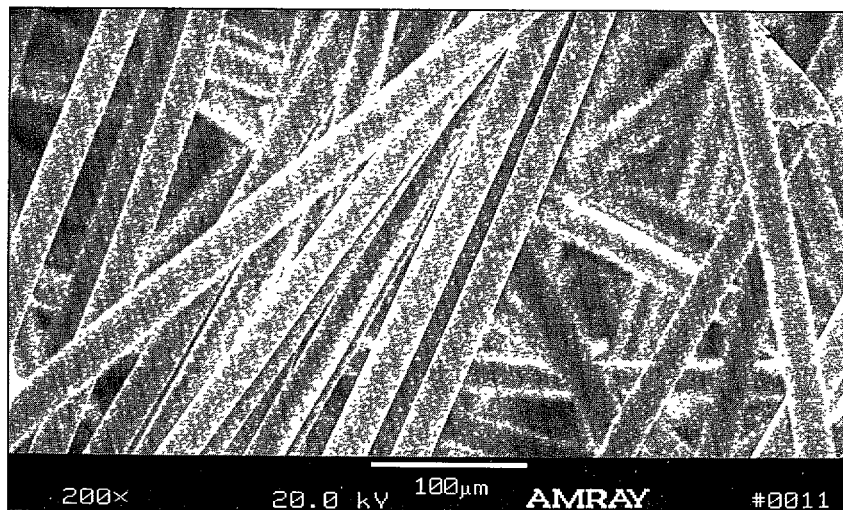


Durability of Geosynthetics for Highway Applications

Interim Report

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U.S. Department of Transportation
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Research and Development
Turner-Fairbank Highway Research Center
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FOREWORD

Geosynthetics as a soil reinforcement material have been used in retaining wall applications since the early 1970's, and more recently in reinforced slope and embankment applications. Because of their cost effectiveness, there is a strong desire to increase the use of geosynthetics in these applications. However, they have not found widespread use in permanent reinforcement applications because of the lack of information and defined test protocols regarding their long-term durability. In 1991, a pooled fund study was initiated through the FHWA to study geosynthetic durability and to develop test protocols to address the long-term durability issue. The scope of the study included both field exhumations of existing installations as well as an intensive laboratory program. This report is interim in nature and provides a background to the study as well as a summary of some of the early test results. This report should be of interest to geotechnical and bridge engineers who are concerned with the durability of geosynthetic reinforced soil structures.



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Research and Development

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16. Abstract <p>This report provides background and summary of the scope of work for the pooled fund study, "Durability of Geosynthetics for Highway Applications." Status and a summary of initial test results for each of the tasks in this study, as well as a summary of the significant accomplishments to date are provided. Finally, discussion of geosynthetic life prediction issues and additional research needs are provided.</p> <p>COVER PHOTO: Nonwoven polyester geosynthetic</p>			
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SI* (MODERN METRIC) CONVERSION FACTORS

APPROXIMATE CONVERSIONS TO SI UNITS

APPROXIMATE CONVERSIONS FROM SI UNITS

Symbol	When You Know	Multiply By	To Find	Symbol	Symbol	When You Know	Multiply By	To Find	Symbol
LENGTH					LENGTH				
in	inches	25.4	millimeters	mm	mm	millimeters	0.039	inches	in
ft	feet	0.305	meters	m	m	meters	3.28	feet	ft
yd	yards	0.914	meters	m	m	meters	1.09	yards	yd
mi	miles	1.61	kilometers	km	km	kilometers	0.621	miles	mi
AREA					AREA				
in ²	square inches	645.2	square millimeters	mm ²	mm ²	square millimeters	0.0016	square inches	in ²
ft ²	square feet	0.093	square meters	m ²	m ²	square meters	10.764	square feet	ft ²
yd ²	square yards	0.836	square meters	m ²	m ²	square meters	1.195	square yards	yd ²
ac	acres	0.405	hectares	ha	ha	hectares	2.47	acres	ac
mi ²	square miles	2.59	square kilometers	km ²	km ²	square kilometers	0.386	square miles	mi ²
VOLUME					VOLUME				
fl oz	fluid ounces	29.57	milliliters	mL	mL	milliliters	0.034	fluid ounces	fl oz
gal	gallons	3.785	liters	L	L	liters	0.264	gallons	gal
ft ³	cubic feet	0.028	cubic meters	m ³	m ³	cubic meters	35.71	cubic feet	ft ³
yd ³	cubic yards	0.765	cubic meters	m ³	m ³	cubic meters	1.307	cubic yards	yd ³
MASS					MASS				
oz	ounces	28.35	grams	g	g	grams	0.035	ounces	oz
lb	pounds	0.454	kilograms	kg	kg	kilograms	2.202	pounds	lb
T	short tons (2000 lb)	0.907	megagrams (or "metric ton")	Mg (or "t")	Mg (or "t")	megagrams (or "metric ton")	1.103	short tons (2000 lb)	T
TEMPERATURE (exact)					TEMPERATURE (exact)				
°F	Fahrenheit temperature	5(F-32)/9 or (F-32)/1.8	Celcius temperature	°C	°C	Celcius temperature	1.8C + 32	Fahrenheit temperature	°F
ILLUMINATION					ILLUMINATION				
fc	foot-candles	10.76	lux	lx	lx	lux	0.0929	foot-candles	fc
fl	foot-Lamberts	3.426	candela/m ²	cd/m ²	cd/m ²	candela/m ²	0.2919	foot-Lamberts	fl
FORCE and PRESSURE or STRESS					FORCE and PRESSURE or STRESS				
lbf	poundforce	4.45	newtons	N	N	newtons	0.225	poundforce	lbf
lbf/in ²	poundforce per square inch	6.89	kilopascals	kPa	kPa	kilopascals	0.145	poundforce per square inch	lbf/in ²

NOTE: Volumes greater than 1000 l shall be shown in m³.

* SI is the symbol for the International System of Units. Appropriate rounding should be made to comply with Section 4 of ASTM E380.

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1. BACKGROUND FOR THE GEOSYNTHETIC DURABILITY RESEARCH PROGRAM

Current codes and guidelines for design of geosynthetic reinforcement [e.g., American Association State Highway Transportation Officials (AASHTO), 1994] require that the long-term allowable strength of the geosynthetic be greater than the load applied to the geosynthetic multiplied by an overall factor of safety. That is, the designer must be reasonably certain that the geosynthetic will have adequate strength to resist the loads applied throughout the life of the structure.

The load applied to the geosynthetic is fairly straight forward to estimate for geosynthetic wall and slope applications as well as for embankment and roadway subgrade reinforcement applications, though typical practice tends to result in conservative estimates. The ability to estimate long-term allowable geosynthetic strength (i.e., its durability) is of direct benefit to the design of these applications, as both sides of the equation would then be known. Currently, the tensile load applied to the geosynthetic cannot be estimated directly for separation, drainage, and erosion control applications. Therefore, the ability to estimate long-term geosynthetic strength would only be indirectly beneficial to the design of these latter applications (i.e., the ability to predict when severe or complete degradation occurs within the desired design lifetime would still be desirable).

All materials degrade with time, including materials used routinely in the construction of retaining walls and other structures such as concrete and steel. For example, steel corrodes, and failures have occurred as the result of the corrosion phenomenon (Weatherby, 1982; Darbin, et. al., 1988). However, much has been learned from these failures as well as the general long-term performance of steel and concrete structures. Due to the available long-term history for concrete and steel structures, designers have the tools and the confidence to use these materials routinely in such structures.

Geosynthetics have been used in reinforcement applications for the past 20 years. Research conducted to date on the durability of geosynthetics includes evaluation of exhumed materials from actual geosynthetic structures as well as evaluation of materials exposed in laboratory environments which are more severe than environments typically encountered in soil. In the case of the field studies, observed long-term strength losses have generally been less than 10 to 15 percent, except in certain extreme soil environments (Allen, 1991). However, the observation period in these studies has typically been 5 to 10 years or less, and extrapolation to typical design lives of 75 years or more, especially considering the many uncontrolled variables, has significant uncertainty. In the case of the laboratory studies in which the variables can be more carefully controlled, severe degradation has been observed in relatively short periods of time. However, the models to relate the severe (i.e., accelerated) laboratory exposure conditions to typical ambient field conditions are not well enough developed to permit accurate extrapolation of the laboratory data. In both cases, extrapolation of degradation data is the key issue.

Perceptions by users concerning geosynthetic durability have caused a significant number of designers and agencies to be reluctant to use geosynthetics for long-term reinforcement applications, to limit reinforcement use to a small number of geosynthetic products which are perceived to be more durable or for which more data is available, or to limit reinforcement use to

noncritical applications (Allen and Diekman, 1994). Furthermore, the lack of geosynthetic durability testing and product evaluation standards does not allow geosynthetic products to be equitably compared regarding long-term durability.

This does not mean that geosynthetics are anticipated to degrade more than materials such as steel and concrete which are at present in routine use. In fact, in some environments, most geosynthetics will likely be significantly more durable than these other materials. The issue is that currently, the available design tools and standards are inadequate to assure the desired long-term design life (typically 75 years or more).

The long-term allowable strength of the geosynthetic is generally determined as follows:

$$T_{allow} = \frac{T_{ult}}{RF} \quad (1)$$

where,

T_{ult} = the ultimate short-term strength of the geosynthetic

RF = a reduction factor which is a function of strength loss due to installation damage, creep, chemical degradation, biological degradation, and joint / connection details.

Installation damage and creep are potentially the largest sources of strength loss; however, current knowledge is adequate to safely design a geosynthetic structure for these two factors. Biological degradation is for the most part a nonissue for the materials used to resist tensile load within the geosynthetic (i.e., Polypropylene "PP," high density polyethylene "HDPE," and polyester "PET"). Current knowledge regarding chemical degradation, and the soil environments which cause chemical degradation, is inadequate for reasonably accurate life prediction and is the main hindrance to routine use of geosynthetic reinforcement. The combined effect of installation damage, creep, and chemical/biological degradation (i.e., the synergism of these factors) on the long-term allowable strength is also not well known, though the current practice of multiplying these factors together (Task Force 27, 1989; AASHTO, 1994) is considered to be conservative.

Routine use of geosynthetics in long-term reinforcement applications such as retaining walls is a desirable objective due to the opportunity for exceptional cost savings relative to the other alternative wall systems currently available. Current experience with geosynthetic walls indicates that on the order of 30 percent or more savings relative to other available wall systems can be realized. Nationwide on Federal and State transportation projects, it has been estimated that savings on the order of \$70 million or more per year could be obtained through routine use of geosynthetic wall systems (Fettig, 1991). Use of geosynthetic reinforced slopes could result in even greater savings. Hence the potential return on investment for the research needed to address long-term geosynthetic strength issues is exceptionally high.

Considering this, a research program to address the issues hindering routine long-term use of geosynthetics in reinforcement applications was developed. The objectives of this research program are three fold:

1. Gain enough understanding of the mechanisms and environments which affect the strength degradation rate of geosynthetic reinforcement in typical in-soil environments to develop an acceptably accurate life prediction model.
2. Develop a practical methodology (i.e., geosynthetic durability testing and evaluation protocols) which can be used to equitably compare geosynthetic products regarding their long-term durability.
3. Develop the detailed research program in a way that considers the expertise nationally of geosynthetics specialists in the geosynthetic industry, academia, transportation agencies, consulting firms, and in related disciplines such as polymer chemistry, materials science, and soil science to ensure nationwide acceptance of the research results.

Due to the size of the research program required and funding limitations, only part of the total research program could be included in the first research contract. However, enough of the total research program was included in the first contract such that the research results obtained would be useable to encourage increased use of geosynthetic reinforced structures. Subsequent research contracts are planned to complete the geosynthetic durability research program.

BACKGROUND FOR FIRST RESEARCH CONTRACT

The first research contract was funded as a Federal Highway Administration (FHWA) HP&R National Pooled fund study. Contributors to the study include the FHWA, 16 State transportation departments, the Forest Service, the National Park Service, the Corps of Engineers, and the geosynthetic industry. At this point, the geosynthetic industry is providing in-kind testing services rather than monetary support. Two of the subcontractors which are part of the selected research team have also contributed in terms of cost sharing. The FHWA is administering the research contract, while the Washington State Department of Transportation is taking the lead among the State transportation departments to provide technical oversight and review for the research.

The scope of the first research contract focuses on the chemical durability, in the laboratory, of the range of geosynthetic products available to gain a better understanding of how the various polymer and environmental factors affect the chemical degradation rate. The chemical durability testing will specifically focus on oxidation of PP and HDPE, and hydrolysis of PET geosynthetics using a sample aging period of approximately 2 years. This understanding would be used to begin to develop life prediction models and to develop well defined, repeatable testing protocols for durability evaluation. A field study of long-term installations of geosynthetics to assess actual degradation rates of geosynthetics in soil at ambient conditions will be conducted to begin to relate the laboratory results to real in-soil behavior. Additionally, a protocol will be developed to evaluate the in-soil creep behavior of various geosynthetics. The in-soil creep test protocol development will provide the necessary groundwork for a future research contract which will evaluate synergism between installation damage, creep, and chemical durability. A summary of the project tasks and workflow is provided in figure 1.

