USING THERMOANALYTICAL TECHNIQUES TO CHARACTERIZE ROOF MEMBRANE MATERIALS

RALPH M. PAROLI
National Research Council of Canada
Ottawa, Ontario, Canada

WALTER J. ROSSITER, JR.
National Institute of Standards and Technology,
Gaithersburg, Maryland

PETER FLÜELER
Swiss Federal Laboratories for Materials Testing and Research
Dübendorf, Switzerland

ANA H. DELGADO
National Research Council of Canada
Ottawa, Ontario, Canada

Many analytical methods are available within the family of thermoanalytical techniques for characterizing roof membrane materials. These techniques include classical methods, such as differential scanning calorimetry (DSC), differential thermal analysis (DTA), and thermogravimetry (TG), and modern methods, such as thermomechanical analysis (TMA), torsion pendulum analysis (TPA), and dynamic mechanical analysis (DMA). This paper summarizes the work of the RILEM/CIB Joint Committee on Membrane Roofing Systems—Thermal Analysis Task Group on TG and DMA Testing of Roofing Membrane Materials. The recommendations from the committee on how TG and DMA can be used to study new and in-service bituminous- and polymer-based roof membrane materials are presented.

KEYWORDS

Analysis, building technology, dynamic mechanical analysis, glass transition temperature, laboratory aging, outdoor exposure, roof membrane, thermal analysis, thermogravimetry, torsional pendulum analysis.

INTRODUCTION

The choice of roofing materials is quite varied, ranging from asphalt-based or polymer modified-asphalt [e.g., atactic polypropylene (APP) and styrene-butadiene-styrene (SBS)] to polymer-based materials, such as thermoplastic olefins (TPO), poly(vinyl chloride) (PVC), and ethylene-propylene-diene monomer (EPDM). This variety motivated the international roofing community to shift toward using both engineering and chemical principles to study roofing materials. In 1988, a joint RILEM/CIB* international roofing committee recommended that a task group be established to investigate the applications of thermal analysis (techniques used for measuring changes in the physical properties of a sample as a function of temperature) in the characterization of roofing membranes.[1]

*RILEM is the French acronym for the International Union of Testing and Research Laboratories for Materials and Structures. CIB is the French acronym for the International Council for Building Research Studies and Documentation.

In 1989, RILEM and CIB formed the Joint Committee on Membrane Roofing Systems, 120-MRS/W.83. It was composed of 40 members representing 22 countries. A task group on thermal analysis was initiated to examine the reproducibility of thermoanalytical techniques when applied to roof membrane materials and to investigate the feasibility of using these methods for detecting changes in membrane materials that may occur in service or under laboratory exposure conditions. This paper highlights the major findings and recommendations of the thermal analysis task group. A complete report from the task group has been issued.[2]

Thermal analysis is gaining popularity in the roofing industry.[3-23] Both the American Society for Testing and Materials (ASTM) and Canadian General Standards Board (CGSB) have initiated the development of standards that will include these techniques to evaluate roof membranes. Thermoanalytical techniques can be used to monitor a wide array of material characteristics. Some of the applications include enthalpy, weight loss, thermal stability, coefficient of thermal expansion (CTE), and the glass transition temperature (T_g). T_g is an important material characteristic, because below T_g, the material is rigid and hard, while above T_g, the material is flexible. Thus, T_g could be useful in evaluating the cold-temperature performance of roof membranes.[15] Other properties that vary above and below the T_g are thermal expansion coefficient, heat capacity, and dielectric constant.[24-28]

The RILEM/CIB committee investigated various techniques, including thermogravimetry (TG), dynamic mechanical analysis (DMA), and torsional pendulum analysis (TPA), to determine and monitor the changes in a roof membrane. A brief overview of TG, DMA, and TPA is presented in the following sections.

Thermogravimetry (TG)

TG measures the change in mass of a material as a function of time at a fixed temperature (i.e., isothermal mode) or over a temperature range using a predetermined heating rate. This technique is useful in monitoring heat stability and loss of components (e.g., oils, plasticizers, and polymers).

Dynamic Mechanical Analysis (DMA) and Torsional Pendulum analysis (TPA)[32-34]

Both dynamic mechanical analysis and torsional pendulum analysis provide similar information on mechanical properties. Both techniques measure a dynamic stress-strain relationship for a viscoelastic material. For both techniques, a load can be applied at predetermined frequency, and modulus data are monitored. The difference between the two methods is that in DMA, either a flexural or tensile load is applied, while in TPA, a torsional load is applied. For simplicity, only DMA will be discussed in the following paragraphs.

