

## Mechanical properties of composite films based on chitosan and poly(L-lactic acid)

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Mechanical properties of chitosan/poly(L-lactic acid) (Ch/PLLA) blend films have been studied in the present research. The films were prepared by dissolving different weight fractions of the polymers in a solvent, which is mixture of formic acid and acetone. The films were casted in Teflon petri dishes and dried at temperature 35°C till constant weight was achieved. Mechanical tests are provided at constant deformation rate 0.1 mm/s and room temperature. Based on the force-deformation curve the rupture point, rupture work and modulus of elasticity have been evaluated. The degree of elasticity was determined using a mechanical hysteresis test. Relaxation test has been also done in order to estimate the average relaxation time and the broadness of relaxation time distribution. Based on the mechanical properties just partial technological compatibility between the compounds could be assumed. For further understanding the structure of chitosan/PLLA films thermal and morphological characterization have been done. Based on the results from the Differential scanning calorimetry (DSC) and SEM analysis it was concluded that no miscibility on molecular level between chitosan and PLLA could be achieved in the proposed solvent.

**Key words:** chitosan, poly(L-lactic acid), mechanical properties, DSC, SEM, compatibility

### INTRODUCTION

Nowadays, the use of natural polymers that are biodegradable and biocompatible has become increasingly important. This is due to their characteristics: natural abundance, low costs, and wide range of applications [1, 2]. These polymers are being widely used in the biomedical area, including wound dressing, drug delivery system, and tissue engineering scaffolds [3, 4].

Chitosan (poly-1, 4-D-glucosamine) is a renewable, natural, nontoxic, edible, and biodegradable polymer which is produced by alkaline deacetylation of chitin. It is one of the most abundant polymers that can be found in nature. Owing to its biodegradability and biocompatibility, chitosan is reported to be an active polymer with antimicrobial and antifungal activities [3, 5–8]. It has a potential as a packaging polymer and, more particularly, as an edible packaging or coating because of its ability to form a film without any use of additives [9]. Moreover, chitosan film has good oxygen and carbon dioxide permeability, which is lower than that of polyethylene film [10, 11], and good mechanical properties, which are comparable with those of many medium-strength commercial polymers [12]. Unfortunately, there are some limitations to the application of chitosan film for packaging, because of its high sensitivity to moisture. Perspective way to overcome this drawback is to

associate chitosan with a moisture resistant polymer, while maintaining the overall biodegradability of the product.

An interesting candidate to blend with chitosan is polylactic acid (PLA). PLA belongs to the family of aliphatic polyester commonly made from lactic acid, which can be produced from renewable resource such as starch via fermentation processes [12]. It is a thermoplastic, high-strength, high modulus polymer and is considered as biodegradable and compostable [13].

Several attempts have been done for blending chitosan and PLA [14, 19]. Unfortunately all of them were unsuccessful and it is difficult to achieve miscibility of these two polymers at micro level. The basic reason for this incompatibility is their very different polarities and high interfacial tension. Normally chitosan can only be dissolved in dilute aqueous acidic solutions and PLLA can be dissolved only in some organic solvents. Even the attempt to mix them in common solvent was unfortunate [20].

The aim of the present study is to investigate the abilities to prepare chitosan polylactic acid casted films using a new two component solvent and to characterize the mechanical properties of the films.

### EXPERIMENTAL

#### Materials

Low molecular weight chitosan (Ch) with degree of deacetylation greater than 75% and viscosity of 20–300 cps (measured for 1% chitosan concentra-

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tion in 1% acetic acid water solution at 25°C) was purchased from SIGMA ALDRICH and used without further purification or characterization. Poly(L-lactic acid) (PLLA) was purchased from LACTEL ABSORBABLE POLYMERS, USA. It is with ester end groups and intrinsic viscosity 0.9–1.2 dL/g, measured at 25°C in CHCl<sub>3</sub>. All other chemicals used are with analytical grade.

