Determination of oxygen permeability of polyethylene and polypropylene nonwoven fabric foils

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Abstract

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The paper deals with determination of oxygen permeability of the polyethylene and polypropylene foils applied in mulching and food packaging. The construction of the equipment of measurement of gas permeability through the foils is discussed. The design and construction of the equipment was realized with applying of the Standard STN EN 77 0333. The measurement of permeability of oxygen through the polyethylene Bralen 2-63 with 9% colored concentrate Maxithen HP 533041 – violet foil, Polyten®MLB black&white foil and black polypropylene nonwoven fabric foil were realized by means of the modified method.

Keywords: gas transmisivity; equipment construction; packaging; mulching

The packaging of foods is very important area of food processing. The mulching of plants is interesting in the agriculture engineering. Materials suitable for food packaging and mulching are applied on the basis of polymers. 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 if needed, with multilayer film; it includes different polymers, surface films and metallic foils. Barrier properties, i.e. protection of the package, are related mainly with the ability to transmit the gases and vapours, which are damaging of the quality of the product. Dissipation of the water may cause undesirable drying-out, which shows by the modification of the food structure. Moistening of the food increases the water activity and the state suitable for expansion of microorganisms is inducing.

Božiková (2011) and Božiková and Hlaváč (2011) are interested in thermal characteristic of cheeses, which are very important at food packaging. Degradation processes of foods are also dependent on time and temperature (Pye et al. 1976; Ashley 1985; Jasse et al. 1994). All components of the food have to be taken into account during selection of package materials, as well as the package material, because they can react with one another under the impression of different storage conditions. Oxygen is harmful for foods of vegetal or animal origin. It causes oxidation of higher fatty acids. Internal atmosphere of gases, such as CO_2 and N_2, is modified of the preservation of the quality of the foods (Jasse et al. 1994). The application of polyethylene plastic foils in horticulture is of great importance; Zeman and Kubík (2007) presented the issue of permeability of polymeric packaging materials and showed the theoretical base of permeability.

MATERIAL AND METHODS

Sorption of gases and their transmission through the polymer depends on the permeability and diffusion. The amount of gas Q (mol), which is transferred through the membrane, is defined by the following equation (Jasse et al. 1994):

$$Q = \frac{D \times \sigma(p_1 - p_2) S \times t}{h} \tag{1}$$

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 ($mol/m^3.Pa$)

t – time (s)

S – area (m^2)

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

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

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

$$P = \frac{Q\,h}{S(p_1 - p_2)\,t} = \frac{Q\,h}{S \Delta p t} \tag{3}$$

After applying equation of state for ideal gas $pV = nRT$, we can obtain equation of the coefficient of permeability P (mol/m.s.Pa) suitable for the experimental measurement:

$$P = \frac{Q\,h}{S \Delta p t} \times \frac{p\,V}{n\,R\,T} = \frac{p\,V\,h}{t\,S\,\Delta p\,R\,T} \tag{4}$$

where:

P – permeability (mol/m.s.Pa)

Q – amout of gas (mol)

S – area (m^2)

t – time (s)

p – pressure (Pa)

V – volume of gas (m^3)

R – universal gas constant, R = 8,314 J/kmol.K

T – temperature (K)

n – amount of gas (mol)

We can also obtain permeability of the membrane P_x ($m^3/m^2.s.Pa$) from the equation:

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

Fick's second law of diffusion can be obtained by first calculating a conservation equation with respect to volume (Cussler 2003),

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

where:

C_h – molar concentration (mol/m^3)

z – coordinate (m)

t – time (s)

D – coefficient of diffusion (m^2/s)

S – area (m^2)

By setting S to be constant, Eq. (6) describes unsteady state diffusion, which is Fick's second law of diffusion. Together, Fick's 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

