EVALUATION OF OXYGEN PERMEABILITY OF POLYETHYLENE FILMS

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A b s t r a c t

The paper dealt with the evaluation of the oxygen permeability of the polyethylene films applied in the mulching and food packaging. The construction of the equipment is discussed. The design and the construction of the equipment was realized with applying of the standard EN STN 77 0333.The measurement of the permeability of the oxygen trough the polyethylene Bralen 2–63 with 9% colored concentrate Maxithen HP 533041 – violet film was realized by means of modified method. The values of the permeability P_x, 1794.25 cm$^3 \cdot$ m$^{-2} \cdot$ d^{-1}. (0.1 MPa)$^{-1}$ of the oxygen trough the film of the thickness of 50 µm was evaluated. The coefficient of permeability P, 4.2560 $\cdot$ 10^{-16} mol $\cdot$ m$^{-1} \cdot$ s$^{-1} \cdot$ Pa^{-1}, coefficient of diffusion D, 4.3999 $\cdot$ 10^{-10} m$^2 \cdot$ s^{-1} and solubility coefficient of the gas in the film S_p, 9.6735 $\cdot$ 10^{-7} mol $\cdot$ m$^{-3} \cdot$ Pa^{-1} were determined.

Introduction

Materials suitable of packaging of foods and mulching are applied on the base of polymers in the present time. The protection of original quality of food against external undesirable effects is the function of the packaging materials. Required protection of foods can be achieved with one layer of polymer, or in the case of need, with multilayer film, includes different polymers, surface films and metallic films. Barrier properties, i.e. protection of the package, are related mainly with the ability to transmit the gases and vapors, which are damaging of the quality of the product. Degradation processes of foods are dependent on the time and temperature (JASSE et al. 1994, ASHLEY 1985, PYE et al. 1976). The oxygen is harmful for foods of vegetal or animal origin. It causes the oxidation of the higher fatty acids. Internal atmosphere of gases, as are CO_2 a N_2, is modified of the preservation of the quality of the foods (JASSE et al., 1994). Polyethylene plastic films have great importance in the horticul-

ture. The plastic films applied as mulch, affect the radiation balance of the environment by means of absorption and reflection of the light by their surface and they change of the microclimate of the cultivated plants (TABER 2010).

WANG et al. (1998) studied oxygen and ethylene permeabilities which have been determined at 19ºC for three kinds of polyethylene films (LDPE, LLDPE and HDPE) and in the temperature range 4–30ºC for LDPE. At constant temperature, ethylene and oxygen permeabilities decrease with increasing density. The temperature dependence of oxygen and ethylene permeabilities was found to be Arrhenius in the temperature range tested, with activation energies of 47.7 and 44.1 kJ/mol for ethylene and oxygen respectively.

PAPIERNIK et al. (2002) describe an apparatus useful for obtaining permeability data. The model may be fitted to the data to determine mass transfer coefficients. The assembled equipment provides a sealed permeability cell, where a sample of the film to be tested is sandwiched between two static half-cells. Vapor is spiked to one side of the film and the concentrations in the spiked and receiving chamber are monitored until equilibrium. The permeability cells described here were gas-tight for >40 d. This approach produces reproducible measures of mass transfer coefficients that are not dependent on the size of the experimental apparatus.

KAMAL et al. (1984) studied oxygen and water vapor permeability on binary polyethylene/polyamide immiscible blends incorporating three polyethylene resins (LDPE, LLDPE, and HDsPE), and three polyamide resins (PA-6, PA-6,6, and modified PA-6,6 m). It was found that the incorporation of PA into PE reduces the oxygen permeability while water vapor permeability is increased. In the range of 0 to 30 weight percent of PA, the oxygen permeability of PE was reduced by a factor of 2.8 to 3.6. Maximum water vapor permeabilities increased: for HDPE by a factor of about 2.6 to 3.1 and for LDPE and LLDPE blends by about 1.6.

