A Laboratory Scale Dynamic Chamber Test for Formaldehyde in Air from Finished Fabrics

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ABSTRACT

Formaldehyde based agents are used in textile treatments to impart durable press (DP) properties, provide dimensional stability and serve as binders in pigment printing. Recent actions by regulatory agencies require the textile industry to monitor formaldehyde (HCHO) in air in the work environment. Development of methods to determine HCHO evolved from DP finished textiles has spanned decades, but there has been little research on methodology for measuring HCHO in air from textiles.

Of the methods for assessing HCHO evolved from fabric, AATCC Test Method 112, Determination of Formaldehyde Release from Fabric: Sealed Jar Method, is the test of commerce in the U.S. A method was devised to measure dynamic emission of HCHO from DP treated fabrics to the air. The results of the new method were compared to static methods. A commercial calibrator and air monitor that analyzes HCHO colorimetrically was used. Fabrics treated with various DP formulations were placed in a small chamber and air pulled into the analyzer at a rate equal to 0.9 exchanges per hour. The effects of differences due to sample size and treatment, process washing, hanging in a strong draft and cure conditions are measurable.

KEY TERMS

DMDHEU
Durable Press
Finishing Cotton
Formaldehyde Emission
Test Methods

Formaldehyde-containing agents are used in textile finishing to impart durable press (DP) properties, provide dimensional stability and serve as binders in pigment printing. Such finishes are relatively low in cost and the resultant fabric properties are durable through the life of the textile when the agent, catalyst and treatment conditions are carefully selected. Through the years, the quality of products and the amount of formaldehyde subsequently evolved has varied considerably. Over time, the industry has developed and modified finishes so that evolution of formaldehyde from such treated fabrics has decreased substantially. Development of methods to determine formaldehyde evolved from DP finished textiles has spanned decades. Of the many methods for assessing formaldehyde evolution from fabric, AATCC Test Method 112, Determination of Formaldehyde Release from Fabric: Sealed Jar Method (1), is the test of commerce in the United States. The results are expressed in μg of HCHO released per gram of fabric under the specified test conditions.

While the first major concerns over evolved formaldehyde in air focused on the wood processing industry, recent actions by regulatory agencies also require the textile industry to monitor formaldehyde in air in the work environment (2). Acute formaldehyde problems associated with wood and textile industry workers involve allergic reactions and dermatitis. Some studies propose that formaldehyde is a probable human carcinogen.

There has been limited research on methodology for quantifying formaldehyde emitted from fabrics to air. The wood industry was one of the leaders with various studies of dynamic chamber tests (3). A few studies involving textiles have been performed (4,5). The chamber used in this study is modeled after the one used in kinetic studies that were performed by Roberts and Rossano (6) who measured formaldehyde evolved in air from treated fabrics.

The purpose of this study was to investigate the measurement of formaldehyde in air evolved from well characterized fabrics using commercially available technology. We needed some preliminary information on what affects the measurement of formaldehyde in air before the industry tries to fine tune procedures for measuring trace amounts in air in the real world and struggles with the regulatory aspects of monitoring formaldehyde in the workplace.

Materials and Methods

Fabrics

The fabric was desized, scoured and bleached 100% cotton print cloth weighing approximately 3.2 ounces per square yard. The majority of samples studied were prepared with three formulations, which are given in Table I. The concentrations of the agent, dimethyloldihydroxyethyleneurea (DMDHEU), were obtained by diluting a 40% solids commercial solution. Magnesium chloride hexahydrate $(MgCl_2 \times 6H_2O)$, citric acid and ethylene glycol (EG) were reagent grade chemicals. All percentages are by weight.

In laboratory scale treatments, fabrics were padded with two dips and two nips to approximately 90% wet pickup. They were dried at 60C for seven minutes and cured at 160C for three minutes in forced draft ovens. A fabric set consists of samples of all three formulations produced in one day.

Another portion of the study was performed on a slightly different set of two fabrics. In this instance, fabrics were treated with 9% DMDHEU at 2.7 or 1.5% $MgCl_2 \times 6H_2O$ alone as catalyst. These specimens had been dried as above and cured from 60 to 180 seconds at 160C.

