

Unclassified

SECURITY CLASSIFICATION OF THIS PAGE

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| <p>→ The U.S. Army Natick Research, Development, and Engineering Center is interested in developing high stacking strength containers to improve space utilization in warehousing and shipping. This contract examined the opportunity to add stacking strength by impregnating a "stiffener" resin into the container linerboard. A successful resin treatment was developed (one of four treatments evaluated) composed of a food grade mixture of sodium silicate and kaolin clay. The treatment added approximately 12% to 14% to the basis weight of the linerboard.</p> <p>Doublewall corrugated containers, measuring 20" x 12" x 10", were manufactured from this treated linerboard and a wet strength corrugating medium, which was not impregnated. Quantities of the containers were submitted to an independent laboratory and to the Natick RD&amp;E Center for testing. To assess</p> |       |                                              |                                                                                                                                                                 |                                                    |                                 |
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16. (cont'd)

Chemical Mixing -

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Container board Impregnation/  
Treatment

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(219) 936-9971

Corrugating

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Physical Testing

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19. (cont'd)

container strength characteristics under environmental extremes, contract compression strength objectives were established for standard conditions (73 degrees F (23°C) & 50% relative humidity (RH)) and for high temperature/high humidity or tropic conditions (100 degrees F (38°C) & 90% RH). Test results from both laboratories found the containers to meet or exceed the contract objectives:

Full Box Compression Values -

Standard Conditions: Objective 1,800 pounds, Test Result 2,205 pounds

Tropic Conditions: Objective 1,000 pounds, Test Result 1,060 pounds.

## SUMMARY

Four different resin treatments were studied as alternatives to create a corrugated container that would sustain high compression loads under standard and high temperature/high humidity conditions. While all four alternatives demonstrated superior compression strength under standard conditions, only one alternative (a treatment of sodium silicate and clay solids) met the compression strength objective of the contract. This container was constructed using a corrugated doublewall, C/B flute combination, dimensions 20" x 12" x 10". The container components were:

|                    |                                                                              |
|--------------------|------------------------------------------------------------------------------|
| Outer liners       | 42-lb linerboard, treated with 12%-14% add-on of sodium silicate/clay solids |
| Middle liner       | 26-lb linerboard, treated with 17% add-on of sodium silicate/clay solids     |
| Corrugating medium | 40-lb wet strength(WS), not treated                                          |

The compression test results appear below.

### Full Box Compression Test Objectives and Results

|                                                                              | <u>Contract Objective</u> | <u>Actual (Treated)</u> |
|------------------------------------------------------------------------------|---------------------------|-------------------------|
| Standard Conditions<br>(73 degrees F (23°C) &<br>50% relative humidity (RH)) | 1,800 lb.                 | 2,205 lb.               |
| Tropic Conditions<br>(100 degrees F (38°C) & 90% RH)                         | 1,000 lb.                 | 1,060 lb.               |

### Chemical Treatment

The linerboard was impregnated with a mixture of sodium silicate and kaolin clay. The saturant was approximately 62% water, 34% sodium silicate, and 4% kaolin clay. The treatment levels were targeted at between 10% and 14% add-on of the basis weight. The corrugating medium was not treated. The resin add-on resulted in a 7% increase in the weight of the combined corrugated board.

The impregnation of chemicals into the linerboard was accomplished using the newly developed MiPly saturation process (U.S. Patent No. 4,588,616 issued May, 1986).

### Corrugation of the Treated Board

The corrugated board was constructed in a C/B flute configuration. The run speed of the corrugator was approximately 200 feet per minute. A special adhesive formulation (National Starch No. 29-9551) was used to combine the treated linerboard.

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## PREFACE

This project to develop a high-stacking strength container was undertaken by MiPly Equipment, Northfield, IL 60093, during the period March 1987 to February 1988 under contract DAAK60-87-R-007 of the U.S. Army Natick RD&E Center, Natick, MA 01760-5018. The funding was under Program element 728012.19, Project no. 537000, Support for the DOD Food Program.

The Natick Project Officer was Mr. Anderson Miller.

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## DEVELOPMENT OF A HIGH-STACKING-STRENGTH CONTAINER

### BACKGROUND

The project entitled Develop A High-Stacking-Strength Container (DHSSC) was undertaken to develop a corrugated container that would withstand high compression loads at standard and high temperature/high humidity conditions.

The need for improved container stacking strength stems from changes in the nature of the unit packaging within the container. Traditional unit packaging has been cans or glass bottles. These cans or bottles would carry the weight of package contents in inventory stacks and during shipment. The role of the corrugated container was truly that of a container, to keep the contents together during shipment. In recent years unit packaging has shifted more to paper and plastic which provide little or no compression strength. The source of stability in inventory stacks and in loads during shipment has therefore shifted to the outside container, the corrugated box. When the outside container is not capable of supporting multiple pallet loads in inventory stacks, pallet support systems (racks) are required. These systems are expensive, take up space, and restrict handling.

High-stacking-strength corrugated containers have been successfully manufactured in the past using combinations of (1) high-weight linerboard and/or corrugating medium, (2) multiple wall structures, and (3) liners and/or dividers within the box. These alternatives all depend on additional fibre, add additional weight to the container, and take up space. Further, the cost of fibre has risen dramatically over the last few years, from \$290/ton (\$.145/lb.) in June, 1986 to \$410/ton (\$.205/lb.) currently.

In this DHSSC project, the potential of chemical additives is examined as an alternative to additional fibre. While it has been understood for some time that additives to paper may provide considerable strength increases in the linerboard, chemical additives may also create brittleness, cracking, and bonding problems. Some chemicals present toxicity concerns in manufacturing, use, and disposal. The DHSSC project objectives required any chemicals used to be food grade and limited the add-on level to 10% of the combined board to minimize scoreline cracking.

### SATURANT CHEMISTRY

#### Many Candidate Chemistries -

An extensive history of chemical treatments to paper exists, primarily from the construction products industry. Additionally, MiPly has conducted a search of the U.S. Patent Office and found 39 patented formulations (Table 1) specifically for the purpose of being impregnated into paper to enhance its characteristics.

