TALLINN UNIVERSITY OF TECHNOLOGY
SCHOOL OF ENGINEERING
Department of Materials and Environmental Technology

ACCELERATED CHEMICAL DEGRADATION OF GEOGRIDS IN VIEW OF CHEMICAL RISKS IN ESTONIAN PAVEMENT CONSTRUCTIONS

GEOVÕRKUDE KIIRENDATUD KEEMILINE VANANDAMINE ARVESTADES KEEMILISI RISKE EESTI TEEDEEHITUSES

MASTER THESIS

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Tallinn 2021

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THESIS TASK

Student: Diane Ogechi Tobias, 194267 KVEM

Study programme: Technology of Wood, Plastics and Textile (KVEM)

Main speciality: Technology of Plastics

Supervisor(s): Early-stage researcher, Merit Rikko. Professor, Andres Krumme (Head of Laboratory).

Thesis topic:

In English: Accelerated chemical degradation of geogrids in view of chemical risks in Estonian pavement constructions

Eesti keel: Geovõrkude kiirendatud keemiline vanandamine arvestades keemilisi riske Eesti teedeehituses

Thesis main objectives:

1. Chemical degradation of geogrids based on their polymeric composition in aqueous environments of potentially harmful chemicals in Estonian road constructions at elevated temperatures
2. Measure the tensile strength of geogrids after their removal from the aging environment
3. Plot the degradation curve at service temperature, to predict the service life of the geogrids in the chemical risk environments of Estonian road constructions

Thesis tasks and time schedule:

No	Task description	Deadline
1.	Literature overview	10th May, 2020
2.	Experimental work	1st Mar, 2021
3.	Analysis of results	1st Apr, 2021

Language: English **Deadline for submission of thesis:** 24th May 2021

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PREFACE

This research was based on contract and funding of Estonian Transport Administration and experimental work was carried out in the Laboratory of Wood, Plastic and Textiles Technology, in Tallinn University of Technology.

The author wishes to thank everyone who aided in the completion of this master’s program. Particularly, the staff members of the Department of Wood, Plastics, and Textile in Tallinn University of Technology.

The master thesis is aimed at assessing the impact of the exposure of geogrids to chemical risks, that are common in Estonian pavement constructions. Geogrids of different polymeric composition were degraded in aqueous environments at elevated temperatures according to accelerated chemical degradation methodology, that was developed in Laboratory of Plastic Technology. Geogrids of polyethylene terephthalate (PET), polypropylene (PP), and high-density polyethylene (HDPE)were tested.

From the results, it can be concluded that the impact on PET geogrid was remarkable, but for HDPE and PP geogrids the time in the aging environment must be extended to see the impact of the potentially harmful chemicals in Estonian road constructions.

Keywords: chemical degradation, geogrids, degradation curve, reinforcement, master thesis.

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List of abbreviations and symbols

°C – Celsius

CEG - Carboxyl End Groups

COOH – Carboxyl

CFB-Circulating Fluidised bed

CMD- Counter machine direction

F_c - mean maximum tensile load of the control specimens

F_e - mean maximum tensile load of the exposed specimens

fPP - Flexible Polypropylene

GCL - Geosynthetics Clay Liner

HDPE-High density Polyethylene

LDPE-Low density Polyethylene

mm – millimeters

nm – nanometres

OH – Hydroxyl

PE – Polyethylene

PET – Polyethylene Terephthalate

PC – Polycarbonate

PP – Polypropylene

PS – Polystyrene

PVC - Polyvinyl chloride

RH – Relative Humidity

R_F - percentage retained tensile strength

UV – Ultraviolet

MD – Machine direction

CMD – Cross machine direction

INTRODUCTION

Geosynthetics are defined as polymeric materials products which are mainly used with geotechnical material for civil engineering projects and structures.

The first modern use of reinforcement was in 1926 when a heavy cotton fabric was placed on a primed soil subgrade and hot asphalt and a thin layer of sand was applied to the fabric. This was in South Carolina, USA. It lasted until 1935 when the fabric started to degrade. The result showed the use of these cotton fabric reduced cracking, ravelling, and other road failures (Koerner, 2005)

In the past 3 decades, geosynthetics have been increasingly used in geotechnical and environmental engineering. These materials have helped designers and engineers to solve several types of engineering problems where the use of conventional construction materials would be restricted or considerably more expensive(Koerner, 2005). They are also designed to eradicate the issue of biodegradation that is common to conventional civil engineering materials over time. The resultant is a more durable construction. Geosynthetics require minimal effort to retain their original strength after mechanical interference, or after the effect of weathering and other sources of degradation.

The durability of most geosynthetics is estimated to be about 100 years of lifetime (Cassidy et al., 1992). However, geosynthetics have only existed for a few decades, and are now used in road pavement construction in Estonia. For this, it is pertinent to assess the chemical risks of geosynthetics used in pavement constructions in Estonia. To do this, all factors peculiar to Estonia that could pose a potential chemical risk have been put into consideration.

In 2008, (Y. Hsuan et al., 2004) were able to carry out a study to ascertain the long-term performance of geosynthetics. The study took into consideration all environments for degradation, which includes weathering, mechanical degradation, and biological degradation and its effect on the aging of geosynthetics. It also highlights the role of antioxidants, plasticizers in the degradation of geosynthetics.

This research has been carried out to evaluate the chemical risks of geosynthetics used in pavement constructions in Estonia for these, geogrids have been used.
With these results, it is possible to make long-time predictions on the aging of geogrids when used in conditions in Estonia.

1. GEOSYNTHETICS

In this chapter, the functions, types of geosynthetics, and common polymers used in geosynthetics manufacturing will be discussed. The methods of degradation of geosynthetics will also be assessed.

1.1 Function of geosynthetics

Geosynthetics have been used to fulfil several functions. There are 5 major functions of geosynthetics according to (Koerner, 2016), they include the following:

i. **Reinforcement:** Geosynthetics are used for soil reinforcement structure, basal reinforcement to support the soil reinforcement structure. Geogrids and geotextile are the principal geosynthetics used for reinforcement. They help in retaining structures such as pavement, walls slope, etc. They reinforce the soil by acting within the soil mass to increase strength, compared to unreinforced soil. They help to stiffen a structure (Koerner, 2016). In the use of geogrids for road reinforcement or road stabilization, the parameters to take into consideration are that the geogrids have a stiff aperture, the ability to maintain high tensile strength at low elongation, and long-time durability. (Šiukščius et al., 2019)

ii. **Filtration:** Geosynthetics are used for filtration in almost every hydraulic structure. Geotextiles are the primarily used geosynthetics for filtration. They help to prevent clogging of hydraulic passages, to ensure constant flow throughout the lifetime of such structures. They can also be used in leachate collection systems, retaining walls, and shoreline structures, also in slopes (Heibaum, 2016).

iii. **Drainage:** Geosynthetics can be used for drainage control. They are used to collect any sewage water coming from the other side of the embankment to avoid contamination on the structural fill. Geonets, geocomposites, nonwoven geotextiles are common geosynthetics used for drainage applications. They are used to build highway edge drains, they are also used as landfill cover, and leachate collection systems.

iv. **Separation:** Geosynthetics are used for separation between the in-situ soil and the imported soil this helps to prevent mixing that decreases the mechanical performance of the soil.

v. **Protection/Erosion control:** Geosynthetics act as erosion control blanket they provide protection to slopes at the top of the earth's crust hence, protecting it from erosion. Geocomposites are mainly used for erosion control. Geosynthetics used for erosion control are classified into 2 types: temporary erosion control materials e.g. erosion control blanket and permanent erosion control materials such as Turf reinforcement mat (B. M. Das, 2011).

1.2 Types of geosynthetics

Geosynthetics are majorly classified according to their functions. However, some geosynthetics can have more than one function. For example, while geogrids are majorly used in reinforcement, geotextiles and geocells can serve the same function as well.
There are different types of geosynthetics on the market, their classification is shown in figure 1.0.

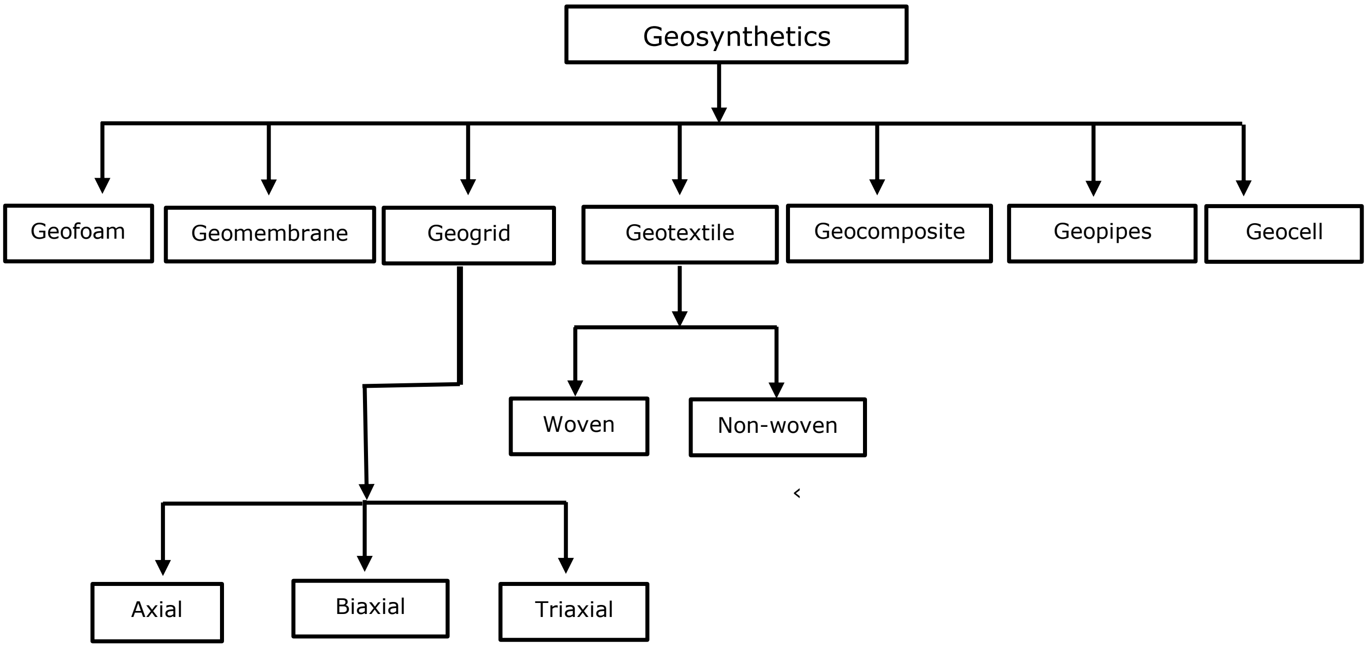


Figure 1.0. Classification of geosynthetics

1.2.1 Geotextile

Geotextile can also function in reinforcement. Geotextiles are permeable synthetic materials made of textile materials such as polypropylene (PP), polyethylene (PE), or polyester. Geotextiles may be woven, nonwoven, or knitted, depending on how they are prepared. (Koerner, 2016; Šiukščius et al., 2019). Geotextiles are mainly used in road and railway construction for separation, filtration, drainage, and soil reinforcement. They are sometimes useful for erosion control alongside river canals and coastal works. They work as a filtering media for drainage in an earthen dam, behind retaining walls, and in deep drainage trenches, they also have their application in agriculture, for mud control (Koerner, 2005).

