A Study on Application of Infrared Sensor Method in Moisture Permeability Test of Food Packaging Materials

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Abstract: Objective This paper is based on the technical improvement of the infrared sensor devices and has combined with the moisture permeability experiments of various food packaging materials, aiming to explore the application performance and effect of infrared sensor method equipment in the moisture permeability test of food packaging materials. Methods The improvement of the temperature control capacity of the equipment together with the sample clamping device helped to ensure a stable temperature during the test. In addition to the test of film/sheet, vessel type samples, the equipment subject to the referee-cup method principle and the equipment of the infrared sensor principle in this study were utilized to test the leather, PET film, sheet, PP film, PET/Al/PA/CPP film and VMPET film with varying moisture permeability levels respectively for their water vapor permeability rates at two different test conditions: 23°C, 90%RH and 38°C, 90%RH. Results The improved infrared sensor method equipment and cup method equipment share a similar relative uncertainty in the test. The water vapor permeability deviations between materials with distinct levels of moisture permeability are within the allowed range after tests with two methods and at different test conditions. Moreover, the homogeneity of test results of each food packaging material is satisfactory. Also, compared with the previous infrared sensor method equipment, the improved equipment witnessed an increase in both uncertainty and precision. Conclusion Therefore, the improved infrared sensor method test equipment can test the water vapor permeability of food packaging materials with different performances in a more accurate and reliable way.

1. Introduction
The moisture permeability of food packaging materials is presented with the water vapor permeability. The property and purpose of contents pose different requirements of the moisture permeability of materials. For example, some food is quite sensitive to vapor, requiring the water vapor permeability of its package to be $10^{-6}$g/(m²·day). As a result, the moisture permeability test of packaging materials is of vital importance. Currently, the main test methods for water vapor permeability include the cup method, electrolytic sensor method, humidity sensor method and infrared sensor method. Compared with the other three methods, the infrared sensor method features a wide variety of applicable food packaging materials, large scope of application, non-consuming property, long service life, strong...
anti-interference performance and good stability. [1] With reference to the referee-cup method test, this verification has optimized and improved the previous infrared sensor method equipment and the application performance of the equipment has also been studied through comparison.

2. Equipment improvement
The infrared sensor method equipment mainly consists of the infiltration cell and the sensor. The existing test equipment manufactured by this principle is mostly subject to the water bath temperature control device to control the temperature of the infiltration cell. However, the disadvantages include a low temperature control precision, long time consumer, poor stability and high energy consumption. In addition, the type of samples that can be tested by such equipment is quite simple, limited to film/sheet or vessel materials only. Thus, in this study, targeted efforts were made to improve the temperature control device and sample clamping device of the equipment.

2.1. Temperature control device
In the conventional water bath temperature control process, a pump is used to feed the constant temperature liquid from the external constant temperature tank equipped to the infiltration cell of the equipment. Nevertheless, heat loss always occurs during liquid flow, affecting the temperature control precision and accuracy. It applies to the temperature control service when the temperature is beyond the room temperature only. This improvement includes the configuration of a heating element, refrigerating element, temperature control element, temperature sensing element and other temperature control devices inside the infrared sensor method equipment, so as to regulate and control the temperature of the infiltration cell. The temperature control element controls the heating and refrigerating element for heating or refrigerating according to the difference between the set temperature value and the test value. Then, P.I.D technology is applied to regulate the entire temperature control process, ensuring that the temperature inside the test cavity quickly and stably reaches the set value. The temperature control range can be lower than the room temperature. The improved equipment has a temperature control precision of ±0.1℃, satisfying the requirement that the temperature of the test cavity must be controlled within ±0.5℃ in GB/T26253, GB/T31355.

