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FORMATION AND PROPERTIES OF CROSS-LINKED POLYMER FILMS BASED ON BIOCOMPATIBLE POLYMERS

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Cross-linked polymer films based on polyvinyl alcohol and polyacrylamide have been prepared via radical cross-linking initiated by peroxide containing reactive copolymers. The influence of temperature, nature and concentration of cross-linking agents onto gel-fraction value and properties of polymer films has been studied. Obtained cross-linked polymer films are characterized by improved physico-mechanical properties that depend on the content of peroxide containing copolymer and on the presence of additional cross-linking agent.

Key words: polyvinyl alcohol, polyacrylamide, polymer films, cross-linking, physico-mechanical properties.

Introduction

Synthetic polymer materials – plastics, fibers, rubbers are widely used in various fields. But usually synthetic polymers such as polyethylene, polypropylene, polystyrene are not biocompatible that restricts their application in biomedicine, biotechnology and other related areas. Besides, the inability of such materials to biodegradation complicates their utilization or recycling. Therefore, the development and application of biocompatible and biodegradable polymers is considered as the most promising way to solve the problem of polymer waste. For the last decades, a lot of different biocompatible and biodegradable materials have been created and many of them are already produced by industry [1, 2]. According to their origin, such polymers are divided into three main categories: (1) synthetic polymers, in particular aliphatic polyesters, such as poly(L-lactide), poly(ε-caprolactone), poly(p-dioxanone), poly(butylene succinate), polyoxazolines [3–7]; (2) polyesters, synthesized using microorganisms – mainly different types of poly(hydroxy alkanoates) including poly(β-hydroxybutyrate) [8]; (3) polymers obtained from natural sources including starch, cellulose, chitin, chitosan, lignin and proteins [9, 10].

However, although significant progress has been made in the development of biocompatible polymers and the situation in this area is very promising, such polymers have not yet become a full-fledged substitute for traditional polymers. The main reason for this is the unsatisfactory properties of these materials, in particular poor mechanical properties, high hydrophilicity and low heat resistance and recyclability, which limit their use. Based on the foregoing, the necessity of modifying such polymers becomes obvious.

Cross-linking is one of the methods for modifying biocompatible polymers, which improves their physicomechanical characteristics, makes it possible to regulate their barrier properties, thermal stability, solubility in an aqueous medium, and biodegradability. George with coworkers have shown that the rate of hydrolytic degradation of cross-linked polylactides (PLLA) is much slower as compared with linear PLLA and depends strongly on the molecular weight between crosslinks [11]. It is reported [12] that cross-linking with succinic acid provides significant improving in mechanical

strength and thermostability of chitosan/collagene scaffold materials. Cross-linked polymers can be used as the components of stimuli-responsive smart polymer systems [13]. In order to improve the properties of polymers specific to their applications, they can be modified with the use of different tecniques: physical cross-linking (high pressure, irradiation) or chemical cross-linking (glutaraldehyde, trisodium trimetaphosphate, ions of polyvalent metals, epichlorohydrin, polycarboxylic acid, for example citric acid, peroxides [14–16]).

Peroxides can be used for cross-linking diverse polymers. Their main advantages as crosslinking agents are the (1) ability to crosslink saturated and unsaturated polymers, (2) high-temperature resistance, (3) good elastic behavior at higher temperature, (4) low moisture uptake [17]. But the use of low-molecular peroxides can result in the formation of volatile decomposition products. Besides, the safety demands during the storage of such peroxides are considerable. Our previous works have shown that polyperoxides with pendant peroxide groups can be used as modifiers and curing agents for diverse types of polymers and copolymers [18, 19].

Therefore, the aim of this work is to study the processes of cross-linking polymer films based on biocompatible polymers, polyvinyl alcohol and polyacrylamide, by polyperoxides and physico-mechanical properties of obtained polymeric materials.

