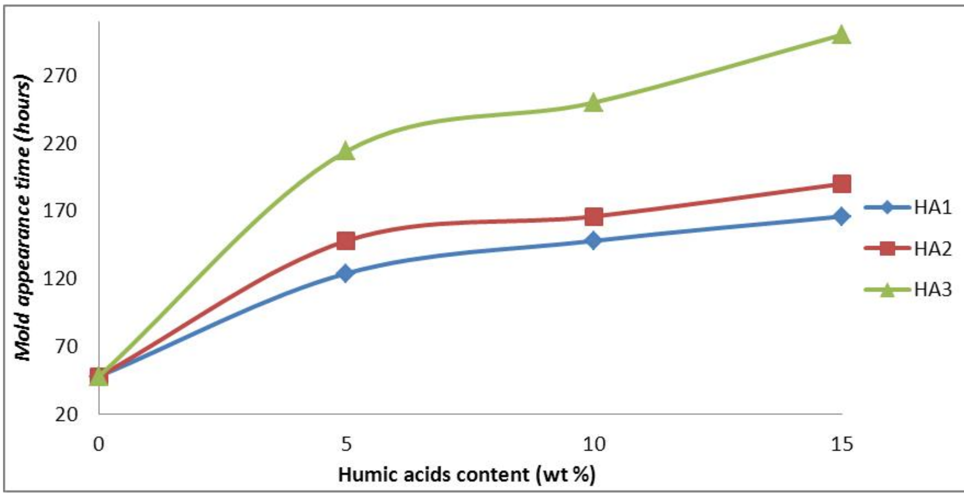


Fig. 6. Mold appearance time of HESBFs with antibacterial properties based on hydroxypropyl methyl cellulose *vs.* the content of humic acids of different types

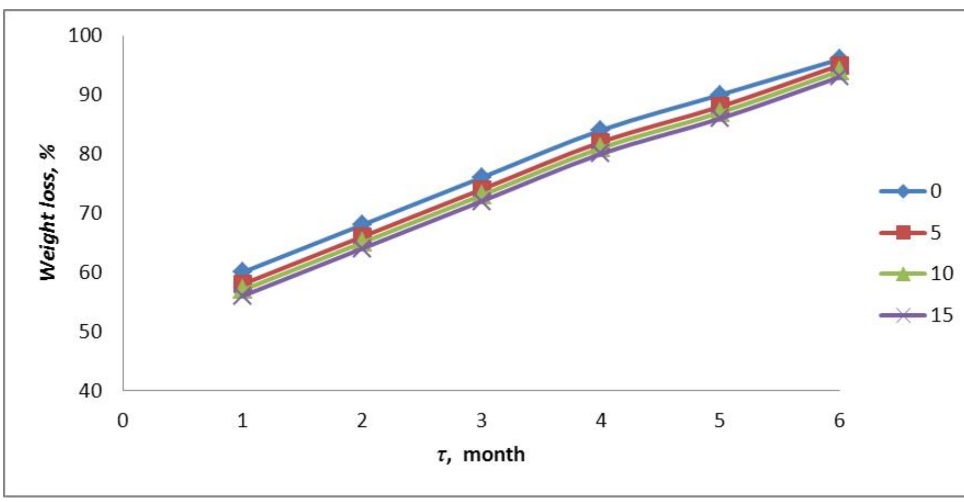


Fig. 7. Degree of biodegradation of HESBFs with antibacterial properties based on hydroxypropyl methyl cellulose *vs.* the HA1 content