1.2.2 Geocells

Geocells are geosynthetics used in soil reinforcement. They are geomaterials placed within cellular confinement(Sonam, 2012). They have a three-dimensional system, strong and lightweight, fabricated from ultrasonically welded high-density polyethylene (HDPE) strips that are expandable on-site to form a structured-like and expandable honeycomb-like structure as shown in figure 1.2. Geocells are filled with compact non-cohesive soils which are confined within the cellular walls. The composite forms a rigid to a semi-rigid structure. The depth of the geocells as well as the size of each cellular unit can vary as per design requirements. Some of the advantages of geocells for reinforcement and support on weak grounds. They reduce pavement sections for all road types. Gravity walls for earth retention and load support. Erosion control on embankment slopes and other slopes also help to channel water (B. Das, 2011).

1.2.3 Geogrids

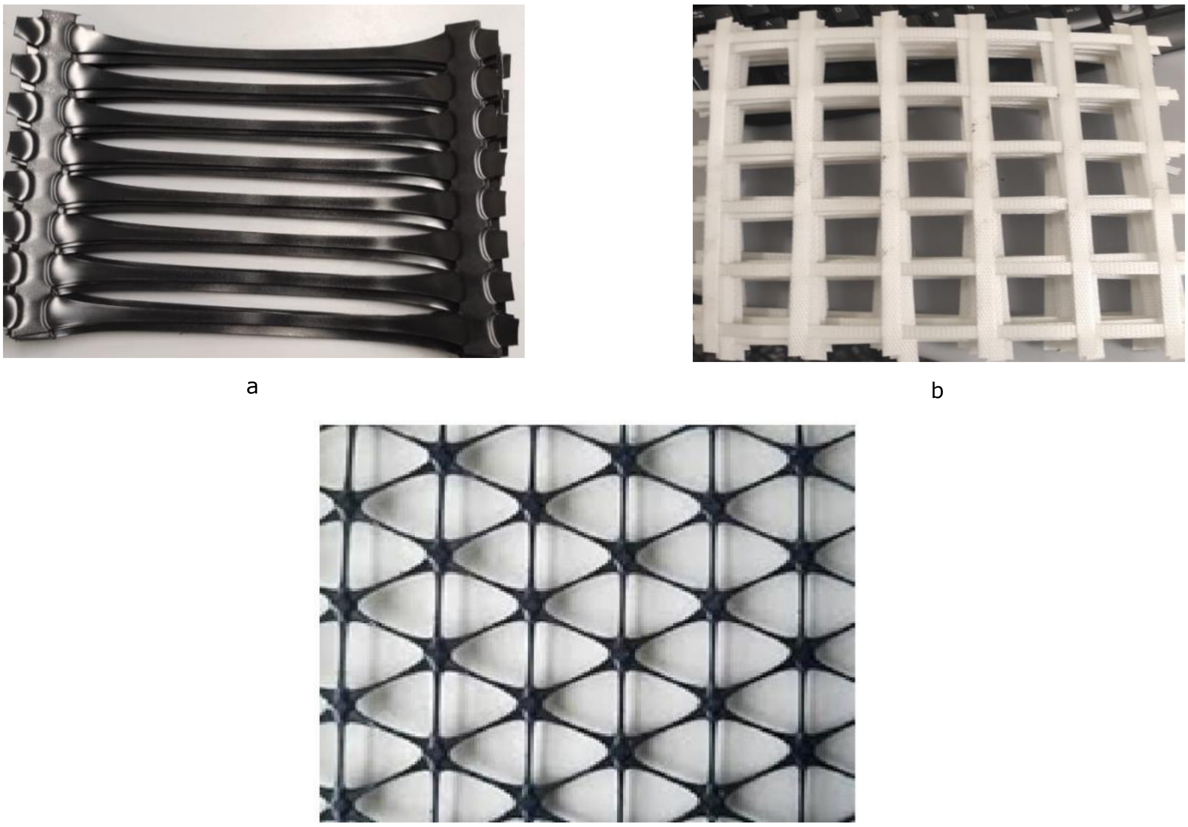
Geogrids are geosynthetics primarily used for reinforcement, they can be produced in three major ways(Jeon et al., 2005). They are produced by:

i. Extrusion
ii. Welded from strips of polymeric material.
iii. Weaving together polymer fibres.

Depending on the direction of the tensile strength, geogrids could be:

i. Uniaxial (axis in one direction),
ii. Biaxial (having its axis in both directions)
iii. Triaxial (axis that forms a triangular aperture)

Uniaxial geogrids have a high tensile strength in one direction as shown in figure 1.1a and are useful for the reinforcement of retaining walls, steep slopes, and road embankments, and for repairing landslides (C.A. Lawrence, 2014). Biaxial geogrids shown in figure 1.1b, have equal tensile strength in both directions and is useful for stabilizing roadways. It distributes loads over a larger area reducing pumping and shear failures while maximizing the load-bearing capacity of the subgrade. Triaxial geogrids, as shown in figure 1.1c, have high radial stiffness across their full 360 degrees, so they have a tensile strength in more than two ortho-direction(Yang, 2012).



a

b

c

Figure 1.1. Types of geogrid: a – Uniaxial geogrid, b-biaxial geogrid and c - triaxial geogrids(Yang, 2012)

Geogrids acts as reinforcement in several ways:

a. They provide lateral confinement by limiting horizontal movement of aggregate and by interlocking, this thus increases stiffness. They also decrease vertical stress applied to subgrade with a corresponding increase in horizontal stress.
b. Geogrids can bolster reinforcement in road pavement by increasing the distribution angle below rails.
c. Geogrids also creates a loading capacity by providing an upward vertical force that provides support to the load applied. This decreases the stress applied to subgrade.

Other types of geosynthetics include

- Geomembranes
- Geocomposites
- Geonets
- Geofoams

Geotextile-geonets Geotextile-geogrids Geomembrane-geotextile

Geocell Drainage geocomposite Composite drain

Geocomposite for planar drainage Glass filament reinforced geocomposite Erosion control geocomposite

Figure 1.2 Various forms of geosynthetics, (Frank K.K., 2004).

Our study focuses on the use of geogrids on paved roads in Estonia. Geogrids are commonly used in the construction of road pavement in Estonia. They have reportedly shown to minimize rutting at high deformation of the pavement surface (Šiukščius et al., 2019). The aim was to study reinforcement geosynthetics used in Estonia pavement construction. Geogrids are the most common of this type used in Estonia, this will be studied based on their different polymer composition. The experimentation involves subjecting geogrids to accelerated chemical degradation, to ascertain their long-term durability when placed in conditions of Estonia.

1.3 Polymers of geosynthetics

The most used polymers in geosynthetic materials production are Polyethylene (PE), Polypropylene (PP), Polyvinyl chloride (PVC), and Polyethylene terephthalate (PET). The characteristics of a geosynthetic depend largely on the parent polymer used, and these determine the application of the geosynthetic as well. Morphology and the types of addictive also determine the properties (Y. Hsuan et al., 2004). Table 1.0 below illustrate common polymers and what geomaterials they are used for.

Table 1.0 Common polymer used for geosynthetics (Y. Hsuan et al., 2004)

Geosynthetics	**Polymer used**
Geogrid	HPDE, PET, PP
Geotextile	LDPE, PET
Geomembrane	PP, PVC
Geopipe	HDPE

- **Polyethylene (PE)** is believed to be the most widely used polymer, it is a polyolefin belonging to the hydrocarbon group. This polymer contains carbon and hydrogen atoms only. They are linear copolymers are produced during polymerization under catalytic conditions with a low temperature and pressure. (Y. Hsuan et al., 2004). With the co-monomers becoming the side chain attaching to the ethylene chain. The types of co-monomers are ɑ-olefins including pentene, hexene, and octene. The type, amount, and distribution of the co-monomers have a strong impact on the chemical and mechanical properties of the resin especially altering the density of the polymers. Thus, leading to PE of different densities such as high-density polyethylene (HDPE), Low-density polyethylene (LLDE), and medium density polyethylene (MDPE). MDPE is best suited for geomembranes and is used as landfill liners. When choosing the right PE to be used for geosynthetic production, the function of the geosynthetic should influence the type of PE to be used.

- **Polyvinyl chloride (PVC)** is produced by free-radical polymerization in suspension. PVC products are usually rigid, firm, and strong thus suitable in the construction of building materials and finishes. Some of the items produced by PVC include pipes, windows, sidings, and door frames. They are highly polar in nature and can be blended with similar compounds. Adding plasticizers to PVC changes the nature of PVC from a rigid to a flexible material.

