THERMAL OXIDATION OF A POLYPROPYLENE GEOTEXTILE USED IN A GEOSYNTHETIC CLAY LINER

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Abstract: Since the strength of geosynthetic clay liners is largely due to the strength of the reinforcing fibers, it is important to understand the long-term behavior of the reinforcement. This paper will address the question of long-term oxidative stability of a polypropylene textile made from fibers used to reinforce a commercial product. Samples were exposed in forced-air ovens at five temperatures for times up to 500 days. The loss of breaking strength and strain were monitored and the temperature dependence of these properties was modelled using 2nd order kinetics. The resulting lifetime prediction suggested that the materials would retain 50% of their strength for 19 years when exposed to air at 15°C under a high air flow rate. However, it was also determined that the amount of fresh air around the samples was a critical factor for oxidation. Results obtained in situations where the amount of fresh air was limited showed a substantial decrease of the oxidation rate. The rate under conditions of 8% oxygen in a static condition, like a buried GCL, was 21 times slower, which means the service life-time in a real application will be around 399 years. Furthermore, the results obtained showed that the materials will still retain over 25% of their original strength after 798 years.

Keywords: Geosynthetic clay liner, thermal degradation, polypropylene, lifetime prediction

INTRODUCTION

Geosynthetic Clay Liners (GCLs) are commonly reinforced with polypropylene (PP) fibers to improve their resistance to shear loads. Since the shear resistance is often used in the design of slopes, the long-term properties of the reinforcement can be the determining factor affecting the service lifetime of the slope. Therefore, it is important to understand the long-term behavior of reinforced GCLs. Two possible failure mechanisms for the reinforcing fibers are creep rupture and oxidation.

Creep rupture is a phenomenon that describes the failure of a material from an applied load that is less than the initial strength of the material. This occurs because the initial strength measured by pulling a material apart depends on the rate at which the material is tested. Faster pulling rates produce higher strength numbers and slower rates produce lower strength values. The long-term, or equilibrium strength can be thought of as the value obtained if one performed a strength test pulling at an infinitesimally slow rate. The value is definitely less than the initial strength, but the only way to determine the actual equilibrium strength is to perform long-term creep tests. Creep rupture occurs when a material is placed under an applied load that is higher than its equilibrium strength, even though it is lower than its initial strength. The material will simply break after a period of time. The time-to-failure becomes longer as the applied load approaches the equilibrium strength. Conversely, the time-to-failure is shortened as the applied load moves towards the initial strength.

Oxidation is a common chemical reaction and how quickly it occurs depends upon the amount of available oxygen and the temperature. It is also highly accelerated from exposure to ultraviolet radiation. Plastics are somewhat susceptible to oxidation so they are protected from this reaction through chemical additives. Additives are available to protect plastics during manufacturing, during long-term service, and from the sun. Plastics commonly become weaker, stiffer, and more brittle as oxidation proceeds. Therefore, a plastic's resistance to oxidation can determine the length of its service lifetime.

Wisse, et al. (1990) investigated both creep rupture and oxidation on a polypropylene geotextile. Their results suggested that at applied loads less than 10% of the initial strength, the oxidation reaction is life-limiting. That means the oxidation reaction is faster than the creep rupture process at low applied loads. The investigators also performed some oven aging tests and made lifetime predictions based on the results. They concluded that generally stabilized polypropylene geotextiles should last from 80 – 120 years at 10°C and that this time could be an order of magnitude larger with special stabilizers. They also calculated energies of activation for the thermo-oxidation reaction to be from 60 to 75 kJ/mol. It should be noted that these predictions applied to underwater applications and their activation energy was determined on materials where the stabilizers were removed by extraction.

A study by Salman, et al. (1998) evaluated the effects of aging at temperatures from 50 to 90°C. There were several important findings reported. First, they showed that for well-stabilized materials, there was an induction time during oven aging where no change in strength was observed. This was seen at temperatures below 80°C but not at 80 and 90°C. This could indicate that the effectiveness of the long-term antioxidants might be lessened at higher temperatures. Because of this induction period, they defined two lifetimes. The first involves additive depletion without a change in strength. The second involves the loss of strength over time after the additives are consumed. They modelled both processes separately and combined the results for an over-all lifetime. They found that the loss in strength after the induction time was best modeled with 1st order kinetics. They also concluded that the overall lifetime could be from 50 to 240 years, including the induction period. Without the induction period they determined a service lifetime at 20°C to be 29 years with an activation energy of 61 kJ/mol.

