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Determination of starch content of starch based plastics

Thermogravimetry (TG)

淀粉基塑料中淀粉含量的测定 热重法(TG)

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Determination of starch content of starch based plastics

Thermogravimetry (TG)

1 Scope

This Standard specifies the method to determine starch content in starch based plastics.

This Standard is applicable to starch based plastics with starch and resin as the main raw materials that have obvious differences in the characteristic temperature of thermal weight loss.

2 Normative references

The provisions in following documents become the provisions of this Standard through reference in this Standard. For dated references, the subsequent amendments (excluding corrigendum) or revisions do not apply to this Standard, however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 6425, *Nomenclature for thermal analysis*

ISO 11358, *Plastics - Thermogravimetry (TG) of polymers - General principles*

3 Terms and definitions

For the purposes of this document, the terms and definitions defined in GB/T 6425 as well as the followings apply.

3.1 starch based plastic

Plastic materials and products such as resins, films, sheets and injection molded products that are processed by molding with starch, plastic resins and additives as the main raw materials.

3.2 characteristic temperatures

The temperature in the TG curve that characterizes a substance, expressed by extrapolated initial temperatures and extrapolated end temperatures.

3.3 extrapolated initial temperatures

The temperature corresponding to the intersection of the TG curve starting mass line extrapolation and the tangent made at the maximum slope of the corresponding transition curve.

3.4 extrapolated end temperatures

The temperature corresponding to the intersection of the TG curve ending mass line extrapolation and the tangent made at the maximum slope of the corresponding transition curve.

4 Principle

Use TG method to determine starch and starch based plastic, respectively. Determine the characteristic temperature and the mass corresponding to that temperature in the TG curve of starch. In the TG curve of starch based plastic, determine the temperature corresponding to the characteristic temperature of starch and the corresponding mass. Use the ratio of the two masses to calculate the starch content in starch based plastic, expressed as mass fraction.

5 Instruments and equipment

5.1 Thermogravimetric analyzer

- High temperature furnace: the temperature can reach 800°C; the heating rate is 20°C/min;
- Thermal balance: the weighing can reach to 50mg; the weighing accuracy is 0.02mg;
- Can record the TG curve.

5.2 Gas source

Specimen gas: nitrogen; analytical grade; the flow is 30mL/min~150mL/min.

5.3 Balance

The accuracy is 0.1mg.

5.4 Other requirements shall be in accordance with the provisions of ISO 11358.

6 Specimen

- Powders, granules or specimens cut directly from the product.

- Quantity: 1 portion for starch, 2 portions for starch based plastic. The mass of each portion is about 10mg.

7 Test steps

- a) Turn on the thermogravimetric analyzer and gas source. Adjust the flow of specimen gas to 50mL/min. Adjust the flow of other gases to corresponding value;
- b) Carry out mass and temperature calibration to the instrument according to ISO 11358;
- c) Use the balance to weigh about 10mg of starch specimen. Put it into the thermogravimetric analyzer crucible;
- d) Heat the temperature from room temperature to 105°C at a constant rate of 20°C/min. Keep it at a constant temperature for 5min and then cool down to room temperature;
- e) Use the thermal balance to weigh the mass (m_s);
- f) Uniformly heat the temperature from room temperature to 600°C at a rate of 20°C/min. Record the TG curve;
- g) Use the balance to weigh about 10mg of starch based plastic specimen. Put it into the thermogravimetric analyzer crucible. Repeat the step of 7d). Use the thermal balance to weigh the mass (m_{s1});
- h) Repeat the step of 7f);
- i) Compare the characteristic temperatures T_A , T_B in the TG curve of the starch specimen with the temperatures T_{A1} , T_{B1} -- corresponding to the starch characteristic temperatures -- in the TG curve of starch based plastic specimen. If the difference between T_A and T_{A1} or T_B and T_{B1} is greater than 20°C, take another specimen and redo it.

8 Result calculation

8.1 Determination of TG curve and relevant parameters

In the TG curve of starch specimen (Figure 1), determine the weight loss transition that characterizes the specimen. At this transition, obtain the extrapolated initial temperature T_A and the extrapolated end temperature T_B as two characteristic temperatures of starch. Determine the corresponding mass m_A and m_B of T_A and T_B . In the TG curve of starch based plastic specimen (Figure 2), find out the weight loss transition at the temperature corresponding to the characteristic temperature of the starch specimen. Obtain the

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