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Survey of Cellulosic Insulation Materials

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Survey of Cellulosic Insulation Materials

I. INTRODUCTION

The properties of commercially available cellulosic thermal insulation materials were evaluated to obtain base level data on the materials and to assess existing specification standards commonly used for testing and purchasing. Cellulosic material has been used for residential building insulation for several decades and currently represents an estimated 30–40 percent of that market (second only to fibrous glass insulation). Nevertheless, very little data about the properties of the product have been published. The results of this survey provide guidance to the manufacturer in the design and manufacturing control of the material; to specification organizations in re-evaluating and improving specifications; and to the consumer in selecting a product.

Cellulosic insulation is manufactured from waste paper products, such as newspaper. Its manufacture is simple, requiring only shredding and milling to convert it into a low-density, fluffy material and the addition of chemicals to provide flame retardancy. When bagged, the material is ready to be installed. Even installation is simple; it can either be poured or blown in place. Because of the relatively low capital cost required for production and the large profitable market for the material, it is estimated that there are over one hundred manufacturers operating throughout the country.

Cellulosic insulation has several advantages which could produce even greater future demand for the product. On the other hand, it has several potential disadvantages which could seriously affect the industry, if not corrected. When properly applied, cellulosic insulation has excellent thermal resistance properties, is manufactured from an inexpensive and readily available waste material, and requires little energy or petroleum base materials in its manufacture. It can currently compete favorably on a cost/performance basis with other insulation materials, and, as future energy costs increase, its competitive position will be enhanced. However, on the negative side, cellulosic insulation has received criticism alleging poor flame retardancy, overstated thermal resistance values and poor manufacturing quality control. Although it is suspected that some of these criticisms are justly deserved, there has been a lack of reliable data to refute or substantiate many of them.

In consideration of the above, this survey of cellulosic insulation properties, though limited in scope, provides base data which will prove valuable in providing a better understanding of the material, in improving the quality of the product, and in promoting the conservation of energy. The specific cellulosic properties addressed in this survey include:

- composition and quantity of fire retardant
- moisture absorptivity
- fire retardance
- thermal conductivity
- corrosiveness, and
- resistance to fungal growth.

I. EXPERIMENTAL APPROACH

A total of nineteen different off-the-shelf samples of cellulosic insulation were obtained from four geographic areas: Colorado, Minnesota, Indiana, and Pennsylvania. All tests, except for thermal conductivity and fire retardancy, were performed under direct ERDA contract at the laboratories of the Naval Weapons Support Center, Crane, Indiana. The thermal conductivity and fire retardance test data were supplied to ERDA by an independent organization, and the sample materials used in their tests were also used in the Naval Laboratory tests.

Whenever possible, tests were performed in accordance with the American Society for Testing Materials (ASTM) specifications C739–73; Cellulose Fiber (Wood Base) Loose-Fill Thermal Insulation. The ASTM C739–73 specification is referenced in the Federal specification HH–1–515C Insulation Thermal (Loose-Fill for Pneumatic or Poured Application) Cellulosic or Wood Fiber, and is the basis for the National Cellulose Insulation Manufacturers Association (NCIMA) specification N–101–73, Standard Specification for Cellulosic Fiber (Wood Base) Loose Fill Thermal Insulation. In some tests, conditions were modified to gain additional information which will be discussed later in the text.

Because it was realized that one sample from each manufacturer might not fairly represent that manufacturer's product, manufacturers' identifications were not given in the report: the data were evaluated as a whole to determine trends and patterns.

I. EXPERIMENTAL RESULTS

A. FIRE RETARDANT ADDITIVES

Samples of cellulosic insulation were analyzed to identify the additives and their relative proportions. This was done by extracting the water soluble content of 5 gram samples and subjecting the residue to a series of analytic procedures described in Appendix A. No attempt was made to analyze for possible water insoluble additives. The analysis scheme included use of X-ray diffraction, atomic absorption, spectrographic and X-ray fluorescence and differential thermal analysis techniques. The analyses were considered to be semiquantitative because:

1. the samples contained various unknown compounds that were also water soluble, such as starches, inks and adhesives;

2. the degree of hydration of the original fire retardant compound was not known (the most probable was assumed) ; and
3. only that quantity retained within or on the cellulose was analyzed. As will be discussed in the next section, it was observed that some of the fire retardant had separated in most of the samples.

