

# Graduation report

## **Influence of water ageing and ultraviolet exposure on the properties of epoxy bio composites**



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## Abstract

Nowadays, composites are mainly produced via petroleum derived chemicals. In a world where petroleum resources are running out, it is essential to find viable options for the replacement of these reserves. Biocomposites are already exploited but they appear to have low resistance to weathering. This resistance has been investigated for a full biocomposite and compare to a regular composite.

In this study, composites were made from Bioepoxy (Greenpoxy 56 from Sicomin) using Flax fibers as reinforcements for the full bio laminate. The regular composite was manufactured from the same resin but using glass fiber as reinforcement. They both have been aged in Bath two-sided immersion at 70°C and room temperature. The Flax composite have also been aged under UV exposure. An experimental device called "POT" has been used in order to counter the edges effect that induces a mistake in the calculation of the diffusion coefficient. At the end of the exposure, they were mechanically tested using tensile, flexural and ILSS tests.

A saturation level of 14,8% and 2,5% have been determined for the Flax composite and Glass composite respectively. The "POT" was not efficient enough to suppress the edges effect. The diffusion coefficient is 11 times faster when the water is at 70°C and the UV exposure didn't have a worrying impact on the composite. However, it turned out that the water ageing was consequently lowering the different mechanical properties of both materials.

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## Abbreviations

**70:** Bath 70°C

**ASTM:** American Society for Testing and Materials

**B:** Test preform in bath at RT

**B7:** Test perform in bath at 70°C

**BS:** Big size sample (approx. L= 200 mm; T= 2 mm; W= 20 mm)

**CP:** Circle pot sample (approx. 78 mm diameter)

**DGEBA:** Diglycidyl ether of bisphenol A

**GF:** Greenpoxy Flax fibers composite

**GG:** Greenpoxy Glass fiber composite

**GreenEpoxy:** Bio based epoxy polymer

**ILS:** Sample for Interlaminar Shear Strength testing (approx. L=20 mm; T= 2 mm; W= 10 mm)

**ILSS:** Interlaminar shear strength

**IR:** Infrared Spectroscopy

**ISO:** International Standardization Organization

**RT:** Room temperature (approx. 23°C)

**S:** Stand for Standardization

**U:** Specimen exposed to Ultraviolet

**UV:** Ultraviolet

**UVB:** UV radiation between (approx. 280 – 315 nm)

# I Introduction

In order to complete the fourth year of chemistry bachelor degree from HZ University, a graduation thesis must be done.

This graduation took place at the composite laboratory of the university Inholland in Delft in The Netherlands from the 22<sup>nd</sup> of February to the 22<sup>nd</sup> of July 2016. The Inholland Composites Laboratory, located at the TU Campus in Delft, is a very well equipped laboratory for the manufacturing of composites. The materials produced are concerning a wide range of applications where the weight reduction is an important factor. Additionally, the Composites Laboratory focus on sustainable product as biocomposites.

In order to decrease greenhouse effect and global warming overall, scientists have started to pay more and more attention to bio-based production. Moreover, Earth's natural resources are running out, new efficient ways to produce materials have to be found out. The usage of biomass products as sustainable source of raw material became a serious topic of research only during the past two decades (Fortes & Baugh, 2004).

Composites are more and more common in nowadays technology because their advantages have been understood. They are considered as an alternative way to materials such as aluminum and steel. Indeed, they have high specific strength, high specific modulus, corrosion resistance, low thermal conductivity (good insulating material) and improved fatigue life (Sadki, 2014). However, the polymer matrices and fibers of composites are still mainly based on petroleum-derived chemicals or others non ecological friendly processes like most of epoxy resins or polyurethanes for polymer matrix and man-made fibers such as glass, carbon or aramid. Therefore, it is important to find new ways of manufacturing composites with materials coming from sustainable resources. The application of plant fibers such as flax, jute or hemp is investigated with growing interest since the last decade, especially the flax fiber. Indeed, the flax reinforcement is the most promising among natural fibers with the highest mechanical properties. Nevertheless, plant fibers cannot become a competitor of the carbon fiber or aramid fiber because of the remarkable properties of both. However, they appear as strong as glass fibers on mechanical properties level when the weight is considered to be an important parameter for the manufacturing of composite. Therefore, they could replace glass fibers in some applications. Unfortunately, these fibers can have an impact on the resistance of the composite by aging differently from conventional fibers such as glass or carbon fibers.

To manufacture a sustainable composite, it is also necessary to use a resin or so-called matrix produced in a way that is respectful of the environment. In the respect of this condition, the epoxy resin was certified biobased by the manufacturer. Therefore, the combination of both, matrix and fiber, resulted in a product that can be classified as biocomposite.

For the comparison, two composites were manufactured. One from bioepoxy with flax fiber reinforcements and another from bioepoxy with glass fibers. These composites were produced in order to compare their properties after accelerated aging using water absorption and UV exposure.

Main question:

Can a biocomposite from bioepoxy and flax fibers be reliable after exposition to water and ultraviolet radiation?

Sub-questions:

- How to manufacture the composites?
- Which procedure is the most adapted to perform the accelerated water aging?
- What is the diffusion rate and the saturation percentage of water in the composite?
- Which procedure is the most adapted to perform the ultraviolet radiation exposure?
- What is the impact of the ultraviolet radiation on the chemical structure and weight of the composite?
- What is the impact of the ageing on the mechanical properties of the composites?

## II Theoretical Framework

### II.1 Composites

#### II.1.1 General informations

Composites are more and more common in nowadays technology because their advantages have been understood. They are considered as an alternative way to materials such as aluminum and steel. Indeed, they have high specific strength, high specific modulus, corrosion resistance, low thermal conductivity (good insulating material) and improved fatigue life besides the fact that they are light (Sadki, 2014). These interesting properties are making composites a growing part in many application fields such as aerospace, automotive, sport, medical field and infrastructure.

Generally, composites are composed of two substances or more. The matrix and the reinforcement. The reinforcing material is stiffer and stronger providing the strength for the composite material. The matrix is the binder or resin that holds the fibers together.

#### II.1.2 Epoxy resin

The reaction between bisphenol A and epichlorohydrin is the major basis for the production of epoxy resins (ASM International, 2001). They are mainly used in composites materials for many different applications such as the automotive, aerospace sectors for instance. This matrix is one of the most versatile available on the market. Indeed, they are offering a wide range of mechanical and physical properties but also a freedom for processing conditions that makes them way above other thermoset resins in term of possibilities (ASM International, 1987). The key chemical group of the matrix is the epoxide group. It is also called oxirane ring. It is a ring composed of one oxygen atom and 2 carbons atoms (Figure 1). The usual starting material for the production of epoxy resins is the prepolymer diglycidyl ether of bisphenol A (DGEBA) from the reaction between bisphenol A and epichlorohydrin.

However, many other prepolymers can be exploited. The prepolymer has two epoxide groups at its extremities which allows the polymerization by using a curing agent (Mallick, 2007). The term epoxy resin is broad because of the wide range of prepolymer and curing agent that can be used in its production resulting in various molecular structures. The following parts will be explained taking DGEBA as prepolymer and a curing agent with amine functional groups.

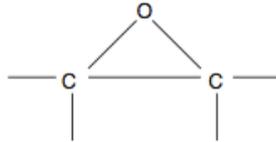


Figure 1: Representation of the epoxide group (Mallick, 2007)

### II.1.2.1 Reaction for DGEBA synthesis

The reaction mechanism of DGEBA production is detailed in Appendix 1

The molecule prepolymer DGEBA results from the step-growth polymerization reaction between bisphenol A and epichlorohydrin in presence of sodium hydroxide (NaOH) catalyst (Figure 2).

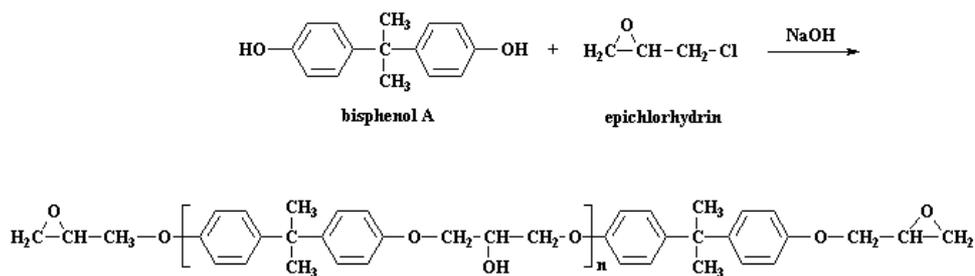


Figure 2: Reaction for the production of DGEBA (University S.M., 2005)

The degree of polymerization  $n$  (Figure 3) is a function of the ratio between Bisphenol A/epichlorohydrin. Depending on its value the molecule of the prepolymer DGEBA is more or less long and its viscosity is affected. Thus, at room temperature, for  $0 < n < 1$  the prepolymer is liquid. For  $1 < n < 1,5$  it is in a viscous state and in a solid state for  $n$  superior to 1,5 (Bardonnnet, 1992). The chemical structure of DGEBA contributes to its good chemical resistance and mechanical properties. The thermal resistance, corrosion resistance and stiffness are provided by the aromatics. The ether groups give also a good stability to the hydrolysis (Figure 3) (Nguyen, 2013).

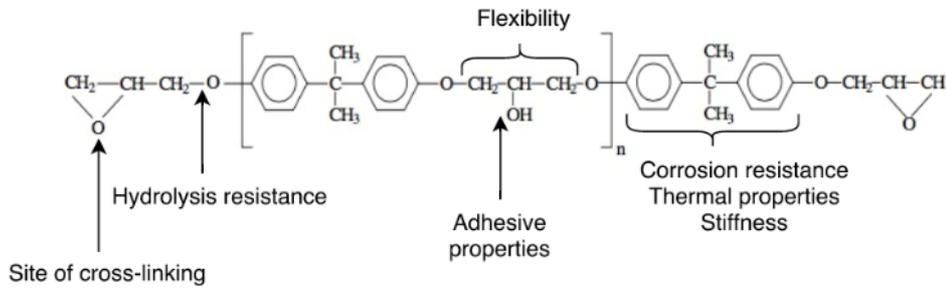


Figure 3: Relation chemical structure and properties (Heman, 2008)

The prepolymer is constituted of two types of sites subject to react: the epoxy groups and the hydroxyls groups. In order to use the DGEBA as matrix, it is essential to open the oxirane ring and this action can be done using amines with labile hydrogen or diacid anhydride. In this study, an amine hardener was used. It is indeed the most common hardener in the production of solid epoxy resin because of numerous advantages such as their low viscosity, their capability to react at room temperature, their reactivity and their price for instance (Debdatta, 2009).

### II.1.2.2 Curing reaction

According to (Nguyen, 2013), the type of polymerization involved in the cure of epoxy resins by diamines is an exothermic and irreversible chain-growth polymerization. This reaction is the transformation of a low molar mass viscous liquid in a viscoelastic amorphous solid with a quasi-infinite molar mass. The epoxy resin being thermoset, the crosslinking network is tridimensional. Several reactions are considered even if the important number of different constituents in commercially available formulations can induce other crosslinking mechanisms (Aufroy, 2005).

The whole reaction mechanism is displayed in Appendix 1.

### II.1.2.3 Bio epoxy resins

A resin or polymer can be considered as bio when one or more of its constituents are obtained by an eco-friendly process using natural resources. In the case of the epoxy, according to (Momtaz, 2014), the label bio is attributed to the resin because the epichlorohydrin involved in the production of the prepolymer DGEBA is 100% biobased. Indeed, the epichlorohydrin produced by Solvay under the brand name Epicerol is synthesized from glycerol. This glycerol is a byproduct from the biodiesel and oleochemicals production. The global warming potential of Epicerol is 61% lower than oil-based epichlorohydrin due to the carbon absorbed by the plants involved in its production and the diminution of greenhouse gas emissions. (Solvay, 2016).

### II.1.3 Fibers

In a fiber-reinforced composite the fibers are the main constituents. They are providing strength and stiffness to the whole material. In general, the properties of the composite are mainly dependent on the fiber chosen since it influences many characteristics such as density, tensile strength and modulus, fatigue strength, electrical and thermal conductivities but also the cost (Mallick, 2007). The fibers can be oriented differently in the matrix (Figure 4) offering a wide range of choices depending on the desired application.

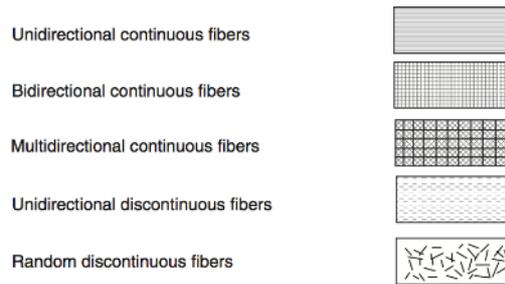


Figure 4: Fiber orientations (Mallick, 2007)

Glass fibers are the most common fibers for application in polymer matrix composite. However, during the past two decades, the use of natural fiber has been actively investigated in order to decrease the impact of man made fibers on the environment. These fibers are already used but only for non-structural applications. They are not suitable for high-performance applications yet due to their hydrophilic feature as well as the poor bonding at the interface matrix/fibers preventing the fiber properties to be transferred into the composite. Once the above mentioned problems are solved, plant fibers, being cheaper and environmentally friendly, are likely to be good candidate to replace the glass fibers. Several plants are studied like hemp, flax, jute for instance but the flax fiber is the most promising one.

#### II.1.3.1 Flax fibers

All Plant fibers are mainly composed of cellulose, hemicellulose and lignin. They also contain low amounts of pectins. The cellulose, hemicellulose and lignin could be referred to as natural polymer since they are produced by the plant itself during their growth. The cellulose is considered as the main components which support the fiber's structure. It controls the resistance, stiffness and structural stability of the fiber and has a high crystallinity. These biopolymers, especially lignin and cellulose have a high level of hydroxyl groups in their chemical structure. Therefore, they have hydrophilic and polar features (Toupe, 2015).

Fibre	Flax
Type	
Cellulose [%]	65-85
Lignin [%]	1-4
Hemicellulose [%]	10-18
Pectin [%]	1-3
Crystallinity [%]	70-75
L/d	1700
Spiral Angle [°]	10
Tensile Strength [MPa]	500-900
Modulus [GPa]	50-70
Elongation [%]	1.5-4.0

The flax fibers are constituted of 65 to 84 % of cellulose, 1 to 4 % of Lignin and 10 to 18 % of hemicellulose (Figure 5). The natural fibers can be compared to a composite material. Indeed, the cellulose plays the role of reinforcement kept together by hemicellulose and lignin which appear to have the same function as a matrix (Van De Weyenberg, 2005). According to (Sparnins, 2006), the fiber alone has a tensile strength from 500 to 900 MPa with a modulus of 50 to 70 GPa. Furthermore, the elongation can be up to 4%. However, concerning a composite flax/epoxy the elastic modulus will be much lower (about 60 MPa for a volume fiber of 50%) (Baley, 2006).

