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An American National Standard

Standard Test Method for Surface Wettability and Absorbency of Sheeted Materials Using an Automated Contact Angle Tester [1]

This standard is issued under the fixed designation D5725; 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.

INTRODUCTION

The property of a liquid to adhere to, or "wet", a sheeted surface, or to be absorbed by that surface, or both, is important in many aspects of paper manufacturing and converting, as well as in the end-use applications of many converted paper products.

Examples include, but are not limited to, the absorption of water or other liquid by an absorbent structure (such as an absorbent tissue or wipe); the adhesion of an ink to a polymer film or a coated or uncoated paper (such as a packaging or wrapping material); the adherence of a polymer film or sizing material to a paper substrate in a laminate or coated structure; the adhesion of a pressure sensitive tape to a release paper; the adhesion of a film to a paper substrate in a composite structure (such as a diaper or other composite structure); and the non-wetting or non-absorbency, or both, of a barrier paper.

The wetting or sorptive behavior between a liquid and a particular sheeted substrate is dependent, at least in part, upon the relationship of the surface energy (tension) of the liquid and the surface energy of the substrate. The theoretical relationship of these energies is complex, and the different mathematical models which have been proposed for adhesion, wettability, and sorption are beyond the scope of this test method, but may be found in standard texts in these areas. In many cases, however, the contact angle of the fluid which will be in contact with the substrate, or the contact angle of a liquid of known surface tension, when placed in contact with a substrate of interest, is used to understand or predict in-process or end-use results of a particular printing, adhesion, or sorptive application.

Contact angle measurements as described in Test Method D724 or Canadian Pulp and Paper Association CPPAF.3H have been used to study and define the printability relationship between an (aqueous) ink and a paper at the water/paper interface. TAPPI T552 and Test Method D2578 use a somewhat different, semi-quantitative approach to provide information regarding the energy relationship between a polymer film and a nonaqueous liquid, the test end-point being the place where the contact angle between a liquid of known surface tension and the test specimen approaches zero under the conditions of the test.

The procedure presented in this test method is a simple, completely automated approach to contact angle measurement applicable to a wide range of sheeted materials and liquids where interfacial contact angles range from near zero to near 180 degrees. The automated procedure shows increased precision and greater ease in use than manual procedures.

1. Scope

1.1 This test method measures the contact angle of a test liquid in contact with a flat specimen of a film or a paper substrate under specified test conditions. This test method may be used with any liquid of interest which is compatible with the equipment used, particularly with regard to liquid viscosity, tackiness, and vapor pressure (evaporation). This test method may be used with any substrate of interest, which can be cut to dimensions compatible with the equipment used.

1.2 For materials which sorb the test liquid under the specified test conditions, the rate of change of the contact angle as a function of time may be significant, and may be determined using procedures described in this test method. It is also possible to evaluate the sorptive properties of a surface, as the

[1] This test method is under the jurisdiction of ASTM Committee D06 on Paper and Paper Products and is the direct responsibility of Subcommittee D06.92 on Standard Documents Relating to Paper and Paper Products.

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remaining liquid volume on top of the specimen surface is measured as a function of time.

1.3 The conditions required in this test method specify reagent water as the test liquid when testing papers designed to be absorbent, such as absorbent tissue grades.

1.4 Conditions are specified for the testing of a wide range of papers considered to be of low absorbance or nonabsorbent, including release papers, sized, coated, or unsized papers designed for printing, writing, wrapping, and similar tasks where the paper surface interaction with aqueous or solvent based inks or other aqueous or nonaqueous liquids is important. In such cases, test liquids other than reagent water, including writing and printing inks, or organic liquids or mixtures of organic liquids may be used as the test liquid upon prior agreement of those involved in the testing, provided the liquid is compatible with the equipment used. Where test liquids other than reagent water are used, the actual liquid used is reported.

