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RESEARCH ARTICLE

Gas permeability of films based on low-density polyethylene–ethylene-vinyl acetate blends with cellulosic fillers

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Abstract

Objectives. The work set out to characterize the gas permeability properties of biocomposite materials based on synthetic polymers and natural fillers.

Methods. The studied materials were blends of low-density polyethylene (LDPE) and ethylene–vinyl acetate (EVA) copolymer, with different LDPE/EVA ratios, as well as biocomposites based on these polymers with natural cellulosic fillers (wood flour (WF) and microcrystalline cellulose (MCC)). The coefficients of gas permeability, diffusion, and oxygen solubility were determined in the obtained composites using the manometric method. The dependence of the diffusion properties of LDPE/EVA blends and biocomposites made of LDPE/EVA/natural filler on the EVA content in the composite was considered.

Results. We demonstrated that, as the EVA content in the polymer matrix increases, so also do its solubility and coefficients of gas permeability and oxygen diffusion. The variation in the diffusion characteristics of biocomposite materials obtained using solid filler particles that differ significantly in shape is characterized. The presented interpretation of the obtained results explains the decrease in diffusion in terms of increased rigidity of biocomposites.

Conclusions. An increase in the EVA content in blends with LDPE leads to a linear increase in the gas permeability for oxygen, as well as enhanced diffusion and solubility of oxygen in the film. Upon adding a cellulosic filler, the gas permeability of the composites drops almost twofold. The decrease in gas permeability is associated with the morphology of the filler particles increasing the path of gas molecules. Oxygen solubility for composites with MCC and WF is not the same due to the shape of the filler particles. Rough and more elongated WF particles form a more rigid, less permeable structure of the biocomposite than smooth spherical MCC particles.

Keywords

gas permeability, diffusion, microcrystalline cellulose, wood flour, ethylene-vinyl acetate copolymer blends, biocomposites

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НАУЧНАЯ СТАТЬЯ

Газопроницаемость пленок на основе смесей полиэтиленов низкой плотности – сэвиленов с целлюлозосодержащими наполнителями

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Аннотация

Цели. Изучение свойств газопроницаемости биокомпозитных материалов на основе синтетических полимеров и природных наполнителей.

Методы. Объектами исследования являлись смеси полиэтилена низкой плотности (ПЭНП) и сополимера этилена с винилацетатом (СЭВА), при различном соотношении ПЭНП/СЭВА, а также биокомпозиты на основе данных полимеров с природными целлюлозосодержащими наполнителями (древесная мука (ДМ) и микрокристаллическая целлюлоза (МКЦ)). У полученных композитов манометрическим методом определяли коэффициенты газопроницаемости, диффузии и растворимости по кислороду. Рассматривалась зависимость диффузионных свойств полимерных смесей состава ПЭНП/СЭВА и биокомпозитов состава ПЭНП/СЭВА/природный наполнитель от содержания СЭВА в композитах.

Результаты. Показано, что с увеличением содержания СЭВА в полимерной матрице увеличивается коэффициент газопроницаемости, коэффициент диффузии кислорода и его растворимость. Показана разница диффузионных характеристик биокомпозиционных материалов, полученных с использованием твердых частиц наполнителей, существенно различающихся по своей форме. Дана интерпретация полученных результатов, объясняющая снижение диффузии повышением жесткости биокомпозитов.

Выводы. С повышением содержания СЭВА в смеси с ПЭНП линейно увеличивается газопроницаемость по кислороду. Также при этом увеличиваются диффузия и растворимость кислорода в пленке. При введении целлюлозосодержащего наполнителя, газопроницаемость композитов падает практически в два раза. Очевидно, что снижение газопроницаемости связано с морфологией частиц наполнителя, увеличивающего путь молекулам газа. Растворимость кислорода для композитов с МКЦ и ДМ не одинакова, что связано с формой частиц наполнителей. Шероховатые и более вытянутые частицы ДМ формируют более жесткую, менее проницаемую структуру биокомпозита, чем гладкие сферические частицы МКЦ.