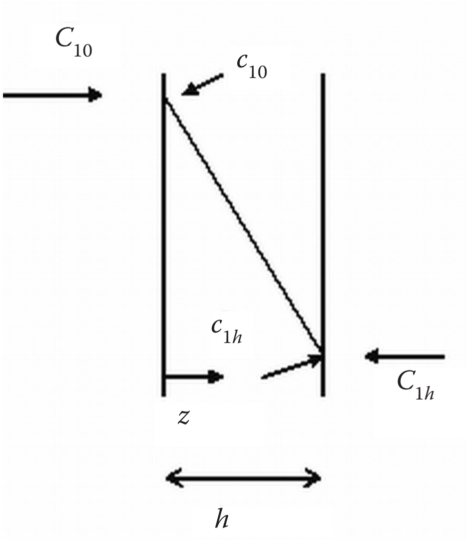

Fig. 1. Steady-state concentration profile in a thin membrane

c_{10} – partion coefficient (mol/m^2); C_{1h} – concentration of downstream solution (mol/m^3); h – thickness of membrane (m); z – coordinate (m) 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 $c_{10} = HC_{10}$.

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 was found that the concentration is negligible. These boundary conditions allow Eq. (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 characteristics of lag time in the diffusion model. After using separation of variables on Fick's second law and imposing the aforementioned conditions to obtain:

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

where:

c_h – concentration on the membrane of thickness h (mol/m^2)

c_{h0} – initial concentration on the membrane of thickness h (mol/m^2)

h – thickness of the membrane (m)

z – coordinate (m)

D – coefficient of diffusion (m^2/s)

n – number of nodes (–)

t – time (s)

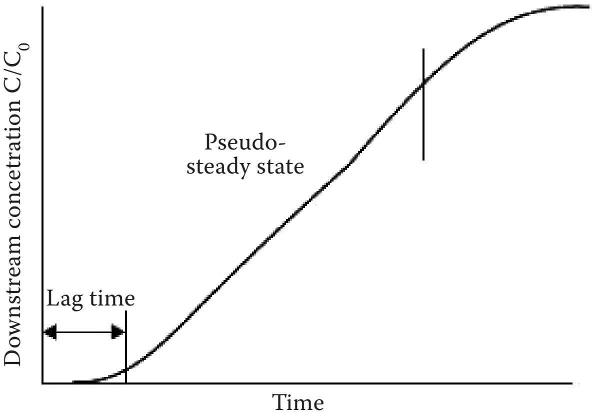

Fig. 2. Typical permeation/lag time curve: normalized downstream concentration versus time in a thin membrane initially free of solute. The code created estimates the pseudo-steady state section (Rutherford, Do 1997)

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

$$\frac{c_h}{c_{h0}} = \frac{S D}{V h}\left(t - \frac{h^2}{6D}\right) \qquad (8)$$

where:

V – ratio of the volume in the air chamber to the area of the fabric

c_h – concentration on the membrane of thickness h (mol/m^2)

S – area (m^2)

D – coefficient of diffusion (m^2/s)

h – thickness of membrane (m)

This limiting equation revealed that there is a lag time for the system to reach the desired pseudo-steady state. Namely, the system cannot be described by an ordinary differential equation when

$$t < \frac{h^2}{6D} \qquad (9)$$

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). Then we can obtain the coefficient of diffusion D (m^2/s) from the Eq. (8):

$$D = \frac{h^2}{6\Theta} \qquad (10)$$

where:

h – thickness of film (m)

Θ – lag time (s)

The solubility coefficient S_p (mol/m^3.Pa) can be calculated using the equation (Pauly 1999):

$$S_p = \frac{P}{D} \qquad (11)$$

where:

P – coefficient of permeability P (mol/m.s.Pa)

D – diffusion coefficient (m^2/s)

The solubility coefficient S_p expresses the solubility of oxygen in the foil. Methods of determination of gas permeability are described in Pye et al. (1976), Koros et al. (1992), Jasse et al. (1994) and Sohail (1997). Polymer's materials present large scale structures and properties which depend on their chemical structure, methods of preparation

