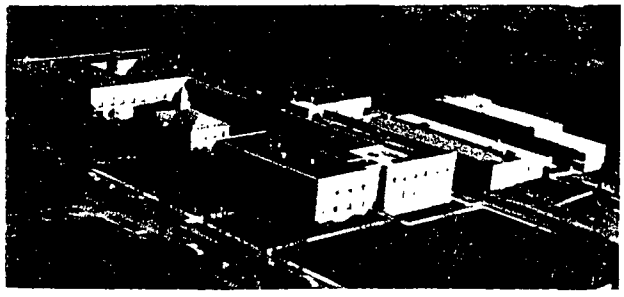

THE INSTITUTE OF PAPER CHEMISTRY, APPLETON, WISCONSIN

Report Three

Monthly Progress Report

to

U. S. ARMY CHEMICAL CENTER PROCUREMENT AGENCY

Project 2256

January 25, 1961

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THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

DEVELOPMENT OF AN IMPROVED DIFFUSION BOARD MATERIAL

INTRODUCTION

During the period ending December 28, 1960, a supply of pulp and charcoal was received, test methods for carbon dioxide diffusivity, and dioctyl phthalate smoke penetration were set up and standardized, and preliminary tests were made for conditions required to make boards with satisfactory diffusion and aerosol protection.

RECEIPT OF MATERIALS

PULP

A shipment of pulp and board samples from Minnesota and Ontario Paper Company consisted of the following:

1. White board wet lap taken from their commercial operation just before the drying phase. Approximately three-hundred pounds were received and stored in a cold atmosphere to reduce any bacterial effects. This pulp, as mentioned in Report Number Two, contains a small quantity of wax-rosin size.

2. Approximately ten pounds of a stock, similar to the above, which had been dewatered by hand and did not contain the wax-rosin size. This pulp was also stored in a cold atmosphere.

3. Six, 12 by 12 by 0.5 inch samples of the finished board made from the wax-rosin sized pulp on their commercial machine.

Samples of the pulps were sent to our Analytical Group for the determination of hot-water extractables, hot water pH, and cold water pH; results of these determinations are not yet available. The board was tested in comparison with laboratory boards made from the same pulp as described below.

CHARCOAL

A drum containing 250 pounds of ASC Grade 1 charcoal fines (through 140 mesh) was received on Voucher 1303-25672-61. Additional information was given as follows:

6810-900592

Charcoal ASC Grade 1

SPEC MIL-C-13724

Through 140 mesh

243 lb. net

Lot NY 5181-5 Pkg. No. 284

Date packaged 8/55

Wt. 293 CU 11.5

STANDARDIZATION OF TESTING

CALIPER AND DENSITY

The caliper values reported are the average of ten individual measurements taken around the perimeter of the board, at least two inches from the edges, with a Cady Exact Automatic Micrometer, recorded to the nearest thousandth of an inch. Thickness suggested in the contract is 0.25 inch.

The face area of each board was determined as the product of the average of three length determinations (to the nearest thirty-second of an inch) and the average of three width determinations (to the nearest thirty-second of an inch). Weights of the board samples were determined to the nearest 0.1 gram on an Ohaus Double Beam balance. These measurements were taken with the board at and in the ambient conditions of the laboratory. The density of each board sample was calculated from the weight and volume. Recommended density is 21 pounds per cubic foot.

CARBON DIOXIDE DIFFUSIVITY

The equipment design and method of operation for this test are based on the information given in "Determination of CO_2 Diffusivity" as furnished us by the Army Chemical Center. However, a metal box was constructed in order to minimize problems of leakage and to give a more rigid base for clamping of samples. Special clamping devices were provided for clamping the sample and for positioning the blank over the sample. In accordance with

the original method, carbon dioxide is introduced into the box until a sufficient concentration is reached and then the time of decay of carbon dioxide concentration in the box is determined by successive tests on an Orsat apparatus. Initial carbon dioxide concentrations in the range of 20 to 30% were found more convenient than the 15% mentioned in the original method. A time of 10 minutes was found to be sufficient to reach equilibrium concentration of carbon dioxide in the box, and 20 minutes was set as a standard diffusion time.

From the dimensions of the test chamber, the change of concentration with time, and the thickness of the sample, diffusivity is then calculated and expressed in centimeters squared per second. Since the thickness of the sample is included in the calculation, this figure should be representative of the diffusivity of the structure of the material independently of the sample size. The amount of diffusion through a given structure will depend, of course, upon the diffusivity and sample thickness. The contract specifies that diffusivity should be as high as possible, consistent with other properties. Previous work indicated that diffusivity should be at least 2.0×10^{-2} centimeters squared per second. Some samples will be tested on this equipment and then referred to the Army Chemical Center for correlation of test results.

D.O.P. SMOKE PENETRATION

The procedure for determining smoke penetration is the same procedure set up by the project officer when the equipment was installed and calibrated in November, 1960. Penetration values are determined at a set flow rate of one liter per minute with the pressure drop noted in millimeters of water, and

at a set pressure drop of 5 millimeters of water with the flow rate noted in liters per minute. The contract specifies that the penetration of a 0.3 micron dioctyl phthalate smoke through the material shall not exceed 0.015% when tested at a pressure drop of 0.2 inch of water gage (5 mm.). In some cases, however, penetration at a flow rate of one liter per minute was almost negligible and the pressure drop was greater than five millimeters of water; in these cases it was not considered necessary or practicable to attempt a penetration measurement with a given pressure drop of five millimeters of water.