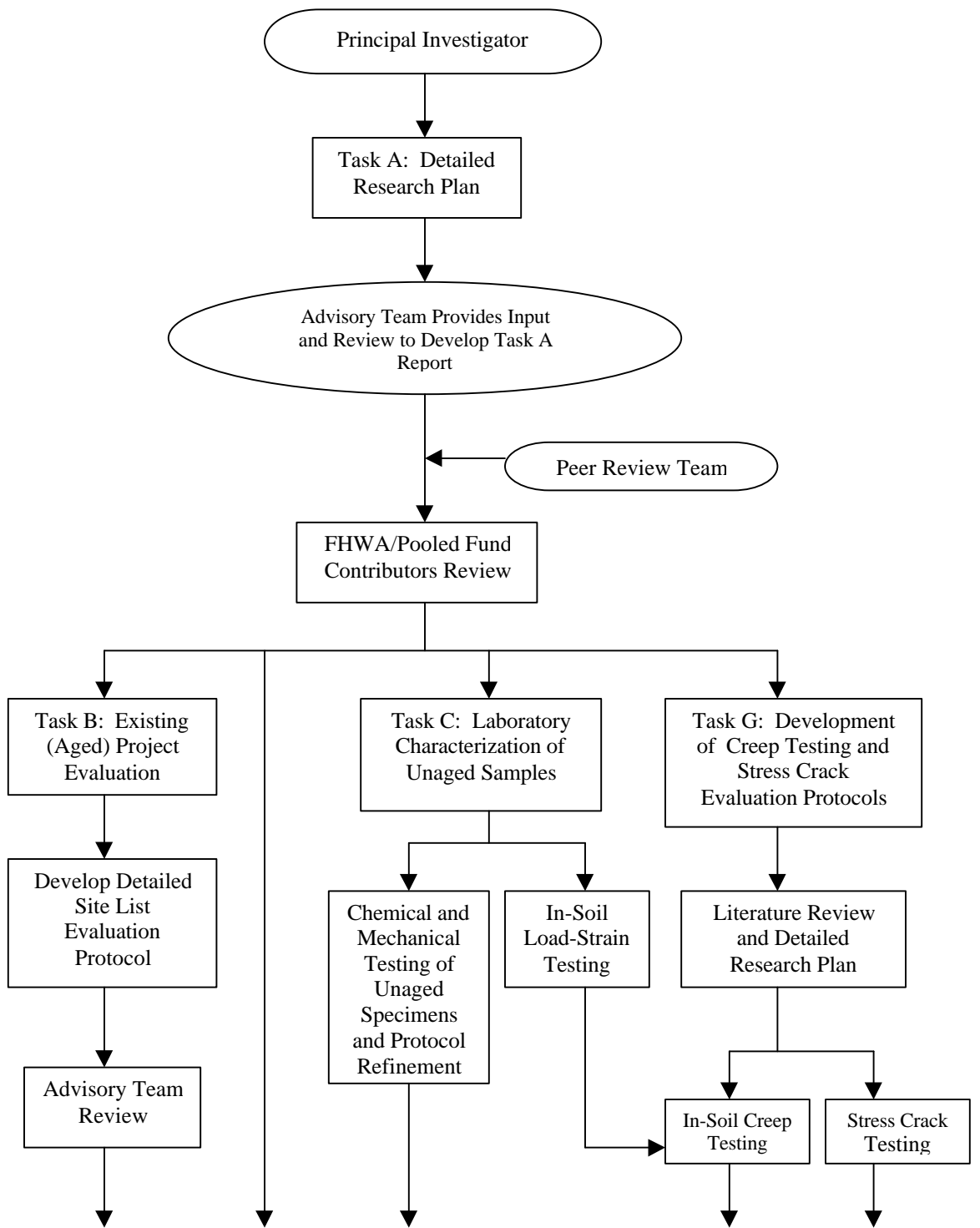


Figure 1. Research project organization and workflow.

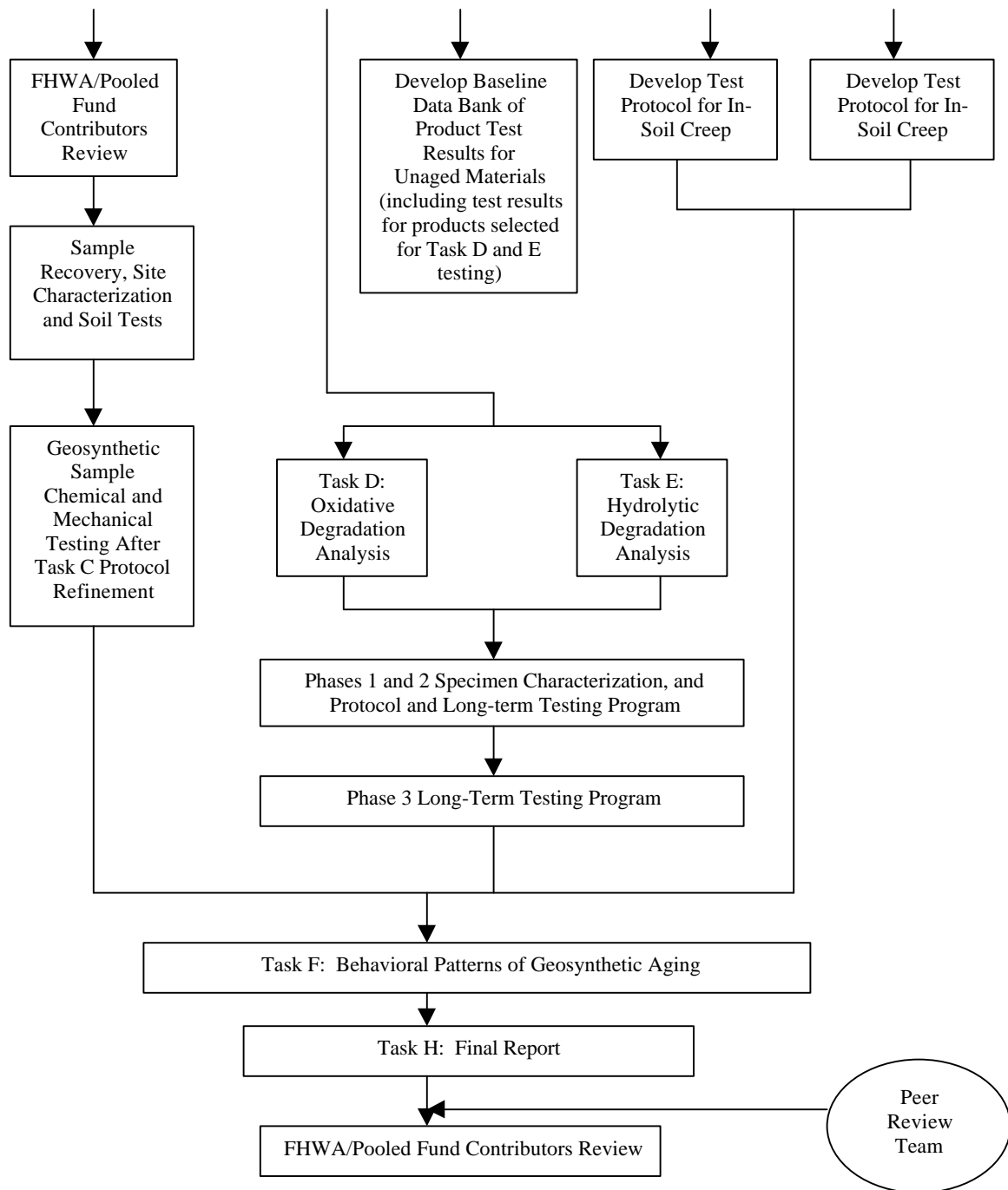


Figure 1. Research project organization and workflow (continued).

The first contract was awarded to the research team on September 23, 1991. The budget for the first research contract was set at \$878,876.00, not including an estimated \$354,514.00 in cost sharing and in-kind testing services provided by some members of the research team. The first contract consists of eight main tasks (Tasks A through H), in which the first task consisted of the development of a more detailed research plan. Once the detailed research plan was developed, an amendment to the initial contract was necessary to implement the detailed research plan. The main changes to the contract resulting from this amendment consisted of increasing the long-term incubation period for oxidation and hydrolysis testing from 2 years to 2.5 years, aging specimens at lower temperatures to accommodate the increased incubation period and to provide an opportunity to extend some specimens to even longer incubation periods, adding two preliminary phases to the oxidation and hydrolysis testing to develop testing protocols and fine tune the long-term testing program, and expanding the in-soil creep testing task to include the development of a stress cracking protocol. This amendment, approved in October 1993, increased the total contract budget to \$1,287,801.00, not including an estimated \$358,517.00 in cost sharing and in-kind testing services from the geosynthetic industry as well as some of the research team members. It is anticipated that as the research project progresses, additional modifications to the contract may be needed. Considering the potential yearly savings nationwide mentioned previously (\$70 million/year), once implemented, the research will pay for itself rapidly. In fact, much of the cost of the research could be recovered from savings produced on one or two large projects.

PROPOSED FUTURE RERESEARCH CONTRACTS

The first research contract does not address all of the objectives envisioned for this research program. The first research contract will provide a geosynthetic life prediction model for the laboratory environment with only an order of magnitude assessment of the relationship between the predicted life in the laboratory environment and the life in typical in-soil environments. This relationship will likely have a great deal of uncertainty, which could result in the estimated geosynthetic life being quite conservative (this will be discussed more fully in a later chapter). This could result in the geosynthetic durability testing and evaluation protocol developed from the first contract being conservative as well. The first research contract will not address the synergism between all of the strength loss mechanisms such as creep installation damage, and chemical durability.

Therefore, the scope of work for future research contracts would need to more fully address the extension of the life prediction model to geosynthetic degradation when buried in soil through laboratory as well as field studies, modifying the durability testing and evaluation protocol as needed. Additionally, these future contracts would need to evaluate chemical degradation rates of geosynthetics under load, both in a virgin state and a roughened state as would result from installation in soil. Finally, depending on the results obtained from the first contract, additional laboratory environments and/or field exhumations to evaluate oxidation and hydrolysis may be needed to address unforeseen questions. The work planned for these future research contracts is currently unfunded.

2. GENERAL BACKGROUND ON CHEMICAL DEGRADATION OF GEOSYNTHETICS

Chemical degradation of geosynthetics is a result of both environmental factors and polymer compositional factors. For a given polymer type, one can expect the greatest amount of chemical degradation to occur, in general, with polymers that have low molecular weights, low percent crystallinity, low density, and low draw ratios (Elias, 1990). Polymer additives also influence the degradation rate. Regarding environmental factors, one can expect the greatest amount of degradation to occur, in general, at relatively high temperatures, in moderate to high moisture conditions, in soils which are active chemically (especially in terms of pH and certain types of ions present), and with the geosynthetic under stress (Elias, 1990). Thickness of the polymer fibers may also have a strong influence on the degradation rate, as degradation mechanisms are dependent on diffusion processes or exposure and removal of surface material (Wrigley, 1987). Key chemical degradation mechanisms in typical soil environments include oxidation, hydrolysis, and environmental stress cracking (ESC).

OXIDATION

The oxidation reaction can either be initiated by ultraviolet radiation or thermal energy. Since the geosynthetic will be buried in most reinforcement applications, thermally activated oxidation is of most interest in the present study. Of the polymers used in geosynthetics, relatively speaking, PP is potentially the most susceptible to oxidation, followed by HDPE and PET which have a relatively low susceptibility. Though in actuality a rather complex reaction, conceptually, polyolefin (i.e., PP and HDPE) oxidation is the reaction of free radicals within the polymer with oxygen, resulting in breakdown and/or crosslinking of the molecular chains and embrittlement of the polymer.

Antioxidants are typically added to the polymer (sometimes multiple types are added to increase effectiveness) to prevent oxidation during processing and use. Broad classes of antioxidants often used in geosynthetics include phenolic stabilizers and hindered amine light stabilizers (HATS). As the antioxidants are used up, resistance of the polymer to oxidation will decrease. The rate of polymer oxidation is dependent on how much and what type of antioxidant is present initially, at what rate it is used up, how well it is distributed within the polymer, and how fast it can be leached out by the flow of fluids, such as water, into and around the polymer (Van Zanten, 1986). Environmental factors which affect the rate of oxidation include temperature, oxygen concentration which in soil can vary from 21 percent in gravels at shallow depth to on the order of 1 percent in fine-grained soils at deeper depths (Yanful, 1993; Yanful, et. al., 1993), and the presence of transition metal ions such as iron (most common) or copper which act as a catalyst and accelerate the oxidation reaction. Thermal oxidation at typical in-soil temperatures appears to be quite slow.

HYDROLYSIS

Of the polymers typically used for geosynthetics, only PET is potentially susceptible to hydrolysis. Hydrolysis occurs when water molecules react with the polymer molecules, resulting in chain scission, reduced molecular weight, and strength loss. Hydrolysis is simply the very slow inverse reaction of the synthesis of PET when water is present. The specifics of the reaction vary

depending on the pH of the liquid. This results in high pH (alkaline) hydrolysis being relatively rapid, whereas neutral or low pH conditions can result in a slow hydrolysis rate. The rate of hydrolysis is also highly temperature dependent and can become relatively rapid at high temperatures in the vicinity of the glass transition temperature or above for the polymer, which is on the order of 70 °C to 80 °C (158 °F to 176 °F). The polymer does not need to be submerged for hydrolysis to occur, as hydrolysis can occur in moderate to high humidity conditions, though the reaction rate becomes slower as the humidity decreases (McMahon, et. al., 1959).

Hydrolysis appears to be the result of both a surface erosional phenomenon as well as a diffusional process of water to the polymer fiber core. These two phenomena have given rise to the terms “outer” and “inner” hydrolysis. Outer, or surficial, hydrolysis is dominant in high pH conditions and is characterized by loss in fiber cross-sectional area with minimal reduction in the molecular weight of the polymer which remains (Anderson, et. al., 1992). Inner, or diffusional controlled hydrolysis is dominant in neutral and acidic conditions and is characterized by significant losses in molecular weight of the polymer with minimal surficial erosion or damage (Anderson, et. al., 1992).