Ключевые слова

газопроницаемость, диффузия, микрокристаллическая целлюлоза, древесная мука, смеси сополимера этилена с винилацетатом, биокомпозиты

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INTRODUCTION

The most inert of the polyolefins, polyethylene undergoes slow degradation in the environment [1]. At the same time, it holds the first place in terms of production volumes among the whole class of polyolefins. The valuable properties combined in this material have led to the widespread use of polyethylene in industry and for domestic purposes [2]. Polyethylene does not react with alkalis, is resistant to acids and salts, and is inert to the action of microbiota from the environment [3–4]. At the same time, polyethylene offers gas and vapor permeability. Gas permeation through the films can be considered as a two-stage diffusion of liquid. In the first stage, the diffusant is equilibrated by the film surface, while in the second stage, it diffuses towards a lower chemical potential. The gas permeability of the films determines whether the material can be used for packaging purposes. For certain packaged products, the gas permeability of the film should be high, while for others, conversely, it should be minimal [5]. Thus, this characteristic determines the scope of application of the material [6]. The gas permeation rate of a film depends largely on temperature [7]. Thus, R. Ahvenainen [8] proposed to conduct a study of automatic packaging at 10, 15, 20, and 25°C to determine O_2 and CO_2 gas permeation rates of selected films and film laminates at these temperatures. Then, as noted by the authors [9], an attempt was made to apply the Arrhenius equation to develop models to predict the gas permeation rates of selected polymer films and film laminates at different temperatures. It is found that film thickness and test temperature have a significant effect on gas permeability. However, the gas permeation rate of films is more affected by their thickness than temperature.

A recent study has shown that in some partially crystallizing polymers (gutta-percha, polyethylene, polyamide) after reaching the limiting orientation of macromolecules, gas permeability decreases due to an additional increase in the degree of crystallinity or an increase in the packing density of amorphous polymer regions [10].

The change in gas permeability values (*P*) is due to the change in the diffusion coefficient (*D*), while the gas solubility coefficient does not change significantly when the film sorbs organic solvents. The gas permeability values of polyethylene for O_2, N_2, CO_2 are not affected by the relative humidity of the gases. Meanwhile, the gas permeability of a mixture of gases often depends on the high solubility of one of the gases included in the mixture. Thus, P.V. Kozlov and G.I. Braginskii in their study of polyethylene in relation to a mixture of ethane and butane-4 showed that the permeability of the mixture increases with increasing butane concentration as compared to the calculated value, which is based on the initial *P* coefficients [11]. The anomaly of the values of gas permeability and CO_2 diffusion coefficients is associated with high values of carbon dioxide sorption by polyethylene. This phenomenon was also noted in other works.

Many researchers have obtained bulk nanocomposite structures by introducing nanoparticles—i.e., fillers having at least one size smaller than 100 nm—into polymer matrices. This has proven to be one of the most promising directions in the development of packaging materials with improved mass transfer properties. The expected role of these inclusions is to achieve a significant reduction in mass transfer properties compared to the original matrix. The filler particles act as physical barriers to diffusion and penetration of diffusing molecules, which must follow a more tortuous path. This phenomenon is called the tortuosity effect or increase in the length of the diffusion path. However, N.N. Barashkov [12] concluded that tortuosity, the main mechanism proposed to explain the decrease in permeability of nanocomposites, often fails to explain the effect of nanoparticles on the barrier properties of nanocomposites.

Note that the analysis of current experience published on various profile forums, websites of organizations, as well as in scientific journals allows us to conclude that the use of biodegradable composites and polymers derived from various renewable sources is currently preferred [13]. The use of eco-friendly materials helps to protect the environment as these materials degrade rapidly in it. Thus, the number of products made from renewable resources has been increasing rapidly in recent years. Applications of green products include food packaging, drug delivery, tissue engineering, medical implants, composite technologies and eco-friendly sorbents [14].

In recent years, interest in the use of biodegradable materials has been increasing due to their eco-friendly, renewable and environmentally friendly properties. In addition, certain specific properties such as low toxicity and density, biocompatibility and biodegradability favor their replacement of synthetic materials. Certain authors have pointed out the importance of cellulose, chitin and starch for the development of environmentally friendly composites [15, 16]. These fillers have played a significant role in the success of developing high strength composites [17].