#### Sample preparation

Blends of chitosan with PLLA were formed in several steps. Firstly a common solvent, which is a mixture of equal volume parts of formic acid and acetone, and 0.5% relative to the total amount of the solution glycerol, was prepared. Then chitosan (2% by weight) and PLLA (2% by weight) were dissolved separately in the common solvent. Dissolution was performed with a magnetic stirrer at 80°C to provide fully transparent and clear solutions. The two solutions were then blended under vigorous stirring. The final homogeneous solution was poured into Teflon petri dishes and dried at atmospheric pressure and a temperature of 35°C until constant mass. Usually, the drying process was continued about 72 h. After drying the films were stored in a desiccator at room temperature and a relative humidity (RH) of 54%. Ten different series of samples were produced and designated as Ch/PLLA 10:0, Ch/PLLA 9:1, Ch/PLLA 8:2, Ch/PLLA 7:3, Ch/PLLA 6:4, Ch/PLLA 5:5, Ch/PLLA 4:6, Ch/PLLA 3:7, Ch/PLLA 1:9 and Ch/PLLA 0:10, where the numbers represent respectively the weight fraction of Ch and PLLA, in the blends.

The thickness of the dried films was measured by digital micrometer Micromaster IP54, Switzerland with resolution 1 μm. Each sample was measured at 10 different point and the average value of the thickness was used in data evaluation. It was in the range of 200 μm.

#### Sample analysis

*Mechanical measurements:* Tensile Machine LS2.5, Lloyd Instruments, United Kingdom equipped with Film Support Rig probe was used for investigation of rheological properties of the Ch/PLLA blend films. The following test were used:

1) Rupture test. The test was carried out at a deformation rate of 0.1 mm/s until a deformation of 10 mm was reached. The instrument was manually stopped immediately after the breaking of the film. The rupture test was used for determining the strength

at the point of breakage of the film (rupture force), the rupture energy (the area under the force-deformation curve) and the modulus of elasticity.

2) Hysteresis test. The hysteresis testing was performed at a deformation rate of 0.1 mm/s until reaching a deformation 0.35 mm. The degree of elasticity was calculated using the dependence:

$$K = A_1/A_2, \quad (1)$$

where  $A_2$  is the area under the unloading section and  $A_1$  is the area under the loading section of the hysteresis.

3) Relaxation test. The test was conducted under pressure, firstly at constant deformation rate while the desired strength was achieved, after which the deformation was kept constant for 60 s. The average relaxation time and the  $\beta$ -parameter, characterizing the relaxation time distribution were calculate using fitting of the experimental data with the stretched exponent model:

$$F = F_\infty + F_1(-t/\tau)^\beta, \quad (2)$$

where  $F_\infty$  is the force at equilibrium,  $F_1$  is the difference between the initial force and the equilibrium force.

All the tests were carried out at room temperature and the results were taken as an average of ten tests.

*Differential scanning calorimetry (DSC):* The thermal properties of the films were investigated by the DSC 204 F1 Phoenix NETZSCH, Germany calibrated with an indium standard. Samples (10–20 mg) were cut from a film after conditioning and placed in sealed aluminum pans. For each sample, the following thermal cycle was applied: the sample was cooled from room temperature to 0°C at cooling rate 10 K/min, 1 min at 0°C, and then heated to 250°C at heating rate 10 K/min. The glass transition temperature ( $T_g$ ), melting temperature ( $T_m$ ) and melting enthalpy ( $\Delta H_m$ ) were calculated from these experiments.

*Scanning electron microscopy (SEM):* The morphology of the chitosan/PLLA blend films was investigated by scanning electron microscopy (SEM). The samples were coated with gold in a vacuum evaporator Jeol JFC-1200 fine coater, the thickness of the gold coating was 0.1 nm. The SEM micrographs were carried out on a scanning electron microscope Jeol JSM-5510, with the cathode voltage of 10 kV and magnification of 5000 and 10000. The cross-sections of the films were investigated.

## RESULTS AND DISCUSSION

### Formation of Ch/PLLA composite films

The basic challenge in a solvent-casting approach is a proper selection of a solvent in which a homogeneous mixture of the two components on micro level is achieved. Therefore the first task, which was performed in the present study was to choose a suitable combination of solvents that dissolve both chitosan and PLLA. Initial experiments were performed to dissolve separate the chitosan in aqueous solution of acetic acid and PLLA in chloroform and to mix the two solutions. It turned out that such a system is unstable and will separate due to the incompatibility of the two solvents. Therefore further experiments were carried out for system, in which both of the polymers are dissolved in common solvent obtained by mixing acetone and formic acid. It was found out that the PLLA could be dissolved in acetone at temperatures about 80°C that necessitated heating the solutions during mixing.