Fabrics were not washed after treatment unless stated otherwise. Washing

Table I. Fabric Formulations

Range of HCHO Expected[a]	DMDHEU (%)	Catalyst (%) (20/1 MgCl₂ × 6H₂O /Citric Acid)	Additive (2%)
High	9	2.7	None
Medium	5	1.5	None
Low	5	1.5	EG

[a]Based on levels of HCHO obtained in Sealed Jar Test (1).

Fig. 1. Effect of sample size of individual specimen on HCHO in air results in the dynamic chamber test: ■ High (9% DMDHEU); ▲ Medium (5% DMDHEU) and ● Low (5% DMDHEU + 2% ethylene glycol).

Fig. 2. Effect of extended time in dynamic chamber test on HCHO in air results of individual specimen: ■ High (9% DMDHEU); * Medium (5% DMDHEU) and + Low (5% DMDHEU + 2% ethylene glycol).

was conducted in a home type automatic washer using deionized water; washed samples were tumble dried. Fabric samples were stored in a plastic bag which was then wrapped in aluminum foil. When a sample was ready for testing, it was removed, a specimen cut and weighed, and the remainder returned to storage.

In one series of tests, treated fabrics were hung on lines in a laboratory hood and removed for testing at intervals between 24 and 192 hours. Replicated intervals included 24, 48 and 144 hours. In addition, a sample was included for either 120 or 192 hours, but not both. At the start of both series there were 12 samples large enough to supply duplicate specimens for testing by three HCHO analysis procedures. A sample from each of the three formulations was removed at four time intervals. Over the course of the experiment, the amount of fabric in the hood diminished. The hood was adjusted for maximum air velocity (263 linear ft/min). The rate of air exchanges per hour (ACH) was 7900.

The test for formaldehyde in air is

described below in general terms and in greater detail in the Appendix. Equipment consisted of a commercially available formaldehyde monitor and calibrator.

Test Chamber

Glass aquaria, each with a capacity of 33.5 liters air, were fitted with Plexiglas covers. Ambient laboratory air drawn through these chambers (see Appendix) produced zero HCHO readings. Fabric samples were tested under ambient conditions which averaged 69F ± 2.6F and 56% ± 4.7% relative humidity.

Monitor

CEA Instrument's Toxic Gas Monitor 555[1] (TGM) was used for this research. For HCHO determinations, three re-

[1]Names of companies or commercial products are given solely to provide scientific information and their use does not imply endorsement by the U.S. Department of Agriculture over others not mentioned.

agents are pumped into the instrument through Teflon tubing using a peristaltic pump and are mixed with air that enters through an internal vacuum pump. Formaldehyde sensitive reagents and sample air are mixed together and reaction time is controlled using one or more glass tubing coils. Reacted liquid moves into a spectrophotometer factory set to read HCHO concentration colorimetrically at 550 nm. Excess air is vented to the outside by the vacuum pump after it is scrubbed of all formaldehyde. At the set rate of 0.5 liters per minute, the air in the test chamber is exchanged 0.9 times per hour.

Calibrator

The gas monitor is standardized using CEA Instrument's SC-100 calibrator. A known formaldehyde source is heated and airflow is adjusted to deliver known HCHO concentrations in air (including
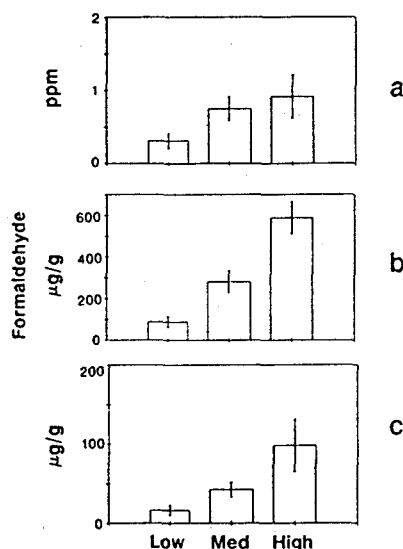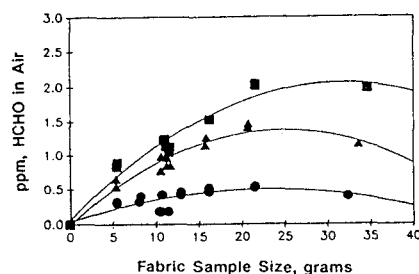

