

is also regenerated in the process without additional cost. This means a saving of about four pounds of sulfuric acid for every pound of chromic anhydride equivalent regenerated. With sulfuric acid at \$14 per ton, this would amount to about three cents for every pound of chromic acid regenerated. Accordingly the continuous process thus developed for the electrolytic regeneration of chromic acid from waste liquor gives promise of valuable technical applications.¹

SUMMARY

Results obtained in the foregoing paper on developing a continuous process for electrolytic regeneration of chromic acid from waste liquor may be briefly summarized as follows:

I—It has been found possible simply by the action of the electric current to destroy the organic matter in waste chromium liquor without subjecting it to any preliminary treatment.

II—Conditions have been worked out, under which the process of regeneration can be conducted successfully as a continuous operation, *i. e.*, the fresh waste liquor is fed in at one part, and the finished solution drawn out at the other part of the cell without interruption. Careful adjustment of conditions and arrangements give good results: current efficiency fair, energy consumption low, chromic acid content of the finished solution up to commercial standard, and, most important of all, sulfuric acid concentration in both chambers practically constant.

It has been found that for every pound of chromic acid regenerated only about 3 k. w. h. of electric energy are required. Furthermore the saving of sulfuric acid, which is also regenerated in the process without additional cost, will amount to nearly as much as the cost of electric energy required (at one cent per k. w. h.) for the regeneration of the chromic acid.

DETERMINATION OF THE WATER RESISTANCE OF FABRICS^{2,3}

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The waterproofing of canvas to be used as coverings for outdoor use has long been practiced, but the World War has naturally increased its importance not only for protective purposes but also as a means of prolonging the usefulness of canvas coverings and thus decreasing the expense of using them. In developing simple and effectual formulas and treatments for waterproofing canvas, both for small scale application with a brush or as a spray and for application in large treating plants, and for testing deliveries of commercially waterproofed fabrics, it has been found necessary to develop practical and simple laboratory tests by which the effectiveness and probable durability of the waterproofing treatment may be judged.

¹ U. S. Patent Application 321,609.

² Acknowledgment is made to H. P. Holman and B. S. Levine, of the Leather and Paper Laboratory, Bureau of Chemistry, U. S. Department of Agriculture, for valuable assistance in developing the methods and equipment.

³ Read at the 57th Meeting of the American Chemical Society, Buffalo, N. Y., April 7 to 11, 1919.

A number of methods have been proposed for testing the water resistance of fabrics. All of them, however, have been applied to the new material without any effort even to remove the first purely transitory resistance, and, furthermore, the literature on the subject does not reveal comparative studies of the value of the several tests. The results obtained by the different methods have not been satisfactorily standardized, nor have the results of laboratory tests apparently been coordinated with the behavior of fabrics exposed to actual weathering or service tests.

Gawalowski¹ describes an apparatus for testing the water resistance of fabrics by attaching a piece of cloth to an open end of a graduated tube such as a burette and filling it with a column of water 12 in. in height. The water dripping through is collected in a graduated measuring glass. He tested a large number of fabrics and found that many allowed from 1 to 6 cc. of water to pass through in 5 hrs., while others required 16 to 23 hrs. to collect this quantity. Other samples allowed sufficient water to pass in 5 hrs. to fill the collecting cylinder.

Dannerth² describes a method for testing the water resistance by stretching and securely fastening a 2½ in. square of the fabric across the mouth of an ordinary thistle tube. The tube is filled with distilled water at 20° C. and the amounts of water that pass through in 5 hrs. and 10 hrs., respectively, are measured. A fabric which allows no water to pass through in 10 hrs. is considered first class. A high grade cravenette allowed all the water to pass through in 5 hrs. Drops of water were visible on the outside of the sample 15 min. after starting the test. With a medium quality "raincloth" the outer surface of the fabric had become damp after 5 hrs. After 10 hrs. half of the water had passed through. With an umbrella cloth no water passed through in 10 hrs.

Heermann³ described several methods:

BAG TEST—A square of the fabric, 50 × 50 cm., or 100 × 100 cm., is tied with strings by the four corners to a frame in such a way that a bag is formed. The bag is filled to a given height with water at the temperature of the room. The height of the column of water used varies, depending on the uses to which the fabric is to be put. No dropping or trickling through of water should take place in 24 hrs., but sweating through or transudation is permitted.