2.2. Sample clamping device
Vessel type packaging materials are characterized by varying forms and rigid structures. The sheet samples are difficult to obtain. However, if the plane clamping structure same to the film/sheet materials is used, bubbles or pores can appear at the fitting face with the plane structure and the vapor at the high humidity side is vulnerable to leakage from the surrounding areas of the sample rather than permeation to the low humidity side through the sample, leading to a large test result. In this study, an independent support structure is applied as the vessel sample clamping device. The air opening on the equipment and the air hole beneath the support bracket is specially designed for seamless jointing, to ensure the airtightness between the vessel and the test cavity. [2] During the test of films or sheets, the test sample is fixed on the plane clamping structure as shown in Figure 1; during the test of vessels, the air opening above the bracket is inserted inside the vessel and the lower air opening is connected to the air hole. Then, dry nitrogen through the air inlet can be permeated to the vessel and form vapor, which will be delivered to the sensor through the air outlet as shown in Figure 2. Under such circumstances, the same equipment can be used to test the film/sheet and vessel type packaging materials.
3. Test method

The cup method is considered the referee method for the moisture permeability test. So, the cup method and infrared sensor method are adopted respectively to test 6 film/sheet materials, namely, leather, PET film, sheet, PP film, PET/Al/PA/CPP film and VMPET film with different moisture permeability performances for their water vapor permeability at two sets of test conditions: 23℃, 90%RH and 38℃, 90%RH pursuant to GB1037, GB/T26253. The uncertainty, test results and deviation of the two types of equipment with distinct principles are analyzed to verify the reliability and stability of the infrared sensor method test equipment application. As the cup method cannot be used to test vessel samples, this verification does not involve the comparison with vessel tests. Equipment used in the test is the W3/031 water vapor permeability tester and the W3/230 water vapor permeability testing system.

4. Result and discussion

The test results of the 6 film/sheet test samples are shown in Table 1-6 respectively.

| Test condition | Cup method | Infrared sensor method |
|----------------|------------|------------------------|
|                | Test result | Test result            |
|                |             | Relative               |
|                |             | Relative standard      |

Table 1 Test Value of Water Vapor Permeability of Leather
| Test condition   | Cup method [g/(m²·24h)] | Infrared sensor method [g/(m²·24h)] | deviation (%) | relative standard deviation (%) |
|------------------|--------------------------|--------------------------------------|---------------|---------------------------------|
| 23°C, 90%RH      | 9.862                    | 9.758                                | -3.3          | 2.1                             |
|                  |                          |                                      | 1.2           |                                 |
|                  |                          |                                      | 2.5           |                                 |
|                  |                          |                                      | 1.1           |                                 |
|                  |                          |                                      | -1.7          |                                 |
|                  |                          |                                      | 0.2           |                                 |
| 38°C, 90%RH      | 29.049                   | 29.804                               | -2.6          | 3.2                             |
|                  |                          |                                      | -0.6          |                                 |
|                  |                          |                                      | 1.2           |                                 |
|                  |                          |                                      | -4.0          |                                 |
|                  |                          |                                      | 5.3           |                                 |
|                  |                          |                                      | 0.7           |                                 |

Table 2 Test Value of Water Vapor Permeability of PET Film

| Test condition   | Cup method [g/(m²·24h)] | Infrared sensor method [g/(m²·24h)] | Relative deviation (%) | Relative standard deviation (%) |
|------------------|--------------------------|--------------------------------------|-------------------------|---------------------------------|
| 23°C, 90%RH      | 7.806                    | 8.020                                | 3.9                     | 4.9                             |
|                  |                          |                                      | 4.8                     |                                 |
|                  |                          |                                      | -8.7                    |                                 |
|                  |                          |                                      | -0.2                    |                                 |
|                  |                          |                                      | 1.5                     |                                 |
|                  |                          |                                      | -1.3                    |                                 |
| 38°C, 90%RH      | 16.191                   | 15.548                               | -2.6                    | 4.3                             |
|                  |                          |                                      | -3.7                    |                                 |
|                  |                          |                                      | 8.1                     |                                 |
|                  |                          |                                      | 0.8                     |                                 |
|                  |                          |                                      | -2.3                    |                                 |
|                  |                          |                                      | -0.3                    |                                 |