ENVIRONMENTAL STRESS CRACKING (ESC)

Rupture of a polymer when under stress is either ductile or brittle in nature. The ductile failure mode occurs when stresses are high enough to cause tie molecules to stretch out, lamellae to separate and start unfolding, resulting in fracture of the spherulites and plastic flow of the molecular structure (Lustiger, 1983). When failure occurs in a brittle manner, stress levels are usually lower, allowing sufficient time for tie molecules to slowly disentangle themselves from adjacent spherulites, initiating crack formation followed by slow crack growth (Bright, 1993).

ESC is the result of an accelerated crack initiation and growth process occurring when a polymer is subjected concurrently to a particular chemical environment and long-term stress. This accelerated crack initiation and growth process can result in premature brittle failure. ESC results in molecular chain disentanglement rather than chain breakage or chemical change.

Evaluation of ESC has been focused on polyethylene due to its use in various critical applications (e.g., telephone transmission cables, natural gas pipe) and the potential sensitivity of some polyethylenes to this phenomenon. The literature indicates that other polymers used for geosynthetics may also experience some sensitivity to this phenomenon (Bright, 1993).

The results of previous studies show that polyethylene resistance to ESC can be improved by increasing its average molecular weight, decreasing its molecular weight distribution, increasing its crystalline content, reducing the crystallite and/or spherulite size, increasing the degree of orientation, and using copolymerization (Wrigley, 1987). Therefore, the potential for ESC in a given polymer can be controlled.

ESC is closely related to the more general phenomenon of stress cracking. The difference between the two is that the chemical present for ESC accelerates the chain disentanglement process, whereas in stress cracking no accelerating chemical is present. Chemicals identified in the literature which can accelerate the stress cracking process include water, weak acids and

bases, alcohols, metallic soaps, and solvents (Wrigley, 1987). Water, and to some extent weak acids and bases, are “chemicals” which need to be considered for ESC in typical in-soil environments.

3. DESCRIPTION AND STATUS OF PROJECT TASKS (CURRENT RESEARCH CONTRACT)

The tasks for the current research contract and their status are summarized as follows:

TASK A - DETAILED RESEARCH WORK PLAN

The focus of this task is to develop a detailed research work plan for the project. The planning involved experts from all of the disciplines encompassed by the subject of study and an extensive literature review to take advantage of all available information and experience in developing the plan. The plan was reviewed by experts internationally as well as by the contributors to the project. The Task A report was completed in November 1992.

TASK B -EXISTING(AGED)PROJECT EVALUATION

This task includes the selection, characterization, and sampling of approximately 20 geosynthetic sites to determine the amount of long-term geosynthetic strength loss occurring in actual geosynthetic installations. The objective of this task is to obtain, as much as possible, aged geosynthetic samples from sites in which the baseline is known, the geosynthetic is under load, and which are as old as possible (i.e., on the order of 5 to 10 years or more). It is unlikely, however, that the baseline can be determined with the needed degree of certainty for all 20 sites. Therefore, the data obtained in this task will also be used to establish an accurate baseline for exhumations performed at those sites in future geosynthetic durability research contracts.

Data to be obtained will include soil chemistry, gradation, strength, temperature, moisture, and structure performance to define the total environment, and geosynthetic laboratory test data such as tensile strength, creep behavior, visual appearance (micro level damage using Scanning Electron Microscopy (SEM) as well as damage at the macro level such as holes, cuts, general abrasion, etc.), and any long-term chemical changes which have occurred through tests such as Differential Scanning Calorimetry (DSC), Oxidation Induction Time (OIT), melt flow index, High Performance Liquid Chromatography (HPLC), molecular weight determination, carboxyl end group (CEG) determination, and Thermogravimetric Analysis (TGA). Specific protocols are to be developed, including exhumation, sample preparation, and testing procedures as needed, as a cooperative effort between the geosynthetic industry and the research team. Since the chemical and mechanical (e.g., strength) tests will be performed by the geosynthetic industry and will involve multiple laboratories, QA/QC procedures for this testing are to be developed to ensure that interlaboratory bias is reduced to a minimum or eliminated. Some duplicate interlaboratory testing may be required to spot check any observed differences between laboratories.

A final plan which identifies the sites selected for exhumation and the test protocols and program for evaluating the exhumed samples was completed in November 1993. The sites selected and the sampling procedures have been approved. The chemical testing protocols and QA/QC procedures to reduce or eliminate any interlaboratory bias have also been developed. Contributors to the study as well as a variety of other agencies, companies and individuals were contacted to identify candidate sites. A total of 60 sites or groups of sites were identified, from which 28 sites or groups of sites were selected for detailed evaluation in this study. The final sites were selected to

ensure that a variety of soil environments and geosynthetic products are evaluated. Preference was given to those sites in which at least some baseline data is available. A list of the selected sites and their characteristics is provided in table 1. To date, 11 of the 28 “sites” have been exhumed. No geosynthetic testing has been conducted thus far, with the exception of two sites in which only the tensile strength testing has been performed. Additional testing will not be conducted until the Task C testing, which will be used to fine tune the test protocols developed as part of this task, is completed. Exhumed samples are being stored in a dark, climate controlled room until the testing can be performed.

TASK C - LABORATORY CHARACTERIZATION OF UNAGED SAMPLES

This task includes the selection and detailed characterization of 14 unaged geosynthetic products in addition to the products to be tested in Tasks D and E to provide a baseline data bank for geosynthetic products typically used in geosynthetic reinforcement applications. In addition, the effects of in-soil confinement on the short-term load-elongation response of various geosynthetics will be investigated.

It is planned to perform the tests provided in table 2 for each of the 14 geosynthetic products. In addition, limited creep testing may be performed for products in which long-term creep test data is not available. This creep data will be used as baseline data for Task G.

The test protocols developed for Task B mentioned previously will also be used for Task C. The Task C testing on unaged materials will be conducted before the testing on aged materials for Task B to allow refinement of the protocols, as it may not be possible to retest the aged materials obtained in Task B due to material quantity limitations should the test protocols need to be refined. The Task C testing has not begun as yet.

The confined geosynthetic load-strain behavior study will consist of a literature review of past attempts to determine this property before developing a detailed testing program and selecting a testing apparatus. The detailed testing program has been developed. It is anticipated that both a triaxial type confined load-extension device as well as a pullout box type device modified to allow loading the specimen at both ends will be used. Test variables such as specimen size, clamping system, soil placement procedures, extension testing displacement rate, and other test calibration details will be evaluated before moving on to other variables. Once this is completed, other variables such as soil type, confining pressure, and geosynthetic type will be investigated. This work will be closely coordinated with the confined creep testing protocol development to be conducted in Task G. The actual in-soil testing proposed for Task C, other than some preliminary protocol development work, has not yet begun.

TASK D- OXIDATIVE DEGRADATION ANALYSIS

This task focuses on the thermally activated oxidative degradation of PP and HDPE geosynthetics. This task consists of a laboratory study to develop test protocols and to evaluate the effect key environmental factors in typical soil environments have on the rate of geosynthetic oxidative degradation.

Table 1. Selected sites for retrievals of geosynthetics.

Site Number	Site Owner	Site Identification	Geosynthetic Type	Comments
1	New York DOT	Thruway Bridge Embankment Failure	PP Slit Film Woven	Sample recovered by NYDOT
2	corps of Engineers, New Orleans District	Test Embankment Bonnet Carre Spillway	PET Multifilament Woven (2 weights)	Sample recovered by COE in Feb., 1993 (approx. 3 years in ground). Have archive data. Site may be available for additional retrievals.
3	Washington DOT	Lab Incubation Samples at 100 percent RH in-soil Taken from Rainier Ave. Wall Site. Includes Installation damaged samples.	PP Slit Film Woven and PET Multifilament Woven	Sample available on demand. Incubations began in June 1989 and are on-going. Short-term virgin and damaged strength data available. Archive samples also available.
4	Colorado DOT	Colorado Test Walls I-70 Glenwood springs	PP Needlepunched Nonwoven, PP Heatbonded Nonwoven, and PET Needlepunched Nonwoven	Geotextile in ground since 1982. Sites excavated in May 1993. Short-term virgin and damaged strength data available.
5	U. S . Forest Service	Shelton Wall, Olympic Peninsula	PP and PET needlepunched Nonwovens	Geotextile in ground since 1975. Samples obtained in May 1993. Archive data available. Site available for additional retrievals.

Table 1. Selected sites for retrievals of geosynthetics (continued).

Site Number	Site Owner	Site Identification	Geosynthetic Type	Comments
6	Pima County, Tucson, AZ	Tanque Verde Walls, Tucson, AZ	HDPE Geogrid	Samples retrieved August 1993. Archive data available. High temperature site. Site available for additional retrievals.
7	OKDOT	State Road 131, OK	PP Needlepunched Nonwoven	Geotextile in ground since 1984. Samples retrieved in June 1993. Site available for additional, retrievals.
8A	Washington DOT	Pacific Way, Cowlitz County	PET Needlepunched Nonwoven	Separator site. In ground since 1982. Exhumed in 1992. Strength test data and samples available.
8B	Washington DOT	Coal Creek Road, Cowlitz County	PP Heatbonded Nonwoven	Separator site. In ground since 1984. Exhumed in 1992. Strength test data and samples available.
8C	Washington DOT	SR-9 Marsh Road	PP Slit Film Woven	Separator site. In ground since 1989. Exhumed in 1992. Strength test data and samples available.
9	AKZO	AKZO Plant Test Wall, Arnhem, The Netherlands	PET Multifilament Woven	Geotextile in ground since 1980. Samples retrieved in fall, 1993. Archive data available.
10 A-D	U.S. Forest Service	Quinalt Forest Subgrade Stabilization Test Site, Olympic Peninsula	Three PP Needlepunched Nonwovens, one PP Heatbonded Nonwoven	Geotextile in ground since 1976. Retrievals made in 1978 with data available. New samples obtained in May 1993.

Table 1. Selected sites for retrievals of geosynthetics (continued).

Site Number	Site Owner	Site Identification	Geosynthetic Type	Comments
11A&B	Remay	Reemay Texas A and Texas B, Harris County, Texas	PP Heatbonded Nonwoven	Site previously excavated and reported by Geosyntec Consult. in 1987. Baseline data published in STP-18.
12	Reemay (DuPont)	Smyrna, Delaware Stabilization Test section	PP Heatbonded Nonwoven	Archive data available.
13A	Georgia DOT	Torres Causeway Brunswick		Archive data available.
13B	Georgia DOT	Dike Area 14B Savannah		Archive data available.
14	Kansas DOT	I-35 and U.S. 119 Slope Repair Project	PP Geogrid	
15A	Montana DOT	Swan Lake	Geotextiles	Geotextile in ground since 1976.
15B	Montana DOT	Helmville	Geotextiles	Geotextile in ground since 1974.
16	Corps of Engineers, Norfolk District	Craney Island Disposal Site	PET and PP Multifilament Woven Geotextiles	In dredge disposal area.
17	corps of Engineers, Philadelphia District	Wilmington Harbor Disposal District	PET Multifilament Woven Geotextile	Geotextile in ground since 1989. Samples Retrieved in 1993.
18	South Carolina DOT	Greenpond Rd. Watersboro, SC	PP Needlepunched Nonwoven	Geotextile in ground since 1978.

Table 1. Selected sites for retrievals of geosynthetics (continued).

Site Number	Site Owner	Site Identification	Geosynthetic Type	Comments
19	Oregon DOT	U.S. 26 Wall at Abandoned Detour Road at Elderberry Inn	PP Needlepunched Nonwoven	Geotextile in ground since 1983
20	Washington DOT	SR-109 Aberdeen/Hoquiam	PP Slit Film Woven	Reinforced embankment in gravel and wood chip environment. In ground since 1987.
21	NCHRP and Geosynthetic Research Institute	NCI-IRP Project 15-13 Edge Drain Study, Sites 63, 90, 30, 29, 83, 82	PP and PET Geotextiles	Samples available.
22	Maryland DOT	Route 50 - Cape St. Clair Interchange A		
23	Louisiana DOT	Test Embankments	Geotextile	
24	Columbia Heights (city)	Jackson Pond	PET Coated Geogrid	
25	North Carolina DOT	Friar Swamp Embankment	PP Multifilament Woven	Geotextile in ground since 1987.
26	Mississippi DOT	U.S. 49, Sunflower Co.	Geotextile	Stabilization application.
27	California DOT	Dumbarton Bridge Fill		
28A	New Mexico DOT	Tierra Amadella Slide	Geogrid	
28B	New Mexico DOT	Raton I/25 Slide	Geotextile	

This task consists of three phases. The first phase develops or fine tunes some of the test protocols to be used for the laboratory testing to be conducted in subsequent phases. Additionally during this phase, equipment will be purchased and calibrated as necessary, and materials and samples will be prepared. Studies will be conducted on both reference polyolefins (HDPE pellets and isotactic PP powder) and some commercial geosynthetic products. Specific preliminary studies are to include:

- Thermal response of reference polymers (TGA and DSC).
- Selection and fine tuning of test methods to chemically track oxidative degradation as it progresses.
- Evaluation of the shrinkage response of PP geotextiles at the highest temperatures anticipated for the testing to determine whether or not specimen framing to prevent shrinkage will be required.
- Evaluation of techniques to remove antioxidants so that the oxidation response of the base polymer can be studied.
- Calibration of OIT and HPLC using reference polymers with premeasured antioxidant concentrations (i.e., model systems).
- Water extraction studies to provide a preliminary evaluation of antioxidant leachability.
- Evaluation of the oxidation response of a model polymer system (PP with known antioxidant concentration) to exposure to various concentrations of a metal salt solution (specifically iron) as an aid to future selection of metal salt concentrations for later long-term studies.
- Mechanical tests conducted to determine the statistically required number of specimens to account for product variability in the Phase 3 testing as well as to provide a baseline.