In [18], lignosulfonates (a byproduct of wood processing) were introduced into polypropylene, which led to an increase in thermal stability. The introduction of more than 50 wt % wood flour (WF) into ethylene-vinyl acetate (EVA) copolymer [19] significantly increased the thermal oxidation resistance of the material due to the diffusion of polyphenolic components (natural antioxidants) from wood into the synthetic polymer during compounding. Thus, biocomposites with wood components are well protected from oxidative degradation during processing and can withstand several recycling cycles.

There are many works devoted to the study of polyethylene composites with natural polymeric fillers such as starch, cellulose, cellulose-containing particles, chitosan, etc. [20–23]. These composites are created to impart biodegradable properties to traditional polyolefins. To improve adhesion between polar natural filler and nonpolar polyethylene matrix, combining additives (compatibilizers) are introduced. One such additive is EVA. In our previous studies, EVA-based superconcentrates filled with WF and microcrystalline cellulose (MCC) were obtained and investigated [24–26]. In these works, biocomposites made from previously obtained superconcentrates were investigated by dilution with low density polyethylene (LDPE). Biocomposites with WF content of 30, 40, 50, 50, 60, 70 wt % were investigated in [27]. When the filler content was higher than 30 wt %, a significant decrease in the complex of mechanical properties was observed. In this study, the filler content for all biocomposites was selected at

Table 1. Characteristics of fillers

Filler	Cellulose, %	Lignin, %	Pentosans, %	Others, %
Wood flour (WF)	46	20	29	5
Microcrystalline cellulose (MCC)	100	–	–	–

Table 2. Characteristics of synthetic polymers

No.	Polymer	Vinyl acetate group content		Melt flow index (MFI), g/10 min	Tensile strength, MPa	Elongation at break, %	Density, g/cm^3
		wt %	mol. %				
1	EVA 28025	28	11	24.8–25.2	>12.0	>850	0.950–0.952
2	LDPE 10803-020	–	–	1.8–2.2	>12.2	>550	0.917–0.920

30 wt % as comprising the limiting filler content in the polymer matrix while maintaining acceptable operational and technological characteristics. The aim of the work was to study their gas permeability in order to determine possible applications.

MATERIALS AND METHODS

For the composites manufacturing, LDPE grade 10803-020 produced by *Ufaorgsintez* (Russia), EVA grade EA 28025 produced by *LG Chem* (South Korea) were used. The fillers used were MCC grade 101 produced by *PO Progress* (Kemerovo, Russia) and WF, comprising a blend of coniferous and deciduous trees supplied by *Novotop* (Moscow, Russia). The fillers were pre-dried at 105°C to constant weight (~5 h), then sieved through a sieve with a mesh diameter of 100 µm. Table 1 shows the composite of the fillers, Table 2 shows the characteristics of the initial synthetic polymers (LDPE and EVA).

Binary (LDPE/EVA) and ternary (LDPE/EVA/filler) composites were created having different ratios of LDPE/EVA in the blend (Table 3).

The composites were made on UBL6175BL laboratory heated rollers (*Dongguan BaoPin International Precision Instruments Co.*, China) at roll temperatures of 130 and 150°C and a mixing speed of 8 rpm. After milling the obtained composites using a PM 120 knife mill (*Vibrotechnik*, Russia), they were thermally pressed using a GT-7014-H press (*Gotech Testing Machines Inc.*, Taiwan) at 140°C and a plate clamping force of 15 tons for 2 min; cooling was performed in air. The resultant films having a thickness of 250–270 µm were then used to determine the density and oxygen gas permeability.

Table 3. Component ratios in binary (LDPE/ EVA) and ternary (LDPE/ EVA/filler) composites

Binary composites		
No.	LDPE content, wt %	EVA content, wt %
1	100	0
2	80	20
3	70	30
4	60	40
5	50	50
6	40	60
7	20	80
8	0	100

Ternary composites			
No.	Natural filler, wt %	LDPE/ EVA ratio in the polymer matrix	
		LDPE, wt %	EVA, wt %
1	30	70	0
2		57	13
3		50	20
4		40	30
5		35	35
6		0	70