Uniform cloth, tent cloth, fabric for knapsacks and bread bags were tested by this method, using pieces 50 cm. square, filled with water to a depth of 75 mm. After 24 hrs. the water may sweat through but should not drip through. The specifications for wagon covers for the Prussian State Railways prescribed that a piece 100 cm. square should be used and that the depth of water should be 10 cm. After 24 hrs. there should be no dripping. Heermann considers one test as usually sufficient but in certain circumstances the same piece is dried and tested for a second or third time, in order to determine how the fabric stands wear.

¹ *Leipziger Monatschrift*, 1893, 221; also "Textile Fibres," Matthews, p. 573, John Wiley & Sons, 1913; and "Technical Testing of Yarns and Textile Fabrics," Herzfeld, p. 155, Scott, Greenwood & Son, 1902.

² *Textile World Record*, 34 (1908), 630; "Methods of Textile Chemistry," Dannerth, p. 53, John Wiley & Sons, Inc., 1908.

³ *Mechanisch- und Physikalisch-Technische Textiluntersuchungen*, Berlin, 1912, 232-239.

BURETTE TEST—This test is similar to Dannerth's described above.

FUNNEL TEST—A piece of the cloth to be tested is folded like a paper filter, placed in a glass funnel, and 300 cc. of water are poured into the cloth filter thus formed. Waterproof fabrics should not wet through in 24 hrs.; the outer side should show only regularly spaced drops.¹

WATER PRESSURE TEST—The pressure at which the water begins to penetrate the fabric is determined. The time in which water passes through the cloth at a constant pressure can also be determined with the apparatus. The apparatus used is fully described by Heermann.

SPRAY TEST—A piece of cloth 50 × 50 cm. is weighed after having been exposed to 65° relative moisture for several hours. It is then spread out smoothly on a frame and set up outdoors in a slanting position. A sprinkling apparatus connected with the water supply is set up at a distance of 6 to 10 meters from the cloth, the nozzle of which is arranged so that a fine spray strikes the goods perpendicularly and equally all over and falls from a height of 2 or 3 meters. The under surface is examined from time to time for penetration of water. If the water has not penetrated the spraying is continued. Whether, and at what time, water appears on the under side during the spraying is also observed. At the end of an hour the spraying is stopped, the material is hung up to dry for 5 min. and weighed. It is claimed that the smaller the amount of water absorbed the more efficient is the waterproofing preparation. Duplicate or triplicate tests are made.

Wosnessensky² describes an instrument for measuring the degree of impermeability of cloth based upon the number of hammer strokes before the water percolates through, the cloth being pressed upward by a column of water.

Villavecchia³ describes a spray test in which the fabric is inclined at an angle of 25° and water allowed to drip upon it for 3 hrs. from a height of 2 meters at a rate of 3 l. per min. on the central part of the fabric so as to cover an area of 3 cm. in circumference. At the end of the experiment no water, or at most a minimum quantity, should have penetrated the fabric.

LeRoy⁴ determines the degree of impermeability of fabrics to still water and imitation rain.

Clark⁵ stretches a piece of fabric over a wide-mouth bottle and allows a heavy artificial rain from a Gooch crucible attached by means of a cork and a tube to a faucet to fall upon it.

Clark also folds a piece of fabric into a pocket into which some heavy articles are placed and immerses as far as possible in water. A resistant fabric should not wet through in 24 hrs.

DEVELOPMENT OF METHOD

From a consideration of the numerous procedures heretofore proposed, it appeared that the funnel test and the spray test could be standardized to be simple in operation and at the same time furnish definite information as to the effectiveness of the waterproofing

¹ This is probably the test described by Matthews and Herzfeld (*Loc. cit.*) as the test prescribed in the official regulations of the German Clothing Department for testing sailcloth.

² *J. Soc. Dyers and Colourists*, **32** (1916), 246.

³ "Applied Analytical Chemistry," **2**, p. 521, P. Blakiston's Sons & Co., 1918.

⁴ *Ann. fals.*, **85** and **86** (1915), 377.

⁵ *Textile World J.*, **53** (1918), 43.

treatment of the fabric. The elimination of additional tests is much to be desired. The new features which have been introduced materially extend the information obtained and increase the reliability of the results as a basis for judging the resistance of the fabric in service.

