Memo to: James Schluter
From : Riad H. Gobran
Date : September 30, 2004
Subject : Florida's DOT Protocol for 100-Year Service Life of HDPE Pipe

Introduction:

Florida's DOT is considering the use of corrugated HDPE as a drain pipe for a 100-year service life. Protocols are proposed to test the pipe to insure it will meet the required service life.

Several factors can affect the service life; the ones studied included stress crack resistance (SCR), oxidation resistance, tensile strength and flexural modulus. These factors were studied separately to simplify an otherwise very complex situation. In the final analysis, the service life of the pipe will be determined by whichever process gives the shortest time to failure. Obviously, when multiple factors are going on at the same time, the service life will be even shorter.

Comments in this report will follow the areas studied individually and final conclusions and recommendations will be presented.

1- Stress Cracking:

Dr. Hsuan, in her report dated August 12, 2003, showed measurements of Stress vs. time for two samples of pipe; P-1 and P-2, made from different resins and under different processing conditions. The measurements were carried out in water at 3 temperatures; 40°, 50°, and 60°C and the results were plotted in Figure 6 and Figure 7 shown on page 12 of Dr. Hsuan's report and reproduced in attached Appendix A

1.1 Stress cracking extrapolated to a site temperature of 20°C:

The results from individual temperatures were transformed into master curves for each pipe, using certain "Popelar shift factors" for time (a_T) and stress (b_T) to reflect a site specific temperature of 20°C. The shifted data are shown in Figures 8 & 9 for P-1 and P-2, respectively (shown in attached Appendix B). From the resulting plots, a relationship was arrived at relating stress and failure time for each pipe:

For P-1 in water: $\sigma = 14227\ t^{-0.426}$

Applying appropriate shift factors for test temperature of 50°C and target site temperature of 20°C according to equations 1 & 2 (page 9 of Dr. Hsuan's report and Appendix C & D attached), the following was calculated:

Stress , σ = 64 psi for 100-year crack free pipe (page 15)

Note: Dr. Hsuan inadvertently used the wrong equation for Pipe-1 by using an exponent of -0.437 instead of -0.426. Had she used the right exponent, the allowed stress would be 74 psi instead of 64 psi for 100-year service life.

In any case, if the site temperature is higher than 20°C, the allowable stress for crack-free 100 year service would be lower as shown below.

1.2 Stress cracking for a site temperature of 26.7°C:

Using the methodology outlined on page 15 for a junction test in water, the shift factors, a_T and b_T, in equations (1) and (2) were calculated assuming an average temperature of 80°F (26.7°C) which is more likely to be the case in Florida. The new values are:

a_T = O.0789 and b_T = 0.1.31

When these values are used, the calculated stress for 26.7°C is:

σ = 50 psi for 100-year crack free pipe

This is a substantially lower allowable stress than the 64 psi (or actually 74 psi) calculated assuming a site temperature of 20°C.

Back calculating the life time, the 64 psi allowable stress for 20°C, would result in a life time of only 56 years for a site temperature of 26.7°C. If the calculated stress is 74 psi as shown above, the life time at 26.7°C would only be 40 years.

1.3 Test protocol for Stress Crack resistance:

In Appendix I entitled "A summary Discussion on the Interim Specification for 100-Year Service Life of High Density Polyethylene Corrugated Pipes", it is stated on page 101 (Attached Appendix E)that:

"From the stress analysis reported in Part I of the test protocol, the long term tensile stress in both longitudinal and circumferential directions of the corrugated pipe is 500 psi after adding a factor of safety of 1.5. This means that the corrugated pipe should not exhibit brittle cracking under a stress of 500 psi for 100 year." This is based on 20°C site temperature.

1.4 Calculations for tests at 80°C and site temperature of 20°C:

In order to speed the results, the test temperature recommended is 80°C. Following is the method used to calculate the corresponding stress and time.

Applying Popelar shift factor backward to obtain stress and failure time at 80°C based on 500 psi and 100 year at 20°C, shift factors were calculated to be:

(a_T) = 0.00144 time shift for 60°C temperature difference;

(b_T) = 2.006 stress shift for 60°C temperature difference.

This leads to the following stress and failure time:

σ_{80} = 250 psi

t_{80} = 1260 hours

Thus, for test purposes at 80°C and assuming site temperature of 20°C, the criteria are:

"At a stress of 250 psi, failure time to achieve 100-year crack-free service has to be above 1260 hours."

1.5 Calculations for tests at 80°C and site temperature of 26.7°C:

Using the same methodology, the shift factors were recalculated to reflect a site temperature of 26.7°C (80°F). The new shift factor values are:

(a_T) = 0.0030 time shift for 53.3°C temperature difference, and

(b_T) = 1.855 stress shift for 53.3°C temperature difference.