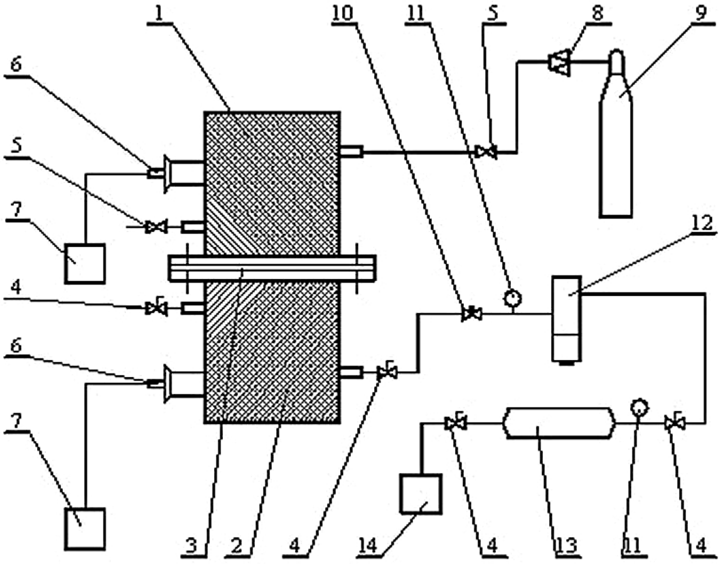

Fig. 3. Design of equipment of measurement of gas permeability
1 – upper chamber, 2 – bottom chamber, 3 – membrane of measured material, 4 – ball valve, 5 – needle valve, 6 – oxygen probe, 7 – digital oxygen meter, 8 –pressure control valve, 9 – pressure oxygen cylinder, 10 – one way throttle valve,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

and conditions of processing. Significant participation of additives and application of polymer mixtures influence barrier properties of films through CO_2, O_2, N_2, or water vapours (Lee 1980).

Design of measurement equipment. Methods of detection and measurement of parameters of packaging materials permeability of gases are specified in the standard STN EN 77 0333 (1987). The method was modified for the conditions of our test. Permeability of the gas was determined 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 was considered universal for the basic measurements. It represents sufficiently the principles of measurement and corresponds with the standard STN EN 77 0333 (1987).

Diagram of design of measurement equipment of gas permeability packaging materials by means of isobaric method is presented in Fig. 3 and equipment is presented in Fig. 4. The bottom chamber was firmly attached on the desk and above it upper chamber was attached by means of nuts. Tested material was inserted between two chambers. Two-part test chamber was made of stainless steel and was thus resistant to corrosion and chemical effects. All parts of the chambers were equipped with the admission valve and the outlet valve and the outlet for the insertion of the oxygen probe. Pressure oxygen cylinder was used as a supply of pure oxygen (99.5%). The amount of oxygen was adjusted by means of the pressure control valve in the upper chamber. The volume of each chamber was 1.128 dm^3. Diameter of the effective area between the chambers was 70 mm. Compressor was used to the perfusion of the bottom chamber with the air. Adjusting the air in this branch was integrated by means of the one way throttle valve and the filter regulator. The digital oxygen meters Mesura (Pierron, Sarreguemines, France) were applied for measurement of the amount of diffused oxygen.

Basis of the isobaric method test is the determination of the concentration of the testing gas, which diffuses through the tested material from the chamber filled with pure testing gas to the chamber with the air. The pressures of the gases at both chambers are equivalent. The samples have to be planar, pure and without mechanical damage. The samples are conditioned during 24 h 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 m^3 with the precision of ± 5%, V_2 is the volume of bottom chamber of the testing vessel measured in m^3 with the precision of ± 5% and S is the testing area of the sample measured in m^2.

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 each of the chambers. The suitable flow of both gases was about 600 cm^3/min. The time of the perfusion had to be at least 30 min and it was prolonged if the material was less permeable. The supply of the testing gas was stopped after the perfusion and all valves of both chambers were closed. The time of the permeation of the testing gas through the sample was

