

MEMBRANE IMMERSION TEST: MODIFIED BITUMEN MEMBRANE ASPHALTIC IMPREGNATION EVALUATION

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Today's low-slope roof systems are being asked to perform for extended periods of time. It is important to be able to predict their long-term performance as accurately as possible.

Many accepted tests are available to evaluate the quality of asphaltic blends before and during manufacture (ring and ball, viscosity, cold-temperature flexibility, and other chemical tests). Various mechanical analysis methods are used to evaluate online manufacturing processes and finished products (tensile, elongation, tear, flex, etc.)

However, it is difficult to predict with these tests how membranes will perform over time (delamination, cracking, etc.). One critical product standard that must be addressed is impregnation of the reinforcements during the manufacturing process. This problem can be the result of the wrong reinforcements, improper asphalt/polymer blend viscosity, poorly adapted process changes, raw material variances, etc.

The authors' company has developed a simple and accurate test, Membrane Immersion Test (MIT), to evaluate impregnation of the reinforcements. This test will help to evaluate the impregnation achieved during manufacture and become an excellent indicator of future ability to resist delamination and related problems.

Specially prepared membrane samples are submerged in hot water 60°C (140°F) for 14 hours and then dried in an oven 110°C (230°F) for 30 minutes. Poorly impregnated membranes will exhibit large-scale blisters or early signs of delamination.

MIT can be an important step forward in the evaluation of modified bitumen membranes' long-term performance. Comparison tests have been completed and an explanation will be discussed in the final report.

KEYWORDS

APP, blisters, delamination, immersion, impregnation, modified bitumen membrane, reinforcement, test method, water resistance, weathering resistance.

INTRODUCTION

Today's low-slope modified bitumen roof systems are being asked to perform for extended periods of time. Advances in manufacturing technology and raw materials provide the opportunity for a longer serviceable life. Numerous manufacturers promote roof systems designed to perform for up to 20 years. It is important, then, to be able to accurately predict the long-term performance of these membranes. Simple and reliable tests that can predict the in-place performance of a finished membrane system are very helpful.

Many accepted tests (e.g., ASTM D 5147) are available to evaluate the quality of asphaltic blends before and during manufacture (ring and ball, viscosity, cold-temperature flexibility, and other chemical tests). Various mechanical analysis methods are used to evaluate online manufacturing processes and finished products (tensile, elongation, tear, flex, weight, thickness). These tests are important in evaluating how well the product has been made in accordance with predetermined manufacturing specifications. But tests designed to simulate real-world rooftop situations should also be a part of product evaluation.

It is difficult, however, with these tests to predict how membranes will perform over time (delamination, cracking, or blistering). One critical product standard that must be addressed is impregnation of the reinforcements during the manufacturing process. Inadequate impregnation of the finished membrane can be the result of inappropriate reinforcements, improper asphalt/polymer blend viscosity, line speed, poorly adapted process changes, or raw material variances.

The authors have developed a simple and accurate test—Membrane Immersion Test (MIT)—for roofing products with a softening point higher than 110°C (230°F) to evaluate impregnation of the reinforcements. This test will assist in evaluating the impregnation of reinforcements achieved during manufacture and can be used as an indicator of a membrane's future ability to resist moisture infiltration or delamination of the blend from the reinforcement or between plies in a composite mat.

MEMBRANE IMMERSION TEST PROCEDURES

Test Method

- A 0.25-m by 0.25-m (10- by 10-inch) square sample is prepared from the membrane to be tested.
- All four edges are kerfed diagonally (45-degree angle) to expose the reinforcement (see Figure 1). The reinforcement must be visible to simulate cuts made during application.
- The sample is submerged in a water bath at 60°C (140°F) for 14 hours (see Figure 2).
- The sample is removed from the water bath and allowed to dry at room temperature (23°C±3°C [73°F±5°F]) for 30 minutes.
- The sample is then placed in an oven (see Figure 3), preheated to 230°F (110°C), for 30 minutes. Note: This test will not provide accurate results for some membranes



Figure 1. All four edges are herfed diagonally (45-degree angle) to expose the reinforcement.