Material and Methods

Fundamental principles of the permeability

Sorption of gases and their transmission trough the polymer depends on the permeability and the diffusion. Amount of gas Q (mol), which is transfer through the membrane, is defined by the equation (JASSE et al. 1994):

$$Q = \frac{D \cdot \sigma \, (p_1 - p_2) \, S \cdot t}{h} \qquad (1)$$

where:
D – coefficient of diffusion, m^2/s,
p_1 – external pressure, Pa,
p_2 – internal pressure, Pa,
h – thickness of membrane, m,
σ – Henry's constant, mol $\cdot$ Pa/m^3,

On the assumption of thermodynamic equilibrium, the coefficient of permeability P is given by the equation:

$$P = D \cdot \sigma \tag{2}$$

Permeability is depended on solubility and diffusivity. These quantities are functions of optional volume, cohesive energy and polymer morphology. Than coefficient of permeability is defined:

$$P = \frac{Qh}{S(p_1 - p_2)\, t} = \frac{Qh}{S\Delta pt} \tag{3}$$

After applying equation of state for ideal gas $pV = nRT$ we can obtain equation of coefficient of permeability P [mol $\cdot$ m^{-1} $\cdot$ s^{-1} $\cdot$ Pa^{-1}] suitable of experimental measurement:

$$P = \frac{Qh}{S\Delta\, pt} \cdot \frac{pV}{nRT} = \frac{pVh}{tS\Delta pRT} \tag{4}$$

where:
P – coefficient of permeability P, mol $\cdot$ m^{-1} $\cdot$ s^{-1} $\cdot$ Pa^{-1},
p – pressure, Pa,
V – volume of gas, m^3,
R – universal gas constant, $R = 8314$ J $\cdot$ kmol^{-1} $\cdot$ K^{-1},
T – temperature, K.

We can also obtain permeability of the membrane P_x [m^3 $\cdot$ m^{-2} $\cdot$ s^{-1} $\cdot$ Pa^{-1}] from the equation:

$$P_x = P\,\frac{V}{Qh} \tag{5}$$

Fick's second law of diffusion can be obtained by first calculating a conservation equation with respect to volume,

$$\frac{\partial C_h}{\partial t} = D \left(\frac{\partial^2 C_h}{\partial z^2} + \frac{1}{S} \frac{\partial S}{\partial z} \frac{C_h}{\partial z} \right) \qquad (6)$$

where:

C_h – molar concentration, $mol \cdot m^{-3}$,

z – coordinate, m.

By setting S to be constant, equation (6) describes unsteady state diffusion, which is Fick's second law of diffusion. Together, Fick' first and second laws describe how much solute moves across the film (diffusion flux) and reveal how the solute concentration changes within the film (concentration profile). To apply Fick's laws, certain assumptions must be made (Fig. 1). Let C_{1h} be the concentration of downstream solution and let C_{10} be the concentration of upstream solution. If H is the partition coefficient which relates pressure in gas to concentration in the film, then $c_{10} = HC_{10}$.
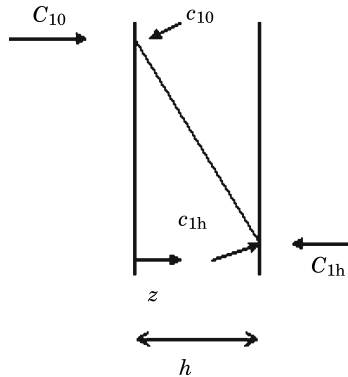

Fig. 1. Steady-state concentration profile in a thin membrane. C_{10} is proportional c_{10} and the partition coefficient H. Infinite reservoirs on both sides are assumed

Initially, the downstream solution is assumed to be free of upstream solution. At time zero, the membrane is also assumed to be free of upstream solution. For $t > 0$ the amount of upstream solution in the membrane is c_{10} at $z = 0$: Furthermore, where $z = l$ the amount of upstream solution is said to be zero.

The boundary conditions restrict the downstream solution to be free of the upstream solution. In experiments it has been found that the concentration is negligible. These boundary conditions allow equation (6) to be transformed into an ordinary differential equation, which will describe a pseudo-steady state in the system.