Another challenge in casting the film turned out to be the strong adhesion of the dried film on the surface of the glass petri dishes and the impossibility to be separated from the dish. Therefore composite films have to be casted in Teflon petri dishes, specially made for the purposes of this study. It was originally planned to prepare films with a weight ratios between the chitosan and the PLLA (Ch/PLLA) as follows: 10:0, 9:1, 8:2, 7:3, 6:4, 5:5, 4:6, 3:7, 2:8, 1:9, and 0:10. It turned out that the addition of PLLA leads to worsen the film forming properties of chitosan, so that in a weight portion of the PLLA greater than 0.6 the formation of films was not observed. Probable cause for the observed effect is insufficiently good dissolving of PLLA in acetone and the difficult formation of a uniform continuous matrix of the polymer. Successful blend with the maximum content of PLLA is a film with composition chitosan/PLLA=4:6.

### Mechanical results

Basic mechanical parameter characterizing the exploitation properties of Ch/PLLA films is their strength, which is characterized by the rupture force and rupture work – (Fig. 1). To take account into the film thickness the last two parameters are normalized to unit sample thickness and therefore they are measured in N/mm and J/mm respectively. Increasing the content of PLLA leads to non-linear decrease of rupture force and rupture work. This observation could

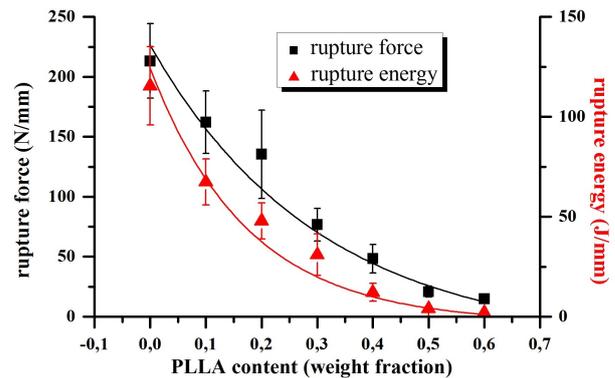


Fig. 1. Rupture force and rupture work for Ch/PLLA blends with different weight fractions of the components.

be interpreted as partial or complete incompatibility of the two materials and occurrence of phase segregation. The poorer solubility of PLLA in the common solvent causes the formation of agglomerates of the PLLA molecules, which is scattered in a continuous matrix of chitosan. When increasing the PLLA concentration, the continuous phase decreases, which results in drastic worsening of the mechanical properties.

The dependence of the modulus of elasticity of the composite films on the weight fraction of PLLA is presented in Fig. 2. The increased content of PLLA results in a decrease of modulus of elasticity and a drastic drop is observed in case of 0.3 weight fraction.

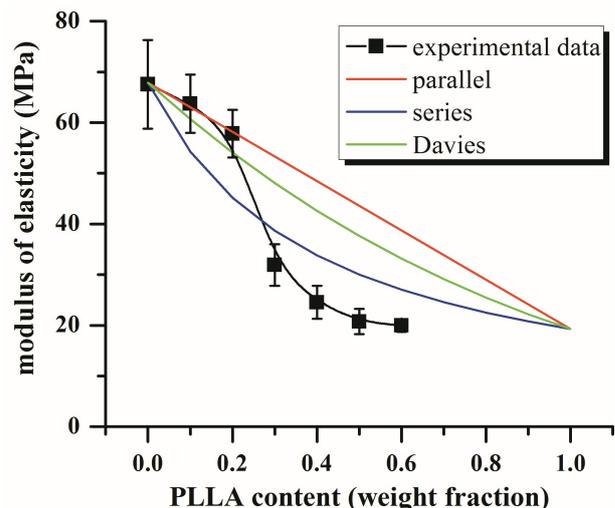


Fig. 2. Experimental modulus of elasticity for Ch/PLLA blends compared with the parallel, series, and Davies models.

No film from pure PLLA was able to be prepared in the common solvent used in this research. Therefore in order to evaluate its elastic properties, films from PLLA dissolved in chloroform were casted and their mechanical properties were examined. The modulus of elasticity designed for these films was 19.3 MPa. Comparing the mechanical characteristics of chitosan and PLLA films it can be seen that chitosan has a better performance than PLLA. As a consequence, all blends show a decrease in the elastic modulus.