Figure 5: Composition and properties of flax fibers (Van De Weyenberg, 2005)

### II.1.3.2 Glass fibers

There are several types of glass fibers existing (E, C, D, F or S...) depending on the composition of the blend for its preparation. The E glass fibers are the most common because of their interesting mechanical properties and price. They are also easier to manufacture than the other kinds. This reinforcement is generally used when strength and high electrical resistivity are required. (Nguyen, 2013).

Table 1: E glass fiber composition (Nguyen, 2013)

SiO <sub>2</sub>	52-62%
B <sub>2</sub> O <sub>3</sub>	0-10%
Al <sub>2</sub> O <sub>3</sub>	11-16%
TiO <sub>2</sub>	0-3%
Fe <sub>2</sub> O <sub>3</sub>	0-1%
HF	0-2%
Na <sub>2</sub> O + K <sub>2</sub> O	<2%
CaO	16-25%
MgO	0-5%

The chemical composition of the E glass fiber is varying up to the manufacturer but it is a family of glasses with a calcium aluminoborosilicate composition and a maximum alkali content of 2% (ASM International, 1987). Other additives can be added such as calcium oxide or boron trioxide which allow to decrease the fusion temperature or aluminum oxide that improves the mechanical properties of the fiber.

The mechanical properties displayed in the Table 2 have been acquired by testing E glass single strand.

Table 2: Mechanical properties of E glass single strand (C.H.R.P.P.G., 1986)

Density	2,60 g/cm <sup>3</sup>
Young Modulus	73 GPa
Tensile strength	3400 MPa
Rupture strain	4,5 %

## II.2 Vacuum Infusion Process

Vacuum Infusion Process (VIP) is the only closed mold process that uses the atmospheric pressure. The vacuum is used to remove all the air in order to compact the reinforcements and helps the resin to migrate through the fibers (Figure 6). The permeability of the fiber mat plays an important role as well as the viscosity of the resin. A low viscosity resin is preferred for this process (Composites World, 2016).

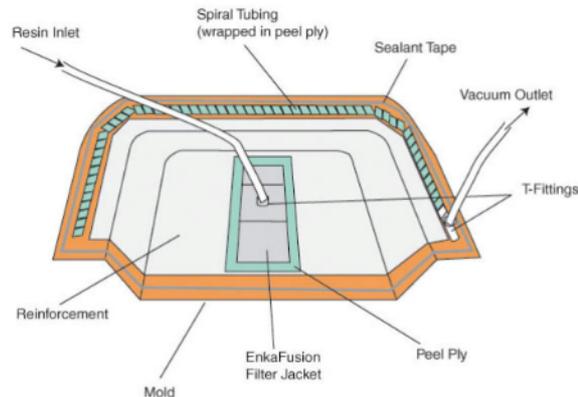


Figure 6: Vacuum Infusion Process (Performance Composites, 2016)

This technique has numerous advantages. There is none or less harmful gas emissions of volatile organic compounds. By reducing the voids content, the composite is stronger. There is also an apparent consistency and repeatability using this technique (Strong, 2008).

## II.3 Environmental ageing

The composite may be exposed to many environments during its life as high temperature and humidity or also ultraviolet exposure for instance. Therefore, before to use the composite in an environment it is important to study its behavior by artificially exposing it to these natural ageing factors (ASM International, 1987). Sunlight and moisture cause millions of euro's material damage every year. Therefore, it is interesting to test products resistance against this type of weathering (Q-Lab Corporation, 2007).

### II.3.1 Hygrothermal ageing on epoxy matrix composites

The degradation of the properties of polymeric composites, after exposure to a combination of moisture and temperature is referred to as hygrothermal ageing (Surathi & Karbhari, 2006). The hygrothermal ageing is impacting, by different mechanisms, the constituents of a composite which are the fibers, the matrix and the interface fiber/matrix.

#### II.3.1.1 Chemical ageing

The chemical ageing is a phenomenon involving a chemical modification of the material under the influence of an environment. Concerning the chemical ageing linked to water exposure, the term hydrolytic ageing is employed. In a composite it is only the matrix which is vulnerable to the chemical ageing. Whenever a matrix system is exposed to water for an extended period of time, chemical reactions are taking place between the adsorbed water and the elements of the matrix system. With an epoxy matrix the water is susceptible to break the oxirane ring to give a diol. However, this reaction of hydrolysis (Figure 7) is limited or even not happening in an epoxy produced with an amine hardener (Figure 4).

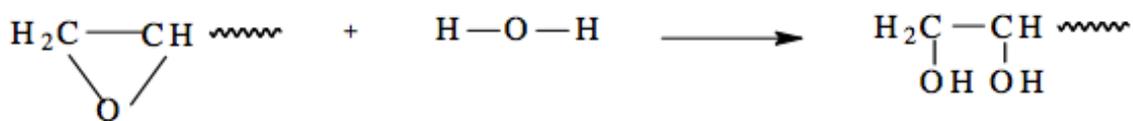


Figure 7: Hydrolysis reaction of an epoxy (Nguyen, 2013)

The hydrolysis reaction can be observed on the kinetic of the water diffusion through the matrix. This resistance can be highlighted when the mass gain curve in function of time is drawn and no mass loss is observed. According to (Nguyen, 2013), a epoxy system subject to hydrolysis tends to loss mass in contrary of some other matrix that gain mass because of the by-product of the reaction. The hydrolysis is irreversible.

#### II.3.1.2 Physical ageing

The physical ageing includes all phenomena in which the chemical structure of the material is not modified. We can distinguish two types of water absorption:

- The absorption in the macromolecular network of the matrix
- The absorption in the heterogeneity of the material (voids, micro-cracks and other impurities).

One of the main physical effect is the plasticization (Figure 8). This effect induces a reorganization of the molecular network by establishing secondary interactions as hydrogen bonds between the water molecules and the polymer replacing the previous bonds between molecular chains of the polymer.

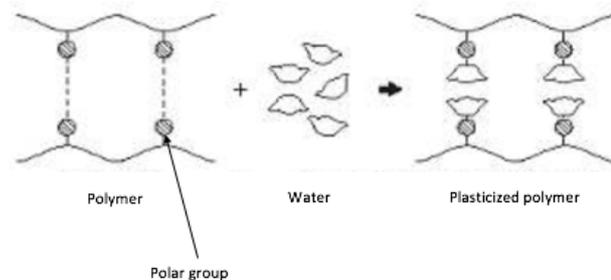


Figure 8: Plasticization of a molecule (Heman, 2008)

There is a partial destruction of the mechanical cohesion which was assured by these bonds as well as an increase of the molecular mobility. However, the plasticization is reversible, so if the material is dried it recovers his properties (Heman, 2008).

As other effect of the water ageing, the absorption by the heterogeneities needs to be mentioned. These heterogeneities are generally voids in a porous material that can be the consequence of an imperfect infusion process. Indeed, the mold not being well prepared or the incomplete degassing step of the resin before injection induce the formation of void in the matrix. The micro-cracks where the water can diffuse are provoked by an inadequate cure cycle. The cycles of cure and post-cure influences the water absorption in the composite (Heman, 2008).

### II.3.1.3 Osmosis

In certain circumstances the laminate acts as a semi-permeable membrane due to uncured or hydrolyzed matrix substances. If exposed to external fluids with different concentrations the difference in concentration causes water molecules to penetrate through the polymer. This penetration can induce a swelling of the composite (Figure 9) and the internal stress caused by the latter affects the mechanical properties of the material. This transportation is typical of the osmotic process. It continues until the osmotic pressure is reached or until the equilibrium is (Heman, 2008).

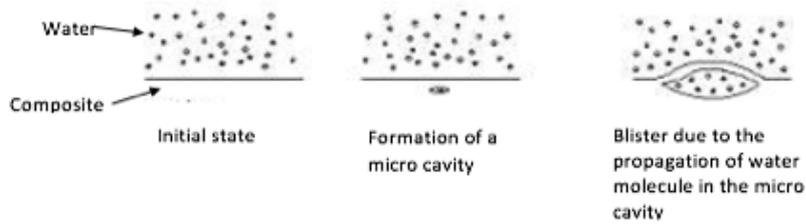


Figure 9: Blister formation (Heman, 2008)

According to (Nguyen, 2013), it seems that in a composite, the osmotic mechanism is localized at the interface, making easier the way for the water to move and weaken the cohesion fiber/matrix. The movement of the water through the interface is known as wicking (Hamer, 2012). The interface plays an important role in composite materials. A weak interface results in relatively low strength and stiffness but can have high resistance to fracture. On the other hand, with a strong fiber matrix bonding the material has high strength and stiffness but might be brittle. This property is depending on the atomic arrangement and chemical properties of the fiber and on the chemical structure of the polymer matrix. As said previously, the flax fibers are hydrophilic. Therefore, it's likely that the interface in the biocomposite will be greatly damaged. Concerning the fiber glass, it is reported that the interface of an epoxy glass composite suffers as well. According to (Springer & Shen, 1980) when the moisture content is above 1% the tensile strength can decrease up to 20%.

#### 11.3.1.4 Effect of water ageing on the fibers

Following the potential damaging of the interface the fibers are exposed to the water. Therefore, susceptible to be corroded and weakened leading to cracks reducing the mechanical properties of the composite.

In this study two kind of fibers or used, the glass fibers and the flax fibers. As mentioned earlier, the flax fiber is very hydrophilic and tends to absorb a lot of water. It results in an important weakening of the interface but not a degradation of the fiber itself. However, the glass fiber is subject to degradation. It is composed of several oxides (Table 1) among them  $\text{SiO}_2$  which react with water (Figure 10) by hydration and destabilizes greatly the fiber.

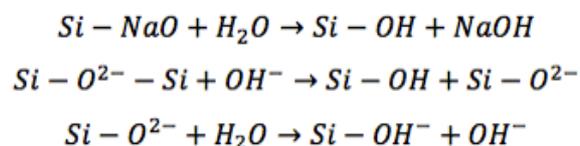


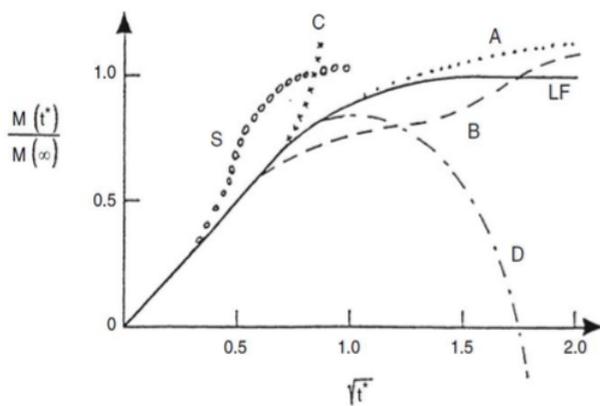
Figure 10: Reaction of  $\text{SiO}_2$  with water (Ishai, 1975)

To conclude, the water can potentially attack:

- The fiber and degrading its resistance
- The matrix by plasticizing it, swelling, micro-cracking or hydrolyzing
- The interface between fiber and matrix because of hydrolysis or osmotic pressure

### II.3.1.5 Diffusion and Fick's law

Concerning the water uptake, the diffusion is the transportation of moisture from an environment of high concentration towards an environment of low concentration by mean of random molecular motion. In this case, the water is diffusing into the composite. These diffusion behaviors have been widely observed and the sorption data is plotted in a graph to give a good perspective of each type of behavior (Figure 9) (Surathi & Karbhari, 2006).



In which:

LF: Linear Fickian diffusion

A: Pseudo-Fickian model

B: Two-stage sorption

C: Rapid increase in moisture content

D: Weight loss attributed to irreversible degradation

S: Sigmoid behavior with single inflection point

Figure 11: Different water uptake behaviors (Surathi & Karbhari, 2006)

The sorption behaviors can be described as following (Hamer, 2012)

- Linear Fickian diffusion: Continues incline with a slow approach towards equilibrium.
- Pseudo-Fickian model: A gradual increase of moisture content which never attains equilibrium.
- Two-stage sorption: The start is a linear uptake of moisture after which it approaches a quasi-equilibrium followed by a final approach to a true equilibrium.
- Rapid increase in moisture content: This is usually accompanied by damage growth leading to material failure.
- Weight loss attributed to irreversible degradation, this is due to the chemical or mechanical breakdown of material. The material leaches along the fiber/matrix interphase regions as well as by hydrolysis.

As shown on Figure 9, some composites and neat resins have simple water absorption behavior that are matching with Fick's second law. This law's basis is that the driving force of diffusion is the fluid concentration gradient:

$$\frac{\partial M}{\partial t} = D \cdot \frac{\partial^2 M}{\partial Z^2} \quad \text{Eq. 1}$$

Where:

D: Diffusion coefficient (mm<sup>2</sup>/s)

M: Moisture content (%)

t: Conditioning time (s)

Z: thickness of the material in which the water is diffusing (mm)

Moisture absorption curves fitting Fick's law have two distinct parts. One part linear and another corresponding to a saturation level where the moisture content stops varying. This curve is obtained by plotting the moisture content in function of the square root of time.

The moisture content of a composite or plastic with a Fickian behavior can be worked out at any time using the Eq. 2 obtained by the method of variable separation from Eq. 1 and assuming that the water doesn't penetrate through the edges of the laminate.

$$M(t) = M_0 + (M_m - M_0) \cdot \frac{4}{h} \cdot \sqrt{\frac{D \cdot t}{\pi}} \quad \text{Eq. 2}$$

Where:

M<sub>0</sub>: initial amount moisture in the solid

M<sub>m</sub>: the saturation moisture content (%)

h: thickness (mm)

t: conditioning time (s)

D: diffusivity in direction of the thickness (mm<sup>2</sup>/s)

In order to determine the moisture content at any time the diffusion coefficient and the maximum moisture content must be known first. It is possible to experimentally determine these constant thanks to a gravimetric analysis.