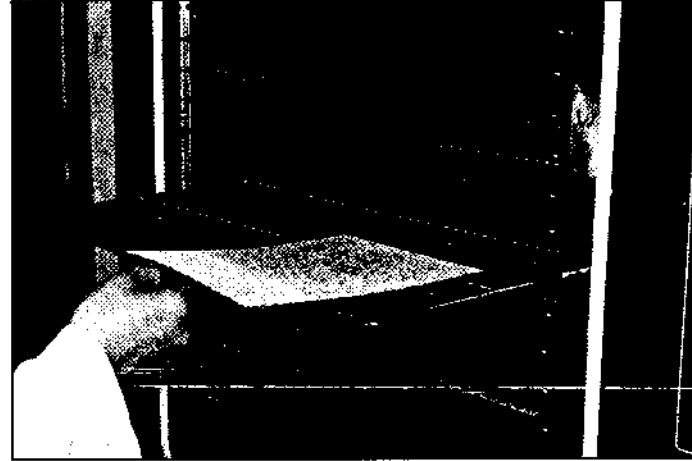


Figure 3. The sample is then placed in an oven, preheated to 110°C (230°F), for 30 minutes.

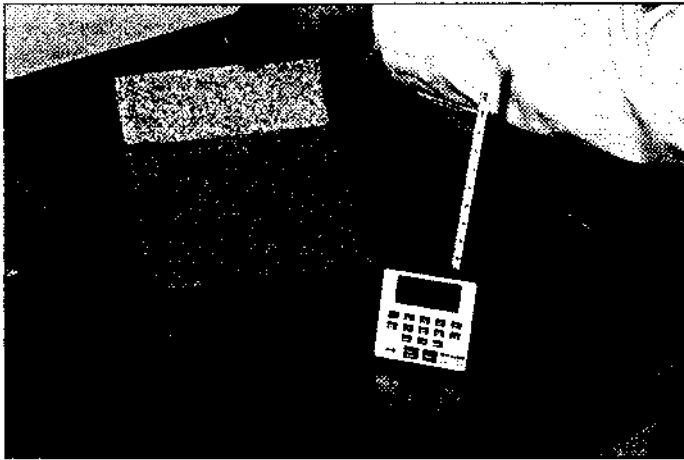


Figure 2. The sample is submerged in a water bath at 60°C (140°F) for 14 hours.

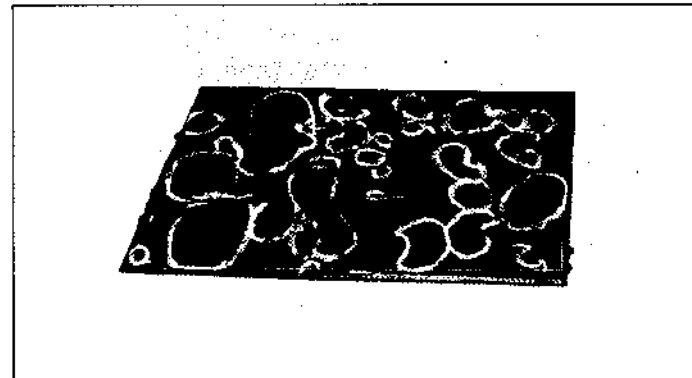


Figure 4. The sample is removed from the oven and visually analyzed. White marks highlight blisters.

(predominantly SBS) because the melt temperature of the finished membrane is below the 110°C (230°F) temperature found in the oven. These membranes will delaminate during the testing.

- The sample is removed from the oven and visually analyzed as discussed in the following section (see Figure 4).

Visual Analysis Criteria

Properly impregnated membranes will exhibit no real change in the membrane surface or structure (thickness, weight).