Based on these new shift factors, the new stress and failure time at 80°C become:

σ_{80} = 270 psi

t_{80} = 2628 hours

Thus, for test purposes at 80°C and assuming a site temperature of 26.7°C, the criteria become:

"At a stress of 270 psi, failure time to achieve 100-year crack-free service has to be above 2628 hours."

This shows that the minimum time to failure for a 26.7°C site temperature is more than twice that for a 20°C site temperature. Stated differently, the life time calculated for a site temperature of 26.7°C would be less than half the 100 years calculated for 20°C site temperature, i.e. less than 50 years.

Note: Measurements at high stress should be avoided since this induces ductile failure.

2. Oxidation Resistance:

According to the representation in Figure 14 of Dr. Hsuan's report, there are 3 conceptual oxidation stages of HDPE (Appendix F):

Stage A represents time to consume all of the antioxidant in the pipe which depends on the type and amount of antioxidant, as well as the surrounding environment.

Stage B is the induction time which is the inherent property of the unstabilized polymer before rapid degradation takes place.

Stage C is the autocatalytic stage of the oxidation process.

In the oxidation test, pipe samples are placed in a forced air oven or in water bath at elevated temperatures to accelerate the oxidation process. After a certain incubation time, the Oxidation Induction Time (OIT) is determined. OIT measures the duration for the polymer to oxidize at a constant temperature of 200°C under oxygen atmosphere and can be used to assess the life time of the antioxidant. It in essence measures Stages A & B discussed above in which Stage B is the inherent property of the polymer without antioxidant which is very short in the case of HDPE.

Typically, the results from a minimum of three test temperatures are plotted in an Arrhenius plot to obtain the activation energy of the process. The activation energy is used to establish the shift factor needed to construct a master curve. However, samples from each pipe were tested only at 85°C. This forced Dr. Hsuan to rely on activation energies obtained from a previous geomembrane study. This methodology is inadequate because:

1. There are many differences between geomembranes and corrugated pipes to make it difficult to justify transforming the results from one to the other.

2. In the geomembrane study, the Arrhenius plot covered a temperature range from 95° to 65°C. The activation energy obtained is not likely to hold at 20°C and thus extrapolation to predict antioxidant depletion at this temperature would be subject to large error.

In actual tests, the pipe samples were incubated in air or in water at 85°C for 30-day intervals and then determining the Oxidation Induction Time (OIT) at 200°C for each sample.

2.1 Calculation of incubation time:

2.2 Oxidation of Geomembrane HDPE without antioxidant in Air:

Using equation 11 on page 25:

$OIT=P*\exp^{(-S*t)}$

For a site temperature of 20°C:

S (the OIT depletion rate) was calculated to be =0.00053 min./day
P, the original OIT without high temperature incubation = 30.895 min. and,
The measured OIT = 0.50 minutes.

Using the above data, Dr. Hsuan estimated the incubation time (t) to reach OIT time of 0.50 minutes to be 21 years for geomembrane HDPE resin, without antioxidant.

Recalculating the same system for a temperature of 26.7°C,
S=0.000747 and, the incubation time (t) = 15 years.

This shows that raising the site temperature from 20° to 26.7°C decreases the oxidation induction time by 28.6%.

2.3 Oxidation of P-2 Pipe in Air vs. Water

From P-2 results (Table 6), OIT dropped from an original 26.39 to 22.50 minutes after incubation at 85°C for 30 days in air oven. Based on an OIT depletion rate in air, S=0.000747 min/day at 26.7°C, this drop in OIT would take 424 days or 1.16 year of incubation at 26.7°C.

For P-2, Table 7 shows a drop in OIT from 26.39 to 14.50 minutes after incubation at 85°C for 30 days in water. This clearly indicates that the antioxidant is depleted much faster in water than air. Unfortunately, it is not possible to extrapolate to the incubation time at 26.7°C in water since the OIT depletion rate in water has not been determined.

Note: Doing the incubation at 85°C is such a high temperature that will cause a change in the morphology of HDPE. This can result in an increase in the amorphous region that is not protected by antioxidant present in the system.

2.4 Conclusions concerning Protocol for Oxidation Resistance of HDPE Pipe

Available results cover very short incubation times. In order to use this methodology to estimate the life time of an antioxidant in HDPE pipe, it will be necessary to:

1. Determine the OIT depletion rate (S), for air and water from measurements extending over several months.

2. Incubate the pipe at high temperature, preferably 50°C, for a long enough time until an OIT close to zero is obtained, i.e., until the antioxidant is close to being depleted and the polymer starts very rapid degradation. This has the advantage of measuring long OIT's and thus minimizing experimental errors.