SIEGEL and CUSSLER (2004) describe the characteristic of lag time in the diffusion model. After using separation of variables on Fick's second law and imposing the aforementioned conditions to obtain:

$$c_h = c_{h0} - \frac{c_{h0}z}{h} - \frac{2c_{h0}}{\pi} \sum_{n=1}^{\infty} \sin\left(\frac{n\,\pi\,z}{h}\right) \exp\left(\frac{-Dn^2\,\pi^2\,t}{h^2}\right) \tag{7}$$

And after calculation the limit of the above equation at large times:

$$\frac{c_h}{c_{h0}} = \frac{SD}{Vh}\left(t - \frac{h^2}{6D}\right) \tag{8}$$

where V is the ratio of the volume in the air chamber to the area of the fabric. This limiting equation revealed that there is a lag time for the system to reach the desired pseudo-steady state. Namely, the system can not be described by an ordinary differential equation when

$$t < \frac{h^2}{6D} \tag{9}$$

The limiting equation also reveals that the permeability of the membrane can be found experimentally by calculating the best-fit slope of the equation. Similarly the lag time Θ (s) can be found by experimentally calculating the x-intercept of the best-fit line of the pseudo-steady state data points (Fig. 2).
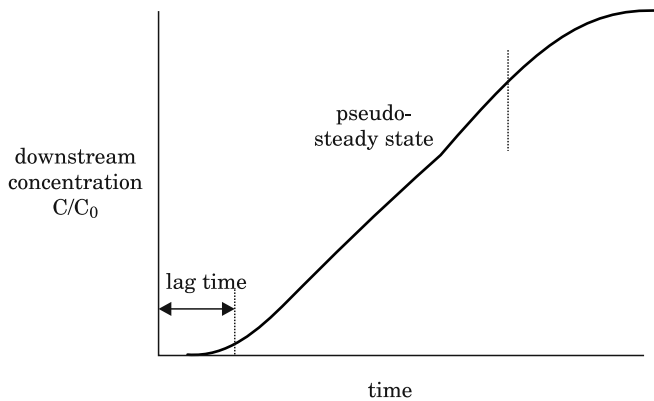

Fig. 2. Typical permeation/lag time curve: normalized downstream concentration versus time in a thin membrane initially free of solute. The code created estimates the pseudo-steady state section (RUTHERFORD, DO 1997)

Then we can obtain the coefficient of diffusion D [m$^2 \cdot$ s^{-1}] (PAULY 1999) from the equation (8):

$$D = \frac{h^2}{6\,\Theta} \tag{10}$$

where:

h – thickness of film, m,
Θ – lag time, s.

The solubility coefficient S_p [mol $\cdot$ m$^{-3} \cdot$ Pa^{-1}] can calculate from the equation (BROŽOVÁ 2008):

$$S_p = \frac{P}{D} \tag{11}$$

where:
P – coefficient of permeability P, mol $\cdot$ m$^{-1} \cdot$ s$^{-1} \cdot$ Pa^{-1},
D – diffusion coefficient, m$^{-2} \cdot$ s^{-1}.

The solubility coefficient S_p express the solubility of the oxygen in the film. Methods of determination of gas permeability are described in JASSE et al. (1994), PYE et al. (1976), KOROS et al. (1992), SOHAIL (1997). Polymer's materials present large scale structures and properties which depend on their chemical structure, methods of preparation and conditions of processing. Significant participation of additives and application of polymer mixtures influence barrier properties of films trough CO_2, O_2, N_2, or water vapors (LEE 1980).

Standard method

The nature of the test of the gas isobaric method is the interferometric determination of the concentration of the tested gas which penetrated trough tested sample from the chamber filled with the pure tested gas to the chamber with the air. Total gas pressures on either side of the sample are equal and the diffusion of the gas trough the tested sample depends on the difference of the partial pressure in the both chambers (EN STN 77 0333).

