

Lifetime Prediction of Geotextiles Using DMA

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ABSTRACT: Dynamic mechanical analysis (DMA) fundamentals are introduced. Various DMA techniques were applied to single polypropylene fibers. The wide range of testing available using DMA provided several methods to monitor material degradation due to chemical exposures and collect data which could be used to estimate the material's lifetime. Time-temperature superposition techniques are introduced and applied to DMA data to predict material property changes over time. Results indicated the usefulness of superposition in predicting long term performance of geotextiles.

1 Introduction

The application of geotextiles in hazardous waste landfills compromises the lifetime of these materials. It is of great importance to determine (or at least estimate) the useful lifetime of these materials.

The integrity of a hazardous waste site is of the utmost importance and is subject to various degradation mechanisms. Exposure to heat and oxygen eventually induces chain scission of the polymer molecules. This reaction results in decreased average molecular weight and increased molecular weight distribution. Such changes on a molecular level transcribe to changes in the bulk material which are manifested in ways such as decreased tensile strength. Since degradation occurs on a molecular level, test methods capable of detecting molecular changes must be employed in aging studies.

This project was undertaken in an effort to advance the science of lifetime prediction of geosynthetics. The intention was to develop test methods capable of producing data that provide insight into the material's service life. Such tests had to be capable of examining the molecular structure of the material since molecular changes precede bulk physical changes.

2 Background

Many studies of polymeric materials are concerned with the physical or mechanical properties of the substance. Quite often, tests such as tensile, tear and melt flow are performed

to assay these characteristics. These methods, however, only provide information about macromolecular behavior. In order to probe the mechanical properties of materials on a micromolecular level, DMA is used.

2.1 Dynamic Mechanical Analysis (DMA)

The operation of a dynamic mechanical analyzer is centered around the sinusoidal application of small amounts of force to a sample. In general, a material's response to stress is expressed in terms of strain. In DMA, the minute sinusoidal stresses (σ) applied to the sample cause a time dependent response in strain (γ). The time difference between application of force and response in strain is referred to as the phase, phase lag, phase angle or the delta (δ). The relationship among these parameters is illustrated in Figure 1.

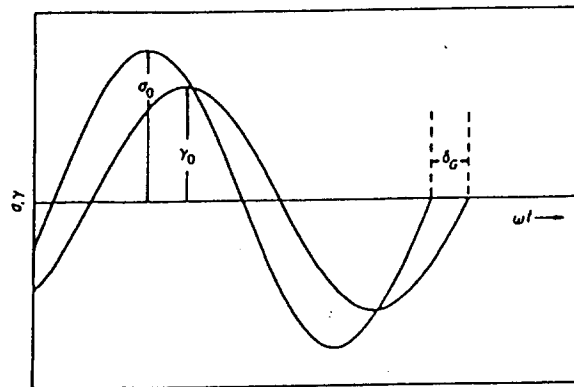


Figure 1. Time Dependence of the Shear Stress (σ) and Shear Strain (γ) (Reed, 1978).

The phase lag is the result of molecular rearrangements and various relaxation phenomena. For instance, a rigid, high modulus material has a large tangent δ whereas pliable, low modulus materials exhibit small values of tangent δ .

The concept of modulus is complicated in dynamic studies due to the time lag between stress and strain. The result of this is that modulus is separated into two components - loss and storage moduli. [Since it is not reasonable to discuss the derivation and ramifications of these moduli in this paper, the reader is referred to various writings on the topic (Sperling, 1992; Nielsen, 1974).] Therefore, dynamic mechanical studies can provide insight into the relationship between dynamic properties and structural parameters such as molecular weight, orientation and crosslinking.

Computer controlled DMA instrumentation is currently available. The instrumentation is commonly of three types: free vibration, resonance vibration and wave propagation. For this study, a resonance vibration type instrument was used. This type of instrument exerts a constant amount of sinusoidal force on a sample.

The instrument was capable of performing tests in five different modes. These were: frequency scan, stress scan, time/temperature scan, thermomechanical analysis (TMA) and stress relaxation.

The three operational parameters for DMA are frequency of applied force, temperature and stress. The relation of operational modes, testing parameters and output measurements are summarized below.

