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Standard Test Method for Stability of Cellulose Fibers in Alkaline Environments[1]

This standard is issued under the fixed designation D6942; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes a procedure for determining the effect of exposure to alkaline environments on the strength of cellulose fibers. An alkaline environment is defined to be any matrix in which the pH is greater than 8 for a period of 2 or more hours.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1348 Test Methods for Moisture in Cellulose

D1695 Terminology of Cellulose and Cellulose Derivatives

2.2 *TAPPI (Technical Association of the Pulp & Paper Industry) Standards:*

T 205 Forming Handsheets for Physical Tests of Pulp[3]

T 231 Zero-span Breaking Strength of Pulp (Dry Zero-span Tensile)[3]

3. Terminology

3.1 *Definitions*—For standard terminology of cellulose and cellulose derivatives, see Terminology D1695.

4. Summary of Test Method

4.1 This test method can be used to compare different cellulose pulp fiber types based on their response to a standard alkaline solution. The stability factor defined below can be used to measure the effect of exposure to alkaline conditions on fiber strength.

4.2 Cellulose fibers are treated with a standard alkaline solution for a specified interval, washed free of alkali, and then formed into standard handsheets (see TAPPI T 205) for strength testing. Zero-span tensile testing (see TAPPI T 231) is used to determine the effect on fiber strength.

4.3 A stability ratio is defined based on the ratio of the zero-span tensile of alkali treated fibers divided by the zero-span tensile of untreated (control) fibers.

5. Significance and Use

5.1 This method is intended to provide a generalized procedure for determining the stability of cellulosic pulp fibers exposed to alkaline environments. Specifically, this method allows various pulp types to be compared with respect to the effect of exposure to alkaline conditions on the strength of individual cellulosic fibers based on a zero-span tensile test. The time intervals listed in the procedure are not critical, and more intervals of shorter or longer duration may be added. In addition, the procedure may be simplified by removing some of the intermediate intervals so long as a range of intervals is determined. An example of a simplified procedure would be to determine 4 intervals (for example, 1 day, 1 week, 2 weeks, 4 weeks; or 1 day, 3 day, 7 day, 14 day).

5.2 The specified solution (1N NaOH) is strongly alkaline. Although this alkali concentration is higher than some environments that would be simulated by this test, the stronger pH provides better differentiation between different cellulose fiber types. Although alkaline stability based on other alkalis (for example, KOH, $Ca(OH)_2$, etc.) at a different concentration could be determined by this method, 1N NaOH is to be considered the standard solution. Alkaline stability results from other treatments may be reported in addition to the standard solution if the additional solution(s) provide useful information.

6. Interferences

6.1 There are no known interferences for this method.

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[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.36 on Cellulose and Cellulose Derivatives.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from Technical Association of the Pulp and Paper Industry (TAPPI), 15 Technology Parkway South, Norcross, GA 30092, http://www.tappi.org.

7. Apparatus

7.1 *Handsheeting Apparatus,* as defined in TAPPI T 205.

7.2 *Zero-span Tensile Tester,* as described in TAPPI T 231.

7.3 *Moisture Balance.*

7.4 *Analytical Balance.*

8. Reagents and Materials

8.1 1N sodium hydroxide (NaOH).

9. Hazards

9.1 Sodium hydroxide solutions are corrosive, and thus harmful to the skin and eyes. Wear safety glasses or goggles, gloves, and lab coat or chemical apron. while working with caustic solutions.

10. Sampling, Test Specimens, and Test Units

10.1 Values stated in SI units are to be regarded as the standard. Values in parentheses are for information only.

10.2 Starting cellulose fibers should be in a dry sheet form (drylap) or in a dry, low-density bulk form. In this context, the term dry means at equilibrium moisture content (see 3.1), which is 6 to 8 % moisture for most pulps.