It is well known that the mechanical properties of the composite films can be used to evaluate the compatibility of polymers by comparing the experimental results and estimates based on various models. The mechanical properties of the polymer blends depend on the inter-molecular forces, the conformation of the molecule (the rigidity of the main chain) and the symmetry of the molecule of the individual polymers used for the preparation of the mixtures. Furthermore, the modulus of elasticity of polymer blends is strongly dependent on the composition and morphology of the mixture and theoretically it is located between the upper limit set by the parallel theoretical model [18]

$$E = \phi_1 E_1 + \phi_2 E_2 \quad (3)$$

and a lower limit given by the series model

$$E = \frac{\phi_1}{E_1} + \frac{\phi_2}{E_2} \quad (4)$$

Moreover, for a co-continuous system, the modulus has to agree with the Davies model, whose the equation is given by

$$E = \phi_1 E_1^{1/5} + \phi_2 E_2^{1/5} \quad (5)$$

in which  $E_i$  and  $\phi_i$  are the elastic modulus and the weight fraction of phase  $i$ , respectively.

In Fig. 2 the experimentally obtained moduli of the chitosan/PLLA films are compared with the theoretical corresponding to ideal homogeneous systems. The following conclusions could be drawn:

1. The addition of a small amount PLLA (up to 0.2 weight fractions) does not interfere the homogeneity of the composite films. In this case, the reduction in elastic modulus is described by the parallel model.
2. Increase the volume of the PLLA in the films above 0.2 leads to degraded mechanical properties that cannot be described with any of the

theoretical models. The reduction of the modulus of elasticity is likely to be due to phase separation, which indicates that chitosan and PLLA are incompatible in the given volume ratios.

The degree elasticity of the Ch/PLLA films (Fig. 3) was determined by hysteresis test performed for force values that are in the linear section of the force-deformation curves. Relatively low degree of elasticity for pure chitosan films is an indication of a strong visco-elastic behavior. A possible reason for this observation could be the strong hydrophilicity of chitosan. As a result bound water in the films acts as a plasticizer which lowers the glass transition temperature and thus enhances the visco-elastic properties at room temperature. The increase of the PLLA weight fraction leads to a linear increase of the degree of elasticity. The addition of PLLA causes hydrophobic effect and reduction of water content; hence increase of films stiffness and strengthening of elastic properties. Except with increased hydrophobicity it can be explained by appearance of phase separation and structural changes of the films. Another possible reason is the presence of a crystalline structure in the PLLA, which reduces the molecules mobility and increase material rigidity.

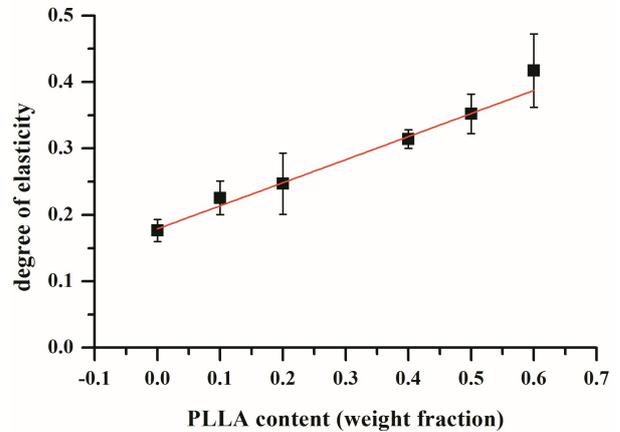


Fig. 3. Degree of elasticity for Ch/PLLA blends with different weight fractions of the components.

Some relaxation parameters of the Ch/PLLA films were determined by generalized Maxwell model in which the exponential function is modified by the stretched exponent [21, 22].

The stress relaxation is more rapid as the higher is the temperature and as the weaker are the macromolecular interactions, which depend on chemical structure of the polymer. In the case of two com-

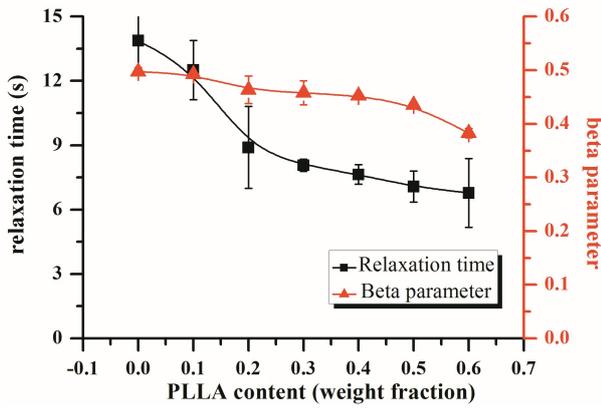


Fig. 4. Average relaxation time and  $\beta$ -parameter for Ch/PLLA blends with different weight fractions of the components.

ponent system the interactions between the components should be taken into account.

