ADHESIVE EVALUATION FOR HEAT RESISTANCE TESTING RESULTS

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OBJECTIVE

The objective of this project was to evaluate the heat resistance of membrane pressed thermoformed vinyl panels manufactured by the Natural Resources Research Institute (NRRI) using polyurethane dispersion adhesives supplied by HB Fuller Company.

METHODS AND MATERIALS

HB Fuller Company supplied four adhesives for evaluation: RK4000, NP4119, NP4135, and NP4136, and the appropriate catalyst for use in manufacturing rigid thermoformed (RTF) panels in the NRRI membrane press.

NP4119 was mixed with 10 percent catalyst by weight, NP4135 was mixed with 5 percent and 10 percent catalyst by weight, and NP4136 was mixed with 5 percent catalyst by weight. They were then applied to routed medium density fiberboard (MDF) manufactured by UniBoard Canada. Each adhesive was applied to two 3/4- x 10- x 13-in boards and two 3/4- x 16- x 20-in boards. A high volume low pressure (HVLP) Binks Mach 1 spray gun was used to spray the adhesives onto the MDF. Two spray passes of adhesive were added to each panel edge resulting in a wet mil thickness of approximately 5-7 mils. Approximately 2-4 wet mils of adhesive was added to the face of the panel using recommended spray techniques. The panels were then lifted from the stack and allowed to air dry for approximately one hour.

The routed doors were covered with a sheet of American Renolit Corporation 16 mil polyvinyl chloride (PVC) thermoforming vinyl. Thermoforming was completed using commercial pressing parameters. The pre-warm cycle length was 15 seconds and the pressure cycle was 50 pounds per square inch (psi) of air pressure for 30 seconds. The cooling cycle length was 30 seconds. When pressing the doors the internal membrane temperature was set at 275°F and 270°F to achieve edge temperatures of approximately 170°F. The control was pressed at 275°F and 270°F to achieve edge temperatures of approximately 170°F. The glueline edge temperatures were verified with paper thermometers placed on the vertical edges of the door. Following the completion of the laminating cycle, the doors were removed from the tray, stacked individually upside-down, and allowed to completely cool before trimming.

Two 5- x 5-in pieces were cut from opposite corners of each panel for a total of four samples for each group. The samples were labeled according to the adhesive type and application on the back side. The uncut edges of the boards were labeled A and B for reporting purposes during the test. Testing was completed according to the NRRI Heat Performance Standard Procedure NRRI 1-96, Standard Method For Heat Resistance Testing of Thermoformed Components (attached).

A forced air oven was pre-heated to 170°F. The specimens were then placed in the oven for 30 minutes at 170°F. One tray of specimens was removed at a time and evaluated for edge creep of the vinyl. A magnifying eyepiece with a scaled reticle was used to measure deformation of the foil at the bottom edge. Then the temperature was increased in 5°F increments until a failure on both edges was recorded. An average of the two sides was taken to give a failed temperature for each piece. Failure was defined as foil deformation of $\geq$0.2 mm (length of visible wood) or other significant foil movement.

RESULTS AND DISCUSSION

Table 1 shows the mean heat resistance and the board size for each of the adhesives tested. NP4136 showed the lowest heat resistance of all the adhesives tested. Ten percent catalyst levels in NP4119 and NP4135 showed no significant improvement in heat resistance compared to 5 percent levels of catalyst. On average the RK4000 adhesive failed at a higher temperature than the other HB Fuller adhesives. Differences in heat resistance values between board sizes were due to lower localized forming temperatures during the pressing process.

CONCLUSION

Based on the trial completed, HB Fuller adhesives NP4119, NP4135, and NP4136 have lower heat resistance than RK4000 adhesive. Increasing the catalyst levels from 5 percent to 10 percent in the NP4119 and NP4135 did not improve the heat resistance of the adhesive.

Table 1.--Average failure temperatures and board sizes for heat resistance testing of membrane pressed doors. Tested December 1998 and March 1999 at the Natural Resources Research Institute.

Adhesive	Average Failure Temperature (°F)	Board Size (in.)	Failure Temperature (°F)
RK4000 5% catalyst	194.40	10 x 13	192.3
		16 x 20	201.7
NP4136 5% catalyst	170.94	10 x 13	170.0
		16 x 20	171.9
NP4119 5% catalyst	181.70	10 x 13	180.8
		16 x 20	183.3
NP4119 10% catalyst	181.25	10 x 13	175.6
		16 x 20	186.9
NP4135 5% catalyst	183.75	10 x 13	180.6
		16 x 20	186.9
NP4135 10% catalyst	186.56	10 x 13	175.6
		16 x 20	197.5

NRRI 1-96 STANDARD METHOD FOR HEAT RESISTANCE TESTING OF THERMOFORMED COMPONENTS

1. Scope

This test method covers the determination of the resistance of adhesives to temperature changes. This procedure primarily applies to thermoformed vinyl door panels manufactured using medium density fiberboard, vinyl, and polyurethane dispersion (PUD) adhesives. Thermoformed vinyl doors are typically manufactured using a membrane pressing process. Generally, this process includes routing medium density fiberboard panels, spraying a PUD on the edges and on the face. Most often two layers of PUD are sprayed on the edges. A sufficient length of open time is required to allow the water to evaporate from the glueline. The minimum open time required is approximately 10 minutes at 68 °F and 50% relative humidity. The door panel is then placed on a tray and supported by a backer board smaller than the door panel. A sufficient amount of vinyl film is placed over the tray. Typical manufacturing cycles are then selected.