Recently, Mueller & Jakob (2000) did an aging study where they monitored both strength and the oxidative induction time (OIT) during oven aging of a polypropylene geotextile. Although the absolute value of the OIT does not relate to service lifetime, it has been suggested that following the change in OIT as a function of oven aging is appropriate (Thomas & Ancelet 1993, Thomas et al. 1993). This was demonstrated for a polyethylene geomembrane (Hsuan & Koerner, 1998). The note-worthy findings of the Mueller & Jakob study were that at 80°C there was no induction period related to strength loss, but there was an induction period before there was a change in the OIT value. They also suggested a requirement that a PP geotextile should retain more than 50% of its strength at 80°C for 1 year to be considered a durable product. Finally, they reported that there was only a small difference in oxidation rates of a high density polyethylene (HDPE) geomembrane when it was aged in a gravity convection oven and a forced air oven. This suggests that the air flow rate is not a variable in the oxidation reaction for HDPE.

The purpose of this project was to determine the oxidation rates of a nonwoven polypropylene geotextile used in GCLs when it was exposed to different temperatures in a forced air oven. Additionally, it would be desirable to use the generated data to make a service lifetime prediction based on the oxidation rates. This paper will report the results obtained and how they relate to the findings discussed above.

EXPERIMENTAL

The material tested was a needle-punched, nonwoven polypropylene geotextile made from fibers used in a commercial GCL product. The fabric weight was about 350 g/m². The specimens for oven exposure were approximately 5 cm x 15 cm and were cut with a standard die from across an entire roll-width of material. Over 500 specimens were cut and then were shuffled to try to minimize the effect of thickness variations in the material. Test specimens were exposed in forced-air ovens at temperatures of 100°C, 90°C, 80°C, 70°C and 60°C and were hung under racks with unfolded steel paper clips. The specimens were spread out evenly around the oven and they were not touching each other. A total of 21 different aging experiments were performed over the course of this project. Table 1 summarizes them.

Table 1. 21 Different Aging Experiments

Temperature (°C)	Atmosphere	Conditions
50	100% oxygen	25 atmospheres in a pressure vessel
60	21% oxygen (air)	High flow oven
70	21% oxygen (air)	High flow oven
	100% oxygen	25 atmospheres in a pressure vessel
	100% oxygen	Jar flow = 300cc/min
80	21% oxygen (air)	High flow oven
	100% oxygen	Jar flow = 300cc/min
90	21% oxygen (air)	High flow oven
	100% oxygen	Jar flow = 300cc/min
100	8% oxygen	Jar flow = 300cc/min
	21% oxygen (air)	Low flow oven
		High flow oven
		5 different flow rates
		4 different oven positions

These 21 exposures can be divided into four groups with different objectives. They are:

- Six of the 21% oxygen experiments were done to perform a service lifetime prediction. These experiments were performed in high flow-rate ovens with the specimens spread apart.
- The experiments at 8%, 100%, and 25 atmospheres were designed to study the effect of oxygen concentration on oxidation rate. The 8% oxygen exposures were performed in canning jars with inlet and outlet tubes sealed in the lid. The gas flowed into the bottom of the jar and out the top at a rate of about 300 cc/min. The 100% oxygen tests were performed in sealed canning jars. And, the 25 atm. exposures were performed in a 1L pressure vessel.
- Five different air-flow rates were studied to determine if a minimum flow rate was required. The jars described above were also used for this study.
- Specimens were placed close together, spread apart, in open tubes, and in open jars to determine how the oxidation rate was affected.

The unexposed and exposed test specimens were evaluated by a strip tensile test. The test grips were 2.5 cm x 10 cm, the strain rate was 10 cm/min, and the initial gage length was 7.5 cm. All tests were taken to failure and the maximum load and the strain at the maximum load were recorded. The exposed specimens were tested in sets of five, as were the baseline specimens. Baseline tests were performed each time a group of exposed specimens were tested. A total of fifteen sets of baseline specimens were tested at different times throughout the five-year study.