The details of the methods as finally developed follow.

MODIFIED FUNNEL TEST—Cut a piece of the fabric one foot square, weigh, crumple thoroughly in the hand and place in an 800-cc. beaker and soak in distilled water at from 70° to 80° F. for 24 hrs., removing, straightening out and recrumpling 4 or 5 times during this period. Remove from the water, straighten out and dry in oven at 45° C. for 24 hrs. Hang in laboratory over night. Crumple, resoak in distilled water, and dry at 45° C. for 24 hrs. and hang in laboratory over night as before. Again crumple, smooth out and place on a piece of absorbent paper (paper toweling) of the same size and fold the two together into the form of a filter, insert in a 6-in. glass funnel having an angle of 60°, and place the funnel in a support¹ (Fig. 1) over a 500-cc. graduated glass cylinder and fill the funnel to a depth of exactly 4 in. with distilled water of 70°–80° F. This depth equals 500 cc. of water.

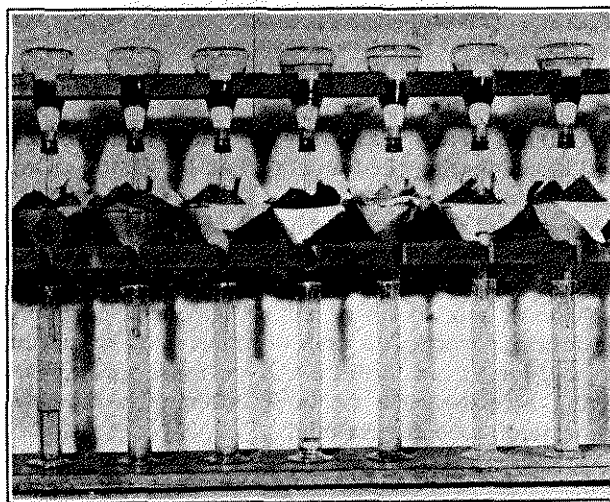


FIG. 1

Maintain a constant water level above the funnel by inverting an Erlenmeyer flask filled with water and closed with a rubber stopper through which passes a glass tube ground at the end to an angle of 45°.

Make the following observations:

- 1—The time elapsed before the paper begins to wet.
- 2—The time elapsed until the paper is entirely wet.
- 3—The time elapsed before the first drop passes into the cylinder.
- 4—The quantity of water in the cylinder in 1, 3, 6, and 24 hrs.
- 5—The time and extent to which the fabric becomes wet above the water level.

At the expiration of 24 hrs., if there has been no dripping, the funnel filled with water is lifted 2 in. and allowed to drop into its support; this is repeated

¹ This support is made of wood, holding ten funnels 6 in. in diameter.

four times and the amount of water that drips through in 3 hrs., if any, is recorded.

Remove the funnel from its support and carefully pour and drain off the water, and then remove the fabric and paper from the funnel, smooth out, and observe:

1—Whether the paper is dry, damp or wet.

2—Whether the fabric on the outside is dry, damp or wet, or whether the water has only sweated through.

The water resistance of fabrics as determined by this method is rated in accordance with the following scale:

Very High 10	The fabric does not become wet above the water level within 24 hrs. No water drips through. No sweating through is apparent except to a very limited extent at the folds. Filter paper under the fabric remains dry, except for slight wetting where the fabric is folded.
High 9	The fabric does not wet above the water level within 24 hrs. Sweating through is sufficiently rapid to cover generally, and especially in the fold, the outside of the fabric with droplets. Filter paper under the fabric becomes wet.
High-Medium 7 and 8	The water dripping through: In 6 hrs. is from 1 cc. to 5 cc. In 24 hrs. is from 1 cc. to 25 cc. In 3 hrs. after raising and allowing the funnel to drop into support 5 times.
Medium 5 and 6	The water dripping through: In 6 hrs. is from 5 to 25 cc. In 24 hrs. is from 25 to 50 cc.
Medium-Low 3 and 4	The water dripping through: In 6 hrs. is from 25 to 75 cc. In 24 hrs. is from 50 to 150 cc.
Low 1 and 2	The fabric wets above the water level readily. The water dripping through: In 6 hrs. is from 75 to 200 cc. In 24 hrs. is from 150 to 300 cc.
Negligible 0	The water dripping through in 24 hrs. exceeds 300 cc.