Materials and methods of research

Synthesis of peroxide-containing reactive copolymers (RC) of butyl methacrylate (BMA), acryl amide (AcAm), maleic anhydride (MA) and peroxidic monomer 5-tert-butylperoxy-5-methyl-1-hexene-3-yne (PM) with different content of monomer links was carried out via radical copolymerization of the aforementioned monomers in 2-propanone at their diverse ratios (total monomer concentration was 3.5 mole/l) at 333 K in the presence of lauroyl peroxide as initiator (C_{PL}= 0.03 mole/l) as described elsewhere [20]. Composition and some chemical characteristics of synthesized RC are shown in Table 1.

Table 1

Synthesis, composition and some characteristics of used peroxide-containing copolymers

RC	Monomer mixture composition, %mol.				Copolymer composition, %mol.				Conver-sion, %	Wpol·10^3, %/c	Intrinsic viscos. in acetone, m^3/kg	M_w ·10^{-3}, g/mole
	AcAm	PM	BMA	MA	AcAm	PM	BMA	MA				
1	30	30	–	40	37.9	24.5	–	37.6	77.8	6.5	0.006	7.0
2	40	20	–	40	44,5	15.5	–	40.0	81.6	8.3		8.5
3	20	20	20	40	25.6	19.5	14.8	40.1	82.1	4.9	0.007	10.6

The content of peroxide groups in RC was determined from the results of gas chromatographic analysis of the final decomposition products obtained at the RC thermolysis at 483K under argon with the use of gas-liquid chromatograph SELMICHROM-1 (Selmi, Ukraine) [21]. The content of MA subunits in RC was determined using a reverse potentiometric titration of carboxylic groups. The content of AcAm subunits was estimated from the results of elemental [C, H, N] analysis. Intrinsic viscosity of RC solutions was measured using Bishoff viscosimeter in acetone at 298K [22]. Molecular weight of copolymers was determined by gel-permeation chromatography using Polymer Standard Service system (PSS, Mainz, Germany, MDS RI detector).

Polyvinyl alcohol (PVA, trademark JP-18 (JAPAN VAM&POVAL Co., Japan)) with molecular weight 80 kDa and polyacrylamide (pAcAm) with molecular weight 200 kDa were used as biocompatible polymers. Hydroxyethyl methacrylate (HEMA) and N,N'-methylene-bis-acrylamide (MBA) supplied by Merck & Co were used as additional cross-linkers.

To obtain cross-linked polymer films 20 % water solution of PVA or pAcAm was prepared with 10-30 % of RC with respect to polymer. The films were cast on a glass surface, and dried at ambient

conditions. As a result, the films were formed with the thickness of 120–160 μm. After that they were cured at 393–413K for 1–3 hours. Cross-linking polymer films is carried out due to chain transfer reactions initiated by free radicals formed as a result of the decomposition of RC peroxide groups.

The kinetics of film curing was investigated by gel fraction content. The soluble fractions were extracted from cured films in a Soxhlet apparatus by water during 12 hours. The gel fraction (H, %) was computed as:

$$H = (m_1/m_2)\bullet 100\ \%\ , \qquad (1)$$

where m_1 and m_2 – the film weights after and before extraction, respectively.

The swelling kinetics of cured polymer films was determined as follows: the weighed sample of the dry film was covered with water and kept in water for the certain time. Thereafter the unabsorbed water was decanted, the swelled film was put on a filter paper to remove the remaining unabsorbed water. The swelling degree (W, %) was calculated as:

$$W = ((m_s - m_f)/m_f)\bullet 100\ \%, \qquad (2)$$

where m_s – the weight of the swelled film and m_f – the weight of the film sample.

In order to determine swelling rate constant and equilibrium (maximum achievable) water content in the swollen film at given conditions (taking into account that the swelling kinetics obeys the second-order equation) the following equation was used [23]:

$$\frac{dW}{dt} = K\cdot(W - W_\infty)^2\ , \qquad (3)$$

where W – water content in swollen film, W_∞ – equilibrium (maximum achievable) water content in the swollen film, t – time, K – swelling rate constant.