Fig. 3. Differences in formaldehyde levels analyzed from four sets of high, medium and low DMDHEU based finishes: results from (a) dynamic chamber test; (b) sealed jar test; and (c) steady state test.
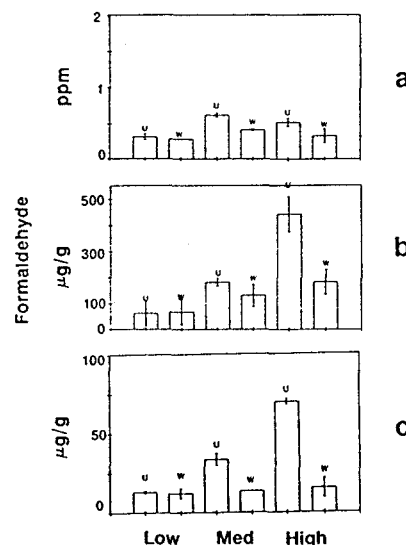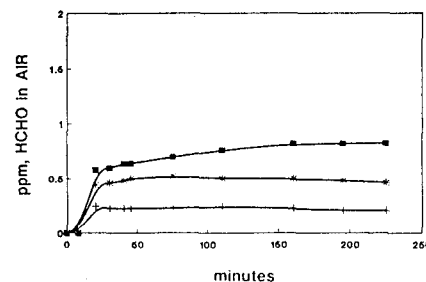

Fig. 4. Effect of one process wash on formaldehyde from high, medium and low DMDHEU based finishes: results from (a) dynamic chamber test; (b) sealed jar test; and (c) steady state test. U = unwashed; W = washed.
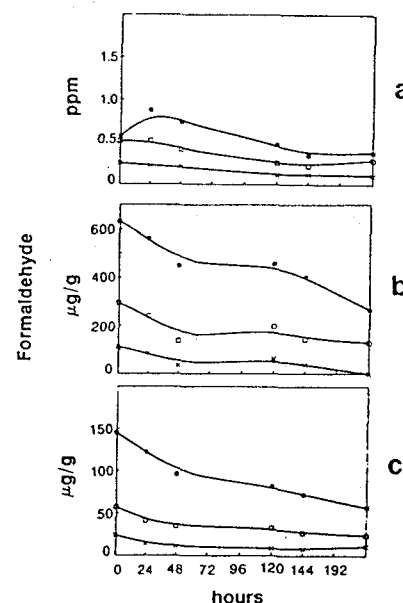

Fig. 5. Formaldehyde test results of high, medium and low DMDHEU based finished fabrics that were hung in hood for up to 192 hours: results from (a) dynamic chamber test; (b) sealed jar test; and (c) steady state test.

zero ppm) to the monitor. A standard curve drawn from the manufacturer's data on a scale of 0-5 ppm HCHO and a standard curve generated in this laboratory from 0-2 ppm, both are first order regression lines with correlation coefficients of $R = 0.99$ (not shown). Output from this calibrator was used daily to standardize at zero ppm in air and with 1 ppm HCHO in air set to read 50 on the TGM-555 digital readout and stripchart recorder.

Dynamic Chamber Test for Fabric

A weighed fabric specimen was hung in the chamber under static conditions for 30 minutes. The tubing was then attached to the HCHO monitor and the air in the chamber was pumped through the instrument for 45 minutes. If the digital readout fluctuated, readings were taken every 10 minutes until the readout stabilized. Final ppm of HCHO was twice the digital readout. Daily start up and clean up times were lengthy. A maximum of six specimens per day were tested.

Two static test methods were performed to compare with results found in the dynamic chamber test. One-gram fabric specimens were tested by AATCC Test Method 112 and steady state (7) formaldehyde analysis procedures. In TM112, samples were suspended over 50 grams water, sealed and heated at 49C for 20 hours. In the steady state test, samples were immersed in 50 grams water at ≤10C for 40 minutes. In both tests, color development using Nash reagent followed. Results of both tests are expressed as µg formaldehyde per gram fabric.

