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An American National Standard

Standard Practice for the Separation and Examination of Stickies[1]

This standard is issued under the fixed designation D 6148; 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 practice is applicable to a wide range of virgin pulps or pulps derived from recovered materials or recovered paper of the type typically used to produce paper, paperboard and other paper products.

1.2 This practice describes laboratory procedures whereby stickies are typically separated from pulp fibers, and then the separated stickies fraction is examined by qualitative or quantitative procedures, or both.

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:*

D 585 Practice for Sampling and Accepting a Single Lot of Paper, Paperboard, Fiberboard, and Related Product[2]

D 1968 Terminology Relating to Paper and Paper Products[2]

D 2019 Test Method for Dirt in Paper and Paperboard[2]

E 122 Practice for Calculating Sample Size to Estimate, With a Specified Tolerable Error, the Average for Characteristic of a Lot or Process[3]

2.2 *TAPPI Documents*[4]

T 205 Forming Handsheets for Physical Testing of Pulp

T 210 Sampling and Testing Wood Pulp Shipments for Moisture

T 412 Moisture in Pulp, Paper and Paperboard

UM 242 Shive Content in Mechanical Pulp (Somerville Fractionator)

3. Terminology

3.1 *Definitions:*

3.1.1 Definitions used in this practice are those found in Terminology D 1968 or the *Dictionary of Paper*.[4]

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *stickies, n*—particulate, generally non-fibrous, contaminants suspended in pulps of the type used in papermaking which adhere to themselves, other components of the paper structure, components of the papermaking machinery, and in the case where the contaminants are incorporated into finished paper or paper products, cause the unexpected and generally undesirable adherence one to another of units of the finished paper or paper product.

3.2.1.1 *Discussion*—The suspended particulate contaminants are usually of low specific gravity and include, but are not limited to, residual materials such as adhesives and coating residues, films, tapes, rubber-like particles, inks, and hydrolysis products of synthetic sizing materials.

4. Summary of Practice

4.1 A slurry of pulp fibers, after mechanical disintegration if necessary, is placed in a separatory device in contact with a plate or screen of defined area having openings of specified number and size.

4.1.1 The characteristics of the plate or screen shall be such that the stickies contaminants are separated from papermaking fibers at an efficiency required to differentiate and discriminate between test materials that are known to contain different levels of stickies, or that have been found to exhibit different performance in the papermaking process due to the levels of stickies incorporated within them.

4.1.2 In some separatory devices, the slurry is recirculated in contact with the plate or screen within the separator device to achieve stickies separation. In other devices, stickies remaining on the plate or screen are washed free from the plate or screen with a supply of fresh water, or mechanically removed with tweezers or a similar device. Sometimes both procedures occur.

4.1.3 The typical plate or screen used for stickies separation in this practice contains slots having a width of 0.15 mm (0.006 in.), however slotted plates or screens having a different width opening, for example 0.10 mm (0.004 in.) or 0.20 mm (0.008 in.), have been successfully used in specific applications.

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[1] This practice is under the jurisdiction of ASTM Committee D06 on Paper and Paper Products and is the direct responsibility of Subcommittee D06.92 on Test Methods.

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[2] *Annual Book of ASTM Standards*, Vol 15.09.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] Available from the Technical Association of the Pulp and Paper Industry, Technology Park, P.O. Box 105113, Atlanta, GA 30348.

4.2 The quantity, qualitative characteristics, or both, of the separated stickies may be determined by any of the following instruments or approaches, singly or in combination, based on the measurement information required:

4.2.1 Touch or visual inspection using tweezers, dissecting needles, size estimation gages, or similar devices (size, color, presence or absence of stickiness),

4.2.2 Automated image analysis (number and size of particles),

4.2.3 Reaction to heat (thermal properties),

4.2.4 Examination under radiant energy including, but not limited to ultraviolet, near infrared, or infrared radiation (spectroscopic [structural] properties),

4.2.5 Examination using chemical reagents including dyes or solvents (chemical properties),

4.2.6 Gravimetric quantification, and

4.2.7 Other suitable means of choice, as defined by the users of this practice.

5. Significance and Use

5.1 The increased use of alternative fiber sources, including but not limited to fiber from recovered material or recovered paper, or both, has increasingly resulted in the presence of stickies in fiber sources of the type typically used in papermaking and, to a lesser extent, in their presence in finished papers and paper products made from such fiber sources.

5.2 This practice provides a consistent format in which to separate and make either qualitative or quantitative measurements, or both, on stickies.

5.3 This practice is suitable for application throughout the entire process of paper production and use, enabling the producer, converter and user of paper to measure, describe, and specify required properties of pulp, paper, and paper products with regard to stickies.

