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Super-absorbent polymer for sanitary towel and diapers

纸尿裤和卫生巾用高吸收性树脂

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Super-absorbent polymer for sanitary towel and diapers

1 Scope

This standard specifies the product classification, requirements, test methods, inspection rules and marking, packaging, transportation and storage of polyacrylate super-absorbent polymers for sanitary towels and diapers.

This standard applies to polyacrylate super-absorbent polymers which are used by all kinds of paper diapers (sheets, pads) and sanitary towels (pads). The super-absorbent polymers which are used by other disposable sanitary products may make reference to this standard.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this standard.

GB/T 6682 Water for analytical laboratory use - Specification and test methods

GB/T 7974 Paper, board and pulps - Measurement of diffuse blue reflectance factor - D65 brightness (Diff/Geometry, outdoor daylight conditions)

3 Product classification

The super-absorbent polymer is divided into the super-absorbent polymer for paper diapers (sheets, pads) and the super-absorbent polymer for the sanitary towels (pads) in accordance with the use of the product. The super-absorbent polymer for paper diapers (sheets, pads) is divided into the super-absorbent polymer for baby diaper (sheets, pad) and the super-absorbent polymer for adult diapers (sheets, pads) in accordance with the objective of use.

6 Inspection rules

6.1 Provisions on inspection batch

The products which are produced by the same raw material, the same process and the same period of time form a batch, the amount of each batch does not exceed 500 t.

6.2 Sampling methods

Take samples evenly from the same batch and not less than 3 packaging bags, the sample amount shall be 1 kg.

6.3 Rules for decision

When the inspection product meets the requirements of this standard or contract, it is judged to be qualified; when any of the inspection items fails, it is judged to be unqualified.

6.4 Quality assurance

The manufacturer shall ensure that the product quality meets the requirements of this standard or contract. The product can only be exit-factory after being inspected qualified and accompanied with quality certificate mark.

7 Marking, packaging, transportation, storage

7.1 Sales mark and packaging

- **7.1.1** The product sales package shall be marked with the following contents:
 - a) Product name, implemented standard number, trademark, product type;
 - b) Name, address and contact information of the enterprise;
 - c) Date of manufacture and shelf life, or production lot number and limited period of use;
 - d) Main raw materials;
 - e) Transportation and storage conditions.
- **7.1.2** The product shall be packed in a packaging bag with a lined plastic film. The packaging bag shall have sufficient strength, to ensure that it will not break or fall off during use. The various identification information on the sales package shall be clear and not easy to fade.

Appendix A

(Normative)

Determination of residual monomers (acrylic acid)

A.1 Principle

The sample is extracted by physiological saline, the residual monosodium acrylate and acrylic acid in the sample are transferred to the solution. After 1 hour, the solution is filtered, the filtrate is taken and subjected to high performance liquid chromatography to measure the content of acrylic acid, the result is based on the content of acrylic acid.

A.2 Reagents and materials

Unless otherwise specified, only chromatographic reagents are used.

- **A.2.1** Water, GB/T 6682, level I.
- **A.2.2** Normal saline, concentration 0.9%. WEIGH 9.00 g (accurate to 0.01 g) of sodium chloride (analytical grade) in a beaker, DISSOLVE and TRANSFER it into a 1 L volumetric flask, USE water to dilute it to the mark, SHAKE it uniformly.
- **A.2.3** Concentrated phosphoric acid, $c(H_3PO_4) = 85\%$ (mass ratio), chromatographic grade or higher.
- **A.2.4** Aqueous phosphoric acid solution, $c(H_3PO_4) = 0.1\%$ (mass ratio). WEIGH 1.0 g of concentrated phosphoric acid in a 1 L volumetric flask, USE water to dilute it to the mark, SHAKE it uniformly.
- **A.2.5** Acetonitrile, chromatographic grade or higher.
- **A.2.6** Acrylic acid, the purity is greater than 99.5%.
- **A.2.7** Acrylic standard stock solution (1000 mg/L): WEIGH 0.1000 g of acrylic acid (A.2.6), accurate to 0.0001 g, PUT it into a 100 mL volumetric flask, USE water to dilute it to 100 mL.
- **A.2.8** Acrylic standard intermediate solution (100 mg/L): PIPETTE 10 mL of acrylic acid standard stock solution (A.2.7) into a 100 mL volumetric flask, USE water to dilute it to 100 mL.