11. Calibration and Standardization

11.1 Calibration and maintenance of the zero-span tensile tester will be accomplished as prescribed in TAPPI T 231. In addition, a control chart of the instrument will be maintained based on breaking paper strips cut from control sheets of paper. A ream of copy paper can be used for this purpose or any other paper with consistent furnish, uniform basis weight, and uniform density (see 3.1). Control paper produced on a paper machine should be tested in the machine direction.

12. Conditioning

12.1 Handsheets are to be conditioned prior to testing as described in TAPPI T 205.

13. Procedure

13.1 For drylap, mechanically disintegrate the pulp sheet to get 150 g of individualized fibers for each sample to be tested. High-density pulp sheets can also be slurried at low consistency, then air-dried to provide a bulk sample of low density. The bulk, air-dry sample can then be disintegrated mechanically or by hand to provide individualized fibers.

13.2 To 10 g (dry basis) of cellulose fibers, add 23.3 g of 1N NaOH and allow to remain for 24 h. (This corresponds to a 30 % consistency, that is, 10 g pulp/33.3 g total. Moisture in the pulp is ignored as long as the moisture content (see TAPPI T 231) is <10 %. A fiber sample larger than 10 g can be used, but the starting consistency must still be 30 %.) The sample may be placed in an uncovered beaker to simulate an environment that is open to the atmosphere, or placed in a sealed bag to simulate an environment in which the consistency would remain constant.

13.3 Repeat step 13.2 to prepare five more samples that will be left to age for time intervals of 3, 7, 14, 21, and 28 days, respectively. Once the time interval has been met, work-up of the samples is accomplished by collecting fibers on a wire screen (100 mesh), washing with tap water until washings are substantially neutral (pH = 7 to 7.5), and then air drying.

13.4 Prepare 2 sets of standard handsheets according to TAPPI T 205 for each time interval. One set will be made from pulp that has not been treated with sodium hydroxide and will be the control set. The other set will be prepared from fibers that have been exposed to alkali for the designated time interval. These 2 sets of handsheets will be prepared on the same day.

13.5 Each set of handsheets will be tested for zero-span tensile according to TAPPI T 231.

14. Calculation or Interpretation of Results

14.1 Determine the zero-span stability ratio (that is, ZSSR) by dividing the zero-span tensile result of the alkali treated sample by the zero-span tensile result from the corresponding untreated (control) sample. The results can be reported as a decimal ratio, such as 0.921 or as a percentage, such as 92.1 %. Reporting 3 significant figures is recommended.

14.2 The zero-span stability ratios will be reported individually for each time interval sample and/or as an average value of all the time interval samples tested.

14.3 Note that higher ratios will be observed for pulps that have greater strength stability in an alkaline environment.

15. Report

15.1 Report the zero-span stability ratios (ZSSR) determined for each time interval sample as a decimal fraction or as a percentage along with the average zero span stability ratio determined from all time interval samples. Since 1N NaOH is the standard test solution, it need not be specified, but if another test solution is used in addition to the standard solution, its composition must be specified.

16. Precision and Bias

16.1 Precision and bias for the zero-span tensile test are given in TAPPI T 231. Repeatability within a laboratory is from 3 to 5 %, and reproducibility between laboratories (30 samples at 3 laboratories) was 10 %.

16.2 Repeatability of zero span tensile tests used to calculate stability ratios was found to be 5 % based on 14 sets of control handsheets made at different times by 2 operators where each set was tested 4 times by cutting 2 test strips from 2 handsheets from each set (64 pulls).

16.3 Repeatability of the stability ratio is partly dependent on the type of fibers tested (SSK, NSK, sulfite, mechanical, etc.) and the duration of the test (1 day, 1 week, 4 weeks). For samples determined according to section 13 in uncovered beakers, the repeatability, expressed as a % coefficient of variation, was 5 to 8 %.

17. Keywords

17.1 alkaline stability; cellulose fibers; zero-span stability ratio; zero-span tensile