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The Influence of Vacuum Degree on Gas Permeability Test of Differential-pressure Method

Abstract: Shortening the period of vacuum pumping will directly lower the vacuum degree of testing chambers and in turn lower the accuracy of test result.
Keywords: gas permeability , gas transmission rate , gtr , vacuum , vacuum degree

Gas permeability of materials is mainly tested with differential-pressure method or equal-pressure method, of which the former one is the most widely used and the most influential method. Being the earliest test method of gas permeability, vacuum method is improved on the basis of time-lagged method of film test technology. It is applicable in permeability testing of many gases. Moreover, it can test parameters of diffusion coefficient and solubility coefficient. With these advantages, vacuum method has become the most acknowledged one around the world. It is also a practical method in the research of material modification.

1 Vacuum Test Environment

Test procedures of vacuum method are mainly as follows: place specimen, vacuumize, fill test gas, record pressure variation of lower chamber when test gas transmits through specimen, finish test, and calculate test results.

Vacuum pumping is an important procedure of the process. It is the only way to form test environment (one side of specimen being test gas of 0.1Mpa and the other side being vacuum). Vacuum pumping can test sealing effect (if there is leakage on specimen, it is hard to realize ideal vacuum degree even if the vacuum pumping is of long time) of specimen effectively. It can also eliminate the impurity and interference gas absorbed by instrument and specimen. Such factors will impose obvious influences on test data, data repeatability, test time, and data validity. Therefore, the effect of vacuum pumping is of great significance. Proper vacuum pumping can improve both data validity and data repeatability.

2 Vacuum Pumping Period

In the mind of some users, the recommended vacuum pumping period of Labthink VAC-V1 is comparatively long. They think it is easy to reach the required vacuum degree in the standards and the procedure of vacuum pumping can be finished quickly. One thing to be noted here is that VAC-V1 can reach the required vacuum degree in a very short period of 30 minutes. However, to obtain accurate test results, it is not good to begin permeability test immediately after the required value of vacuum degree is reached. This is determined by the special nature of vacuum.

According to specifications of GB/T3163-93, vacuum can be roughly divided as follows:

Low vacuum10^{5}Pa-10^{2}Pa

Medium vacuum10^{2}Pa10^{-1}Pa

High vacuum10^{-1}Pa-10^{-5}Pa

Ultra-high vacuum＜10^{-5}Pa

VAC-V1 works in higher 'medium vacuum' region. If 'low vacuum' or lower 'medium vacuum' is chosen for the test, vacuum pumping period can be greatly shortened. Different 'vacuum degree' requires different vacuum pumping period. Shortening vacuum pumping period will lower vacuum degree of testing chambers and in turn lower testing precision. Therefore it is rather one-sided to emphasize vacuum pumping period while omit vacuum degree.

The required vacuum degree of testing chamber in ASTM D 1434-82（2003）is less than 26Pa. While in ISO 2556:2001 and GB/T 1038-2000, it is required to be less than 27Pa. The lower testing chamber must reach required vacuum degree before the test gas is filled into upper testing chamber. Otherwise, test results will have no significance. According to standard GB/T 1038-2000, when the required vacuum degree is reached, vacuum pumping should continue on for more than three hours. It can be seen that vacuum pumping period should be longer than three hours.

3 The Influence of Vacuum Pumping Period on Test Data

The longer the vacuum pumping period is, the lower the vacuum degree of lower chamber and the more accurate and stable the test data will be. To confirm the influence of vacuum pumping period on test data, the author carried on permeability test to several specimens with different vacuum pumping period. Here we only take some of 25μm PET film data as an example for explanation.

Vacuum pumping periods are set into 24h, 18h, 2h, 1h and 0.5h respectively. Under each test environment, the test is carried out for three times at least. The statistic data are listed in Table 1.

Table 1. Test Data of PET Oxygen Permeability in Different Vacuum Pumping Period

Vacuum Pumping（h）	Test Temperature（℃）	Test Humidity <>（%RH）	Test Time（min）	Oxygen Permeance 1	S	CV
24	22	25.3	112	41.063	0.313	0.762
18	22	24.4	102	41.085	0.874	2.13
2	23	27.8	77	45.081	1.35	2.99
1	23.3	29.2	53	45.629	<2>0.804	1.76
0.5	23.4	29.9	52	45.645	1.29	2.83

Note: the unit of oxygen permeance is $cm^3/m^2 \cdot 24h \cdot 0.1Mpa$.

It can be seen from table 1 that as the period of vacuum pumping is shortening, test time presents a tendency of shortening while oxygen permeance presents an obvious increase, which is identical with the variation tendency of theoretical analysis. When vacuum period is shorter than four hours, oxygen permeance increases obviously. Data stability is not good.

4 Conclusion

Although vacuum pumping period is only one of the parameters of vacuum method gas permeability instruments, it should be specially noted by the users; otherwise, not only accuracy of the test data is lowered, mistaken test data will sometimes appear as well. For the application of VAC-V1,

Labthink has provided empirical data list of vacuum pumping period for the user references. It is comparatively a reasonable way to decide the period of vacuum pumping according to empirical operations.