In conducting a DMA experiment, two components of modulus can be obtained by resolving the stress-strain components:

$$\sigma = \varepsilon_o E'\sin(\omega t) + \varepsilon_o E'' \cos(\omega t)$$

Where:

- σ is the stress;
- ε is the strain;
- ω is the angular frequency;
- t is the time;
- E' is the *storage modulus* [$E' = (\sigma_o/\varepsilon_o)\cos\delta$] and is a measure of recoverable strain energy in a deformed body;
- E" is the *loss modulus* [$E'' = (\sigma_o/\varepsilon_o)\sin\delta$] and is associated with the dissipation of energy as heat due to the deformation of the material;
- δ is the phase angle.

The ratio E"/E' yields the loss tangent or damping factor ($\tan\delta$), which is the ratio of energy lost per cycle to the maximum energy stored and, therefore, recovered per cycle. A typical dynamic mechanical analysis curve shows either E', E" or $\tan\delta$ plotted as a function of time or temperature. In general, the most intense peak observed for either E" or $\tan\delta$ in conjunction with a relatively pronounced drop in E' corresponds to the glass-transition temperature.[34]

Note that when conducting a TPA experiment, storage modulus and loss modulus are determined. However, because it is in torsional mode, G is used instead of E' and E"; this could affect the T_g values and, hence, the technique used needs to be reported.

The transition temperature determined by dynamic techniques is both heating-rate-dependent and frequency-dependent. Therefore, the test method, heating rate, frequency, and the mechanical/rheological property (E', E", G", G" or $\tan\delta$) used to determine the T, must be specified. E" has been preferentially used for T_g. In this regard, it has been found that the E" peak maximum at 1 Hz corresponded closely with the T_g obtained from volume-temperature measurements obtained by TMA.[21]

Sample Selection and Preparation

The RILEM/CIB thermal analysis task group included samples of EPDM, PVC, APP polymer modified bitumen, and SBS polymer modified bitumen roof membrane materials for the study. One commercial product of each type was included and subjected to the following:

- Outdoor Exposure

  The outdoor exposures were performed by Committee-member volunteers at: Haifa, Israel; London, UK; Tokyo, Japan; and Washington, D.C., USA. It was initially planned that, for a period of at least four years, the membrane samples were to be removed from the exposure racks every year and sent for analysis to the participating laboratories.[2] However, difficulties associated with sample retrieval precluded their being exposed at every site for the predetermined periods. (Please see Table 3 in Reference 2 for average weather data at the exposure sites.)

- Laboratory Aging

  The Building Research Establishment, UK, conducted the heat aging and water submersion exposures. For heat aging, the samples were placed in a forced-air ventilated oven at 70°C±2°C (158°F±4°F) (temperature variations are absolute limits) in ambient humidity for periods of 90 days and 180 days. For water submersion, the samples were hung vertically in separate tanks filled with deionized water maintained at both room temperature and 40°C±2°C (104°F±4°F) for periods of 90 days and 180 days. Each tank incorporated a stirrer to ensure even temperature distribution. The deionized water was changed every seven days.

EXPERIMENTAL

The task group selected thermogravimetry (TG), dynamic mechanical analysis (DMA), torsional pendulum analysis (TPA), and differential scanning calorimetry (DSC) to characterize the materials. They were analyzed "as received" (i.e., prior to exposure/weathering), after exposure in the laboratory to heat aging and water submersion, and after exposure to natural weathering in climates of four countries. The following experimental procedures were used:

- TG—Specimens weighing approximately 25 mg (apothecary/troy) (0.0008 ounces) were heated using a heating rate of 20°C/minute (36°F per minute) from room temperature to about 900°C (1652°F). The atmosphere was an inert gas (e.g., nitrogen or argon) for temperatures up to 600°C (1112°F) and air for temperatures beyond 600°C (1112°F). Granules or other surfacings (where appropriate) were removed. The analysis included reporting the mass percentage of the pyrolyzable organic, combustible organic, and residue constituents, as well as the temperature ranges over which they were lost. Five laboratories participated in the testing (identified as Laboratory Nos. 1, 3, 4, 5, and 6).
- DMA/TPA—The specimens were conditioned in an oven for 1 hour at 80°C (176°F) before testing. Low-temperature stabilization was achieved in the instrument test chamber before beginning an analysis. The clamping pressure varied from sample to sample but was applied in accordance with equipment manufacturers' recommendations. Where present, granules were removed before testing. Specimens were tested at 1 Hz with a heating rate of 2°C/minute (4°F per minute) from -80°C (-122°F) to 50°C (112°F). All specimens were cut in the machine direction. The analysis included reporting T_g, frequency, E' or G' below T_g (value at T_g minus 10°C [18°F]), and E' or G' above T_g (value at T_g plus 10°C [18°F]). Four laboratories conducted DMA testing, although two laboratories provided only limited data that, consequently, was not included in the final analysis (identified as laboratory Nos. 5 and 7). Only one labora-