After integrating and rearrangement of equation 3, the following equation is obtained:

$$\frac{t}{W} = \frac{1}{K\cdot W_\infty^2} + \frac{t}{W\infty}\ . \qquad (4)$$

By plotting the graphical dependence of $t/W \rightarrow t$, one can determine the swelling rate constant and the equilibrium (maximum achievable) water content in the film under the given conditions.

The film hardness (T) was determined using M-3 pendulum apparatus:

$$T = \tau_p/\tau_g, \qquad (5)$$

where τ_p – a time of M-3 pendulum swings on polymer film cured on a glass plate (s), τ_g – a time of M-3 pendulum swings on a glass plate (s).

Tensile strength (*P*, kPa) and relative elongation at break (*ε,* %) of cross-linked films were determined using TIRA test 2200 testing machine.

Results and Discussion

The study of the dependence of crosslinking processes of biocompatible polymers on temperature reveals that the course of the process is determined by the nature of biocompatible polymer and composition (primarily the content of peroxide fragments) and concentration of RC.

If curing is carried out at 393K the gel-fraction value of the films based on PVA and pAcAm is very low and increases with the temperature enhancement. The temperature dependence of cross-linking value is especially clearly seen for PVA films. In the case of pAcAm, if only peroxide-containing copolymers are used for curing the gel-fraction value of polymer films cured at 413K was 4–15 % increasing slightly with the rise of RC concentration and the content of peroxidic groups in RC (Fig. 1), while in the case of PVA the maximum values reaches 40–60 % depending on concentration of RC used and content of peroxidic groups in it. Low gel-fraction value for pAcAm curing with RC is caused most likely due to its tendency to β-decay reactions [24, 25]. As a result of hydrogen atom abstraction from pAcAm macromolecules by free radicals formed via RC decomposition, the AcAm macroradicals are formed that easily undergo to β-chain scission leading to the decomposition of polymer chain and, respectively, the decrease in AcAm molecular weight. Therefore, the formation of network structures in such system is hampered, instead the degradation of polymer matrix occurs.

The cross-linking degree of films based on PVA films increases sharply with increasing content of RC peroxide groups of which serves as the initiators of cross-linking processes generating free radicals at elevated temperatures. With increasing RC concentration from 10 % up to 30 % with respect to polymer weight the gel-fraction of films rises from 23 % to 60 % at 413K.

The influence of small (3–5 %) quantities of vinyl monomers – hydroxyethyl methacrylate and N,N'-methylene-bis-acrylamide onto cross-linking processes has been studied. One can see that the introduction into the system of HEMA and especially bifunctional MBA provides a significant increase in the value of gel-fraction. For example, at the MBA introduction into PVA film the maximum gel-fraction value is 74 %, while in the case of pAcAm films it reaches 62 % (Fig. 1, 2).