- **Polyethylene terephthalate (PET)** PET formation process involves condensation polymerization. Polymerization between terephthalic acid and ethylene glycol. The transition temperature of PET is relatively high, and this affects the properties of PET. The molecular weight, carboxyl end groups (CEG), crystallinity, content of copolymerized acids and alcohols, moisture content, soil water chemistry, orientation, and the product design also affect the properties of PET (Y. Hsuan et al., 2004).

- **Polypropylene (PP)** is the second most widely used polymer after PE. It is synthesized by catalytic polymerization that resembles that of PE. PP provides excellent flexibility, elongation, cold temperature resistance, long term UV stability, and advanced chemical resistance to geosynthetics. The unsupported styles of PP have highly flexible materials used in the manufacture of geomembrane and making complicated part easy to attach. PP can also be installed and welded in cold conditions (B. Das, 2011).

1.4 Degradation of geosynthetics

Polymer degradation can be described as any process that brings alteration in the property of a polymer (e.g., shape, colour and tensile strength) by undergoing biological, chemical or physical reactions resulting in bonds breakage with further chemical transformations (Carneiro et al., 2018).

Geosynthetics undergoes degradation in many ways. The three major ways of geosynthetics degradation are namely:

- Chemical degradation.
- Weathering
- Mechanical degradation

The strength of a geosynthetics reduces as the geosynthetics undergo degradation (Schroeder et al. 2001). The degradation of geosynthetics can be divided into three modes. Figure 1.3 describes each mode, and they are divided into three:

a) **Mode 1:** This mode of degradation results in an abrupt reduction in strength, then insignificant further reduction with time(Iso Tr 20432: 2007). This type of degradation is usually seen in installation. In this type of degradation, it is convenient to reduce the tensile strength by an appropriate time-independent reduction factor.

b) **Mode 2:** This is a gradual, though not necessarily constant but progressive reduction in strength. An example of mode 2 degradation is creep damage(Y. Hsuan et al., 2004). The tensile strength will also be reduced by a time-dependent reduction factor.

c) **Mode 3:** This this type of degradation, there is no reduction in strength for a long period; after a certain period, rapid degradation starts occurring. This is common in geosynthetics that have be coated with additives. For Mode 3, it is not appropriate to apply a reduction factor to the tensile strength, however, the service lifetime will be restricted.

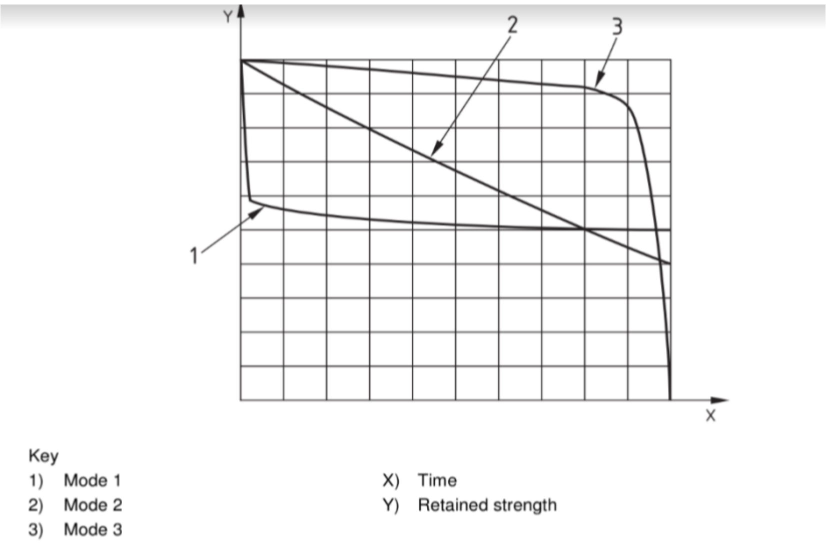


Figure 1.3 Retained strength plotted against time for the three modes of degradation (EN ISO TR 20432:2006)

1.4.1 Chemical degradation

Hydrolysis and oxidation are the primary mechanism for the chemical degradation. The process of oxidation and hydrolysis in geosynthetics are described in more detail in the following subchapters.

1.4.1.1 Oxidation

Oxidation is predominant in acidic environment from a pH below 6.5. The mechanism of oxidation of geosynthetics follows that of the polymer the geosynthetic is made of. It begins with initiation, propagation, chain branching, and termination reactions as seen in figure 1.4. Initiation could be triggered by radiation, temperature, a contaminant, or a catalyst ion. (Cassidy et al., 1992).

Chain initiation **Chain termination** **Chain branching**

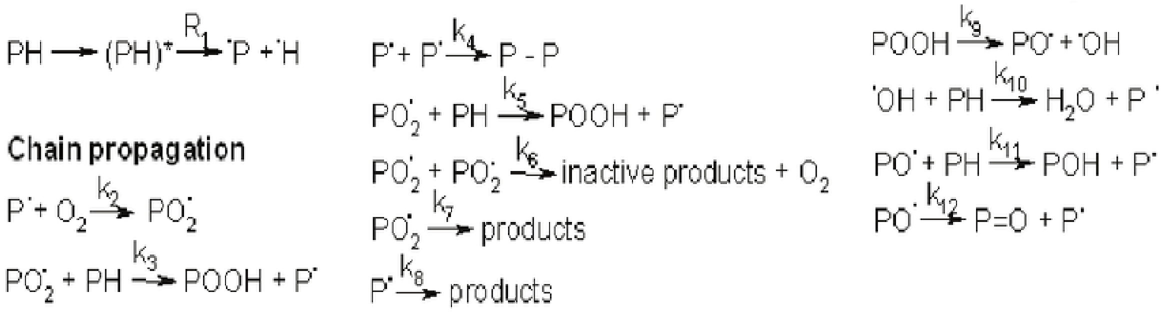


Figure 1.4 Mechanism of propagation of oxidation in polyolefin

When oxidation occurs in geosynthetics, the molecular weight of the polymer decreases, thus decreasing the strength of the geosynthetics (Bonaparte et al., 2002).

The nature of the polymer a geosynthetics is made from also plays a role in the oxidation of geosynthetics. For example, the amorphous nature of Polyethylene (PE) tends to retard the onset the oxidation. Furthermore, elevated temperature results in faster oxidation degradation at the surface (Bonaparte et al., 2002; Y. Hsuan et al., 2004).

Factors that affect oxidation of geosynthetics

Some of the factors that affects the rate of oxidation in polyolefins include:

- Polymer Morphology: The presence of tertiary hydrogen atom in polyolefin strongly encourages oxidation. The energy required for oxidation is largely determined by the number of tertiary hydrogen bonds present in a certain polymer. The tertiary hydrogen requires lower dissociation energy as compared to secondary and primary hydrogen atoms to form free radicals thus depending on the number of tertiary hydrogen atoms in polyolefin, they generate more free radicals than other polymers with primary and secondary atoms. PP will produce more free radicals than PE under the same conditions(Y. Hsuan et al., 2004).
- Orientation: The orientation of the polymer during manufacturing affects oxidation.
- Temperature: At a lower temperature, the oxygen consumption rate is relatively moderate, higher temperatures tends to speed up oxidation, thus decreasing the strength of the polymer.(Y. Hsuan et al., 2004)
- Oxygen Concentration: Environmental oxygen is essential to the oxidation degradation of geosynthetic materials.
- Antioxidant and UV stabilizer: The addition of antioxidant UV stabilizer as addictive minimizes degradation(Y. Hsuan et al., 2004).

1.4.1.2 Hydrolysis

PET can react with water and revert to compounds with carboxyl (− COOH) or hydroxide (− OH) end groups. This reverse reaction is the hydrolytic reaction which leads to a decrease in molecular weight.

The chemical and physical structure of the polymeric product affects PET hydrolysis. The presence of carboxyl end groups (CEG) in PET is the major factors that results in the decrease of the molecular weight of PET. Other factors that affect PET includes comonomers or other chemical modifications, the presence of additives and impurities may also affect the molecular weight, crystallinity and orientation also influences the rate of hydrolysis. The reaction is occurring mainly in the amorphous regions

The hydrolysis of PET can occur in 2 ways depending on the pH of the environment:

i. Inner hydrolysis, as the name implies, occurs mainly inside the PET product. Inner hydrolysis is largely affected by the presence of water molecules in the PET product. It can also occur in the presence of only vapor. The process is catalyzed by protons attacking the ester oxygen bond to yield ester alcohol as shown in figure 1.5. Thus, there is a simultaneous attack of water oxygen on the neighboured carbon atom forming an acid also. This reaction occurs at a pH < 9 and a hydrolysis reaction forms at the end of a polymer chain (Y. Hsuan et al., 2004). Inner hydrolysis breaks the chain of the polymer, leading to a decreased strength of the geosynthetics.

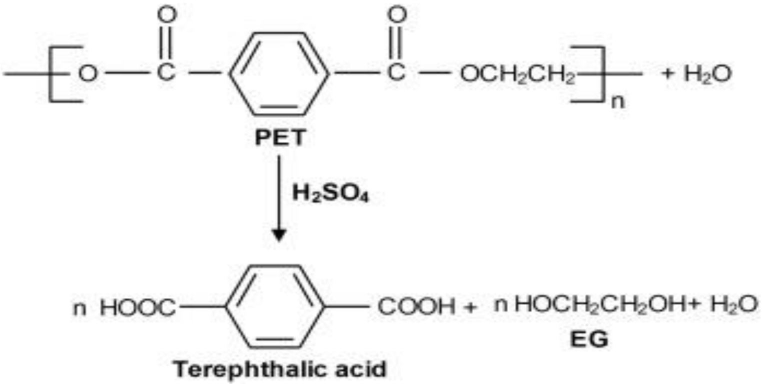


Figure 1.5 Inner hydrolysis (Y. Hsuan et al., 2004)

ii. The second kind of PET-hydrolysis called alkaline hydrolysis or surface hydrolysis is seen in figure 1.6. This reaction takes place at the surface. It happens when PET-product gets in contact with alkaline liquids. Hydroxyl- anions attack PET on the surface. This results in holes on the surface of the polymer and the geosynthetic product. This reaction occurs at a pH > 9 (Kanelli et al., 2015).