MODIFIED SPRAY METHOD—Dry the piece of fabric used in conducting the funnel test at 45° C. for 24 hrs., hang in laboratory over night and clamp loosely in a frame. Set the frame in a holder attached to a trough at an angle of 45°. The trough used held six frames (Fig. 2). Allow clear tap water at room temperature to fall from a height of 6 ft. upon the central portion of the fabric, covering an area of about 8 in. in circumference, for 24 hrs., from a 2 3/4 in. brass spray nozzle having 25 holes, each 1.9 (0.75 in.) mm. in diameter, at a rate of 1000 cc. per min.

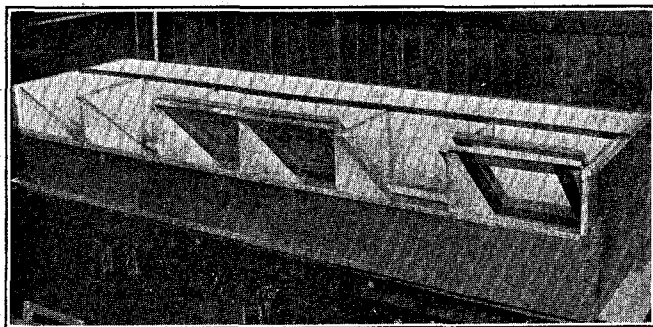


FIG. 2

Inspect the condition of the under side of the fabric at the end of 5 min., 1/2 hr., 1 hr., 3 hrs., 7 hrs., and 24 hrs. Note at each inspection whether the under surface is dry, damp or wet with no dripping; damp or wet with dripping.

The water resistance of fabrics as determined by

the modified spray test is rated on a scale of 10 as follows:

- 10 Undersurface of fabric remains dry for 24 hrs.
- 9 Undersurface remains dry for 7 hrs. but is damp or wet in 24 hrs. No dripping
- 8 Undersurface remains dry for 7 hrs. but is damp or wet in 24 hrs. Dripping
- Undersurface remains dry for 3 hrs. but is damp or wet in 7 hrs. No dripping
- 7 Undersurface remains dry for 3 hrs. but is damp or wet in 7 hrs. Dripping
- 6 Undersurface remains dry for 1 hr. but is damp or wet in 3 hrs. No dripping
- 5 Undersurface remains dry for 1 hr. but is damp or wet in 3 hrs. Dripping
- 4 Undersurface remains dry for 1/2 hr. but is damp or wet in 1 hr. No dripping
- 3 Undersurface remains dry for 1/2 hr. but is damp or wet in 1 hr. Dripping
- 2 Undersurface remains dry for 5 min. but is damp or wet in 1/2 hr. No dripping
- 1 Undersurface remains dry for 5 min. but is damp or wet in 1/2 hr. Dripping
- 0 Undersurface damp to dripping in 5 min.

DISCUSSION OF GENERAL PRINCIPLES

MODIFIED FUNNEL TEST—Canvas for covering equipment, wagons, etc., is often bent in various ways or may be folded in short points. The crumpling, wetting, drying, rewetting, and redrying are carried on because in this way the temporary resistance of new fabric is partly removed, and the conditions of actual service are imitated to some definite extent, at least. The paper behind the fabric serves a double purpose of imitating the effect of contact with an absorptive material while the fabric is wet, and, furthermore, promptly shows any wetting through of the fabric.

TABLE I—COMPARISON OF THE FUNNEL AND THE SPRAY TESTS WITH ACTUAL EXPOSURE TO RAIN

Number	Funnel Test Rating	Spray Test Rating	Actual Rain Test (Condition of Blotting Paper)
32340	0	0	Wet
32745	0	0	Wet
33253	0	0	Wet
33330	0	0	Wet
33433	0	0	Wet
33676	4	2	Wet
33886	0	0	Wet
33965	0	4	Wet
34751	0	0	Wet
34772	0	0	Wet
34780	0	0	Wet
34791	0	0	Wet
34792	0	0	Wet
34964	0	2	Wet
35261	0	0	Wet
35729	0	0	Wet
35730	0	0	Wet
35731	0	0	Wet
35756	0	0	Wet
33617	0	4	Dry
34748	9	9	Dry
34749	10	7	Dry
34810	4	9	Dry
34824	10	9	Dry
34826	4	8	Dry
34828	10	8	Dry
34831	9	9	Dry
34856	0	6	Dry
35737	0	9	Dry