RESULTS
Strength Tests on Unexposed Samples

Strength tests on unexposed (baseline) specimens were performed each time a set of exposed specimens was tested. This was done to ensure that no significant changes in the test method or instrument occurred. A total of fifteen sets of baseline tests were performed. The breaking strength and strain for these test sets are shown in Table 2.

Table 2. Strength properties of unexposed samples

Set Number	Breaking Strength (daN)	Breaking Strain (%)
1	125 +/- 18	93 +/- 5
2	134 +/- 14	92 +/- 9
3	127 +/- 6	100 +/- 5
4	131 +/- 12	99 +/- 9
5	132 +/- 9	88 +/- 8
6	137 +/- 6	95 +/- 9
7	137 +/- 6	90 +/- 9
8	129 +/- 6	90 +/- 4
9	130 +/- 13	96 +/- 7
10	139 +/- 12	98 +/- 5
11	135 +/- 12	93 +/- 3
12	127 +/- 11	100 +/- 7
13	129 +/- 16	94 +/- 4
14	128 +/- 8	96 +/- 3
15	135 +/- 8	87 +/- 3
Mean	132 +/- 10	94 +/- 6

These results demonstrated that the baseline properties had a coefficient of variation of less than 7.5% throughout the 5 year study. These average values were used to calculate all the retained strengths and strains reported. The individual fabric weights of some exposed and unexposed specimens were obtained in an effort to determine if the precision and accuracy of the strength measurement could be improved. This was done by correlating the specimens' masses to their strengths. The results on five dozen test specimens are shown in Figure 1.
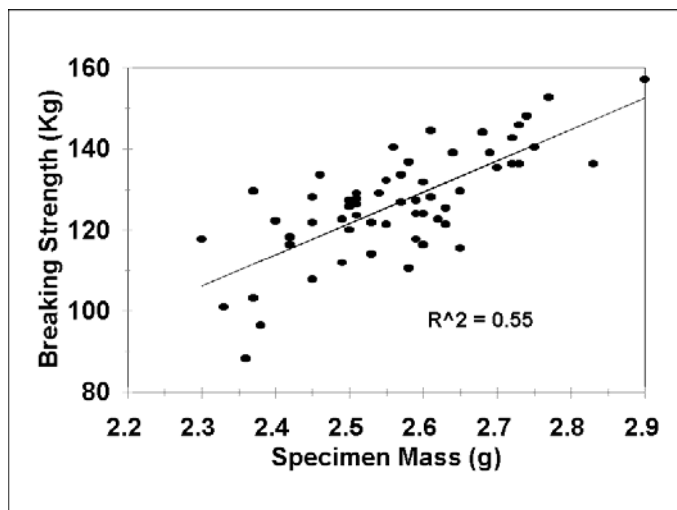

Figure 1. Correlation between specimen breaking strength and mass

This figure clearly shows that the strength of the test specimen is not significantly related to the fabric weight. It is believed that this lack of correlation is important because it is often suggested that normalizing strength data on nonwoven materials is a way to improve the results. In this particular case, there is no improvement. When these results were applied to the strength averages, the resulting coefficients of variability were sometimes higher and sometimes lower. This showed that normalizing based on fabric weight did not improve the quality of the results.

Effects of Forced-Air Oven Exposure

Samples were aged at 100°C, 90°C, 80°C, 70°C, and 60°C for times up to 504 days. The changes in breaking strength and strain were monitored as a function of aging time. The results for each of the exposure temperatures are shown in Tables 3-7. Each value of strength and strain is an average of 5 individual test specimens.