Table 3 Test Value of Water Vapor Permeability of Sheet

| Test condition   | Cup method [g/(m²·24h)] | Infrared sensor method [g/(m²·24h)] | Relative deviation (%) | Relative standard deviation (%) |
|------------------|--------------------------|--------------------------------------|-------------------------|---------------------------------|
| 23°C, 90%RH      | 1.171                    | 1.208                                | -3.5                    | 4.7                             |
|                  |                          |                                      | 2.2                     |                                 |
|                  |                          |                                      | 4.6                     |                                 |
|                  |                          |                                      | -4.1                    |                                 |
|                  |                          |                                      | 5.7                     |                                 |
|                  |                          |                                      | -4.4                    |                                 |
| 38°C, 90%RH      | 1.282                    | 1.263                                | 2.9                     | 3.6                             |
|                  |                          |                                      | -3.1                    |                                 |
|                  |                          |                                      | -4.5                    |                                 |
|                  |                          |                                      | -2.6                    |                                 |
|                  |                          |                                      | 1.1                     |                                 |
|                  |                          |                                      | 3.7                     |                                 |
### Table 4 Test Value of Water Vapor Permeability of PP Leather

| Test condition | Cup method | Infrared sensor method |
|----------------|------------|------------------------|
|                | Test result [g/(m²·24h)] | Test result [g/(m²·24h)] | Relative deviation (%) | Relative standard deviation (%) |
| 23℃, 90%RH    | 1.940      | 1.986                  | 7.9                    | 5.1                          |
|                |            |                        | 3.8                    |                              |
|                |            |                        | -5.3                   |                              |
|                |            |                        | -4.1                   |                              |
|                |            |                        | -2.5                   |                              |
|                |            |                        | 0.2                    |                              |
| 38℃, 90%RH    | 5.318      | 5.727                  | -7.1                   | 4.8                          |
|                |            |                        | -2.1                   |                              |
|                |            |                        | -0.2                   |                              |
|                |            |                        | 4.4                    |                              |
|                |            |                        | -1.1                   |                              |
|                |            |                        | 6.1                    |                              |

### Table 5 Test Value of Water Vapor Permeability of PET/Al/PA/CPP Film

| Test condition | Cup method | Infrared sensor method |
|----------------|------------|------------------------|
|                | Test result [g/(m²·24h)] | Test result [g/(m²·24h)] | Relative deviation (%) | Relative standard deviation (%) |
| 23℃, 90%RH    | 0.097      | 0.101                  | -9.9                   | 6.5                          |
|                |            |                        | 0.0                    |                              |
|                |            |                        | 5.0                    |                              |
|                |            |                        | -5.0                   |                              |
|                |            |                        | 2.0                    |                              |
|                |            |                        | 7.9                    |                              |
| 38℃, 90%RH    | 0.108      | 0.110                  | 3.3                    | 3.0                          |
|                |            |                        | -0.3                   |                              |
|                |            |                        | -3.9                   |                              |
|                |            |                        | 2.4                    |                              |
|                |            |                        | -3.0                   |                              |
|                |            |                        | 1.5                    |                              |

### Table 6 Test Value of Water Vapor Permeability of VMPET Film

| Test condition | Cup method | Infrared sensor method |
|----------------|------------|------------------------|
|                | Test result [g/(m²·24h)] | Test result [g/(m²·24h)] | Relative deviation (%) | Relative standard deviation (%) |
| 23℃, 90%RH    | 0.245      | 0.232                  | -4.0                   | 5.3                          |
|                |            |                        | 5.9                    |                              |
|                |            |                        | 6.7                    |                              |
|                |            |                        | -5.7                   |                              |
|                |            |                        | 0.7                    |                              |
|                |            |                        | -3.6                   |                              |
| 38℃, 90%RH    | 0.447      | 0.436                  | -4.6                   | 4.5                          |
|                |            |                        | -0.5                   |                              |
|                |            |                        | -5.3                   |                              |
|                |            |                        | 5.9                    |                              |
|                |            |                        | 3.9                    |                              |
|                |            |                        | 0.6                    |                              |