Poor impregnation of the finished membrane results in the appearance of blisters on either surface of the membrane. Blistering (Figures 5 and 6) would indicate a product that may not perform as intended because of inadequate impregnation of the reinforcement.

Samples with inadequate impregnation may exhibit a total delamination of the asphaltic blend from the reinforcement (Figure 7).

To characterize the results, the authors quantify the percentage of the blistering area by measuring the ratio between the blistering area and the total area of the sample (expressed as a percentage). Manufacturers can develop a standard for measuring blisters or delamination that reflects their acceptable risk level.



Figure 5. Sample A after MIT.

This simple test can be an important measurement of a finished membrane's ability to perform as originally intended. The test simulates the environment found in many areas where modified bitumen roofs are used. The heat and presence of moisture create a real-world test for roofing membranes. The exposed 45-degree kerfed edge simulates the real-world actions of a mechanic when fabricating or cutting a membrane during installation.

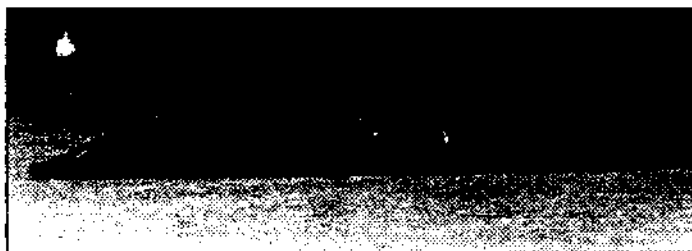


Figure 6. Sample showing excessive blistering. The blend separates from the reinforcement.



Figure 7. Complete delamination of the blend from the reinforcement.

DISCUSSION

In the course of conducting regular quality assurance tests to determine the level of impregnation, numerous membranes have been tested. Three major factors that contribute to inadequate impregnation of the reinforcements and, thus, the potential for a shortened life of the modified bitumen roof membranes have been identified.

- asphalt and polymer final blend viscosity
- production line speeds
- type of binder utilized in the membrane reinforcement

FINAL BLEND VISCOSITY

Experience has shown that the final viscosity of a blend or batch prepared for a manufacturing run affects the impregnation of the reinforcements used in the membrane. With the experience of thousands of batches of production, this factor has become a reliable indicator of potential impregnation problems.

VISCOSITY TESTING

A test was conducted as an example to demonstrate this concept.

A specific reinforcement, in this case a polyester and polyester/glass scrim mat, was utilized. The line speed was held constant at 1600 m/hr (5250 feet/hour). Asphaltic and polymer blends were developed at the viscosities shown in Table 1.

Blend 1	6 Pa.s. (6000 cps) at 182°C (360°F)
Blend 2	10 Pa.s. (10000 cps) at 182°C (360°F)
Blend 3	16 Pa.s. (16000 cps) at 182°C (360°F)

Table 1.

Three membranes were manufactured; the only variation was the viscosity of the asphalt and polymer blend.

VISCOSITY VARIATION RESULTS

The three trial membranes were then subjected to the MIT described previously. The results show a marked difference in impregnation based on the viscosity of the blend, as shown in Table 2.

		Area of Blisters
Blend 1	6 Pa.s. (6000 cps) at 182°C (360°F)	0%
Blend 2	10 Pa.s. (10000 cps) at 182°C (360°F)	20%
Blend 3	16 Pa.s. (16000 cps) at 182°C (360°F)	35%

Table 2.

The level of visible blisters varies from 0 percent at the lower viscosity (6000 cps) to 35 percent at the higher viscosity (16 Pa.s.) of the asphalt and polymer blend. It is understood that differences in reinforcements, viscosity of blends, and line speeds may vary by manufacturer, but the concept that higher-viscosity materials run at the wrong speeds can create a poorly impregnated membrane is valid.