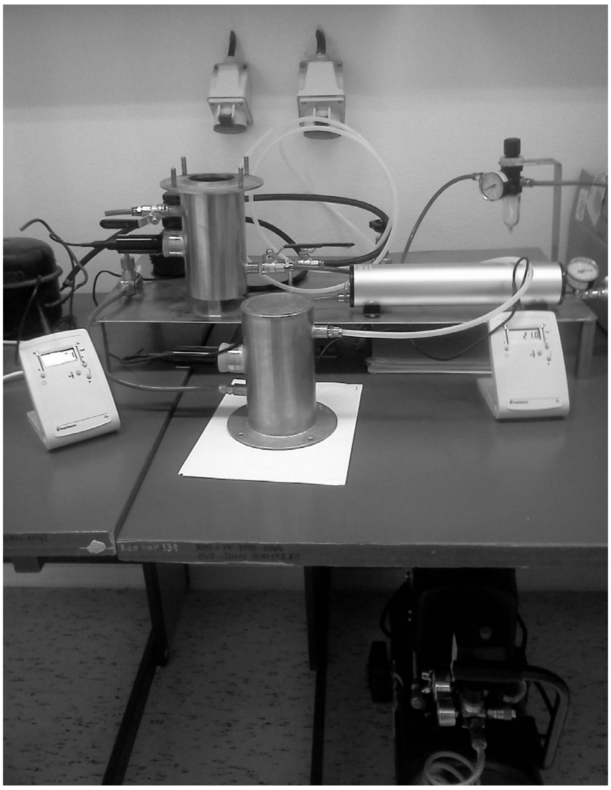

Fig. 4. Equipment of measurement of gas permeability

started in the moment. The diffusion of the gas between both chambers was in the progress for a period of 24 hours.

The coefficient of permeability P is determined on the basis of Eq. (4) from the equation:

$$P = \frac{\Delta p_p}{\Delta t}\frac{V\,h}{S\,p_i}\frac{1}{R\,T} \tag{12}$$

where:

p_i – pressure of the gas in the initial volume (Pa)
V – calibrated volume (m^3)
h – thickness of the foil (m)
Δp_p – increase of the pressure of the gas transferred into calibrated volume V (Pa)
Δt – time of the duration of the diffusion (day)
S – area of the foil (m^2)
R – universal gas constant, R = 8,314 J/kmol.K
T – temperature (K)

The coefficient of permeability P was obtained from the increase of the concentration $\Delta\rho_p$ (m^3/kg) of the gas permeated through the foil to the calibrated volume V in the time Δt because the oxygen probes measured the relative concentration of oxygen and the absolute values of concentration had to be calculated from the tabulated values of the air and oxygen at the real conditions. After applying the equation of state for ideal gas the coefficient of permeability P can be obtained from the equation:

$$P = \frac{\Delta\rho_p}{\Delta t}\frac{V\,h}{S\,\rho_i}\frac{1}{R\,T} \tag{13}$$

where:

ρ_i – density of the gas in the initial volume V (kg/m^3)
$\Delta\rho_p$ – increase of the density of the gas transferred into calibrated volume V (kg/m^3)
V – volume of the vessels (m^3)
h – thickness of the foil (m)
S – area (m^2)
R – universal gas constant 8,314 J/kmol.K
T – temperature (K)

We can also obtain permeability of the membrane P_x (m^3/m^2.s.Pa) after applying Eqs. (5) and (13) and equation of state for ideal gas $pV = nRT$, from the equation:

$$P_x = \frac{\Delta\rho_p\,V\,M}{\Delta\,t\,S\,\rho_i^2\,R\,T} \tag{14}$$

where:

M – molecular mass (kg/mol)
R – universal gas constant 8314 (J/kmol.K)
T – temperature (K)
t – time (s)
ρ_p – density of the gas in the calibrated volume V (kg/m^3)
ρ_i – density of the gas in the initial volume V (kg/m^3)

RESULTS AND DISCUSSION

The permeability of pure oxygen (99.5%) through polyethylene and polypropylene foils was studied.

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 foil sample
S	m^2	0.0038465	area of the foil sample
h	µm	50 and 90	thickness of the foil
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 of (A) the PE Bralen 2-63 with 9% coloured concentrate Maxithen HP 533041 – violet foil (thickness 50 μm), (B) PE Polyten®MLB black&white foil (thickness 90 μm) and (C) PP polypropylene nonwoven fabric foil (thickness 50 μm) at the air temperature 295 K

Symbol	Unit	Description	Quantity A	Quantity B	Quantity C
Δt	day	duration of the permeation	1.0382	1.0000	1.0000
ρ	kg/m^3	air density	1.1965	1.1965	1.1965
ρ_{O_2}	kg/m^3	O_2 density	1.3386	1.3386	1.3386
ϕ_{1O_2i}	% v	O_2 initial relative concentration, UC	98.0	96.4	95.7
ρ_{1O_2i}	kg/m^3	O_2 initial density, UC	1.3118	1.2904	1.2810
ϕ_{2O_21}	% v	O_2 initial relative concentration, BC	23.0	21.9	21.0
ρ_{2O_21}	kg/m^3	O_2 initial density, BC	0.3079	0.2620	0.2811
ϕ_{2O_22}	% v	O_2 final relative concentration, BC	23.7	21.9	21.9
ρ_{2O_22}	kg/m^3	O_2 final density, BC	0.3173	0.2620	0.2932
$\Delta\rho_{O_2p}$	kg/m^3	O_2 density increase in the bottom chamber	0.0094	0.0000	0.0121