6. Equipment

NOTE 1—Various pieces of equipment may be used in performing the procedure described in this practice. The exact equipment required varies based upon the portions of the procedure used.

6.1 *Disintegrator*, as described in TAPPI T 205, Appendix A.

NOTE 2—The disintegrator may not be required or even desirable when slurried pulps from the papermaking process are being evaluated.

6.2 Separatory instrument or device for removing stickies from the pulp slurry. The instrument or device shall separate pulp from stickies based upon a screening technique, and shall incorporate a screen or plate with the following characteristics:

6.2.1 Slots in the screen or plate of the instrument or device are of such dimension that all fibers in the slurry are allowed to pass through,

NOTE 3—In spite of this requirement, long fiber strings or fiber bundles will frequently be retained on the plate or screen.

6.2.2 The plate or screen in the instrument or device retains stickies at a level and efficiency that efficient and statistically reliable differentiation among pulps known to contain measurably different stickies levels is possible,

6.2.3 The orientation and size of the slots in the plate or screen may be precisely described, verified, and reliably reproduced.

6.2.4 The Somerville Fractionator (TAPPI UM 242)[5] and the Pulmac MasterScreen[5] have been found to comply with these requirements and are suitable instruments for use in this practice. There may be other suitable separatory units which meet the requirements, as well.

NOTE 4—No screening or separating device has yet demonstrated the ability to quantitatively remove from a slurry of pulp fibers every sticky particle that may result in the probability of negative performance characteristics in some application of interest for a particular pulp or paper material. What the practically useful stickies separating system does is to remove some reproducible portion of the stickies present. The portion of the stickies removed will be a function of the specific interaction of the separating system's screen or plate opening size, the amount of solids (both pulp fibers and stickies) present in the test specimen, and characteristics of the stickies separating system including flow rate, recycling and washing system used, and the particle size distribution of the stickies particles present. Since two stickies separating systems with different operational characteristics are unlikely to produce the same specific interaction between the screening system and the test specimen, nonidentical separatory systems will generally produce results which are different quantitatively, qualitatively, or both. However, separating screens or plates chosen to meet the general criteria above will rank different test specimens in the same general order with respect to stickies, even though there will be differences between separation systems in the specific qualitative and quantitative information derived.

6.3 *Filter Paper* to fit the system used (as required).

6.4 *Particle Size Comparison Chart*, such as the dirt speck comparison chart described in Test Method D 2019 for visual estimation of size of stickies particles separated from specimen (as desired).

6.5 *Common Laboratory Equipment*, such as tweezers, dissecting needles, glassware, and similar.

6.6 *Specific Instrumentation*, such as balances, spectrophotometers, thermal analysis units, and similar, as required for any specific stickies identification procedures which are to be used as agreed by the users of the practice (as desired).

NOTE 5—The specific performance capabilities of instrumentation used for examination of the stickies fraction are not specified here because of the wide variety of equipment available and in use for stickies examination. The user must be judicious in the choice of the equipment used, however. For example, because of the very small amount of stickies collected from some specimens, use of a semi-micro balance capable of weighing to ±0.00001 g, rather than the usual analytical balance weighing to ±0.0001 g may be required to perform gravimetric quantitation (see 4.2.6).

6.7 *Microscope* (monocular- or stereomicroscope) or magnifying light stand (if desired).

6.8 *Image Analysis System* to measure number and size distribution, or both, of collected stickies particles (if desired).

[5] The device known as the Somerville Fractionator was first disclosed in 1932 (see Bibliography, Ref 2). It is described in TAPPI UM 242, and is available from vendors of paper testing equipment. The sole source of supply of the device known as the Pulmac MasterScreen known to the committee at this time is Pulmac Instruments International, Inc., Box 50 HCR 34, Montpelier, VT 05602. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at meeting of the responsible technical committee, which you may attend.

7. Sampling

7.1 *Sampling*:

7.1.1 The exact sampling plan followed will depend upon the material being evaluated, and must be agreed upon in advance by parties involved in the use of this practice. The following are recommended:

7.1.1.1 When sampling a lot of pulp in roll, bale, or similar form, follow TAPPI T 210, Section 8.

7.1.1.2 When sampling a lot of paper or paper product at anytime following the papermaking process, follow Practice D 585.

7.1.1.3 When sampling for other than acceptance purposes, as an alternative, use Practice E 122.

7.2 *Sample Preparation*:

7.2.1 Determination of the percentage solids present in the material to be evaluated using an appropriate standardized procedure such as TAPPI T 210 or TAPPI T 412.