A.3 Instruments and equipment

A.3.1 High performance liquid chromatography.

Appendix B

(Normative)

Determination of volatile content

B.1 Instrument and equipment

- **B.1.1** Oven, it can keep the temperature at 105 °C ± 2 °C.
- **B.1.2** Balance, the sensitivity is 0.001 g.
- **B.1.3** Dryer, with active desiccant (e.g., silica gel).
- **B.1.4** Specimen container, for the transfer and weighing of specimen. The container is made of a lightweight material that is water-repellent and resistant to changes under experimental conditions. The inner diameter of the container is $55 \text{ mm} \pm 5 \text{ mm}$.

B.2 Determination step

WEIGH approximately 4.0 g of the specimen, accurate to 0.001 g, LOAD it into a specimen container (B.1.4) which has reached to constant weight. PLACE the container which contains the specimen in an oven (B.1.1) at a temperature of 105 °C, OPEN the container lid, DRY it for 3 hours. After 3 hours, OPEN the oven, COVER the container lid in the oven, then PLACE the container into the dryer (B.1.3) for 30 minutes, WEIGH the mass of the container and the specimen. PERFORM two parallel determinations simultaneously.

B.3 Calculation of results

The volatile content is calculated in accordance with formula (B.1):

$$M = \frac{m_1 - m_2}{m_1} \times 100$$
 (B.1)

Where:

M - Volatile content of the specimen, %;

m₁ - The mass of the specimen before drying, in grams (g);

m₂ - The mass of the specimen after drying, in grams (g).

The arithmetic mean of the two determinations is used as the result, the result is rounded off to one decimal place. The absolute error of the two determinations shall not exceed 0.4%.

Appendix D

(Normative)

Determination of distribution of particle size

D.1 Instrument and equipment

- **D.1.1** Balance, the sensitivity is 0.01 g.
- **D.1.2** Screen vibrator, the amplitude is 1 mm and the frequency is 1400 r/min.
- **D.1.3** Screen, standard screen has a diameter of 200 mm and a mesh size of 45 μ m and 106 μ m.
- D.1.4 Receiving base and cover.
- **D.1.5** Brush.

D.2 Determination step

- **D.2.1** CLEAN the screen (D.1.3) before each use, CHECK the entire surface of the screen under the light source, CHECK the damage of each screen. If any cracks or holes are found, DISCARD the damaged screen and REPLACE it with a new one. If the screen is not clean, it needs to be cleaned.
- **D.2.2** STACK the screens on the screen vibrator (D.1.2), PLACE the receiving base (D.1.4) at the bottom, STACK the screens from top to bottom in the order of 106 μ m to 45 μ m. USE a 250 mL glass beaker to weigh 100 g of specimen, accurate to 0.01 g. Gently POUR the specimen into the top screen, ADD the cover (D.1.4) and START the screen vibrator for 10 min. Then carefully REMOVE the screen, respectively WEIGH the mass of the specimen above the 45 μ m screen and on the receiving base. During determination, AVOID ventilation airflow. USE the brush (D.1.5) to collect the specimen below screen into a waste dish, CLEAN the screen.

D.3 Calculation of results

The distribution of particle size is calculated in accordance with formula (D.1) and formula (D.2):

$$S_1 = \frac{m_2 + m_3}{m_1} \times 100$$
(D.1)

$$S_2 = \frac{m_3}{m_1} \times 100$$
(D.2)

Appendix E

(Normative)

Determination of density

E.1 Instrument and equipment

- **E.1.1** Balance, the sensitivity is 0.01 g.
- **E.1.2** Density test device.
- **E.1.3** Funnel, with a capacity greater than 120 mL, with perforated throttling damping or baffle, inner diameter of the orifice is $10.00 \text{ mm} \pm 0.01 \text{ mm}$.
- **E.1.4** Density cup, with the cup cylinder capacity $100 \text{ cm}^3 \pm 0.5 \text{ cm}^3$.