tory participating in the study had access to a TPA (identified as Laboratory No. 3).

RESULTS AND DISCUSSION

TG Results

In a typical TG analysis, mass loss occurred over two temperature ranges: 1) from initial heating to about 600°C (1112°F) and 2) after the addition of air at about 600°C (1112°F) to the end of the test (see Figure 1). These ranges divide the data into three compositional groups: 1) pyrolyzable organic components liberated in the inert atmosphere, 2) combustible organic components oxidized in air, and 3) residue (i.e., ash) that remained after oxidation. The important compositional group is the pyrolyzable organic group. For the synthetic single-layer sheets (i.e., EPDM and PVC), they include the base polymer and additives such as processing oils, stabilizers, and plasticizers. For the modified bitumens, the pyrolyzable organics include the asphalt and the polymer modifiers. Variations that occur in the mass percent organic compositional group during exposure may be indicative of basic changes in the composition of the sheet including the base polymer. Thus, in considering the within and between laboratory variability, the TG analyses and discussions were limited to the findings for the pyrolyzable organic constituents. These TG data were from three laboratories (Nos. 3, 4, and 5). Other laboratories participated in the TG testing, but provided data insufficient for inclusion in the within and between laboratory variability analyses.

Variability within a Laboratory

To examine the question of variability within a laboratory, for each material, the relative standard deviation, also called the coefficient of variation, (i.e., the ratio of the standard deviation to the average times 100) for each set of measurements was plotted as a function of laboratory and either the exposure condition or time of exposure (see Reference 1b for plots). The variability within a laboratory for the sets of TG measurements was small. EPDM showed the least scatter while SBS displayed the most. With one exception, the average relative standard deviations for all laboratories for all sets of measurements were less than 1.7 percent.

The data from all laboratories showed slightly lower (less than 1 percent) average relative standard deviations for TG measurements of the synthetic materials (EPDM and PVC) than for the polymer-modified bituminous materials (~1 to 1.7 percent). This slight difference could be due either to material variability (i.e., the synthetic materials were somewhat more homogeneous than the polymer modified bitumens) or to the laboratories' techniques in conducting TG analyses of the synthetic samples vs. the bituminous samples.

Variability between Laboratories

To examine the question of variability between laboratories, the difference between average mass percent pyrolyzable organics for a single laboratory and that of all laboratories was calculated. The question addressed was whether the values for a given laboratory were consistently higher or lower than those of the other laboratories. This provided a measure as to whether there is a bias in the data from that laboratory versus the other laboratories.

Statistical analysis showed no statistically significant differences (99.9 percent confidence level) between the average measurements of Laboratory Nos. 3, 4, and 5 for the EPDM and PVC materials. In the case of outdoor exposed APP, Laboratory No. 4 reported higher values than Laboratories Nos. 3 and 5, thus resulting in a significant difference between the average measurements of the laboratories. Also, a statistically significant difference was found between the laboratories for the analyses of the laboratory-exposed SBS material because of the slightly higher values reported by Laboratory No. 5. These statistically significant differences were not practically important.

In spite of the two cases where a statistically significant difference was found between the three laboratories, in general, the results of the data analysis of between-laboratory variability were good. In the worst case of the analysis of the outdoor-exposed APP, the bias from one laboratory vs. the others was about 6 percent. The data for the synthetic EPDM and PVC products displayed slightly less bias than those for the APP- and SBS-modified bituminous materials. This finding was similar to that of the data analysis of the within-laboratory variability.