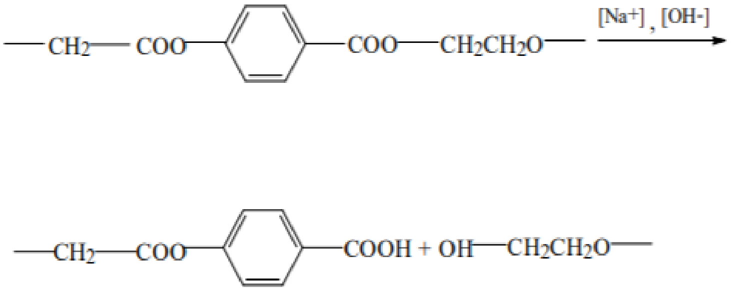


Figure 1.6 Surface hydrolysis (Y. Hsuan et al., 2004)

1.4.2 Weathering

Another important factor that affects the degradation of geosynthetics is the exposure geosynthetics to ultraviolet (UV) light. All polymers are susceptible to UV degradation when exposed to sunlight, but the degree vary from polymer to polymer. This is because UV light possesses sufficient energy to break chemical bonds within the polymers. Polymers with the shorter wavelengths are more prone to degradation than those with longer wavelength (Y. Hsuan et al., 2004). The table 1.1 below shows the wavelengths or wavelength ranges that cause photodegradation of the four types of polymers commonly used in geosynthetics.

Table 1.1 Polymers and their wavelength (Hsuan, G., *et al*. 2020)

Polymer	**Wavelength (nm)**
PE	330 – 360
PP	335 – 360
PVC	320
PET	325

1.4.3 Mechanical degradation

Mechanical degradation includes damage caused during installation and creep damage. They include:

a) Installation damage

The damage caused during installation of geosynthetics does not in actuality affects lifetime prediction. This is because the effect is a single, one time reduction in strength at the stage of installation. It is important however, to take this into account as it also a factor. Mechanical degradation caused by installation is a mode 1 degradation (Y. G. Hsuan & Koerner, 2001).

b) Creep

Creep can be described as the deformation of a geosynthetic under a constant load. The creep behaviour of a geosynthetic, is affected by various factors such as polymer type, temperature, duration of testing, humidity, manufacturing method, and percentage of tensile strength applied. Geosynthetics of polyolefins such as PP and PE are more susceptible to creep behaviour than polyester. Geogrids manufactured from PP or PP are more likely to exhibit creep behaviour. (Y. Hsuan et al., 2004; Naughton & Kempton, 2006).

1.4.4 Biological degradation

Geosynthetics are rarely affected by biological degradation, this is due to their high molecular weight. As such, they are rarely affected by bacteria and fungi. Geosynthetics reinforcement also possess high tensile strength, as a result, they are rarely damaged by rats, rabbit or other burrowing animals(B. Das, 2011). The use of additives can sometimes expose geosynthetics to a more rapid biological degradation if the additives are susceptible to these types of degradation, however, this degradation is still negligible. (Y. G. Hsuan & Koerner, 2001).

2 CHEMICAL RISKS IN ESTONIAN PAVEMENT CONSTRUCTIONS

Estonia is located in the Baltics, there are certain risk factors peculiar to Estonia that could result in possible chemical degradation of pavement constructions made with geosynthetics. Here are three possible chemical risks in Estonian pavement constructions:

i. **Sodium Salt (NaCl):** Sodium salt are used in road maintenance. They are applied as de-icing in winter season to enable vehicle stability while moving, they are also used as dust suppressant in summer. Road pavement are fed with huge amounts of salt in the winter season which are usually washed away by rain or water (Denby et al., 2016). Sodium salts are also believed to prevent biological degradation of many materials however, they could pose a chemical risk for pavement construction, because they are used throughout the year in Estonia pavement.

ii. **Bog water:** Bog water is acidic in nature. It is estimated that one-fifth of Estonia drylands is covered with bogs of peat (Orru & Orru, 2008). Thus, comparing the risk of bog water to the chemical aging of geogrids used in pavement construction is necessary. Bog waters are found in peatlands, peats are formed from the accumulation of decomposed organic matter from the plant detritus, they are a result of anaerobic respiration. They are usually found in cool climate such as Northern Europe and in Canada. Peat bog drainage water is coloured and contains lots of nutrient. Due to the presence of humic acid, they are believed to be potential factors for degradation (Kuokkanen et al., 2015). In Estonia, soft peaty soil exist and they have been gradually reinforced with geogrids to strengthened the soil and to prevent the soil from cracks potholes and bumps (Šiukščius et al., 2019), however, bogs of peat could pose a threat by causing degradation of geogrids used in Estonia road construction.

iii. **Circulating fluidized bed (CFB) fly ash**: CFB can pose a chemical risk to geosynthetics used for pavement construction as their leachate is highly alkaline. CFB is a combustion technology used to generate electricity by burning petroleum coke. Residual ash from circulating fluidized bed is gotten after electric manufacturing (Lin et al., 2017). They are usually disposed as land fill. The major component of fly ash is Calcium oxide, Sulphur oxide and Silicon oxide. They are used for landfills and sometimes used for cement constructions (Lin et al., 2017; Tumolva et al., 2014). In Estonia, fly CFB is produced by burning oil shale. According to Estonia statistics report, oil shale production was 15.9 million tonnes in 2019.

The ash from this production is used to for construction, sometimes used as land fill. Leachate from CFB fly ash is alkaline and can result in degradation of geosynthetics by hydrolysis (Tumolva et al., 2014).

3 EXPERIMENTALS

The chapter focuses on the materials and methods used for the accelerated degradation of geogrids. Methodology of accelerated chemical degradation was developed in the Laboratory of Polymers and Textile technology of TalTech and is based on the following standards:

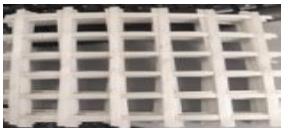
- ISO/TR 20432 *Guidelines to the determination of long-term strength of geosynthetics for soil reinforcement*
- EN ISO 12226 *Geosynthetics — General tests for evaluation following durability testing*
- ISO/TR 12960 *Geotextiles and geotextile-related products – Screening test method for determining the resistance to liquids*
- EN ISO 9862 *Geosynthetics - Sampling and preparation of test specimens*
- EN ISO 10319 *Geosynthetics - wide-width tensile test*

3.1 Materials, environment and apparatus

3.1.1 Materials

Table 1.2 shows detailed information about geogrids, which were studied, and all the environments used.

Table 1.2 Technical information on geogrid used during experiment

Polymer	**Axis of geogrids /Production method**	**Product name**	**Producer**	**Dimension of the specimen**	**Photo of geogrid**
PET coated with PVC	Biaxial/ Woven	Armostab AR2P. 40/40	MIAKOM.	20 x 22 cm	
PP	Biaxial/ Welding	Secugrid® Q (PP) 40/40	NAUE GmbH and Co.	21 x 21 cm	
HDPE	Uniaxial/ Extrusion	RE 520	Tensar Technical.	19 x 25 cm	

3.1.2 Environment

Geogrids were chemically degraded in the following environments:

a) Salt: To study the effect of NaCl, 36.5% (by weight) saturated aqueous solution with NaCl was prepared. Aqueous sodium chloride is neutral in pH.

b) Bog water is acidic and was gathered by digging at least three 80 cm deep holes to fetch out water from Pääsküla bog as seen in figure 1.7. The water is not diluted and there are not any proportions or is there a special storage needed for bog water, rather they are kept in 5L cans.

c) Leachate of CFB fly ash was prepared in the weight ratio of 1:10 ash to water. The solution was prepared in 20L plastic bucket, and stirred continuously for about 3 minutes, allowed to settle for at least an hour, after which the liquid part was used, and the residual ash was discarded. The pH of the leachate was recorded, the leachate of CFB ash is highly alkaline.

Figure 1.7 Bog water from Pääsküla raba matkarada

3.1.3 Apparatus

Our experiment implored the use of the following apparatus (Figure 1.8):

i. Scissors, 26 cm (Fiskars® Power Arc)
ii. 20L plastic buckets
iii. Rotating rollers (E-10, ~ 50 Hz, Labor® Hungary)
iv. 4-Litres and 5-litres jars with Metal lids
v. Thermal oven, SNOL 120/300 (220 V ~ 50 Hz)
vi. Measuring scales (Max 15kg d=5g, Manufactured by GWB Mettler)

vii. Micro scale (capacity 1200g x 0.1g, A & D Company Limited, Japan)
viii. Micro pipette

ix. pH meter: The pH meter used was PeakTech P5305, which is a product of PeakTech Germany, they are manufactures of portable and stationary measuring instruments. The P5305 is a 2 in 1 pH meter as it can measure pH and temperature with single hand operations. It can measure the pH value of a liquid using the galvanic potential difference between acidic, neutral or basic aqueous liquids. It measures the temperature of an environment, displaying both readings of temperature and pH on LCD display with backlight illumination. The device is also waterproof.

x. Instron 5866 tensile testing machine: The Instron 5688 universal testing machine as seen in figure 1.9a is an electromechanical and hydraulic systems that are used to perform static testing, tensile compression, bend, peel, tear, and other mechanical tests. The test involves the use of tensile force to pull a material or specimen and notes its response to the stress being applied. This determines the strength of a material and is elongation. Tensile testing of specimens was Carried out using the Instron 5866 universal testing machine according to standard 10319 (P. Kosky et al., 2013). The specimens are placed in the tensile testing machine, then it is being pulled to obtain the tensile properties of the material. This results in a graph showing the stress/strain curve and how the specimen reacted to the forces being applied.