E.2 Determination step

- **E.2.1** PLACE the density test device (E.1.2) on the platform, ADJUST the screws on the three feet to keep them level. PLACE the washed and dried funnel (E.1.3) vertically at a height of 40 mm \pm 1 mm above the center of the density cup (E.1.4), to ensure the funnel level.
- **E.2.2** WEIGH the mass of the empty density cup, accurate to 0.01 g, RECORD its mass as m₁. Then PLACE the weighed empty density cup directly below the funnel.
- **E.2.3** Accurately WEIGH 100 g (accurate to 0.01 g) of specimen, gently ADD it to the funnel. The orifice throttling damping or baffle under the funnel is closed.
- **E.2.4** Quickly OPEN the orifice throttling damping or baffle under the funnel, so that all the specimens in the funnel fall naturally.
- **E.2.5** USE glass rod to scrape off the excess specimen on the top of the density cup. DO not tap or shake the density cup.
- **E.2.6** WEIGH the mass of the density cup which contains the specimen, accurate to 0.01 g, RECORD the mass as m_2 .

E.3 Calculation of results

The density of the specimen is calculated in accordance with formula (E.1):

$$\rho = \frac{m_2 - m_1}{V}$$
 (E.1)

Appendix F

(Normative)

Determination of yellowing value

F.1 Principle

The color of the specimen changes after a certain period of time under the specified temperature and humidity conditions. The ratio of the D65 brightness difference before and after the specimen treatment to the D65 brightness value before the specimen treatment is used to represent the yellowing value.

F.2 Instrument and equipment

- **F.2.1** Balance, the sensitivity is 0.01 g.
- **F.2.2** Constant temperature and humidity chamber, which can keep the temperature at 50 $^{\circ}$ C \pm 2 $^{\circ}$ C, maintain the relative humidity at 90% \pm 2%.
- **F.2.3** Test container, in glass or stainless steel, cylindrical, with a bottom diameter of 35 mm \pm 5 mm and a height of 10 mm \pm 2 mm.
- **F.2.4** Reflectance photometer, in accordance with the provisions of GB/T 7974.

F.3 Determination step

- **F.3.1** TAKE a sufficient amount of specimen, PLACE in and FILL the test container (F.2.3), to ensure that the surface of the specimen is flat and level with the top edge of the test container.
- **F.3.2** Carefully PLACE the test container which contains the specimen in the test area of the reflectance photometer (F.2.4), DETERMINE the D65 brightness R₁ of the specimen.
- **F.3.3** SET the constant temperature and humidity chamber (F.2.2) to a temperature of 50 °C and a relative humidity of 90%. TURN on the constant temperature and humidity chamber. After reaching to the set temperature and humidity, PLACE the test container which contains the specimen into the constant temperature and humidity chamber for conditioning.
- **F.3.4** After 28 d, TAKE out the test container which contains the specimen, USE a glass plate to press the surface of the specimen flatly, to make the surface of the specimen level with the upper edge of the test container, then carefully PLACE the test container which contains the specimen in the test area of reflectance photometry, to determine the D65 brightness R₂ of the specimen.

Appendix G

(Normative)

Determination of absorption speed

G.1 Reagents and materials

Standard synthetic test solution: see Appendix K.

G.2 Instrument and equipment

- **G.2.1** Balance, the sensitivity is 0.001 g.
- **G.2.2** Beaker, the capacity is 100 mL.
- G.2.3 Measuring cylinder, 10 mL.
- G.2.4 Stopwatch.