Under conditions of steady state temperature, the fluid uptake in a material can be expressed as a percentage of the original dry weight using:

Where:

$W_i$ : Mass wet specimen

$W_d$  mass dry specimen

$M$ : Percentage of fluid uptake or loss

$$M = \left( \frac{W_i - W_d}{W_d} \right) \quad \text{Eq. 3}$$

According to (Shell, 2016), the Eq. 4 is assuming the classical single free phase model of absorption in which the molecules are not combined with the polymer. The diffusion coefficient can be determined using the equation:

$$D = \pi * \left( \frac{wt}{4 \cdot M_e} \right)^2 * \left( \frac{M_1 - M_2}{\sqrt{t_1} - \sqrt{t_2}} \right)^2 \quad \text{Eq. 4}$$

where  $M_1$  and  $M_2$  are the percentage of fluid uptake at times  $t_1$  and  $t_2$  respectively,  $wt$  is the wall thickness and  $M_e$  is the fluid equilibrium/saturation level. The term (Eq. 5) is the slope of the linear portion of the plot of  $M$  against the square root of time:

$$\left( \frac{M_1 - M_2}{\sqrt{t_1} - \sqrt{t_2}} \right) \quad \text{Eq. 5}$$

An approximation of the time needed to reach the stabilization period can be determined by using Eq. 6:

$$t_{stab} = \frac{\pi}{16} * \frac{wt^2}{D} \quad \text{Eq. 6}$$

However, these equations are concerning the case of isotropic thin sample strips where the thickness of the sample is way smaller than the length and the width ( $wt \ll l$  and  $wt \ll b$ ). Indeed, Eq. 4 provides the one-directional diffusion coefficient by omitting the edges effect. A correction factor called the "size factor" needs to be added to the previously calculated diffusion coefficient:

$$D_r = D * \left( 1 + \frac{wt}{l} + \frac{wt}{b} \right)^{-2} \quad \text{Eq. 7}$$

There are several factors that are influencing the water uptake. The fiber volume fraction, the fiber orientation, the voids, the matrix composition and finally the cure state are playing a role as internal factors. As external factors, it is assumed that the temperature, the type of water and the manufacturing process induce more or less water sorption into the composite (Hamer, 2012)

## II.3.2 Ultraviolet exposure

The purpose of this part is to give a clear and accurate explanation on the impact of UV radiations on polymer material, more precisely on an epoxy matrix.

### II.3.2.1 Photochemistry

The photochemistry studies the transformation of molecules exposed to light. When the material is irradiated, molecules absorb energy to a certain extent until the transition level. This so called transition level means that the molecule moves from its stable state to its excited state. Therefore, the molecule has no a reactivity that it doesn't have in its fundamental state. The process can be written as following:

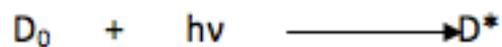


Figure 12: Molecule moving to its excited state

**hv** comes from the Planck's equation (Eq. 8)

Where:

**E**: Energy of radiation

**h**: Planck's constant

**v**: The frequency of the radiation

**c**: Velocity of light

**λ**: The wavelength of the radiation

$$E = hv = \frac{hc}{\lambda} \quad \text{Eq. 8}$$

In some cases, the excited molecule  $D^*$  is able to transfer its energy to a molecule non absorbent as the Figure 15 shows:

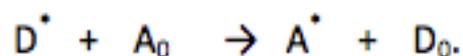


Figure 13: Transfer of excited state

Ultraviolet light is electromagnetic radiation and as such is part of the spectrum of the sunlight's spectrum. Ultraviolet radiations are subdivided into 3 types. The UVA from 315 to 380 nm, the UVB from 280 to 315 nm and UVC from 10 to 280 nm. Because of the absorption in the Earth's atmosphere, 99% of the UV-light that reaches the surface consists of UVA (Gzik, 2007).

The ageing from UV exposure is because of the photo-oxidation mechanisms involved. In fact, it is the energy of UV light which is facilitating the oxidation of the polymer (Nguyen, 2013). This mechanism is based on 3 steps

- Initiation. Where the polymer or an impurity gives a radical  $R\bullet$  as seen in Figure 14.
- Propagation. During this step the primary radical reacts with oxygen to give  $RO_2\bullet$  radicals. Then, these  $RO_2\bullet$  attacks chemical groups with hydrogen atoms to form hydrogen peroxide  $ROOH$ . These molecules are particularly unstable to UV and release new radicals.
- Termination. Diverse mechanisms of termination can be suspected

The entire mechanisms are described in Appendix 2.

### II.3.2.2 Photo-oxidation of epoxy resin

All the formation mechanisms are displayed in Appendix 3.

Concerning epoxy resin from DGEBA/amine, the fundamental products from the photo-oxidation are amides (visible at  $1650-1670\text{ cm}^{-1}$  on the Infrared spectrum), carbonyls ( $1730-1740\text{ cm}^{-1}$ ) and structures containing quinone methide ( $1658\text{ cm}^{-1}$ ). According to (Rivaton, Moreau, & Gradette, 1997), phenyl formate ( $1735\text{ cm}^{-1}$ ) is produced as well. The mechanism of the formation of these photo-products are involving mainly the ether group and the  $\text{CH}_3\text{-CH}$  bond of the epoxy polymer. UV radiations is able to change the structure of the resin because of the labile hydrogens from DGEBA composition (Figure 14).

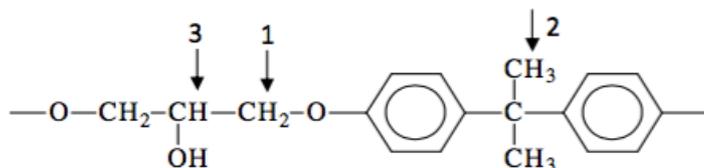


Figure 14: The 3 potential reactive sites under UV exposure

Moreover, the hydrogens on the oxirane ring from non cured DGEBA into the polymer are labile enough to react with UV radiation forming alcohols or aldehyde groups. The UV ageing is resulting in a decrease of the  $T_g$  and mechanical properties. A yellowish color can be observed as well.

## II.4 Characterization

### II.4.1 Tensile strength test

A force is applied on a sample of standardized size. The tensile strength is the resistance of this material while a force tends to tear it apart. It measured the maximum tension the material can withstand without tearing. A graph is drawn on which one the elastic and plastic behavior can be observed (Appendix 4). The young modulus (Eq. 9) is also an important value that can be worked out from this graph. It compares the strain (mm) against the force (MPa) (Jacquot, 2010).

Where:

$$E = \frac{\Delta\sigma}{\Delta\varepsilon} \quad \text{Eq. 9}$$

E: Young's modulus

$\Delta\sigma$ : Difference in tensile stress between two point of the curve

$\Delta\varepsilon$ : Difference in strain between two point of the curve

Different fracture modes can occur during this test. They are all displayed in Appendix 4.

### II.4.2 Flexural strength test

The flexural test can either be three points or four points. Here, the test was performed in the three points set up. It allows to measure the breaking strength. The sample is placed on 2 support (2 points) and the third point applies a force on the middle of the sample (Jacquot, 2010). The flexural modulus can be worked out thanks to the Eq. 10.

$$E_{\text{bend}} = \frac{L^3 F}{4wh^3 d} \quad \text{Eq. 10}$$

Where:

L: Distance between the two supports

d: The deflection due to the load

wh: Width and thickness of the specimen

F: Load applied

### II.4.3 Interlaminar shear strength test

The interlaminar shear strength test or short beam test is based on the flexural strength test. The difference is that the distance from the two lower supports is much less. This test is used to determine interlaminar shear strength of parallel fibers. In other words, the bonding between fibers and matrix is tested. A plan is available in Appendix 5 as well as the different fracture modes.

This test allows to determine the short beam strength by using the following equation:

$$F_{sbs} = \frac{3P}{4 \times W \times H} \quad \text{Eq. 11}$$

Where:

F<sub>sbs</sub>: Short beam strength

P: Maximum load

W: Width of the specimen

H: Thickness of the specimen

## III Methods and Materials

A flowchart overview of all methods is displayed in Appendix 6.

### III.1 Composites manufacturing

#### III.1.1 Vacuum infusion

Both composites have been manufactured by vacuum infusion using the resin Greenpoxy 56 from Sicomin. The hardener diamine SD 4771 from Sicomin as well has been used to cure the resin. For each composites 4 plies of 0° unidirectional flax fibers and glass fibers have been placed on the glass plate. Manufacturer of fibers is “Biotex” for flax and “SGL carbon company” for the glass fibers (UD ES 600/300). Approximately 800 g of resin were necessary to complete the infusion. It resulted in composite plate of approximately 0,25 cm<sup>2</sup>. The laminates have been cured for 24 hours at RT followed by a post-cure treatment at 60°C for 8 hours and 100°C for 4 hours. The aim was to produced 2 mm thick plate.

The full protocol for the infusion process is displayed Appendix 7 as well as the manufacturer data sheet of the resin/hardener and flax fiber.

#### III.1.2 Cutting of specimens

##### III.1.2.1 *Flax fibers composite*

The flax fibers composite specimens have been cut by means of a laser cutter “Lion 1250 iX” using the software Lasercut 5.3 Lion laser systems.

##### III.1.2.2 *Glass fibers composite*

The glass fibers composite specimens have been cut by a water jet cutter company named “Alleswatersnijden” based in De Lier in The Netherlands.

## III.2 Fiber content determination

The fiber content of both composites have been determined by two different methods.

### III.2.1 Determination of Glass fiber content by ignition loss

The Glass fiber content has been worked out using the method of Ignition Loss. This method is described in the ASTM D 2584 – 94 (Ignition Loss of Cured Reinforced Resins). The epoxy resin has been totally vaporized when heating 3 replicate specimens placed in crucibles at 565°C in a “Vecstar” furnace. The difference between the mass of the residue (glass fiber only) and the sample allows the determination of the fiber content in the composite using the following equation:

$$Mf = \frac{Mr}{Mt} \times 100$$

Eq. 12

Where:

*Mf*: Fiber mass fraction in %

*Mr*: Mass residue

*Mt*: Mass total of the specimen

### III.2.2 Determination of Flax fiber content

The content of Flax fiber in the composite has been determined by weighing 4 plies of Flax fiber of dimensions equal to BS. The mean of 10 BS and the mean of 10\*4 plies of Flax fibers have been necessary to determine the fiber content using the following equation:

$$Mf = \frac{Mf}{Mbs} \times 100$$

Eq. 13

Where:

*Mf*: Fiber mass fraction in %

*Mf*: Mass flax fibers

*Mbs*: Mass BS specimen

### III.3 Water ageing

The following procedure is an example for one type of composite. All specimens' dimensions (mean of 3 thickness, mean of 3 width and length) have been measured using a digital caliper from "HBM Machines" before exposure. The samples have been dried at 70°C for 12 hours, then cooled down in a desiccator at RT in order to know the dry weight. This process has been repeated until the dry mass stabilized. All weight measurements were made using the same scale AG204 from "Metler Toledo" to the nearest 0,1 mg. The planned dimensions of specimens have to be found in Appendix 6. This method is in accordance with ISO 62:2008 (Plastics – Determination of water absorption) and ASTM D 570 – 98 (Water Absorption of Plastics). The weight gain in percentage was worked out from the average of 10 specimens for BS samples and 6 specimens for ILS samples. The dimensions were taken again after the ageing for the 10 laminates immersed in the RT Bath to measure the potential swelling of specimens.

#### III.3.1 Ageing in bath 70°C and RT

The samples (10 BS and 6 ILS for each bath) were immersed in one beaker of 5L for 70°C bath (B7) and in two beakers of 3L for the RT bath. The beaker of 5L has been placed in an oven "Ecocell Fisher Bioblock Scientific". The water temperature was  $70 \pm 5$  °C. Before measuring the weight, B7 specimens were systematically immersed in water at RT for 5 minutes. Specimens for both, RT bath and 70°C Bath, were always wipe dry of the water on their surface. The water used was filtrated by reverse osmosis principle by means of a filtration device from the company "Merck Millipore". The pH was measured using "Macherey-Nagel" pH paper. The water was changed after 3 days' exposition.

The mass of the specimens was frequently measured during the first 24 hours and once everyday afterwards. The moisture content (%) (Eq.3) has been plotted against the square root of time. The slope of the linear part of the curve was determined using Eq. 5 in order to calculate the diffusion coefficient.

#### III.3.2 POT Ageing

The POT is an experimental device to measure the water uptake suppressing the edge effect by mean of a one-sided exposition. The design of this apparatus is displayed in Appendix 8. Only the GF composite has been tested at RT as the speed of diffusion was very slow. Measurements were taken only once a day.

## III.4 UV exposure

Only the GF composites have been exposed to UV radiations because this type of ageing affects exclusively the matrix. The specimens have been dried following the same procedure as for the water ageing before exposure. Five BS samples have been introduced in a UV black box designed by Inholland CompositeLab, as well as one sample of neat epoxy resin. The bulb used was an UVA-340 Lamp from Q-Lab Corp with an irradiance peak at 340 nm. The technical datasheet of this lamp can be found in Appendix 9. The pure epoxy resin has been analyzed by infrared spectroscopy using a Spectrum 100 FT-IR Spectrometer from PerkinElmer before and after UV ageing. The BS composites placed in the UV box were designed to be mechanically tested with a tensile test and weighed everyday. The mass change has been plotted against the time. The specimens have been exposed to UV light for 504 hours.

## III.5 Mechanical properties testing

All mechanical tests have been performed in the Aerospace Structures and Materials Laboratory of Delft University of Technology.

### III.5.1 Tensile test

The tensile tests have been performed in accordance with ASTM D3039/D3039M – 14 (Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials). The calculation mentioned page 23 – 24 are directly applied by the software of the apparatus.

#### III.5.1.1 GF composites

The Flax fibers composites have been tested on a 10 kN tensile bench developed by “ZwickRoell” using the software testXpertII. The followings specimens have been tested: 5 BS for standardization, 5 BS aged by UV, 5 BS aged in 70 °C bath. A speed of 1,3 mm/min and a gauge length of 140 mm were chosen. A pre-loading of 2 N was applied before testing.

#### III.5.1.2 GG composites

The Glass fibers (2 BS standards/2 BS aged in 70°C bath) have been tested on a 20 kN tensile bench with extensometer developed by “ZwickRoell” using the software testXpertII. A speed of 1,3 mm/min and a gauge length of 140 mm were chosen. A pre-loading of 2 N was applied before testing.

### III.5.2 Flexural test

The Flexural tests or 3-point bending tests have been performed in accordance with ASTM D 790 – 03 (Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials).

Both, GF and GG composites (5 BS Standard/5 BS aged in 70°C Bath), have been tested on the “ZwickRoell” 10 kN bench. A speed of 1 mm/min and a 32 mm width span between the two lower support were used. The test was set up to stop when 5% deflection were reached.

### III.5.3 Interlaminar shear strength test

The short-beam shear tests have been performed in accordance with ISO 14130:1997(E) (Fibre-reinforced plastic composites –Determination of apparent interlaminar shear strength by short-beam method).

Both, GF and GG composites (6 ILS Standard/6 ILS aged in 70°C Bath) have been test on the “ZwickRoell” 10 kN bench. A speed of 1 mm/min and 10 mm width span between the two lower supports were used. A preload of 2 N was set up as well.

## IV Results and Discussion

### IV.1 Determination of fiber content

#### IV.1.1 Fiber content of GG composite by Ignition Loss

The results as well as statistic data are displayed in the table below.

