Labthink Instruments CO.,LTD.
144 Wuyingshan Road, Jinan, China
Tel: +86-531-85811021 85864214
Fax: +86-531-85812140
Website: www.labthink.cn
E-mail: trade@labthink.cn

Features of Permeability Test for high barrier Films

Permeability of films can be classified into high barrier, middle and low barrier according to the gas transmission rate of film at 25μm thick. To high barrier film, the gas transmission rate is less than $5cm^3/m^2 \cdot 24h \cdot 0.1MPa$; to barrier film, it is between $5 \sim 200\ cm^3/m^2 \cdot 24h \cdot 0.1MPa$; and to low barrier film it is greater than $200cm^3/m^2 \cdot 24h \cdot 0.1MPa$. In the last few years, low barrier films has become a key developing point to the plastic packaging industry, and they are used widely in various industries such as food, pharmacy, cigarette, chemical, electronics, military and so on.

Since for most raw materials of package, they can be used in packaging industry only after the reprocessing (such as coextrusion process and lamination process). The reprocessing process could cause the alteration to both physical and chemical properties of raw materials, and result in the change in their permeability. It is necessary to test the permeability of the "finished product" to control the quality of package. This is fully proved by tests of various specimens in the Labthink Permeability Laboratory. Among these specimens more than 70% are high barrier films including aluminum foil, aluminum coating film, K-coated film and laminated film containing PVDC, EVOH, etc.

High barrier films have the below features in permeability testing (Take the permeability testing using differential method for an example):

1. Prerequisite

A high barrier specimen has strict requirements on the test condition for permeability test. To obtain scientific and accurate data, you must pay attention to the factors as following:

1.1 Test method

Test results vary with different test methods employment. For the permeability tester using vacuum-differential pressure method, the degree of vacuum in test chamber is the most important parameter. In the procedure V of ASTM D 1434-82 (2003) the degree of vacuum in the lower pressure side(test chamber) should be limited to below 26Pa; For ISO 2556:2001and GB/T 1038-2000, it should be not less than 27 Pa. Since the degree of vacuum is directly interrelated to the state of specimen, whether the specified degree according to standards of vacuum can be reached in the test chamber is a key factor affecting the test results. The precision of the vacuum gauge is also specified too. In ISO 15105-1 the minimum precision is 5Pa; in GB/T 1038-2000 the precision of the vacuum gauge shall be not lower than 6Pa.

1.2 Equipment

Suitable range of the vacuum gauge must be selected. Because the precision of the vacuum gauge is specified in the standards, so this limits the range of the vacuum gauge in fact. To most of the high quality vacuum gauge, its precision is generally equal to 0.1% of its full range. For examplem, if the resolution of the vacuum gauge you choose is 6 Pa, the full scale

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of it will be less than 6kPa. The vacuum gauge whose resolution exceeds 0.1% of its full range is seldom seen in the market. It should be specially indicate that the display resolution and the real resolution of the vacuum gauge are totally different. The display resolution depends on AD transducer and the resolution of the vacuum gauge is the actual limit of detection. For example, if the full range of one vacuum gauge is 6kPa, the instrument employing this degree of vacuum can have a display below 6Pa or even below 1Pa; but the error between the display value and the actual value is an times value which multiple the resolution of vacuum gauge.

1.3 Test temperature

Test gas in the test chamber is as rarefied as near to the Ideal Gas Model and follows the Ideal Gas Equation:

$$pV = nRT$$

Where : p —— gas pressure

V —— gas volume

n —— moles of gas

R —— gas constant

T —— temperature of gas

If the volume of gas in the test chamber is invariable, we can get $\frac{P_1}{T_1} = \frac{P_2}{T_2}$, and this reflects that gas pressure changes with temperature obviously. Fluctuation of temperature could affects the permeability test obviously, According to Arrhenius Law, this could cause the alteration of P, S and D. If the stability of temperature cannot be maintained during the entire test period, the equipment itself must have a function to resist the interference of temperature fluctuation. The best way is to put the test chamber into a heat-insulated cell with a temperature self-control function. Practice has proved that the temperature-controll performance of VAC-V1 gas permeability tester can reduce the requirements of test environment effectively, and is beneficial to resist the interference of temperature fluctuation due to an excellent design in temperature insulation even if the temperature control function is not being used.

2. Features of testing Procedure

There are two obvious features in high barrier testing procedure: comparing with other specimens (barrier films and high barrier films), the pressure in test chamber changes more little and the test duration time is longer. This feature is shown in permeability test using not only differential method but also equal method.

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The features of high barrier films determine the little change values of pressure and the long test time. If the permeability of the tested specimen is low, the quantity of gas permeating through in the whole test process is little in total, so the pressure at the side of low concentration to test gas changes very little. We need quite a long time to collect enough gas pressure for the vacuum gauge detection. The wider the range of a vacuum gauge is, the bigger the value of resolution is, and the longer test time we need. A group of actual test results of high barrier are shown in Table 1. For each film, the maximum pressure change is not bigger than 20Pa.

Table1. Actual data of oxygen permeability

Film	Temperature °C	Relative humidity %RH	Thickness μm	Test time h	End pressure of test Pa	Oxygen permeance $ml/m^2 \cdot 24h \cdot 0.1MPa$
A	24.2	63.8	90	22.0	2.47	0.05
B	24.3	53.6	100	7.5	8.22	1.04
C	24.1	58.6	100	6.4	15.07	2.01
D	21.4	42.3	250	2.1	9.32	3.467
E	23.1	27.1	60	2.7	12.60	5.44

Where : A : PET/AL/PE (AL=0.009mm) B : coextrusion film

C , D : film for milk package E : film from Thailand Package Center

Well, the most important difference between tests of high barrier specimens and other specimens is that when you do permeability test to high barrier film, strict requirements are demanded on test condition and equipment, and needs a long test time. So when you select testing instruments please pay attention to following technical parameters: range and resolution of vacuum gauge, function of temperature compensation, and how to deal with the specimen clamping, sample sealing and so on.