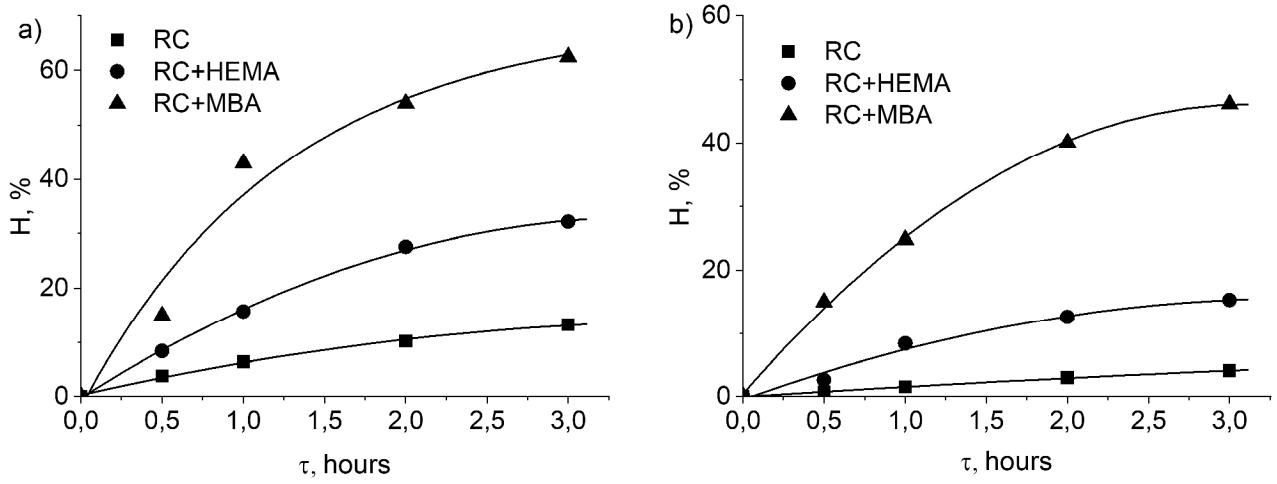

Fig. 1. Dependence of gel-fraction of pAcAm polymer films cross-linked with the use of RC (C_{RC} = 30 %) and vinyl monomers (5 % HEMA, 3 % MBA) on curing time: RC-1(a), RC-2 (b); T = 413K

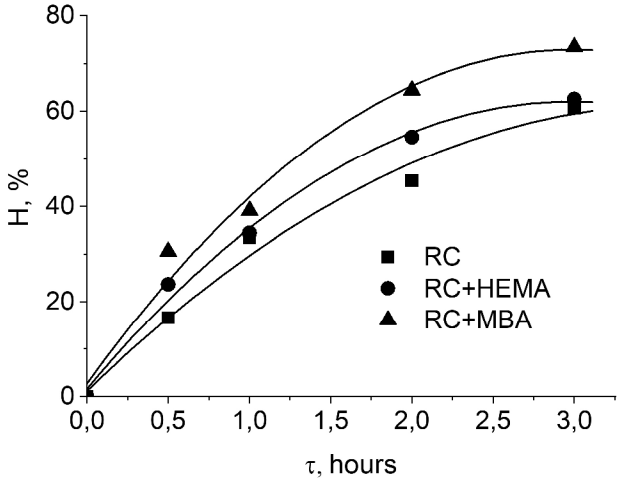

Fig. 2. Dependence of gel-fraction of PVA films cross-linked with the use of RC-1 (C_{RC} =30 %) and vinyl monomers (5 % HEMA, 3 % MBA) on curing time (T = 413 K)

If the films of biocompatible polymers are cross-linked in the presence of vinyl monomers the dependences of gel-fraction value on the concentration of RC in the film and on the content of peroxide fragments in RC are analogous to those observed at the absence of vinyl monomers (Fig. 3, *a, b*).

One can see (Fig. 3, *a*) that with increasing RC concentration in the films from 20 % to 30 % the rate of cross-linking PVA films in the presence of MBA rises essentially and gel-fraction after 3 hours curing increases from 57 % to 74 %. The cross-linking degree also increases sharply with increasing the content of peroxide groups in RC at the constant concentration of RC in the film. So, for pAcAm films in the case of RC-1 containing 24,5 % peroxidic groups the gel-fraction value is essentially higher than if RC-2 was used containing 15,5 % of peroxide groups and such enhancement is observed for both additional monomers – HEMA and MBA (Fig. 3, *b*).

Cross-linked films based on PVA are characterized by high degrees of swelling in water – 500–2000 % depending on cross-linking values and nature of added additional monomer (Table 2, Fig. 4). Swelling degree diminishes with the introduction in the films of vinyl monomers participating in the cross-linking. The lowest degree is for the films obtained in the presence of MBA for which the highest gel-fraction value is observed, i.e. the most rigid network is formed.