Table 3: Results of GG ignition loss

N°	Mt (g)	Mr (g)	Mf (%)
1	0,5805	0,4611	79,43
2	0,5804	0,4631	79,79
3	0,6006	0,4760	79,25

The statistic study shows a average fiber content  $M_f = 79,49 \pm 0,2750$  %. The coefficient of variation is equal to 0,003459. This result is acceptable because  $0,2750 \ll 3,0$  (ASTM). The fiber content will be rounded to 80 %.

## Calculations

Example for N°1 using Eq. 12:

$$Mf = \frac{0,4611}{0,5805} \times 100 = 79,43 \%$$

$$s = \sqrt{\frac{\sum(X - Av)^2}{(n - 1)}} = \sqrt{\frac{((79,43 - 79,49)^2 + (79,79 - 79,49)^2 + (79,25 - 79,49)^2)}{(3 - 1)}} = 0,2750 \%$$

Where:

*s*: Standard deviation

*X*: Value

*n*: Number of observations

*Av*: Average

### IV.1.2 Fiber content of GF composite

The average mass found for 20 BS specimens was equal to 8,504 g. The average for 10 measurements of 4 fibers plies was equal to 4,190 g. A fiber content of 49,30 % has been found. The fiber content will be rounded to 50 % for the rest of the report.

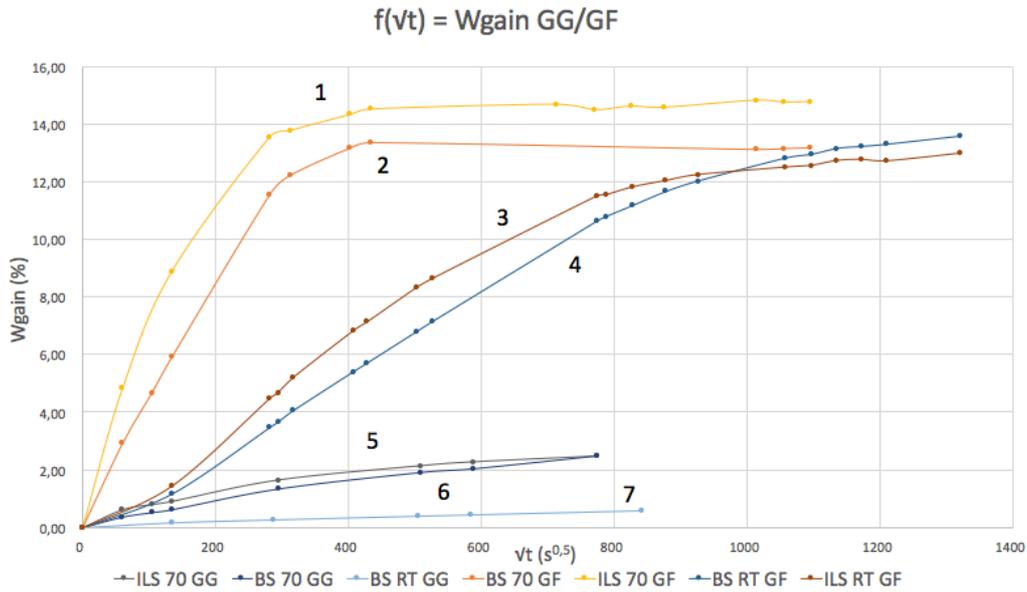
## IV.2 Effect of water ageing

Tables with all dimensions of specimens before and after exposure are to be found in Appendix 10. All detailed data of the water uptake experiments have to be found in Appendix 10 as well. Only the GF composite has been aged using the POT.

### IV.2.1 Swelling

The dimensions of specimens after 676 hours and 197 hours of exposure in Bath RT for GF and GG composites respectively were measured. No swelling has to be reported for the Glass/epoxy composite. However, for the GF laminate, a volume swelling of  $14,62 \pm 2,08 \%$  has been measured. The difference between both composites is because of the exposure time 3,4 times higher for the GF composite. The main reason is, of course the type of fibers. Indeed, as said previously the flax fiber has a hydrophilic behavior much more pronounced than the glass fiber. Furthermore, it is believed that the fiber content is playing a role as well. The diffusion of water in the epoxy matrix is greater than in the glass fibers. Therefore, with 80% fiber content, the water is suspected to not diffuse as easily as in the GF composite. In the Flax/epoxy composite, a high internal load is suspected because of the swelling of these fibers. The flax/epoxy specimens were also twisted due to the water absorption (Appendix 11).

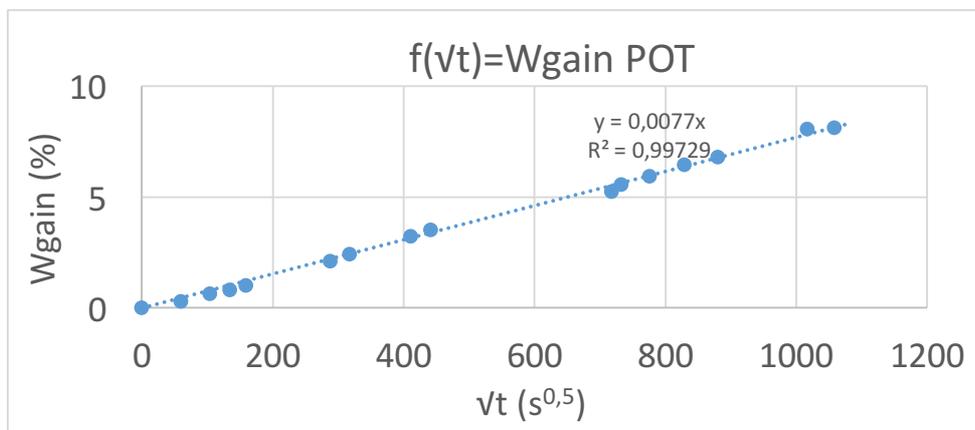
## IV.2.2 Diffusion coefficient and moisture content equilibrium



Graph 1: Water content percentage in function of the square root of time for Bath 70°C and RT exposure

Table 4: Determination of the diffusion coefficient

N°	GF/GG	70/RT	$Wt_{\text{av}}$ (mm)	$L_{\text{av}}$ (mm)	$b_{\text{av}}$ (mm)	Slope	$M_e$ (%)	Diffusion coefficient $D$ ( $\text{mm}^2/\text{s}$ )	Diffusion coefficient with edges effect $D_x$ ( $\text{mm}^2/\text{s}$ )
1	GF	70	1,90	19,99	9,93	0,0525	14,8	$8,92 \cdot 10^{-6}$	$5,39 \cdot 10^{-6}$
2	GF	70	1,88	200	19,85	0,0421	14,8	$5,65 \cdot 10^{-6}$	$4,64 \cdot 10^{-6}$
3	GF	RT	1,80	20	9,97	0,0155	14,8	$6,98 \cdot 10^{-7}$	$4,32 \cdot 10^{-7}$
4	GF	RT	1,89	200	19,87	0,0134	14,8	$5,75 \cdot 10^{-7}$	$4,70 \cdot 10^{-7}$
5	GG	70	1,62	19,97	9,77	0,0061	2,5	$3,07 \cdot 10^{-6}$	$1,98 \cdot 10^{-6}$
6	GG	70	1,62	200	19,86	0,0046	2,5	$1,75 \cdot 10^{-6}$	$1,47 \cdot 10^{-6}$
7	GG	RT	1,66	200	19,97	0,0008	2,5	$5,54 \cdot 10^{-8}$	$4,65 \cdot 10^{-8}$



Graph 2: Water content percentage in function of the square root of time for POT exposure

Tableau 5: determination of diffusion coefficient for POT

	GF/GG	70/RT	$W_{t_{av}}$ (mm)	Slope	$M_e$ (%)	Diffusion coefficient $D_x$ (mm <sup>2</sup> /s)
<b>POT</b>	GF	RT	1,87	0,0077	14,8	<b><math>7,43 \cdot 10^{-7}</math></b>

As can be seen, all the specimens have a linear water absorption at the beginning which slows down and then reaches the saturation level. The diffusion process can be described as Fickian.

According to the Graph 1, the saturation level for the composite GF is 14,8% (Graph 1) at 333h of exposition in 70°C bath. This saturation is valid for all Flax/epoxy composites (Curve 1,2,3,4) because they will, eventually, reach this percentage. Therefore, it is used for the determination of the diffusion coefficient. However, the final saturation level could be slightly different in reason of the inconsistency of the flax fibers properties. Indeed, the flax fiber composition in cellulose, lignin and hemicellulose or the surface density can vary, depending on the conditions of plant's growing. Therefore, it can change from one specimen to another.

In comparison, the saturation level for Glass/epoxy composite (Curve 5,6,7) is about 2,5% for 166h exposure. This percentage has been taken as approximation because it is the last value observed even though the moisture content will slowly increase until a saturation level suspected to be slightly higher. These moisture content equilibriums are supposed to be the same for immersions at 70°C or at RT for the same composite. However, it is reached 11 times faster at a temperature of 70°C. This high temperature offers a reliable way to accelerate the water ageing and evaluate the saturation level.

The difference between the saturation levels of GG and GF composites is mainly caused by the fiber type and fiber content. Indeed, these results highlight the behavior extremely hydrophilic of the natural fiber. Moreover, the higher content of fiber (80%) in the GG composite plays a role in the slow water diffusion where the water can only diffuse in the matrix and at the interface. Furthermore, flax fibers have swelled by 14,6% creating internal stress and micro-cracks in the epoxy matrix and therefore, the diffusion was even greater. The internal stress applied by the swelling might also have an impact on the mechanical properties of the laminate. Also, Flax fibers have lost their original pigmentation because of their expansion as shown in pictures in Appendix 11.

In Graph. 2, the behavior of GF composite is shown for one-sided exposure. Unfortunately, by comparing the diffusion coefficient  $D_4$  and  $D_{x4}$  of  $n^{\circ}4$  (Table 4) with  $D_{X_{POT}}$  (Table 5), a higher deviation is reported between  $D_{x4}$  and  $D_{X_{POT}}$  than between  $D_4$  and  $D_{X_{POT}}$ . The diffusion coefficient  $D_{X_{POT}}$  is closer from the diffusion coefficient without edges effect correction  $D_4$ . Therefore, it is assumed that the POT doesn't protect from edge effects as expected.

### Calculations

The examples of calculations are for  $N^{\circ}1$  (Table 4) and POT (Table 5).

Eq.4, 5, 7 from page 20 are used.

$$D_1 = \pi \times \left( \frac{wt}{4 \times Me} \right)^2 \times Slope^2 = \pi \times \left( \frac{1,90}{4 \times 14,8} \right)^2 \times 0,0525^2 = 8,92 \times 10^{-6} \text{ mm}^2/s$$

$$D_{1x} = 8,92 \times 10^{-6} \times \left( 1 + \frac{1,90}{19,99} + \frac{1,90}{9,93} \right)^{-2} = 5,39 \times 10^{-6} \text{ mm}^2/s$$

$$D_{POT} = \pi \times \left( \frac{2 \times wt}{4 \times Me} \right)^2 \times Slope^2 = \pi \times \left( \frac{2 \times 1,87}{4 \times 14,8} \right)^2 \times 0,0077^2 = 7,43 \times 10^{-7} \text{ mm}^2/s$$

### IV.3 Effect of UV exposure

The specimens and samples of neat resin have been exposed to UV radiation for 504h hours at RT. The BS specimens were tested with tensile test and the neat resin was analyzed by Infrared Spectroscopy. No weigh changes induced by the UV have been recorded during the exposure (Appendix 12).

### IV.3.1 Infrared analysis

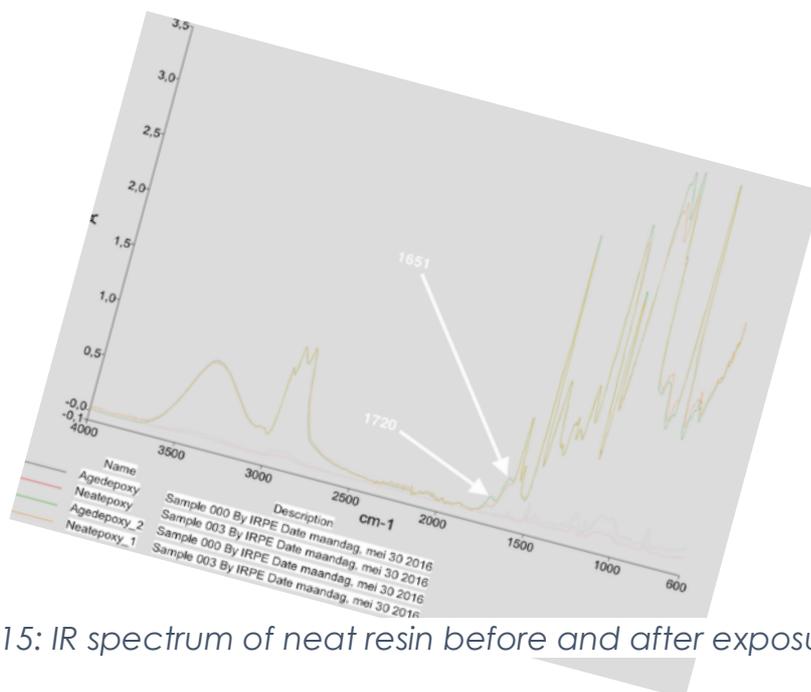


Figure 15: IR spectrum of neat resin before and after exposure

On the Infrared Spectrum (Figure 15), two differences are noticeable between the resin and the UV aged resin. Two additional peaks (1720 and 1651  $\text{cm}^{-1}$ ) are present in the spectrum of the aged resin. The exposure has been too short to see fundamental changes in the structure of the resin. According to (Nguyen, 2013), the peak at 1651  $\text{cm}^{-1}$  could be the signature of amides or quinone methide function. However, the latter's formation takes a longer time than the formation of amides from amines group (Reaction mechanism Appendix 3). The time of exposure being short, it is likely that the peak at 1651  $\text{cm}^{-1}$  belongs to the amide group. Concerning the peak at 1720  $\text{cm}^{-1}$ , it can be from both, carbonyls (1730-1740  $\text{cm}^{-1}$ ) and phenyl formate (1735  $\text{cm}^{-1}$ ). Only a further analysis could say more about this additional peak.