Results and Discussion

Fabric Sample Size

Preliminary testing was performed to learn what effect sample size had on the determination of HCHO in air from treated fabric by our method. Fabric samples from the three different formulations that weighed from approximately 5 to 30 grams each were tested. The largest single piece of unfolded fabric that could

fit into the chamber was approximately 9 × 16 inches and weighed 10 to 11 grams. The average sample size of 96 specimens was 10.40 ± 0.79 grams. Variations in sample total weights were divisions and multiples of this sized sample.

Fig. 1 shows the results when individual specimen sample size (g) was plotted against HCHO concentration (ppm) in air. There was an initial proportional increase in HCHO in air (ppm) as sample size increased to 15 grams for all three formulation levels. However, all 30-gram samples produced HCHO readings that were equivalent to or lower than those of the 20-gram samples, indicating that we were approaching chamber saturation. Regression lines on the figure are second order. The sample with the highest formaldehyde concentration (top curve) had the lowest correlation ($R = 0.784$). The other curves had R values >0.97. All subsequent samples were cut to approximately 9 × 16 inches to maintain a constant surface area.

Residence Time

We wanted to learn whether the readings would change significantly if a sample were held in the dynamic chamber for more than the standard 45 minutes. A specimen from each level of treated fabric was run up to five times longer. The results are shown in Fig. 2. No substantial differences were found for the low or medium HCHO level samples. There was a modest increase in the high HCHO level sample that will be investigated in the future.

Differences in Finish Formulation Level

With sample size held constant in dimension/weight and readings taken at constant 45 minute intervals, we examined the differences in HCHO evolved from the three finish formulations (low, medium and high level HCHO). The results of four sets of treated fabrics run in duplicate are given in Fig. 3. The standard deviations were ±0.10, 0.16 and 0.30 ppm respectively for the low to the high level finishes. Overall differences in the amount of formaldehyde in air (Fig. 3a) were measurable between the low level and the medium/high HCHO level fabric samples. Fabric finished with 2% ethylene glycol in the formulation (low level finish) had substantially lower HCHO in air values. We could not always distinguish between the medium and high level finishes. Averaged HCHO results of some individual sets of samples were highest for the medium level finish.

For comparison, the same fabric treatments were tested in the sealed jar (TM112) and steady state tests (Figure 3b and 3c). These tests were able to distinguish among the three HCHO levels. The standard deviations for the sealed jar test results were ±26, 52 and 77 µg/g; steady state tests had standard deviations of ±8, 11 and 36 µg/g for the low, medium and high HCHO level finishes, respectively.

Single Wash Cycle

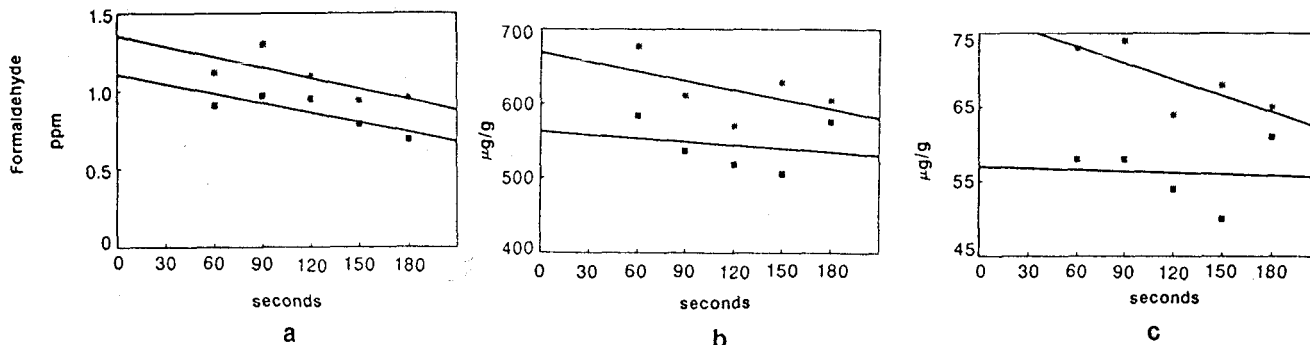Two sets of fabrics were prepared and one set was washed. All three formaldehyde