DMA/TPA Results

Typical DMA and TPA plots are shown in Figures 2 and 3, respectively. Two laboratories (Nos. 5 and 7) provided the

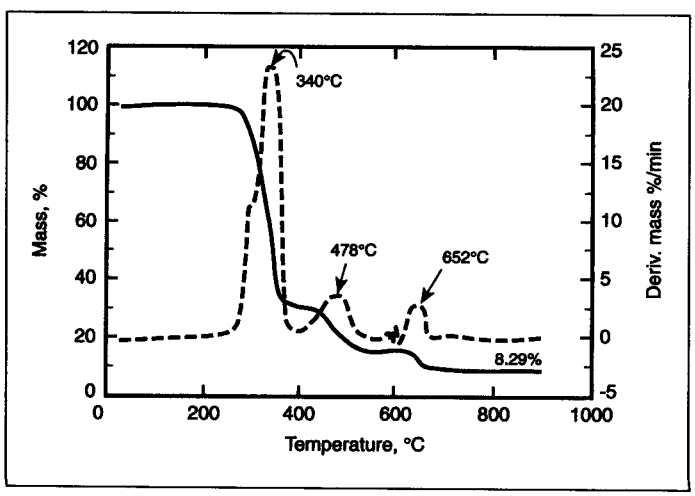

Figure 1. Typical TG (solid line) for the analysis of PVC; the dashed line is the derivative of the TG curve (DTG).

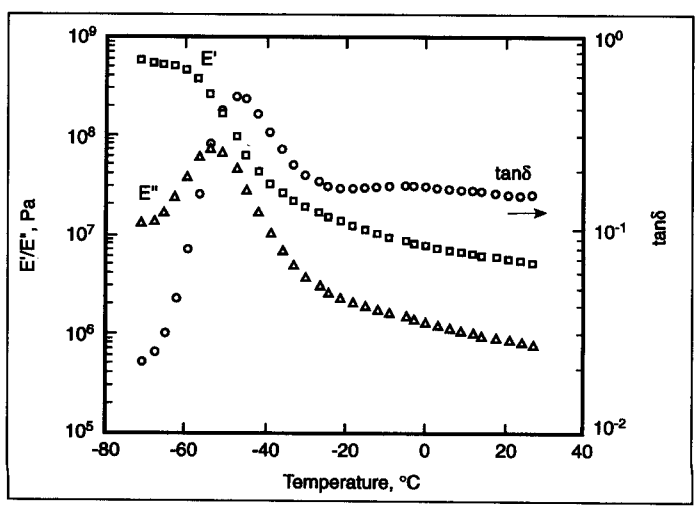

Figure 2. Typical DMA curves for the analysis of EPDM.

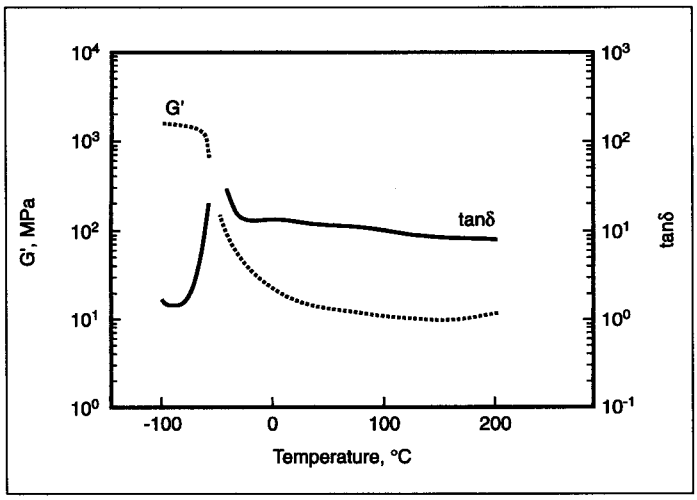

Figure 3. Typical TPA curves for the analysis of EPDM. The break in the two curves over the temperature range of -40°C to -60°C (-40°F to -76°F) indicates where data were not obtained. ***Note: The laboratory conducting the TPA tests did not plot the G" curves when reporting to the CIB/ RILEM Committee.***

DMA data. A greater number of specimens were analyzed by Laboratory No. 5, as Laboratory No. 7 volunteered to participate in the study after it was under way. The TPA data were provided by Laboratory No. 3. Because of the limited number of laboratories participating in the DMA/TPA testing, statistical analyses of the variability between laboratories were not conducted. Nevertheless, some qualitative statements comparing the results within laboratories were made.