Gas permeability was measured on a VAC-V1 instrument (*Labthink*, China) according to ISO 15105-2:2003[1]. The gas permeability was measured in oxygen (O_2) at a chamber temperature of 30°C with a measurement range of 0 to 100000 $cm^3/m^2 \cdot 24\ h \cdot 0.1\ MPa$. In addition to the gas permeability coefficient, the diffusion coefficient and solubility coefficient were also determined. ISO 15105-2:2003 provides a formula for calculating gas permeability:

$$\mathrm{GTR} = \frac{V_C}{R \cdot T \cdot P_h \cdot A} \cdot \frac{dp}{dt},$$

where GTR is gas transmission rate; V_C is the volume of the lower pressure chamber, liters, T is temperature, K; P_h is the gas pressure in the high pressure chamber, Pa; A is the area of the sample through which the diffusion gas passes, m^2; R is the universal gas constant, $J \cdot K^{-1} \cdot mol^{-1}$; $\frac{dp}{dt}$ is the pressure change over time in the low-pressure chamber, Pa/s. Multiplying GTR by the average thickness of the sample, we obtain the gas permeability coefficient P.

Microphotographs of filler particles were obtained by scanning electron microscopy (SEM). A Vega 3SB scanning electron microscope (*TESCAN GROUP*, Czech Republic) was used, having an accelerating voltage of 20 kV, a working distance from 9 to 15 mm, and a platinum coating.

RESULTS AND DISCUSSION

The oxygen gas permeability of films with double (PE/EVA) and ternary composites (PE/EVA/filler) was studied. Figure 1 shows the dependencies of the gas permeability coefficient on the EVA content in the polymer matrix for double and ternary composites. The gas permeability coefficient values increase with increasing EVA content in double blends with LDPE, this dependence has a linear form.

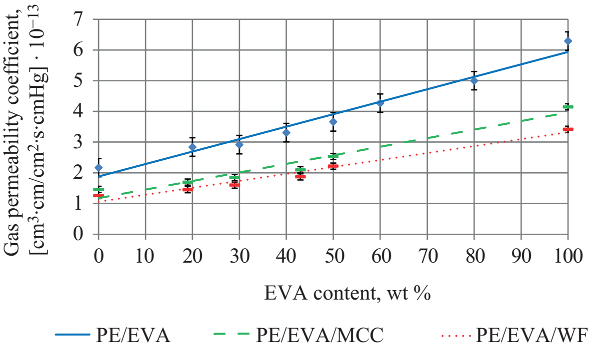
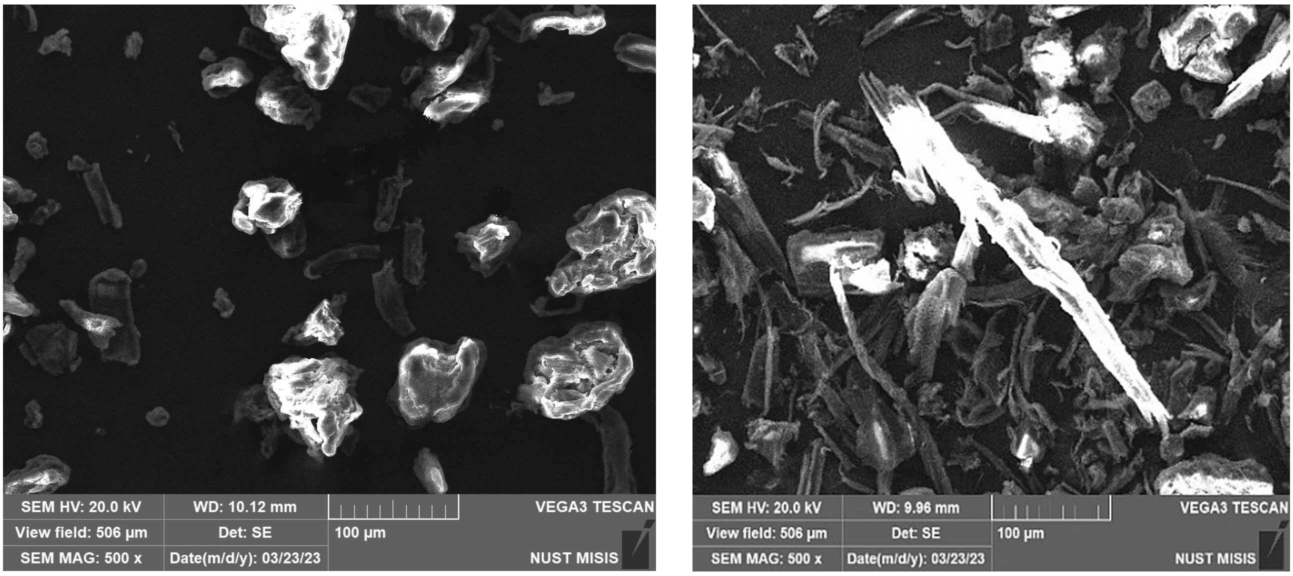