The second phase provides limited preliminary laboratory tests conducted on commercial geosynthetic samples to provide a baseline for the Phase 3 testing. This baseline data includes chemical and mechanical properties of products to be tested in Phase 3, and preliminary short-term oven aging tests in extreme conditions to fine tune the long-term oven aging protocol and test program in Phase 3. Table 2 provides a list of the tests anticipated to be conducted (with the exception of the tests only applicable to PET) in this phase.

The third phase consists of a long-term (i.e., up to 2.5 years in length as currently planned) systematic experimental program to evaluate the effect various environmental variables have on the oxidative degradation rate of PP and HDPE geosynthetic products in an oven environment. Elevated temperature will be the primary environmental variable used to accelerate property changes such that measurable strength losses occur within the desired time frame. The researchers will attempt to model this data as a function of temperature (i.e., the Arrhenius model combined with a rate expression, discussed later in this report) to permit extrapolation to typical in situ temperatures.

Table 2. Tests for evaluation of geosynthetic products in task C.

Test	Geosynthetic Polymer	Purpose of Test
Mass per Unit Area	All Polymers	Overall physical characterization
Thickness	All Polymers	Overall physical characterization
Density/Specific Gravity	All Polymers	Overall physical characterization
Wide Width Tensile Strength	All Polymers	Overall physical characterization. The load-strain behavior of the geosynthetic is determined.
Grab Tensile Strength	All Polymers	Overall physical characterization. This is an index test used to evaluate strength, mainly to assess the survivability of the material regarding installation conditions.
Thermogravimetric Analysis (TGA)	All Polymers	TGA permits continuous weighing of a specimen as the specimen temperature is increased. The increasing temperature causes volatile components of the polymer to leave the polymer, decreasing the polymer mass. This test will provide information regarding decomposition temperatures of the various polymer components and can help to identify these components, their quantities, as well as the amount of carbon black and ash present.
Differential Scanning Calorimetry (DSC)	All Polymers	DSC permits continuous measurement of the power required to maintain a temperature balance between the specimen and a reference cell as the temperature in both cells is increased. In this way heat flow into and out of the specimen can be measured. Any reaction or phase change in the polymer which involves the releasing or absorbing of heat can be identified. Polymer characteristics such as the glass transition, decomposition, and melting temperatures, as well as the percent crystallinity can be determined using this method.

Table 2. Tests for evaluation of geosynthetic products in task C (continued).

Test	Geosynthetic Polymer	Purpose of Test
Oxidation Induction Time (OIT)	PP and HDPE	This test utilizes the DSC apparatus to measure the oxidative stability of the polymer. The temperature of the specimen is continuously increased in an inert (nitrogen) atmosphere until the OIT test temperature of 175 °C (347 °F) for PP or 200 °C (392 °F) for HDPE is reached and the specimen is in a molten state, at which point oxygen is introduced and the time to the exothermic oxidation reaction peak is obtained. This peak approximately represents the point at which the antioxidants in the polymer are used up and rapid oxidative degradation of the main polymer begins. The OIT test measures the response of the entire polymer system with various additives (e.g., antioxidants, carbon black, etc.) in the molten state to oxygen and can give an indication of the resistance (i.e., antioxidants remaining) of the polymer to oxidation.
High Performance Liquid Chromatography (HPLC)	PP and HDPE	HPLC permits identification and quantification of antioxidants in polyolefins. In general, the antioxidant type, especially if more than one additive is present, must be known in advance to accurately identify and quantify the antioxidants present. This method is especially useful to determine the degree of antioxidant consumption which has occurred due to oxidative degradation.
Melt flow Index	PP and HDPE	The melt flow test is used to measure the flow rate of a molten polymer at a specified temperature and pressure. The melt flow index can be qualitatively related to the molecular weight of the polymer.

Table 2. Tests for evaluation of geosynthetic products in task C (continued).

Test	Geosynthetic Polymer	Purpose of Test
Gel Permeation Chromatography (GPC) or Intrinsic Viscosity	PET	GPC essentially allows the molecules within a polymer, which are placed in solution, to be separated according to size in a way analogous to soil gradation using sieves. From this test, the molecular weight distribution can be determined directly. The average molecular weight can also be determined by measuring the intrinsic viscosity of the polymer in solution with a given solvent at a given temperature and using a correlation to relate viscosity to molecular weight. This property is a key indicator of resistance to hydrolysis and appears useful to track hydrolytic degradation as it progresses.
Carboxyl End Group (CEG) Determination	PET	This test measures the concentration of carboxyl end groups. This property may be a key indicator of resistance to hydrolysis.
Oven Aging Classification Test	PP and HDPE	This test will be similar to the single high temperature oven aging tests conducted in Phase 2 of Task D in which strength loss as a function of time during oxidative degradation of specimens placed in a forced air oven is determined. This test measures the response of the entire geosynthetic specimen in the solid state to oxidative degradation. The test is an index test and may not be directly relatable to long-term product life. However, the usefulness of this test for this purpose will be evaluated in Task D.

Phase 3 will evaluate four or five polyolefin geosynthetics such that a range of chemical properties, fiber properties and overall geosynthetic constructions are evaluated. Two of these products will, in addition to the commercial version, be specially produced without end-use antioxidants to allow assessment of the oxidative resistance of the base polymer so that the effect of the antioxidants on oxidative resistance can be determined. The key environmental variables and test acceleration techniques evaluated in this task would be used to allow the protocol developed to be related, at least to some degree, to typical in situ conditions. Environments to be evaluated include four temperatures, two oxygen concentrations, a dry and high humidity condition, and two metal salt concentrations. Forced air ovens will be used to expose the geosynthetic specimens to these conditions. Specimens will be allowed to degrade until a strength loss of 50 percent is obtained, if possible. Typically, five sample retrievals performed during the duration of the test exposure time, with five specimens per retrieval, are planned for each environmental condition and geosynthetic product tested. The test program may be adjusted as early results become available. Some additional samples for additional retrievals will be included at the lowest test temperature should longer incubation times than originally planned be required.

It is not the intent of this study to evaluate every possible combination of polymer chemical makeup currently available. Instead, the study is to develop a test protocol which can be used by various geosynthetic manufacturers and geosynthetic users to assess the potential long-term performance of any given geosynthetic product.

Wide width tensile strength will be used to characterize the strength of the geosynthetics as oven aging progresses. Specimen weight loss will also be measured. Scanning electron microscopy (SEM) will be conducted occasionally to evaluate the physical nature of the oxidative damage as it becomes more severe. HPLC and OIT tests will be conducted to attempt to track the consumption of antioxidants as degradation progresses.

To date, Phases 1 and 2 of this task have been completed, and the specimens to be evaluated in Phase 3 have been placed in the ovens and have been incubating since early January, 1994. The test program has been adjusted based on the results of Phases 1 and 2 (provided in the next chapter), with additional minor adjustments based on the early results from the Phase 3 testing.

TASK E - HYDROLYTIC DEGRADATION ANALYSIS

This task focuses on the hydrolytic degradation of PET geosynthetics. This task is strictly a laboratory study to develop test protocols and to systematically evaluate the effect key environmental factors of concern in typical soil environments have on the rate of geosynthetic hydrolytic degradation.

This task consists of three phases. The first phase provides the groundwork for the fine tuning of the experimental plan to be implemented in subsequent phases. During this phase, detailed test protocols are to be developed or fine tuned, equipment will be purchased and calibrated as necessary, and materials and samples will be prepared. Studies will be conducted on both a reference polymer (e.g., PET flakes) and some commercial geosynthetic products, both in the form of fibers as well as complete geosynthetic products. Specific preliminary studies are to include:

- Thermal response of reference polymers (TGA and DSC).
- Selection and fine tuning of the best test methods to chemically track hydrolytic degradation (specifically molecular weight loss and surficial erosion) as it progresses.
- Evaluation of the shrinkage response of PET geosynthetics at the highest test temperatures to determine whether or not specimen framing to prevent shrinkage will be required.
- Characterization of the PVC coating on coated geogrids, the nature of its degradation, and evaluation of experimental techniques to remove the PVC from the geogrid.
- Initial studies using PET fibers to select environments appropriate for Phase 2 and 3 experiments.
- Preliminary development of long-term immersion test equipment and procedures.
- Mechanical tests conducted to determine the statistically required number of specimens to account for product variability in the Phase 3 testing as well as to provide a baseline.

The second phase provides limited preliminary experiments conducted on commercial geosynthetic samples to provide a baseline for the Phase 3 testing. This baseline data includes chemical and mechanical properties of products to be tested in Phase 3, and preliminary short-term immersion testing in “worst case” conditions to fine tune the long-term hydrolytic degradation protocol and test program in Phase 3. Table 2 provides a list of the tests anticipated to be conducted (with the exception of the tests only applicable to PP and HDPE) in this phase. A specific plan to evaluate the effect the PVC coating has on hydrolytic degradation of coated PET geogrids will also be developed in this phase.

The third phase consists of a long-term (i.e., up to 2.5 years in length as currently planned) systematic experimental program to evaluate the effect various environmental variables have on the hydrolytic degradation rate in laboratory immersion environment of typical PET geosynthetic products. Elevated temperature, and to some extent the pH of the immersion liquid, will be the primary environmental variables used to accelerate degradation such that measurable strength losses occur within the desired time frame. This data will be modeled as a function of temperature (i.e., the Arrhenius model combined with a rate expression, discussed later in this report) to permit extrapolation to typical in situ temperatures and/or as a function of pH to permit extrapolation from alkaline (and to some extent acidic) conditions to more typical neutral conditions found in situ.

It is specifically planned in Phase 3 to evaluate three PET geosynthetics such that a range of chemical properties, fiber properties and overall geosynthetic constructions are evaluated. The one coated geogrid product will be tested with and without the PVC coating to assess the long-term effect the coating has on hydrolysis of PET geosynthetics. It is not the intent of this study to evaluate every possible combination of polymer chemical makeup currently available, but instead is to develop a test protocol which can be used by various geosynthetic manufacturers and geosynthetic users to assess the potential long-term performance of any given geosynthetic

product. The key environmental variables and test acceleration techniques evaluated in this task would be used to allow the protocol developed to be related, at least to some degree, to typical in situ conditions. Environments to be evaluated include four temperatures, three alkaline immersion solutions (both sodium and lime), one acidic solution (sulfuric acid), a neutral solution (water), a moderate and high humidity condition, and a limited study of hydrolysis of one product in soil at neutral pH. Hydrolysis reactors (i.e., an immersion tank) in which the solution is continuously stirred will be used to expose the geosynthetic specimens to these conditions. Specimens will be allowed to degrade until a strength loss of 50 percent is obtained, if possible. Typically, five sample retrievals performed during the duration of the test exposure time, with five specimens per retrieval, are planned for each environmental condition and geosynthetic product tested. The test program may be adjusted as early results become available. Some additional samples for additional retrievals will be included at the lowest test temperature should longer incubation times than originally planned be required.

Wide width tensile strength will be used to characterize the strength of the geosynthetics as hydrolysis progresses. Specimen weight loss will also be measured. Scanning electron microscopy (SEM) will be conducted as needed to evaluate the physical nature of the hydrolytic damage, especially in alkaline conditions, to help differentiate between inner and outer hydrolysis degradation mechanisms. Hydrolysis will be tracked chemically using intrinsic viscosity to determine molecular weight and by measuring the CEG.

To date, Phases 1 and 2 of this task have been completed, and the specimens to be evaluated in Phase 3 have been placed in the ovens and have been incubating since early January, 1994. The test program has been adjusted based on the results of Phases 1 and 2 (provided in the next chapter), with additional minor adjustments based on the early results from the Phase 3 testing.

TASK F- BEHAVIORAL PATTERNS OF GEOSYNTHETIC AGING

In this task, all of the data generated in Tasks A through E will be analyzed to establish behavior patterns of geosynthetic aging. New procedures and design methodologies to account for and/or predict changes in the long-term geosynthetic strength and deformation characteristics due to aging will be developed.

TASK G- DEVELOPMENT OF CREEP TESTING PROTOCOL

This task will parallel the confined in-soil load-elongation testing conducted in Task C, except that the focus will be on time dependent behavior under constant load (i.e., creep). Additionally, unconfined creep tests to evaluate the stress crack resistance of HDPE geogrids and to evaluate testing protocols will be conducted.

Regarding the confined in-soil creep tests, a test program will be developed by conducting a literature review to define key variables and identify the most appropriate apparatus and preliminary test protocols to investigate. These preliminary test protocols will be developed with the following considerations in mind:

- Simplicity, reliability, and availability of test equipment.

