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On Water Vapor Permeability Test Methods for Plastic Films (package)
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Abstract: water vapor permeability is of vital importance to food and medicals. Based on his work experience, the author analyzes influencing factors of water vapor permeability testing methods for plastic films (package). Meanwhile, some suggestions for improvement are put forward.

Key Words: water vapor permeability, water vapor transmission rate, cup method, sensor method, desiccant method and water method

1.Overview:

Packaging is a key application field of plastic films (package). The main function of packaging materials is to protect the quality of packed products. Some products such as food, pharmaceuticals and some cosmetics are unstable in chemical properties because they contain some active substances, for which special demand on barrier property of packaging materials are raised. What affects those active substances most are oxygen and water vapor. Therefore, current plastic packaging enterprises make it an important research subject to improve barrier property of films. They are also constantly searching for new test methods to verify the validity of their research. Today, as all edible plastic package manufacturers are required to pass QS authentication, summing up and analyzing present test methods while searching for more scientific and reasonable test methods can provide practicable support to QS authentication.

2.Water Vapor Barrier Property Testing Methods for Film

2.1Cup Method (gravimetric method)

Test principle of Cup Method (gravimetric method) is as follows: under specified temperature and humidity, a certain water vapor pressure difference is maintained on two sides of specimen. Water vapor transmitting through the specimen is then measured for users to calculate water vapor transmission rate and water vapor permeability coefficient. Cup method (weighing method) can be further divided into the desiccant method and the water method. However, the only national *standard* GB1037-1987 being appointed for determination of water vapor transmission rate for plastic film and sheets, adopts desiccant method. First, add specified desiccant (particle size and dry processing) into permeable cup, the top of which is sealed with film specimen using sealing wax. The cup is then placed in standard environment of 38 ° C, 90% RH or 23 ° C, 90% RH. Weigh the cup repeatedly until the water absorption becomes stable. In the mathematical model of desiccant method, relative humidity (R.H.) of inner cups (of between desiccant and film specimen) is generally considered as 0 and external relative humidity is 90%. Specimen in this method bears an outside-inside water vapor pressure difference of 90%RH. In water method, permeable cups contain distilled water or saturated saline solution. For distilled water, inner cups is considered as 100%R.H. Test environment is 38℃、10%R.H. Specimen in this method also bears an outside-inside water vapor pressure difference of 90%RH. Although test data of desiccant method and water method measure in accordance with test procedures and calculation formula of ASTM E96 should be identical in ideal situation, water method has not been adopted by domestic standards and even current QS identification investigation rules for food package yet. The commonly used method in China at present is still desiccant method.

2.2 Sensor Method

Sensor method directly measures humidity of dry chamber using humidity sensor. Generally, relative

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humidity inside wet chamber is maintained using certain amount of distilled water or saturated saline solution, or saturated sponge sometimes. What is similar to water method is that distilled water or solution is not allowed to contact specimen. At present, there is no relevant sensor method standard in our country. Sensor method includes electrolytic analysis method and infrared analysis method. Corresponding international standards are ASTM F1249-01、ASTM E 398-03 and ASTM F372-99, which are more suitable for medical films and sheets with smaller barrier property.

3. Limitations of Test Methods

3.1 Limitation of specimen thickness: relevant documents show that both cup method and sensor method are not suitable for excessively thick specimen. As to cup method, specimen with excessive thickness may cause great error resulting from sealing failure.

3.2Environmental variation causes error: in traditional cup method, permeable cups are repeatedly moved between test environment and weighing environment, for which the test is unable to be carried out in a stable condition. Taking condition A in GB 1037 as an example, under the temperature of 38℃ and relative humidity of 90%, theoretical value of outside-inner permeable pressure on specimen is 90%. If weighing environment is 27℃、60%R.H., the transmission pressure difference should be 60%R.H., which will destroy the original transmission equilibrium and diffusion equilibrium of specimen. Meanwhile, because of the passing in and out of specimen, the temperature and humidity of constant temperature and humidity container itself requires a period of time to reestablish to its preset condition. Therefore, accuracy of test result is affected.