The dependence of the relaxation time of the content of the PLLA is presented in Fig. 4. It is noteworthy that the relaxation time decreases with increasing content of the PLLA. Reducing of the relaxation time shows that the films become more elastic and lose their viscous properties. This fact is fully consistent with the increase of the degree of elasticity that occurs with increased content of PLLA.

It is well known that spectrum of relaxation times is observed in the polymers, the width of which depends on the molecular weight distribution, the presence of cross-linkings, the presence of a crystalline phase, etc. The parameter, which describes the spectrum of relaxation times in the “stretched exponential” model, is the exponent  $\beta$ .

The dependence of the parameter  $\beta$  on the PLLA content in the composite film is shown in Fig. 4.

Increasing the weight concentration of PLLA leads to reduced values of the  $\beta$ -parameter. This fact should be analyzed as widening of the relaxation times distribution. Wider distribution could be con-

nected to the existence of two types of molecules with different flexibility, molecular weight and tendency to crystallize. It is also possible to relate it with inhomogeneous phase-separated areas with different size and hardness.

#### Thermal properties

Thermal properties of the Ch/PLLA composite films are investigated by DSC. It is known from the literature that in addition to the mechanical properties of polymer blends their glass transition temperature  $T_g$  is an important criteria for the miscibility of the components. In case of fully miscibility, just one value of  $T_g$  which is between the  $T_g$  values of the pure polymers is observed. If the two components are just partially miscible the  $T_g$  value of each component phase should be affected by the other one, and it is usually composition dependent [23].

The values for  $T_g$  the Ch/PLLA films are presented in Table 1. It is extremely difficult to determine the glass transition temperature of chitosan because the associated heat capacity changes are very small to be measured. The reason is the stiffness of the macromolecules and the strong intramolecular hydrogen bonds on the backbone [14,24]. Therefore the chitosan  $T_g$  was not determined in this work and only the changes in the PLLA thermal behavior will be discussed. However, according to some authors the  $T_g$  of chitosan is about 194°C and in case of miscibility increasing in the  $T_g$  values of PLLA is expected.

Based on the experimental results the increase of the chitosan weight fraction in the composite films lead’s to a decrease in the  $T_g$  values. Therefore an assumption could be done that there is no compatibility of the components on microlevel. The decreasing  $T_g$  could be explained by some additional action of chitosan – most probably the increased hydrophilicity of the composite films causes higher amount of bound water, which acts as a plasticizer.

Table 1. Thermal properties of Ch/PLLA composite films, investigated by DSC.

Ch/PLLA weight fraction	$T_g$ (°C)	$T_1$ (°C)	$\Delta H_1$ (J/g)	$T_2$ (°C)	$\Delta H_2$ (J/g)	Crystallinity (%)
10:0	-	—	—	—	—	—
9:1	41.68	145.21	1.78	166.03	4.17	64
8:2	45.12	143.90	1.66	165.25	10.27	64
7:3	48.07	143.31	2.34	163.32	13.94	58
6:4	51.23	143.54	3.43	163.90	19.70	62
5:5	64.53	144.67	3.91	165.86	23.88	60
4:6	65.51	149.42	4.82	167.51	28.44	60

For all composites two endothermic peaks at about 145°C and 165°C are observed. They are related to the fusion process of the PLLA. The lower temperature of fusion could be due to some degradation processes of PLLA (which are engaged at the time of dissolving) or to different crystal form. The clarification of the observed melting behaviour will be an object of further investigations.

Crystallinity may influence strongly the properties of PLLA-based materials, including its glass transition dynamics and mechanical behavior [25, 26]. From the DSC results it is possible to calculate the crystallinity degree ( $X_c$ ) of the PLLA fraction by applying the following equation:

$$X_c = \frac{\Delta H_m}{\Delta H_m^0 \omega}, \quad (6)$$

where  $\Delta H_m^0$  is the melting enthalpy of fully crystalline PLLA (93 J/g [27]) and  $\omega$  is the weight fraction of PLLA in the blend.

These values are also included in Table 1. It can be seen that the degree of crystallinity of PLLA is not affected by the presence of chitosan and is relatively high. Thus, the PLLA phase in the blends prepared by the developed method was present predominantly in the crystal state. This observation could be associated with the increased elastic properties of composites with higher PLLA content.

#### Morphological analysis

To get a clear picture of the morphology of the composite films SEM analysis has been done. SEM micrographs for cross-sections of various films are shown in Fig. 5.