The uncertainties of the infrared sensor method equipment and cup method equipment are compared to verify the reliability of the improved new infrared sensor method equipment. With the test data of PET film at 38℃, 90%RH as an example, the measured data and all calibrated measuring
parameter values are put into the relative uncertainty calculation formula calculated by the uncertainty propagation law during the test respectively, concluding that the relative uncertainty introduced in the water vapor permeability test process is 7.7% for the infrared sensor method equipment and 7.5% for the cup method equipment. The uncertainty indicates an evaluation of the uncertain degree of the test result. Based on the above results, the two types of testing equipment share similar uncertainty, showing that their reliability resembles. This also proves that the change in the corresponding mechanical structure guarantees reliability when the infrared sensor method testing equipment is applied to test the water vapor permeability of packaging materials.

From the test results under two sets of test conditions, the water vapor permeability of leather and PET film is around 7-30g/(m²·24h), showing their high moisture permeability performance; the vapor permeability of sheet and PP film is around 1-6g/(m²·24h), being materials with relatively low moisture permeability performance; the vapor permeability of PET/Al/PA/CPP and VMPET films is smaller than 1g/(m²·24h), also materials with low moisture permeability performance. Based on the test data of the infrared sensor method equipment in Tables 1-6, it can be found that under two sets of test conditions, the relative deviation for test results of each material is within ±10% and the relative standard deviation is lower than 10%, indicating that the degree of dispersion of test results for every single test is relatively small and the homogeneity is satisfactory. Therefore, the infrared sensor method equipment can be applied to test the water vapor permeability of materials with varying moisture permeability performance with good repeatability and stability.

With the vapor permeability measured by the cup method equipment as the reference, it is available to calculate the relative deviation between test results of the infrared sensor method equipment and the reference, so as to evaluate the accuracy of the test results of the infrared sensor method equipment. The relative deviation among the average test values for the 6 types of test samples is shown in Table 7. The maximum relative deviation among the 12 groups of data in the table is 7.7% and the minimum value is -6.9, not exceeding ±10%. According to GB1037 that the allowed deviation of test results may not exceed ±10%, the test results of the infrared sensor method equipment are consistent with those of the cup method equipment, meaning that it is accurate and effective to apply the infrared sensor method equipment to test the water vapor permeability of films and sheets with distinct barrier properties.

Table 7 Deviation between Test Results of the Cup Method and the Infrared Sensor Method

| Test sample           | 23°C, 90%RH | 38°C, 90%RH |
|-----------------------|-------------|-------------|
|                       | Cup method  | Infrared sensor method | Relative deviation (%) | Cup method  | Infrared sensor method | Relative deviation (%) |
| Leather               | 9.862       | 9.758       | -1.1          | 29.049      | 29.804      | 2.6                 |
| PET                   | 7.806       | 8.020       | 2.7           | 16.191      | 15.548      | -4.0                |
| Sheet                 | 1.041       | 1.044       | 0.3           | 2.381       | 2.216       | -6.9                |
| PP                    | 1.940       | 1.986       | 2.4           | 5.318       | 5.727       | 7.7                 |
| PET/Al/PA/CPP         | 0.097       | 0.101       | 4.1           | 0.108       | 0.110       | 1.9                 |
| VMPET                 | 0.245       | 0.232       | -5.3          | 0.447       | 0.436       | -2.5                |

5. Conclusion

In this paper, the temperature control device and the test sample clamping structure of the infrared sensor method water vapor permeability testing equipment have been improved, enabling the equipment to quickly reach and precisely control the test temperature and satisfying the sealed sample clamping requirements for the film/sheet and vessel type test samples. The scope of application of the equipment has also been expanded. The cup method water vapor permeability testing equipment has been compared with the improved infrared sensor method testing equipment, resulting in the uncertainties, relative deviations and relative standard deviations of 6 materials with the high, medium and low moisture permeability performance concerning their water vapor permeability. It proves that
the improved infrared sensor equipment features relatively high reliability, accuracy, repeatability and stability in testing the different levels of water vapor permeability.

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