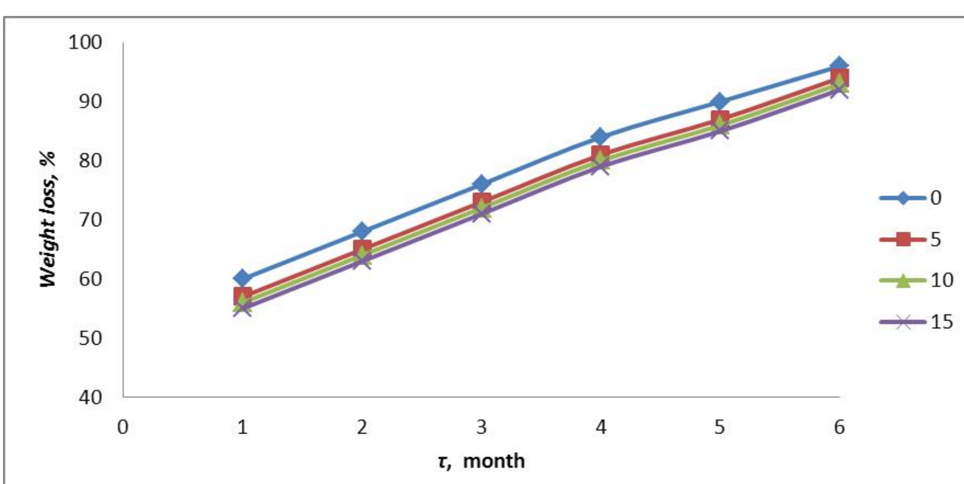


Fig. 8. Degree of biodegradation of HESBFs with antibacterial properties based on hydroxypropyl methyl cellulose *vs.* the HA2 content

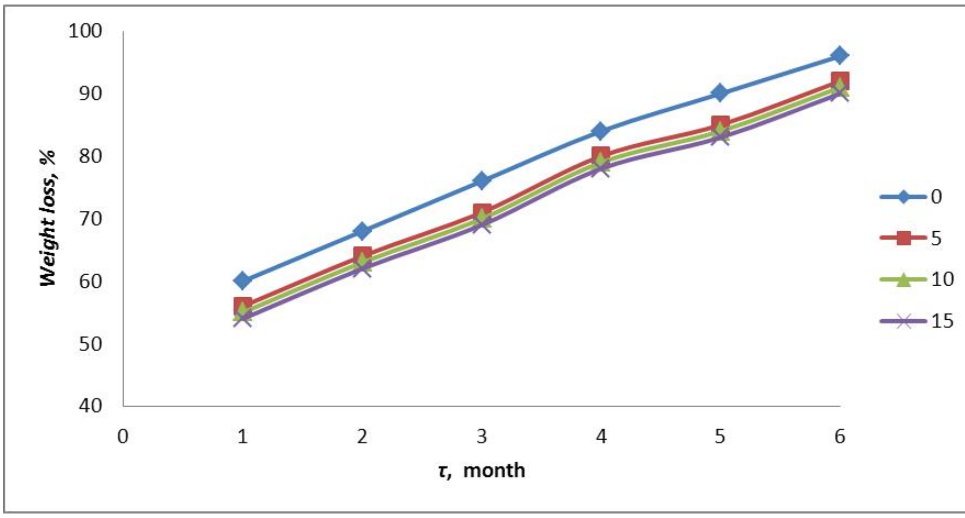


Fig. 9. Degree of biodegradation of HESBFs with antibacterial properties based on hydroxypropyl methyl cellulose *vs.* the HA3 content

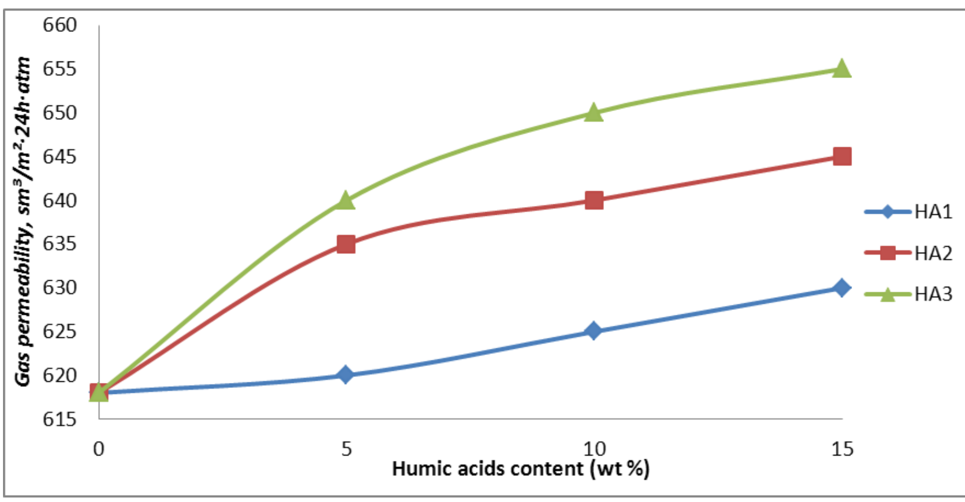


Fig. 10. Gas permeability of HESBFs with antibacterial properties based on hydroxypropyl methyl cellulose *vs.* the content of humic acids of different types

In order to determine the optimal content of humic acids, the biodegradability of HESBFs with antibacterial properties based on hydroxypropyl methyl cellulose has been examined as well (Figs. 7–9).

