Viscosity of starch and starch products (C.I.V. test)

1. Scope and summary

This method is for measuring the viscosity of starch solutions or suspensions by means of the Corn Industries viscometer (C.I.V. test). The viscosity is recorded automatically on a graph which gives viscosity characteristics during any desired period of cooking and cooling. The method is applicable to unmodified and modified starches and starch derivatives from cereal grains and tubers. For measuring the viscosity of starch solutions or suspensions by means of the Visco-Amylo-Graph method, see TAPPI T 676 “Viscosity of Starch and Starch Products (Visco/Amylo/Graph Test).”

2. Apparatus

2.1 Corn Industries viscometer\(^1\), with water bath, as shown in Fig. 1. The water bath in which the starch is cooked is electrically heated and the temperature automatically controlled by a variable thermoregulator so that any preset temperature can be maintained within limits. The dynamometer, which is of the pendulum type, is located in the recorder case. Supplementary weights are provided so that the recorder chart at full scale has four ranges of viscosity units: 225 (standard weight on instrument, no additional weight required), 450, 900, and 1800 g-cm, respectively. Weights corresponding to each scale can be readily added or removed without change in the calibration of the viscometer. This permits testing a variety of starches over a wide range of concentrations. One advantage of this is that starches of widely different viscosities can often be tested at the same concentration so that a direct comparison can be made of their properties.

2.2 Container, 1-L capacity, such as an enameled graduate with handle.

2.3 Forced circulation oven having uniform heating distribution, maintained at 105 ± 3ºC.

2.4 Balance accurate to 0.001 g.

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\(^1\)Names of suppliers of testing equipment and materials for this method may be found on the Test Equipment Suppliers list in the bound set of TAPPI Test Methods, or may be available from the TAPPI Technical Operations Department.
2.5 Moisture dishes, preferably aluminum, approximately 5 cm (2 in.) in diameter and equipped with covers.

3. Calibration

Periodically calibrate the instrument, using the procedure below, with samples from one or more lots of pretested starch materials which have been set aside for this purpose.

4. Test specimen

4.1 In accordance with TAPPI T 657 “Sampling of Fillers and Pigments,” obtain representative samples of shipment weighing at least 750 g for a duplicate test. Place each sample in an air-tight container as it is taken, and keep the container sealed until ready for test.

4.2 Weigh to the nearest 0.1 g a specimen intermediate between those required in Table 1 for the particular type of starch being tested (see also Note 1).
5. Procedure

5.1 Transfer the test specimen to a preweighed 1-L container and add sufficient distilled water at room temperature to make a total net weight of 1000 g.

5.2 Place the viscometer cup in the water bath preheated to 50° ± 0.5°C. Insert the stirring device, swing the viscometer head in place over the cup, and attach the stirring mechanism to the shaft in the viscometer head. Start the stirrer motor. Before pouring the starch-water mixture into the viscometer cup, stir the slurry briefly with a stirring rod to ensure a uniform suspension. Start the chart-drive motor of the recorder as soon as the mixture is poured into the viscometer cup. Set the thermoregulator to 92° ± 0.5°C. Cover the cup with the metal top to prevent cooling and evaporation.

5.3 The viscosity characteristics of the starch are given by a continuous graph from which the following information can be read:

5.3.1 Time required for the initial viscosity rise and the temperature at this point.

5.3.2 Viscosity range: the time and temperature change from the initial viscosity rise to the maximum viscosity.

5.3.3 Maximum viscosity and viscosity at the end of 30 min.

5.3.4 Viscosity at all times during any desired period of cooking, thus showing the effect of heat and shear.

5.4 Viscosities are given directly on the recorder chart in gram-centimeters of torque.

5.5 Moisture

5.5.1 Grind down any hard granular pellets, taking precautions to prevent significant loss of moisture.

5.5.2 Weigh to the nearest 0.001 g a 5 g specimen into a predried, cooled, and tared moisture dish. Place the dish and cover (removed) in the forced air oven, 105 ± 3°C, for 3 hours. Cover the dish, cool in a desiccator about 30 min and weigh.

5.5.3 Calculate and report the moisture as a percentage of the original (undried) sample to the nearest 0.1%.

NOTE 1: If necessary, adjust the concentration of the specimen to obtain a viscosity value at the end of the test, within the range of 50 to 150 g-cm of torque.