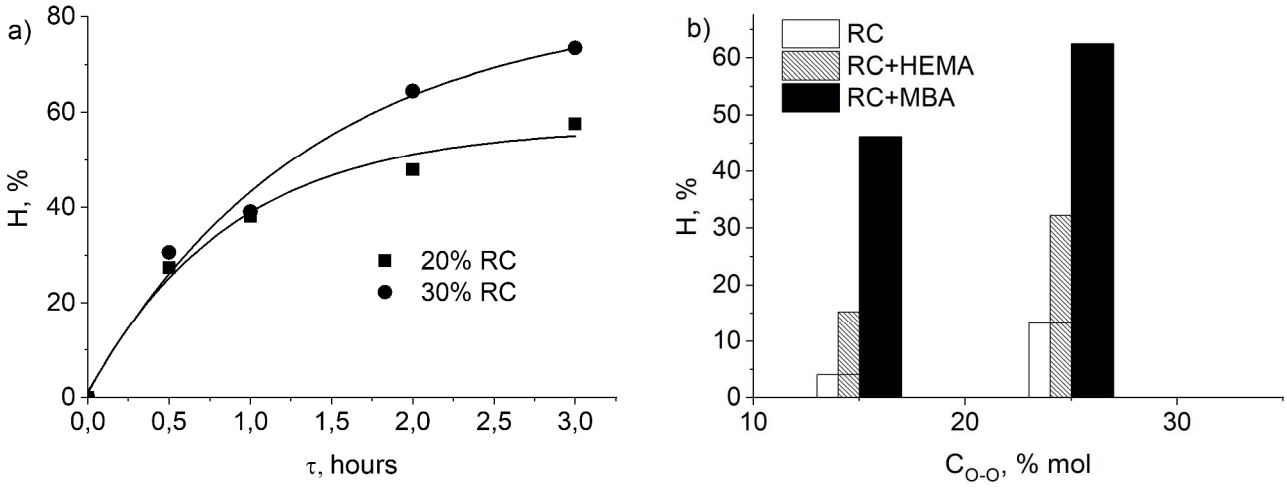

Fig. 3. Dependence of gel-fraction of PVA films cured in the presence of 3 % MBA on RC-1 content in the film (a) and pAcAm films cured in the presence of vinyl monomers (5 % HEMA, 3 % MBA) on the content of peroxide groups in RC (C_{RC} = 30 %) (b)

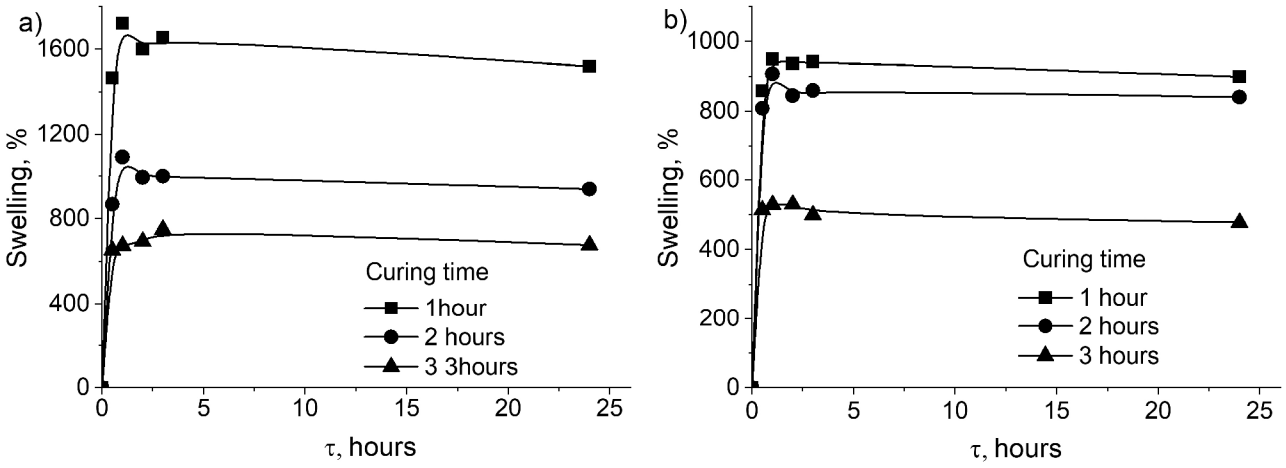

Fig. 4. Dependence of swelling of polymer films on time (C_{RC} = 30 %): PVA film with RC-1 (a); PVA film with RC-1 and 3 % MBA (b)

It should be noted that the maximum swelling degree is reached after 1–2 hours and then its insignificant decrease is observed that evidently is explained by leaching of the water-soluble fraction and as a result by the decrease of the content of polymer capable of water sorption (Fig. 4). Moreover, the relative reduction in the swelling degree is minimal for films with MBA which are characterized by the highest gel-fraction values that witnesses in favor of our assumption.