Variability within a Laboratory

In the case of DMA, the T_g measurements from the $E"_{max}$ illustrated the within-laboratory variability. Under the experimental conditions, the two laboratories conducting the DMA analysis found that the T_g values generally were reproducible within ±2°C (4°F) for the four types of membrane samples. Further work is required to determine the reproducibility of the E' and E" modulus data over the given temperature range.

In the case of TPA, Laboratory No. 3 generally only performed one or two analyses per sample per exposure. As a measure of within-laboratory reproducibility, this laboratory conducted a triplicate analysis of the PVC sample exposed outdoors in London for two years (Table 1).

The results in Table 1 showed that:

- the coefficient of variation (CoV) of G' at about 23°C (73°F) was 10 percent;
- the CoV of G' at peak maximum was 6.0 percent;
- the CoV for G' at G_{max} (i.e., the value of G' at T_g) was 2.3 percent.

That is, as the G' value increased, the CoV decreased. Additionally, the standard deviation for the temperature at $tan\delta_{max}$ and at $G"_{max}$ was 0.4°C (0.07°F) or less, which is small.

Variability between Laboratories

Because of the limited number of participating laboratories and DMA/TPA data obtained, the variability between laboratories could not be determined.

Comparison of Glass Transition at $E"_{max}$ vs. $G"_{max}$.

Recall that the value of the T_g may be dependent upon the testing method, heating rate, and frequency. In this study, because both DMA and TPA were used and both obtain similar modulus data, it was of interest to compare the values of T_g at $E"_{max}$ measured by Laboratory No. 5 and those of T_g measured at $G"_{max}$ by Laboratory No. 3. The comparison indicated that, in general, the T_g values at $G"_{max}$ obtained using TPA were higher (i.e., warmer temperature) than those measured at $E"_{max}$ by DMA. The exception was some of the EPDM samples. The differences in T_g values measured using the two methods was as follows:

- for EPDM, 1 to 4°C (2 to 7°F)
- for PVC, 1 to 10°C (2 to 18°F)
- for APP, 2 to 8°C (4 to 14°F)
- for SBS, 1 to 22°C (2 to 40°F)

Reasons for the differences were not investigated.

		G´ at +23 °C		Temp. at tanδ Peak Maximum			Temp. at G´´ Peak Maximum		
	Thickness mm	Temp. °C	G´ MPa	Temp. °C	tanδ	G´ MPa	Temp. °C	G´´ MPa	G´ MPa
Rep. 1	1.134	23.0	14.4	-10.2	0.343[a]	89.9	-40.7	105.4[b]	610
Rep. 2	1.131	23.4	17.0	-10.3	0.301[a]	89.3	-40.7	108.0[b]	589
Rep. 3	1.135	22.7	17.5	-10.9 -14.0	0.304[a] [0.307[c]]	99.2 124.2	-41.3	109.2[b]	615
Mean	1.133	23.0	16.3	-10.5	0.316	92.8	-40.9	107.5	605
sd[d]	0.002	0.4	1.7	0.4	0.023	5.6	0.3	1.9	14
CoV, %[e]	0.18	0.17	10.0	3.8	7.3	6.0	0.73	1.8	2.3

[a] Maximum of data set in series plateau or approx. linear increase of values
[b] Maximum of a temperature interval of about 15 to 20°C
[c] Absolute maximum value
[d] sd is the standard deviation
[e] CoV is the coefficient of variation

Table 1. Analysis of PVC exposed outdoors in London for two years as an illustration of TPA reproducibility.

Changes in Membrane Properties Brought about by Laboratory and Outdoor Exposure

One objective of the thermal analysis task group's activities was to determine whether the results of measurements obtained from the TG and DMA/TPA are applicable to demonstrating the stability of materials and to comparing performance of materials under both laboratory and outdoor exposure. Consequently, the TG and DMA/TPA data were analyzed to determine whether the laboratory and outdoor exposures brought about changes in the measured membrane material properties. Because more data were obtained from the TG measurements, a formal statistical analysis was conducted using the TG data. In the case of the DMA/TPA data, where fewer points were available, general comparisons were made without the use of statistics. As will be discussed in the paragraphs to follow, only minor changes in membrane material properties as a function of the type of exposure were found for the products tested.