1.5 Conditions are also specified for the testing of polymer films, polymer-coated papers, paper laminates, felt, textiles and non-wovens, using water or other fluids compatible with the equipment and important to the end-use applications of the materials tested, including gluing and printing.

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

1.7 *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]

D 528 Test Method for Machine Direction of Paper and Paperboard

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

D 685 Practice for Conditioning Paper and Paper Products for Testing

D 724 Test Method for Surface Wettability of Paper (Angle-of-Contact Method)

D 1193 Specification for Reagent Water

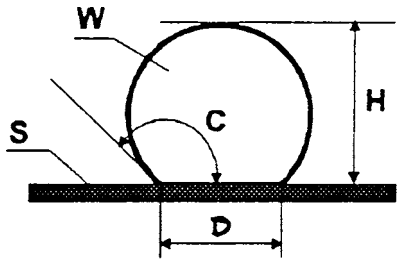
D 1968 Terminology Relating to Paper and Paper Products

D 2578 Test Method for Wetting Tension of Polyethylene and Polypropylene Films

D 5039 Test Methods for Identification of Wire Side of Paper

E 122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method



W—water droplet
S—specimen
D—diameter of the surface of contact of the droplet
H—height of droplet
C—contact angle

NOTE 1—For materials exhibiting sorptive properties with respect to the test liquid used, the values for contact angle, droplet diameter, and droplet height may vary as a function of time following drop deposition on the material substrate.

FIG. 1 Principle of Measurement

2.2 *TAPPI Standard:*

T 552 Determination of Wetting Tension of Polyolefin Films and Coated Surfaces via the Mayer Rod Technique[3]

3. Terminology

3.1 *Definitions:* For definitions used in this test method, refer to Terminology D 1968 or the *Dictionary of Paper*.[3]

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *contact time*, *n*—the length of time the droplet has been in contact with the specimen surface.

3.2.2 *droplet diameter*, *n*—diameter of the surface of contact between the specimen surface and the droplet, shown as distance "D" in Fig. 1.

3.2.3 *droplet height*, *n*—height of the droplet in contact with the specimen surface, shown as distance "H" in Fig. 1.

3.2.4 *drop motion time*, *n*—the time it takes for the droplet to reach the specimen surface after the drop application has been triggered.

4. Summary of Test Method

4.1 A drop of a specified volume of water or another agreed upon test liquid is applied to a test specimen surface using a liquid delivery system and specified deposition parameters. Images of the drop in contact with the substrate are captured by a video camera at specified time intervals following deposition.

4.2 At a specified time after drop deposition, which is varied based upon the sorptive or barrier properties of the substrate/liquid interface, the test is terminated. The contact angle between the drop and the substrate at various time intervals following drop deposition are determined by image analysis techniques on the captured images, and the contact angle at specified time(s), the rate of change of the contact angle change as a function of time, and changes in droplet height and diameter, as well as other test variables are analyzed, based on specific information requirements for the materials being tested.

[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.

4.3 The test method is divided into two parts, Procedures A and B, which vary only in certain procedural aspects and allow the use of the procedure over the wide range of sample types described in the Introduction and Section 1.

4.4 To identify the applicable procedure, a drop of the standardized size is formed at the tip of the liquid delivery system. The drop is then slowly lowered towards the specimen surface until contact is initiated between the liquid and the specimen. Use Procedure A if the drop releases immediately from the tip on contact with the specimen surface. Use Procedure B if the drop remains attached to the tip on contact with the specimen surface.

4.5 In order to measure the highest contact angle possible, the drop should be applied as gently as possible. With Procedure A, the drop may be applied with a very short stroke, as the drop will release from the liquid delivery system tip immediately upon contact with the specimen surface. Therefore, Procedure A should be tried as the first option.

4.6 Procedure A gives specific conditions for the testing of sheeted materials having contact angles with water less than about 100°. Materials of this type are generally sorbent papers.

4.7 Procedure B gives specific conditions for testing of sheeted materials having contact angles with water above about 100°. Procedure B is applicable when the drop is not immediately released from the liquid delivery system tip upon contact with the specimen surface.