While many different chemistries are viable candidates for adding strength to paper, most that have been identified include phenolic resins as a component. Phenolic resins build considerable strength and are largely impervious to moisture. However, at the time of this project none of the identified phenolic resin saturants had been accepted by the Food & Drug Administration (FDA) for use in food packaging. Application for FDA approval was beyond the time scope of this project.

TABLE 1. List of Patented Saturant Chemistries  
and Their Holders

| <u>Patent No.</u> | <u>Holder(s)</u>      |
|-------------------|-----------------------|
| 2,049,217         | Meunier               |
| 2,567,097         | Berglund              |
| 2,672,427         | Bauling et al         |
| 2,709,141         | Burks, Jr.            |
| 2,739,908         | Marsh                 |
| 2,786,786         | Nova et al            |
| 2,808,350         | Seiler                |
| 3,009,829         | Gouveia               |
| 3,269,860         | Richardson et al      |
| 3,346,443         | Elmer                 |
| 3,560,328         | Anderson et al        |
| 3,676,055         | Smith                 |
| 3,676,182         | Smith                 |
| 3,793,057         | Wheelock              |
| 3,914,518         | Haskell               |
| 3,934,067         | Goldman et al         |
| 3,936,561         | Cotton                |
| 3,989,416         | Louden                |
| 4,002,785         | Grossmann et al       |
| 4,024,307         | Brahm et al           |
| 4,058,648         | Louden                |
| 4,071,651         | Hicklin et al         |
| 4,119,746         | Bleyle                |
| 4,123,592         | Rainer et al          |
| 4,158,712         | Degens                |
| 4,212,916         | Tamaka et al          |
| 4,242,380         | Courtroy              |
| 4,245,744         | Daniels et al         |
| 4,246,311         | Hirst                 |
| 4,318,963         | Smith                 |
| 4,342,805         | McCartney             |
| 4,343,403         | Daniels et al         |
| 4,362,778         | Andersson et al       |
| 4,376,148         | McCartney             |
| 3,870,557         | Fink et al            |
| 4,407,697         | Sadler et al          |
| 4,423,112         | Luthringshauser et al |
| 4,496,624         | McCartney             |
| Japanese          | 52-68722              |

### Sodium Silicate -

As an alternative to phenolic resins, MiPly Equipment has conducted extensive trial work with sodium silicate as a stiffening agent for packaging. Sodium silicate has been used in the packaging industry for many decades, primarily as an adhesive. Food and Drug Administration (FDA) approval is below on the Code Federal Regulations (CFR).

Relevant information from FDA regulations, Volume 21, Subpart D "Specific Usage Additives"

#### 21 CFR 175.390

Lists sodium silicate as an optional substance, which may be used in the formulation of coatings used as the food-contact surface of articles intended for use in handling food.

#### 21 CFR 182.90

Lists sodium silicate as a substance which may migrate to food from paper and paperboard products used in food packaging.

Sodium silicate is available in many grades, each grade being a variation of the ratio between silicate ( $\text{SiO}_2$ ) and soda ( $\text{Na}_2\text{O}$ ). Generally, the higher the ratio of silicate to soda, the larger the crystal formed and the stronger the paper. For this DHSSC project two different silicates were selected: (1) 3.2  $\text{SiO}_2:\text{Na}_2\text{O}$  and (2) 3.8  $\text{SiO}_2:\text{Na}_2\text{O}$ . The 3.2 silicate is commonly available and has been the basis of most of MiPly's work with silicate. The 3.8 silicate is a specialty grade which is manufactured only on request. Constructing containers alternately from linerboard treated with 3.2 and 3.8 silicate would test the potential for additional strength from the special grade.

Tests of the repulpability of paper treated with sodium silicate were conducted by The Black Clawson Company, Fulton, NY with the finding that, for treatment levels under 20%, there is no difference between treated and untreated stocks.

### Moisture Sensitivity of Sodium Silicate -

Sodium silicate does, however, have its drawbacks. Silicate is hygroscopic, it attracts moisture. In the presence of adequate moisture the crystalline structure dissolves and the strength enhancement is lost. Conversely, in the absence of moisture the silicate crystal becomes very brittle. Paper that is treated with sodium silicate and subjected to very low humidity will become excessively brittle and subject to cracking. Finally, due to its rapid moisture pickup, silicate-treated paper is difficult to bond. The treated paper absorbs the moisture from the adhesive too rapidly, leaving the adhesive solids on the surface. The hygroscopic nature of silicate must be modified to be useful as a saturant for corrugated containers.

### Clay As An Additive To Silicate -

Many chemistries offer good potential as an additive to sodium silicate. Not surprisingly, phenolic resins are at the top of the list. One such promising alternative is provided by an adhesive formulation used in the manufacture of

plywood. This formulation, which provides a "high temperature and water resistant fluid adhesive," is described in Canadian Patent No. 689,002 (issued June 16, 1964, now expired) granted to United States Plywood Corporation. This formulation calls for a phenol-formaldehyde and silicate composition. Upon inspection, the patent reveals that the silicate is first mixed with a "filler clay", the phenol resin added later. The phenol-formaldehyde is added to provide a thermosetting capacity to the adhesive. We suspect that the alumina in the clay provides a reaction which displaces the soda in the silicate, providing reduction in moisture sensitivity. If we were to make a saturant solution, such as described in this patent but without the phenol-formaldehyde, we may find superior moisture stability, less moisture degradation of compression strength, and superior bonding characteristics.

Therefore, as an alternative to sodium silicate alone, a formulation of 3.2 silicate and kaolin clay was proposed. Kaolin clay is a Generally Recognized As Safe material, FDA Reg. No. 21 CFR 186.1256. The proposed formula is given below.

#### Sodium Silicate - Kaolin Clay Mixture

|                            |     |
|----------------------------|-----|
| 3.2 sodium silicate solids | 34% |
| Georgia kaolin clay        | 4%  |
| Water                      | 62% |

### **CHEMICAL PREPARATION**

#### 3.2 Sodium Silicate

The 3.2 sodium silicate is commonly used by the saturating facility, Menser Industries, Plymouth, Indiana, and is maintained there in large quantity. At the time of the saturation work the necessary 3.2 sodium silicate was taken from the Menser stores.