- Ability to conduct a large number of tests.
- Ability to test a variety of geosynthetic and soil types.
- Ability to standardize soil preparation procedures.
- Ability to measure and evaluate patterns of relative displacement between the geosynthetic and the confining soil and to directly measure load in the geosynthetic.
- Ability to decouple the confined tensile response from the soil/geosynthetic interface shear,

Equipment used will likely be either a triaxial or a pullout type device similar to that used for Task C, modified as appropriate. A trial test program is planned to more fully develop or adjust the selected protocol. The final protocol will be developed with consideration of the possibility of testing either chemically degraded specimens or to chemically degrade specimens in the device at an elevated temperature or through long-term chemical submergence during the creep test. Once the final test protocol is developed, test variables such as soil type, geosynthetic type, confining pressure, and geosynthetic load level will be evaluated. Some tests may be conducted in a submerged condition.

To date, the literature review and preliminary test protocols for the confined creep testing have been completed, Preliminary testing to evaluate and refine the preliminary test protocols has begun.

Regarding the stress crack resistance testing and protocol development, the testing program will be conducted using a four step approach. In the first two steps, HDPE resin materials will be evaluated, whereas in Steps 3 and 4, geogrid product will be evaluated. In Steps 1 and 4, notched specimens will be tested, whereas in Steps 2 and 3, unnotched specimens will be tested. Each of the four steps are summarized as follows:

1. Notched creep rupture resistance of three HDPE resins, of which one is a resin used for HDPE geogrids, one is a resin known to have low stress crack resistance, and one is a resin known to have high stress crack resistance, will be determined at 50 °C (122 °F) in water. Five load levels will be tested which vary from 25 to 65 percent, and three specimens will be tested at each load level. This testing is intended to verify that the selected protocol (ASTM D5397, modified to use water rather than Igepal as the surface wetting agent) will discriminate between a crack resistant and a crack sensitive material and that the resin used for geogrids is crack resistant. Because the specimens are notched, this testing only evaluates crack propagation.
2. The testing proposed for this step is the same as that to be performed in Step 1, except that all specimens will be unnotched. Therefore, evaluates both crack initiation and crack propagation. Comparison with Step 1 results will allow the effects of crack initiation to be separated from the effects of crack propagation. This testing is intended to additionally determine if the test, with the added step of crack initiation, which is a more realistic situation

for geogrids in situ, can still discriminate between crack resistant and crack sensitive resins. It is hoped that these results will support the practice of notching to accelerate the test,

3. In this step, unnotched geogrid specimens will be tested using (creep) stress rupture testing. Test conditions will be similar to ASTM D5397, except that the clamping arrangement will not pierce the specimens, water will be used as the environment, and the specimens are unnotched. Temperatures tested will be 50 °C (122 °F), 65 °C (149 °F), and 80 °C (176 °F), and the testing times will be extended to as much as 10,000 h. This testing is intended to assess the stress crack resistance of unnotched geogrid product at temperature levels known to accelerate stress cracking. It is anticipated that this data can be used to make life predictions and to establish the safe design stress level which will preclude slow crack growth failures. This data will provide a baseline for the development of accelerated test procedures. Furthermore, comparison of this data to data from Steps 1 and 2 will allow the effect of molecular orientation on stress crack resistance to be determined.
4. Tests performed in this step on geogrid specimens are the same as those performed in Step 3, except the geogrid will be notched in the relatively nonoriented geogrid nodal material. Some initial parametric studies will be conducted in this step to determine the specific notch geometry and location for the notched specimen testing. The objective of this step is to qualify an accelerated test which utilizes notched specimens that can be used to obtain quantitative design oriented data.

Scanning electron microscopy will be used to evaluate fracture surfaces. Index testing such as ultimate tensile strength, polymer density, melt flow index, and percent crystallinity will be used to characterize the materials tested.

To date, the specimens to be tested in Steps 1 and 2 have been made, and the parametric study to evaluate notch details has begun. Unnotched specimens were loaded in May, 1994, and creep data is being obtained.

TASK H-FINAL REPORT

This task consists of the development of a final report which summarizes the overall test program and research approach, a description of how each task was performed including test procedures and apparatus used, a summary of all of the data obtained in the study, a discussion of the behavior observed, and any conclusions which can be made. Detailed test results are to be provided in a separate report.

A design chapter is also to be provided. This chapter will present methods for the determination of long-term geosynthetic strength and deformational characteristics, including a step-by-step approach to be used in any reinforcement application. Recommended procedures for short-term tests needed to predict long-term geosynthetic performance for both design and quality control purposes, as well as geosynthetic specification guidelines, will be included.

This task is anticipated to be completed by the end of 1996.

4. TASK D, SUMMARY OF PHASE 1 AND 2 RESULTS

The specific testing program for this task is provided in chapter 3.0 of this report. This task focuses on the oxidation degradation mechanism. It must be understood that test results from the Phase 1 and 2 testing discussed, or in limited cases presented, herein are preliminary in nature and intended only for the purpose of developing the Phase 3 long-term testing program. The relationship of the results from the Phase 1 and 2 testing to potential long-term product life is at this point unknown.

CHARACTERISTICS OF PRODUCTS SELECTED FOR TESTING

Candidate products were initially selected during the Task A planning such that a variety of product macrostructures and polymer compositions are evaluated. Due to budget limitations, only four to five geosynthetic products can be evaluated in this task in Phase 3. Testing performed in Phases 1 and 2 was used to refine the product selection process. In addition to product variety, potential statistical product variability was also considered. It was desirable to select products with relatively low property variability to minimize the number of specimens required. Strength reported later in this chapter was used to determine this. In general, it was felt that chemical property variations would not be as great as the strength property variations and that strength testing needs would control the number of specimens required. A summary of the final product selection, including the reference materials used only for Phase 1 and 2 test protocol development, is provided in table 3.

PHYSICAL AND MECHANICAL CHARACTERIZATION OF PRODUCTS IN “AS-RECEIVED” CONDITION

Up to 10 specimens of each geosynthetic product were tested to measure unit weight (ASTM D3776), thickness (ASTM D 1777), and tensile strength. Regarding tensile strength, the wide strip method (ASTM D4595, but modified for geogrids) was used. Additionally, the geogrid was tested to evaluate the effect the number of ribs tested in a given specimen had on the measure tensile strength.. Both single rib and an eight rib wide specimens were tested. No statistical difference in the measured strength per rib for the different specimen widths was observed. Therefore, all geogrid testing will utilize one rib wide specimens. A summary of the physical testing is provided in table 4.

The number of specimens required for strength testing was determined using the Student's t distribution as outlined in ASTM D4595. The coefficient of variation obtained for the sample for wide width strength was compared to the manufacturer's reported values from quality control testing, which was assumed to be representative of the variation for the population, as available (this data was available for two PP products). The sample and manufacturer values were found to be similar. Furthermore, all specimens were taken from the center of the roll width, and all future specimens will be taken from the same area from the same geosynthetic roll. This was done to minimize specimen variation and to ensure that the recommended number of specimens will be statistically adequate for the future testing.

Table 3. Geosynthetics and reference polymers selected for oxidative degradation experiments.

Code Number	Polymer	Geosynthetic Macrostructure
P-1	HDPE	410 g/m ² (12 oz/yd ²) Uniaxially Drawn Geogrid
P-3	PP	270 g/m ² (8 oz/yd ²) Continuous Filament Needleponched Nonwoven Geotextile
P-4	PP	270 g/m ² (8 oz/yd ²) Staple Filament Needleponched Nonwoven Geotextile
P-9	PP	200 g/m ² (6 oz/yd ²) Slit Film Woven Geotextile
P-10	PP	270 g/m ² (8 oz/yd ²) Continuous Filament Needleponched Nonwoven Noncommercial Fabric Produced with Minimal Antioxidants for Research Purposes Only
P-11	PP	270 g/m ² (8 oz/yd ²) Staple Filament Needleponched Nonwoven Noncommercial Fabric Produced with Minimal Antioxidants for Research Purposes Only
S-1	HDPE	Pellets (reference material), Molecular Weight of 125,000, Density of 0.95, and Melt Index of 0.3
S-2	PP	Powder (reference material), Isotactic, Intrinsic Viscosity of 2.2 to 2.5, Density of 0.9, Tg of -26 °C, and Tm of 189 °C

Table 4. Physical and mechanical properties of polyolefin materials as-received.

Product Code Number	Number of Specimens Tested	Weight/Area ASTM D3776 (oz/yd²)	Thickness ASTM D1777 (in.)	Tensile Strength ASTM D4595 (lbs/in.)	Strain ASTM D4595 (%)	Coeff. of Variation (%)	Minimum Required Number of Specimens for Strength
P-1	9 9 8 8	12.66	0.07	339	37.8	9.65 4.03 4.86 15.3	5
P-3	9 9 7 7	10.01	0.074	83	117	1.2 1.3 4.7 13.1	5
P-4	10 10 10 10	11.09	0.1	99	83.5	4.52 4.79 3.08 9.02	5
P-9	10 10 10 10	7.23	0.026	232	21.1	1.24 3.35 2.51 3.88	5

1 oz/yd² = 33.9 g/m²

1 inch = 25.4 mm

1 lb/in = 0.175 kN/m

PROTOCOL DEVELOPMENT TO CHEMICALLY TRACK OXIDATIVE DEGRADATION

The focus of this subtask was to select and fine tune test methods which could be used to chemically track oxidation as it progresses. Chemically tracking oxidation as it progresses in polyolefins is a very complex endeavor. Oxidation results mainly in antioxidant consumption at first but transitions to mainly degradation of the main polymer in the later stages of oxidation. Chemical tracking can either focus on the consumption of antioxidants or it can focus on changes which occur in the main polymer as oxidation progresses. Since a given polymer may contain a complex mixture of antioxidants which work synergistically together, and since antioxidant packages are usually considered proprietary, making antioxidant details difficult to obtain, quantitative tracking of antioxidant depletion is difficult. In the past, attempts were made with limited success to track antioxidant depletion using OIT or HPLC.

Quantification and interpretation of changes in the main polymer as oxidation progresses are also fraught with difficulties. For example, though molecular weight can be measured, oxidative degradation does not always cause molecular weight to decrease, as either chain scission or crosslinking can occur. Furthermore, oxidation occurs primarily on the polymer surface, leaving the core of the polymer unaffected. Since the tests available to assess changes in the main polymer must analyze the entire polymer specimen, not just its surface material, degradation must progress to relatively high levels to raise the average response of the material to a sufficiently high level to detect.

Several test methods were investigated, and in some cases tests were conducted, to evaluate their viability to track oxidative degradation of the main polymer. TGA and DSC tests were performed in both nitrogen (an inert environment) and air to assess the sensitivity of polymer decomposition and melting temperatures to oxidation. Though these test methods did show some sensitivity to the presence of oxygen, these tests could not detect significant differences between products. These test methods are still somewhat useful for overall characterization of the polymers, however. Fourier Transform Infrared spectroscopy (FTIR) was also investigated as to its viability for tracking oxidation, but no tests were performed. FTIR was considered unsuitable for detecting oxidation as it progresses because of interpretation difficulties and inadequate sensitivity. It was concluded that, in general, measurement of changes to the main polymer material was not a viable approach to tracking degradation chemically.

OIT and HPLC were investigated regarding their viability to track antioxidant depletion as oxidation progresses. Detailed studies and geosynthetic product testing were conducted to investigate this. OIT tests were conducted on reference polymers and geosynthetic products using the most widely used procedure, ASTM D3895 as modified by Gray (1990). It was found that the test could not differentiate between different products, with OIT's for P-1 and S-1 varying from 5.12 to 5.26 min, respectively. Therefore, an investigation to determine how to modify the test to better differentiate between products and ensure repeatability was conducted. The heating program, nitrogen and oxygen flow rates, rate of shifting from nitrogen to oxygen, and the metal used in the specimen pan were changed as a result of the investigation, causing the OIT to now vary from approximately 50 to 70 min for the HDPE products and 2.5 to 5.5 min for the PP products. This method still did not provide adequate differentiation between PP products. It was found that the test temperature needed to be lowered from 200 °C (392 °F) to 175 °C

(347 °F) for the PP materials to provide adequate differentiation between products, yet stay above the melting temperature of PP. OIT calibration curves developed using the reference polymer S-2 and known percentages of Irganox 1010 showed that the OIT test as modified was sensitive in a consistent manner to antioxidant content. However, due to the complexities of antioxidant packages in polymers, it was concluded that OIT could only be used as a qualitative assessment of the overall response of the polymer to oxidation and could not be the sole criterion used to track oxidation.

Investigation of the HPLC method showed that antioxidant loss could be quantitatively tracked using this method, even if more than one antioxidant is present, provided that the type of antioxidant is known going into the test or that the components present can be isolated and identified, and provided that the antioxidants in the mixture do not overlap in their responses in the test. Preliminary studies to investigate the ability of the HPLC device to be calibrated to measure antioxidants typically used for geosynthetics were conducted. Based on these studies, HPLC appears to be the best method available to monitor antioxidant depletion as oxidation progresses.

PRELIMINARY STUDIES OF ANTIOXIDANT LEACHABILITY IN WATER

A preliminary assessment of antioxidant leachability for PP geosynthetics immersed in water was conducted. Specimens of three different PP products were placed in distilled water at 70 °C (158 °F) with stirring for 18 days. Changes in the product polymers with time were monitored using only the modified OIT test. The results indicated a rapid decrease in OIT in the first 2 to 5 days followed by only slow changes. Though there were differences in the OIT decrease rate between the three products tested, the significance of those differences is unknown. Leaching by this test method appears to be temperature, product, and antioxidant dependent and is therefore not an appropriate method to obtain antioxidant free specimens for the Phase 3 testing. Furthermore, the relationship between antioxidant leaching in such severe test conditions or other similar types of data reported in the literature to realistic in situ conditions is unknown. It is not clear at this point if leaching of antioxidants through exposure to water in soil at in situ temperatures is really a significant issue at all. However, because of the available published information (Wisse, 1990) which indicates the potential for leaching to occur, a limited study under more realistic test conditions (i.e., high humidity oven aging experiments) will be conducted in the Phase 3 testing.