UC – measured by the oxygen probe in the upper chamber; BC – measured by the oxygen probe in the bottom chamber

Polyethylene samples of foils which contained 91% polyethylene Bralen RA 63 and 9% coloured concentrate Maxithen HP 533041 – violet, were used. Thickness of the samples was 50 μm. The foils were made in the company Slovnaft, a.s., Bratislava, Slovak Republic and the coloured concentrate was made in the company Gabriel-Chemie, Lázně Bohdaneč, Czech Republic. Then the polyethylene Polyten®MLB black&white foils were studied, which were made in the company Chemosvit Fólie, a.s., Svit, Slovak Republic. The thickness of the foils was 90 μm. Black polypropylene nonwo-

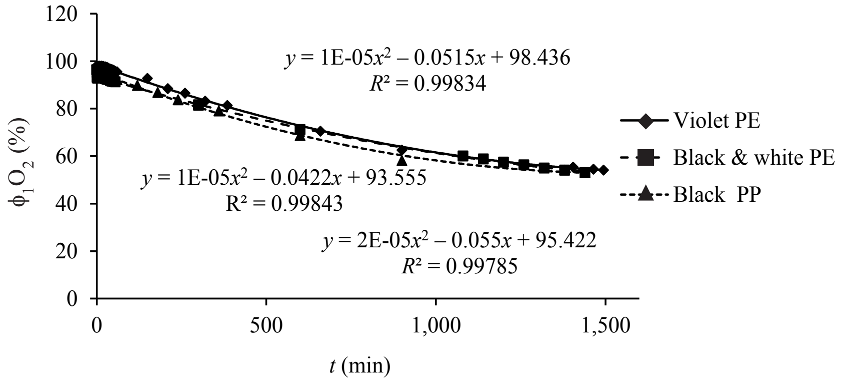

Fig. 5. Dependence of the oxygen volume concentration of on the time in the upper chamber during the test
PE – polyethylene foils, PP – polypropylene foil

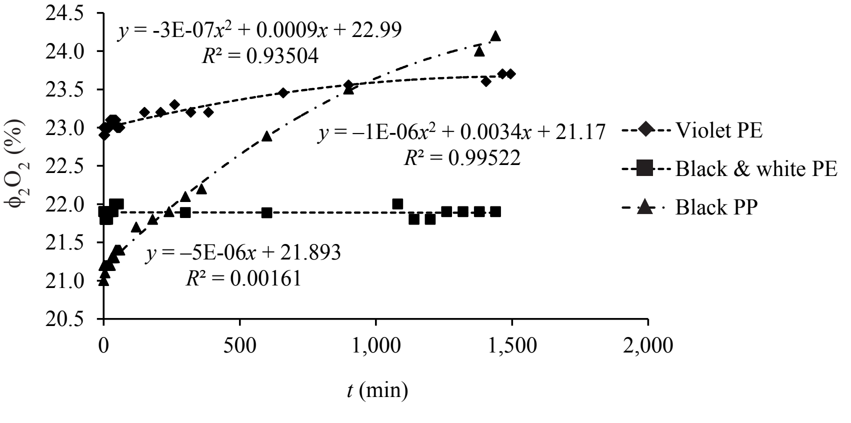

Fig. 6. Dependence of the oxygen volume concentration of on the time in the bottom chamber during the test
PE – polyethylene foils, PP – polypropylene foil