This saturant was used to provide the treatment for Box #1.

#### 3.8 Sodium Silicate

The 3.8 sodium silicate is a specialty grade, manufactured only on order. Order inquiries were made with The P.Q. Corporation and Occidental Chemical. The P.Q. Corporation expressed reservations about the material remaining in solution during shipment and storage awaiting the impregnation into linerboard. Occidental Chemical expressed a higher level of confidence in maintaining the solution. Therefore the order was placed with Occidental Chemical, Chicago, Illinois 60693, for 4,000 pounds of #34 silicate of soda, which was delivered to Menser Industries in seven 55 gallon drums.

This saturant was used to provide the treatments for Boxes #3 and #4.

### 3.2 Sodium Silicate - Kaolin Clay Mixture

This mixture had been under consideration by Menser Industries and some limited laboratory work had been completed prior to the award of this contract. A serious concern was to maintain a sufficiently small particle size for the clay during mixing and while in solution. Preliminary work had revealed the tendency of the clay to form larger aggregates when mixed with the silicate solution.

As a first step it was decided to attempt mixing a small quantity of the silicate/clay saturant, impregnate the mixture into linerboard, and analyze the results. The impregnation would be made at several different add-on levels (5%, 10%, and 15%) to assess the relationship between add-on level and strength improvement. This work was undertaken in April, 1987 and submitted for laboratory testing at the Container-Quinn Testing Laboratories, Inc., Wheeling, Illinois.

The compression strength of the linerboard was tested, using the standard 6" ring crush test<sup>1</sup>, at standard (73 degrees F (23°C) & 50% relative humidity (RH)) and high temperature/high humidity (90 degrees F (32°C) & 90% RH) conditions. While the high temperature level was not as high as that required in the DHSSC (100 degrees F (38°C)), it was considered adequate to evaluate the treatment at this preliminary stage. The Container-Quinn test report is presented as Appendix A.

#### Analysis of Test Results:

##### Preliminary Treatment With Silicate/Clay Saturant

The following ring crush values are given for cross machine direction (CMD).

TABLE 2. Compression Strength Tests of Linerboard  
Treated with Sodium Silicate/Clay Saturants

| Add-on<br>Percent                                   | Add-on<br>Lb/MSF* | Pounds Per 6 Lineal Inches |                        |             |                               |
|-----------------------------------------------------|-------------------|----------------------------|------------------------|-------------|-------------------------------|
|                                                     |                   | Measured<br>Ring Crush     | Expected<br>Ring Crush | Improvement | Improvement<br>Per Lb. Add-on |
| <u>A. Standard Conditions</u>                       |                   |                            |                        |             |                               |
| 5%                                                  | 2.1               | 96                         | 74                     | 22          | 10.5                          |
| 10%                                                 | 4.2               | 122                        | 74                     | 48          | 11.4                          |
| 15%                                                 | 6.3               | 146                        | 74                     | 72          | 11.4                          |
| <u>B. High Temperature/High Humidity Conditions</u> |                   |                            |                        |             |                               |
| 5%                                                  | 2.1               | 47                         | 33                     | 14          | 6.7                           |
| 10%                                                 | 4.2               | 58                         | 33                     | 25          | 6.0                           |
| 15%                                                 | 6.3               | 68                         | 33                     | 35          | 5.6                           |

\* Thousand square feet

These results were very encouraging. MiPly noted that untreated linerboard is expected to retain approximately 45% of its compression strength under high temperature/high humidity (33 out of 74). The strength enhancement due to the silicate/clay treatment maintained approximately 55% (ave. 6.1 out of ave. 11.1). Under previous testing, sodium silicate alone has been shown to be severely sensitive to moisture. It is apparent that the clay is improving the moisture resistance of the treated linerboard.

Disappointing results were (1) the apparent pattern of reducing moisture resistance at the higher add-on level; 64% retention at 5% add-on (6.7 out of 10.5) compared to 49% retention at 15% (5.6 out of 11.4), and (2) the appearance of substantial clay on the surface of the treated linerboard. It appeared that during treatment much of the clay was being filtered out onto the surface. Perhaps, at the higher treatment levels, a higher percentage of clay was filtered in this manner.

To overcome the filtering effect, the clay would have to be prevented from aggregating into the larger particle size during mixing with the silicate. Menser Industries does not have the specialized equipment for such mixing. A jobber was sought out and the Niles Chemical Paint Company, Niles, Michigan was selected. Niles Chemical employed a Cowles High Speed Disperser mixing unit, which successfully maintained the clay at its original particle size.

To facilitate the handling of the silicate/clay mixing, Niles Chemical mixed the components at high concentration. This concentrated mixture (at very high viscosity) was shipped to Menser Industries where it was diluted with silicate solution until a ratio of silicate solids to clay of 7.3:1 (88% silicate solids to 12% clay solids) was achieved in a 30% solids aqueous solution.

This mixture, then, served as the saturant solution to be impregnated into linerboard for Box #2.

#### CHEMICAL IMPREGNATION OF LINERBOARD

##### The Difficulties With Sodium Silicate -

The impregnation of sodium silicate into paper has been an impossible task - at reasonable speeds and cost - until the development of the MiPly saturation technology (U.S. Patent No. 4,588,616 issued May, 1986). The difficulty in impregnating sodium silicate is due to its high viscosity, where the viscosity is extremely sensitive to moisture content. When sodium silicate comes into contact with the fibers of the paper, the fibers immediately absorb a substantial portion of the water of the sodium silicate solution. The silicate turns into a gelatinous mass, which presents a barrier to further impregnation. The following table illustrates how dramatically the viscosity changes as the percent solids changes with water gain or loss.

TABLE 3. Moisture Sensitive Viscosity of Sodium Silicate

| <u>Percent Solids</u> | <u>Viscosity (Centipoise)</u> | <u>Pounds Water/MSF (Loss) Gain</u> |
|-----------------------|-------------------------------|-------------------------------------|
| 41%                   | 2,000 plus                    | (.98)                               |
| 40                    | 710                           | (.50)                               |
| 39*                   | 385                           | -                                   |
| 38                    | 185                           | .53                                 |
| 37                    | 120                           | 1.08                                |

\*The 3.2 sodium silicate is manufactured and delivered as a 39.3% solids solution.