The results of the analyses did provide a measure of the kinds and relative proportions of fire retarding additives as shown in Table I. Also included in Table I are the pH* values of the samples when contacted with water. The procedure for measuring pH is given in Appendix B.

These analyses showed that boric acid ** and ammonium sulfate were the most common additives and were used singly or in various combinations with other additives such as calcium sulfate, aluminum sulfate and sodium carbonate. Comparison of the pH values of the respective samples showed that the resulting pH was not always in accordance with the kind and quantity of the additives. Whereas this could be, in part, a result of inaccuracies in the quantitative analyses, it is also possible that impurities in the cellulose stock material contributed to the final pH.

B. Separation of Fire Retardant Additives

Of the nineteen samples received for analyses, thirteen showed visible evidence that some of the fire retardant chemical had separated from the cellulosic matrix; quantities of the additives were found at the bottoms of the containers. Because each sample had undoubtedly been handled differently from the time of its manufacture, no attempts were made to measure the quantity of the separated material nor to determine the effects such separations may have on the properties of the samples. The referenced standard specifications (ASTM, Federal and NCIMA) do not include a test or requirement for non-separation of the fire-retardant additive.

C. Moisture Absorptivity

The nineteen samples were tested for water absorption in accordance with ASTM C739–73, section 10.5. According to that specification, weight gain should not exceed 15 percent. For the standard test, samples of approximately 100 grams were pre-conditioned at 50 percent relative humidity (R.H.) and 120°F to a constant weight. The samples were then explosed to 90 percent R.H. at 120°F for 24 hours and the weight gain recorded. In addition to the standard 24 hour tests, cumulative weight gain data were also obtained after 8 days and 15 days.

Since the ASTM C–739–73 procedure does not specify the sample configuration during testing, the samples were contained in 9″ x 12″ x 2½″ open containers to allow a low-packing density similar to that found in attic installations. For several of the sample materials, additional test specimens were packed in either 1000 ml or 2000 ml beakers to evaluate the effect of different packing densities and configurations.

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* A measure of the relative acidity of samples; 7.0 indicates a neutral solution and decreasing values indicate increasing acidic activity.

** The boron contents were reported as boric acid; however, the original compound may have been other boron compounds such as "borax."

TABLE I
Composition and pH of Cellulosic Insulation Samples

Sample Identification	Total Water Solubles %	pH	Fire Retardant Chemical, % Ammonium Sulfate	Boric Acid	Calcium Sulfate	Aluminum Sulfate	Sodium Carbonate
526-1	18	4.4	18	—	—	—	—
526-5	20	8.0	—	11	1	—	5
527-A	22	8.2	—	16	—	—	3
527-B	31	4.8	—	23	—	—	—
527-C	28	8.1	—	22	—	—	5
527-C1	24	8.2	—	20	—	—	5
527-D	22	8.0	—	13	—	—	5
527-E	26	4.5	26	—	—	—	—
527-F	21	5.9	—	10	5	—	2
527-G	19	4.4	19	—	—	—	—
527-H	21	7.8	—	16	3	—	1
527-I	20	5.0	—	4	1	—	—
535	24	7.4	—	17	4	—	—
562	22	3.7	18	1	2	—	1
563-4	24	4.0	—	10	—	7	2
563-5	19	7.7	12	4	1	—	2
563-6	17	5.9	—	4	6	—	—
563-7	23	6.1	—	5	8	—	—
593	17	7.7	—	—	—	17	—

The results of the moisture absorptivity tests are given in Table II and shown graphically in Figure I.

Examination of these data show the following:

1. There was a wide range in moisture absorption between samples when tested in the low density configuration. After the standard 24 hour test, moisture gains ranged from 3.5 to 38 percent and six of the samples exceeded the 15 percent limit given in the standard specification.
2. The differences in moisture absorption increased with increasing time—some samples had moisture gains in the 75 percent range after 8–15 days exposure. Also, in some samples the moisture gains reached a maximum and then decreased. The mechanism for this behavior is not known, but may be a result of some moisture-induced separation of the fire retardants from the cellulose matrix.
3. Generally, samples containing primarily boric acid had lower, and acceptable, moisture absorptivities, whereas those containing primarily ammonium sulfate had excessive gains in moisture. The one sample containing only aluminum sulfate showed excessive moisture gains but less than those containing ammonium sulfate.
4. The differences between moisture gains in samples containing similar additives suggested that factors other than composition of fire retardants also affect moisture gain, such as the size and distribution of the additives and the characteristics of the cellulose matrix.
5. The apparent moisture gains observed during testing were dependent upon the specimen configuration. For example, sample 563–5, when tested in the low-density configuration had an unacceptable 22 percent moisture gain. However, when tested in 1000 and 2000 ml beakers, the moisture gains were an acceptable 6 and 8 percent respectively.