In the case of the TG data, plots of the mass percent pyrolyzable organic constituent as a function of time for each material and outdoor-exposure location were prepared, and the data were fitted to a linear model. Examination of the plots did not support using a more complex model. In most cases, it was found that the slopes of the lines were not statistically significantly different from zero (i.e., the magnitude of the slope was not at least three times greater than its standard deviation). In those cases where a statistically significant difference existed, no pattern to the direction of the change was observed. In some cases, the slope was positive, and in others, it was negative. In addition, when the slopes were statistically significantly different from zero, the estimated changes (increase or decrease) in the mass percent pyrolyzable organic constituents for the outdoor exposed samples were generally of the order of about 1 percent per year, with the maximum being about 2 percent.

Similar statistical analyses of the data set for the laboratory-exposed samples were not performed, because the data over time were limited (i.e., only two exposures, 90 days and 180 days, were conducted). Figure 4 presents plots of the average mass percent pyrolyzable organic constituents as a function of exposure as determined by all laboratories. Averaging all data from all laboratories was considered acceptable because, as previously discussed, little variability existed between the average TG results from the laboratories. For each membrane material, note in Figure 4 that the mass percent pyrolyzable organic constituent shows little variability regardless of exposure or time. Note also that variations, where apparent, show either an increase or decrease in the average mass percent pyrolyzable organic constituents for the exposed samples vs. those of the controls.

In the case of the DMA/TPA data, the comparison between the controls and exposed samples were consistent with the results of the TG analyses. Generally, only minor changes in the measured membrane material properties were found.

To illustrate this finding using DMA data, Figure 5 contains plots of T_g at E''_{max} vs. outdoor exposure time. With the exception of the SBS material, little variation in E''_{max} over time is observed. In the case of SBS, considerable scatter in the data was found. The apparent decrease in T_g over time may be, in part, due to the relatively low value measured at time zero (i.e., the control sample). One hypothesis is that the SBS sample underwent a slight change initially because of heating; another hypothesis is that the apparent decrease in T_g over time was due to variability in the SBS sample.

Figure 6 shows values T_g at G''_{max} vs. outdoor exposure time as determined by TPA. With the exception of PVC exposed

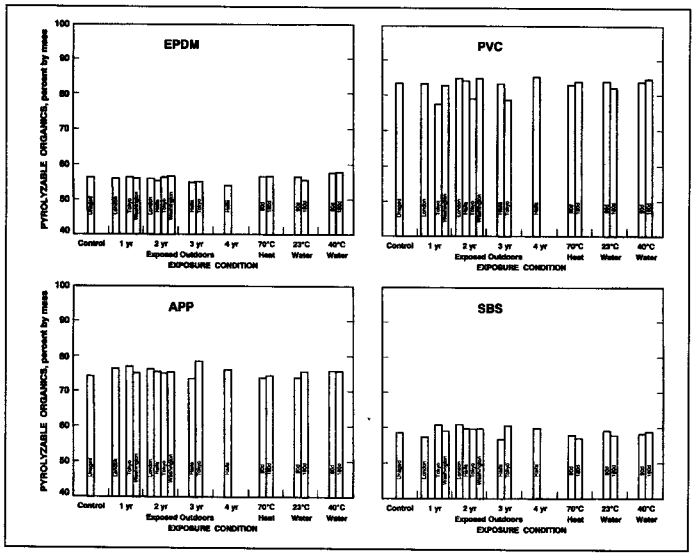

Figure 4. TG analysis as a function of exposure and time.

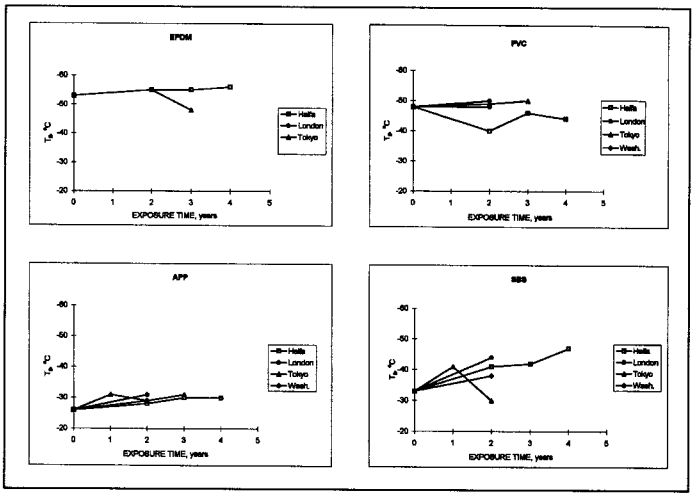

Figure 5. T_g at E" as a function of outdoor exposure time.