Constants of swelling rate and equilibrium (maximum achievable) water content in swollen films at given conditions were calculated from plots t/W→t as it is described in experimental part (Fig. 5, Table 2).

One can see (Table 2), that constant of swelling rate decreases with increasing curing time i.e. with increasing film cross-linking degree. Equilibrium water content decreases in the same way that witnesses in favor of more cross-linked rigid polymer film network. It is interesting that after 1 hour curing the swelling rate constant for films obtained with HEMA participation is 3–7 times higher than for films obtained in the absence of vinyl monomer or with MBA. Evidently, the introduction of HEMA links which are characterized by high hydrophilicity into the films promotes the increasing ability of polymer films to water-swelling. At the same time, for the films cured during 3 hours (highly cross-linked films) the swelling rate constant becomes two times lower than for films without vinyl monomers and close to the constant for films obtained with MBA participation.

Table 2

Dependence of kinetics of PVA film swelling on the composition and curing time

№	Nature of cross-linker	Curing time, hours	K, 1/(%·min)	Swelling for 24 h, %	W_∞,%
1	RC-1	1	$2.86 \cdot 10^{-2}$	1520	93.5
2	–//–	2	$1.94 \cdot 10^{-2}$	940	90.3
3	–//–	3	$1.90 \cdot 10^{-2}$	677	87.7
4	RC-1+5% HEMA	1	$19.3 \cdot 10^{-2}$	1397	93.5
5	–//–	2	$9.7 \cdot 10^{-2}$	869	89.3
6	–//–	3	$0.95 \cdot 10^{-2}$	681	87.0
7	RC-1+3% MBA	1	$5.16 \cdot 10^{-2}$	899	90.9
8	–//–	2	$2.83 \cdot 10^{-2}$	841	89.1
9	–//–	3	$0.97 \cdot 10^{-2}$	478	82.6

