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Surface wettability and absorbency of sheeted materials using an automated contact angle tester

1. Introduction

1.1 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.

1.2 This test method is an 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°.

2. Scope

2.1 This test method measures the contact angle of a test liquid in contact with 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.

2.2 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 here after. It is also possible to evaluate the sorptive properties of a surface, as the remaining liquid volume on top of the specimen surface is measured as a function of time.

2.3 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.

Approved by the Standard Specific Interest Group for this Test Method
TAPPI



Documents

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3. Applicable documents

- TAPPI T 400 "Sampling and Accepting a Single Lot of Paper, Paperboard, Containerboard, or Related Product";
- TAPPI T 402 "Standard Conditioning and Testing Atmospheres for Paper, Board, Pulp Handsheets, and Related Products,"
- TAPPI T 409 "Machine Direction of Paper and Paperboard;"
- TAPPI T 455 "Identification of Wire Side of Paper;"
- TAPPI T 552 "Determination of Wetting Tension of Polyolefin Films and Coated Surfaces via the Mayer Rod Technique;"
- TAPPI T 698 "Determination of Wetting Tension of Polyethylene and Polypropylene Films and Coatings;"
- TAPPI T 458 "Surface Wettability of Paper;"
- TAPPI T 1200 "Interlaboratory Evaluation of Test Methods to Determine TAPPI Repeatability and Reproducibility."
- ASTM D 1193 "Specification for Reagent Water;"
- CPPA F.3H "Surface Wettability of Paper and Paperboard;"
- ASTM E 122 "Practice for Choice of Sample Size to Estimate a Measure of Quality for a Lot or Process."

4. Definitions

4.1 *Contact angle*, the angle formed by the substrate and the tangent to the surface of the liquid drop in contact with the substrate, shown as "C" in Fig. 1.

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

4.3 *Droplet height*, height of the droplet in contact with the specimen surface, shown as distance "H" in Fig. 1.

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

4.5 *Contact time*, the length of time the droplet has been in contact with the specimen surface.

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.

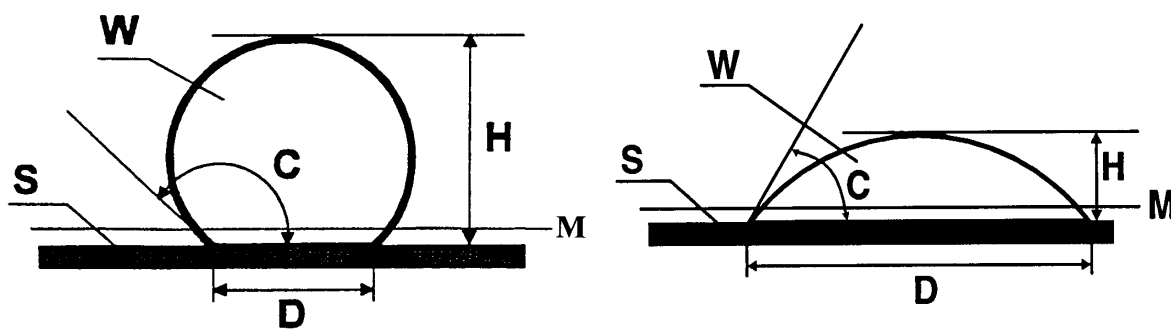


Fig. 1. Principle of measurement: W: water droplet; S: specimen; H: height of droplet; C: contact angle; D: diameter of the surface of contact of the droplet; M: minimum height of drop to be analyzed.

5. Summary

5.1 A drop of a specified volume of water or another agreed test liquid is automatically 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.

5.2 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 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.

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

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

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

5.6 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.

5.7 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 on contact with the specimen surface.

5.8 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.

6. Significance

6.1 Contact angle measurements can be used to study the relative sorptive rates of uncoated sorbent papers, or to study the relative printing or writing characteristics of coated or sized printing and writing papers.

6.2 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.

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

6.4 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.

6.5 It is generally found that papers having contact angles with water-based inks in the range 90 to 110° work best (1). 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°.

6.6 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.

6.7 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 different 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.

6.8 The complex interaction between a liquid and a surface can be looked upon as a combination of three different processes; wetting, absorption and adsorption. Wetting is best explained with a drop of water on a plastic film. Here 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.

7. Apparatus¹

7.1 An automated contact angle tester is required to perform the testing described in the procedure and consists of the following components, each of which are described in detail below: 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.

7.1.1 The *light source* is described as follows:

7.1.1.1 A halogen lamp is 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.

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

7.1.2 The *video camera* is described as follows:

7.1.2.1 The video camera is equipped with a lens to achieve an image view of about 10 x 7.5 mm.

7.1.2.2 The video camera is equipped with an electronic shutter. The shutter is set for a one millisecond exposure time for the purpose of this method.

7.1.2.3 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.

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

7.1.2.5 The depth of the focal plane must be sufficient. If this is not properly arranged, the base of the droplet will be influenced by the forward edge of the specimen.

7.1.3 The *specimen stage* is described as follows:

7.1.3.1 The specimen must be positioned so the test surface appears flat and horizontal to the video camera.

7.1.3.2 To avoid the influence of capillary forces, the specimen must be freely suspended across the wetted test area.

7.1.3.3 When the specimen is moved to a new area, the previous drop's wetted area must be avoided.

7.1.4 The *liquid delivery system* is described as follows:

7.1.4.1 The pump drives a micro-syringe. By moving the plunger forward, a droplet containing $4.0 \pm 0.1 \mu\text{L}$ is delivered at the tip of a PTFE tube with an inner diameter of $0.50 \pm 0.05 \text{ mm}$, and an outer diameter of $0.70 \pm 0.05 \text{ mm}$.

7.1.4.2 When a droplet of different volume is requested, it is possible to use a drop size of $0.2\text{--}20 \pm 0.1 \mu\text{L}$.

7.1.4.3 For certain liquids and/or drop sizes a PTFE tube with different dimensions may be used.

7.1.4.4 The drop size, tubing material and tubing dimensions stated in 7.1.4.1 are standard for this test method, and any deviations from the conditions stated therein must be included in the report.

7.1.5 The *drop applicator* is described as follows:

7.1.5.1 The purpose of the drop applicator is to apply the droplet onto the specimen surface with a down-going motion ("stroke"). The length of this stroke should be as short as possible in order to minimize the force exerted to the droplet. Depending on the wetting properties between the liquid and the specimen surface, there are two different procedures, A and B, for the application of the drop. Depending on stroke length and acceleration, there are some timing considerations.

NOTE 2: Stroke, droplet velocity and acceleration are important to the application of the drop. The correct approach is to capture the image of the highest contact angle as early as possible.

7.1.5.2 To achieve the requested timing, the drop applicator is activated (triggered) by the sync pulse from the

¹ Names of suppliers of testing equipment and materials for this method may be found on the Test Equipment Suppliers list, available as part of the CD or printed set of Standards, or on the TAPPI website general Standards page.