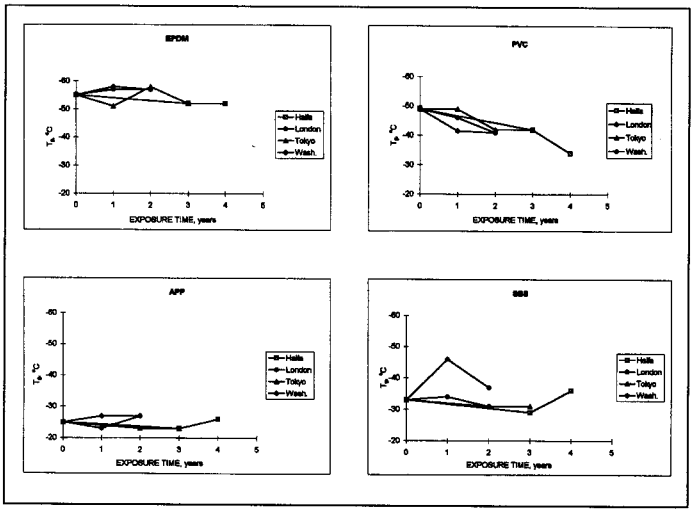

Figure 6. T_g at G" as a function of outdoor exposure time.

in Haifa, the T_g values at G''_{max} were relatively constant over time. For example, the PVC samples exposed at London, Tokyo, and Washington showed increase of T_g of about 7°C to 9°C (13°F to 16°F) after exposure. In contrast, the sample exposed at Haifa had an increase in T_g of 15°C (27°F), which was the largest measured change (perhaps because of Haifa's high temperatures). In comparison, the largest change in T, measured for the Haifa-exposed PVC by DMA was an increase of 8°C (14°F). Finally, in the case of the SBS analyzed by TPA, Figure 6 shows considerable scatter (similar to the DMA results), which was due to the determination of G''_{max}. The peak in the data output was often broad, as well as flat, and often contained more than a single maximum.

In conducting either a DMA or TPA analysis, the thickness of the sample must be measured accurately. Laboratory No. 3 plotted the thickness data associated with its TPA measurements. The plots are given in Figure 7 for the samples exposed outdoors. Note that thickness measurements were not conducted on the unexposed samples before they were set on exposure. The initial thickness measurements (i.e., zero time) in the plots are taken from the control samples. Thus, it is possible that the trends in the plots are overly influenced by those zero time data. A summary of the thickness results is: for the synthetic EPDM and PVC materials, the samples exposed at Haifa showed a relatively high decrease in thickness, about 6 and 4 percent, respectively. The other exposure sites showed a slight trend of increasing thickness over time, and for the bituminous APP and SBS materials, a decrease of about 8 percent in thickness was found over four years' exposure.

Comment on the Lack of Change with Exposure Time

In discussing the effects of laboratory and outdoor exposures on the membrane material properties, the committee considered that the membrane materials were relatively stable under the exposure conditions. The TG and DMA/TPA methods are well-established as being sensitive for characterizing polymeric and similar materials and changes they may undergo during environmental exposures. Consequently, if major changes had been brought about by the exposures, they would have been observable by one or more of the methods. For example, both TG and DMA identified an EPDM product that was experiencing problems in the field in Canada.[8] This EPDM product has been withdrawn from the market.

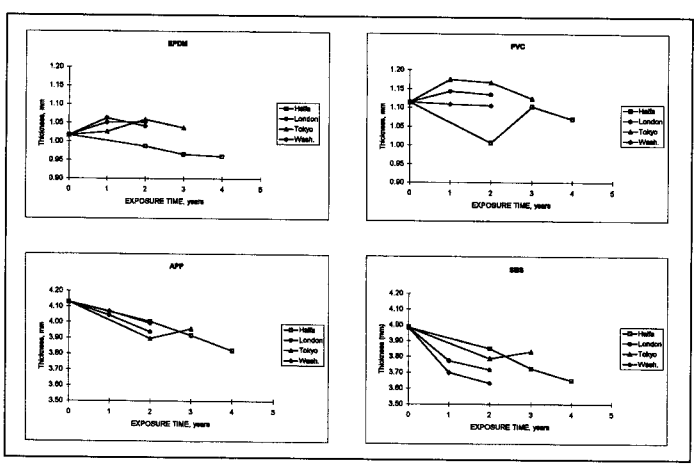

Figure 7. Specimen thickness as a function of outdoor exposure time.