#### The MiPly Saturation Technology -

The MiPly saturation technology is simply a very powerful method which pushes the silicate solution into the paper so rapidly that water loss is not a factor. The MiPly technology was a recipient of the IR-100 AWARD as one of the 100 most significant new technologies developed, worldwide, in 1986.

Menser Industries, a licensee of MiPly Equipment, has constructed the first operating process line utilizing the MiPly process. Menser has provided demonstration and research trials for companies interested in chemically enhanced papers for their product areas. Some fifteen companies with corrugating divisions have conducted trials at the Menser facility.

#### The Chemical Treatment -

Once all chemistries were available for impregnation, Menser Industries treated the 42-lb. and 26-lb. linerboard stocks. In the treatment process, the chemical saturant is impregnated into the board through one surface and driven toward the other surface. The depth of penetration depends on the volume of chemical to be added and the solids percentage of the saturant solution. In the case of the stock treated under the DHSSC, penetration is expected to be approximately 60% of the way through the stock. Where the fibers of the linerboard are not so densely packed, the penetration will likely proceed all the way through the stock. This will create a linerboard with one surface heavily treated and the other surface either untreated or only very modestly treated.

Preparation of linerboard stock with the four different treatments for each of the three unwind stations of the corrugator created 12 separate rolls of treated material, as listed in the following table. The linerboard stocks used varied in width from 63-3/8" to 75" due to the odd lot purchasing that was necessary for the project.

TABLE 4. Treated Materials for Corrugating

| Roll<br>No. | Linerboard*<br>Basis Weight | Chemical**<br>Treatment | Width   | Length |
|-------------|-----------------------------|-------------------------|---------|--------|
| 1           | 42#                         | #42 SS 10%              | 63 3/8" | 4,500' |
| 2           | 42#                         | #42 SS 10%              | 63 3/8" | 4,500' |
| 3           | 42#                         | #34 SS 14%              | 66 5/8" | 4,500' |
| 4           | 42#                         | #34 SS 14%              | 66 5/8" | 4,500' |
| 5           | 42#                         | #34 SS 14%              | 66 5/8" | 4,500' |
| 6           | 42#                         | #34 SS 10%              | 66 5/8" | 4,600' |
| 7           | 42#                         | #34 SS 10%              | 66 5/8" | 4,600' |
| 8           | 26#                         | #42 SS 10%              | 63 3/8" | 5,500' |
| 9           | 26#                         | #34 SS 10%              | 66 5/8" | 4,500' |
| 10          | 26#                         | #42 SC 10%              | 75"     | 5,000' |
| 11          | 42#                         | #42 SC 10%              | 75"     | 5,000' |
| 12          | 42#                         | #42 SC 10%              | 75"     | 5,000' |

\* 42# is 42 pound per 1,000 square feet(MSF) linerboard  
 26# is 26 pound per 1,000 square feet(MSF) linerboard

\*\* #42 SS 10% is Occidental Chemical Product No. 42 silicate of soda (3.2  $\text{SiO}_2\text{:Na}_2\text{O}$ ) with a target add-on of 10% of the linerboard basis weight.  
 #34 SS 14% is Occidental Chemical Product No. 34 silicate of soda (3.8  $\text{SiO}_2\text{:Na}_2\text{O}$ ) with a target add-on of 14% of the linerboard basis weight.  
 #34 SS 10% is Occidental Chemical Product No. 34 silicate of soda (3.8  $\text{SiO}_2\text{:Na}_2\text{O}$ ) with a target add-on of 10% of the linerboard basis weight.  
 #42 SC 10% is Occidental Chemical Product No. 42 silicate of soda (3.2  $\text{SiO}_2\text{:Na}_2\text{O}$ ) mixed with Georgia Kaolin Clay with a target add-on of 10% of the linerboard basis weight.

TABLE 5. Schedule for Combining Treated Rolls on the Corrugator

| Treatment        | Outside<br>Liner | Corr.<br>Medium | Inside<br>Liner | Corr.<br>Medium | Outside<br>Liner |
|------------------|------------------|-----------------|-----------------|-----------------|------------------|
| <u>Box No. 1</u> |                  |                 |                 |                 |                  |
| 275# C/B flute   |                  |                 |                 |                 |                  |
| #42 SS 10%       | 42#              | 40#WS           | 26#             | 40#WS           | 42#              |
| Roll No.         | 1                |                 | 8               |                 | 2                |
| <u>Box No. 2</u> |                  |                 |                 |                 |                  |
| 275# C/B flute   |                  |                 |                 |                 |                  |
| #42 SC 10%       | 42#              | 40#WS           | 26#             | 40#WS           | 42#              |
| Roll No.         | 11               |                 | 10              |                 | 12               |
| <u>Box No. 3</u> |                  |                 |                 |                 |                  |
| 275# C/B flute   |                  |                 |                 |                 |                  |
| #34 SS 10%       | 42#              | 40#WS           | 26#             | 40#WS           | 42#              |
| Roll No.         | 6                |                 | 9               |                 | 7                |
| <u>Box No. 4</u> |                  |                 |                 |                 |                  |
| 350# C/B flute   |                  |                 |                 |                 |                  |
| #34 SS 14%       | 42#              | 40#WS           | 42#             | 40#WS           | 42#              |
| Roll No.         | 3                |                 | 4               |                 | 5                |

## CORRUGATING ADHESIVE SEARCH

Prior work with sodium silicate has revealed a difficulty in bonding a silicate treated board using the common starch adhesives normally used by corrugating operations. While the cause of this difficulty is still not fully resolved, there is evidence that silicate-treated board absorbs moisture at a far higher rate than untreated board. Moisture at the surface of the board is necessary for the starch to gelatinize properly. This being true, the proper formulation for an adhesive to be used with a silicate-treated board would either (1) contain elements which would retain the moisture necessary for gelatinization or (2) contain bonding agents which would react properly under the high moisture loss condition.