Table 3. Tensile properties over time for samples aged at 100°C in 21% oxygen

Exposure Time (days)	Breaking Strength (Kg)	Relative to Baseline	Breaking Strain (%)	Relative to Baseline
0	132 +/- 10	1.00	94 +/- 6	1.00
26	111 +/- 17	0.84	64 +/- 8	0.68
53	94 +/- 7	0.71	55 +/- 7	0.59
81	100 +/- 7	0.76	57 +/- 2	0.61
118	73 +/- 6	0.55	52 +/- 7	0.52

Table 4. Tensile properties over time for samples aged at 90 °C in 21% oxygen

Exposure Time (days)	Breaking Strength (Kg)	Relative to Baseline	Breaking Strain (%)	Relative to Baseline
0	132 +/- 10	1.00	94 +/- 6	1.00
31	107 +/- 12	0.81	94 +/- 3	1.00
63	106 +/- 10	0.80	67 +/- 3	0.71
90	101 +/- 14	0.77	64 +/- 4	0.68
124	90 +/- 5	0.69	50 +/- 2	0.53
152	84 +/- 6	0.64	54 +/- 3	0.58
181	74 +/- 8	0.59	47 +/- 5	0.51

Table 5. Tensile properties over time for samples aged at 80 °C in 21% oxygen

Exposure Time (days)	Breaking Strength (Kg)	Relative to Baseline	Breaking Strain (%)	Relative to Baseline
0	132 +/- 10	1.00	94 +/- 6	1.00
63	104 +/- 12	0.79	65 +/- 4	0.69
124	94 +/- 8	0.72	50 +/- 9	0.58
181	84 +/- 15	0.66	56 +/- 7	0.61
243	87 +/- 6	0.66	55 +/- 2	0.58
328	70 +/- 15	0.55	50 +/- 5	0.55
424	71 +/- 7	0.54	46 +/- 1	0.49

Table 6. Tensile properties over time for samples aged at 70 °C in 21% oxygen

Exposure Time (days)	Breaking Strength (Kg)	Relative to Baseline	Breaking Strain (%)	Relative to Baseline
0	132 +/- 10	1.00	94 +/- 6	1.00
99	105 +/- 11	0.79	71 +/- 7	0.75
120	94 +/- 7	0.71	56 +/- 2	0.60
152	102 +/- 10	0.77	66 +/- 2	0.70
261	90 +/- 6	0.68	57 +/- 4	0.61
300	87 +/- 7	0.66	59 +/- 3	0.63
401	76 +/- 10	0.58	54 +/- 7	0.57
503	70 +/- 3	0.53	56 +/- 2	0.60

Table 7. Tensile properties over time for samples aged at 60 °C in 21% oxygen

Exposure Time (days)	Breaking Strength (Kg)	Relative to Baseline	Breaking Strain (%)	Relative to Baseline
0	132 +/- 10	1.00	94 +/- 6	1.00
100	102 +/- 8	0.77	63 +/- 3	0.67
202	104 +/- 6	0.79	65 +/- 2	0.69
504	81 +/- 5	0.61	55 +/- 2	0.58

Plots of relative strength and strain versus time were prepared and these are shown for all five temperatures in Figures 2 and 3. After the entire series of plots were prepared, they were collectively examined and some points were omitted to generate smoother curves for the lifetime predictions.
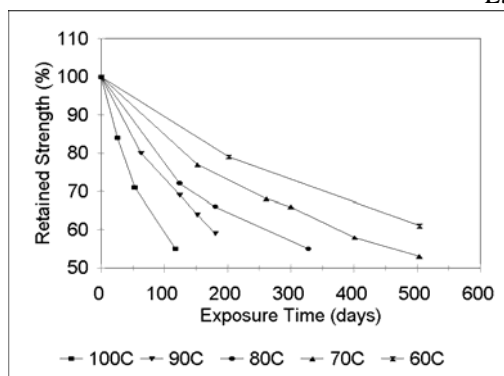
Figure 2. Residual strength over time during oven aging in 21% oxygen

One aspect of these curves is that there was no induction period for any of the temperatures. These were observed in earlier work (Salman, et al. 1998). The significance of this is not yet known. It is possible that different additives behave in different ways. It is known that different additives have different temperature ranges of activity (Fay & King, 1994). It is also possible that the exposure temperatures are higher than the upper temperature limit for a particular additive. If that were true, the additive would appear invisible during the aging study but could still be a very effective long-term stabilizer. This has been seen before. For example, hindered amine light stabilizers (HALS) are not detectable during the OIT test at 200° C but are know to be one of the best classes of stabilizers at ambient temperatures (Thomas & Ancelet 1993).