It is noting that the film of pure chitosan is possesses very homogeneous and dense structure. The observed fractures and micro-cracks are due to fracture of the specimen during sample preparation. Therefore it could be concluded that the selected two-component solvent (formic acid/acetone) completely dissolves the chitosan.

The addition of PLLA rises to a porous structure, in which no structured texture is observed. Pores are irregular in shape and their sizes are of the order of several microns. The size and the surface roughness of the domains forming the film differs depending PLLA content.

Increasing the amount of the added PLLA leads to an increase in the size of the domains. It is clear from the microphotographs, that the domains are built-up from both polymers. Domains at a low concentration of PLLA apparently seem homogeneous, and only in rare cases smaller spherical formations are observed on the surface of the domains. In this case, most probably the beginning of the phase separation is observed, assuming that the entities are precipitated on the surface PLLA drops. The increase of PLLA leads to a drastic change in the surface topography of the domains. Spherical formations with dimensions of the order of 0.5-1.0 microns grow on the surface, which later merged to form a rough unordered topography.

The observed by SEM morphology is evidence for the occurrence of phase separation (as was suggested on the basis of mechanical characteristics), which subsequently influence the macroscopic properties of the films. Most probably the broader spectrum of relaxation time, which is noted during the mechanical relaxation experiments, is a result of proved phase separation and immiscibility of PLLA and chitosan.

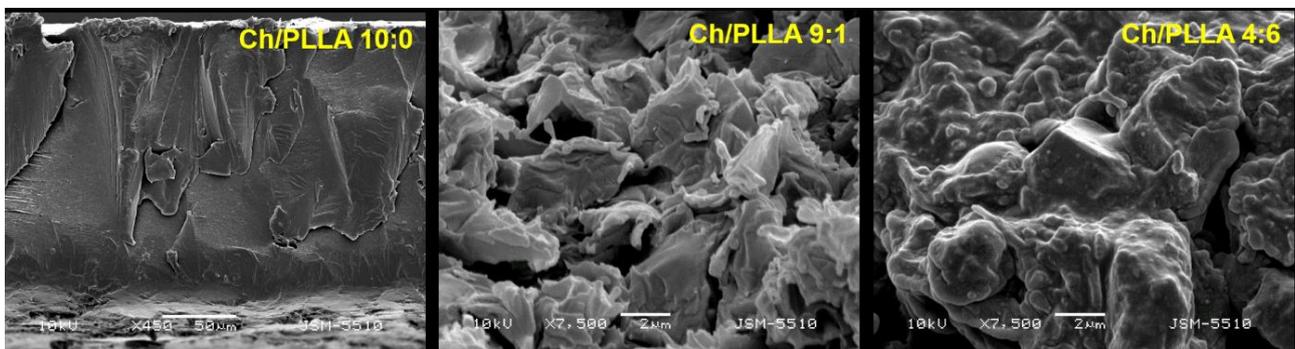


Fig. 5. SEM micrographs of pure chitosan, Ch/PLLA blend film, weight fraction 9:1 and Ch/PLLA blend film weight fraction 4:6.

## CONCLUSIONS

A new two component solvent system to mix chitosan and PLLA for casted blends is presented. The proposed method resulted in macroscopically homogeneous blends, which possess technological miscibility at low PLLA content (less than 20%). Thermal and morphological analysis pointed to phase separation at the microscopic level between the two polymers, independently of the composition of the studied blends.

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РЕОЛОГИЧНИ СВОЙСТВА НА КОМПОЗИТНИ ФИЛМИ НА ОСНОВАТА  
НА ХИТОЗАН И БИОРАЗГРАДИМИ ПОЛИЕСТЕРИ

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(Резюме)

В настоящата работа е изследвано реологичното поведение при едноосна деформация на композитни филми от хитозан/полимлечна киселина и хитозан/поли-ε-капролактон. Филмите са получени при различни масови части на двата полимера в разтворител, представляващ смес на мравчена киселина и ацетон в обемно отношение 1:1. Филмите са отляти в петрита от тефлон и изсушени до постоянна маса в сушилня при температура 35°C. Реологичните тестове са проведени при постоянна скорост на деформация 0.1 mm/s и стайна температура. От основната физико-механична крива за композитните филми са определени точката на разрушаване и модула на еластичност. От проведените тестове на механичен хистерезис са изчислени коефициента на еластичност и остатъчната деформация. Получените реологични параметри са пряко свързани с експлоатационните характеристики на филмите при приложението им за опаковъчен материал.