<table>
<tr><td colspan="5">Table II. HCHO in Air, Originals (Test 1) & Same Samples Rerun One Month Later (Test 2)</td></tr>
<tr><td rowspan="2">Cure Time (sec)</td><td colspan="2">2.7% Catalyst[a]</td><td colspan="2">1.5% Catalyst</td></tr>
<tr><td>Test 1</td><td>Test 2</td><td>Test 1</td><td>Test 2</td></tr>
<tr><td>60</td><td>1.12</td><td>0.58</td><td>0.91</td><td>0.34</td></tr>
<tr><td>90</td><td>1.30</td><td>0.60</td><td>0.97</td><td>0.36</td></tr>
<tr><td>120</td><td>1.10</td><td>0.56</td><td>0.95</td><td>0.30</td></tr>
<tr><td>150</td><td>0.94</td><td>0.68</td><td>0.79</td><td>0.38</td></tr>
<tr><td>180</td><td>0.96</td><td>0.60</td><td>0.69</td><td>0.26</td></tr>
</table>

[a] 9% DMDHEU with $MgCl_2 \times 6H_2O$

Fig. 6. Effect of varying cure time on formaldehyde tests results with two 9% DMDHEU finishes (* 2.7% and ■ 1.5% $MgCl_2 \times 6H_2O$ catalyst): (a) dynamic chamber test; (b) sealed jar test; and (c) steady state test.

Fig. 7. Relationship between formaldehyde data found in sealed jar test *vs* ppm formaldehyde in air from the dynamic chamber test.

tests were performed. The results of duplicate samples are shown in Fig. 4. Washing lowered the HCHO in air slightly (Fig. 4a). Here was an example of a medium level finish producing larger HCHO in air readings than the high level finish. The dynamic chamber test could not consistently distinguish between those two treatments. On the contrary, greater differences were recorded in the sealed jar (Fig. 4b) and steady state (Fig. 4c) test results. This means that the static tests might predict a greater benefit due to washing than would be found in a real time, dynamic test.

Hanging Fabrics in Strong Drafts

In a second experiment involving time, samples were hung in a laboratory hood for up to 192 hours before testing. There were some differences that might be explained because sample placement inside th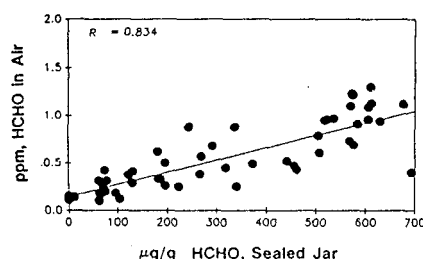e hood was random. For example, it is likely that samples hung in the front were blown harder than those placed in the middle. The HCHO in air results are shown in Fig. 5a. The rapid air exchanges that occurred in this environment produced noticeable changes in ppm HCHO in air evolved from treated fabrics. Not surprisingly, the greatest loss of HCHO was found with the high level finish samples, and samples from all three finish formulations showed lowered evolution of HCHO over time. Only small differences were measured after 120 hours in the hood. One set of fabrics showed an initial rise in HCHO when hung in the hood and then tested in the chamber test (24 to 48 hours). Perhaps we are measuring a degree of vapor transport of formaldehyde as described by Reinhardt (8). Further tests may help us understand how this could occur. Other researchers have reported formaldehyde evolution from fabric that peaked over time in a dynamic test (9).

The results for the same treatments tested by the sealed jar and steady state methods are shown in Figs. 5b and 5c. Static results may level off faster than those in air. Sealed jar and steady state formaldehyde levels did not increase during the first 24 hours as did one set in the dynamic chamber test.