The strain-at-break was also plotted and these are shown in Figure 3. These lines have more defined curves than the strength plots but they also show less differences between different temperatures. Just by visual appearance alone, a decision was made to use the strength results for a service lifetime prediction.
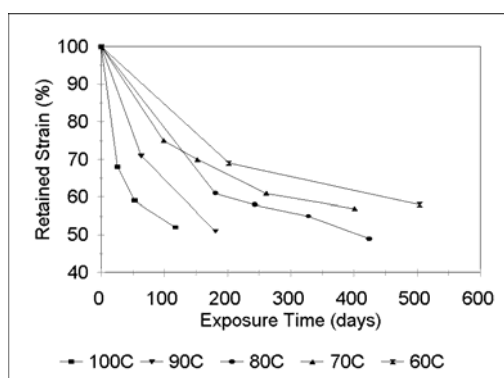

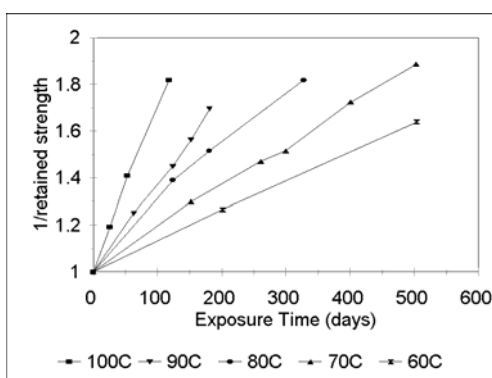**Figure 3.** Residual breaking strain over time during oven aging in 21% oxygen

Figure 4. Second order plot

The first step towards a lifetime prediction is to determine how to model the results. The simplest way is to assume that the chemical reaction involved follows zero, first, or second order kinetics. Each of these processes has a specific way to treat the data (Daniels & Alberty, 1975). Zero order reactions are linear when a simple linear plot is used. First order reactions are linear when the natural logarithm of the strength is plotted versus time. And, second order reactions are linear with a plot of the inverse of strength versus time. It was determined that in this case, the best fit came with a second order plot of 1/Strength vs. Time. The plots of inverse strength versus time are shown in Figure 4 and the calculated line parameters are shown in Table 8.

Table 8. Regression parameters for a second order fit of the strength results

Exposure Temp.	Slope (%/day)	Y-intercept	Corr. Coef. (R^2)
60C	-0.00127	1.00	0.999
70C	-0.00175	1.01	0.998
80C	-0.00248	1.04	0.984
90C	-0.00373	1.00	0.998
100C	-0.00694	1.01	0.997

One can determine the quality of the fit by noting how close the y-intercept and the correlation coefficient is to 1.0. A value of 1.0 would indicate a perfect fit. The results show a good fit to a second order equation. Salman et al. (1998) used 1st order kinetics to determine their lifetime predictions. When both were examined, there was little difference between the two models. A 2nd order model was used because the fit was slightly better than the 1st order model.

The results can also be looked at in terms of the aging requirement suggested by Mueller & Jakob (2000). They proposed a requirement that a geotextile maintain over 50% of its strength when exposed at 80°C for 1 year. The results from the 80°C exposure showed that this material retained over 60% after 365 days. This shows that the evaluated product would be considered acceptable for use in a long-term project.

Arrhenius Plot and Service Lifetime Prediction

The slopes of the lines generated in Figure 4 are the rates of oxidation for this material at each of the four temperatures. The next step in a lifetime prediction is to prepare a plot of the natural logarithm of the rate versus reciprocal temperature, in absolute temperature units (Kelvin). This plot shows the temperature dependence of a chemical reaction, in this case oxidation of polypropylene. If the resulting line is nearly linear, one can assume the fit is appropriate and the reaction mechanism is consistent throughout the temperature range evaluated. The Arrhenius plot for the results from Table 8 is shown in Figure 5.
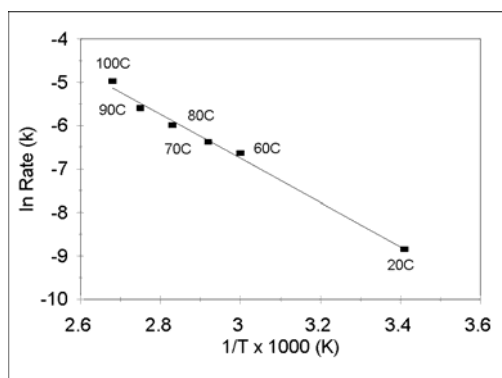

Figure 5. Arrhenius plot for aging in 21% oxygen

The data used to develop this relationship are found in Table 9.