Operational Mode	Testing Parameter			Output Measurement
	Freq.	Temp.	Stress	
Frequency Scan	Δ	C	C	Strain, Phase angle
Temp. Scan	C	Δ	C	Strain, Phase angle
Stress Scan	C	C	Δ	Strain, Phase angle
TMA	N/A	Δ	C	Strain
Creep-Recovery	C	C	Δ/C	Strain, Time

Δ - Changes, C - Constant

Each of these five modes can provide different information about the analyzed material.

In order to understand the various types of phenomenon occurring during dynamic mechanical analysis, some insight into viscoelastic theory is helpful. However, it is possible to interpret DMA data by starting with complex modulus. Complex modulus, E^* , relates storage modulus, E' , and loss modulus, E'' using vector displacements and speed (for DMA in extension mode with a fiber) as follows:

$$E' = \frac{ky}{\pi d^2} \quad ; \quad E'' = \frac{\omega D}{k} E' \quad ; \quad E^* = \sqrt{(E')^2 + (E'')^2}$$

where k is the spring constant, y is the displacement along the axis of the fiber (strain), d is the fiber diameter, $\omega = 2\pi\nu$; ν = frequency in Hz (radians/sec). and D is the inertial

damping factor of the material. From this, complex viscosity, η^* , can be defined.

$$\eta^* = \frac{E^*}{\omega}$$

Other reporting variables can be calculated such as compliance, shear modulus and tangent delta.

2.2 Time-Temperature Superposition

Time-temperature superposition is a method that can be applied to lifetime prediction work. The theoretical basis for this lies in the viscoelastic nature of polymers.

Viscoelastic materials simultaneously exhibit elastic and viscous behavior (Ferry, 1980). Sperling states, "Generally, viscoelasticity refers to both the time and temperature dependence of mechanical behavior." (Sperling, 1992) It is this conclusion of viscoelastic theory which provides the basis for time-temperature equivalence.

Time-temperature equivalence in its simplest form implies that viscoelastic behavior at one temperature can be related to that at another temperature by a change in the time scale only. As an example, consider the creep compliance curves of an ideal polymer at two temperatures, T_1 and T_2 (Figure 2).

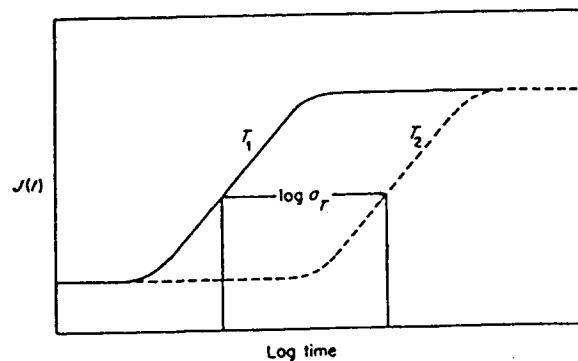


Figure 2. Schematic Illustrating the Simplest Form of Time-Temperature Equivalence (Ward, 1993).

In the case of ideal time-temperature equivalence, these curves can be superimposed (exactly) by a horizontal displacement ($\log a_T$). The superposition comes from applying the Boltzmann superposition principle to equations involving the temperature dependence of linear properties. The $\log a_T$ factor here is the same as that expressed in the Williams-Landel-Ferry (WLF) equation (Ward, 1993). This results in showing that all linear viscoelastic properties obey a time-temperature superposition principle within degrees of T_g (Dealy, 1990). The reader is directed to the literature for a thorough treatment of time-temperature relationship, theory and application (Sperling, 1992; Ferry, 1980).

3 Experimental

Polypropylene fibers were obtained from Spartan Technologies, Spartanburg, SC. The five denier material was received in non-needlepunched form.

Dynamic mechanical analysis was performed on a Perkin-Elmer DMA 7. Computer control, data acquisition and calculations were provided by the Thermal Analysis Controller 58 (PE 7700 computer) running Thermal Analysis Data Software[®] (TADS) version 2.43.

4 Results and Discussion

Application of DMA to single geotextile fibers was performed here for the first time. The instrument's fiber extension measuring system, sensitive control and detection electronics made this possible. As mentioned previously, the instrument was capable of running in five modes. The frequency scan mode was used for the work reported here. The available range was 0.01-51 Hz. Examination into the various reporting variables was required.