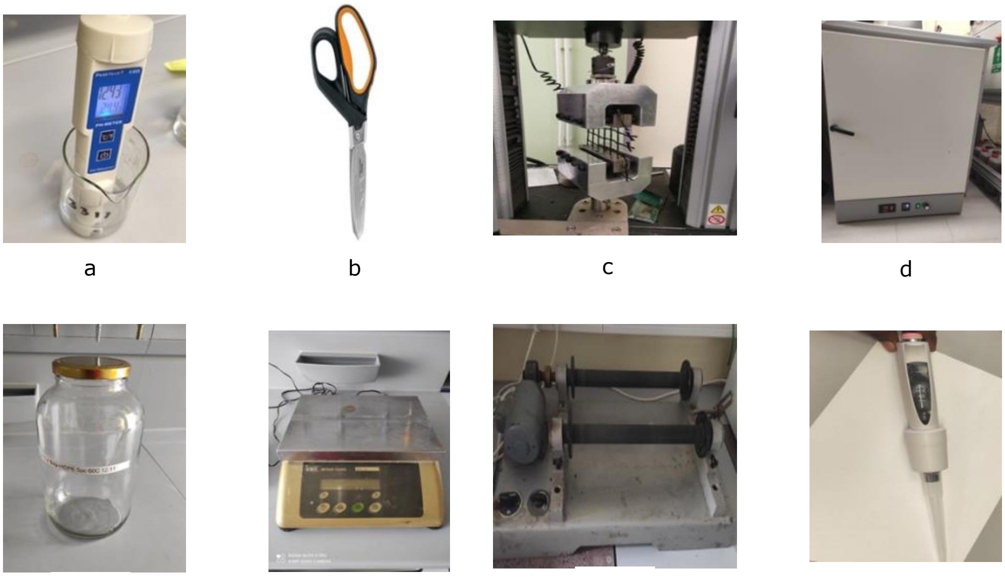

a b c d
e f g h

Figure 1.8. Apparatus used during experiment. a – ph meter, b-scissors, c – tensile testing machine, d-thermal oven, e – jar with metal lid, f – scale, g – rotating rollers, h – micropipette

3.2 Testing conditions

This sub-topic focuses on all environments the specimen has been subjected to. The preparation of the environment, temperature, and duration.

3.2.1 Testing Temperature

The geosynthetic service temperature is 20ºC and to accelerate the chemical degradation elevated temperatures were used. In all environments, aging was done at 60°C, 70°C, 80°C, and 90°C.

During homogenization of samples in the respective environment, care was taken to ensure that the glass vessels containing geogrids were not taken out of their testing temperature for a long time. So, each sample was immediately returned to the oven after mixing.

3.2.2 Testing duration

At each temperature, specimens were withdrawn at 5 different times. Table 1.3 shows the duration in the environment at all tested temperatures.

Table 1.3 Testing temperature and duration for accelerated degradation of geosynthetics

Takeout (time) / **Temperature**	**1**	**2**	**3**	**4**	**5**
90°C	3 days	6 days	13 days	20 days	27 days
80°C	7 days	14 days	21 days	28 days	32 days
70°C	14 days	24 days	36 days	42 days	55 days
60°C	24 days	36 days	55 days	68 days	83 days

3.3 Procedure

In this sub-chapter, specimen preparation, the quantity of specimens, and washing of the specimens after removal from the aging environment are described. All the tested geogrids were supplied in rolls. Care was taken when handling the materials, and observation was done to ensure that each roll selected was without damage and the wrapping was intact. Also, the machine direction was noted as this aided the direction of cutting.

The specimens were cut using Fiskars® Pro Power Arc scissor (26 cm). Before cutting specimens, the first two turns of the roll were excluded from sampling before cutting was done. Also at least 10cm was removed from each side of the roll. This was to prevent the effect of mechanical damage affecting materials. in accordance with the principles in this standard ISO 9862.

3.3.1 Quantity of specimen

The tensile strength of all the geogrids in all environments and at all temperatures, was tested in machine direction (MD). Only at 90°C, 5th takeout 5 specimens of PP and PET in all environments were tested in counter machine direction (CMD).

As 3 types of geogrids were tested in 3 environments, at 4 temperatures, at 5 different durations in the environment and each time 5 specimens were taken out at once. In MD was tested: 3 x 3 x 4 x 5 x 5 = 900 test specimens and5 x 3 = 15 reference specimens

In CMD 5 specimens of PP and PET geogrids were tested in 3 environments, at 90 °C after 27 days in the environment: 5 x 2 x 3 x 1 x 1 = 30 test specimen and5 x 2 = 10 reference specimens

3.3.2 Quantity of test liquid

The quantity of test liquid was done in line with the standard ISO 12960. It is required that the liquid shall be 30 times the weight of the specimens and they must be fully immersed in the solution. A 4-liter and 5-liter jars were used to achieve these results.

3.3.3 Positioning and conditions of specimen placement

The positioning was done according to the following procedure:

- The immersion of the specimen is done in line with the standard ISO 12960 it follows the principle of completely immersing the specimen in liquid.
- The weight of the solution of the testing environment was at least 30 times that of the specimen in a jar.
- On an average, 7, or 8 HDPE specimens are kept in a jar. About 9-10 PP specimens in one Jar and about 10 specimens of PET in a Jar. (Figure 1.9a).
- Daily, the jar was taken out of the thermal oven and the solution was manually mixed to maintain homogeneity of the material and that of the liquid and to ensure the interaction between the material and the liquid. (Figure 1.9b)
- The jar to be used, its cover were all be resistant to the test chemicals. The exact test temperature has been used.
- pH was measured weekly, and the environment was replaced when the pH changes were more than 0.3 units.


a b

Figure 1.9 Positioning and mixing of specimen in two stages (a) specimen in solution and (b) homogenization

3.3.4 Washing and drying of specimen

Washing of the specimens was done after removal from the environment, which was done according to schedule seen in the procedure below.

- Specimens are first thoroughly washed with tap water as shown in figure 2.0b.
- Each sample material is then subjected to a 0.01 M solution of sodium carbonate for at least 5 minutes.
- After washing with sodium carbonate, they were rinsed in water again, to ensure that the materials have been thoroughly cleaned.

The only exception in washing procedure was for PET geogrids in leachate of Ash water because calcium terephthalate salt crystals were formed on the specimens (Figure 2.0a). Then the washing procedure was as follows:

- Specimens are thoroughly washed with tap water as shown in figure 2.0b.
- After rinsing with tap water, specimen was rinsed with a 10% solution (by weight) of trisodiumnitrilotriacetate (NTA) in a hood for 5 minutes.
- After taken out, the sample is placed in a 3% (by weight) acetic acid solution for another 5 minutes.
- The specimen was rinsed with water again.


a b c

Figure 2.0 Washing and drying of sample. a – PET specimen in ash water, b – washing, c - drying

3.4 Characterization

3.4.1 pH measurement

The pH of the environment was measured when the jar containing geogrids and the aging environment was prepared and homogenised once a week. This was done by extracting 50 ml of the solution using a micropipette and to measure the environment pH. All changes in the pH of the environment were noted. The environment of sample placement is discarded and replaced when the change was greater than 0.3 units.

3.4.1 Tensile strength testing

Tensile testing was done in accordance with ISO 10319 to determine of mechanical properties of specimen. The test was done using 200 mm wide-width strip. In the tensile testing the Instron 5866 universal tensile machine is used. As shown in figure 2.1, a specimen is held across its entire width in a set of clamps of a tensile testing machine operated at a constant displacement speed, and a longitudinal force is applied to the test specimen until the specimen ruptures. For specimen of PP and PET, one of the ribs from the right side is left out of the clamps, when placing the specimen in the tensile testing machine. This is to accommodate pitch size, so it can fit perfectly into the clamp. However, this was not done for HPDE specimen. Also the excluded rib is cut along its horizontal axis either in 4 places (for PET) or in three places (for PP). The tensile properties of the test specimen are calculated from machine scales, an interfaced computer. Table 1.4 highlights the specifications of the testing machines during testing.

Table 1.4 Specification for tensile strength testing

Extension rate	20 mm/min
Atmospheric condition	23 ±3 ^{0}C, (15 ±5) % relative humidity (RH)
Gauge length	PP and PET – 10 cm, HDPE – 17 cm

Tensile strength test of PET Tensile strength test of PP

Figure 2.1 Tensile strength test of PET and PP geogrid

Determination of changes in mechanical properties

The standard BS EN 12226 recommends the formular for the calculation of the retained strength of samples. This was done by calculating the mean tensile load of specimen exposed to the environment represented by F_e and its standard deviation and those of the control sample F_c and its standard deviation.

The percentage retained tensile strength is therefore:

$R_f = F_e / F_c \, x \, 100$ (1)

Where:

R_f is the percentage retained strength

F_e is mean of samples

F_c is mean of control standard

The rate of change is determined by interpolating the retained strength against time, that is the exact time to the desired retained strength which is 90% retained strength.

The time at 90% retained strength is plotted against the inverse of the absolute temperature T_K in K (Arrhenius' formula). A straight line is obtained if Arrhenius' formula applies, and then extrapolation to service temperature can be done.

Since a straight line was obtained, the equation of a straight line was calculated.

Using $y = \log t_{90}$ (2) and

$x = 1/T_j$ (3),

as $y = y + b_a\,(x - \bar{x})$ (4)

Where,

$b_a = S_{xy}/S_{xx}$ (5)

$S_{xx} = \sum(x - \bar{x})^2$ (6)

$S_{yy} = \sum(y - \bar{y})^2$ (7)

$S_{xy} = \sum(x - \bar{x})\,(y - \bar{y})$ (8)

We calculated the lower confidence limit (LCL) of the line:

$$y = \bar{y} + b_a(x - \bar{x}) - t_{n-2}\sigma_0\sqrt{1 + {}^{1}/_{n} + (x - \bar{x}}\,)^2/\,S_{xx} \quad (9)$$

Where,

t_{n-2}= Student's t for n – 2 degrees of freedom and a stated probability.

n = the number of creep-rupture or Arrhenius points.

$\sigma_0 = \sqrt{[(S_{yy} - S_{xy^2}/S_{xx})/(n - 2)]}$ (10)

4 RESULTS AND DISCUSSION

4.1 Visual Examination

The specimen was examined using the eye according to the standard BS EN 12226-2012. Some of the changes that was observed include salt crystals in PET exposed to ash water conditions. PP shows no significant change when compared to the standard. PET showed the most significant change as there was discoloration from black to grey, shrinkage in sample size. PP showed slight peeling.