3.3 It is difficult to maintain the stability water vapor pressure difference a long-term, especially for specimen with big hygroscopic capacity. There are two reasons for this: the first one is personal operating habits, for example, not quick enough in weighing and vibrating desiccant. Second, although it is specified in GB 1037 that hygroscopic capacity of desiccant should not exceed 10%, it has not been verified whether the hygroscopic capacity is lowered when it reaches 7～9%. Either of the two factors may cause the actual pressure difference deviate a lot from 90%, affecting accuracy of test data.

3.4 Poor sealability of Permeable cups: the composition and quality of sealing wax have great impact on test results. Meanwhile, during specimen sealing, the high-temperature treated desiccant with great hygroscopic capacity directly exposed in air for certain time. If wax sealing cannot be completed in a very short instant, effective hygroscopic capacity of desiccant would be lowered. In addition, for thicker specimen, improper edge treatment of specimen is another source of error for specimen.

3.5Poor Repeatability of test result: different positions (printed pictures and thickness of film) of sampling and the uniformity of temperature and humidity of constant temperature and humidity chamber are important factors affecting test results.

3.6 Longer test period. Based on common test experience, if water vapor transmission is measured with gravimetric method , test period for specimens with WVT not exceeding2g/24h ·m^2 ,such as aluminum coated film and three layer barrier film, is generally between 7 to ten days. Moreover, data repeatability of individual specimen is lower than that of single-layer film. Even to common single-layer film such as PE and BOPP, test period is usually longer than three days. Since Laminated film has become the mainstream in present packaging, especially in a time when all edible plastic package manufacturers are required to pass QS authentication, longer test period cannot satisfy the requirements of product manufacturers and test institutions in terms of rapidness, accuracy and high efficiency, making it imperative to improve test methods and increase test efficiency.

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4. Suggestion for Improvement

Replace desiccant method with water method. In desiccant method, in order to maintain the **hygroscopic capacity** of desiccant, users must vibrate desiccant at set intervals during test, making it difficult to complete desiccant method automatically. In contrast, water method not only can realize full automatic testing, but also avoid many human interfering factors. At the same time, water method uses distilled water or saturated saline solution to supply stable temperature and the other side of specimen keeps dry through external means. Test environment in water method is stable and reliable. A stable pressure difference can be maintained in water vapor on two sides of specimen in a longer period, thus avoiding the error in desiccant method caused by environmental variation and human inference. For the above-mentioned reasons, water method is a better way to test water vapor barrier property of packaging materials. Recently, the WVT data comparison of some authoritative institutions shows that test results of desiccant method and water method are very close. Moreover, since there are various influencing factors existing in desiccant method, it needs further verification as to which method's result is closer to the real value. Recently, CNSA organizes another data comparison for barrier property of materials, registering the great importance attached to barrier property of materials.

At present, automatic water method testers have been developed by some tester manufactures, for example, Labthink has developed the 12-chamber automatic water vapor permeability tester TSY-T3, which not only efficiently solves the problem of human interference in desiccant method, but also fills up the deficiency of traditional cup method in terms of lower efficiency.

Now, the only domestic test standard for water vapor permeability in China is GB1037. Comparing with international ISO and ASTM standards, the methods involved in domestic standard is rather rare. New standards are in urgent need to satisfy the rapid market development and accurate test requirement. Only in this way can test methods be more practical and accurate to reflect product quality in a timely, accurate and highly efficient way.

Recently, national Department of Quality Supervision and National Committee for Standardization have entrusted China packaging product Quality Supervision & Testing Center (Jinan), China packaging development and testing center, and Jinan Labthink Mechanical and Electronic Center to formulate the standard named *standard test method for the determination of water vapor transmission rate for plastic films and sheets-electrolytic sensor method*. This standard will be formulated in accordance with ISO15106-3：2003, while taking domestic situation into account. The basic principle is as below: place specimen to the permeable chamber so that permeable chamber is divided into dry chamber (lower concentration of water vapor) and wet chamber (high concentration of water vapor). Dry carrier gas carrying water vapor transmitted from wet chamber flows through dry chamber into electrolytic tank, where the water vapor is decomposed into oxygen and hydrogen. Water vapor transmission rate is calculated with the recorded value of Faradaic current.

Electrolytic method can not only accurately measure water vapor transmission rate, but also greatly improve test efficiency and test level. This method can fill the needs of automatic production line. Let us wait expectantly for the introduction of this standard.