EFFECTS OF METAL SALT CONTAMINATION

The literature has indicated that certain transition metals in contact with polyolefins can accelerate the rate of oxidation. Iron is the most common transition metal in soil and is therefore the focus of this study. Preliminary studies of this phenomenon were conducted to develop a preliminary protocol to prepare iron salt contaminated specimens, to assess the general effect concentration of the iron solution and time of exposure to the iron solution have on the potential degradation rate, and to select specific iron concentrations in the specimens for Phase 3 testing.

To this end, reference polymer S-2 was combined with a 0.10 percent Irganox 1010 concentration (i.e., antioxidant) and soaked in FeCl₃ solution to form specimens with various percentages of

FeCl₃ on a weight of FeCl₃ per weight of polymer basis. The specimens were then pressed into a film and subjected to OIT testing. The results indicated that iron contamination reduced OIT times by a factor of 3 to 10 for FeCl₃ concentrations in the specimens of 0.2 percent to 1.0 percent, which is similar to what has been reported in the literature. For iron concentrations greater than 1.0 percent, there was little additional decrease in OIT. Therefore, iron concentrations of 0.2 percent and 1.0 percent were selected for testing in Phase 3.

PRELIMINARY OVEN AGING EXPERIMENTS UNDER SEVER EXPOSURE CONDITIONS

The purpose of this subtask are to:

- Identify the best oven features to ensure uniformity of the test environment and to ensure that the selected equipment will allow the desired test program to be accomplished.
- Determine specimen installation procedures and other test protocol details for the oven aging experiments.
- Obtain preliminary product performance data to help estimate the incubation times and define the test program in Phase 3.
- Compare the results obtained from chemical tracking of the oxidation to the observed sample strength losses to refine the chemical tracking program.

Based on previous experience, a forced air oven which meets the requirements of ASTM D3045 and ASTM E145, Type II-B was selected for the experiments. Specifically a Blue-M 0.23 m³ (8 ft³) Stabil-Therm Bench type oven with a temperature uniformity of ~~off~~ 1 percent and a horizontal air flow was selected. It was determined that specimens should be placed in the oven parallel to the air flow spaced no closer together than 13mm (0.5 in.) apart.

A study to evaluate the shrinkage response of the geosynthetics at 90°C (194 °F) was conducted. Based on the literature, significant shrinkage can affect the mechanical properties as well as the molecular structure of the polymer, and it is desirable to avoid such effects. Specimens are sometimes framed to prevent shrinkage can create load in the specimen, which can also affect the response of the specimen to oxidation. Therefore, specimens of all the PP geotextiles to be evaluated were placed in an oven at 90°C (194 °F) for 40 days. No shrinkage was observed. It was therefore decided that the specimens need not be framed.

Once the preliminary oven aging protocol was developed, a limited oven aging experiment was conducted utilizing specimens of all the geosynthetic products listed in table 3. An exposure temperature of 110 °C (230 °F) for 40 days. No shrinkage was observed. It was therefore decided that the specimens need not be framed.

Once the preliminary oven aging protocol was developed, a limited oven aging experiment was conducted utilizing specimens of all the geosynthetic products listed in table 3. An exposure temperature of 110 °C (230 °F) was selected, which is similar to what is currently proposed in Europe for an oven aging index test. A high temperature was selected to insure that results could be obtained in a relatively short time and to eventually determine, after the Phase 3 testing is complete, if this relatively short-term high temperature test could be used to estimate the long-term performance of products in the future. Key issues regarding this determination are the proximity of this high temperature to the melt range for some products and the validity of extrapolation of such data to in situ (i.e., low temperature) performance.

Specimens were allowed to incubate for up to 80 days. Three retrievals of three specimens per product were performed. Wide width strength tests, weight loss, and OIT tests were conducted for each set of specimens. These results showed that considerable variation in product response to the rather severe oxidative environment occurred. Whether or not such a wide spread in product response will occur at lower temperatures is unknown, but will be investigated in Phase 3. Note also that the spread may not be significant in a practical sense if future estimates of product life, which will not be possible to make until the Phase 3 results are available, indicate that even the product with the most rapid degradation rate has an acceptable life.

Comparison of the strength loss data with OIT and weight loss measurements indicate only a general, qualitative correlation. The correlation was not adequate for quantitative analysis. Therefore, OIT and weight loss measurements will have only a limited usefulness in tracking oxidative degradation.

This preliminary oven aging data was used to refine the testing program for Phase 3. Furthermore, early Phase 3 test results have required that some additional changes be made to the test program. The revised test program for Phase 3 as it currently stands is provided in table 5.

Table 5. Revised matrix of test conditions for task D, phase 3.

Product Code Number	Antioxidant	FeCl₃ Contaminated	Environment	Temperature (°C)	Estimated Test Duration (days)
P-3, P-9	Yes	No	21 percent oxygen, dry	60	850
				70	600
				80	300
				90	100
P-4	Yes	No	21 percent oxygen, dry	40	800
				50	400
				60	200
				70	100
				80	80
P-1	Yes	No	21 percent oxygen, dry	80	850
				90	600
				100	400
				110	200
P-10	No	No	21 percent oxygen, dry	50	500
				60	300
				70	200
				80	100
P-11	No	No	21 percent oxygen, dry	40	400
				50	200
				60	100
				70	50
				80	25
P-3, P-9	Yes	No	7 percent oxygen, dry	60	1000
				70	500
				80	250
P-4	Yes	No	7 percent oxygen, dry	60	600
				70	400
				80	200

Table 5. Revised matrix of test conditions for task D, Phase 3 (continued).

Product Code Number	Antioxidant	FeCl ₃ Contaminated	Environment	Temperature (°C)	Estimated Test Duration (days)
P-1	Yes	No	7 percent oxygen, dry	90 100 110	500 250 150
P-10	No	No	7 percent oxygen, dry	60 70 80	400 200 150
P-11	No	No	7 percent oxygen, dry	60 70 80	300 150 75
P-3, P-4	Yes	Yes (0.2 percent, 1.0 percent concentration)	21 percent oxygen, dry	40 50 60	400 200 100
P-10, P-11	No	Yes (0.2 percent, 1.0 percent concentration)	21 percent oxygen, dry	40 50 60	200 100 50
P-9	Yes	No	21 percent oxygen, 90 percent RH	60 70 80	200 150 100

Note: For each condition, 5 retrievals with 5 specimens per retrieval are planned.

$$^{\circ}\text{F} = (9/5)(^{\circ}\text{C}) + 32$$

5. TASK E, SUMMARY OF PHASE 1 AND 2 RESULTS

The specific testing program for this task is provided in chapter 3.0 of this report. This task focuses on the hydrolytic degradation mechanism. It must be understood that test results from the Phase 1 and 2 testing discussed, or in limited cases presented, herein are preliminary in nature and intended only for the purpose of developing the Phase 3 long-term testing program. The relationship of the results from the Phase 1 and 2 testing to potential long-term product life is at this point unknown.

CHARACTERISTICS OF PRODUCTS SELECTED FOR TESTING

The product selection process for this task was the same as that used for task D (see chapter 4.0). A summary of the final product selection for Task E, including the reference materials used only for Phase 1 and 2 test protocol development, is provided in table 6.

PHYSICAL AND MECHANICAL CHARACTERIZATION OF PRODUCTS IN "AS-RECEIVED" CONDITION

Up to 10 specimens of each geosynthetic product were tested to measure unit weight (ASTM D3776), thickness (ASTM D1777), and tensile strength. Regarding tensile strength, the wide strip method (ASTM D4595, but modified for geogrids) was used. All geogrid testing utilized one rib wide specimens. A summary of the physical testing is provided in table 7.

The number of specimens required for strength testing was determined using the Student's t distribution as outlined ASTM D4595. The coefficient of variation obtained for the sample for wide width strength was compared to the manufacturer's reported values from quality control testing, which was assumed to be representative of the variation for the population, as available (this data was available for one PET product). The sample and manufacturer values were found to be similar. Furthermore, all specimens were taken from the center of the roll width, and all future specimens will be taken from the same area from the same geosynthetic roll. This was done to minimize specimen variation and to ensure that the recommended number of specimens will be statistically adequate for the future testing.

PROTOCOL DEVELOPMENT TO CHEMICALLY TRACK HYDROLYTIC DEGRADATION

The focus of this subtask was to select and fine tune test methods which could be used to chemically track hydrolysis as it progresses. Based on the available literature, molecular weight measurement has been most often used to track hydrolysis and with success, at least for hydrolysis in low to moderate pH solutions. Since hydrolysis results in chain scissions in the main polymer, thereby directly reducing the molecular weight and tensile strength, it should be relatively easy to track hydrolysis by monitoring this property. CEG contents have also been shown to be a factor in the rate of hydrolysis and could also be used to evaluate hydrolysis as it progresses.

In general, two methods have been successfully used to monitor changes in molecular weight: Gel Permeation Chromatography (GPC) and Intrinsic Viscosity (IV). The molecular weight and its distribution can be determined directly using GPC, whereas, the molecular weight must be

Table 6. Geosynthetics and reference polymers selected for hydrolytic degradation experiments.

Code Number	Polymer	Geosynthetic Macrostructure
P-5	PET	540 g/m ² (16 oz/yd ²) Continuous Spunbonded Filament Needlepunched Nonwoven
P-6	PET	340 g/m ² (10 oz/yd ²) PVC Dipped Woven Geogrid
P-7	PET	370 g/m ² (11 oz/yd ²) Multifilament Woven Geotextile
P-8	PET	Single Fiber Corresponding to P-6
S-3	PET	Flakes (reference material), Inherent Viscosity 0.7, Density 1.385, and T _g of 81 °C (178 °F)

Table 7. Physical and mechanical properties of polyester materials as-received.

Product Code Number	Number of Specimens Tested	Weight/Area ASTM D3776 (oz/yd ²)	Thickness ASTM D1777 (in.)	Tensile Strength ASTM D4595 (lbs/in.)	Strain ASTM D4595 (%)	Coeff. of Variation (%)	Minimum Required Number of Specimens for Strength
P-5	9 9 9 9	16.44	0.177	204	75.3	2.26 2.34 4.59 2.1	5
P-6	9 9 9 9	10.83	0.05	1037 (lbs/rib)	17	5 5.1 10.3 15.4	15
P-7	9 9 8 8	10.64	0.031	544	18.8	3.1 3.8 4.3 7.4	5

1 oz/yd² = 33.9 g/m²

1 inch = 25.4 mm

1 lb/in = 0.175 kN/m

determined through a correlation/calibration using IV. This calibration is dependent on the solvent system and temperature used to perform the test. Several solvent systems were evaluated for use in this study. Some differences in the molecular weight calculated, even after calibration, will result from use of different solvent systems. However if the same solvent system and temperature are always used, changes in molecular weight can be tracked with acceptable accuracy using this method. Since IV is considerably easier to perform than GPC, the experimental work focused on fine tuning the IV test for use in this study. It was decided that only one solvent system would be selected and used for the remainder of the research program to insure repeatability and ease of interpretation of the test results. The solvent system selected was Phenol/TCE=4/6 (wt./wt.) at 25 °C (77 °F). Therefore, IV will be the primary method used to chemically track hydrolysis. CEG contents will also be measured as needed to track hydrolysis.