Regarding the present study, field experience has shown that the four membrane materials have generally performed well in service for considerable lengths of time (e.g., 10 years or more). As a consequence, it might be expected that they would be found to be stable under the relatively short laboratory and outdoor exposures used in the study.

CONCLUSIONS AND RECOMMENDATIONS

Based on the results of the TG and DMA/TPA testing and analyses of the EPDM, PVC, APP modified bitumen, and SBS modified bitumen membrane materials, the RILEM/CIB Committee made the following conclusions and recommendations.

TG Conclusions

Applicability

The method was found to be readily applicable to roof membrane materials.

Variability

The method showed rather low variability within- and between-laboratory.

TG Recommendations

TG is an acceptable method for characterizing new and aged roof membrane materials. It is complementary to other mechanical, chemical, and physical methods used for membrane material characterization. It may be used for a variety of applications, such as evaluating stability under laboratory and outdoor exposure or providing quality control measurements.

The change in the mass percent of the organic constituent after exposure (i.e., aging) should not exceed ±3 percent (absolute) if a material is to be considered stable to the exposure conditions. Changes greater than 3 percent may indicate changes in the material resulting from the exposure.

Test Procedure

The parameters selected for conducting the analyses were suitable for roofing membrane materials. A standard test procedure needs to incorporate the RILEM/CIB committee test parameters which are:

- heating rate: 20°C (36°F) per minute
- heating range: room temperature to about 900°C (1652°F)
- atmosphere: an inert gas (e.g., nitrogen or argon) for temperatures up to 600°C (1112°F) and air for temperatures beyond 600°C (1112°F)
- specimen size: 25 mg (~0.0008 ounces); full thickness of the sheet; remove granules or other surfacings, where appropriate

Analysis

Report the mass percentage of the pyrolyzable organic, combustible organic, and residue constituents, and the temperature ranges over which they are lost.

DMA/TPA Conclusions

Applicability

The method was found readily applicable to roof membrane materials.

Variability
The number of participating laboratories was limited so that definite statements on within-laboratory and between-laboratory variability are not made.

DMA/TPA Recommendations
DMA and TPA are acceptable methods for characterizing new and aged roof membrane materials. They are complementary to other mechanical, chemical, and physical methods used for membrane material characterization. They may be used for a variety of applications, such as evaluating stability under laboratory and outdoor exposure or providing quality control measurements. Recall that DMA provides tensile modulus data while TPA provides shear modulus data. Hence, if a laboratory begins a series of analyses using one of those methods, then it should use the same method throughout the series.

After exposure, the change in T_r should not exceed 8°C (14°F), and change in the storage modulus (E'/G') at T_r ±10°C (18°F) should not be more than a factor of ten, if a material is to be considered stable to the exposure conditions. Changes greater than these values may indicate changes in the material resulting from the exposure.

Test Procedure
The parameters selected for conducting the analyses were suitable for roof membrane materials except that the temperature range of -100°C to 100°C (-148°F to 212°F) was not necessary. A standard test procedure needs to incorporate the following RILEM/CIB committee test parameters:

- heating rate: 2°C (4°F) per minute
- temperature range: -80°C to 50°C (-112°F to 112°F)
- frequency: 1 Hz (if possible)
- specimen orientation: machine direction
- conditioning of the specimens: in an oven for one hour at 80°C (176°F) before testing
- low-temperature stabilization: achieve before testing
- clamping pressure: unspecified; applied in accordance with equipment manufacturer's recommendations
- where present, remove granules before testing

Analysis
Report T_g, frequency, E'/G' below T_g (value at T_g minus 10°C [18°F]), and E'/G' above T_g (value at T_g plus 10°C [18°F]).

SUMMARY

Thermal analysis shows much promise in providing quick and reliable data regarding the stability of roof membranes. TG, DMA, and TPA provide information, which is complimentary to the other, as well as mechanical tests. Thus, these techniques can be of assistance in quality control and when trying to understand why a membrane is exhibiting unusual behavior. In the near future, one would anticipate that the recommendations of the RILEM/CIB would be implemented into the relevant international membrane standards. In Canada, the next version of the PVC standard will be addressing this issue. In the United States, ASTM D-08 (Roofing) has initiated the development of standards, which will include these techniques to evaluate roof membranes based on the RILEM/CIB recommendations.

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