

Present Situation and Prospects of Gravimetric Method Water Vapor Permeability Testing

Abstract: test accuracy and test repeatability of water vapor permeability testing using gravimetric method are controversial at present. The expansibility of gravimetric method and whether gravimetric method will be replaced by new methods become the focus of concern. This article presents a systematic analysis to test principle, present situation and prospects of gravimetric method.

Key Words: GB 1037, water vapor permeability, gravimetric method, cup method

Oxygen and water vapor have obvious influence on active substance of food, pharmaceuticals and some cosmetics, which makes chemical property of these products unstable. Therefore, commercial packaging usually raises demand on barrier property of specimen materials. At present, the fundamental method of water vapor permeability testing-gravimetric method is provoking criticism in terms of its test efficiency, test repeatability and etc. The expansibility of gravimetric method and whether gravimetric method will be replaced become the focus of concern. This article presents a systematic analysis to test principle, present situation and prospect of gravimetric method.

1. Gravimetric Method

1.1 Principle of Gravimetric Method

In gravimetric method, the two sides of specimen maintain certain pressure difference at specified temperature. Then the weight variation of permeable cup is measured using gravimetric balance or analyzing balance. Based on the size, thickness, weighing interval, and humidity difference on two sides of specimen, water vapor permeability parameters can be calculated. Since the characteristic testing component in gravimetric method is permeable cup, this method is also called cup method. Although certain relative humidity difference is specified in gravimetric method as its testing condition, it is not pointed out which side should be the high humidity side. Therefore, whether the inner permeable cup to be the high humidity side and outer cup to be the dry side (water method) or vice versa(desiccant method) conforms to test principle of gravimetric method.

1.2 Data Comparison Between Desiccant Method and Water Method

Whether test data obtained by two methods based on the same test principle, i.e., desiccant method and water method, are consistent becomes the concern of the whole testing industry. According to test process and calculate formula provided in ASTM E96, test data of the two methods should be consistent under perfect condition. However, such deduction is less persuasive than actual comparison. In 2007, proficiency testing to 'barrier property testing for plastic package material-determination of oxygen and water vapor transmission rate' is held in our country nationwide, which is sponsored by CNCA and organized by National Packaging Product Inspection and Testing Center (Ji Nan). There are altogether 67 labs (all the domestic authoritative testing institutions participated) participating the testing of water vapor transmission rate (66 labs provided effective test data and 10 labs did not adopt gravimetric method). This proficiency testing is unprecedented in the world in terms of its scale, testing level of the members and authority of its organizers. Based on the results of data comparison, field test data of desiccant method and water method have no obvious difference.

2. Advantages of Equal-pressure Method

2. Present Situation of Gravimetric Method

2.1 Present Situation

It is undeniable that the formulation and the implementation of GB/T 1037 play a rather important role in the wide application of gravimetric method. At the same time, the salient features of gravimetric method in terms of its easy operation and lower price of test instruments also accelerate the popularization of instruments using this method. During its application in the past twenty years, the negative evaluation of gravimetric method is occurred now and then. Moreover, with the introducing of new testing methods and the intensifying need to replace traditional instruments, such negative evaluation becomes more prominent. The negative side mainly exists in aspects of lower test efficiency, accuracy and repeatability. Thus the newly rising sensor method instruments become more favorable, which is thought to be more suitable to the booming development of present flexible packaging industry.

2.2 Analyzing Present Application Situations

Is gravimetric method is really impractical as is reported? When analyzing the reasons, it is not difficult to discover that the so said defects of gravimetric method mainly result from the using of desiccant method instruments. In fact, there are indeed weak points in desiccant method. However, these problems are mainly result from the difference between testing environment and weighing environment and the difficulty to maintain a stable pressure difference of water vapor on two sides of specimen in a long term (test environment is not stable). This is especially obvious for specimens with bigger hygroscopic capacity. Although consolidating testing environment can solve errors caused by environmental change, concentration difference of water vapor on two sides of specimen is maintained totally relying on manual operation, which makes automatic testing and the consolidation of test environment difficult to realize. In addition, although it is specified in GB/T 1037 that hygroscopic moisture content of desiccant should not exceed 10%(there is similar specification for hygroscopic capacity of other desiccant), there is no data proofing whether hygroscopic capacity of desiccant changes obviously when its hygroscopic moisture content approaches the upper limit.