7.3 *Preparation of Fiber Slurry*:

7.3.1 The exact procedure for preparation of the fiber slurry will vary based upon the fiber source and the separation system used. Typically, 25 to 50 g of dry fiber weight are used when the 0.10 mm (0.004 in.) slotted screen is used, and 100 g when the 0.15 mm (0.006 in.) slotted screen is used.

7.3.2 Fibers are disintegrated, when required or agreed, using a standard disintegration procedure such as TAPPI T 205.

7.3.2.1 Disintegration is particularly used to separate individual fibers from fiber clumps of bundles that would be retained on the screen with the stickies.

7.3.2.2 It is always advisable to disintegrate pulp received in sheet form, regardless of the solids content.

7.3.3 In some cases where the sample is received in pulp slurry form, it is simply diluted without further disintegration and processed as described in 7.3.4

7.3.4 After disintegration, if required, the pulp slurry is diluted to the optimum solids content recommended for use with the separation system used, such that the total dilute slurry volume results in a dry fiber content in the range specified in 7.3.1. Dilution is typically to a fiber solids content (dry fiber weight basis) of 0.5 to 1.0 % (50 to 100 g of dry fiber in 10 L of water).

7.4 *Separation of Stickies Material*:

7.4.1 Stickies material may be separated from pulp fibers using a variety of commercially available instruments (6.2).

7.4.1.1 It must be understood that the stickies material separated from specific pulps will vary widely in composition, and may include numerous materials not fitting the definition of stickies found in 3.2.1, such as fiber strings and bundles (Note 3), plastic, wood, shives, metal, and other materials.

7.4.1.2 Only after thorough examination of the contaminant fraction using appropriate techniques and the equipment in 6.4 through 6.8 may an exact estimation of the actual amounts and types of stickies materials present in a sample be made.

7.4.2 The Pulmac MasterScreen is an automated, high flow rate, low consistency screening device which creates and maintains a rigidly controlled screening environment whereby stickies are separated and collected from a pulp slurry. Typical slurry concentration is 100 g dry fiber in 8 to 10 L of water. The screen most frequently used for stickies collection is one having a slot width of 0.15 mm (0.006 in.). Other concentrations of pulp or screen slot width may be used upon the agreement of the persons using this practice, and must be shown in the report of the testing done.

7.4.2.1 The instrument automatically screens and then collects contaminant materials from a pulp slurry.

7.4.2.2 The contaminant materials are automatically deposited on a filter paper and may be removed from the instrument at the completion of the analysis for examination using the general techniques described in 6.6 through 6.8.

7.4.2.3 After making certain the instrument (particularly the slotted plate) is clean, separate the contaminant materials from a pulp slurry prepared as in 7.3.4 using the instructions accompanying the instrument.

7.4.3 The instrument known as the Somerville Fractionator was originally proposed by Somerville in 1932, and is described in some detail in TAPPI UM 242.

7.4.3.1 Make certain that the instrument is clean and equip it with the 0.15 mm (0.006 in.) screen plate unless a different screen plate has been agreed to in advance.

7.4.3.2 Using a dilute pulp slurry prepared as in 7.3.4, and containing about 50 g of dry fiber in 10 L of water, pour about 5 L of the slurry into the Somerville Fractionator and process the slurry using the manufacturer's instructions until the filtrate is clear.

7.4.3.3 Remove the screen plate from the Somerville instrument and wash both of its sides using a jet of water.

7.4.3.4 Collect the washings and transfer the solids in them to one or more pieces of filter paper using a Buchner funnel. The number of pieces of filter paper used will depend upon the amount of stickies present. Use sufficient pieces of filter paper to keep the stickies particles sufficiently separated for easy examination.

7.4.3.5 When filtration of the washings is complete, visually examine the screen, removing with tweezers any remaining stickies particles seen and adding them to the particles on the filter paper.

7.4.4 For samples having very high or very low levels of stickies, a reduction or increase in the amount of dilute slurry processed, its concentration, or both may be required to provide the level of stickies required for the examination that will be done in Section 8.

8. Examination of the Collected Stickies

8.1 Visual examination is frequently the first technique used to evaluate stickies.

8.1.1 Stickies may be examined on the filter paper sheet on which they are collected.

8.1.1.1 Techniques to enhance visualization, as agreed by users of the practice, such as spraying the filter paper to color it, for example, black, and then coating the stickies with a dry white powder, my be used.

8.1.2 Stickies may be examined after transfer to a sheet of paper of contrasting color such as orange or green.

8.1.2.1 Transfer of the stickies to a sheet of paper of contrasting color is generally done by placing the (dry) paper of contrasting color in contact with the surface of the (wet) filter paper upon which the stickies rest and passing this "sandwich" through a suitable nip press. Separate the filter

paper from the colored paper upon completion of pressing. The stickies will remain on the colored paper.