PRODUCTION LINE SPEED

If the speed of the line is too fast, air bubbles can be trapped within the membrane when the reinforcement enters the impregnation tank. That is, the roof membrane appears to be well-impregnated, but air is encapsulated in the blend. In practice, this entrapped air creates small craters when the membrane is heat-welded. A microscopic evaluation of a transverse cut of the membrane reveals these small air pockets.

If these air bubbles are located at the interface of the reinforcement and the blend, they can collapse and create a path for moisture to penetrate the membrane. During the 30 minutes in the oven, the air encapsulated in these pockets expands and creates blisters. The aspect of the test membrane after the MIT is different from the results described in the previous section. In place of a few big blisters, a lot of small blisters are observed.

This example also shows that by adapting production line speeds to accommodate variations in blend viscosity, it is possible to also produce a properly impregnated membrane.

The ideal situation is to match the viscosity of the blend with the appropriate line speed to achieve proper impregnation of the membrane. The MIT can help to determine if this has been done.

BINDER SELECTION FOR MEMBRANE REINFORCEMENTS

There are different types of binders used to manufacture reinforcements for the modified bitumen roofing industry today. These binders are used to bond the various strands of fiberglass and polyester materials together to form a mat or reinforcement. Once again, extensive manufacturing records have been evaluated to develop this position, but for clarity of purpose, a specific test was run to demonstrate the importance of proper binder selection in the modified bitumen membrane manufacturing process.

REINFORCEMENT AND BINDER TEST PARAMETERS

All of the tests were made utilizing readily available reinforcements (fiberglass mat or polyester mat and polyester and/or fiberglass scrim) from today's modified bitumen reinforcement market.

The two most important parameters affecting the impregnation capability of the reinforcement in this study are the type of binder and the residual moisture present in the selected reinforcements after manufacturing.

Configuration and designation of samples are as shown in Table 3.

Sample Designation	Binder and Reinforcement Used
A	Glass Mat GM1/Polyester scrim PS1/Glass scrim GS1 Binder: Polyvinyl Alcohol
B	Glass Mat GM1/Polyester scrim PS1/Glass scrim GS1 Binder: SB Latex/Urea
C	Glass Mat GM2/Polyester scrim PS3/Glass scrim GS3 Binder: SB Latex 1
D	Glass Mat GM2/Polyester scrim PS3/Glass scrim GS3 Binder: SB Latex 2
E	Polyester Mat PM1/Polyester scrim PS2/Glass scrim GS2 Binder: PVC Latex 1
F	Polyester Mat PM1/Polyester scrim PS2/Glass scrim GS2 Binder: PVC Latex 1
G	Polyester Mat PM2/Glass scrim GS3/Polyester Mat PM2 Binder: Thermally Bond
H	Polyester Mat Binder: Acrylic or Spunbond
I	Polyester Mat Binder: Acrylic or Spunbond

Table 3.

These configurations were used to manufacture membranes for the test. The manufacturing parameters were the same for the nine samples tested and listed in Table 3. (Note: The impregnation temperature and line speed were estimated for Samples H and I.)

- viscosity of the blend: 8 to 9 Pa.s. (8000 to 9000 cps) at 182°C (360°F)
- impregnation temperature: 179°C (355°F)
- line speed: 1600 m/hr (5250 feet/hour)

After manufacture, the nine samples were tested in accordance with the MIT described previously.

DISCUSSION OF TEST RESULTS

Reinforcements A and B are identical except for the binder used during manufacturing. The curing (or drying) temperature of the reinforcement during manufacture was 179°C (355°F) in both cases. There is an important difference

MIT Test Results:		
Sample	Blisters	Delamination of the scrim
A (Figure 5)	70%	yes
B (Figure 8)	0%	no
C	35%	no
D	0%	no
E	0%	no
F	70%	yes
G	0%	no
H	50%	no
I	0%	no

Table 4.