Samples of the treated linerboard were sent to the National Starch & Chemical Corporation development laboratories in Bridgewater, New Jersey. National was given the assignment of identifying an adhesive that had the best probability of success. Normally, we would place a number of additional qualifiers on the adhesive, such as minimum cost, compatibility with the corrugator's normal adhesive, modest application rate, and ease of handling. In the DHSSC, however, MiPly was concerned to evaluate the resin treatments. The identification of an optimal adhesive would wait until confirmation of the value of the resin treatment.

National Starch recommended their product No. 29-9551. This adhesive worked well in their laboratory tests, particularly well with the board treated with the silicate/clay mixture. The No. 29-9551 adhesive is not compatible with normal starch adhesives and therefore requires a complete cleanup before and after use.

## ADHESIVE TRIAL

### Singleface Trial at Lawrence Paper -

Since the No. 29-9551 adhesive requires a special setup on the corrugator, it was decided to conduct an adhesive trial to test the adequacy of the setup procedures and the bonding obtained. To minimize the quantity of materials consumed, the trial was conducted on a singleface corrugator.

The adhesive trial was conducted at the Lawrence Paper Company corrugating plant in Lawrence, Kansas on January 28, 1988. The trial was conducted on a singleface, A flute, corrugator. A portable adhesive system was used to prepare the adhesive and feed it directly into the reservoir of the corrugator. Four runs were made: (1) 3.2 silicate/clay treated board, bonding to the treated side, (2) bonding to the untreated side, (3) 3.8 silicate-treated board, bonding to the treated side, and (4) bonding to the untreated side.

The "green bond" obtained on the corrugator appeared quite weak. However, it was evident that the adhesive cured very well and developed a strong bond. An evaluation of the bonding was obtained by conducting a series of pin adhesion

tests<sup>2</sup> approximately one hour after the corrugator trial. While pin adhesion values ranged from a low of 85 to a high of 240, the majority of values tended to be around 170. The expected value for this A flute board would be 140. These tests were conducted quite informally and admittedly too soon after the corrugating to allow a full curing of the adhesive.

#### Adhesive Analysis at National Starch -

Samples of the singleface corrugated board were sent to National Starch & Chemical Corporation, Bridgewater, NJ, for analysis. MiPly and National met to review the results of the analysis on February 4, 1988. National's review found the bonding to be superior: average pin adhesion values of 160 compared to the expected 140 for A flute board. National did not find the extreme variation of pin adhesion values found at Lawrence Paper. This may be due to the fact that the Lawrence Paper tests were conducted very shortly after the corrugating trial and the adhesive had variations in cure development.

National did note instances of "dry" streaks associated with the finger lines. There were no suggestions of the cause or cure, other than to bring this to the attention of the corrugator operator. Also noted in the MiPly/National meeting was an apparent ridging of the adhesive on the medium. When individual flutes were examined, it appeared that the adhesive was spotty. However, when a number of flutes were reviewed together, a pattern developed showing the spots to be lined up across the flute tips. A suggestion was made that there may be a high surface tension in the adhesive, which could be adjusted in its formulation. It was agreed that this was a subject for future work. The bond obtained was quite adequate for the evaluation of the resin treatment.

#### **CORRUGATING TRIAL AT LAWRENCE PAPER**

The corrugating trial was held at Lawrence Paper, Lawrence, Kansas at 7:00 AM, Monday, February 8, 1988. Being run as the first job on a Monday morning meant that the corrugator was washed up and most easily prepared for the special adhesive. As with the previously run adhesive trial, the No. 29-9551 adhesive was prepared in mobile adhesive systems, a separate independent system for each of the four glue lines.

The sequence for running the different treatments began with Box No. 2 (where the treated linerboard was 75" wide), then Boxes No. 3 & 4 (where both sets of treated linerboard were 66-5/8" wide), and then finally Box No. 1 (where the treated linerboard was 63-3/8" wide). This schedule was undertaken because corrugator operators prefer to schedule jobs starting with the widest stock and to then work in order of successively narrowed stocks.

At the conclusion of the corrugating run approximately 25 sheets of the corrugated board made from each different treatment type were cut into blanks, scored, slotted, and folded into containers. These containers were stapled at the manufacturer's joint. Each 60" x 90" corrugated sheet yielded two 20" x 12" x 10" containers, with substantial waste. With the telephone approval of Mr. Anderson Miller of the U.S. Army Natick RD&E Center, the container dimensions were changed from outside dimension (O.D.) to inside dimension (I.D.). This change from O.D. to I.D. was requested by Lawrence Paper as it is their customary practice to use inside dimensions when laying out the container.

The completed containers were sorted so that 15 of each treatment type would be shipped to Pro-Pack Testing Laboratories for compression analysis. The remainder were shipped to MiPly Equipment along with all corrugated sheets produced. Subsequently, at the request of the Project Officer, 10 containers from each of the four treatment types were shipped to the Natick Accountable Property Officer.

#### PHYSICAL PROPERTIES TESTING AT PRO-PACK TESTING LABORATORY

Fifteen (15) containers of each treatment type were sent to ProPack Testing Laboratory, St. Louis, Missouri for analysis. The 15 containers were divided into three groups of 5 each: (1) 5 containers for full box compression<sup>3</sup> testing at standard conditions, (2) 5 containers for full box compression<sup>3</sup> testing at tropic conditions, and (3) 5 containers to be (a) analyzed for fabrication defects, (b) bonding failures, (c) short column compression<sup>4</sup> values at standard and tropic conditions, (d) caliper, and (e) basis weight. The Pro-Pack Report is presented as Appendix B.

A summary of the compression test results follows.