4.8 In cases where a liquid other than water is used, the specific procedure applied will depend on the contact angle between the liquid and the specimen substrate. For example, where the film side of a paper-film laminate, or a polymer film itself, is tested with a liquid whose surface tension is approximately equal to or below that of the film, the contact angle at the liquid/substrate will approach zero, and Procedure A would be used. If the same film were tested with water as the liquid, Procedure B might be appropriate. The procedure is chosen based on the resulting interfacial wetting properties, not the identity of the liquid or specimen substrate.

5. Significance and Use

5.1 Contact angle measurements may be used to study the relative sorbtive rates of uncoated sorbent papers, or the relative printing or writing characteristics of coated or sized printing and writing papers.

5.2 The complex interaction between a liquid and a surface may be looked upon as a combination of three different processes of wetting, absorption, and adsorption. Wetting is best explained with a drop of water on a plastic film. The liquid volume remains the same, the drop base diameter will increase, and the contact angle will decrease as a function of time. When the liquid volume is reduced as a function of time, the base diameter of the drop is studied. When this diameter remains constant, the absorption is dominating. When the drop is spreading across the surface (increasing base diameter), the interaction is based on adsorption.

5.3 For sized papers, an increase in feathering is likely as the rate of change in the contact angle with time increases, indicating a relative increased degree of liquid transport or penetration (absorption) into the paper.

5.4 For sorbent papers, the change in contact angle with time may be very rapid, with those papers showing the greater relative change per unit time having the fastest rate of sorption.

5.5 For hard sized papers, little change in contact angle with time may be seen, and for laminates or polymer coated and barrier papers, release papers, or other similar specialty grades, there may be no change in contact angle over the time interval of a typical test.

5.6 It is generally found that papers having contact angles with water-based inks in the range 90 to 110° work best in printing and writing applications. Feathering may be expected for contact angles less than 90°. Breaks in the flow of ink onto the paper may occur for contact angles greater than 110°.

5.7 Because of the wide range of paper coating possibilities and ink compositions, further generalizations are difficult. However, contact angle is a precise empirical tool for use in studying specific liquid/substrate combinations for product and process improvements.

5.8 In addition, contact angle measurements on films are used to determine printing and gluing characteristics of films with specific printing inks or adhesives. In such applications, the procedure may use a constant film substrate with various test liquids of significance to a specific end-use application. By measuring substrate surface free energy and then monitoring and controlling any surface treatment of the material using contact angle measurements, improved end-use performance in gluing or printing applications is possible.

6. Apparatus

6.1 *Automated Contact Angle Tester*, consisting of the following components, each of which are described in detail: a light source, a video camera, a specimen stage, a liquid delivery system consisting of a pump and micro-syringe and a computer and associated software suitable for video image capture, image analysis, and reporting.

6.1.1 *Light Source*:

6.1.1.1 *Halogen Lamp*, sealed in a separate lamp housing with its own ventilating fan. Room temperature air is circulated inside the lamp housing and the warm air is then returned outside the instrument so it cannot reach the test specimen or the test liquid.

6.1.1.2 Other designs are possible, using heat dissipating filters or similar equipment to eliminate heating of the specimen or test liquid.

6.1.2 *Video Camera*:

6.1.2.1 The video camera is equipped with a lens to achieve an image view of about 10 by 7.5 mm and an electronic shutter. The shutter is set for a 1-ms exposure time for purposes of this test method.

6.1.2.2 The video camera will, depending on the video standard used, send out video images continuously at a rate of 50 (CCIR) or 60 (EIA) images per second. Hence, the time between two consecutive images is 20 ms (CCIR) or 16.7 ms (EIA). Either of these video standards may be used. The CCIR timing has, however, been used throughout this description in the timing examples.

6.1.2.3 When a droplet of a different size than standard is used, other magnifications may be needed.