However, water method of gravimetric method does not have the above defects and has realized automatic testing at present. Then why the improvement of water method could not attract enough attention? The author thinks that this is related with the misunderstanding of GB/T 1037 among some insiders. ASTM E96 is the most widespread and historic water vapor permeability-testing standard. It is also the most excellent standard in terms of its completeness of describing test methods. This standard simultaneously introduced these two test methods: desiccant method and water method. Both of these two methods are considered as fundamental test methods, which should be selected according to application environment of materials in actual test. Other water vapor permeability testing standards, such as GB/T 1037, do not have such complete description of test methods. The first edition GB 1037-70 'determination of water vapor transmission rate for plastics' simultaneously includes gravimetric method and water method. However, the revised edition GB 1037-88 'Determination of Water Vapor Transmission Rate for Plastic Films and Sheets-Cup method' simplifies its description of water method and only lists the method of putting desiccant into permeable cup when explaining how to realize relative humidity, which makes some users think that it is not allowable to use distilled water or other reagents. However, the stressing of GB 1037-88 on 'cup method' in the standard title indicates that its content does not confine to 'desiccant method' only. It is because the above-mentioned misunderstanding that results in the cold reception of water method, which actually conforms to GB/T 1037. In the same time, various investigation rules including present market access QS certification requires to implement GB/T 1037 gravimetric method, not confining to desiccant method. In addition, the impact of sensor method makes some people violate justice in evaluating desiccant method. Although some people speak highly of sensor method, they do not have any actual application experience. Test

parameters and the reasonability of precision will not be discussed in this article for the present. The limitation of desiccant method in terms of specimen thickness and specimen sealing also exist in those traditional sensor methods. Moreover, the sealing problem is still a key factor restraining lower test limit of testing instrument. Therefore, in terms of test principle, the lower test limit of several water vapor testing methods should be consistent.

3. Prospects of Gravimetric Method

In barrier property proficiency testing in 2007, 85% of the participators use gravimetric method instruments. And system error of all these laboratories is within the allowable range of our country. Statistical data from this proficiency testing have confirmed the fundamental position of gravimetric method, which simultaneously refuted those reports about lower accuracy of gravimetric method .

However, we must admit that the improvement space of desiccant method is becoming more and more restricted. With the accelerating of automatic testing process, its development is obviously lagging behind other test methods. The pleasant thing is that problems encountered by desiccant method during the course of its development can be well solved by water method. Liquids such as distilled water contained in permeable cups of water method can maintain long-term water vapor concentration stably without frequent human interference. Now the highly efficient and convenient automatic testing instrument using water method has been introduced to the market. Actual application has proved that desiccant method testing needs three days while full automatic water method instruments only needs several hours to complete the testing. Test efficiency of water method instruments is no less inferior to that of sensor method instrument. Moreover, full automatic instruments possess advantages in terms of test accuracy and repeatability.

To develop water method, the content of GB/T 1037 should be further improved to eliminate misunderstandings of GB/T 1037 in the society, avoiding the ideas that 'water method does not belong to gravimetric method'. At present, to update their testing instruments, several authoritative testing institutions in our country already began to purchase full automatic instruments using water method and stopped purchasing desiccant method instruments, which indicates that they believe water method conforms to GB/T 1037. Although electrolytic sensor method water vapor permeability testing standard was approved in 2007, but the application of this new method would not shake the position of gravimetric method, just as the issuing of GB/T 19789 did not shake the authoritative position of differential-pressure method(in the 2007 proficiency testing of barrier property, 74% participators use differential pressure method instruments) in gas permeability testing during the past two years. Therefore, those who want to completely develop water vapor permeability testing must start with gravimetric method, in which popularizing and improving water method is the best way.