Figure 3 shows a typical frequency scan composite of three unexposed polypropylene fibers with complex modulus as the reporting variable.

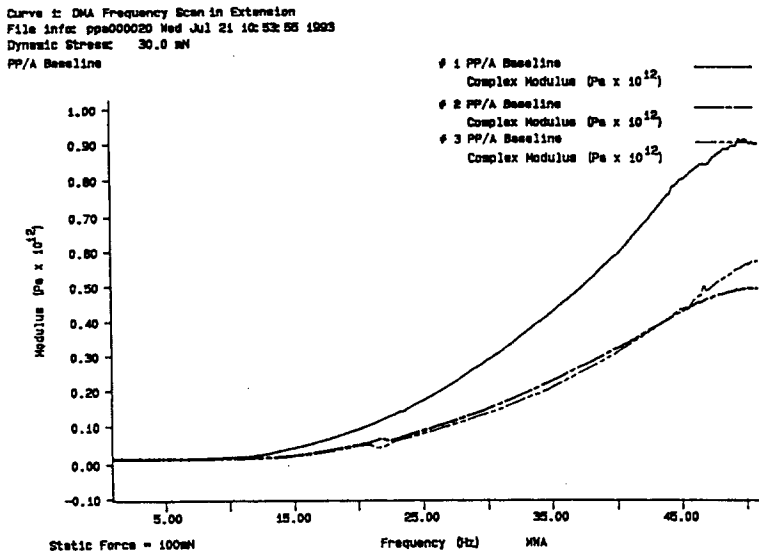


Figure 3. Complex Modulus vs. Frequency for Unexposed Polypropylene Fibers.

At 22.40 Hz, the material's resonance frequency is attained, resulting in a small, sharp signal. It should be noted here that this characteristic is present in all frequency scans of this material. At 33 Hz, another transition (β transition) is observed which is followed by the primary (α) transition at 45 Hz. The broadness of the peaks is due to the presence of plasticizers which have a limited solubility in the polypropylene resin (Ward, 1993).

This composite indicates good reproducibility of data, particularly in the cases of #2 and #3. The offset of #1 is

unexpected since single fiber testing should eliminate differences among specimens. The sample mounting technique may have some limitations which are reflected in these data.

In order to apply time-temperature superposition, a series of scans was required, each at different temperatures. Test temperature limits were -65 °C to 500 °C.

Frequency scans were performed using complex viscosity as the reporting variable. This was chosen since it demonstrates a linear response with respect to frequency. For each scan, logarithmic data of frequency (0.01, 0.02, 0.05, 0.07, 0.10, 0.25, 0.50, 0.75, 1.00, 2.50, 5.00, 7.50, 10.00, 20.00, 30.00, 40.00, and 50.00 Hz) vs. complex viscosity (η^*) were plotted and are shown in Figure 4.

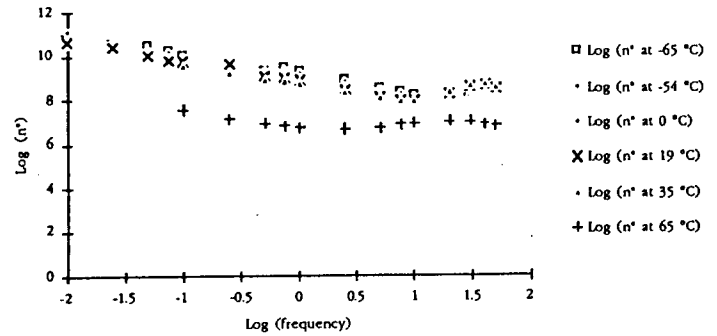


Figure 4. Log η^* vs. Log frequency for Acid Exposed Material at 22 °C for 30 days.

The data are then regressed in order to provide straight lines for further work. The result of this is shown in Figure 5 below.