TABLE 6. Summary Full Box Average Compression Values  
(pounds)

| <u>Conditions</u>                      | <u>Treatment Variations*</u> |                  |                  |                  |
|----------------------------------------|------------------------------|------------------|------------------|------------------|
|                                        | <u>Box No. 1</u>             | <u>Box No. 2</u> | <u>Box No. 3</u> | <u>Box No. 4</u> |
| Standard<br>(73 degrees F<br>& 50% RH) | 2,265                        | 2,205            | 2,175            | 2,350            |
| Tropic<br>(100 degrees F<br>& 90% RH)  | 920                          | 1,060            | 940              | 800              |

- \* Box No. 1 - 275# - 10% add-on of 3.2 Sodium Silicate
- Box No. 2 - 275# - 10% add-on of 3.2 Sodium Silicate/Kaolin Clay
- Box No. 3 - 275# - 10% add-on of 3.8 Sodium Silicate
- Box No. 4 - 305# - 14% add-on of 3.8 Sodium Silicate

Of the four treatment variations, Box No. 2 (88% silicate/12% clay in an aqueous solution) meets the contract requirements of 1,800 lbs. at standard and 1,000 lbs. at tropic.

The caliper of the corrugated boards of all box types ranged from .258 to .267, well under the maximum allowable caliper of .300.

The linerboard weights were 42 lbs/msf and 26 lbs/msf, well under the maximum allowable linerboard weight of 90 lbs/msf.

The corrugating medium was 40 lbs/msf, the maximum allowable.

The resin add-on, as a percentage of the combined corrugated board was: Box No. 1, 6.5%; Box No. 2, 6.9%; Box No. 3, 9.8%; Box No. 4, 11%. Thus, Boxes No. 1, 2, & 3 were under the 10% maximum resin add-on, while Box No. 4 was slightly over.

The fabrication analysis found no manufacturing defects in the squareness of the containers or in the slots. Slight scoreline cracking was found in Box No. 1. Serious concern was focused on problems with the adhesive application. Severe dry streaks were found at both singlefacers and severe spotty application was found at the B flute doublebacker. These application problems may be due to the corrugator crew's lack of familiarity with the adhesive and the special treatment of the linerboard. These adhesive problems may also be due to formulation problems as noted in the discussion of the adhesive trial. Failures under the full box compression tests appeared to be linked to the adhesive problems. This suggests that the resin treatment may actually provide a greater strength enhancement than that revealed in these tests.

## CONCLUSIONS AND RECOMMENDATIONS

The resin treatment has provided a very substantial increase in the compressive strength of the corrugated board. This strength increase is substantial for all treatments, but most notable for the silicate/clay saturant treatment. The short column compression for the silicate/clay treatment shows a 38% greater strength than that expected for the same combination of linerboard weights and corrugating medium. This percentage increase is reduced to 18% in the full box compression tests. The lower full box values are most likely due to failures in the adhesive application.

### Cost/Savings Implications -

If we assume that the short column values will be reflected in full box compression once the adhesive problems are resolved, we can forecast a dramatic savings in fibre costs to construct such a high-stacking-strength container. To obtain equivalent compression values with untreated linerboard,

we would expect a configuration of 90/33/42/33/90 C/B flute doublewall. This configuration would have 92 more pounds of fibre per 1,000 square feet of corrugated board than the configuration used to construct Box No. 2.

The resin treatment for the silicate/clay utilized a chemical saturant that has an expected cost in volume of approximately \$0.20 per dry weight pound. The expected cost, in commercial application, to process the saturant into the linerboard is \$0.18 per pound. In Box No. 2 approximately 16 dry weight pounds of silicate/clay were used. Thus the expected cost, again in volume, for this treatment is  $(16 \times (\$0.20 + \$0.18)) = \$6.08$ . The alternative cost to use heavy weight linerboard is  $92 \times \$0.205 = \$18.86$ . (Note: current cost of linerboard is \$410/ton; therefore  $\$410/2000 = \$0.205/\text{lb.}$ ) Therefore a cost savings opportunity of  $\$18.86 - \$6.08 = \$12.78$  per 1,000 square feet of corrugated board is available. This savings is reduced by any increased costs for the adhesive and any increased operating expense at the corrugator. The savings is increased by the value of the superior compression strength under tropic conditions.

It is clear that an effort to resolve the adhesive problems offers a handsome reward.

#### Saturant Opportunities -

The silicate/clay formulation provided the most significant strength enhancement. This formulation used a ratio of 7.3:1 silicate to clay. This ratio was chosen both as a matter of expediency (trying to be sure that we would get the clay dispersed without building particle size) and a concern that too high a level of clay may cause difficulty in rigidity and/or repulping. However, a higher level of clay may well provide superior strength in tropic conditions and not cause those other problems. It should be tried.

Excellent opportunities are available with other silicate mixtures. MiPly has recently begun investigating a silicate/starch formulation where uncooked starch is mixed with silicate, impregnated into the linerboard, and dried - with the starch cooking, gelatinizing, in the drying oven. MiPly expects this treatment to have reduced moisture sensitivity, superior strength, good flexing capability without cracking, and better bonding characteristics.

# REFERENCES

| Ref.<br>No. | Standard<br><u>Physical Properties Tests</u> | <u>TAPPI</u> | <u>ASTM</u> |
|-------------|----------------------------------------------|--------------|-------------|
| 1           | 6 Inch Ring Crush Test                       | T818         | D1164       |
| 2           | Pin Adhesion Test                            | T821         |             |
| 3           | Full Box Compression Test                    | T804         | D642        |
| 4           | Short Column Test                            | T811         | D2808       |

APPENDIX A

C-Q No 3437

ESTABLISHED 1923

DATE May 11, 1987

 **CONTAINER-QUINN**  
**TESTING LABORATORIES, INC.**  
170 Shepard Ave. • Wheeling, Illinois 60090  
Telephone 312/537-9470

TESTS CONDUCTED FOR:

MiPly Equipment, Inc.  
820 Frontage Road  
Northfield, IL 60093

Attn: Eliot R. Long  
Vice President

ITEM TESTED: Three (3) sample lots of 42# treated linerboard identified as:  
5%/10%/15%.

Object of Tests: To determine the Ring Crush Resistance at Standard Conditions  
and after exposure to High Humidity conditions.

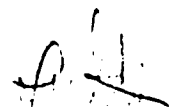
Test Procedures: All specimens prepared, conditioned and tested in accordance  
with applicable ASTM and/or TAPPI Standard Test Methods.

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FINDINGS

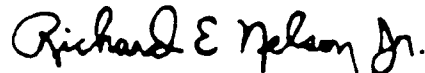
Please see attached Laboratory Data Sheet for detailed  
Test Results.