NOTE 2: For the effect of cooling on the viscosity of the paste, turn off the heater for the water bath after any desired period of cooking and continue to cool in a constant temperature bath at about 10°C.

Table 1. Starch data

<table>
<thead>
<tr>
<th>Kind of starch</th>
<th>Concentration of starch*, g moisture-free starch/1000 suspension</th>
<th>Dynamometer scale</th>
<th>Approximate maximum viscosity to be expected</th>
</tr>
</thead>
<tbody>
<tr>
<td>Corn, unmodified</td>
<td>55</td>
<td>225</td>
<td>220</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>1800</td>
<td>1700</td>
</tr>
<tr>
<td>Corn, moderately thin-boiling</td>
<td>60</td>
<td>225</td>
<td>35 to 150</td>
</tr>
<tr>
<td></td>
<td>110</td>
<td>1800</td>
<td>1200 to 1600</td>
</tr>
<tr>
<td>Corn, thin-boiling</td>
<td>70</td>
<td>225</td>
<td>35 to 80</td>
</tr>
<tr>
<td></td>
<td>140</td>
<td>1800</td>
<td>1600 to 1800</td>
</tr>
<tr>
<td>Corn, very thin-boiling</td>
<td>180</td>
<td>225</td>
<td>50 to 150</td>
</tr>
<tr>
<td></td>
<td>815</td>
<td>1800</td>
<td>1200 to 1800</td>
</tr>
<tr>
<td>Wheat, unmodified</td>
<td>50</td>
<td>225</td>
<td>50</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>1800</td>
<td>1700</td>
</tr>
<tr>
<td>Tapioca, unmodified</td>
<td>40</td>
<td>225</td>
<td>175</td>
</tr>
<tr>
<td></td>
<td>65</td>
<td>1800</td>
<td>1500</td>
</tr>
<tr>
<td>Potato, unmodified</td>
<td>40</td>
<td>225</td>
<td>175</td>
</tr>
<tr>
<td></td>
<td>65</td>
<td>1800</td>
<td>1500</td>
</tr>
</tbody>
</table>

*These concentrations are approximations of the extreme limits of representative starches which can be tested in the Corn Industries viscometer. All intermediate concentrations can also be tested.

6. Report

6.1 Report the maximum viscosity and the viscosity at the end of 30 min, in gram-centimeters of torque at 92°C, and state the starch concentration used. Other data may be included as desired.
7. Precision

7.1 The precision of this method has not been investigated (see 8.3).

8. Keywords

Viscosity, Starch, Dispersion, Solutions

9. Additional information


9.2 The viscosity of a liquid system is its resistance to flow. It may be regarded as fluid or internal friction. While starch solutions are often spoken of as having viscosity, they do not have a true (Newtonian) viscosity of a perfect fluid. The so-called viscosity of a starch solution is the combined effect of a number of inherent properties which cause it to deviate from Newtonian viscosity. These effects occur from the residual granule particles or from new colloidal groupings that take place as a result of cooking the starch. Many starches possess a varying degree of thixotropy, which is the property of becoming thinner with agitation or when under shear. In a starch paste, the observed viscosity depends upon the rate of shearing to which it is subjected in the viscometer; in a thixotropic paste, the greater the rate of shearing, the less the viscosity will be.

9.3 Viscosity data obtained for any starch depend upon the arbitrary selection of conditions of preparing the paste. Some types of starches reach a relatively stable viscosity, while others tend to decrease in viscosity on continued heating and agitation, eventually reaching a value near that of water. The time required for these changes to occur varies with different types of starches, and the same cycle of variation is evident even when the same starch is prepared in different concentrations. Also, some starches have a much greater tendency than others to form organized structures or gels on cooling, which increases the viscosity. Variables such as rate of heating, final maximum temperature, and rate and kind of stirring cause different observed viscosities. The method of preparing the specimen is very important and must be strictly observed if reproducible data are to be obtained, even with the same type of viscometer. Repeatability can be demonstrated by checking the viscosity of the same specimen in duplicate or triplicate as desired on the same instrument; to show reproducibility between instruments, determine the viscosity of the same specimen on another instrument. Results can be determined to a repeating accuracy of ± 1%.

9.4 This method, formerly T 637 su-70, has been reaffirmed as classical in 1997 by committee action.

Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Technical Operations Manager.