D. CORROSIVENESS

The nineteen samples were tested for corrosiveness in accordance with ASTM C739–73, section 10.7, except that thicker metal test coupons were used. The thicker coupons (0.25 inches/0.6 cm) were selected to allow more detailed evaluation of the mechanisms and rates of possible corrosion. The test coupons specified in ASTM C739 are only 0.003 inches thick and failure of test is based on visual observance of perforation of the coupon.

The coupons, tested in duplicate for each cellulosic samples, were:

1. Steel, AISI type 1018, cold rolled, 5 cm x 6.3 cm x 0.6 cm.
2. Aluminum, alloy 2024-0, (annealed) 5 cm x 5 cm x 0.6 cm.
3. Copper, type K tubing, 5 cm x 1.5 cm O.D.

As specified in the standard, the coupons were placed in contact with the cellulosic samples that had been moistened with water and held for thirty days at 120°F and 96% R.H. Because of the difficulty in expressing the corrosion results adequately in terms of a single number or term, the data were reported by corrosion type; general or uniform, pitting, and subsurface.

TABLE II

Moisture Weight Gains in Cellulosic Insulation Samples

A. Moisture Gain (%) in Low-Density Packing Configuration

Sample Identification	Pre-Test Conditioning (50% RH)	Test Exposure (90% RH) 24 Hour *	8 days	15 days
526-1	2.4	38.5	76.5	43.7
526-5	1.1	9.5	18.2	19.3
527-A	1.1	5.6	7.7	6.7
527-B	1.3	5.6	7.2	6.0
527-C	–0.5	7.8	9.0	8.4
527-C1	0.1	7.3	8.4	7.9
527-D	2.2	10.9	16.4	20.4
527-E	2.2	29.0	65.9	70.0
527-F	1.1	10.0	9.7	8.5
527-G	2.2	24.1	43.2	37.4
527-H	2.6	7.0	5.5	3.8
527-I	2.5	24.8	50.7	42.5
535	–0.3	10.3	16.4	14.9
562	1.8	11.2	20.2	19.6
563-4	0.5	11.6	16.8	14.4
563-5	0.7	21.6	55.1	29.5
563-6	0.7	10.6	15.0	10.1
563-7	0.6	12.3	13.1	10.3
593	1.2	19.6	30.3	29.7

B. Moisture Gain for Different Packing Configurations

Sample Identification	Pre-Test Conditioning (50% RH)	Test Exposure (90% RH) 24 Hour *	8 days	15 days
527-A, Low Density	1.1	5.6	7.7	6.7
527-A, 2000 Ml Beaker	1.4	4.0	6.9	6.9
527-A, 1000 Ml Beaker	1.5	3.5	6.4	6.5
562, Low Density	1.8	11.2	20.2	19.6
562, 2000 Ml Beaker	2.2	4.9	13.1	17.1
562, 1000 Ml Beaker	1.8	4.3	12.2	15.2
563-5, Low Density	0.7	21.6	55.1	29.5
563-5, 2000 Ml Beaker	1.1	7.6	23.5	29.2
563-5, 1000 Ml Beaker	1.1	6.0	21.1	26.4

* Standard ASTM test, acceptance level set at less than 15% gain.