## IV.4 Evolution of mechanical properties

### IV.4.1 Tensile strength test

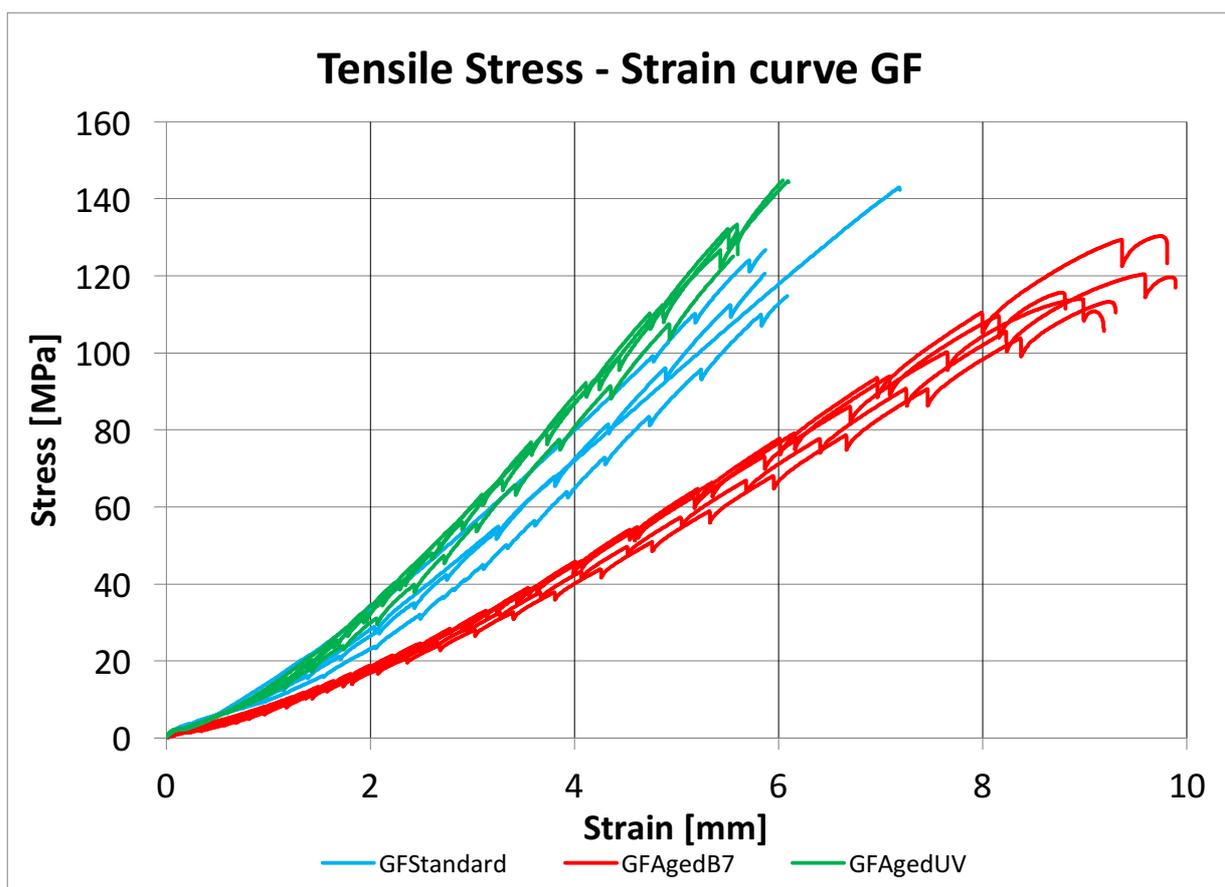


Figure 16: Tensile Stress - strain curve of Standard and Aged UV/B7 GF

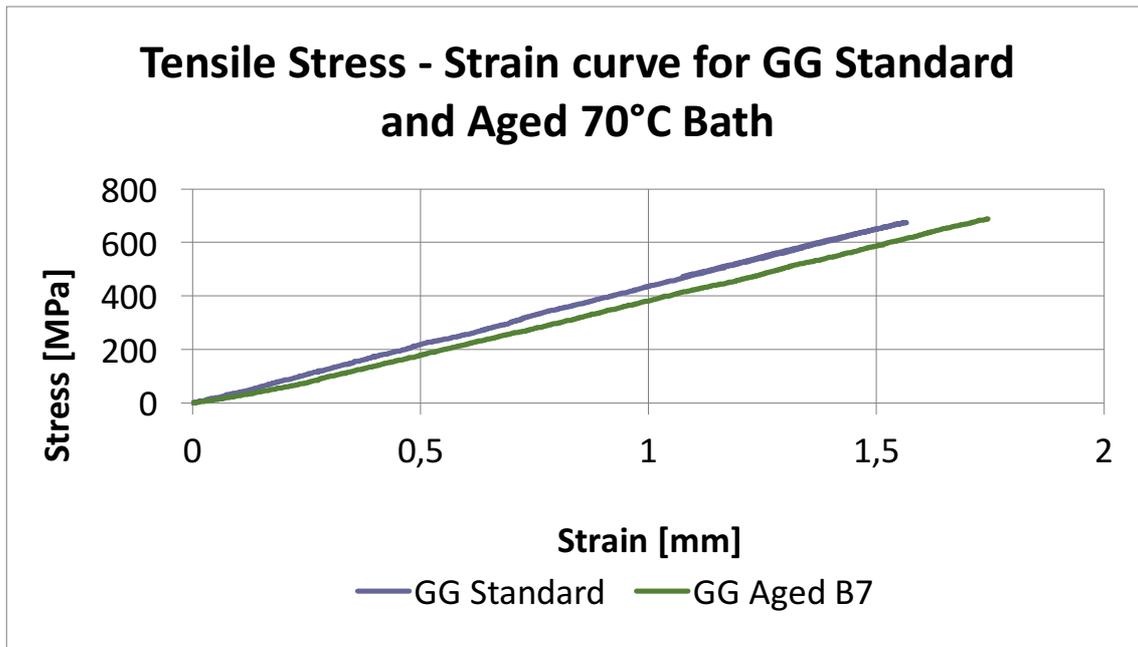


Figure 17: Tensile Stress - strain curve of Standard and Aged UV/B7 GG

Table 6: Values of tensile properties of GF composite

	$F_{max}$ (MPa)	$E_t$ (MPa)	$\epsilon_{max}$ (mm)
Standard	126	22	5,99
Aged UV	132	25	5,84
Aged B7	119	14	9,40

$F_{max}$ : Maximum tensile strength;  $E_t$ : Young's modulus;  $\epsilon_{max}$ : fracture strain

Concerning the UV ageing, an increase of 4,8% for the tensile strength and 13,6% for the Young's modulus was observed. However, the rupture elongation decreased about 2,5%. The UV radiations is suspected to have triggered a second reticulation of the matrix's structure resulting in a stronger tridimensional molecular network. This exposure is believed advantageous for a short period of time but will damage the composite if prolonged. The mechanical properties of the GF composite were impacted by the water ageing. Indeed, the tensile strength has decreased of 5,6% and the Young's modulus is 36,4% lower. The fracture strain however is 59,6% higher after ageing. It is believed that this diminution is due to the plasticization of the matrix and the fibers degradation that affects greatly the tensile strength. However, these values need to be taken carefully because the specimens were slipping from the tensile device's grips due to the non flat surface of flax/epoxy composite. It might has induced some errors in the test. The reason for the gap between the Young's modulus experimental value and the one found page 13 can be explained by the inexperienced of the operator with the vacuum infusion process.

Unfortunately, the GG composite couldn't be tested correctly on the 20kN tensile bench because the fracture point was not reached. Indeed, the strength needed to break the glass/epoxy composite was higher than 20 kN. Therefore, the results between standards and aged is not reliable. Just a subtle diminution of the modulus and a higher elongation can be observed. However, it is sufficient to say that the GG composite has better tensile properties than the GF composite with  $F_{max} = 675$  MPa and a Young's modulus of 436 MPa.

IV.4.2 Flexural strength test

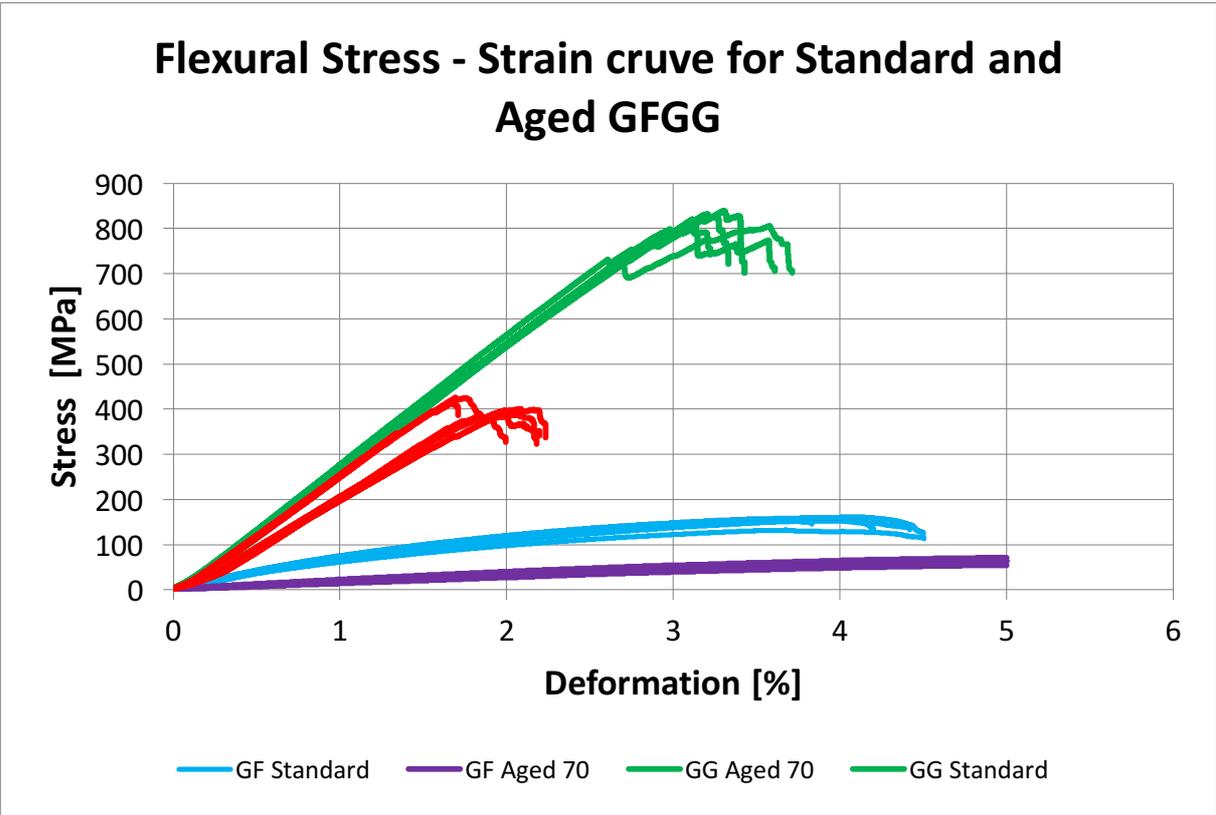


Figure 18: Flexural Stress - Strain curve for GF/GG composites

Table 7: Values of flexural properties of GF/GG composite

	$S_M$ (MPa)	$E_H$ (MPa)	$S_1$ (MPa)
<b>GF</b> standard	151	7534	68
<b>GF</b> Aged B7	64	1694	18
<b>GG</b> standard	819	22833	263
<b>GG</b> Aged B7	404	17426	222

$S_M$ : Maximal flexural strength;  $E_H$ : Flexural modulus;  $S_1$ : Flexural stress at 1% deformation.

According to Table 7, for the GF composite, the flexural strength and the flexural modulus decreases of 57,6% and 77,5% respectively. The  $S_1$  value gives information about the composite's flexibility, the stress is lower (64 MPa for standard against 18 MPa for Aged B7) for 1% deformation. It needs less energy to bend to the same point. The final deformation of the GF Aged composite is not available because the test was stopping at 5% deformation and it didn't break before. After ageing, the composite was too flexible to be tested by flexion test which is a test that is more suitable for brittle composite.

The ageing impacts the GG laminate as well. A 50,7% decrease for  $S_M$  and 24% for  $E_H$  were reported.  $S_1$  is also lower with a diminution of 15,6%. The specimens broke twice before when aged. This diminution might be because of a degradation of the interface fiber/matrix as the specimens failed by debonding of the fiber mat from the matrix. The type of failure occurs because the fiber content is too high (80%).

For flexural properties, the GG composite is stronger than the GF composite as standard but tends to be weakened more easily than the GF composite as the debonding occurs. The fact that the GF laminate was aged for a longer period than the GG laminate needs to be kept in mind.

IV.4.3 Short-Beam shear strength test

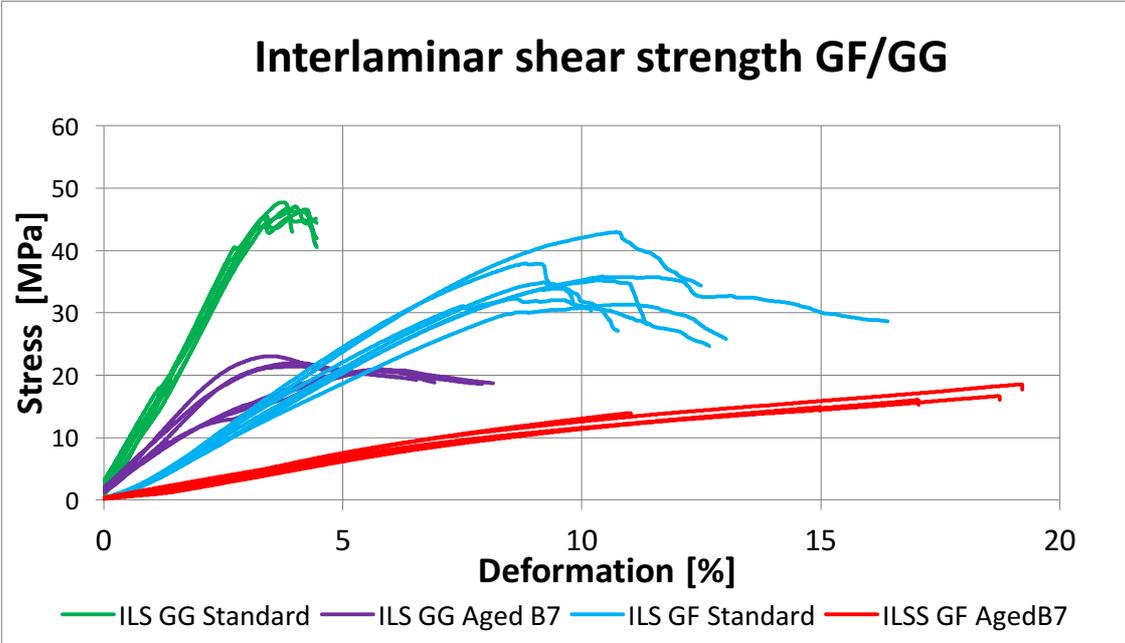


Figure 19: Interlaminar shear strength test for GF/GG composite

The results for GF composite are not interpretable for this test because the failure mode was a tensile failure mode (Appendix 5).

However, the GG composite failure mode was interlaminar. The interlaminar shear strength for the standard specimens is 10562 MPa against 5333 MPa for the aged one. This represents a 49,5% diminution. The ILSS test confirms the hypothesis that the interface fiber/matrix of the GG composite is greatly damaged during the ageing. The high fiber content is the main reason.