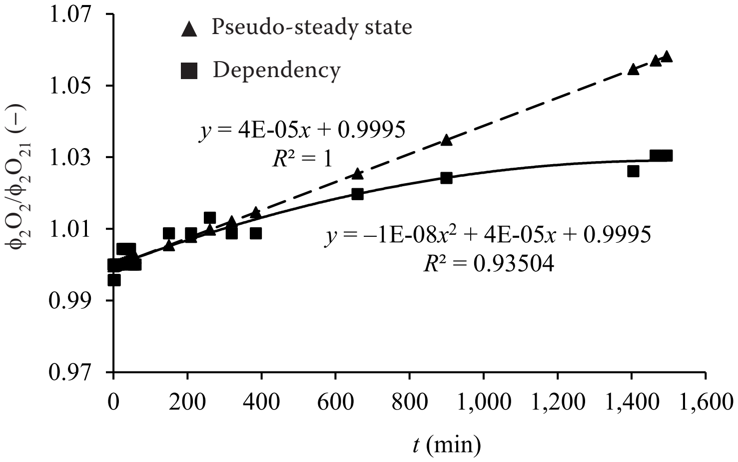

Fig. 7. Dependence of normalized concentration on the time of the PE Bralen 2-63 with 9% colored concentrate Maxithen HP 533041 – violet foil and the tangent of pseudo-steady state in the point $t = 40$ min

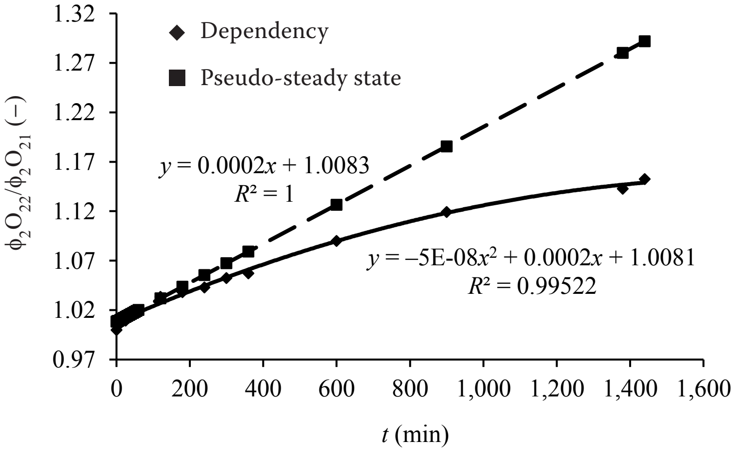

Fig. 8. Dependence of normalized concentration on the time of the polypropylene nonwoven fabric foil and the tangent of pseudo-steady state in the point $t = 60$ min

ven fabric foils were also realized with the thickness of 50 µm. The conditions of measurement are described in the Table 1. The measurements were realized at the temperature 22°C, 30% air moisture and the barometric pressure 102,600 Pa. Measured quantities needed of calculating of the result quantities are presented in Table 2. The basic absolute values of the volume concentration of oxygen at the temperature of 295 K were 1.1965 kg/m^3 of the air and 1.3386 kg/m^3 of oxygen. These values were calculated and extrapoled by means of the equation of state for ideal gas. The absolute values of measured densities were calculated from the base absolute values and the relative values measured by the oxygen probes. The oxygen probes were calibrated on the value of 20.9% of oxygen in the air at the beginning of the test. The perfusion of the equipment by the air and oxygen during 30 minutes at the beginning of the test caused an increase of the initial values of the oxygen probes from 20.9 to 23% in the bottom chamber and to 98% in the upper chamber for the Bralen PE foils, then an increase of the initial values of oxygen probes from 20.9 to 21.9% in the bottom chamber and to 96.4% in the upper chamber for the Polyten®MLB foils, and an increase of the initial values of the oxygen probes from 20.9 to 21.0% in the bottom chamber and to 95.7% in the upper chamber for the polypropylene nonwoven fabric foils. Values of the amount of oxygen in the upper chambers are real, but the values in the bottom chambers were occasioned by means of sensibility of the oxygen probe to speed of the air flow and represent the error of the measurement.

Dependencies of the volume concentration of oxygen on the time in the upper chambers during the test are presented in Fig. 5. Dependencies of the volume concentration of oxygen on the time in the bottom chambers during the test are presented in Fig. 6. The dispersion of the curves in Fig. 6 is caused by the influence on the air perfusion on the beginning of the measurement. The coefficients of permeability P were calculated by the Eq. (13). The permeabilities of the foils P_x were calculated from the Eq. (14). The coefficient of diffusion D of the membrane was determined on the basis of the Eqs (8) and (10). The solubility coefficient S_p of the membrane was determined from the Eq. (11). The measured values from Tables 1and 2 were used for the calculation.

