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**Characterisation and testing methods of recycled poly
(ethylene terephthalate) (PET)**

聚对苯二甲酸乙二醇酯(PET)塑料回收料的表征特性及检测方法

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Characterisation and testing methods of recycled poly (ethylene terephthalate) (PET)

1 Scope

This Standard specifies the characterization characteristics, testing methods and quality assurance of recycled poly (ethylene terephthalate) (PET) used for production of finished or semi-finished plastic products.

This Standard applies to the evaluation of PET plastic recycled material in the transaction process. The relevant parties may select the applicable characterization characteristics and negotiate the delivery conditions in accordance with this Standard.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 1632.5, *Plastics - Determination of the viscosity of polymers in dilute solution using capillary viscometers - Part 5: Thermoplastic polyester (TP) homopolymers and copolymers*

GB/T 2035, *Terms and definitions for plastics*

GB/T 3682, *Plastics - Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics*

GB/T 6005, *Test sieves - Metal wire cloth, perforated metal plate and electroformed sheet - Nominal sizes of openings*

GB/T 12006.2, *Plastic polyamide - Part 2: Determination of moisture*

ISO 11664-4, *Colorimetry - Part 4: CIE 1976 L*a*b* Colour space*

3 Terms and definitions

For the purposes of this document, the terms and definitions defined in GB/T 2035 and the following apply.

3.1 recycled material

Annex A

(normative)

Determination of particle size and particle size distribution of PET recycled material fragments by sieving method

A.1 Scope

This appendix specifies a method for determining the particle size and particle size distribution of PET recycled material fragments.

A.2 Principle

With the help of mechanical shaking, the PET recycled material is sieved through a single sieve or a set of test sieves with different mesh sizes. If several test sieves are selected to form a sieve group, the test sieves shall be assembled in the order of increasing sieve size, so that the test sieve with the largest mesh size is placed on top.

The result can be expressed as sieve residue on a single test sieve.

A.3 Instruments and equipment

A.3.1 Weighing balance: The accuracy is 0.1g.

A.3.2 Test sieve: According to GB/T6005, the diameter of the screen frame is 200mm, with a cover and a receiving tray. The mesh sizes are 1mm, 2mm, 3.15mm, 4mm, 6.30mm, 8mm and 12.5mm.

A.3.3 Vibrating screen machine.

A.4 Steps

A.4.1 Check the test sieve (A.3.2) or the sieve group for damage to the sieve mesh or deformation of the sieve body. If the above defects are found, the test sieve shall be replaced.

A.4.2 Accurately weigh the mass of each test sieve (A.3.2), accurate to 0.1g.

A.4.3 Accurately weigh the mass of the receiving tray, accurate to 0.1g.

A.4.4 Assemble test sieve (A.3.2) or sieve group and receiving tray. Increase layer by layer. When assembling the sieve group, make sure to install it in the order of increasing sieve size, so that the sieve with the largest sieve size is placed on the top.

A.4.5 Accurately weigh 100g~300g (accurate to 0.1g) of the fragmented specimen to prepare for testing.

A.4.6 Move the weighed specimen into the top test sieve (A.3.2) to avoid spilling.

A.4.7 Cover the test sieve (A.3.2) with the cover. Install it on the vibrating screen machine (A.3.3).

A.4.8 Set the vibration time of the vibrating screen machine to 12min.

A.4.9 After vibrating the sieve, carefully separate the test sieve (A.3.2) or sieve group from the top. Weigh each test sieve, receiving tray, and sample on top of the test sieve or set of sieves together.

A.4.10 Determine each specimen twice.

A.5 Result representation

A.5.1 Calculate the sample mass in each test sieve or receiving tray according to formula (A.1):

$$(m_1 - m_2) = m_3 \text{ \& } (m_4 - m_5) = m_6 \text{ (A.1)}$$

Where,

m_1, m_4 - The mass of the test sieve or the receiving tray plus the remaining fragments during the first and second determinations, in grams (g);

m_2, m_5 - The mass of the test sieve or the receiving tray during the first and second determinations, in grams (g);

m_3, m_6 - The mass of the fragments remaining in the test sieve or the receiving tray during the first and second determinations, in grams (g).

Calculate the average value of the two determination results according to formula (A.2):

$$\frac{(m_3 + m_6)}{2} = m_r \text{ (A.2)}$$

Where,

m_r - The average value of the sieve allowance of each test sieve or the allowance of the receiving tray, in grams (g).

A.5.2 According to formula (A.3), calculate the percentage R of the sieve balance of each test sieve or the balance of the receiving tray to the total sample amount:

$$R = \frac{m_r}{m_s} \times 100\% \text{ (A.3)}$$

Where,

Annex B

(normative)

Determination of water content by gravimetry

B.1 General

This appendix specifies a method for the determination of the water content in PET recycled material fragments.

B.2 Principle

Place a certain mass of samples in a blast oven at 150°C. After the water evaporates, measure the weight loss of the sample to obtain the water content.

B.3 Instruments and equipment

B.3.1 Analytical balance: The accuracy is 0.0001g.