In general, it is clearly seen that the hybrid modification of hydroxypropyl methyl cellulose with humic acids for the development of biodegradable films with antibacterial properties allows preserving the biodegradability for the all types of used humic acids. According to the State Standard of Ukraine (DSTU EN 14995:2018), the weight loss of all samples exceeds 90 % within 6 months.

Fig. 10 shows findings of the study on gas permeability of HESBFs based on hydroxypropyl methyl cellulose modified with humic acids.

The findings in Fig. 10 indicate that the increase in gas permeability has been observed for all films due to growing the intensity of structurization during the hybrid modification. This stems from the formation of a more polar film surface with the increasing content of polar groups (C–O, O–H, C=O,–NH_2, *etc.*). The effect of enhanced gas permeability of hydroxypropyl methyl cellulose-based HESBFs with antibacterial properties depends on the HAs characteristics. Gas permeability increases in the range HA1 < HA2 < HA3 due to the higher content of volatile substances in initial lignite 3 and the higher content of the polar C–O, O–H, C=O, and –NH_2 groups in the HA3 sample of humic acids as mentioned above. The enhanced gas permeability of HESBFs upon the modification with HAs may prevent fogging of food. On the other hand, food packed in such materials should have a longer shelf life.

4. Conclusions

The FTIR spectroscopic data has revealed that hybrid structures of hydroxypropyl methyl cellulose modified with the different types of humic acids are formed by the mechanism of a matrix synthesis. This process is accompanied by the crosslinking of hydroxypropyl methyl cellulose *via* the multipoint interaction with the carboxyl groups of humic acids.

It has been found that the hybrid modification of hydroxypropyl methyl cellulose with humic acids by the mechanism of a matrix synthesis for the development of biodegradable films allows reducing their water absorption, increasing the major strength characteristics along with imparting the antibacterial properties as evidenced by the findings on mold appearance time for the films.

It has been determined that the optimal content of humic acids in the hybrid environmentally safe biodegradable films with antibacterial properties based on hydroxypropyl methyl cellulose is 10 wt.%. At this content, the maximum strength at break of 17 MPa, the relative elongation at break of 19%, the mold appearance time of 250 hours, and the minimum water absorption of 360 % have been achieved.

It has been also shown that the hybrid modification of hydroxypropyl methyl cellulose with humic acids of different types for imparting antibacterial properties ensures the film biodegradability exceeding 91 % within 6 months at the optimal content of humic acids of 10 wt.%.

Due to their operational characteristics, the developed environmentally safe biodegradable films with antibacterial properties based on hydroxypropyl methyl cellulose and humic acids are promising candidates for packaging materials for dry food (bread, cereals, nuts, *etc.*) with an extended shelf life.

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ДОСЛІДЖЕННЯ ГІБРИДНОЇ МОДИФІКАЦІЇ ГУМІНОВИМИ КИСЛОТАМИ ЕКОЛОГІЧНО БЕЗПЕЧНИХ БІОДЕГРАДАБЕЛЬНИХ ПЛІВОК НА ОСНОВІ ГІДРОКСИПРОПІЛМЕТИЛЦЕЛЮЛОЗИ

Анотація. *Розглянуто можливість підвищення комплексу міцнісних та експлуатаційних властивостей екологічно безпечних біодеградабельних полімерних матеріалів на основі гідроксипропілметилцелюлози через модифікацію різними типами гумінових кислот (ГК) з бурого вугілля. Уперше одержано гібридні екологічно безпечні високоміцні плівки з антибактеріальними властивостями. Фізико-хімічними дослідженнями та ІЧ-спектроскопією встановлено формування гібридних структур гідроксипропілметилцелюлози, модифікованої різними типами ГК. Виявлено закономірності зміни водопоглинання, міцності на розрив, відносного подовження під час розриву та часу появи цвілі екологічно безпечних біодеградабельних полімерних матеріалів на основі гідроксипропілметилцелюлози залежно від вмісту різних типів гумінових кислот. Також встановлено, що гібридна модифікація гідроксипропілметилцелюлози різними типами гумінових кислот із наданням їм антибактеріальних властивостей дає змогу зберегти в них властивості до біодеградації. Одержані екологічно безпечні біодеградабельні плівки з бактерицидними властивостями на основі гідроксипропілметилцелюлози та ГК за експлуатаційними характеристиками перевершують відомі аналогічні біодеградабельні плівки на основі природних біополімерів.*

Ключові слова: *екологічно безпечні, біодеградабельні плівки, гідроксипропілметилцелюлоза, бактерицидні властивості, гумінові кислоти.*