Table 3. Transport properties of the polyethylene Bralen 2-63 with 9% coloured concentrate Maxithen HP 533041 – violet foil for the oxygen permeability (thickness 50 μm) and black polypropylene nonwoven fabric foil (thickness 50 μm)

Transport properties	Bralen 2-63		Black polypropylene	
	experimental values	experimental values related to the foil thickness	experimental values	experimental values related to the foil thickness
Permeability coefficient (mol/m.s.Pa)	4.7771×10^{-16}	2.3889×10^{-20}	5.7812×10^{-16}	2.8906×10^{-20}
Permeability (cm^3/m^2.d.0.1MPa)	2013.6703	0.1165	2822.0832	0.1633
Diffusion coefficient of (m^2/s)	5.8666×10^{-10}	2.9333×10^{-14}	2.9333×10^{-9}	1.4667×10^{-13}
Solubility coefficient (mol/m^3.Pa)	8.1429×10^{-7}	4.0714×10^{-11}	1.9709×10^{-7}	9.7297×10^{-12}

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 basis of the Eq. (8) as it is presented in Fig 2.

The tangents in Figs 7 and 8 represent pseudo – steady state of polyethylene Bralen and polypropylene nonwoven fabric foils and their slopes represent the slope of the Eq. (8). Then the diffusion coefficients D were determined:

$$D = \frac{V \times h \times a}{S} \qquad (15)$$

where:
V – volume (m^3)
h – thickness of the foil (m)
a – slope (1/s)
S – area (m2)

The dependence of Polyten®MLB foil presented in Fig. 6 is linear and horizontal, so the permeability was minimal and the tangent and the diffusion coefficient D were not possible to determine.

The tangents were determined from the analytical equations of the tangent to the curve in the point $[x_T, y_T]$:

$$y = k_t\left(x - x_T\right) + y_T \qquad (16)$$

where:
k_t – $f'(x_T)$
y – dependent variable (–)
x – independent variable (min)
x_T – tangent coordinate (min)
y_T – tangent coordinate (–)

The point of the tangent contact was determined in the steepest area. The coordinate of the point in the curve in Fig. 7 was selected as t = 40 min and in Fig. 8 as t = 60 min. Results of the diffusion coefficients and the transport coefficients of oxygen through the PE and PP foils are presented in the Table 3. Transport properties are also related to the thickness of the foil which was 50 μm for polyethylene Bralen 2-63 and the polypropylene nonwoven fabric foil and 90 μm for Polethylene Polyten®MLB black&white foil. The polyethylene Bralen 2-63 with 9% coloured concentrate Maxithen HP 533041 – violet foil and the polypropylene nonwoven fabric foil showed the high values of the coefficients. The foils were highly permeable for oxygen. Bhadha (1999) presented the values of coefficient of permeability P (mol.m/m.s.Pa) in the range from 0.4120 to 0.0375. The foils are not very suitable for the food packaging but they are suitable for mulching of the plants. Polyethylene Polyten®MLB black&white foil showed the null values of the coefficients of the permeability; it was not permeable. The foil is suitable for food packaging and is usually used for packaging of the milk.

CONCLUSION

The design of the measurement equipment 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 STN EN 77 0333. The measurement of the permeability of oxygen through the polyethylene Bralen 2-63 with 9% coloured concentrate Maxithen HP 533041 – violet foil, polyethylene Polyten®MLB black&white foils and black polypropylene nonwoven fabric foil were 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 for measurement of the permeability of oxygen, but the improvement of the gas concentration measurement will be needed for the measurement of absolute concentration of gases. The permeability of oxygen through the polyethylene Bralen 2-63 and the polypropylene nonwoven fabric foils were high. The permeability of oxygen through the polyethylene Polyten®MLB black&white foils was minimal. The polyethylene Bralen 2-63 and the polypropylene nonwoven fabric foils are suitable for mulching technologies. The polyethylene Polyten®MLB black&white foils is suitable for food packaging.

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