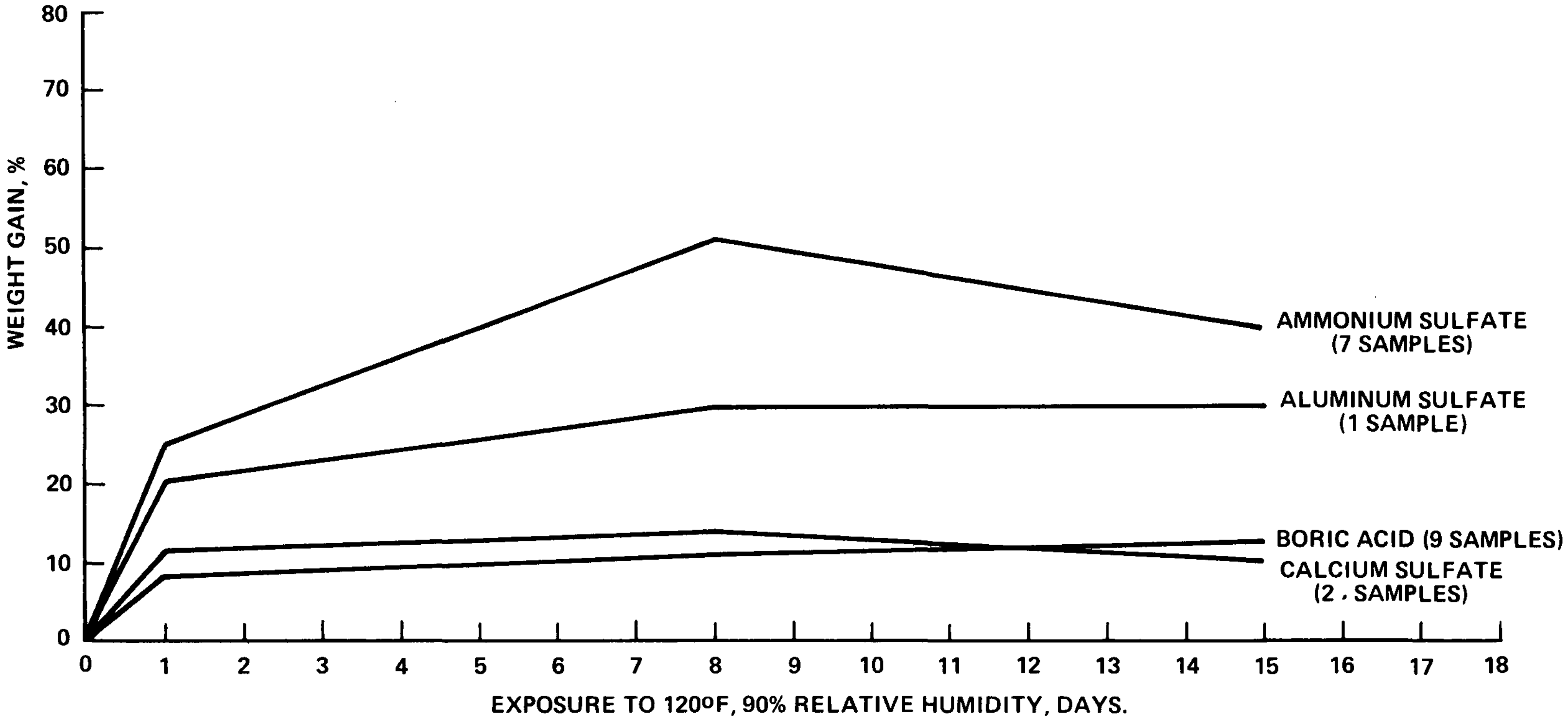

FIGURE 1. EFFECT OF FIRE RETARDANT COMPOSITION ON MOISTURE WEIGHT GAIN.

General corrosion was determined by measuring the coupon weight loss during the test, and calculating the equivalent uniform loss of thickness over all surfaces of the test coupon. Pitting corrosion was determined by microscopic measurement of the depths of individual surface pits below the final coupon surface. Subsurface corrosion was determined by metallographic analysis of cross-sections of the coupons thereby indicating both the depth and mechanism of corrosion; e.g., intergranular. Again, all measurements were made from the final coupon surface. The general corrosion data are given in Table III and Figure 2. Pitting corrosion data are given in Table IV.

Examination of the corrosion test data showed the following:

General Corrosion:

1. The corrosion experienced by the individual test coupons was not uniform, but rather much greater on one of the surfaces, and, in many cases variable over the surface. Apparently corrosion was greater at points of intimate physical contact between the coupon and the cellulose. Consequently, the calculated general corrosion reported in Table III and Figure 2 represents conservative corrosion values: it is roughly estimated that the corrosion areas were approximately twice those reported.
2. A wide range of corrosion rates of the test materials were observed between the cellulosic samples. Generally, the steel coupons experienced the greatest rate of corrosion.
3. There was poor correlation between corrosion rates, composition of fire retardant, and pH.
4. Based on the calculated general corrosion rates, eleven of the nineteen samples showed corrosion rates greater than allowed under ASTM C739–73 for one or more of the test coupon materials. (The corrosion rate to completely dissolve the 0.003 inch thick test coupons specified in ASTM C739–73 in the 30-day test period is equivalent to 0.45 mm per year.)