4.2 pH measurements

The result in table 1.5 shows PET in different environment of acidic, neutral, and alkaline environment.

Table 1.5 PET in aging environments at temperatures 60-90°C.

Specimen	Environment	pH range	Observation and pH fluctuations
PET	Bog Water	3.92-4.34	Significant and constant rise in pH of the environment, solution was continuously replaced at all temperatures.
	NaCl	7.10-7.51	pH of the environment was mostly stable in all temperature.
	Ash water	11.94-12.66	Significant drop in pH of the environment at all temperature mostly weekly, solution was replaced when the pH difference was remarkable.

The table 1.6 shows the result of PP in different environment of acidic, neutral, and alkaline environment. Most environment were quite stable in pH when PP was submerged into it. Compared to PET geogrids, PP geogrids are relatively inert/unaffected by exposure to chemical attack. The same result is was concluded by (Mathur et al., 1994) when studying the chemical effect of aging on geosynthetics.

Table 1.6 PP in aging environments at temperatures 60-90°C.

Specimen	Environment	pH range	Observation and pH fluctuations
PP	Bog Water	3.94 - 4.14	Mostly stable, but there is a rise in pH of the environment from time to time. Solution was replaced when the pH difference was remarkable
	NaCl	6.98 - 7.16	pH of the environment was mostly stable.
	Ash Water	12.4 - 12.62	pH of the environment was mostly stable.

The result of HDPE in different environment of acidic, neutral, and alkaline environment is largely like those PP in all environments at all temperature as seen in table 1.7.
HDPE is highly resistant to aging under alkaline, acidic, and neutral environment. This explains why the environment solutions were hardly replaced.

Table 1.7 HDPE in aging environments at temperatures 60-90ºC.

Specimen	Environment	pH range	General observation and pH fluctuations
HDPE	Bog Water	3.93 - 4.13	Mostly stable, but there is a rise in pH of the environment from time to time. Solution was replaced when the pH difference was remarkable
	NaCl	6.97 - 7.26	pH of the environment was stable.
	Ash Water	12.32 - 12.65	pH of the environment was mostly stable. Solution was replaced a few times when the pH difference was remarkable

4.3 Tensile testing

This sub-topic shows the results and discussion of tensile testing measurements. First it compares the result of the retained strength in both machine and cross machine directions, and then discusses the result of mechanical testing done in machine direction. Arrhenius plot is expressed and used to plot a degradation curve.

4.3.1 Geogrids (PP and PET) across MD and CMD

The table 1.8 compares the mean retained strength for PP and PET samples in MD direction and that in CMD for specimen at 90°C. The result from the table shows that there is no significant difference in strength both in machine and in counter-machine direction in all environments. For example, the retained strength of PET in bog water at MD direction is 100.9 while that in CMD direction is 94.5 This is also the manufacturers stated tensile strength of equal strength in both directions.

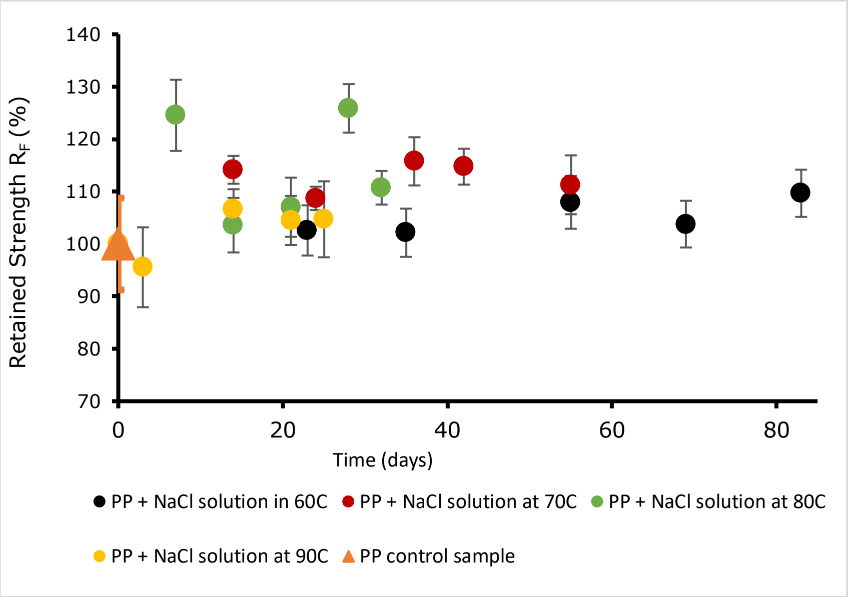
Table 1.8 Average retained strength of PP and PET geogrids in MD and CMD at 90C after 27days in the environment

Temperature	**Geogrid**	**Days in the environment**	**Environment**	**Material direction**	**Average retained Strength R_f (%)**	**Standard deviation (%)**
90°C	PET	27	Bog water	MD	100.9	8.445
				CMD	94.5	9.039
			NaCl	MD	103.2	8.379
				CMD	97.1	8.459
			Leachate of fly ash	MD	1.9	0.848
				CMD	1.7	0
	PP		Bog water	MD	108.2	3.109
				CMD	115.4	3.106
			NaCl	MD	103.6	8.286
				CMD	109.2	16.623
			Leachate of fly ash	MD	106	6.221
				CMD	111.6	5.089

4.3.2 NaCl aqueous solution environment

Sodium chloride has a pH of around 7. When geogrids specimen was submerged in conditions of neutral pH, the tensile strength shows that there was no significant loss in the strength of the specimen to that of the control specimen. A mostly upward R_f value is seen in PP, HDPE and PET in NaCl, as shown in figure 2.2. However, there is a decline in the R_f value of PET at 60°C to 92.0 % after an 83-day period, but no specimen had a decline below a 90% retained strength.

The decline could be attributed to the type of PET geogrid used as woven geogrids are weaker than extruded or welded geogrids, also geogrids with thicker surface have more chemical resistance than those with thinner surface (ISO DTR 18228-1, 2017) . This results also means that increasing the testing time could also lead to further possible degradation, as the specimen were in the environment the longest. A degradation curve could not be plotted, because no dependence occurred between temperature and duration in the environment (Y. G. Hsuan & Koerner, 2001).



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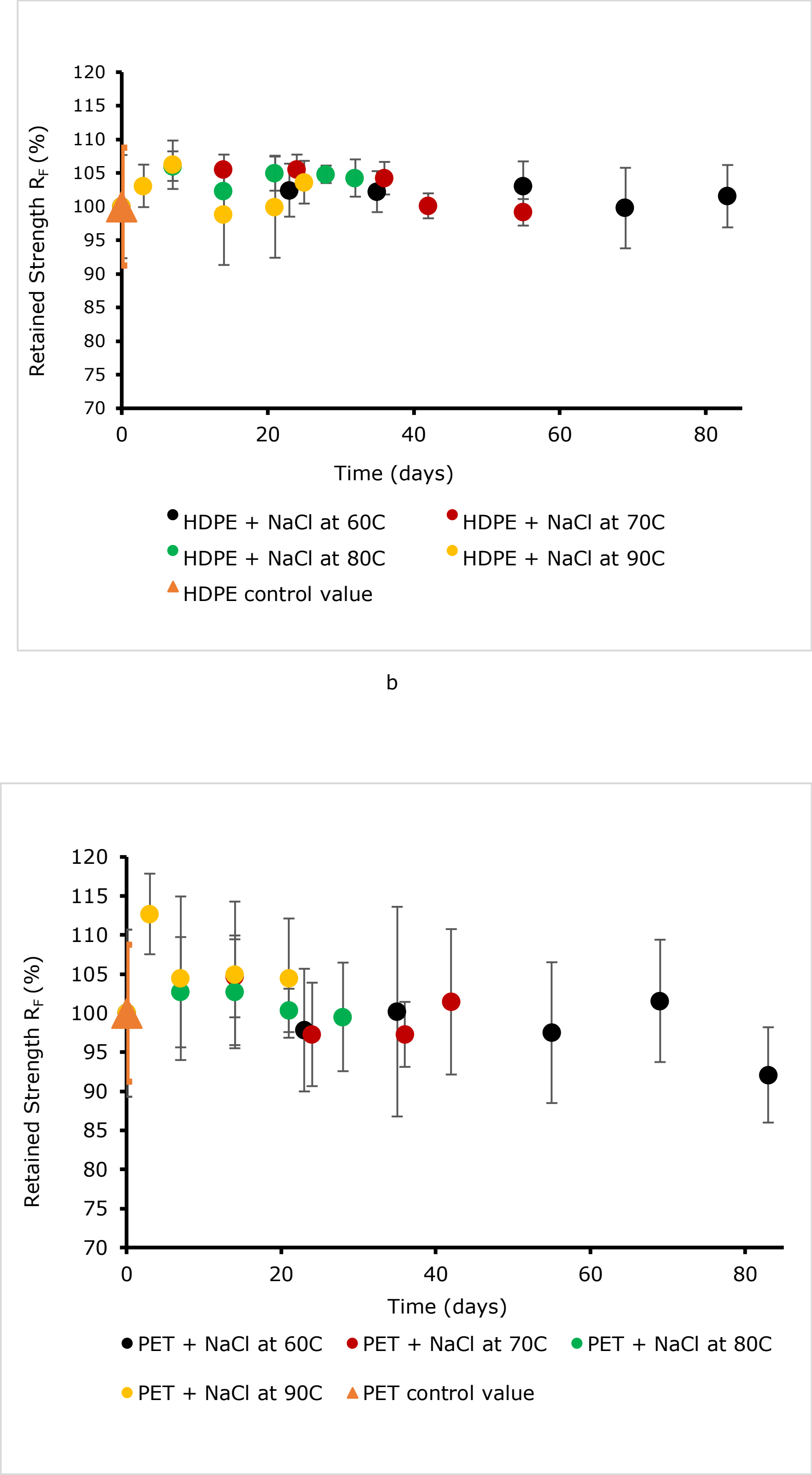