There is no consensus on standard testing procedures for determining the heat resistance of the substrate and vinyl adhesive line. Each adhesive manufacturer and panel manufacturer have developed internal evaluation procedures. This method is being developed to standardize a heat resistance testing method. It is open to modification.

It is recognized that no accelerated procedure for degraded materials corresponds exactly to actual service conditions and that no single or small group of laboratory procedures will simulate all service conditions. Consequently, care should be exercised in the interpretation of the data obtained in this test.

2. Referenced Documents

2.1 *Laminating Materials Association:*
Voluntary Standards for PVC Thermoforming Vinyl, Laminating Materials Association, Oradell, NJ.

3. Definitions

3.1 *Thermoformable overlay film,* calendered or extruded solid color rigid vinyl films in single ply or multi ply construction. Film may be rigid or semirigid formulation. Gauges run from 0.010 - 0.040 inches and film may be embossed and may be coated with scratch and stain resistant coatings. Designed for thermoforming with heat and pressure in the bladder press or vacuum forming process.

3.2 *Substrate*, medium density fiberboard (MDF) and particleboard
Medium density fiberboard (MDF), a panel manufactured from wood fibers combined with a synthetic resin or other suitable binder. and bonded under heat and pressure to a density of 31 to 55 pounds per cubic ft (pcf) Because of its homogenous construction, MDF features an extremely smooth face and tight, machinable core.
particleboard, a panel manufactured from wood or agro particles or combinations of wood or agro particles and fibers, bonded together with synthetic or other suitable binders under

heat and pressure to a density of 28 to 55 pounds. Particleboard offers a smooth surface with a good machinable core. MDF is favored over particleboard for contour thermoforming applications.

3.3 *Polyurethane dispersion (PUD)*, an aqueous dispersion of fully reacted urethane polymers containing hydrophilic anionic, cationic, or nonionic groups noted for their high performance properties, excellent adhesion, chemical resistance, outstanding toughness, and low temperature flexibility. Generally used for the membrane pressing of vinyl to an MDF core. PUD's can be used with a hardener (usually 5% by weight) for higher heat resistance if required. Typically sprayed at a rate of 2-4 wet mils and then air dried before pressed with thermoformed vinyl in a membrane press.

4. Apparatus

4.1 *Circulating Air Ovens*, capable of being maintained at the required temperatures.
4.2 *Magnifying Eyepiece*, with a scaled rectacle to measure deformation of the thermoformed foil.

5. Test Specimens

5.1 The number and type of test specimens shall be of sufficient number to interpret the test data. It is recommended that two specimens be selected from opposite corners of each panel evaluated. These specimens should be five-by-five inches. For NRRI purposes, five panels will be tested for each manufacturing variable combination. This will result in the testing of ten samples for each manufacturing variable combination.

6. Conditioning

6.1 Condition all specimens for seven days after manufacturing to ensure complete curing of the adhesive. Panels manufactured at the NRRI should be conditioned for seven days at a relative humidity of 50 ± 2% at 73.4 ± 2°F (23 ± 1.1°C).

7. Procedure

7.1 Label each specimen with the appropriate specimen number. Each of the two edges should be labeled to differentiate failure location.
7.2 Place each specimen in a forced air oven that has been preheated to 170°F. The specimens should be placed horizontally so that all edges have free access.
7.3 The specimens should be left in the oven for thirty minutes at 170°F. One tray of specimens should be removed at a time and evaluated for performance. Specific information recorded may include: deformation of vinyl at the panel edge or corner, deformation of vinyl in routed center areas, and visual presence of the substrate. These may be qualitative or quantitative measurements. A magnifying eyepiece will be used at the NRRI to measure deformation of the foil at the bottom edge. Other evaluation techniques may include photocopying the panel profile or taking pictures of the edges and profile.

7.4 Following evaluation, the specimens will be returned to the oven and allowed to equilibrate at the current temperature setting until 45 minutes have elapsed since the start of the current temperature. Then the temperature will be increased in 5° F increments until a failure is identified. For NRRI purposes, a failure may be defined as foil deformation of greater than 0.2 mm (length of visible wood) or other significant foil movement.

7.5 When a specimen has been considered failed, the specimen will be removed from the testing and retained.

8. Calculations

8.1 The mean, standard deviation and coefficient of variation of the failure temperature for each group of panels will be determined according to standard statistical procedures.

9. Report

9.1 The report shall include the following:
9.2 Title and company for which the testing was performed,
9.3. Adhesive type and application method,
9.4 Foil type, thickness and supplier,
9.5 Manufacturing parameters,
9.6 Number of specimens for each manufacturing variable combination,
9.7 Modifications to testing procedure NRRI 1-96,
9.8 Changes in appearance to each specimen,
9.9 Photocopies or pictures of specimens before testing and at failure if desired,
9.10 Failure temperature of each specimen, and
9.11 Mean, standard deviation and coefficient of variation of failure temperature for each group tested.