3. Base the calculations on a realistic site temperature for Florida, namely, about 26.7°C (80°F) instead of 20°C.

Only when these conditions are met will it be possible to extrapolate the results to assess the life time of the pipe under field conditions.

3. Effect of Hydrocarbons in Runoffs:

Petroleum Hydrocarbons in runoff from urbanized auto-intensive land uses is well documented. (Petroleum Hydrocarbon Concentrations Observed in Runoff From Discrete, Urbanized Automotive-Intensive Land Uses. By David L. Shepp, Senior Environmental Engineer, Metropolitan Washington Council of Governments, Washington, D.C.). The data presented point to a relationship between auto exposure and total hydrocarbon concentration. The median concentrations can be as high as 12.4 mg per liter, without accounting for as much as 50% of the lighter fractions which are presumed lost due to evaporation.

The presence of hydrocarbons in the runoff, as small as it might seem, would have a dramatic effect on the failure of HDPE pipe. This is because PE has no affinity for water, as their solubility parameters are significantly different, but has a great affinity for hydrocarbons as their solubility parameters are much closer as shown in the following table:

Material	**Solubility Parameter** (calorie/cubic cm)$^{1/2}$
Polyethylene	7.9
n-octane	7.4
Cyclohexane	8.2
Toluene	8.9
Water	23.4

The close similarity between the solubility parameters (or polarities) of Polyethylene and hydrocarbon fluids, will result in the hydrocarbons being absorbed by polyethylene. It was shown by Isaksen, that the higher the % absorption of the fluid in HDPE, the shorter is the time to failure. [Isaksen R.A., Newman S., Clark R.J., J Appl. Polym. Sci.,Vol. 7, p.515 (1963)] The results in the table below show some of the results they obtained by studying time to failure using static tensile fatigue.

Absorption and Time to Failure for HDPE in various Fluids

Fluid	Absorption,%	Time to failure, Hours
Benzene	74.5	0.77
Xylene	60.4	1.1
Ethanol	8.7	13.2
Water	0.2	55

Note: The effect of hydrocarbons on failure of HDPE pipe has not been taken into account in the studies conducted or the suggested protocols

Conclusions:

1. For 100-year crack-free pipe, a calculated stress at 20°C of 64 psi becomes 50 psi for a site temperature of 26.7°C or 80°F.

2. Back calculating the life time, the 64 psi allowable stress for 20°C, would result in a life time of only 56 years for a site temperature of 26.7°C. (Due to a miss calculation, the calculated stress is 74 psi instead of 64 psi. This would result in a life time at 26.7°C of only 40 years).

3. The minimum time to failure for a 26.7°C site temperature and stress of 270 psi, is more than twice that for a 20°C site temperature and a stress of 250 psi. This translates to a life time at 26.7°C of less than 50 years compared to 100 years calculated for 20°C at approximately the same stress.

4. In the oxidation study for geomembrane HDPE resin the incubation time to reach OIT time of 0.50 minutes is calculated to be 21 years for a temperature of 20°C. This becomes 15 years at a temperature of 26.7°C. Thus, raising the site temperature from 20° to 26.7°C decreases the service life by 28.6%.

5. OIT measurements show that the antioxidant is depleted three times faster in water than in air based on 30 days incubation at 85°C.

6. Short incubation times studied at high temperature, makes extrapolation to site temperature very unreliable.

7. The presence of hydrocarbons in runoffs can considerably affect the life time of HDPE drain pipe but was not considered.

Recommendations

1. It is necessary to establish correlations between pipe performance and the type of resin used as well as the processing conditions employed. This should lead to tight specifications for resin and processing conditions. The major discrepancies between OIT data obtained of 14 commercially new pipe samples are cause for serious concern.

2. The maximum amount of regrind allowed should be determined and specified. Regrind from other polymers should not be allowed.

3. The type and amount of antioxidant used should be specified in the protocol. High molecular weight antioxidants insoluble in water should be considered.

4. Specify the amount of carbon black to be used and its particle size.

5. Shift factors should be determined based on activation energies obtained from HDPE pipe.

6. Extend the range of Arrhenius plots to cover as low a temperature as possible.

7. Pipe failure should be examined in hydrocarbon fluids in order to represent runoffs in urbanized, auto intensive areas.

8. All extrapolations should be based on an average site temperature of at least 25°C to reflect Florida's climate.

9. Determine the OIT depletion rate, for air and water from measurements extending over several months.

10. Incubate the pipe at high temperature, preferably 50°C, for a long enough time until an OIT of close to zero is obtained, i.e. until the antioxidant is close to being depleted and the polymer starts very rapid degradation. This has the advantage of measuring long OIT's and thus minimizing experimental errors and making extrapolation to site temperature more reliable.