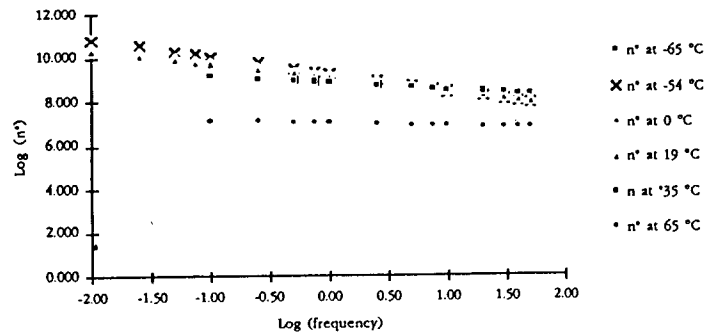


Figure 5. Regressed Data from Figure 4.

To satisfy the requirements of the superposition principle, three temperatures are chosen which do not demonstrate overlap. It was not expected to require such large temperature differentials (> 30 °C) in this work.

The shifting of the data can now be performed. The reference temperature is arbitrarily chosen to be 65 °C. The curves for the other temperatures are shifted with respect to the one at 65 °C. This is performed by finding the difference in both the abscissa and ordinate values of the left-most point of the reference curve and the right-most

point of the curve to be shifted (35 °C). These two points are then brought together. This action produces a new leftmost point that is used for the next shift. The result of all shifting is shown below.

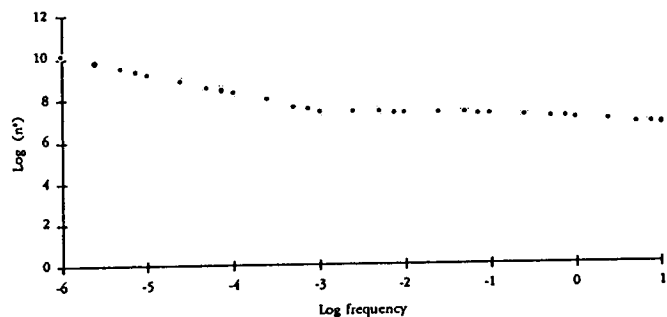


Figure 6. Result of Shifting Data of Acid Exposed Polypropylene Fibers

Following shifting, the anti-log (of the abscissa and ordinate) is calculated and frequency is inverted to units of time. This results in a master curve of complex viscosity vs. time (>500 hours).

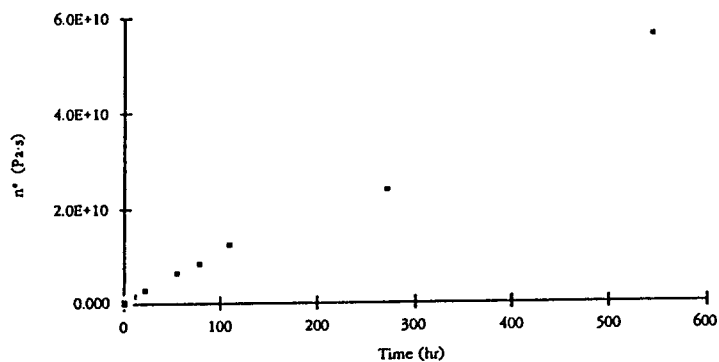


Figure 7. Master Curve for Polypropylene Fibers in H_2SO_4 .

Here we see that the complex viscosity of polypropylene fibers can be estimated at 65 °C in sulfuric acid after 500 hours.

5 CONCLUSIONS

5.1 Dynamic Mechanical Analysis

The application of DMA complements current physical property test methods already in place. Since DMA is a mechanical analytical technique, the data are indicative of mechanical performance. The ability to apply minute amounts of stress to the sample, however, increases its usefulness for microstructure analysis. In this study, DMA proved its ability to probe the microstructure of the materials and report on various physical properties.

This study employed single fiber testing in DMA. This method proved worthy of further study. It is now evident that DMA is a valuable tool in geotextile testing.

5.2 Time-Temperature Superposition

Time-temperature superposition was introduced as a method of predicting the long term performance of geosynthetic materials. The treatment of data illustrated here determined that mechanical properties could be predicted over long periods of time from data collected in a very short period of time. In this fashion, it could prove very useful.

Although not discussed here, additional work showed that incorrect data manipulation can lead to extreme results. The project reported here serves only as an introduction into the usefulness of time-temperature superposition.

6 ACKNOWLEDGEMENTS

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