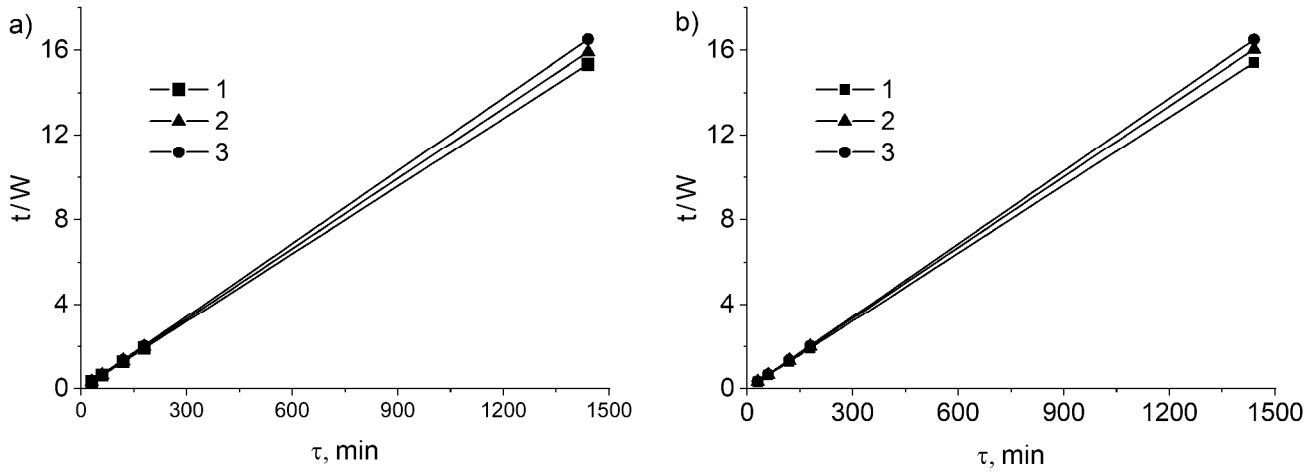

Fig. 5. Kinetics of swelling polymer films based on PVA and RC-1 (a), PVA and RC-1 with MBA in coordinates t/W→t. Curing time: 1 hour. (1), 2 hours (2), 3hours (3)

Swelling degree depends essentially on the content of peroxidic groups in RC composition and decreases with its increase (Fig. 6). As it was shown earlier, the degree of film cross-linking rises with increasing content of peroxide fragments in RC that causes the decrease in degree and rate of water-swelling.

Therefore, the obtained data witnesses that according to the rate and degree of swelling in water, the obtained polymer films can be classified as superabsorbent polymers [26] and swelling degree and rate are determined by the nature of polymer matrix and cross-linking degree.

Cross-linked films based on biocompatible polymers are characterized by high hardness (Table 3). Films based on pAcAm possess high values of hardness even before curing that is explained by high temperature of glass transition for this polymer (Tg ~ 200 °C).

The hardness of PVA films rises with increasing curing time, that is with increasing cross-linking degree. Besides, film hardness rises with increasing concentration of RC in films and increasing content of peroxidic groups in RC that is explained by the enhancement of cross-linking degree. The introduction of additional vinyl monomers enhances slightly the hardness of cross-linked PVA films that is in a good agreement with the values of gel-fraction, in other words – with film cross-linking degree (Fig. 7).

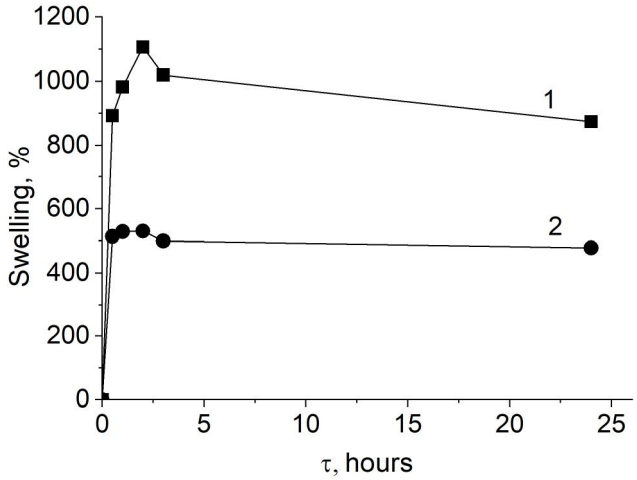

Fig. 6. The influence of peroxide group content in RC on the water-swelling kinetics of films based on PVA with RC-2 ($C_{O\text{-}O}$= 15.5 % mol) (1) and PVA with RC-1 ($C_{O\text{-}O}$= 24.5 % mol) (2). C_{RC} = 30 %

Table 3

Dependence of hardness of PVA films on composition and curing time

Curing time, hours	C_{RC}, % per polymer	Relative hardness, a.u.
Pure glass		1.0
RC-3 (AcAm-PM-BMA-MA (25:20:15:40))		
0	20	0.21
1	–//–	0.34
2	–//–	0.67
3	–//–	0.77
RC-1 (AcAm-PM-MA (38:24:38))		
0	20	0.23
2	–//–	0.81
3	–//–	0.83
0	30	0.40
2	–//–	0.82
3	–//–	0.84
0*	20	0.77
3*	–//–	0.82

* *Films based on pAcAm*

Obtained cross-linked polymer films are characterized by high enough tensile strength (Table 4) which depends on curing time and film composition. Increasing curing time leads to the increase in tensile strength of films based on PVA from 30–40 MPa to 50–65 MPa, and the films obtained in the presence of additional cross-linker MBA have higher values of tensile strength. At the same time, the relative elongation at break decreases sharply – from 70–190 % for uncured to 1–4 % for cross-linked films that can be explained by the increase of cross-linking density and rigidity of polymer network.