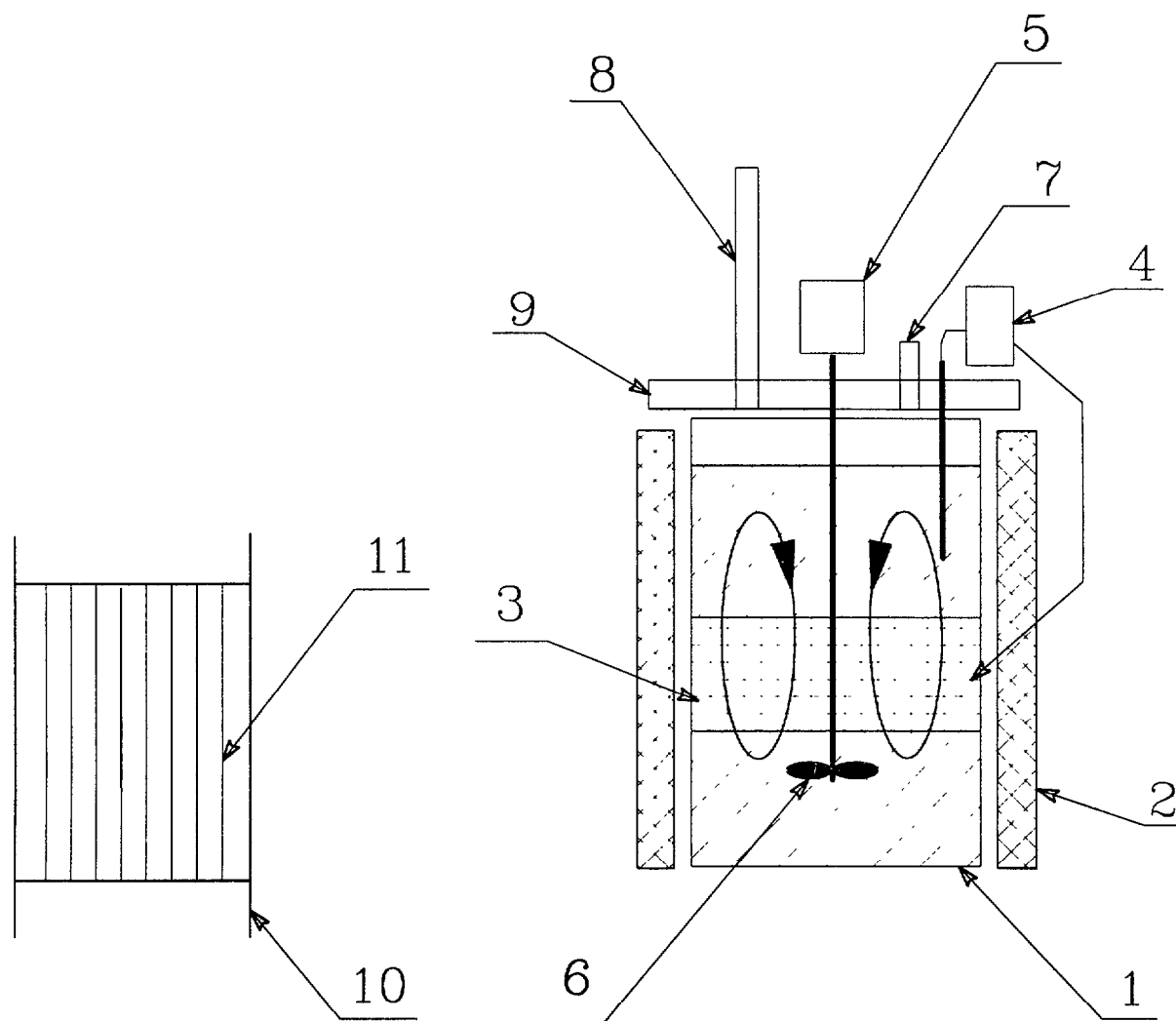
Evaluation of changes in molecular weight as hydrolysis progresses are likely to work well for hydrolysis in low to moderate pH conditions. However, as stated previously, surficial erosion of the polymer can become a more dominant factor than molecular weight loss in alkaline (i.e., high pH) conditions. Therefore, especially at high pH conditions, weight loss measurements and a visual assessment of the fiber surface using SEM will be performed in combination with IV measurements as part of the overall hydrolysis tracking protocol.

PRELIMINARY HYDROLYSIS EXPERIMENTS UNDER SEVER EXPOSURE CONDITIONS

The purpose of this subtask was to identify and fine tune apparatus (i.e., a reactor) which could be used to expose the geosynthetic specimens, determine specimen installation procedures and other test protocol details for the hydrolysis experiments, obtain preliminary product performance data to help estimate the incubation times, select incubation environments, and define the test program in Phase 3, and to compare the results obtained from chemical tracking of the hydrolysis to the observed sample strength losses to refine the chemical tracking program as needed.

Due to the special needs of the study in terms of the reactor size needed as well as other details, a custom reactor was designed for use in the Phase 3 testing. The reactor design principles used were similar to those used for standard, commercially available reactors. Special attention was paid to temperature uniformity (i.e., ± 1 percent) and stability during long-term use. The volume of the reactor developed is 50 L (13 gal) which allows storage of up to 40 full-sized specimens, which complies with a minimum solid/liquid ratio of 1:40. Pyrex glass was used for the reactor body for acidic solutions and stainless steel for neutral and alkali solutions. Nitrogen was poured in the space between the reactor head and the liquid to minimize possibilities for oxidation. Specimens were suspended on a Teflon or stainless steel frame with minimum separation distance of 13 mm (0.5 in.). The solution was intensively stirred to ensure solution uniformity. The reactor is illustrated in figure 2.

Preliminary studies were conducted prior to attempting full-scale, high temperature incubations to assess the best way to remove the coating from the coated geogrid. The coating was characterized by weight content of PVC on the geogrid. It was determined that the best way to remove the coating was by soaking the specimen in tetrahydrofuran (THF) and then washing. However, removing the coating tends to change the overall structure of the PET yarn (i.e., the fibers don't stay together well.)



- 1 - PYREX glass 50-liter jar; 2 - thermal insulation; 3 - outer heating tape;
 4 - temperature controller; 5 - stirrer motor; 6 - turbine blade agitator; 7 nitrogen inlet port;
 8 - condenser; 9 - Teflon head with inlet ports; 10 - Teflon frame; 11 - specimens

Figure 2. Schematic of temperature controlled reactor for hydrolytic degradation.

The two most likely ions to be encountered in practice where PET geosynthetics could be used include calcium and sodium. Therefore, preliminary studies were conducted to evaluate the practicality of using sodium and/or calcium in solutions of various pH levels for long-term hydrolysis studies. Special consideration was given to the development and long-term maintenance of high temperature, high pH solutions incorporating these ions (i.e., calcium hydroxide and sodium hydroxide) to be used for accelerated hydrolysis tests. The studies indicated that the pH of a saturated lime solution is a maximum of 11.5 at 90 °C (194 °F) and will decrease continuously to a pH of 9.6 in 20 days due to precipitation of the lime out of solution, unless the solution is frequently replenished. Furthermore, PET fibers placed in this type of solution were observed to become coated with the lime precipitate. Both of these factors make high pH lime solutions impractical for use, and the direct comparison in the literature of PET hydrolysis results in lime (i.e., calcium) and sodium solutions which show lime to be a more aggressive media appear to be erroneous. However, lime solutions do become more stable at lower temperatures and moderately alkaline conditions (i.e., pH of 10 or less), and could be used in that case.

Once all these protocol details were developed, full size geosynthetic specimens were placed in the prototype reactor to conduct preliminary hydrolytic degradation experiments at high temperature and pH. A temperature of 90 °C (194 °F) at a pH of 12.7 in sodium hydroxide (0.1 N) was selected to insure that significant property changes would be observed in a relatively short period of time. Four retrievals of three specimens each were performed. The test results were used to begin the assessment of the effect of the coating on the rate of hydrolysis at the high pH, high temperature condition, to fine tune the estimated incubation periods for the Phase 3 testing, and to assess how well viscosity, weight loss, and SEM track hydrolytic degradation.

Regarding strength loss, the test results confirmed what has been reported in the literature (Risseuw and Schmidt, 1990; Anderson, et. al., 1992; Jailloux, et. al., 1992). A wide spread in product response occurred. Whether or not this relatively wide spread in product response will occur at lower temperatures is unknown, but will be investigated in Phase 3. Note also that the spread may not be significant in a practical sense if future estimates of product life, which will not be possible to make until the Phase 3 results are available, indicate that even the product with the most rapid degradation rate has an acceptable life.

Only viscosity measurements were obtained for the evaluation of the effect of the coating on the hydrolysis rate of PET geogrid in severe conditions. Both sets of specimens prepared for this test were 13 cm (5 in) long and were fully immersed in the solution. The PET fibers were exposed to the solution at the cut ends of the coated geogrid specimens. This data indicates that the coating is at least initially a partial barrier to hydrolytic degradation. However, longer term testing will be required to fully evaluate this issue.

The viscosity data appeared to correlate only roughly with the strength loss data obtained in the high pH environment, indicating that overall weight loss and degree of surficial erosion observed from SEM photographs in combination with viscosity changes must be used to track the progress of hydrolysis in high pH environments. This, as well as viscosity losses occurring in low to neutral pH conditions, will be more fully investigated in Phase 3. Photomicrographs of the fiber

surfaces indicated that significant surface erosion (i.e., “outer” hydrolysis) was occurring in the high pH solution.

This preliminary hydrolytic degradation data was used to refine the testing program for Phase 3. Furthermore, early Phase 3 test results have required that some additional changes be made to the test program. The revised test program for Phase 3 as it currently stands is provided in table 8.

Table 8. Revised matrix of test conditions for task E, phase 3.

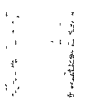
Product Code Number	PVC Coating Removed?	Environment	Temperature (°C)	Estimated Test Duration (days)
P-S	N/A	Distilled Water (pH=7)	50	900
			60	700
			70	350
			80	200
P-6, P-7	No for P-6, N/A for P-7	Distilled Water (pH=7)	60	1000
			70	700
			80	250
			90	100
P-5	N/A	H ₂ SO ₄ (pH = 1)	50	700
			60	450
			70	250
P-6	No	H ₂ SO ₄ (pH= 1)	50	700
			60	450
			70	250
P-7	N/A	H ₂ SO ₄ (PH = 1)	60	700
			70	450
			80	250
P-5	N/A	NaOH (pH=12)	30	300
			40	150
			50	75
P-6	No	NaOH (pH=12)	50	500
			60	350
			70	250
P-6	Yes, but No Mechanical Tests to Be Performed	NaOH (pH=12)	50	500
			60	350
			70	250
P-7	N/A	NaOH (pH=12)	50	500
			60	350
			70	250

Table 8. Revised matrix of test conditions for task E, Phase 3 (continued).

Product Code Number	PVC Coating Removed?	Environment	Temperature (°C)	Estimated Test Duration (days)
P-5	N/A	NaOH (pH=10)	50	700
			60	500
			70	250
P-6	No	NaOH (pH=10)	50	850
			60	700
			70	600
P-7	N/A	NaOH (pH=10)	50	850
			60	700
			70	600
P-5	N/A	Lime (pH=10)	50	600
			60	400
			70	200
P-7	Yes	Lime (pH=10)	50	850
			60	700
			70	600
			80	400

Note: For each condition, 5 retrievals with 5 specimens per retrieval are planned.

$$^{\circ}\text{F} = (9/5)(^{\circ}\text{C}) + 32$$



6. ISSUES TO BE ADDRESSED TO ACCOMPLISH LIFE PREDICTION OF GEOSYNTHETIC REINFORCEMENT

Engineering design dictates that the length of time a material used as an integral part of a structure will remain functional be known with some degree of certainty. In the case of geosynthetic reinforcement, functionality is defined in terms of its strength, and to some extent its load-strain characteristics. The length of time the material remains functional is defined as the life of the material. For example, if the material degrades and becomes too weak to support the loads placed upon it, the material fails and loses functionality. Life prediction of a given material is the ultimate goal of tests to assess material durability.

Conditions in laboratory durability research studies are typically severe when compared to actual in-soil conditions so that test results can be obtained in a relatively short period of time. Temperature is usually the main accelerator used in these tests. The key to the prediction of the life of a given polymeric product is the availability of proven extrapolation procedures. For example, if degradation tests are conducted at high temperatures, the test data must be extrapolated or otherwise somehow related to behavior at lower temperatures typical of in situ conditions to be of any value. Furthermore, if property changes are measured over a relatively short period of time, say 2 or 3 years, as would be the case in a laboratory study, or even 10 years, such as would be the case in a field exhumation study, that short-term data must be extrapolated to the desired design life of the material (typically on the order of 75 years or more).

How does one go about developing an extrapolation model to make use of the available short-term data? Such a model must consider the properties of the material itself in terms of how the environment affects that material, and must also consider the differences between the laboratory environment and the anticipated in situ environment. At least for chemical reactions which can result in material degradation, considerable data shows that most chemical reactions of this nature have a strong temperature dependence as well as a dependence on the concentration/quantity of the chemicals involved in the reaction. In fact, such dependence can be used advantageously to develop relationships which can be used for extrapolation purposes.

One relationship (i.e., equation) which is often used to develop extrapolation models is the Arrhenius equation, which addresses the temperature dependency of the degradation reaction, combined with an appropriate rate expression, which addresses the time dependency of the reaction (Koerner, et. al., 1992; Shelton and Bright, 1993). A common form of this important extrapolation tool is as follows:

$$\ln \frac{t}{t_o} = \frac{U}{R} \left(\frac{1}{T} - \frac{1}{T_o} \right) \quad (2)$$

where,

t = time to a given strength loss, usually 50%, at the test conditions

T = temperature of the test environment, in °K

t_o = time to the same given strength loss as for t , but in the in - situ environment

T_o = temperature of the in - situ environment, in °K

R = universal gas constant, which is 8.314 J / mole

U = effective activation energy, in J / mole

Equation 2 has been simplified by assuming that the reaction is first order by constituent. Should the reaction become autocatalytic (i.e., the reaction accelerates due to the catalytic effects of one or more of the reaction products themselves on the reaction) with time or after a certain magnitude of property change, the reaction order could change, affecting the accuracy of extrapolations performed with this equation. This issue will need to be considered when developing a life prediction model for geosynthetics.

An Arrhenius plot, in which degradation data is plotted as the logarithm (base “e”) of the reciprocal of time versus the reciprocal of temperature using equation 2, is often used to represent data extrapolated using this model. A conceptual example of an Arrhenius plot is provided in figure 3. Note that temperature has an exponential effect on the time to a given level of degradation based on this model, and that the data used in the equation is obtained at a constant level of degradation in the material as represented in this case by the strength loss. This equation could also be directly applied, for example, to the time required to reach a given loss in viscosity in the case of PET geosynthetics.

Data which fits this particular model well will plot as a straight line, with the slope of the line related to the effective activation energy. For this model to be completely valid, the activation energy must not be a function of temperature or other factors within the time and temperature range of interest. Also note that the activation energy determined using this model is really a combined or average activation energy resulting from a number of degradation processes all going on at the same time, hence the term “effective.” Due to this complexity in the activation energy for a given material, it is quite possible to have nonlinearity in the Arrhenius plot, especially as the difference between the lowest test temperature and the anticipated in situ temperature becomes large or if a transition such as the glass transition occurs within the temperature range of interest. It is therefore desirable to keep this difference in temperatures to a minimum.