GAS LIFE

Protective effectiveness against toxic gases will be determined against cyanogen chloride gas by the Army Chemical Center. Samples will be kept in equilibrium with 80% R.H. and used with a test gas at the same R.H. and at a test temperature between 70 and 80°F. A test area of 100 sq. cm. will be used, with a gas concentration of 4 mg. per liter and a test flow to give 0.2 inch of water gage pressure drop or, if the flow under these conditions is less than one liter per minute, with a flow of one liter per minute. Under these conditions, a minimum protective life of 20 minutes is desired.

FORMATION OF BOARDS

UNTREATED BOARD

Two sets of six boards each were formed from the Minnesota and Ontario wet lap in order to make a preliminary test of the conditions necessary for the production of board with physical characteristics within the limits set in the contract. A quantity of wet lap was slushed at 4.0% in a Somat pulper and the proper amount for one board diluted to 0.7% in the deckle box. Drainage was effected with a water-leg of approximately three feet until the forming water level was even with the upper surface of the formed board, at which time a 20-inch Hg vacuum was applied until drainage was complete. Pressing and drying conditions, caliper, density, carbon dioxide diffusivity, and smoke penetration values are given in Table I. All of these tests were made without removing any surface skin.

It appears from these preliminary tests that a pressing condition of 10 minutes at 100 p.s.i. will yield a board with approximately the correct density and with a diffusion (without removing surface skin) slightly below specified value. It is known that the addition of carbon tends to improve the diffusion and consequently it was hoped that conditions adequate for evaluation of additives could be maintained without removing the surface skin. It also appears that the pressing and drying conditions on the commercial Minnesota and Ontario operation are approximately equivalent to 50 p.s.i. pressing under laboratory conditions. However, a better diffusion was obtained with the commercial board than would be expected.

TABLE I

PRELIMINARY FORMATION OF BOARDS FROM MINNESOTA AND ONTARIO PULP WITHOUT CHARCOAL

Sample No. 2256-	Wet Pressing		Drying		Caliper, in.	Density, lb./ft.^{3}	CO_2 Diffusivity, cm.^{2}/sec. x 10^{-2}	Smoke Penetration		
	Time, min.	Pressure, p.s.i.	Time, hr.	Temperature, °C.				Flow, l./min.	Pressure Drop, mm. H_2O	Penetration, %
10-1	15	50	overnight	59	0.207	17.4	-	1	17	0.002
10-2	10	50	overnight	59	0.204	18.0	-	-	-	-
10-3	5	50	overnight	59	0.208	17.5	-	-	-	-
10-4	15	25	overnight	59	0.230	16.1	1.66	1 0.270	12 5	0.023 0.003
10-5	10	25	overnight	59	0.254	15.2	-	-	-	-
10-6	5	25	overnight	59	0.234	14.9	-	-	-	-
12-1	10	100	overnight	59	0.228	21.5	1.57	-	-	-
12-2	5	100	overnight	59	0.248	20.3	1.88	1 0.090	31 5	<0.001 <0.001
12-3	10	100	overnight	59	0.236	21.4	1.56	1	56	0.001
12-4	10	100	overnight	59	0.225	21.1	1.57	-	-	-
12-5	10	100	overnight	59	0.238	21.3	-	-	-	-
12-6	10	100	overnight	59	0.256	21.1	-	-	-	-
19-1	Commercial sample M & O Board				0.50	17.6	2.63	-	-	-

BOARDS FORMED WITH CHARCOAL FURNISH

A set of seven boards was formed with 25% ASC Grade 1 Charcoal, based on ovendry fiber weight. No specific problems were encountered in handling the charcoal; it dispersed quite readily. Contact times between the charcoal and the water in the slurry were not noted specifically; the time required to produce all seven boards was approximately four hours from the addition of the charcoal to the slurry to the removal of the last board from the mold, with a time lapse of roughly one-half hour between the addition and the production of the first board--the boards were coded in the order of their formation.

The charcoal was added to a 4.0% stock slurry having a Canadian Freeness value of 645 cc. and a pH of 5.2. After the charcoal addition, the slurry was agitated for 15 minutes in a Somat repulper; a check on the pH after agitation gave a value of 7.3. The boards were formed in the sheet mold at a consistency of 1.0%. Drainage on one group was effected with the combination of a water-leg and a vacuum application in the manner previously explained; the other group was drained into a receiver under 20-inch Hg vacuum. Pressing and drying conditions, caliper, density, and carbon dioxide diffusivity values are given in Table II. Addition of carbon has caused a decrease in density at the same pressing conditions and an increase in diffusivity. Assuming that this change would be accompanied by an increase in smoke penetration, some boards were made by pressing to a slightly higher density.

TABLE II

BOARDS FORMED USING 25% CHARCOAL ADDITIONS

Sample No. 2256-	Drainage	Wet Pressing		Drying		Caliper, in.	Density, lb./ft.^{3}	CO_2 Diffusivity, cm.^{2}/sec.
		Time, min.	Pressure, p.s.i.	Time, hr.	Temp., °C.			
17-1	Water-leg	10	100	3.75	105	0.322	20.2	2.55×10^{-2}
17-2	Water-leg	10	100	2.25	105	0.319	20.35	2.53×10^{-2}
17-3	Water-leg	10	150	2.0	105	0.289	22.0	2.36×10^{-2}
17-4	Water-leg	10	150	16.75	105	0.292	21.85	2.45×10^{-2}
17-5	Vacuum	10	150	16.25	105	0.288	21.8	2.33×10^{-2}
17-6	Vacuum	10	150	15.75	105	0.311	21.15	2.43×10^{-2}
17-7[a]	Vacuum	5	200	15.25	105	0.250	20.90	1.96×10^{-2}

[a] Sample 17-7 was formed from the last of the stock slurry; the consistency was uncertain and composition may have varied.

THE INSTITUTE OF PAPER CHEMISTRY

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