## V Conclusion

In this study, the manufacturing by vacuum infusion has been proved to be a good process to manufacture plate of regular dimensions. The GG composites fiber content was 80% and the GF composite fiber content was about 50%.

Furthermore, the saturation level for Bioepoxy/Flax composite and Bioepoxy/Glass have been found. They are equal to 14,8% and 2,5% respectively. The ageing in bath at 70°C has resulted in a satisfying acceleration in order to reach the saturation level for both composite. The experimental device "POT", however, didn't allow to suppress the edges effect and therefore to calculate a diffusion coefficient close to the reality. It has been proved that the water uptake caused a swelling of 14,6% in the flax composite, impacting the mechanical properties of this one. The mechanical properties have been greatly altered for both composites. The tensile properties were found to decrease with the water absorption for both composite. However, the flexural deformation of GF composite was highly increased but resulting in a very flexible material with no resistance to bending. In contrary, the water ageing damaged the interface of fiber/matrix for the GG composite.

The UV ageing increased the tensile properties of the epoxy/flax laminate because of a second reticulation and caused the formation of by-product as amides in the matrix.

Finally, the GG composite appears to have better mechanical properties than the GF composite as standard and a lower moisture saturation level. However, the interface glass/epoxy is weaker than the interaction between flax and epoxy. The difference in fiber contents have to be taken in account. Despite the interface degradation of GG composite, it is still preferable to use a glass/epoxy composite than a flax/epoxy composite in a humid environment.

## VI Recommendations

First of all, the composites should be made with the same fiber content to allow a more accurate comparison between them. The difference 80% for glass composite against 50% for the flax composite is too important and therefore impacts the water uptake.

Concerning the water uptake experiment, the composite should have been immersed at the same time in the beginning and withdrawn also, at the same time. It is believed that a longer time of exposure is needed to see if the saturation level stays intact. Some phenomenon like hydrolysis might have occurred if the laminates were let longer in immersion.

The UV exposure should be made in a clean environment, free of dust or humidity to have a real insight on the weight change. It could also be interesting to combine the effect of water absorption, temperature and UV exposure resulting in accelerated ageing.

A very interesting experiment would be to mechanically test the specimens at each water content measurements in order to follow the degradation of the mechanical properties. However, it requires a well-equipped laboratory and an important number of specimens if they are tested by tensile, flexural and ILSS.

The mechanical properties could also have been measured after a desorption period for the specimens to dry and, therefore, allows to see if the diminution of the properties is reversible. The tensile fracture would have been interesting to investigate using the Scanning Electron Microscopy technology.

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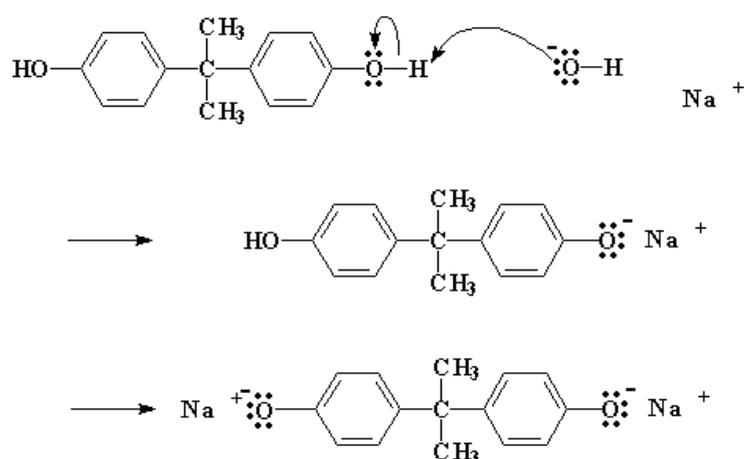
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## VIII Appendices

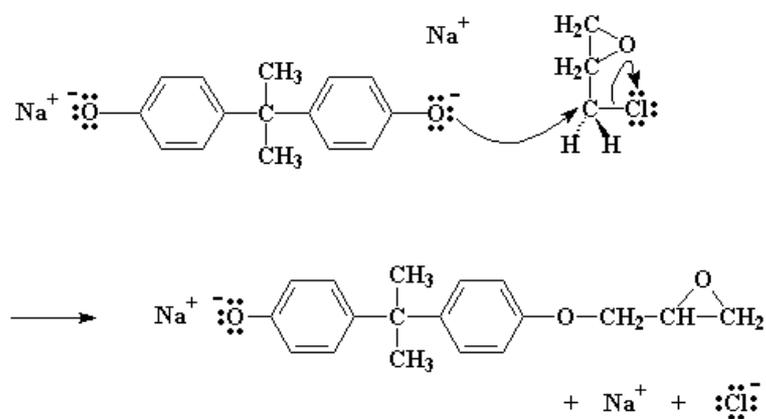
### Appendix 1

Here under, the detailed reaction mechanism for DGEBA and the curing mechanism.

Firstly, the nucleophilic OH group from the sodium hydroxide attacks the partially positively charged hydrogen at the end of the Bisphenol A. Water is formed. It is due to the high electronegativity of the oxygen atom. It results in a sodium salt of Bisphenol A.

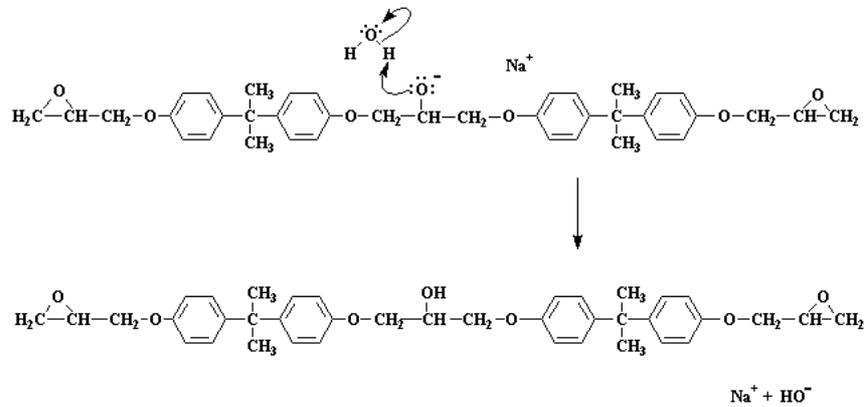


The chlorine atom is, as well as the oxygen, very electronegative. Therefore, in the epichlorohydrin molecule, the carbon atom attached to it, has a partial positive charge. The oxygen atom of the bisphenol A being nucleophilic replace the chlorine atom by nucleophilic substitution. A Bisphenol A molecule with an epoxy group is formed as well as a NaCl by-product.



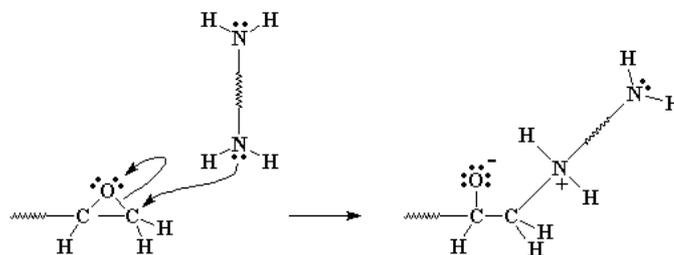


The water from the first step of the reaction will react with this nucleophilic oxygen. As a result, the oxygen forms an alcohol group and the sodium hydroxide catalyst is formed again. The prepolymer DGEBA is synthesized with a maximum degree of polymerization equal to 25 depending on the amount of Bisphenol A and epichlorohydrin introduced at the beginning.

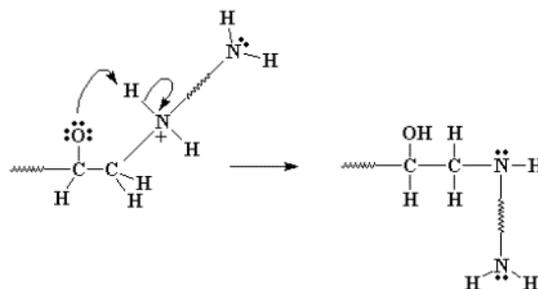


## Curing mechanism

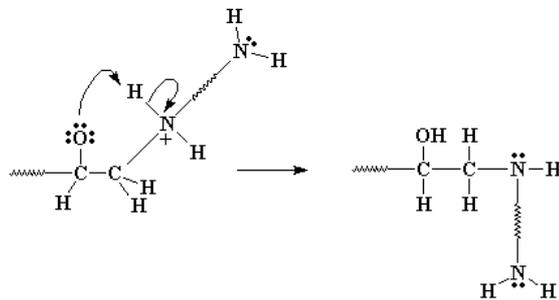
The first mechanism is the attack of the less saturated carbon of the epoxy by the amine's lone pair of electrons. Resulting in a positively charged nitrogen atom and a nucleophilic oxygen atom.



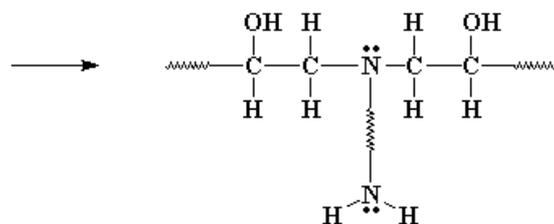
Still part of the first mechanism, this oxygen reacts with the hydrogen of the amine function. As a result, a secondary amine and secondary alcohol groups are created from the epoxy ending group of DGEBA.



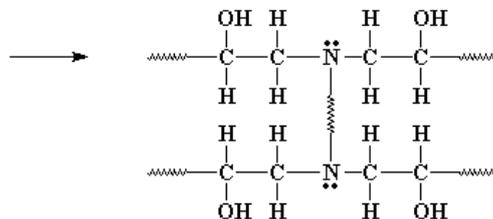
Still part of the first mechanism, this oxygen reacts with the hydrogen of the amine function. As a result, a secondary amine and secondary alcohol groups are created from the epoxy ending group of DGEBA.



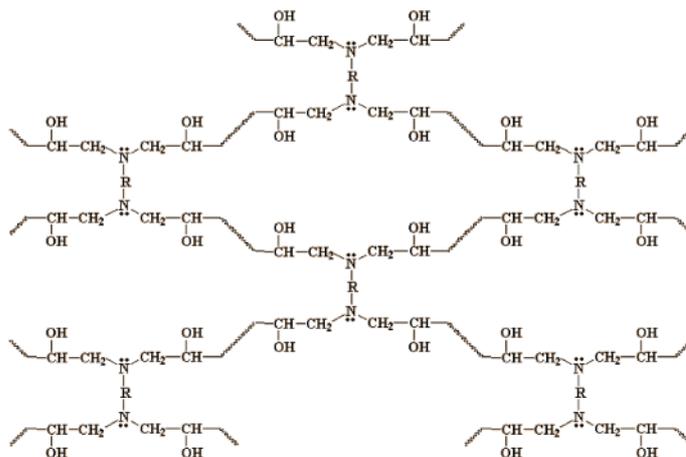
The amine group still has a hydrogen left. Therefore, it reacts with another epoxy group as well.



In turn, the other amine group of the diamine molecule reacts the same way.

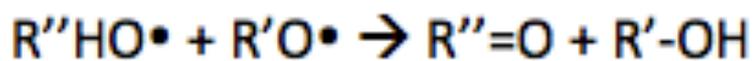
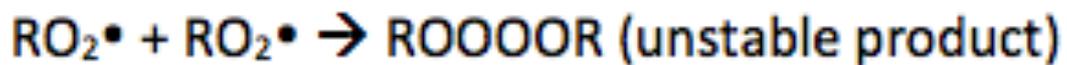
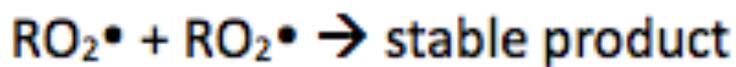
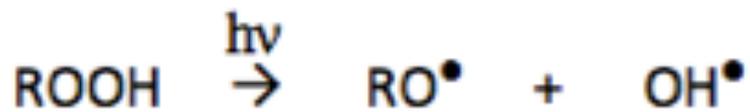
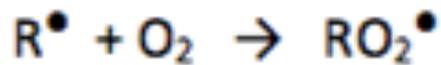
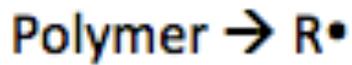


As long as there are epoxy groups and amine groups with labile hydrogens the reaction goes on building up the molecular chain.



## Appendix 2

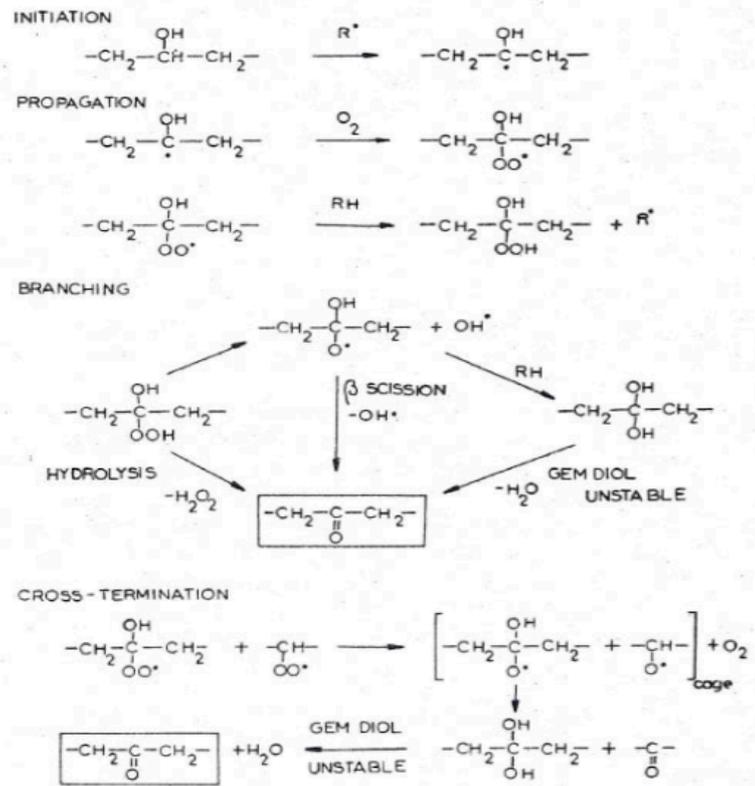
Mechanism of Photo-oxidation



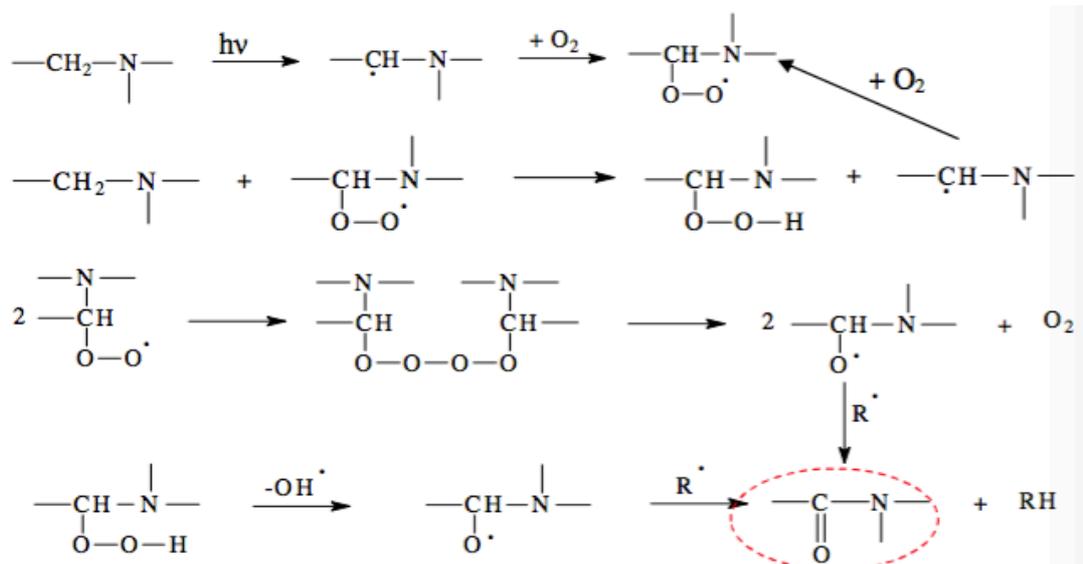
## Appendix 3

Hereunder are displayed all the reaction mechanisms linked to the UV exposure.