Instrument as well as standard procedures for measuring the oxygen permeability are available but measurement of the concentration of the oxygen is proposed by means of optical interferometric method with the laboratory

interferometer with two-part gas chambers of the length 100, 50, 25 and 10 cm. This method is difficult and the interferometer is not available in common. The digital oxygen meter Mesura which measured the relative concentration of the oxygen was used instead of interferometer and the absolute values of concentration had to be calculated from the tabulated values of the air and oxygen at the real conditions. All others conditions of the standard were kept to the terms.

Design of measurement equipment

Methods of detection and measurement of parameters of permeability of packaging materials of gases are specified in the standard EN STN 77 0333. The method was modified for the conditions of our test. Permeability of gases is determinated at the barrier tests of the materials as one of the specific parameter of the protective efficiency of the packaging. The design of measurement equipment was projected. The design is considered universal for the basic measurements. It represents sufficiently the principles of measurement and correspondents with the standard EN STN 77 0333.

Diagram of design of equipment of measurement of gas permeability packaging materials by means of isobaric method is presented in the Figure 3 and
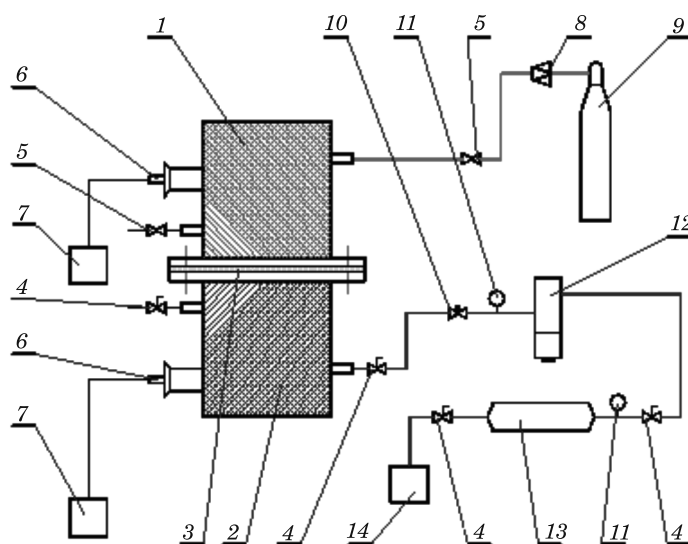

Fig. 3. Design of equipment of measurement of gas permeability: *1* – upper chamber, *2* – bottom chamber, *3* – membrane of measured material, *4* – ball valve, *5* – needle valve, *6* – oxygen probe, *7* – digital oxygen meter, *8* -pressure control valve, *9* – pressure oxygen cylinder, *10* – one way throttle valve, *11* – manometer, *12* – filter regulator, *13* – air tank, *14* – compressor

equipment is presented in the Figure 4. The bottom chamber (2) was firmly attached on the desk and above it upper chamber (1) was attached by means of nuts. Tested material (3) was inserted between two chambers. Two – part test chamber was made of stainless steel and so resistant to corrosion and chemical effects. All parts of the chambers were equipped with the admission valve and the outlet valve (4, 5) and the outlet for the insertion of the oxygen probe (6). Pressure oxygen cylinder (9) was used of supply of pure oxygen (99.5%). The amount of the oxygen was adjusted by means of the pressure control valve in the upper chamber (1). The volume of the each chamber was 1.128 dm^3. Diameter of the effective area between the chambers was 70 mm. Compressor (14) was used to the perfusion of the bottom chamber with the air. Adjusting of the air in this branch was integrated by means of the one way throttle valve and the filter regulator (10, 12). The digital oxygen meters Mesura (7) were applied of measurement of the amount of diffused oxygen.
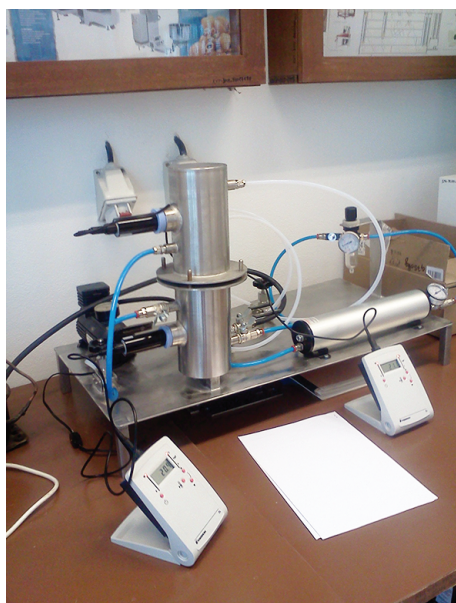