Fig. 1. Dependence of oxygen gas permeability coefficient on EVA content in the polymer matrix

At introduction of 30 wt % of natural filler, the gas permeability decreases almost two-fold. The gas



(a)

(b)

Fig. 2. Microphotographs of filler particles:
(a) MCC, (b) WF. Scanning electron microscopy (SEM)

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[1] ISO 15105-2:2003. Interstate Standard. Plastics. Film and sheeting. Determination of gas-transmission rate. Part 2: Equal-pressure method. Moscow: Standartinform; 2010. 12 p. (in Russ.).]

permeability of composites with WF is less than that of composites with MCC. At the same time the type of dependence does not change, while the curve is unaffected by the introduction of filler. The difference in the values of gas permeability coefficients for composites with MCC and WF can be explained in terms of the different morphology of filler particles. The shape and size of filler particles were investigated using SEM. Microphotographs of the particles are presented in Fig. 2.

As can be seen from Fig. 2, WF particles are more elongated and branched, while MCC particles are more spherical in shape and have a smooth surface. Accordingly, the greater the filler particle length, the longer the diffusion path of the gas passing through the film. When films are fabricated by pressing, the WF particles are arranged uniformly in its plane, i.e., perpendicular to the direction of oxygen flow through the film. It was shown in [28] that the diffusion rate was 1 or 2 orders of magnitude higher in the axial direction of the wood fibers than in the radial direction. The diffusion of oxygen through wood was studied in [29] to evaluate the oxidation of wine stored for extended periods of time in oak barrels. The study shows that the very low rate of oxygen diffusion through wood depends on the type of wood. An approximate scheme of the gas path through films with spherical and cylindrical filler particles is shown in Fig. 3. The oxygen diffusion path length can additionally be influenced by the contact area of the filler particles with the polymer matrix. Here, the free volume of the polymer is smaller when the contact area with the filler is larger. The interface surface of biocomposites made of polyethylene with WF was studied in [30] along with the structure of biocomposites based on polyethylene filled with WF or oilseed flax stems. When the free volume of the polymer decreases, the gas permeability should decrease. These factors explain the lower gas permeability of biocomposites with WF compared to MCC. It was shown in [30] that biocomposites with WF absorbed oxygen significantly. Oxygen sorption with oxidation of low molecular weight substances can explain the lower gas permeability of biocomposites with WF.

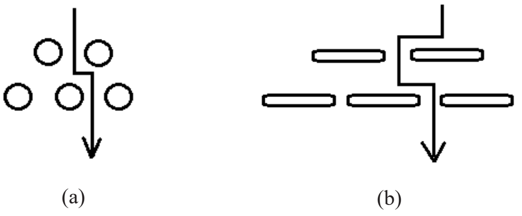
(a) (b)

Fig. 3. Model of diffusion of a gas molecule in a polymer filled with particles of spherical (a) and cylindrical (b) shapes

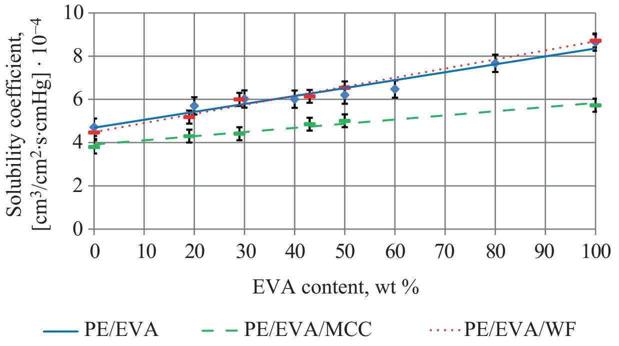

Fig. 4. Dependence of oxygen solubility coefficient on EVA content in the polymer matrix