In order to obtain consistent and reliable results by this method, it is necessary to follow closely the directions as outlined, especially as to drying in the oven at 45° C. (113° F.) for 24 hrs. Many comparisons have been made on samples of various treatments by (1) soaking and drying in the air in the laboratory for 24 hrs. and (2) by drying in the oven at 45° C. for 24 hrs. The results show a low water resistance on many samples when dried in air only, and a high water resistance when dried in the oven at 45° C. In no case has the reverse been true.

TABLE II—DATA ON THE WATER RESISTANCE OF A TREATED COTTON DUCK

NUMBER	Method of Application (Brushed)	Applied Per cent	Pin Holes	Condition of Dry Fabric			Condition of Wet Fabric	Water Resistance		Water Absorbed Per cent	Decrease in Water Absorbed Due to Treatment Per cent
				—10° C. for 24 Hours	Room Temperature	45° C. for 24 Hours		Funnel Test Rating	Spray Test Rating		
36095 ¹	Used as blank	...	Numerous	...	Satisfactory	Satisfactory	...	0	2	61.0	...
36164	1 coat on each side	34.2	Numerous	Satisfactory	Satisfactory	Satisfactory	Slightly sticky	7	6	33.0	46.0
36164	2 coats on one side	36.4	Several	Satisfactory	Satisfactory	Satisfactory	Slightly sticky	9	8	32.0	46.0
36166	1 coat on each side	33.5	Numerous	Stiffens slightly	Satisfactory	Satisfactory	Slightly sticky	10	10	30.0	51.0
36166	2 coats on one side	34.9	Several	Stiffens slightly	Satisfactory	Satisfactory	Slightly sticky	9	10	31.0	49.0
36168	1 coat on each side	30.0	Numerous	Stiffens slightly	Satisfactory	Satisfactory	Slightly sticky	10	9	33.0	46.0
36168	2 coats on one side	34.9	Several	Stiffens slightly	Satisfactory	Satisfactory	Slightly sticky	10	9	32.0	48.0
36170	1 coat on each side	34.9	Several	Stiffens slightly	Satisfactory	Satisfactory	Slightly sticky	7	9	29.0	50.0
36170	2 coats on one side	34.9	None	Stiffens slightly	Satisfactory	Satisfactory	Slightly sticky	8	9	30.0	51.0
36172	1 coat on each side	35.7	Several	Satisfactory	Satisfactory	Satisfactory	Slightly sticky	8	9	28.0	54.0
36172	2 coats on one side	41.3	None	Satisfactory	Satisfactory	Satisfactory	Slightly sticky	9	9	30.0	51.0
36174	1 coat on each side	32.8	Several	Stiffens slightly	Satisfactory	Satisfactory	Satisfactory	9	10	30.0	51.0
36174	2 coats on one side	28.6	Numerous	Stiffens slightly	Satisfactory	Satisfactory	Satisfactory	10	10	32.0	48.0
36176	1 coat on each side	25.8	Numerous	Stiffens slightly	Satisfactory	Satisfactory	Satisfactory	10	9	33.0	46.0
36176	2 coats on one side	17.9	Numerous	Stiffens slightly	Satisfactory	Satisfactory	Satisfactory	10	9	33.0	46.0
36178	1 coat on each side	34.2	Numerous	Stiffens	Stiffens slightly	Satisfactory	Satisfactory	10	10	31.0	49.0
36178	2 coats on one side	30.8	Numerous	Stiffens	Stiffens slightly	Satisfactory	Satisfactory	10	10	34.0	44.0
36180	1 coat on each side	33.5	Numerous	Stiffens	Satisfactory	Satisfactory	Satisfactory	9	9	31.0	49.0
36180	2 coats on one side	33.5	Numerous	Stiffens	Satisfactory	Satisfactory	Satisfactory	9	9	30.0	51.0
36182	1 coat on each side	27.9	Numerous	Stiffens slightly	Satisfactory	Satisfactory	Satisfactory	9	10	32.0	48.0
36182	2 coats on one side	29.3	Numerous	Stiffens slightly	Satisfactory	Satisfactory	Satisfactory	9	10	34.0	44.0
36140	1 coat on each side	34.2	Several	Stiffens	Satisfactory	Satisfactory	Rubs off slightly	8	10	30.0	51.0
36140	2 coats on one side	36.0	Several	Stiffens	Satisfactory	Satisfactory	Rubs off slightly	10	10	32.0	48.0
36229	1 coat on each side	22.4	None	Stiffens considerably	Stiffens slightly	Satisfactory	Stiffens slightly	0	10	40.0	34.0