Fig. 4. Equipment of measurement of gas permeability

Basis of the test of the isobaric method is the determination of the concentration of the testing gas, which diffused trough the tested material from the chamber filled with pure testing gas to the chamber with the air. The pressures of the gases on both are equivalent. The samples have to be planar, pure and without mechanical damage. The samples are conditioned during 24 hours in the laboratory conditions. The temperature and the moisture are

continually controlled. The dimensions of volumes V_1, V_2 and S are determined before the test, where V_1 is the volume of the upper chamber of the testing vessel measured in m³ with the precision of ± 5 %, V_2 is the volume of bottom chamber of the testing vessel measured in m³ with the precision of $\pm 5\%$ and S is the testing area of the sample measured in m².

Procedure of the test

The upper chamber was perfused with the testing gas and the bottom chamber with the air before the measurement as long as the homogenous environs was reached in the each of the both chambers. The suitable flow of the both gases is about 600 cm³ · min⁻¹. The time of the perfusion has to be at least 30 minutes and it is prolongs if the material is less permeable. The supply of the testing gas is stopped after the perfusion and all valves of the both chambers are closed. The time of the permeation of the testing gas trough the sample is started in the moment. The diffusion of the gas between both chambers is in the progress for a period of 24 hours.

The coefficient of permeability P is determined on the base of the equation (4) from the equation (BROŽOVÁ 2008):

$$P = \frac{\Delta p_p}{\Delta t} \frac{Vh}{Sp_i} \frac{1}{RT} \qquad (12)$$

where:
p_i – pressure of the gas in the initial volume V, Pa,
V – calibrated volume, m³,
h – thickness of the film, m,
Δp_p– increasing of the pressure of the gas transferred into calibrated volume V, Pa,
Δt – time of the duration of the diffusion, d,
S – area of the film, m²,
R – universal gas constant, $R = 8314$ J · kmol⁻¹ · K⁻¹,
T – temperature, K.

The coefficient of permeability P was obtained from the increasing of the concentration $\Delta \rho_p$ [kg · m⁻³] of the gas permeated trough the film to the calibrated volume V in a time Δt because the oxygen probes measured the relative concentration of the oxygen and the absolute values of concentration had to calculated from the tabulated values of the air and oxygen at the real conditions. After applying equation of state for ideal gas the coefficient of permeability P can obtain from the equation:

$$P = \frac{\Delta\rho_p}{\Delta t} \frac{Vh}{S\rho_i} \frac{1}{RT} \tag{13}$$

where:

ρ_i – density of the gas in the initial volume V, kg · m^{-3},

$\Delta\rho_p$ – increasing of the density of the gas transferred into calibrated volume V, kg · m^{-3}.

We can also obtain permeability of the membrane P_x [m^3 · m^{-2} · s^{-1} · Pa^{-1}] after applying the equations (5) and (13) and equation of state for ideal gas $pV = nRT$, from the equation:

$$P_x = \frac{\Delta\rho_p}{\Delta t} \frac{VM}{S\,\rho_i^2 RT} \tag{14}$$

where:

M – molecular mass, kg · mol^{-1}.

Results and discussion

The permeability of pure oxygen (99.5%) trough polyethylene films of the thickness of 50 µm was measured. Samples of film contained 91% of polyethylene Bralen RA 2 – 63 and 9% colored concentrate Maxithen HP 533041 – violet. The films were made in the company Slovnaft, a.s., Bratislava and the colored concentrate were made in the company Gabriel-Chemie, Lázně Bohdaneč.