B.3.2 Porcelain crucible: 80mL~90mL.

B.3.3 Dryer.

B.3.4 Oven: With blast system. The maximum temperature is 250°C.

B.4 Steps

Dry the porcelain crucible (B.3.2) to constant weight. Use the balance (B.3.1) to weigh (accurate to 0.0001g) and record its mass. Place about 50g of specimen into the crucible. Weigh its mass (accurate to 0.0001g). Put it in the oven (B.3.4). Dry at 150°C for 4h. After taking out the crucible, place it in the dryer (B.3.3) for 30min. Use the balance to reweigh its mass (accurate to 0.0001g).

B.5 Result representation

Calculate the water content C according to formula (B.1), expressed in %.

$$C = \frac{m_1 - m_2}{m_1 - m_0} \times 100\% \quad \dots\dots\dots (B.1)$$

Where,

m_0 - The mass of the porcelain crucible, in grams (g);

m_1 - The mass of the porcelain crucible and the specimen before drying, in grams (g);

Annex C

(normative)

Rapid determination for residual impurities

C.1 General

This appendix specifies a method for the determination of the residual impurity content in PET recycled material fragments.

This appendix is applicable to the detection of residual PVC and polyolefin in PET recycled material fragments.

C.2 Principle

Put a certain mass of specimen into a blast oven at 220°C for heat treatment. During this process, various impurities are degraded and discolored. It can be distinguished from the specimen with the naked eye. The changes that take place in this process are as follows:

- PVC discoloration;
- Polyolefin melting and discoloration.

C.3 Instruments and equipment

C.3.1 Blast oven: The maximum temperature is 250°C.

C.3.2 Weighing balance: The accuracy is 0.1g.

C.3.3 Analytical balance: The accuracy is 0.0001g.

C.4 Steps

Accurately weigh 1000g of specimen fragments, to the nearest 0.1g. Pack the fragments evenly into aluminum or another metal container. Put in the oven (C.3.1) preheated to 220°C. After 1 h, taken it out and cool to room temperature in the dryer. The impurities are separated as follows:

- Separate the black carbonized particles. Use the analytical balance (C.3.3) to weigh its mass. Record it as m_1 ;
- Identify polyolefins by characteristics such as their shape and thickness. After heat treatment, the loose and yellowish decomposed or melted parts that stick to the PET are separated out. Use the analytical balance (C.3.3) to weigh its mass. Record it as m_2 .

Annex D

(informative)

Potentiometric determination for residual alkalinity

D.1 General

This appendix specifies a method for the determination of residual alkalinity of PET recycled material.

D.2 Principle

Add a certain mass of specimen to a certain volume of distilled water. Stir well. Residual alkaline substances on the surface of the specimen will increase the pH of the water. Use a glass electrode to measure its pH value.

D.3 Instruments and equipment

D.3.1 Magnetic stirrer, or other devices with the same function.

D.3.2 Weighing balance: The accuracy is 0.1g.

D.3.3 Beaker: 1000mL.

D.3.4 Graduated cylinder: 500mL.

D.3.5 pH meter with glass electrode.

D.4 Reagents

Distilled water.

D.5 Steps

Calibrate the pH meter with a buffer solution of known pH value. Accurately weigh 100g of sample. Put it into a 1000mL beaker. Add 500mL of distilled water. Make sure all pieces are submerged in water. Stir for 10min.

After stirring, filter off the solution. Immerse a glass electrode in a solution to measure the pH value. When the value is stable, record the test result.

D.6 Result representation

Residual alkalinity is expressed as the pH of the solution. Record the initial pH value of distilled water for blank control.

Annex E

(informative)

Determination of infusible impurities by filtration method

E.1 General

This appendix specifies a method for determining the content of non-melting impurities (such as aluminum, paper, carbonized PVC) in PET recycled material fragments by melt filtration.

This appendix also applies to the detection of PET raw material or its compounds. But it is not for polymers that thermally decompose under test conditions.

E.2 Principle

The change of extrusion pressure of molten polymer in a certain size filter is a function of the content of solid particles in the polymer. When the test conditions are constant, the purity of the sample can be reflected according to the change of pressure.

E.3 Instruments and equipment

Single screw extruder: It can control the extrusion temperature. It comes with feed pump, temperature, pressure recorder and metal filter. The mesh size is 35μm.

E.4 Steps

Install a metal filter with a mesh size of 35μm at the extrusion port. According to the model of the extruder, adjust the feed pump to the best working condition. Set the temperature of the extruder and filter head. It shall be ensured that the temperature of the polymer in the filter head can reach $(290 \pm 1)^\circ\text{C}$. Turn on the extruder. When the parameters are stable, the pressure at the filter is recorded at regular intervals. The duration is 2h~3h.

E.5 Result representation

Calculate the pressure change ΔP according to formula (E.1), in MPa per hour square centimeter [$\text{MPa}/(\text{h} \cdot \text{cm}^2)$].

$$\Delta P = (P - P_0) \times Q_p / (M_p \times A) \quad \dots\dots\dots (E.1)$$

Where,

P - The pressure after t time, in megapascals (MPa);

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