Table 9. Data Used for Arrhenius Plot

Exposure Temp.	1/T x 1000 (K)	Rate (%/day)	ln Rate
60C	3.00	0.00127	-6.67
70C	2.92	0.00175	-6.35
80C	2.83	0.00248	-6.00
90C	2.75	0.00373	-5.59
100C	2.68	0.00694	-4.97

The plot has been extended to reach a temperature of 20°C. This is how one extrapolates higher temperature results to a service temperature. These results are very close to a straight line. It is also seen that the slope is consistent throughout the temperature range studied (60 – 100°C). This indicates that the oxidation mechanism is likely the same throughout this temperature range. An assumption is made that the reaction mechanism is the same from the exposure temperature to the service temperature.

It is now possible to predict the service lifetime of this material at a use temperature of 20°C. From Figure 5, it is known that the natural logarithm of the rate at 20°C is –8.85. Next, the rate is found by taking the antilog of –8.85. This gives a rate of 1.43×10^{-4} /day. The time it takes for the material to lose 50% of its strength is the half-life. The half-life can be determined for a second order reaction by simply taking the inverse of the rate. This is 6993 days or 19 years. Extending the plot further to 10°C gives a service lifetime prediction of 35 years.

One can also predict lifetimes at different strength loses. For example, it takes two half-lives to reach a point where the material retains 25% of its original strength. These results suggest that the time to reach this point would be 38 years at 20°C and 70 years at 10°C. It should be emphasized that this lifetime prediction is based on a high air-flow around the specimens continuously. The actual service environment would be much different. The amount of air would be limited to 8% or less (Yanful, 1993). And, there would be no air flow around the specimen to bring oxygen to the surface and remove the oxidation products. The energy of activation for the oxidation reaction can also be calculated. By definition, the slope of the Arrhenius plot is equal to the energy of activation divided by the gas constant (E_{act}/R). Therefore, multiplying the slope by the gas constant (8.3 J/mol) produces the energy of activation, which is about 42 kJ/mol in this case. This is lower than the values reported by Wisse, et al. (1990) or Salman, et al. (1998). They reported values of 60-70 kJ/mol and 60 kJ/mol, respectively. What this means is the temperature dependence is not as great in the current study as it was in the previous ones. This could be caused by the additive package or the specific type of polypropylene.

Effect of Oxygen Concentration

Another part of this study was to evaluate the effects of oven aging in atmospheres consisting of 8% oxygen, 100% oxygen and 100% oxygen at 25 times atmospheric pressure. This is believed to be the nominal oxygen concentration

found in buried applications (Yanful, 1993). The exposure was done by placing the test specimens in 1L canning jars fitted with both an inlet and outlet tube. A flow meter was placed in line after the jars to adjust the flow rate. A custom-made gas mixture of 8% oxygen in pure nitrogen was continuously passed through the jars at a rate of about 300 cc/min. This means the atmosphere in the jar was replaced every 3 – 4 minutes. The results from the exposure are shown in Figure 6, along with the 100°C/21% results, used for comparison.
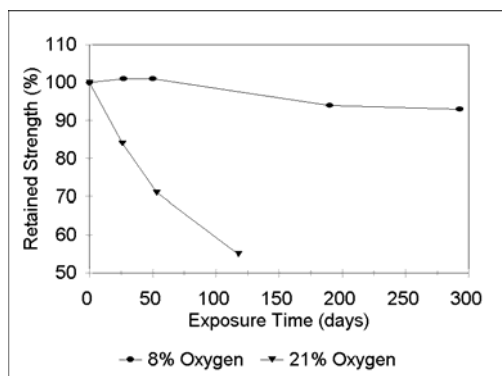
Figure 6. Residual strength over time during oven aging at 100°C in 8% oxygen