The conditions of measurement are described in the Table 1. The measurement was realized at the temperature 22ºC, 30% of moisture of the air and the barometric pressure 102 600 Pa. Measured quantities needed of calculating of the result quantities are presented in the Table 2.The absolute values of densities were calculated from the relative values measured by the oxygen probes. The oxygen probes were calibrated on the value of 20.9% of oxygen in the air on the beginning of the test. The perfusion of the equipment by the air and oxygen during 30 minutes on the beginning of the test caused the increasing of the initial values of the oxygen probes from the 20.9% to the 23% in the bottom chamber and to the 98% in the upper chamber. Value of 98% of the amount of the oxygen in the upper chamber is real, but the value of 23% in the bottom chamber was occasioned by means of sensibility of the oxygen probe on the speed of the air flow.

Table 1

Conditions of measurement

Symbol	Unit	Quantity	Description
V_1	m^3	0.0011283	volume of upper chamber
V_2	m^3	0.0011283	volume of bottom chamber
d_s	m	0.07	diameter of the film sample
S	m^2	0.0038465	area of the film sample
h	μm	50	thickness of the film
b	Pa	102 600	barometric pressure of the air
T	K	295	temperature of the air
φ	%	30	relative moisture of the air
M	kg/kmol	32	molar mass of the O$_2$
R	J/kmol · K	8314	universal gas constant

Table 2

Measured quantities

Symbol	Unit	Quantity	Description
Δt	d	1.0382	time of the duration of the permeation
ρ	kg/m^3	1.1965	density of the air at the temperature 295 K
ρ_{O2}	kg/m^3	1.3386	density of the O$_2$ at the temperature 295 K
ϕ_{1O2i}	% (vol.)	98.0	initial relative concentration of the O$_2$ at the temperature 295 K measured by the oxygen probe in the upper chamber
ρ_{1O2i}	kg/m^3	1.3118	initial density of the O$_2$ at the temperature 295 K measured by the oxygen probe in the upper chamber
ϕ_{2O21}	% (vol.)	23.0	initial relative concentration of the O$_2$ at the temperature 295 K measured by the oxygen probe in the bottom chamber
ρ_{2O21}	kg/m^3	0.2752	initial density of the O$_2$ at the temperature 295 K measured by the oxygen probe in the bottom chamber
φ_{2O22}	% (vol.)	23.7	final relative concentration of the O$_2$ at the temperature 295 K measured by the oxygen probe in the bottom chamber
ρ_{2O22}	kg/m^3	0.2836	final density of the O$_2$ at the temperature 295 K measured by the oxygen probe in the bottom chamber
$\Delta\rho_{O2p}$	kg/m^3	0.0084	increasing of the density of the O$_2$ at the temperature 295 K in the bottom chamber

Dependence of the volume concentration of the oxygen on the time in the upper chamber during the test is presented in the Figure 5. Dependence of the volume concentration of the oxygen on the time in the bottom chamber during the test is presented in the Figure 6. The coefficient of permeability P was calculated by the equation (13).

dependence of volume concentration of O_2 on time of upper chamber – PE violet foil

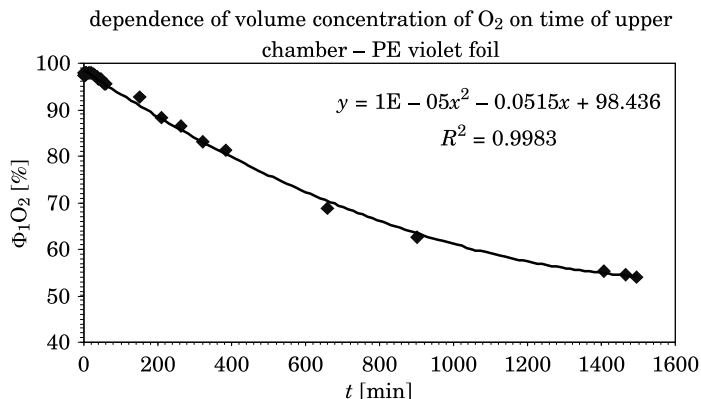$$y = 1E - 05x^2 - 0.0515x + 98.436$$

$$R^2 = 0.9983$$

Fig. 5. Dependence of the volume concentration of the oxygen on the time in the upper chamber during the test

The permeability of the film P_x was calculated from the equation (14). The coefficient of diffusion D of the membrane was determined from the equations (8) and (10).The solubility coefficient S_p of the membrane was determined from the equation (11). The measured values from the Table 1 and Table 2 were used for the calculation.