¹ Untreated cotton duck used for all treatments. Weight, 14.3 ounces per sq. yd. Threads per inch: warp, 36 (double threads, each one ply); filling, 24 (single threads, one ply).

While it is realized that this test as conducted in this laboratory is a severe one, no canvas which withstands it has been found to fail in actual service.

MODIFIED SPRAY TEST—The spray test establishes, approximately at least, in what time hard rain will penetrate the fabric when it is placed in a slanting position.

Important features of this method are: (1) the rate of flow of water is definitely established and controlled; (2) the water is not subject to uncontrollable fluctuations of pressure; and (3) the temperature of the water remains about the same at all times.

A few typical results in which the funnel and spray tests are compared with an actual rain test on samples which have been exposed to the weather for several months are given in Table I. The actual rain test was carried on by securely attaching a 12-in. square sample flat on the board. Between the board and the fabric was placed a piece of blotting paper. The board on which the fabrics were attached was exposed at an angle of 30° to a gentle continuous rain for 6 hrs. At the end of the rain the blotting paper was examined, with the results recorded in Table I.

The results in Table I show that both the funnel and spray tests give a rating of zero on all except three of the samples under which the blotting paper became wet during the actual rain test. On samples that did not wet through during the same rain, the funnel test ratings range from zero to 10, and the spray test ratings range from 4 to 9.

It will be noted that several of the fabrics which did not become wet on the undersurface during this rain showed a rating of zero by the funnel test. On the other hand, whenever the fabric proved of no value on actual exposure to weather conditions, it has always had a low rating by the funnel test.

The spray test appears to check better with the results obtained by exposure to an actual rain than the funnel test.

In Table II are reported some results by the funnel and spray tests on fabrics treated by formulas developed in this laboratory. The percentage of water absorbed, determined by the method outlined below, is also recorded.

All except one of the treatments herewith reported show a rating of 7 to 10 by the funnel test. This treatment gave a rating of zero.

The spray test gives a rating of 6 to 10 on all samples, the lowest (36164) corresponding to the lowest by the funnel test.

The water absorption of the fabrics was determined as follows:

Dry the sample used in the funnel and spray tests for 24 hrs. at 45° C., weigh, and lay flat in distilled water at a temperature of 70° to 80° C. for 5 hrs., remove, hold by two corners and give it four vigorous, snappy swings by hand; place between two sheets of 50-pound blotting paper on a flat surface, cover with a flat surfaced weight 13 in. square and weighing 5 lbs. Allow to remain under this weight for 30 sec., remove, weigh immediately, and calculate the per cent of water absorbed.

The percentage decrease in water absorbed due to the treatment on all samples herewith reported, while considerable, does not vary to any great extent. The difference is within the error of manipulation, except treatment 36229. This sample shows a percentage decrease in comparison with the untreated cloth of 34 per cent, while all the other treatments range from 44 to 54 per cent. These results confirm a more extended series on the same subject. It is interesting to note also that this treatment (36229) has a rating of zero by the funnel test.

The pressure test essentially as described by Heermann, the water being applied from a height of 20 in., to the undersurface of a circular area of fabric (2³/₄ in. in diam.), was also studied. The results, however,

added nothing to the information supplied by the funnel and spray tests, and it was abandoned.

Other tests and observations made on fabrics which have been treated to increase the water resistance are briefly as follows:

Pin holes: The fabric is held before an electric light, and pin holes are rated as numerous, several, or none.

Condition of the dry fabric at -10°C. , at ordinary room temperature and at 45°C. is rated as stiff, or supple, and as greasy, sticky, or rubs off.

Condition of the wet fabric is rated as stiff, or supple, and as greasy, sticky, or rubs off.

A reaction to litmus paper of the water extract is noted.

The sulfates are tested for.