This plot shows that the oxidation rate in 8% oxygen is much slower, as expected. If one were to compare the slopes of the two lines in Figure 6, the slope of the 21% line is 21 times greater than the slope of the 8% line.
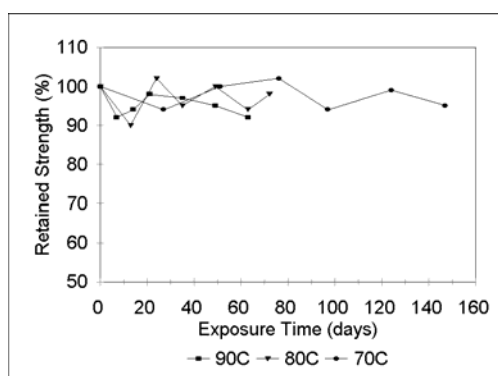

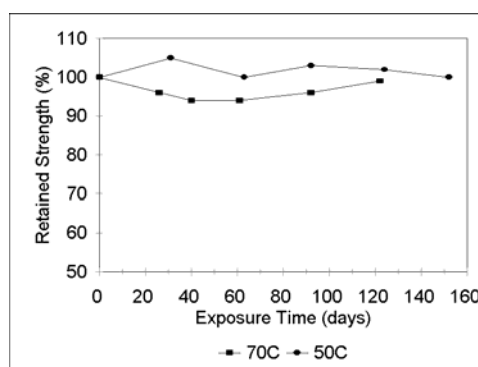**Figure 7.** Exposure with 100% Oxygen at three temperatures

Figure 8. Exposure to 2.48 MPa in a pressure vessel

Exposures were also performed in 100% oxygen in sealed jars and in a pressure vessel under 25 times atmospheric pressure (360 psi, 2.48 MPa). Plots for these conditions are shown in Figures 7 and 8.

Notice that very little oxidation occurred during these exposures. These results are similar to the 8% oxygen ones because very little oxidation occurred during the exposures. They are a little different experimentally. The 8% oxygen was a gas mixture that was pushed through the exposure jars at a rate of about 300 cc/min. The other two experiments were in sealed containers. It is obvious from these results that the oxidation reaction needs more than oxygen to take place. It appears as if there also needs to be a means for fresh oxygen to reach the surface of the specimen and for the oxidation products to be swept away from the specimens. These results suggest that without a high flow of gas around the specimens, the oxidation reaction is self-quenching.

Effect of Fresh Air Flowrate

During the early stages of this study, exposures were performed at 100°C in air for 120 days, resulting in only a small change in strength. It was discovered that the vents designed to draw in fresh air were partially blocked. This reduced the amount of fresh air circulating inside the oven. The vents were cleared and the exposures repeated. The effect of the restricted air flow is seen in Figure 9.
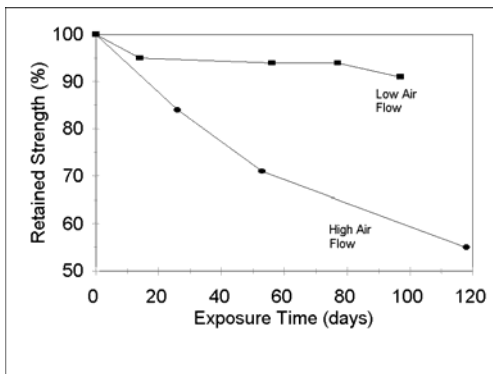
Figure 9. The effect of fresh air flow on the oxidation rate

Once again, it was shown that the amount of fresh air is a variable and that the reaction essentially stopped when there was not enough fresh air available. This is a different result than was reported by Mueller & Jakob (2000), who found that air flow was not significant. However, they evaluated HDPE sheet while this study looked at PP nonwoven geotextiles. The textiles have less oxidative stability to begin with and have much higher surface area for attack by oxygen. The author feels strongly that this phenomenon is real because it has been observed under a variety of different conditions.