The time lag Θ was calculated by performing linear regression analysis on only the data points that contribute to the pseudo-steady state. The model implied the regression should be linear and the general form used was $y = b + ax$, where a and b were estimated on the base of the equations (8) as it is presented in the Figure 7.

The tangent in the Figure 7 represents pseudo-steady state and its slope represents the slope of the equation (8). Then the diffusion coefficient D is determined:

$$D = \frac{V\,ha}{S} \tag{15}$$

where:
a – slope.

Results of the transport coefficients of the oxygen trough the PE film are presented in the Table 3. Transport properties are also related to the thickness of the film. The polyethylene Bralen 2 – 63 with 9% colored concentrate Maxithen HP 533041 – violet film showed the high values of the coefficients. The film was high permeable for the oxygen. The film is not very suitable for the food packaging but it is suitable for the mulching of the plants.

Table 3

Transport properties of the polyethylene Bralen 2 – 63 with 9% colored concentrate Maxithen HP 533041 – violet film for the oxygen permeability, thickness of the film 50 μm

Transport properties	Experimental values	Experimental values related to the film thickness	
Coefficient of permeability P [mol $\cdot$ m^{-1} $\cdot$ s^{-1} $\cdot$ Pa^{-1}]	$4.2560 \cdot 10^{-16}$	coefficient of permeability P [mol $\cdot$ m^{-1} $\cdot$ s^{-1} $\cdot$ Pa^{-1}]	$1.8387 \cdot 10^{-15}$
Permeability P_x [cm^3 $\cdot$ m^{-2} $\cdot$ s^{-1} $\cdot$ (0.1 MPa)$^{-1}$]	1794.2500	permeability P_x [cm^3 $\cdot$ m^{-1} $\cdot$ s^{-1} $\cdot$ Pa^{-1}]	0.1038
Coefficient of diffusion [m^2 $\cdot$ s^{-1}]	$4.3999 \cdot 10^{-10}$	coefficient of diffusion [m^3 $\cdot$ s^{-1}]	$2.2000 \cdot 10^{-14}$
Solubility coefficient S_p [mol $\cdot$ m^{-3} $\cdot$ Pa^{-1}]	$9.6735 \cdot 10^{-7}$	solubility coefficient S_p [mol $\cdot$ m^{-2} $\cdot$ Pa^{-1}]	$4.8368 \cdot 10^{-11}$

The developed instrument was not compared with the instrument described in the standard (EN STN 77 0333) because the construction of the instrument is not trivial thing. The developed instrument was only constructed and tested and the obtained data were compared with the data in the literature. BHADHA (1999) presented the values of the permeability P_x in the range from 0.4120 to 0.0375 [cm^3 $\cdot$ m/m^2 $\cdot$ s $\cdot$ Pa] and the values obtained by means of the developed instrument were 0.1038 [cm^3 $\cdot$ m/m^2 $\cdot$ s $\cdot$ Pa]. Advantages of the developed method are the simple detection of the relative oxygen concentration. Disadvantage of the developed method is the necessity to calculate the absolute values of the oxygen concetration from the tabulated values of the air and oxygen at the real conditions. The accuracy of the developed method depends on the accuracy of the measurement of relative concentration of the oxygen by means of instrument Mesura, depends on the accuracy of determination of the density of oxygen at the same condition and depends on the applied theoretical methods for determination of the quantities. The electronic accuracy of the instrument Mesura was 0.1% of oxygen on the scale 100% of oxygen. The accuracy of the determination of the density of the oxygen depends on the accuracy of the measurement of the temperature of the gas and the pressure of the gas. The accuracy of the measurement of the temperature was ±0.5ºC and the accuracy of the measurement of the pressure of the gas was ±50 Pa. Density of oxygen was calculated from the equation of state for ideal gas. The accuracy of the determination of the density was ±0.0029 kg/m^3, i.e. ±0.22% related to the value of the oxygen density 1.3386 kg/m^3 or ±34.5% related to the value of the oxygen density 0.0084 kg/m^3 (increasing of the density of the O$_2$ at the temperature 295 K in the bottom chamber).