G.3 Determination step

- **G.3.1** USE balance to weigh 1.000 g of specimen, accurate to 0.001 g, POUR it into a 100 mL beaker. SHAKE the beaker to evenly spread the specimen at the bottom of the beaker.
- **G.3.2** USE the measuring cylinder to measure 5.0 mL of 23 °C standard synthetic test solution, quickly POUR it into the beaker which contains the specimen, quickly SHAKE the beaker to evenly distribute the specimen. POUR in the standard synthetic test solution and start timekeeping at the same time. When the liquid mirror disappears in the beaker, STOP timekeeping immediately and RECORD the time.

Note: When shaking the beaker, AVOID the specimen from being attached to the wall of the beaker.

G.3.3 Each sample is determined twice.

G.4 Calculation of results

The arithmetic mean of the two determinations is taken as the determination result, the absorption speed is expressed in seconds (s), the result is rounded off to an integer position.

Appendix J

(Normative)

Determination of extractable content

J.1 Reagents and materials

Unless otherwise specified, only analytical reagents are used.

- J.1.1 Water, GB/T 6682, Level III.
- **J.1.2** Sodium hydroxide solution, c(NaOH) = 0.1 mol/L.
- **J.1.3** Hydrochloric acid solution, c(HCI) = 0.1 mol/L.
- **J.1.4** Normal saline, concentration 0.9%. WEIGH 9.00 g (accurate to 0.01 g) of sodium chloride in a beaker, DISSOLVE and TRANSFER it to a 1 L volumetric flask, USE water to dilute it to the mark, SHAKE it uniformly.
- **J.1.5** Standard buffer solution, with a pH of 4.00, 6.86, 9.18 at 25 °C.

J.2 Instrument and equipment

- **J.2.1** Balance, the sensitivity is 0.0001 g.
- J.2.2 pH meter.
- **J.2.3** Beaker, the capacity is 250 mL.
- J.2.4 Parafilm.
- J.2.5 Magnetic stirrer and stir bar.
- **J.2.6** Vacuum filter bottle and Buchner funnel.
- J.2.7 Filter paper, quantitative filter paper.
- J.2.8 Vacuum pump.
- J.2.9 Burette, 10 mL or 20 mL, readable accuracy of 0.01 mL.

J.3 Determination step

- **J.3.1** USE the measuring cylinder to accurately measure 200 mL of normal saline, POUR it into a 250 mL beaker, PUT in a stir bar.
- **J.3.2** WEIGH 1.000 g of specimen, accurate to 0.001 g, then POUR all into the

V_{HCl,s} - The volume of hydrochloric acid solution required to titrate the supernatant of the filtered sample to pH 2.7, in milliliters (mL);

V_{HCl,b} - The volume of hydrochloric acid solution required to titrate the blank solution to pH 2.7, in milliliters (mL);

c_{HCl} - The concentration of hydrochloric acid solution, in moles per liter (mol/L).

J.4.3 The total amount of carboxylic acid which is neutralized in the filtrate is calculated in accordance with formula (J.3):

$$n_{\text{COONa}} = n_{\text{tot}} - n_{\text{COOH}}$$
(J.3)

Where:

n_{COONa} - The total amount of carboxylic acid which is neutralized in the filtrate, in moles (mol);

J.4.4 The relative mass of carboxylic acid and carboxylate is calculated in accordance with formula (J.4) and formula (J.5):

Where:

mcooh - The relative mass of acrylic acid, in grams (g);

mcoona - The relative mass of sodium acrylate, in grams (g);

Mcooн - The molar mass of acrylic acid, in grams per mole (g/mol) (Mcooн = 72.0);

 M_{COONa} - The molar mass of sodium acrylate, in grams per mole (g/mol) (M_{COONa} = 94.0);

 F_{dil} - The dilution factor, which is 200/50 = 4.

J.4.5 The extractable content in the super-absorbent polymer is calculated in accordance with formula (J.6):

$$w = \frac{m_{\text{COOH}} + m_{\text{COONa}}}{m_s} \times 100$$
(J.6)

Where:

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