between Sample A with polyvinyl alcohol binders and Sample B with the SB latex/urea binder. The former binder is water-soluble, and the latter is emulsified. The curing (or drying) temperature of the reinforcements during manufacture for Sample A was not high enough to dry the polyvinyl alcohol binder. During the oven test, the residual moisture in the Sample A finished reinforcement was determined to be higher than 2 percent (minimum moisture content for PVA even "dry"—moisture is easily picked up from the ambient humidity). Moreover, as the reinforcement in Sample A is directly exposed to water for 14 hours in the MIT water bath, the binder is hydrated, and water is transported into the roofing membrane by capillarity. At 110°C (230°F), in the MIT oven test, the residual moisture present in the uncured polyvinyl alcohol binder of Sample A begins to boil, and a complete delamination of the blend from the reinforcement is seen. In addition, there is a complete delamination within the reinforcement of the scrim from the fiberglass mat.

From this experience, the use of polyvinyl alcohol as a reinforcement binder in a modified bitumen roof membrane is quite suspect. These results show that the impregnation is affected.

Samples C and D are exactly the same, but the manufacturing of the reinforcements was slightly different, and the residual moisture was higher in Reinforcement C. Even when SB latex emulsion binders are used, problems can occur if the binder is not completely dried.

Reinforcements in Samples E and F are exactly the same. Sample E utilized a reinforcement batch that was more thoroughly cured than that used in Sample F.

This was determined through the use of differential scanning calorimetry, which is a reliable method to determine the moisture level and the curing (or drying) of the binder in a reinforcement. The binder used to manufacture reinforcements has to be dry and/or completely cured.

The reinforcement in Sample G is thermally bonded, and no blistering was observed during the MIT test. In this particular sample, the weave of the mat is more open; this facilitates impregnation of the membrane.

Samples H and I were manufactured from standard polyester mats as single, mid-reinforced modified bitumen membranes. Sample H exhibited 50 percent blisters after the MIT, while Sample I exhibited no blistering or physical change after the test.

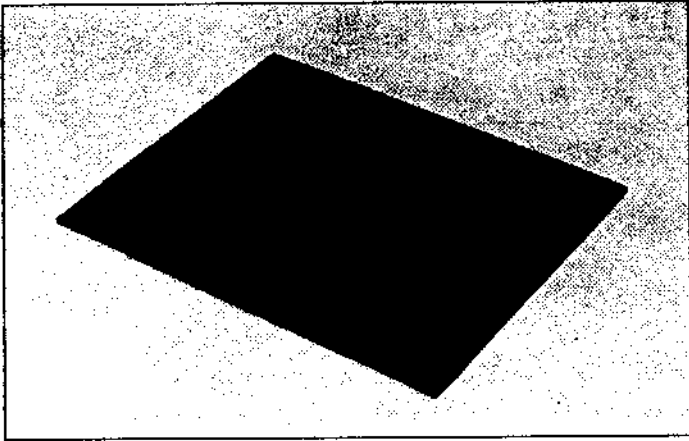


Figure 8. Sample B after MIT.

In addition to the tests discussed previously, other exploratory tests were made on reinforcements containing excessive amounts of binder. If the reinforcement fibers or strands have been heavily coated with excessive binder during manufacture, the finished reinforcement becomes coated by a continuous layer of binder. In some instances, the melting point of the binder is higher than the final impregnation temperature during membrane manufacture. The binder layer remains intact, and the heavy coating of binder acts as a barrier and will not allow a thorough impregnation of the reinforcement, because the blend cannot pass through the mat. When this type of product is subjected to the MIT, a complete delamination of the blend from the reinforcement (Figure 7) can occur.

CONCLUSION

The Membrane Immersion Test is an important step forward in the evaluation of a modified bitumen membrane's potential to perform over the long haul. Heat and moisture are key factors in the life of a roof membrane. Materials that have difficulty standing up to this kind of testing will have difficulty performing in real-world roof membrane situations. Although this test has been developed to measure impregnation performance in the factory, it may have potential for use in evaluating problem jobs.