Pitting Corrosion:

5. The observed pit depths are conservative, since measurements were taken from the final coupon surfaces.
6. Only the aluminum test coupons showed any significant amount of pitting corrosion.
7. All nineteen cellulosic samples produced pitting in the aluminum test coupons to an extent greater than allowed in ASTM C739–73 (The ASTM standard test coupon is 0.003 inches or 0.076 mm thick.) However, the control sample, which was exposed only to the test atmosphere, also showed excessive pitting. Of the nineteen coupons tested against the cellulose samples, twelve exhibited pitting depths greater than that observed on the control coupon. The standard specifications do not require the evaluation of control coupons in the corrosion tests.

8. Essentially no correlation was observed between the pitting of aluminum, composition of fire retardant, and pH.

Subsurface Corrosion:

9. There was extensive subsurface corrosion in the form of intergranular attack in the aluminum test coupons. Maximum observed depths of subsurface attack were in the 0.008–0.010 inch range. Although no attempt was made to correlate depth of attack with fire retardant composition and pH, deep attack was observed in test coupons exposed to samples containing boric acid and ammonium sulfate.
10. Subsurface corrosion of the copper test coupons was limited to an observed maximum of about 0.0005 inches and was considered negligible.
11. The steel test coupons had no observable subsurface corrosion.

TABLE III
Rates of General Corrosion of Aluminum, Copper and Steel Exposed to Cellulosic Insulation Samples, Millimeters per Year*

Sample Identification	Aluminum	Copper	Steel
Control**	0.18	0.05	0.05
526–1	0.29	0.75	1.57
526–5	0.14	Nil	0.77
527–A	0.05	Nil	0.19
527–B	0.16	0.07	1.53
527–C	0.06	Nil	0.46
527–C1	0.29	Nil	0.38
527–D	0.25	0.03	0.36
527–E	0.10	0.78	0.33
527–F	0.07	Nil	0.43
527–G	0.11	0.47	1.57
527–H	0.06	Nil	0.15
527–I	0.39	0.30	0.98
535	0.24	0.04	0.58
562	0.26	0.20	0.16
563–4	0.06	0.10	1.12
563–5	0.20	0.36	0.38
563–6	0.14	0.03	0.87
563–7	0.11	0.03	0.34
593	0.22	0.33	2.24