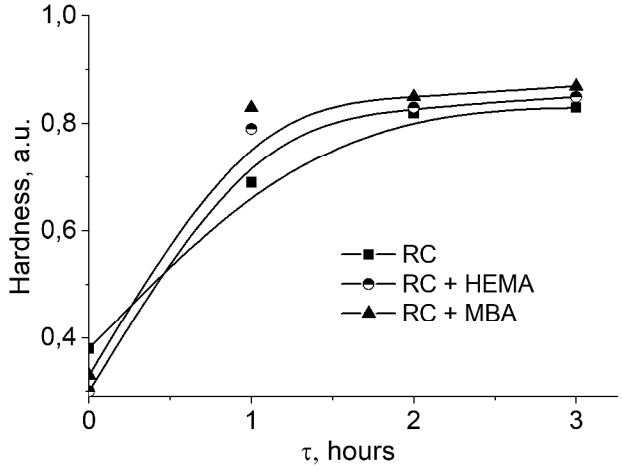

Fig.7. Dependence of hardness of films based on PVA and RC-1 (C_{RC} = 30 %) on curing time and film composition

Table 4

Dependence of physico-mechanical characteristics of PVA films on composition and curing time (T = 413 K)

Film composition	Curing time, hours	Section area, м^2	Breaking force, N	Tensile strength, MPa	Relative elongation*, %
RC	0	$1.8^{\bullet}10^{-6}$	72.8	41.5	74.5
	1	$2.2{\cdot}10^{-6}$	124.4	56.3	6.4
	3	$1.\ 9{\cdot}10^{-6}$	97.8	51.6	0.9
RC+HEMA	0	$3.0{\cdot}10^{-6}$	87.2	29.1	107.0
	1	$2.3{\cdot}10^{-6}$	139.8	60.5	7.5
	3	$2.2{\cdot}10^{-6}$	134.1	61.8	6.4
RC+MBA	0	$2.7{\cdot}10^{-6}$	88.1	33.1	194.4
	3	$1.8{\cdot}10^{-6}$	120.3	66.8	3.84

Conclusions

The processes of cross-linking polymer films based on biocompatible polymers by peroxide containing curing agents have been studied. It has been shown that the degree of cross-linking rises with increasing concentration of peroxide-containing copolymer in the film and increasing content of peroxidic groups in copolymer. Introduction of additional vinyl monomers such as HEMA and MBA into the film composition provides the increase in cross-linking degree that witnesses in favor of the participation of these monomers in the reactions of film cross-linking. Obtained cross-linked polymers are able to swell in water and according to the rate and degree of swelling they can be classified as superabsorbent polymers. The degree and the rate of swelling is determined by the nature of polymer and degree of cross-linking. Films based on cross-linked biocompatible polymers are characterized by high enough physico-mechanical properties – hardness, tensile strength. These parameters can be regulated by changing the content and nature of RC which serve as cross-linking agents.

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ФОРМУВАННЯ ТА ВЛАСТИВОСТІ СТРУКТУРОВАНИХ ПОЛІМЕРНИХ ПЛІВОК НА ОСНОВІ БІОСУМІСНИХ ПОЛІМЕРІВ

Структуровані полімерні плівки на основі полівінілового спирту та поліакриламіду отримані за допомогою радикального структурування, ініційованого пероксидовмісними реакційноздатними кополімерами. Досліджено вплив температури, природи і концентрації зшивальних агентів на величину гель-фракції та властивості отриманих плівок. Отримані структуровані нанокомпозитні плівки характеризуються покращеними фізико-механічними властивостями, що залежать від вмісту пероксидвмісного кополімеру та присутності додаткового зшиваючого агента.

Ключові слова: полівініловий спирт, поліакриламід, полімерні плівки, структурування, фізико-механічні властивості.