Cure Duration

A series of fabrics that were cured at various intervals from 60-180 seconds were tested for HCHO in air values. Only two formulations were used for the cure study—9% DMDHEU with two concentrations of $MgCl_2 \times 6H_2O$ (without citric acid) as catalyst. The results of the chamber test are shown in Fig. 6a and indicate a general lowering of HCHO in air level as cure times increased for each finish. Fig. 6b shows the sealed jar and Fig. 6c the steady state HCHO results for the same fabrics. These two tests showed the same general trend.

We had occasion to put the *same* test specimens from the cure study back into the chamber and run them again. These results are shown in the Table II. Test 2 samples had been stored approximately one month after the first chamber test. We found the formaldehyde in air values were less than half when a specimen was retested a second time. Similarly, Vail and Andrews (10) reported lowered sealed jar results when specimens were retested.

Dynamic Chamber vs Static Test Results

We plotted the HCHO in air values against those from the sealed jar test for all standard sized specimens. The result is shown in the Fig. 7. Included in this figure are all samples regardless of protocol—i.e., washed, hung in hood, cured differently—as long as they were tested for 45 minutes in the chamber. The correlation was high ($R = 0.834$) considering that the tests really do not measure the same thing. The sealed jar test measures both hydrolyzable and free formaldehyde and the chamber test measures the real time evolution of formaldehyde.

A single plot containing all the values of HCHO in air *vs* steady state results had a poor correlation coefficient ($R = 0.5$) (not shown). However, the data obviously were grouped in two sections, those samples that received timely testing and those that were tested after storage. When the two groups were plotted individually, the correlation coefficients of both were >0.7. We previously reported that samples which were sealed, stored and then tested had increased steady state formaldehyde values (11). We found similar results. This area will be the subject of a future study.

Summary

Using only finishes based on DMDHEU, we produced samples with three levels of

Appendix

- **Chamber:** Three glass aquaria holding 33.5 liters of air were purchased. Plexiglass lids were fabricated, a handle was installed and 20 holes (2 mm) were drilled at one end to allow outside air to be drawn into the chamber. The chamber lids were not coated. Zero formaldehyde was measured in a frequently used chamber after sitting overnight. A hole large enough for an inverted glass Y tube to fit was drilled in the other end. The top, single arm was connected to the instrument with Teflon FEP tubing. Inside the chamber, the two arms were fitted with Teflon tubing long enough to approach the floor of the chamber but not touch it. Fabric specimens were hung lengthwise in the chamber suspended by alligator clips attached to strips of Teflon. The strips were draped over the chamber's side and held in place by the lid.

- **Toxic Gas Monitor TGM 555:** The formaldehyde analysis reagent system module was factory installed. Formaldehyde in the air is absorbed in a solution of tetrachloromercurate in sodium sulfite. Pararosaniline (acid bleached) is added and the resultant color is measured at 550 nm. This procedure is based on one by Lyles et al. (12). They claim no known interferences of significance. The output can be read on a digital display and/or sent to a stripchart recorder.

- **SC-100 Calibrator:** Permeation tubes containing HCHO are heated in an internal oven set at constant temperature (in this study it was 100C) and airflow is adjusted to dilute the gas and deliver precise and reproducible concentrations of HCHO in air to the analyzer. Using special chemical cartridge scrubbers, zero HCHO in air can also be delivered.

  The system is cleaned daily by running deionized water through the three reagent lines. In addition, household strength hypochlorite bleach is hand pumped through the glass coils and the spectrophotometer cells to remove any precipitated color which can introduce error. Deionized water flushes the last of this agent from the system.

formaldehyde release in finished print cloth. According to the manufacturer's directions we ran the toxic gas monitor and calibrated it with known concentrations of formaldehyde from permeation tubes. We built sample chambers and learned that we could monitor formaldehyde in the chamber under dynamic conditions when treated fabric was present. Concentrations of HCHO in air were generally stabilized after 45 minutes and remained basically unchanged up to four times longer if they were not read right away.

Differences based on sample size and evidence of a chamber loading effect were noted. When a standard sample size was used, we could observe differences in HCHO concentration due to finish formulation, exposure to strong drafts, a process washing cycle and cure times. Retesting of specimens after storage gave lower HCHO in air results.