b

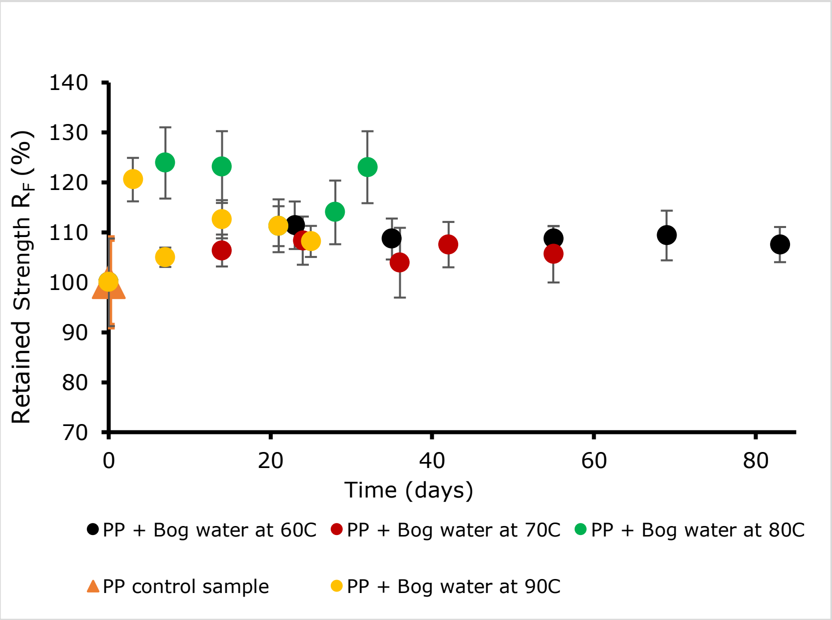
c

Figure 2.2 Retained strength in aqueous NaCl environment with different geogrid. a – PP, b – HDPE, c - PET

4.3.3 Bog water environment

The pH in bog water environment stayed in the range of 3.0 - 4.5. In bog water, geogrids specimen of PP and HDPE did not show any significant loss in their strength. In this environment, all specimen of PP and HDPE had a retained strength of above 100%, as shown in figure 2.3 (a-b). A degradation curve is not possible in this case as there is no dependence between temperature and duration in the environment.

PET on the other hand showed a slight drop in the retained strength value of some specimen at 60^{0}C, with the most significant decline at an 83-day period to an Rf value of 83%, the degradation is as a result of inner hydrolysis when PET comes in contact with an acid, this causes PET polymer to loss its molecular mass causing a decrease in strength. The loss in retained strength value of PET in bog water is higher than that of PET in NaCl. It appears that bog water environment causes more chemical aging to PET, compared to environment of NaCl environment, which can be related to the fact that PET has better chemical resistance to salts that to acidic environment (Mathur et al., 1994).



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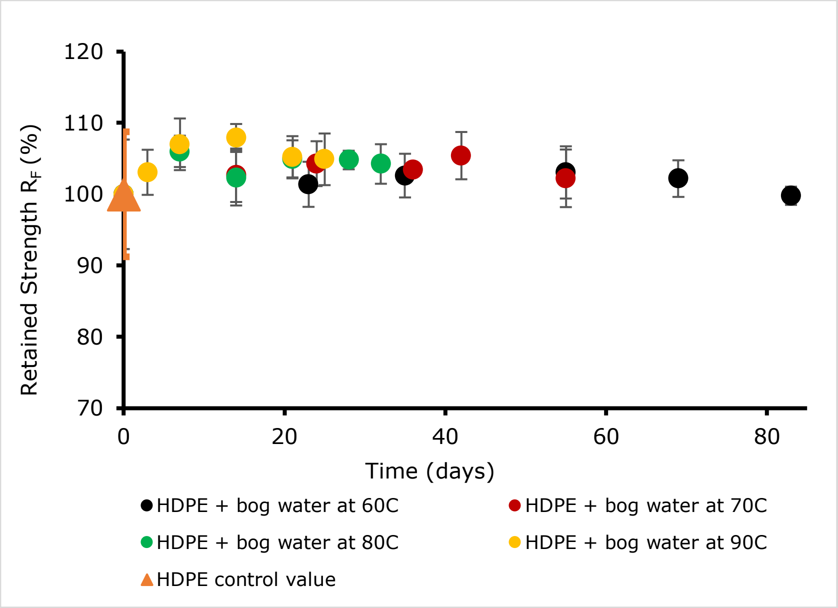

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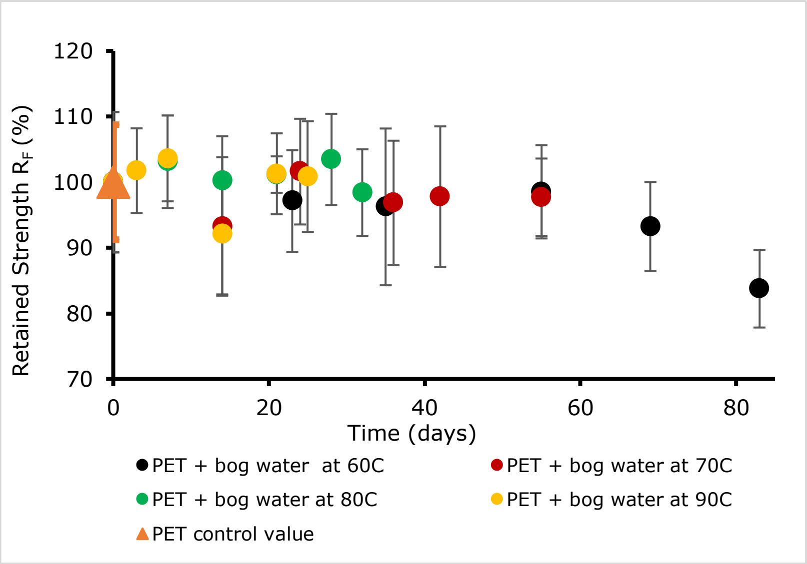


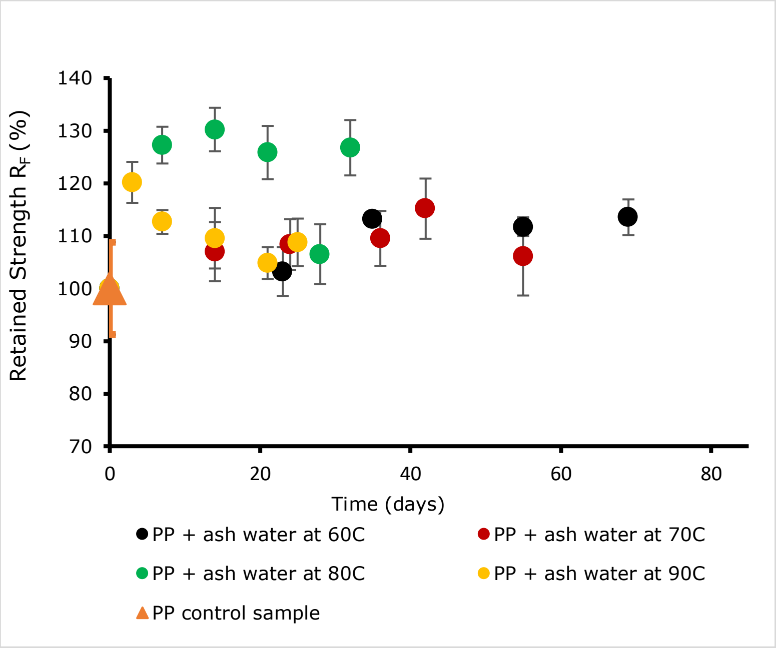
c

Figure 2.3 Retained strength in bog water environment with different geogrid. a – PP, b – HDPE, c – PET

4.3.4 Leachate of CFB fly ash environment

Ash leachate is highly alkaline, around a pH of over 12. The effect of strongly alkaline leachate on HDPE and PP geogrids was minimal as the average retained strength was stayed above 90% (Figure2.4 a-b). They show chemical resistance to alkaline environment. Since there is no dependence between temperature and degradation of PP and HDPE in this environment, a degradation curve cannot be plotted.

PET is highly susceptible to aging by alkaline environment. The result of chemical aging is seen in figure 2.4c as the value of the retained strength has declined significantly for all temperature. The mechanism of this decline is surface hydrolysis which occurs when PET polymers come in contact with a strong alkaline. Hydroxyl- anions attacks PET leading to the formation of terephthalate salt. This chemical degradation of PET causes the molecular weight of PET to decrease further causing a decrease in the retained strength. A temperature dependant degradation is seen to have taken place (Y. G. Hsuan & Koerner, 2001).