It is anticipated that this model, or a modification of it, will be the primary tool used for analysis of the data obtained in Phase 3 of Tasks D and E, and the development of a life prediction model for geosynthetics. One of the key objectives for the Phase 3 testing is to define the activation energies which can be expected for typical geosynthetics and if in fact the Arrhenius plots for the

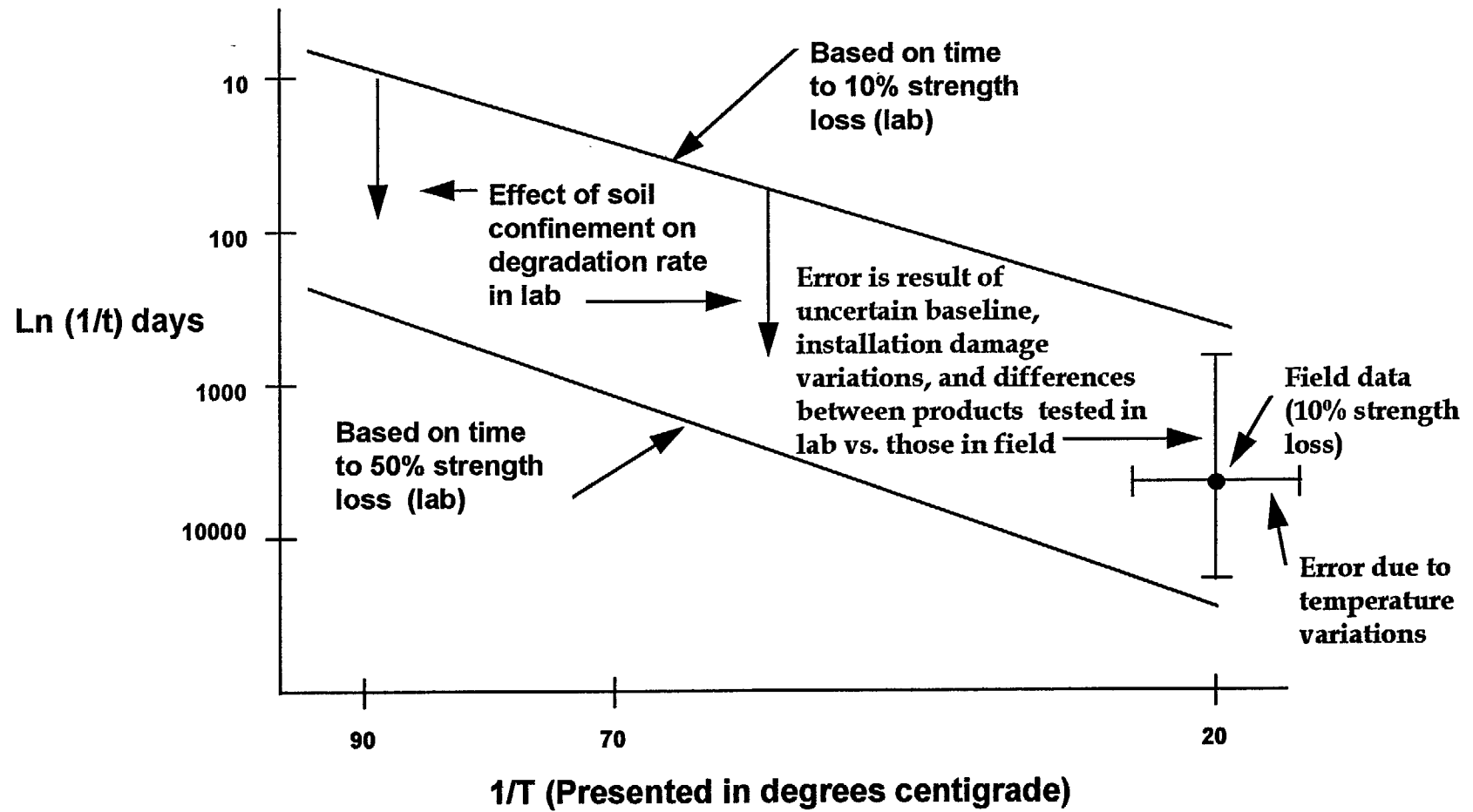


Figure 3. Conceptual Arrhenius plot showing likely relationship between laboratory degradation data and data from field exhumations.

data are reasonably linear. Based on the test program currently planned for Phase 3, the temperature range of interest is on the order of 10 °C to 110 °C (50 °F to 230 °F with the lowest test temperatures in relatively neutral conditions being on the order of 50 °C (122 °F) depending on the situation. Within this range, previous studies reported in the literature indicate that for the geosynthetic fibers, yarns, or related materials tested, only slight nonlinearity exists in their Arrhenius relationships, if any (McMahon, et. al., 1959; Gilroy, 1979; Wisse and Birkenfeld, 1982; Wisse, et. al., 1990; Van Langenhove, 1990; Risseeuw and Schmidt, 1990). However, most of this previous work used time to embrittlement (i.e., complete strength loss) rather than time to a given reduced strength which is the focus of the current study. The range of activation energies for oxidation and hydrolysis for PP, HDPE, and PET reported in the literature or which can be calculated based on data provided in the literature, which were based on some type of strength loss data (i.e., time to embrittlement or to some percent strength loss), are as provided in table 9.

Table 9. Previously published activation energies for oxidative and hydrolytic degradation of geosynthetic polymers.

Polymer	Degradation Process	Range of Activation Energy Measured (KJ/mole)	Reference
PP	Oxidation	50 to 75	Wisse and Berkenfeld, 1982; Wisse, et. al., 1990
HDPE	Oxidation	75 to 90	Grievesson, et. al., 1961; Gilroy, 1979
PET	Hydrolysis	60 to 90	McMahon, et. al., 1959; Jailloux, et. al, 1992; Anderson, et. al., 1992

1 KJ/mole = 0.24 KCal./mole

Extrapolation of laboratory data on the basis of temperature using the Arrhenius equation only provides a partial solution to the life prediction problem, as the relationship between the laboratory environment and the in situ soil environment has not been addressed. Extrapolation of high temperature laboratory test data to in situ temperatures only provides an estimation of material life in the laboratory environment at the in situ temperature. Other environmental factors must be addressed to realistically estimate the life of the geosynthetic in situ. The actual in-soil environment must be better defined as well to improve the accuracy of life predictions.

For oxidation testing, which is usually carried out with full exposure of the specimens to air in an oven with continuous replenishment of the air, these other factors include the oxygen content (in air, the oxygen content is approximately 21 percent, but in soil, the oxygen content may be as low

as 1 percent). Preliminary data relating the rate of oxidation in air to that in water (7 percent) based on laboratory studies in a gaseous environment indicates that lower oxygen content does significantly reduce the rate of oxidation (Van Langenhove, 1990; Wisse, et. al., 1990). Apparently, the air circulation velocity in the oven may also affect the measured life of the material tested. A study by Forsman (1964) indicates that substantially shorter oven lives are obtained in a forced air oven relative to a convection oven. Obviously, in soil, there is no air velocity. Furthermore, the ability of oxygen to diffuse through the soil to the geosynthetic to replenish the oxygen supply as the geosynthetic oxidizes may affect the rate of degradation. Based on recent studies of oxidation of mine tailings, oxygen diffusion rates could significantly affect oxygen availability as the soil becomes finer grained (Yanful, 1993). Measurement of oxygen levels in actual wall or slope backfill soils is needed to better define the in-soil environment regarding this issue. Finally, the total volume of oxygen in the soil in the vicinity of the geosynthetic is less than that in open air because the soil particles take up a portion of the volume (i.e., the oxygen is only in the pore spaces between the soil particles). These factors could cause polymer life regarding oxidation to be much greater than predicted using high temperature oven aging results and simple Arrhenius modeling using only temperature.

For hydrolysis testing, which usually carried out with full exposure of the specimens to water or other chemical solutions with vigorous stirring of the solution, these other factors include the volume of water available to the soil (water is contained only within the pores between soil particles), the ability of water to be replenished in the soil near the geosynthetic (soil permeability may restrict water replenishment near the geosynthetic), and possibly the stirring action in the laboratory test (water moves very slowly in typical backfill soils). There is no existing data which relates the laboratory hydrolysis immersion conditions to in-soil conditions. In dry climates, lack of saturation may also be a factor which reduces the rate of hydrolysis. Elevated pH levels are sometimes used to help accelerate the hydrolysis reaction in the laboratory. However, as yet there have been no models developed which account for the combined effects of pH and temperature, though some preliminary data is available (Jailloux, et. al., 1992; Anderson, et. al., 1992). These factors could cause polymer life regarding hydrolysis to be much greater than predicted using high temperature laboratory hydrolysis test results and simple Arrhenius modeling using only temperature.

The effect of aggressive soil conditions on the rate of degradation such as a high iron content soil for oxidation or a high pH soil for hydrolysis is not well known and adds additional complexity to developing life estimates. The effect of these particular soil conditions on polymer life will be investigated in the current research contract.

Preliminary experimental work will be conducted in the Phase 3 testing of Tasks D and E and compared with the results from Task B to begin to relate the laboratory environment to the in situ soil environment. The relationship between the laboratory environment and the in situ soil environment is conceptually shown in figure 3 in the form of an Arrhenius plot. Note that the field data obtained from Task B only provides one data point, which potentially has a great deal of uncertainty associated with it, to relate the laboratory experimental data to the real performance expected in situ. The problem is that the in situ data from Task B only represents one temperature. Laboratory studies at elevated temperatures and which are allowed to degrade to say, 50 percent strength loss, in which the laboratory environment is adjusted to better simulate

true in situ soil conditions, are needed to accurately relate the laboratory environment to the in situ environment. Therefore, the Task B results can only be used as a reality check for the laboratory simulations.

Obviously, reasonably accurate life prediction of geosynthetics for in situ conditions is currently not possible based on the available published data, and life prediction based on extrapolation of laboratory test data will likely produce excessively conservative results. Better long-term data is needed if reasonably accurate estimates of geosynthetic life are to be made and is hence one of the main reasons for performing Phase 3 of the current research contract.

Once the life prediction problem, considering all of the variables mentioned previously which affect life prediction, has been adequately tackled, must long-term (i.e., 2 to 5 years) laboratory aging studies be conducted every time the life of a new product is to be estimated, or can a single temperature short-term index test which has been “calibrated” based on the long-term testing be used to assess the potential long-term performance of a given product? The answer to this question will depend on the variation in the activation energy for various products as well as the variation from product to product regarding the effect various other environmental variables have on product life. This will be investigated, at least partially, in Phase 3 of Tasks D and E. A short-term test to assess the potential life of a given new product is certainly a desired goal of the Phase 3 testing.

7. SUMMARY OF SIGNIFICANT ACCOMPLISHMENTS AND LEARNING FROM THE STUDY TO DATE

Considering the information provided in this report, much has already been learned and accomplished through this project. This learning and accomplishment are summarized as follows:

- Based on the literature review conducted for this project and the experience of those involved in the project, the polymer and soil environmental factors which affect the geosynthetic degradation rate have been identified as well as tests which potentially could be used to assess and quantify many of these factors.
- The literature review has helped to identify the issues which must be addressed to develop life prediction models for geosynthetics, especially with regard to relating the laboratory environment to the in-soil environment.
- The study has created an environment which has promoted improved communication between the industry, in particular their polymer experts and suppliers, and the portion of the engineering community which routinely uses geosynthetics. This has allowed the geosynthetic industry to better understand the needs of those who use geosynthetics regarding durability issues, and has helped geosynthetic users to know what questions to ask and what can be realistically expected from the industry. A team spirit between the industry and the geosynthetic users has begun to develop from this which was not present before the study began.
- Variations in the test protocols used to chemically characterize polymers, with specific reference to geosynthetics, which could significantly affect test results or make test results difficult to interpret have been identified and corrections to the protocols made (e.g., the OIT test). These protocol variations help to explain why comparison and use of data provided in the literature is difficult.
- From the effort to better define the test protocols, preliminary standardized test and QC/QA procedures to be applied industry wide have been developed and will be further evaluated during the remainder of the research program. This has set into motion the ability of the industry to perform the tests which will likely be recommended for future evaluation of geosynthetic durability, and the ability to develop a consensus on how those tests should be performed and quality control maintained.
- A preliminary assessment has been made as to which chemical characterization tests (see table 2 for the types of tests available) appear most useful to at least roughly characterize the potential durability of a geosynthetic product with a minimal amount of effort. Initially, these potential tests appear more promising for durability characterization of PET products than for PP and HDPE products.

- The issues which must be addressed to chemically track geosynthetic polymer degradation as well as the best tests to perform this tracking, and their limitations, have been preliminary identified, and attempts are being made to determine their usefulness.
- A preliminary test protocol for long-term durability performance testing of geosynthetics (i.e., oven aging and immersion testing) has been developed.
- The long-term testing program needed to develop a model for life prediction of geosynthetics and to establish a practical protocol to assess the durability of all geosynthetic products has been set into motion.
- A separate but parallel effort by Transportation Research Board (TRB) committee A2K07 has already been initiated to develop the frame work necessary for rapid implementation of the results of this study. This effort has also helped to identify just where the real needs in terms of geosynthetic durability knowledge are so that once the study is completed, a coherent recommended practice to determine the long-term strength of geosynthetics can be developed.
- Based on the initial data and data provided in the literature, it appears that most geosynthetic products will have the durability needed for critical reinforcement applications, especially in relatively neutral environments. In severe environmental conditions, preliminary results appear to indicate a fairly wide range of degradation rates. However, there is much to be learned yet before such preliminary predictions can become final conclusions.

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