With these limited experiments, we compared results from this dynamic chamber test to those of the sealed jar and steady state formaldehyde tests. Correlations were good. Work with the dynamic chamber will proceed with testing under standard conditions of temperature and relative humidity. In the future, studies of finishes from other formaldehyde based reactants, experiments using a lower temperature in the calibrator to improve the accuracy of standards at lower ppm levels and measurements of effects of storage will be performed. ○○○

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⧗ I Remember When

● In the 1950s, I was in a small plant in Rhode Island, printing cotton, rayon, Bemberg and nylon for apparel. A customer wanted a nylon plissé (a fabric with a randomly distributed pucker) which was made by printing a pattern of a paste containing phenol which would cause the nylon to shrink in the printed areas and create the plissé effect. From the odor, workers coming into the plant thought they were entering a hospital ward. There was no EPA back then. Little thought was given to pollution, so the unused paste ended up downstream.

GEORGE T. ROBERTS
Taylors, S. C.

● In 1932, I was working for Dutchess Bleachery in Wappingers Falls, N. Y., where in a minor capacity I was associated with the very early development of continuous J-box peroxide bleaching, even before the original patent was issued to BECCO (Buffalo Electro-Chemical Co.). In fact, no operating millman had an earlier start with this form of bleaching than I did.

The basic laboratory work of the BECCO process, which led to the granting of the original patent, was carried out at Dutchess as follows.

At first, swatches of cloth were saturated with various concentrations of the chemical solutions and steamed for various times and temperatures in a small autoclave. As the helper, I was directed by BECCO personnel. Then, the metal shop at Dutchess constructed a small J-box that was attached to a steam line provided with a temperature gauge. The J-box could be buttoned-up to contain the steam for various lengths of time. To feed the cloth through the saturating chemical tank and into the little J-box, I manually turned a set of squeeze rolls from an old fashioned home laundry setup. Once again, a long series of trial variations were made in the chemical saturating bath, the steaming conditions and the timing. This simple approach established the basic data for the BECCO patent.

Later we installed the big BECCO continuous J-box system at Dutchess and the entire installation was most successfully performed by Dutchess' own shop personnel. I, personally, did all the engineering drawings for that installation and supervised the work as it progressed. (Mr. Damon sent a copy of the *BECCO ECHO*, Vol. 1, No. 1, August 1950, which details this development at Dutchess Bleachery and his contributions to it.)

During the construction of Deering Milliken's Magnolia Plant at Blacksburg, S. C., I was technical advisor in the design and construction of the plant and became its first technical manager, entrusted with establishing the Operating Manual and getting the plant started up. I had a very long and notable career with Milliken and am now 81 years of age and retired 16 years ago.

A. DURFEE DAMON
Hampton, N. H.

● When the textile industry first started to resin treat fabrics in the late 1930s, the commonly used resins were urea formaldehyde (UF) pastes and syrups, as well a malamine formaldehyde (MF) powder and syrups. When applied to 100% cotton and 100% rayon, they produced wrinkle and shrink resistant fabrics.

Such resin products were sold to textile mills by a number of chemical manufacturers. In addition, the British firm Tootal Broadhurst Lee, which had invented the process for treating cellulose with UF resins, licensed its manufacturing technology to textile mills.

Initally, UF and MF resins served admirably, but were gradually replaced by products having improved chlorine resistance and washfastness, reduced odor and longer shelf life. Among those improved reactants were triazones, modified triazones, ethylene urea, urons, propylene urea and carbamates, as well as many more.

In the late 1950s, Sun Chemical Corp. (now Sequa Chemicals) introduced the imidazolidone type resin which is now generally known as the glyoxal or DMD-HEU type. Through many modifications and improvements over the next 35 years, the glyoxal type gradually replaced all of the other types and has now captured virtually 100% of the textile resin market.

HERMAN B. GOLDSTEIN
Chester, S. C.

- Finishing for Added Properties
- Effects of Dye Substantivity
- Drycleaning Fastness of Pigments
- Measuring Formaldehyde Emissions
- Direct Dyes and Their Application

The Magazine of the Wet Processing Industry

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