---

  
Larry Staffin - Laboratory Mgr

APPROVED BY:

CONTAINER-QUINN TESTING LABORATORIES, INC



Richard E. Nelson, Jr.



Ring Crush Resistance (LBS/6"x½" Specimens)

Specimens Tested at Standard Conditions (73±2°F + 50±2%RH)

|           | 5%  |     |  | 10% |     |  | 15% |     |  |
|-----------|-----|-----|--|-----|-----|--|-----|-----|--|
|           | MD  | CMD |  | MD  | CMD |  | MD  | CMD |  |
| High :    | 138 | 106 |  | 159 | 129 |  | 193 | 154 |  |
| Low :     | 129 | 90  |  | 149 | 115 |  | 184 | 140 |  |
| Average : | 134 | 96  |  | 154 | 122 |  | 187 | 146 |  |

C-Q Laboratory  
Expectency for  
Untreated 42#  
Linerboard :

|     |    |     |    |     |    |
|-----|----|-----|----|-----|----|
| 108 | 74 | 108 | 74 | 108 | 74 |
|-----|----|-----|----|-----|----|

Specimens Tested After Conditioning at 90±3°F + 90±3%RH

|           | 5% |     |  | 10% |     |  | 15% |     |  |
|-----------|----|-----|--|-----|-----|--|-----|-----|--|
|           | MD | CMD |  | MD  | CMD |  | MD  | CMD |  |
| High :    | 67 | 49  |  | 76  | 62  |  | 96  | 75  |  |
| Low :     | 60 | 44  |  | 63  | 55  |  | 81  | 66  |  |
| Average : | 64 | 47  |  | 71  | 58  |  | 88  | 68  |  |

C-Q Laboratory  
Expectency for  
untreated 42#  
Linerboard :

|    |    |    |    |    |    |
|----|----|----|----|----|----|
| 50 | 33 | 50 | 33 | 50 | 33 |
|----|----|----|----|----|----|

# APPENDIX B

## PRO-PACK TESTING LABORATORY, INC.

7208 WEIL AVENUE • ST. LOUIS • MISSOURI • 63119

(314) 645-3622

February 29, 1988

MI-PLY Equipment, Inc.  
820 Frontage Road  
Northfield, IL. 60093

Attn: Mr. Eliot Long

Project No. 88-2-15

Subject: Special Tests on Four Sets of Experimental Containers.

### Objective

To measure various physical properties of both the corrugated board structures and boxes representing four different chemical treatments. These evaluations were performed on the specimens following conditioning periods at both standard and tropic conditions. The boxes were marked as #1, #2, #3 and #4 for identification purposes.

### Summary

The sets exhibited these average top load compressions: (pounds)

|             | <u>#1</u> | <u>#2</u> | <u>#3</u> | <u>#4</u> |
|-------------|-----------|-----------|-----------|-----------|
| a. Standard | 2265      | 2205      | 2175      | 2350      |
| b. Tropic   | 920       | 1060      | 940       | 800       |

### Procedures

Tests applicable to this project were performed in accordance with the following methods\*

|                            |      |
|----------------------------|------|
| Thickness in Caliper       | T411 |
| Basis weight determination | T410 |
| Edge Crush Test (ECT)      | T811 |
| Box compression            | T804 |

The conditioning environments used were 50% RH, 73°F for standard and 90% RH, 100°F. for the tropic condition. (Exposure time - 72 hours)

All flaps of the boxes were sealed with a weather-resistant adhesive that maintained closure at the tropic condition and the standard condition.

### Test Results and Discussions

| <u>Materials Analyses</u> | <u>#1</u> | <u>#2</u> | <u>#3</u> | <u>#4</u> |
|---------------------------|-----------|-----------|-----------|-----------|
| Flute                     | BC/DW     | BC/DW     | BC/DW     | BC/DW     |

#### Materials\*\* (Lbs./MSF)

|                |           |           |            |            |
|----------------|-----------|-----------|------------|------------|
| Out Facing     | 44.8(2.8) | 47.0(5.0) | 53.8(11.8) | 53.1(11.1) |
| Corr. Med. (B) | 40.7(0.7) | 41.5(1.5) | 42.8(2.8)  | 40.9(0.9)  |
| Mid Facing     | 29.4(3.4) | 30.4(4.4) | 30.6(4.6)  | 50.3(8.3)  |
| Corr. Med. (C) | 39.8      | 38.5      | 39.7       | 40.8(0.8)  |
| In. Facing     | 49.5(7.5) | 48.0(6.0) | 47.4(5.4)  | 48.7(6.7)  |

\*Only differences in some instances were the number of determinations.

\*\*Theoretical amount of additive using rated component basis weight are in parenthesis.

Project No. 88-2-15 (continued)

|                         | <u>#1</u> | <u>#2</u> | <u>#3</u> | <u>#4</u> |
|-------------------------|-----------|-----------|-----------|-----------|
| <u>Corr. Adhesive</u>   | WRA       | WRA       | WRA       | WRA       |
| <u>Ply Sep. Rating*</u> | Pass      | Pass      | Pass      | Pass      |

Sets #1, #2, and #3 reportedly were a 42(40)26(40)42 combination while #4 was 42(40)42(40)42.

Caliper, In.

|             |      |      |      |      |
|-------------|------|------|------|------|
| Theoretical | .263 | .263 | .263 | .266 |
| Actual      | .263 | .258 | .260 | .267 |

Edge Crush, P.L.I.  
(Short Columnr.)

Standard

|      |     |     |     |     |
|------|-----|-----|-----|-----|
| High | 120 | 124 | 101 | 112 |
| Low  | 91  | 92  | 85  | 85  |
| Ave. | 105 | 108 | 92  | 100 |

Typical Regular Untreated

|    |    |    |    |
|----|----|----|----|
| 78 | 78 | 78 | 84 |
|----|----|----|----|

Diff.(Act. vs typical)

|      |      |      |      |
|------|------|------|------|
| +35% | +38% | +18% | +19% |
|------|------|------|------|

Tropic

|      |    |    |    |    |
|------|----|----|----|----|
| High | 50 | 56 | 58 | 56 |
| Low  | 40 | 48 | 46 | 46 |
| Ave. | 45 | 51 | 52 | 52 |

The caliper of the experimental boards were reasonably close to the calculated or theoretical, as derived by using typical component thicknesses and minimum flute heights.