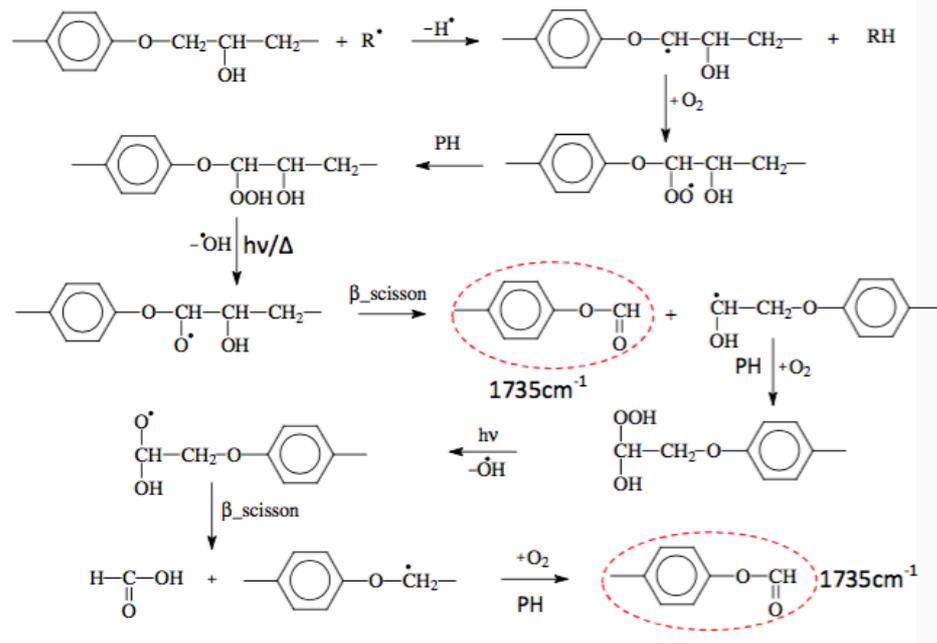
Mechanism of formation of carbonyl groups from secondary hydroxyl groups



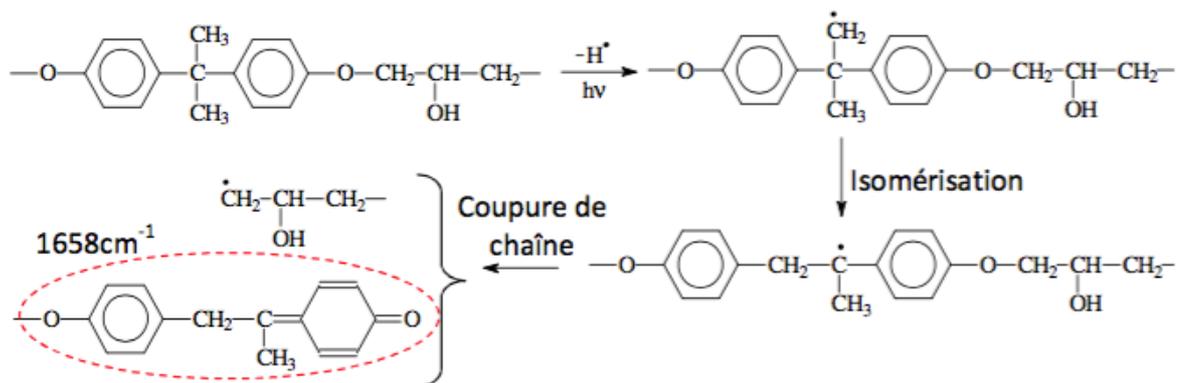
Mechanism of formation of amides



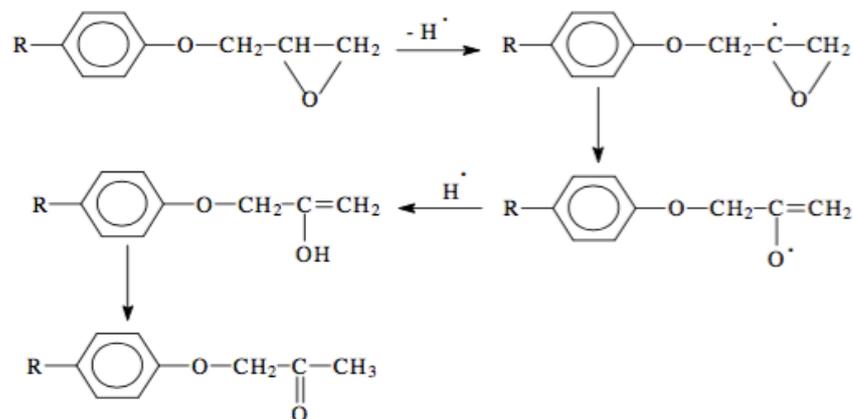
### Mechanism of phenyl formate formation



### Mechanism of quinone methide formation

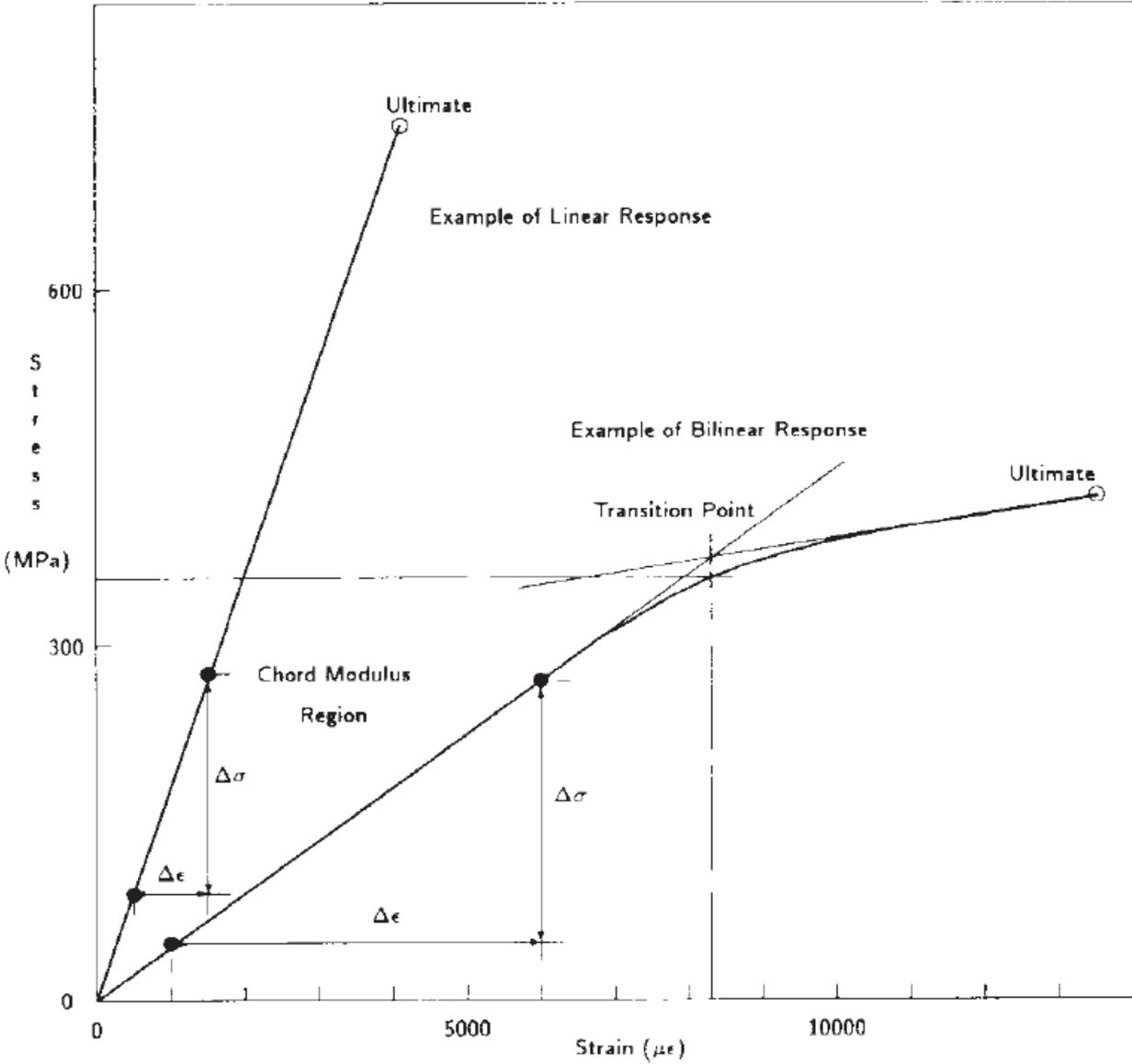


### Mechanism of oxirane ring opening



# Appendix 4

Example of stress strain curve for the tensile test.



## Tensile test failure modes



LIT



GAT



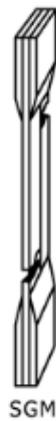
LAT



DGM



LGM



SGM



AGM(1)



AGM(2)



XGM

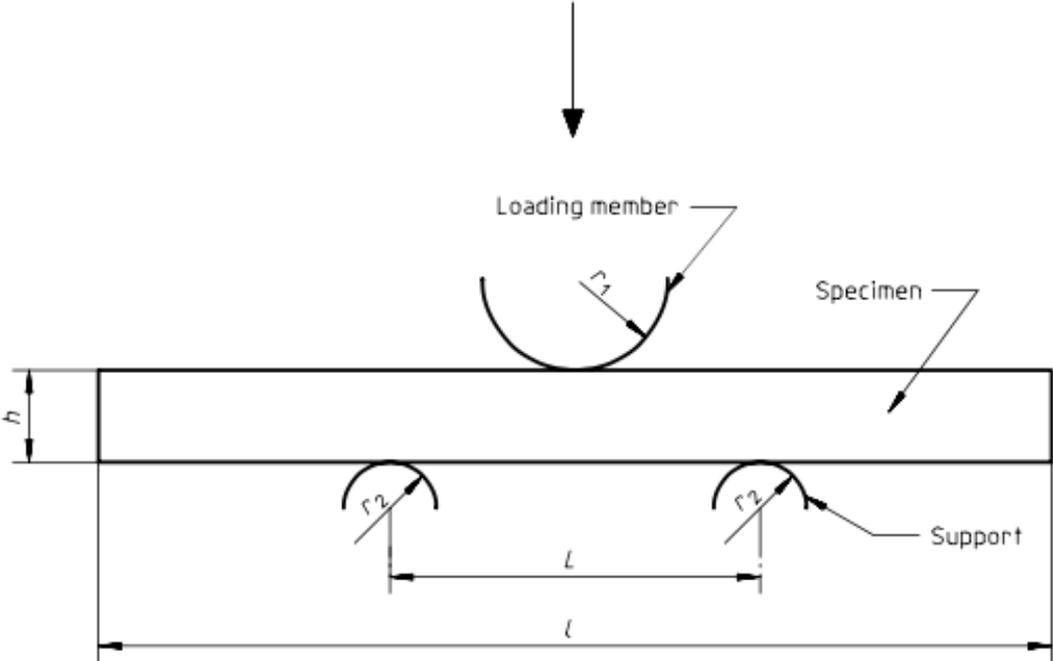
First Character	
Failure Type	Code
Angled	A
edge Delamination	D
Grip/tab	G
Lateral	L
Multi-mode	M(xyz)
long. Splitting	S
eXplosive	X
Other	O

Second Character	
Failure Area	Code
Inside grip/tab	I
At grip/tab	A
<1W from grip/tab	W
Gage	G
Multiple areas	M
Various	V
Unknown	U

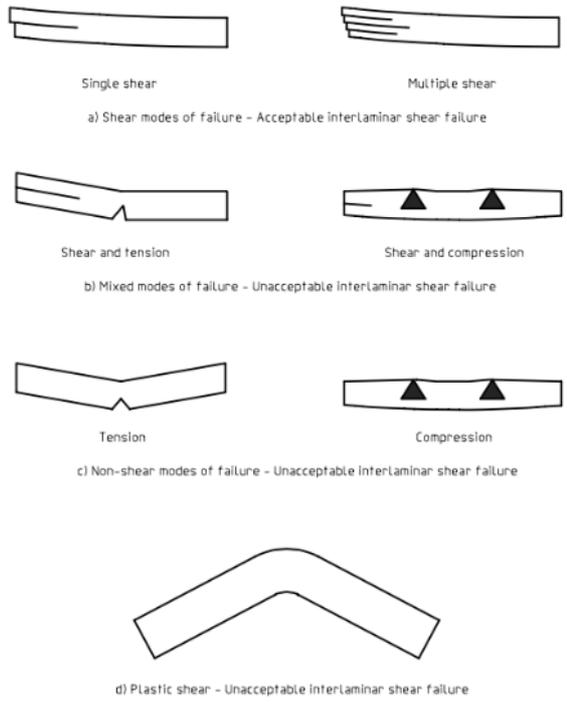
Third Character	
Failure Location	Code
Bottom	B
Top	T
Left	L
Right	R
Middle	M
Various	V
Unknown	U