* Base on 30-day test period

** Control samples exposed only to test atmosphere

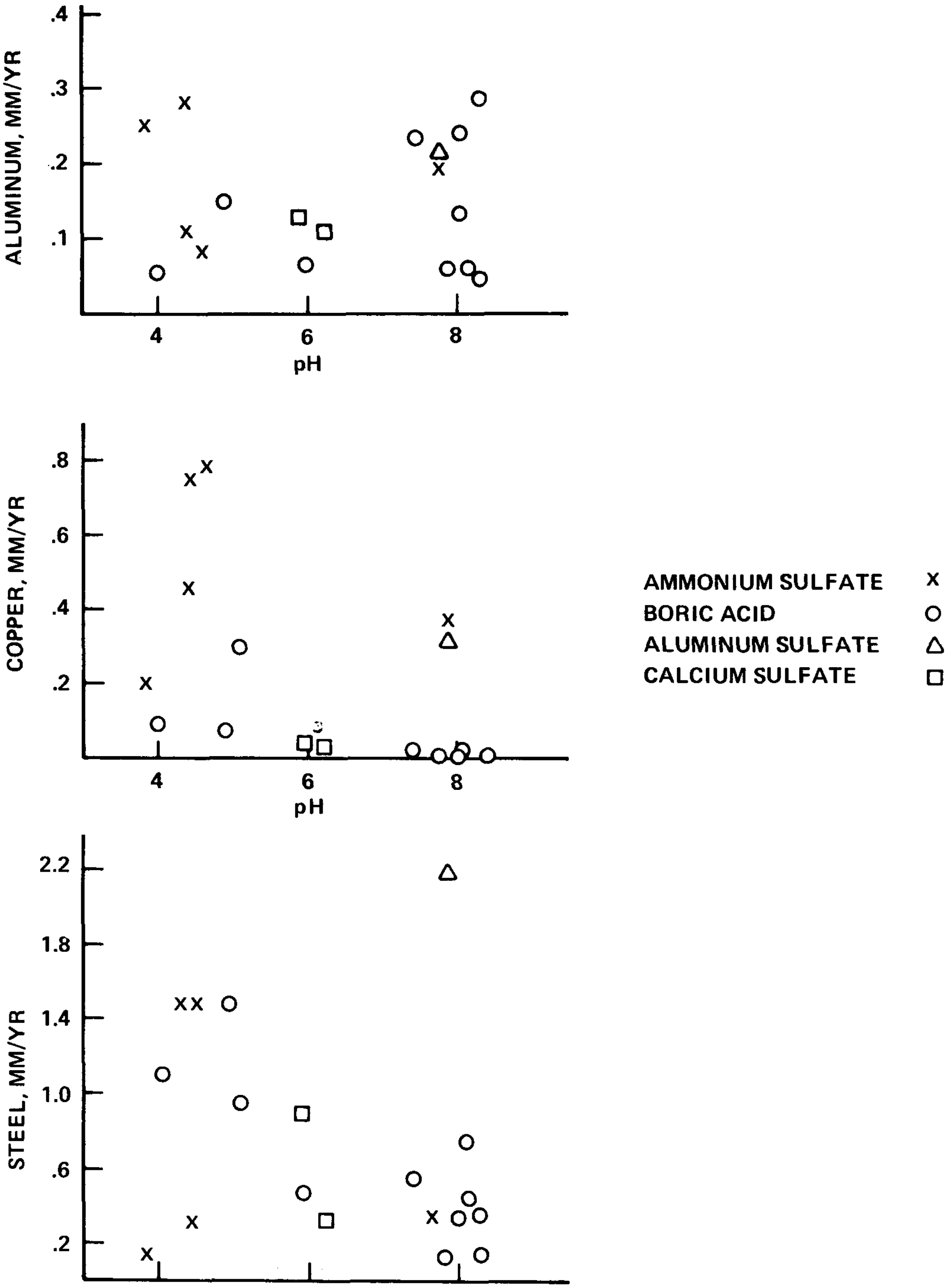

FIGURE 2. EFFECT OF COMPOSITION AND pH ON GENERAL CORROSION RATES.

TABLE IV
Maximum Pit Depth on Aluminum Exposed to Cellulosic Insulation Samples for 30 Days

Sample Identification	Pit Depth Millimeters
Control	0.38
526–1	0.25
526–5	0.86
527–A	0.15
527–B	0.15
527–C	0.33
527–C1	0.76
527–D	0.99
527–E	0.28
527–F	1.04
527–G	0.63
527–H	0.33
527–I	0.66
535	0.48
562	0.96
563–4	0.66
563–5	1.07
563–6	0.61
563–7	0.55
593	0.81

E. RESISTANCE TO FUNGAL GROWTH

Fungi which degrade cellulose are widespread and are found in virtually all environments. Generally, these fungi require temperatures in the 50–100°F range and a relative humidity of 60 percent or greater. It is possible that fungal growth on cellulosic insulation could cause the following undesirable conditions:

a. provide a source of fungal spores which can penetrate the living area and cause health problems,
b. degrade the thermal properties of the insulation by destroying the structure of the cellulose, and
c. increase the corrosive action of the insulation material through accomulation of metabolic products.

Testing for fungal growth is not included in the ASTM, Federal or industry specifications.

The nineteen cellulosic samples were tested on a "go, no-go" basis to determine the propensity for fungal growth. The samples were tested at 86°F and 95 percent relative humidity for 28 days in accordance with military testing specification Military Standard 810B, method 508. Results of those tests showed the following:

1. Cellulosic samples containing primarily boric acid were resistant to fungal growth.

2. Cellulosic samples containing primarily ammonium sulfate supported fungal growth. Visible indications of growth were observed after 2–3 weeks exposure in sample 526–1, 527–E, 527–G, 563–5 and 562.
3. The cellulosic sample containing primarily aluminum sulfate (593) supported fungal growth; visible indications of growth were observed after 2 weeks exposure.
4. The existence of fungal growth after 28 days exposure would be difficult for the untrained or casual observer to detect because of the coloration and texture of the cellulosic matrix.