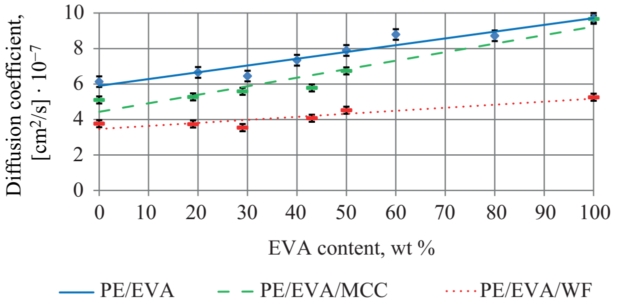

Fig. 5. Dependence of oxygen diffusion coefficient on EVA content in the polymer matrix

Figures 4 and 5 show the dependencies of the diffusion coefficient and oxygen solubility coefficient in the films of ternary and double composites on the EVA content in the polymer matrix. With increasing EVA content in the blend, a linear increase in the values of these two parameters is observed. Oxygen solubility in biocomposites containing WF is the same as in composites without filler, while the presence of MCC particles markedly reduces oxygen solubility in the composite. Fragments of EVA macromolecules, which have a certain mobility, can shift upon introduction of gas molecules. This occurs for the pure polymer and for biocomposites with MCC. However, the stiffer polymer chains have less mobility. Biocomposites with WF particles are less elastic (stiffer), as can be seen when comparing their mechanical properties. Thus, it was shown in [24] that the elongation at break of biocomposites with WF is about 5 times lower than that with MCC. At the same time, the elastic modulus of biocomposites with WF is significantly higher than that of composites with MCC (Table 4). Prior to the creation of ternary biocomposites, we investigated the change in elastic modulus of highly filled binary EVA biocomposites with natural fillers (WF and MCC). Table 4 shows the values of elastic modulus of highly filled biocomposites with filler content (WF or MCC) of 50 wt %. The presented values allow us to conclude that biocomposites with WF are much stiffer than biocomposites with MCC. It can be seen that the composites with WF have a higher modulus. Due to the increased stiffness of biocomposites with WF and the increased path length of diffusing gas molecules, a significant difference with the original polymer matrix in terms of diffusion coefficient is observed (Fig. 5).

Table 4. Modulus of elasticity of EVA 28025 based composites

Tensile modulus of elasticity, MPa		
Without filler	MCC 50 wt %	WF 50 wt %
23 ± 2	108 ± 5	176 ± 5

The obtained results are not only important for the physical chemistry of polymers, but also have applied significance. When using biocomposites as raw materials for the production of packaging films, the oxygen gas permeability index for such products is of crucial importance since determining the shelf life of food products. By obtaining an understanding the dependence of gas permeability on the shape of filler particles and the ability to vary their shape and content in the polymer matrix of the composite, it is possible to create biocomposite materials with specified gas-permeability parameters.

CONCLUSIONS

The diffusion properties of biocomposite materials based on LDPE and EVA and having MCC or WF as fillers were studied. An increase in the EVA content in blends with LDPE was shown to lead to a linear increase in the oxygen gas permeability along with enhanced diffusion and oxygen solubility coefficients in the film. Upon adding cellulose-containing filler, the gas permeability of the composites is reduced approximately two-fold. The decrease in gas permeability is probably due to the morphology of the filler particles. While the oxygen solubility for composites with MCC and WF is not the same, it may also be related to the morphology of filler particles. The rough and elongated WF particles form a stiffer and less permeable structure than the smooth spherical MCC particles. The established regularities can be used to evaluate and predict the barrier properties of film materials made of biocomposites with fillers having different morphologies.

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Authors' contributions

P.G. Shelenkov—conducting the study.

P.V. Pantyukhov—approval of the final version of the article.

A.A. Olkhov—preparing and editing the text of the article.

A.A. Popov—development of the research concept.

The authors declare no conflicts of interest.

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