This phenomenon was further explored with two additional experiments. First, an air manifold was constructed, five different flow rates were produced, and specimens were aged at 100°C for 104 days under the five different conditions. Secondly, four sets of five specimens were strategically placed in an oven and also aged for 100 days at 100°C. The manifold and oven set-up are shown in Figures 10 and 11.
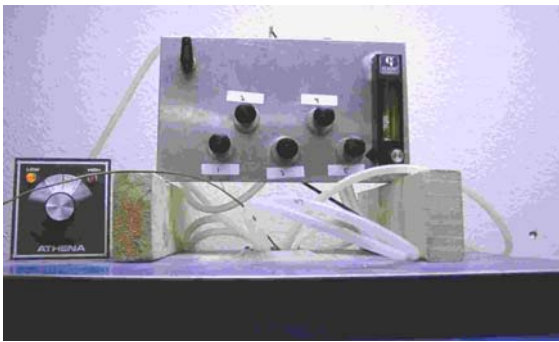

Figure 10. Air Flow Manifold

Figure 11. Different flow rates

The results obtained at flow rates of 52, 152, 292, 880, and 1960 cc/min of air are shown in Figure 12.
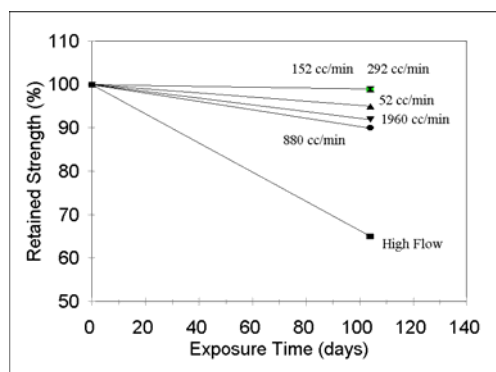

Figure 12. Residual Strength at different air flow rates

The line for 152 cc/min falls directly on top of the 292 cc/min line. Notice that there is no order to the strength losses and that all of the five produced oxidation rates much slower than the high flow oven did.

The second experiment was designed to see how blocking or limiting airflow around individual specimens would effect the oxidation rate. Specimens were set up in an oven in four different ways:

- Spread apart
- Clustered together
- Placed in an open tube
- Placed in an open jar.
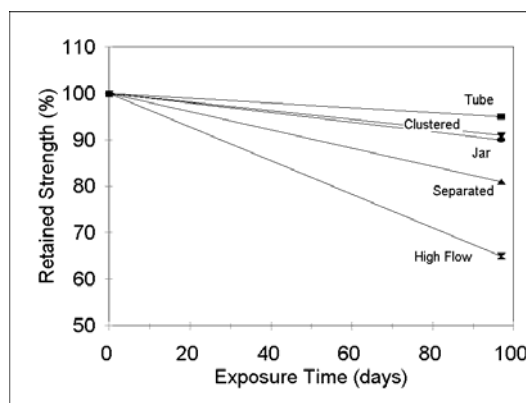
Figure 13. Open Jars and Open Tubes

Figure 14. Effect of different oven positions on oxidation

A photograph of the set-up is shown in Figure 13. There were five specimens of each type and all were exposed to 100°C for 97 days. The results of the strength tests are shown in Figure 14. These results show that the air flow around the test specimens affects the oxidation rate. Notice that even the separated specimens did not oxidize as much as the high flow samples used for comparison. It is possible that the oven was too full to allow adequate airflow around the separated specimens.

CONCLUSIONS

This study has contributed to the understanding of the durability of PP geotextiles in the following ways:

- There was no induction period seen for this system at 70°C, even though this was a well stabilized PP geotextile. This suggests that an induction period may not be seen with every type of additive package.
- The data best fit a 2nd order kinetic model and there was only a small difference seen between zero, first, and second order fits.
- The temperature dependence of the oxidation reaction seen in this study is not as great as those reported before. The specific additives found in the PP geotextile could cause this.
- The amount of fresh air circulated in the oven dramatically affected the oxidation rate.

In terms of service lifetime, it was found that a sample of this material could be expected to last a very, very long time in a buried application with limited oxygen and air-flow. With the use of 2nd Order kinetics and the Arrhenius model, the following prediction can be made from the data obtained.

Table 10. Service Lifetime Predictions

Service Temp.	Condition	Retained Strength	Service Lifetime
20°C	High flow of fresh air	50%	19 years
		25%	38 years
	Stagnant air at 8%	50%	399 years
	oxygen	25%	798 years

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