The possible effect of the treatment is illustrated by comparing the actual vs a theoretical ECT value derived from typical ECT values for untreated 275 DW having 40 lb. mediums. These differences (increases) ranged from 18-35% of the commercial stocks.

All the experimental boards were adversely affected in ECT by serious fabrication conditions, namely, dry streaks on the C and B flute singlefacers and spotty glue coverages on the B flute doublebacker sides.

Box Compression Tests  
(Lbs. @ Defl.in.)

| <u>Standard</u> | <u>#1</u> | <u>#2</u> | <u>#3</u> | <u>#4</u> |
|-----------------|-----------|-----------|-----------|-----------|
| Box 1.          | 2325/.90  | 2140/.70  | 2400/.75  | 2480/.74  |
| 2.              | 2200/.87  | 2185/.73  | 2100/.77  | 2395/.74  |
| 3.              | 2385/.77  | 2075/.67  | 2140/.77  | 2419/1.4  |

\*Using conventional Fed. Stds. e.g. PPP-F-320

Project No. 88-2-15 (continued)

Box Compression Tests (continued)  
(lbs. @ Defl. in.)

|                               | <u>#1</u> | <u>#2</u> | <u>#3</u> | <u>#4</u> |
|-------------------------------|-----------|-----------|-----------|-----------|
| Box 4                         | 2210/.73  | 2310/.76  | 2170/.73  | 2075/.79  |
| 5.                            | 2215/.70  | 2325/.67  | 2070/.77  | 2390/.73  |
| Ave. (nearest 5 lbs.)         | 2265/.79  | 2205/.71  | 2175/.76  | 2350/.88  |
| <u>Estimated*</u>             | 2530      | 2575      | 2205      | 2425      |
| <u>Diff. (Actual vs Est.)</u> | 90%       | 86%       | 99%       | 97%       |

Tropic

|                               |          |          |          |         |
|-------------------------------|----------|----------|----------|---------|
| Box 1.                        | 900/172  | 1020/.72 | 1040/.77 | 875/.80 |
| 2.                            | 1125/.84 | 1105/.64 | 1045/.77 | 760/.80 |
| 3.                            | 960/.75  | 1015/.72 | 840/.72  | 665/.80 |
| 4.                            | 770/.70  | 940/.70  | 845/.72  | 990/.73 |
| 5.                            | 860/.80  | 1215/.78 | 925/.74  | 700/1.0 |
| Ave. (nearest 5 lbs.)         | 925/.76  | 1060/.71 | 940/.74  | 800/.83 |
| <u>Estimated*</u>             | 1085     | 1215     | 1245     | 1260    |
| <u>Diff. (Actual vs Est.)</u> | 85%      | 87%      | 76%      | 63%     |

Moisture contents for materials in tropic rooms ranged from 17.8 - 19.7%

The actual vs the estimated top loads at standard conditions were comparable overall. At tropic conditions the difference was relatively greater which could indicate that the fabrication defects were more sensitive to the humid conditions thereby having a more adverse affect on board stiffness. In the latter case this would apply to both the ECT values and the box compression levels.

As previously illustrated, the ECT's of the treated material reflected possible stiffness increases ranging from 18-35% above other untreated, commercial boards. Making a similar comparison, but using the top load compression data, at standard conditions, indicates the following improvements:

| <u>Standard-Untreated</u>                                                                        | <u>#1</u> | <u>#2</u> | <u>#3</u> | <u>#4</u> |
|--------------------------------------------------------------------------------------------------|-----------|-----------|-----------|-----------|
| 275 DW/Base (Assuming a 42(40)26(40)42 board for 275 DW and 42(40)42(40)42 for untreated 350 DW) |           |           |           |           |
|                                                                                                  | +21%      | +18%      | +15%      | +26%      |
| 350 DW/Base                                                                                      | +13%      | + 9%      | + 8%      | +17%      |

This same contrast could be made with the top load data at tropic conditions. (In this example the dry strength retention of the untreated material at the humid condition was arbitrarily selected as 45%) These differences could be in error because of the serious fabrication defects in this case.

\*Using Institute formula and applicable ECT, Caliper values and box size of 20 x 12 x 10 in.

Project No. 88-2-15 (continued)

| <u>Tropic-Untreated</u> | <u>#1</u> | <u>#2</u> | <u>#3</u> | <u>#4</u> |
|-------------------------|-----------|-----------|-----------|-----------|
| 275 DW/Base             | +9%       | +26%      | +12%      | -5%       |
| 350 DW/Base             | +2%       | +17%      | + 4%      | -13%      |

Fabrication Analyses

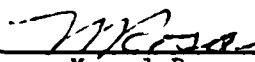
As mentioned previously, there were fabrication defects consisting of the following in all sets. These factors, their severities and locations appeared directly related to the failure lines in the boxes.

| <u>Flute/Side</u> | <u>Conditions</u>  |
|-------------------|--------------------|
| B/DB              | Severe spotty glue |
| B/SF              | Severe dry streaks |
| C/SF              | Severe dry streaks |

Also, there were significant "build-ups" of corrugating adhesive at gluelines of these facings. The excessive water may have been a contributor to the cockling effect noted on some outer facing(s) and the middle facing.

Boxmaking inspections also indicated the following:

| <u>Conditions</u>     | <u>#1</u> | <u>#2</u>  | <u>#3</u> | <u>#4</u> |
|-----------------------|-----------|------------|-----------|-----------|
| <u>Slots</u>          | O.K.      | O.K.       | O.K.      | O.K.      |
| <u>Squareness</u>     | O.K.      | O.K.       | O.K.      | O.K.      |
| <u>Liner Cracking</u> |           |            |           |           |
| Flap Scores           | Slight    | O.K.       | O.K.      | O.K.      |
| Panel Scores          | Slight    | Noticeable | O.K.      | O.K.      |

  
Manuel Rosa, Pres.

END

DATE

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