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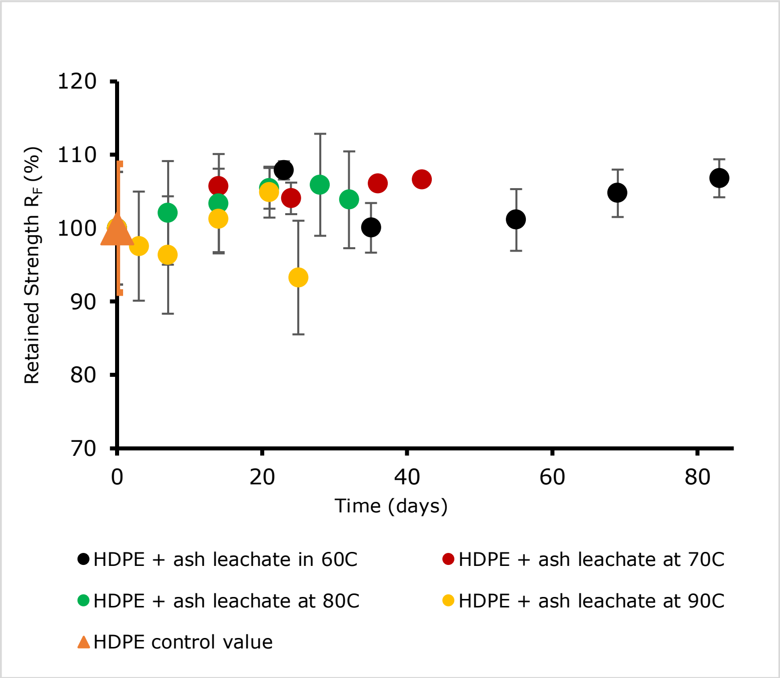

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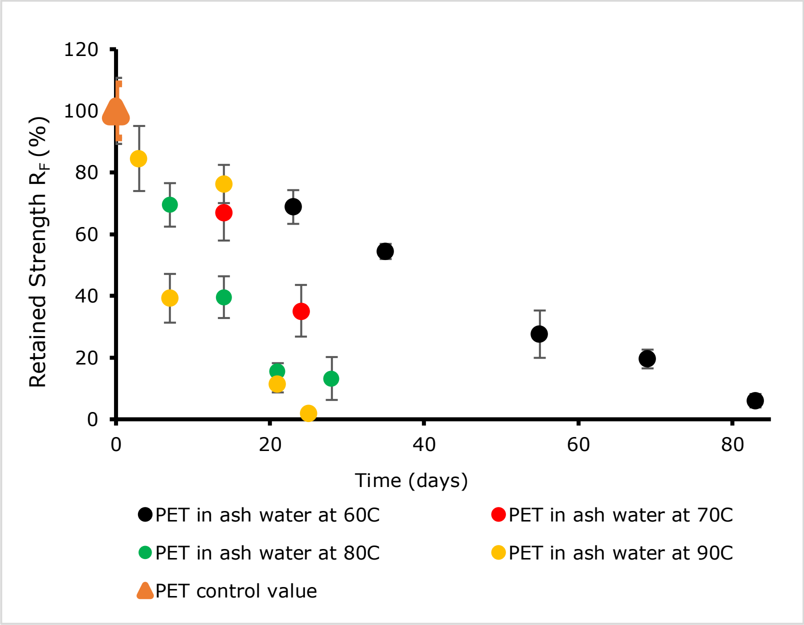

c

Figure 2.4 Retained strength in leachate of CFB fly ash environment with different geogrid. a – PP, b – HDPE, c – PET

For PET in ash leachate, a degradation curve could be plotted which was used to predict the durability of PET when exposed in chemical risk of ash leachate. To do this, the time needed to reach 90% retained strength was plotted against the inverse of the absolute temperature T_K in K using Arrhenius' formula (figure 2.5), it was then, extrapolated to the service temperature which is 20^{0}C. At 60°C, the specimen reached their 90% retained strength after 190 hours, specimen at 70^{O}C reached their 90% retained strength after 100 hours. For 80°C, specimen, they got to their 90% retained strength at 50 hours while specimen at 90°C reached their 90% at 19 hours. The lower confidence limit for PET at the service temperature of 20^{O}C specimen to reach 90% at 286 hours.

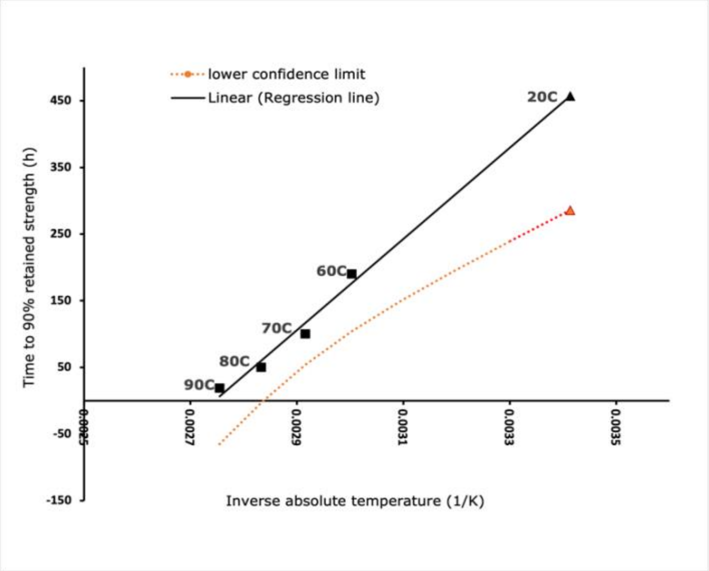

Figure 2.5 Arrhenius diagram showing inverse of absolute temperature to retained strength

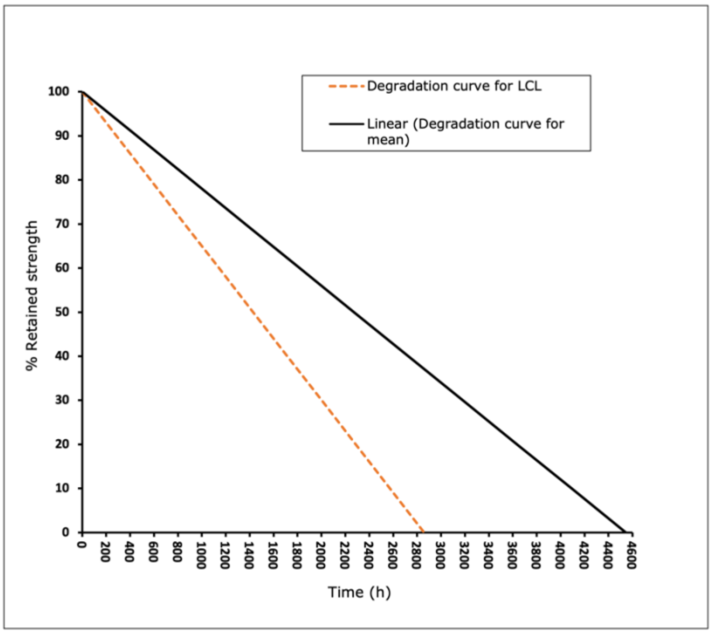

Figure 2.6 Degradation curve for PET in leachate of fly CFB ash

The degradation curve (figure 2.6) shows that PET geogrid will degrade completely to a 0% retained strength within 4545 hours and according to LCL within 2858 hours. The reduction factor and the uncertainty ratio $R_2 = T_{char} / T_{LCL}$ cannot be calculated, because these PET geogrids in fly CFB ash leachate environment degraded a lot faster than the manufacturers stated lifetime prediction.

The manufacturers predicted lifetime was 100 years, however this does put into consideration the lifetime when PET geogrids are subjected to alkaline environment, only within a pH of 4 – 9, however, our environment for testing had a pH over 12.
For this, it is advisable to not use woven PET geogrids as they will be greatly affected by chemical risk of hydrolysis.

CONCLUSION

The aim of this master’s thesis was to accelerate chemical degradation in reinforcement geosynthetics used in Estonian road constructions using geogrids as the geosynthetics to study. For this study, the following conclusions were drawn:

Geogrids of PP had a high chemical resistance to neutral, acidic and basic environment, not temperature dependent degradation occurred in this environment.
Geogrids of HDPE also had a high chemical resistance to neutral, acidic and basic environment and did not show any temperature dependent degradation.
Geogrids of PET had a chemical resistance to neutral environment of NaCl; however, it was slightly sensitive in acidic environment, and highly sensitive to alkaline environment.

A degradation curve was plotted for PET in alkaline environment of leachate of CFB fly ash. We are able to make predictions of the possible degradation of PET showing that it reached a 90% retained strength only after 490 hours, after 4545 hours, there should be no strength left in them. The lower confidence limit also had a predicted strength of 90% after 300 hours and after 2858 hours specimens are predicted to have no strength left in them.

Future research can be carried out with increased testing time, since geogrids are affected by duration rather than by temperature alone. Also, if possible, field retrieved samples can be used to generate data that can be used for lifetime prediction of aging. Samples retrieved from the field would confirm the predictions gotten from laboratory testing.

SUMMARY

Geosynthetics have been actively used in construction over the last three decades. Reinforcement geosynthetics are increasingly being used in pavement construction in Estonia. As a result, the life-time durability of geosynthetics used in Estonia pavement construction needs to be assessed. However, the assessment is focused on the chemical risk peculiar to Estonia that could result in possible degradation of for reinforcement and in road construction.

This study focused on the potential chemical risks of Estonian road construction that could shorten the lifetime of geosynthetics. The aim of the study was to assess the impact of potential chemical risks on the most common geogrids in Estonian road construction, based on their polymeric nature.

The results obtained from the experiment showed that geogrids of PP and HDPE are resistance to chemical aging of Estonia risk factors, while PET geogrids were more sensitive to chemical degradation, particularly to alkaline environment.

From the research was also concluded that duration in the environment has more impact on degradation than elevated temperatures. Therefore, in future studies the time in the environment should be longer to see the aging impact of possible chemical risks on geogrids, especially on HDPE and PP, but also on PET in acidic and saline environment.

KOKKUVÕTE

Geosünteete on viimase kolme aastakümne jooksul aktiivselt ehituses kasutatud. Eesti teedeehituses kasutatakse üha enam geosünteete armeerimise eesmärgil, mistõttu on vajalik hinnata Eesti teedeehituses kasutatavate geosünteetide pikaajalist vastupidavust.

Antud uuringus keskenduti Eesti teedeehituse võimalikele keemilistele riskidele, mis võivad geosünteetide eluiga lühendada. Töö eesmärgiks oli hinnata potensiaalsete keemiliste riskide mõju Eesti teedeehituses enamlevinud geovõrkudele, lähtudes nende polümeersest olemusest.

Läbiviidud katsetustest järeldus, et PP ja HDPE geovõrgud on võimalike keemiliste riskikeskkondade suhtes vastupidavad. PET geovõrgud on aga tundlikumad, eriti just aluselise keskkonna suhtes.

Uuringust järeldus ka, et ajaline kestvus riskikeskkonnas mõjutab geosünteedi vananemist rohkem kui kõrgendatud temperatuur. Sellest tulenevalt peaks tulevastes uuringutes geosünteetide ajalist kestvust keemilise riskikeskkonnas pikendama. See võimaldaks riskikeskkondade mõju paremini hinnata, eriti HDPE ja PP geovõrkudele, aga ka PET geovõrgule happelises ja soolases keskkonnas.

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