The accuracy of the determination of the coefficient of permeability P was ±5.1145.10^{-17} [mol/m $\cdot$ s $\cdot$ Pa], i.e. 12.02%. The accuracy of the determination of

the permeability P_x was ±15.684 [cm^3/m^2 · d · (0.1 MPa)], i.e. 0.8%. The accuracy of the determination of the coefficient of diffusion D was ±16.07 · 10^{-11} [m^2/s], i. e. 36.53% and accuracy of the determination of the solubility coefficient S_p was ±1.1624 · 10^{-7} [mol/m^3 · Pa], i.e. 12.01%.
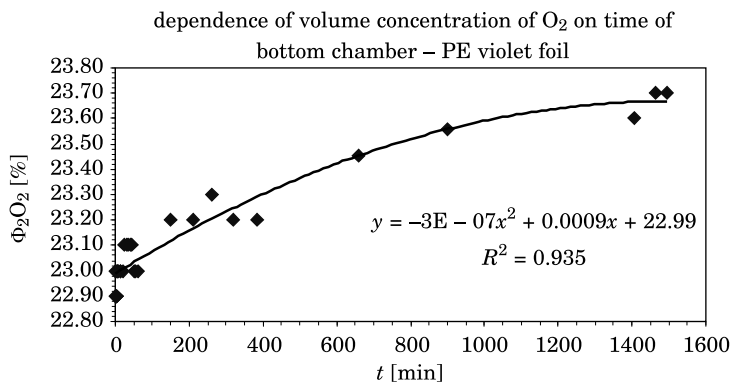

Fig. 6. Dependence of the volume concentration of the oxygen on the time in the upper chamber during the test
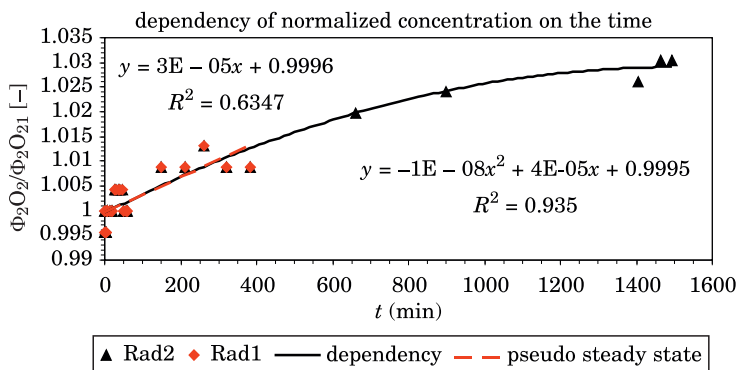

Fig. 7. Dependence of normalized concentration on the time

Conclusion

The design of the equipment of the measurement of the oxygen permeability of the packaging and mulching material was realized and the equipment was constructed. The design and the construction of the equipment was realized with applying of the standard EN STN 77 0333. The measurement of the permeability of the oxygen trough the polyethylene Bralen 2 – 63 with 9% colored concentrate Maxithen HP 533041 – violet film was realized. The

measurements and the calculations of the permeability were modified and personal access was applied. Modified measurement enabled to obtain the experimental values comparable with other authors. The measurements confirmed that the designed equipment is suitable of measurement of the permeability of the oxygen, but the improvement of the measurement of the gas concentration will be needed for the measurement of the absolute concentration of gases. The permeabilities of the oxygen trough the studied films were high. The polyethylene films are suitable for mulching technologies and no suitable for the food packaging.

Translated by Lubomír Kubík

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