F. THERMAL CONDUCTIVITY AND FLAME SPREAD

Only eight of the nineteen cellulosic samples were tested for thermal conductivity and flame spread by a non-Federal organization prior to the foregoing tests performed at the Naval Laboratory. The thermal conductivity tests were reportedly performed in accordance with ASTM C518–70, Thermal Conductivity of Materials by Means of the Heat Flow Meter Method. Likewise, the flame spread tests were performed in accordance with ASTM E84–75, Test For Surface Burning Characteristics Of Building Materials.

Both of the above tests are included in the ASTM, Federal and Industry specifications. The results of the tests are given in Table V.

Examination of these data show the following:

1. For most of the samples, the thermal conductivity values obtained through independent testing were significantly higher than corresponding values reported by the manufacturers. The differences in value were greater than could be expected from differences in test densities.
2. Flame spread data reported by the manufacturers were in good agreement with those obtained through independent testing. The data indicate that both boric acid- and ammonium sulfate-based fire retardants are capable of affording the flame spread levels defined in the standard specifications.

TABLE V
Thermal Conductivity and Flame Spread
Values for Cellulosic Insulation Samples

A. Thermal Conductivity

	Manufacturer's Data		Independent Data		
	Test Density pcf	"K" BTU-in Hr-ft^2-°F	Test Density pcf	"K" BTU-in Hr-ft^2-°F	Deviation in "K" Values, %
527–A	3.0	.25	2.9	.35	40
527–B	2.3	.20	2.8	.31	55
527–C	3.0	.19	2.6	.31	63
527–C1	–	–	2.4	.30	–
527–D	2.4	.27	2.7	.32	18
527–E	–	.24	2.2	.29	21
527–F	1.8	.26	2.2	.29	12
527–G	2.2	.27	2.3	.27	0

B. Flame Spread

	Manufacturer's Data*	Independent Data**
527–A	15	10
527–B	10	20
527–C	38	15
527–C1	–	–
527–D	30	20
527–E	–	15
527–F	20	15
527–G	–	10

* Test method not known
** ASTM E84 25-foot tunnel tester

IV. CONCLUSIONS

A survey of cellulose thermal insulation materials has provided base level property data heretofore unavailable and an assessment of the suitability of specification standards for defining the properties and quality of the material. Since the survey included samples of only nineteen commercially available products from an estimated one hundred manufacturers, the results and conclusions cannot be interpreted as pertaining to every manufacturer. But rather, the results must be reviewed in total to identify overall trends or patterns and serve as a base from which manufacturers can improve their products and consumers can better choose a product. The results also provide a basis from which organizations responsible for preparing cellulosic insulation standard specifications can evaluate the adequacy of their standards.

Specific conclusions derived from this survey were as follows:

1. A variety of fire retarding chemicals are added to cellulose in quantities ranging to about 25 percent. The chemicals are added either singularly or are combined. Types of chemical additives used include:

   ammonium sulfate
   "borates", e.g., boric acid and borax
   aluminum sulfate
   calcium sulfate
   sodium carbonate

2. The pH of the samples, when contacted with water, ranged from 3.7 to 8.2. The standard specifications neither limit nor require reporting of pH values.
3. Some separation of fire retardant chemicals from the cellulosic matrix occurred in thirteen of the nineteen samples surveyed after handling under normal conditions. The standard specifications do not include criteria for retention of fire retardant chemicals.
4. Six of the nineteen samples exceeded the moisture absorption criteria of the standard specifications when tested in a low-density configuration (e.g., as may be found in attic installations). Excessive moisture absorption rates were generally found in samples containing ammonium sulfate and aluminum sulfate. The standard specifications do not adequately define the testing conditions for moisture absorption tests.
5. The capacity of cellulosic materials to absorb moisture is variable. Extended testing at 120°F and 90 percent relative humidity showed weight gains ranging from 5 to 76 percent. The standard specifications do not provide limits for moisture absorption over long-term exposure.
6. The cellulosic materials exhibited a wide range of corrosiveness against aluminum, copper and steel when tested in accordance with the standard specifications. Three types of corrosion were observed:

   a. general corrosion (dissolution of the metal)
   b. pitting corrosion, and
   c. subsurface corrosion (intergranular)

   When compared with the limits of corrosion provided in the standard specifications, eleven of the samples produced excessive general corrosion, primarily when in contact with steel. Excessive pitting and subsurface corrosion were observed on aluminum coupons when tested against most of the cellulose samples. The standard specifications do not differentiate between different types of corrosion, nor do they consider the full extent of possible corrosion. Because of the poor correlation between composition and pH of the cellulose samples and observed corrosion, composition and pH cannot be used as indicators of corrosiveness.