8.1.3 Stickies may be examined after transfer to a (transparent) sheet of polyester film.

8.1.3.1 One convenient way to transfer the stickies to a sheet of polyester film is by placing a polyester film sheet on the surface of the (wet) filter paper containing the stickies. Lay this "sandwich", polyester film sheet up, on a flat surface. Pass a TFE–fluorocarbon coated surface having a surface temperature of approximately 235° F over the polyester film sheet 2 or 3 times exerting minimal pressure on the "sandwich." A TFE-fluorocarbon coated home iron adjusted to low heat and used at a speed typical of that used in ironing large flat fabrics while exerting no pressure other than that of the iron itself, is generally suitable.

8.1.3.2 Immediately after passing the 235°F TFE-fluorocarbon coated surface over the "sandwich" 2 or 3 times, separate the filter paper from the polyester film sheet. The stickies will remain on the polyester film sheet. Place a second polyester film sheet over the transferred stickies materials to protect the stickies from becoming dislodged or contaminated.

8.2 The color of the separated stickies, other physical characteristics such as stickiness or ability to be stretched, or other visually observable parameters, may be determined using material prepared as in 8.1.1, 8.1.2, or 8.1.3, however the type of further testing which can be done may be dependent upon which of the procedures in 8.1.1 through 8.1.3 was used.

8.2.1 In many cases, the first task undertaken is to separate from the stickies material any non-stickies particles such as wood, shives, metal, or others present. This is done under magnification, using either a magnifying light stand or a low power stereo microscope, frequently as part of 8.1, and before transfer of the stickies to colored paper or polyester film, although non-stickies components can also be separated from the stickies after transfer.

8.3 Quantification may be based on the mass of stickies collected.

8.3.1 Stickies may be collected on filter paper which has been previously dried at 105°C. After redrying the filter paper with the stickies present, the difference in weight may be taken as a measure of the stickies present. If there are significant non-stickies materials present in the material on the filter paper, they must be removed before determination of the dry stickies weight. In the case where the collected stickies contain materials that are volatile at 105°C, the gravimetric estimation of stickies will be too low. The balance used must be suitable for the level of stickies being weighed. (See Note 5)

8.4 Quantification may be based on the number, the area, or both, of the stickies collected.

8.4.1 The number of stickies (number of stickies/gram of dry pulp) can be done visually by simply counting the stickies in the collected stickies fraction while using a stereomicroscope or magnifying light stand. Non-stickies particles (wood, metal, etc.) are not counted. This determination may be done as part of 8.1.

8.4.2 The area of stickies present may be determined using the Dirt Estimation Chart and the techniques described in Test Method D 2019. The area of non-stickies particles is not included in the material measured. This determination may be done as part of 8.1.

8.4.3 The number, size, and size distribution of the stickies present may be done using an image analyser after transferring the stickies on to polyester film (8.1.3.1). When image analysis is done, prior removal of non-stickies material (8.2.1) is of great importance, because the image analyser will not discriminate between stickie and non-stickies.

8.5 *Spectroscopic or Chemical Testing*:

8.5.1 Spectroscopic testing may be done on individual stickies particles removed from the filter paper using tweezers or a dissecting needle.

8.5.1.1 The exact spectroscopic measurement technique used depends upon the information required.

8.5.1.2 For example, infrared spectroscopy would be suitable for differentiation of particles of hydrocarbon wax and synthetic adhesives in collected stickies.

8.5.2 Chemical testing such as spot tests or extraction with a solvent may be done on individual stickies particles or the entire stickies fraction collected.

9. Report

9.1 Report the following information:

9.1.1 The identification of the material tested,

9.1.2 The stickies separation technique used (7.4.1), including the instrument or device used for the separation and the characteristics of the separating plate or screen including:

9.1.2.1 The dimension of the slots in the plate or screen,

9.1.2.2 The number of slots in the plate or screen, and

9.1.2.3 The effective dimensions of the screen or plate itself; that is the area of plate or screen that is available to come into contact with stickies.

9.1.3 The concentration and volume of slurry from which the stickies fraction was separated (7.4.2, 7.4.3.1, or 7.4.4), and

9.1.4 The specific results of the stickies examination done and the testing procedure used (Section 8).

10. Keywords

10.1 paper; paper products; pulp; recovered paper materials; recycled paper; stickies

Bibliography

(**1**) Brittner, Mark J., "Stickies Collection and Quantification within a Production Environment", 1994 TAPPI Pulping Conference.

(**2**) Somerville, J. L., Proc. Tech. Section Papermaker's Assoc. 13:37 (1932).