Weight per square yard in ounces is determined before and after washing.

Threads per inch, warp and filling are counted.

Percentage of ash, qualitative tests for lead, zinc, copper, chromium, aluminum, and iron are determined when desirable.

Quantitative determination of copper is also made when put in mildew-proof fabrics.

An attempt is also made to determine the nature of the waterproofing material as well as the quantity present.

SUMMARY

The modified funnel test and the modified spray test when conducted under the standardized conditions described are simple of execution, give results within a reasonable time, and yield more information on the water resistance of cotton duck than the other tests which have been tried in the Bureau of Chemistry.

ADDENDUM

Since the presentation of this paper at the meeting of the AMERICAN CHEMICAL SOCIETY at Buffalo, April 7 to 11, 1919, there has appeared an article by Martin and Wood¹ entitled "Notes on the Quantitative Testing of Rainproof and Waterproof Cloth." These writers review briefly the various methods for determining the waterproof value of cloth, including the "drop test," "dash test," "trough or bag test," "filter test," "thistle tube test," and the Gawalowski apparatus. These methods, with the exception of the "drop test" and the "dash test" have already been reviewed in this paper. The "drip test" according to Martin and Wood is known as the "War Office test" and it is claimed that it is especially valuable, "in that it furnishes a fairly accurate numerical value of the degree of waterproofing."

The authors describe the "drop test" in which water is allowed to drip from a burette from a height of about 5 ft. at a rate of 20 drops per minute on to the fabric. The cloth is placed at an angle of 45° under which is laid a piece of white blotting paper. The dripping of water is continued until the water passes through the cloth and stains the blotting paper.

A similar test to this has been given a trial in this laboratory but it was discontinued for the spray test because it was believed the latter imitates much better the conditions of an actual rain. The drop test as described by Martin and Wood was, no doubt, devised for waterproofed garments and not for waterproofed cotton duck.

The "dash test" as described consists of holding the fabric horizontally and pouring water on it. Martin and Wood do not consider it of any particular value.

UTILIZATION OF ASPHALTIC BASE ACID SLUDGE OBTAINED IN REFINING PETROLEUM AND SHALE OILS¹

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At certain stages in the process of refining petroleum and shale oils after treatment with sulfuric acid the "acid sludge," as it is usually called, is cooked up by means of live steam, with concentrated sulfuric acid to separate any refinable oil retained by the usually pasty mass and to carbonize the remaining material. This is the common method of disposing of a residuum obtained in large amounts in oils especially of an asphaltic, or partially asphaltic base nature.

The process above outlined may be regarded as taking place in two stages, although they so merge one into the other that it, as practiced, appears as one. As the temperature of the mixture of acid sludge and acid is raised the retained oil escapes from the pasty mass, which becomes quite liquid with the elevation of temperature. The separated oil rises on top of the original mixture. This is effected within about an hour or an hour and a half. The raising of the temperature by means of steam is continued and the acid sludge and concentrated sulfuric acid are mixed intimately. This treatment is continued for several hours longer during which time the refuse or residuum (middle portion) becomes more or less decomposed and carbonized as shown by its change in character and the constant escape of sulfur dioxide, the latter being due to the reduction of sulfuric acid. On ceasing the agitation, the carbonized acid sludge and excess of concentrated acid used separate into two layers, the concentrated sulfuric acid being the bottom layer. The sulfuric acid is drawn from beneath to be used again, and the acid sludge thus produced is run out into a suitable conveyor trough and washed once with water, whereby usually about one-half of the retained acid is removed and is sometimes recovered. This washed, acid-cooked residue is usually burned with coal to get rid of it. It contains from 3 to 15 per cent of free sulfuric acid.

Many investigations have been carried out looking toward utilization of this cooked acid sludge, but the most economical practice to date appears to be its destruction by burning. Its production involves large and expensive lead-lined kettles and its disposition entails much labor. In fact, it is a nuisance to the refiner of petroleum. The products of the combustion are unusually rich in sulfur dioxide.

It has been determined that this expensive treatment is unnecessary and furthermore that the acid sludge, if obtained from an oil with an asphaltic, or partially asphaltic base, may be converted almost directly into a product of commercial value.

¹ Presented at the 57th Meeting, American Chemical Society, Buffalo, N. Y., April 2 to 7, 1919.