7. Six of the nineteen samples supported fungal growth when tested at 86°F and 95 percent relative humidity for 28 days. Samples containing boric acid were resistant to fungal growth, whereas samples containing primarily ammonium sulfate or aluminum sulfate supported fungal growth. The standard specifications do not include criteria for fungal growth resistance.
8. Thermal conductivity values for seven of the eight samples tested exceeded the values reported by the manufacturers; the range of deviations was 11–63 percent. The standard specifications allow only a 5 percent deviation.
9. Flame spread ratings obtained from the eight samples tested were in good agreement with values reported by the manufacturers. The chemicals used in the tested samples were effective in providing flame spread resistance as defined under the standard specifications.

V. RECOMMENDATIONS

The results of this survey show the need for a better understanding of the parameters which control the performance of cellulosic insulation and the need for improved standard specifications. Therefore, the following recommendations are offered:

1. That the performance criteria of cellulosic insulation be reevaluated and, where necessary, redefined and/or new performance criteria identified. Items to be included in the reevaluation should be:
    - retentivity and stability of fire retardant additives
    - moisture absorptivity
    - resistance to fungal growth
    - corrosiveness
2. That the standard specifications be modified to insure effective specifications for product quality and performance.
3. That manufacturers of cellulosic insulation place greater emphasis on compliance with standard specifications.
4. That consumers of cellulosic insulation insist that purchased materials are in compliance with the standard specifications and, when feasible, check for such compliancy by independent testing.

APPENDIX A—PROCEDURE FOR ANALYZING FIRE RETARDANT ADDITIVES

(1) Check sample for organic additives by extraction with suitable solvents. Check sample for ethanol solubles in particular.

(2) Oven dry a weighted sample at 80°C for one hour to determine moisture level "as received."

(3) If results of (1) through (3) are negigible, proceed with water extraction.

(4) Combine 5 grams of insulation sample with 100–200 ml distilled water in suitable container and warm on hot plate for 5 minutes. Stir occasionally.

(5) Remove water and dissolved material from wet cellulose by vacuum filtration. Retain washed cellulose on filter paper in buchner funnel.

(6) Set aside 100 ml of the filtrate for analysis.

(7) Wash cellulose with three 100 ml portions of hot distilled water and one 50 ml portion of 95 percent ethanol.

(8) Transfer cellulose to watch glass and dry one hour at 100°C.

(9) Weigh watch glass and cellulose. Place cellulose in labeled bottle, wipe dust from watch glass and weigh.

(10) Computer weight of dried cellulose and percent water solubles by difference.

(11) Heat filtrate retained in step (6) at 80–90°C until water has evaporated. Remove dried solids, grind and mix to assure uniformity. Place in labeled vial.

(12) Perform emission spectrographic and X-ray fluorescence analyses to detect elements.

(13) Confirm compounds and elements detected in steps (11) and (12) using X-ray diffraction and differential thermal analysis.

(14) Quantitatively determine metallic elements using atomic absorption spectrophotometry.

(15) Quantitatively determine metallic elements by using the quantity of that element present and the molecular weight of the most likely molecular form and hydration state.

(16) Quantitatively determine those compounds which do not contain metallic elements by subtracting known substances in those cases where all residue components are identifiable.

APPENDIX B—PROCEDURE FOR DETERMINING pH OF CELLULOSIC SAMPLES

(1) Allow a quantity of distilled water to equilibrate with atmospheric CO_2 (approximately pH 5.7).

(2) Add 50 ml of water to one gram insulation sample and stir for approximately five minutes using an electric stirrer.

(3) If the sample does not wet readily, add 0.1 ml of a 5 percent Trident Solution to the mixture prior to stirring. (This will not alter the final pH).

(4) Using a Beckman Zeromatic pH meter and Combination Electrode (Beckman Part No. 39013), determine the pH of the suspension.