# Appendix 5

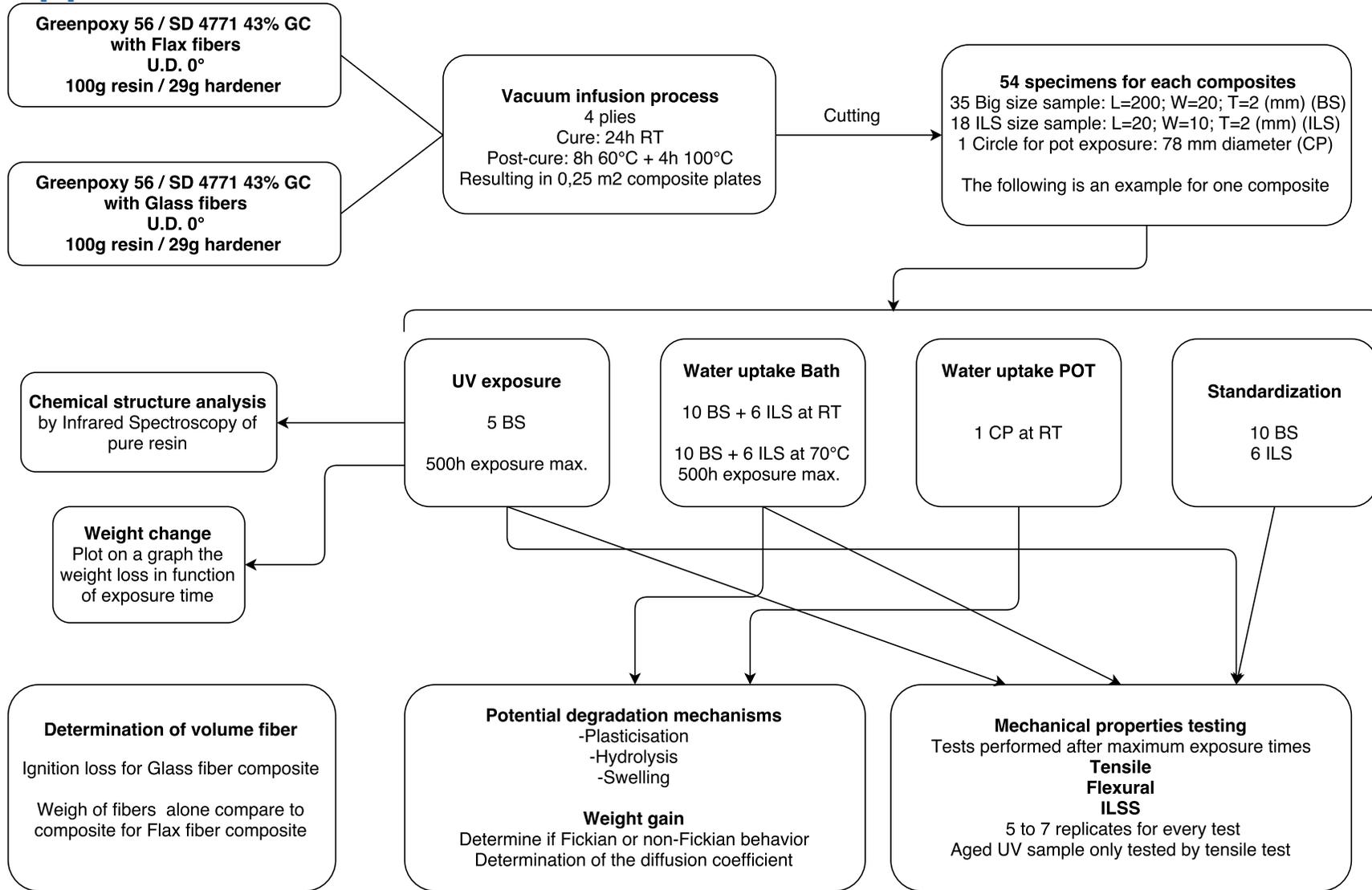
Representation of a short beam strength test



Potential modes of failure during a interlaminar shear strength test



# Appendix 6



## Appendix 7

### Protocol for composites manufacturing

<b>Preparing the glass plate</b>
- Clean residue on the plate
- Wax plate for easier unmolding
<b>Preparing the laminates</b>
- Cut fibers to required dimensions
- Dry fibers at 70°C for 12 hours
- Cool fibers for 15 minutes
- Place fibers on the plate
<b>Preparing the resin infusion</b>
- Apply tacky tape
- Apply peel ply
- Apply flow mesh
- Connect resin transfer tube
- Place vacuum bag
- Apply vacuum
- Check for leak
<b>Preparing the matrix</b>
- Weigh amount of resin
- Mix with correct ratio of hardener (100g/29g)
- Degassing of the resin
<b>Infusion</b>
- Connect resin pot to inlet
- Let until complete infusion of the fibers
- Disconnect feed pot
<b>Curing</b>
- Let vacuum for 24h
- Unmold the plate
- Post-cure for 8h at 60°C
- Post-cure for 4h at 100°C

## GREENPOXY 56 High Biomass Carbon Content

The system **GREENPOXY 56** is out coming from the latest innovations in green chemistry.

The system **GREENPOXY 56** is a produce with a high content of carbon from vegetable origin.

The Green Carbon content of our system is certified by an independent laboratory using Carbon 14 measurements.

This is a significant technological advance on the following points: clarity, color, performance and guarantees of industrial tonnages availability.

SICOMIN obtains a rate of 56% of the molecular structure from plant origin, with the pair SR **GREENPOXY 56** / SD GP 505 v2.

Other systems **GREENPOXY 56**, SD XXXX are possible (please see TDS).

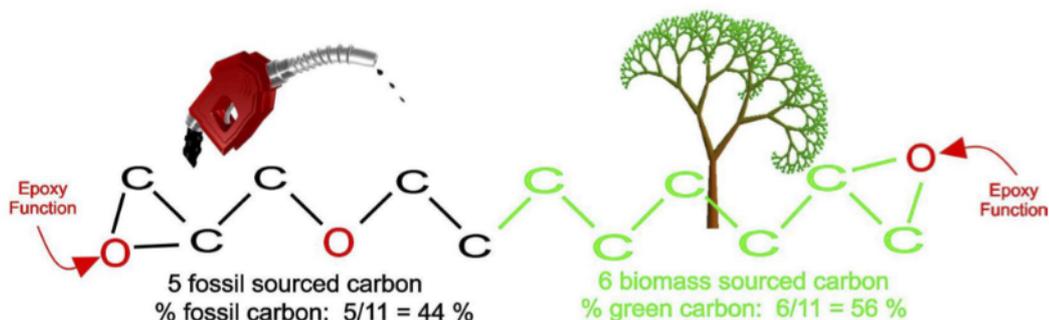
The rate of final Green carbon content will depend on the hardener choice.

SR **GREENPOXY 56** is an epoxy resin which has 56% of its molecular structure coming from plant origin.

This percentage is function of the origin of the carbon contained in the molecule.

Below, as shown in this hypothetical molecule of 11 carbons, 6 being coming from green chemistry, so it contains 56% green carbon.

Hardener SD GP 505 v2 follows the same logic and contains the maximum rate possible of biomass carbon.



**GREENPOXY 56** has an average viscosity enabling the manufacturing of various parts

**Applications**

- Hand lamination for tooling or industrial parts
- RTM processes (infusion, injection...)
- Filament winding
- Hot or cold press
- Casting
- Bonding

**SR GREENPOXY 56 Resin**

Aspect / colour		Yellow liquid
Chemical nature		Epoxy
Storage		2 years, cristalisation free
Viscosity (mPa.s)	15 °C	2500 ± 500
Rheometer CP 50 mm	20 °C	1400 ± 280
Shear rate 10 s <sup>-1</sup>	25 °C	800 ± 160
	30 °C	500 ± 100
	40 °C	250 ± 50
% Green Carbon		56 ± 2
Color (Gardner) ISO 4630		2 max
Density NF EN ISO 2811-1	20 °C	1.198 ± 0.005
Refractive index DIN 514423-2	25 °C	1.535 + 0.002

**SD GP 505 V2 Hardener**

Aspect / colour		Reddish yellow liquid
Typical reactivity		Fast
Viscosity (mPa.s)	15 °C	1800 ± 360
Rheometer CP 50 mm	20 °C	1200 ± 240
Shear rate 10 s <sup>-1</sup>	25 °C	820 ± 165
	30 °C	580 ± 115
	40 °C	320 ± 60
% Green carbon		58 ± 3
Color (Gardner) ISO 4630		17 max
Density NF EN ISO 2811-1	20 °C	0.986 ± 0.005

### SR GREENPOXY 56 / SD GP 505 V2 Mix Properties

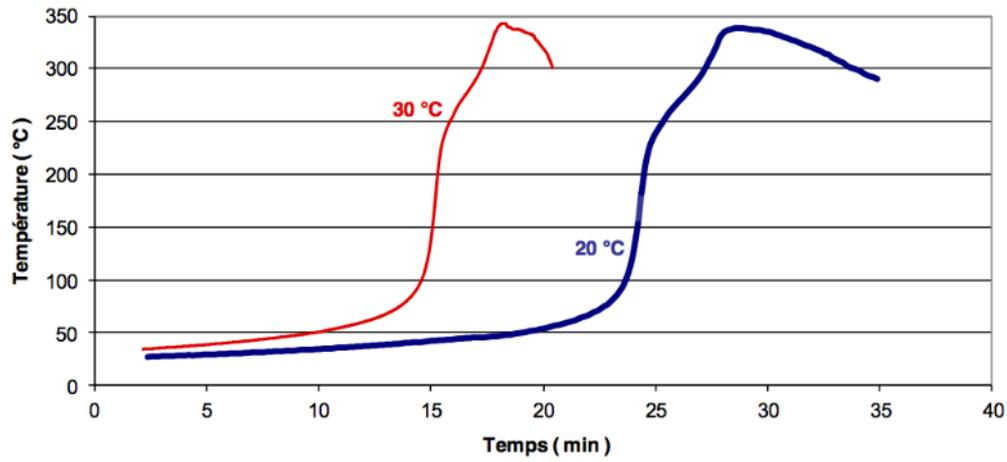
Weight ratio		100 / 42 g
Volume ratio		100 / 50 ml 2 / 1
% Green carbon		> 56 %
Mix viscosity (mPa.s)		
Rheometer CP 50 mm	20 °C	1400 ± 280
Shear rate 10 s <sup>-1</sup>	30 °C	780 ± 160

### Reactivity – Mass Exotherm On 500 G Mix

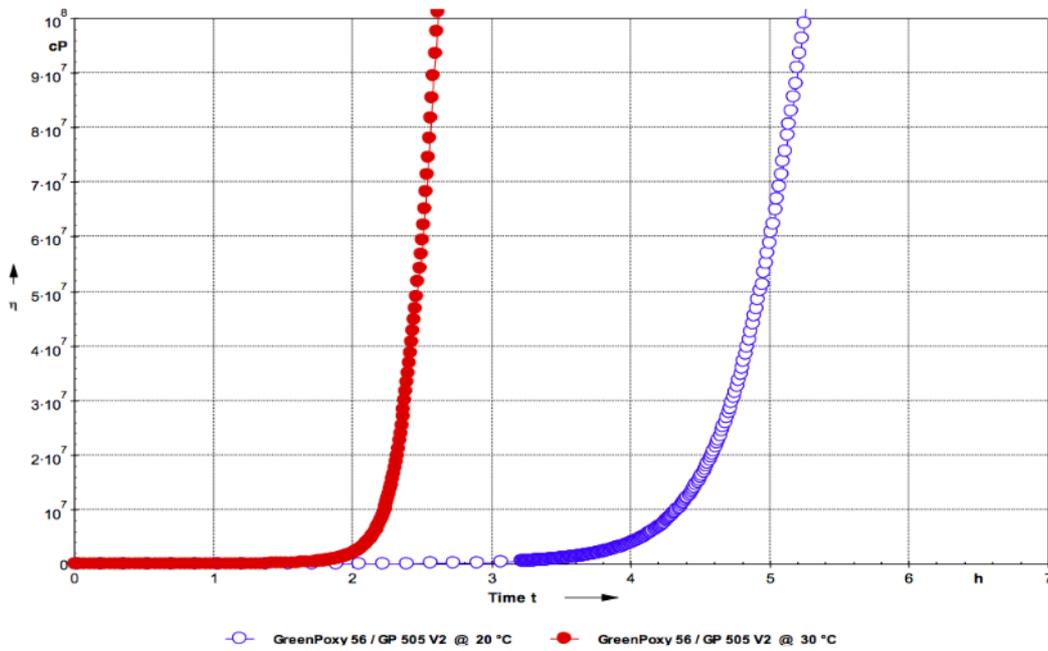
SR GREENPOXY 56 / SD GP 505 V2		
Exothermic peak:		
	20 °C	340 °C
	30 °C	340 °C
Time to reach exothermic peak :		
	20 °C	28'
	30 °C	18'
Time to reach 50 °C ::		
	20 °C	19'
	30 °C	10'

**Exotherms on 500 g mix @ 20 et 30 °C:**

NB: Large casting develop very exotherm temperature and high smokes densities



**Reactivity – 1 mm film viscosity evolution**



**Gel time on 1 mm thickness:**

@ 20 °C            3 h 20'  
 @ 30 °C            1 h 45'

Gel time: intersection G' G'' / rheometer CP50 - Shear rate 10 s<sup>-1</sup>

### Other possibilities

	Mixing By weight	% C Green	Tg 1 maximum or Onset (°C)	Best use
<b>SR GREENPOXY 56 / SD Surf Clear</b>	100 / 37	41	SD SC : 75	Clear laminates
<b>SR GREENPOXY 56 / SD Glass One</b>	100 / 42	40	SD GO: 69	Clear laminates
<b>SR GREENPOXY 56 / SD GP 505</b>	100 / 47	56	DP 505: 71	
<b>SR GREENPOXY 56 / SD 1213</b>	100 / 50	37	1213 : 43	Large clear casting
<b>SR GREENPOXY 56 / SD 280x</b>	100 / 37	41	2806 : 66 2803 : 72 2801 : 80	Multipurpose
<b>SR GREENPOXY 56 / SD 477x</b>	100 / 29	43	4775: 80 4771: 74	Multipurpose
<b>SR GREENPOXY 56 / SD 550x</b>	100 / 37	41	5505 : 78 5503 : 85 5502 : 84	Multipurpose
<b>SR GREENPOXY 56 / SD 597.20</b>	100 / 21	46	597.20 : 100	Very large casting
<b>SR GREENPOXY 56 / SD 720x</b>	100 / 37	41	7206 : 84 7203 : 82 7201 : 80	Multipurpose
<b>SR GREENPOXY 56 / SD 860x</b>	100 / 37	41	8605 : 67 8601 : 56	Multipurpose
<b>SR GREENPOXY 56 / SD 882x</b>	8824 100 / 21 8822 100 / 31	46 43	8824 : 90 8822 : 71	Infusion

### Mechanical properties on pure cast resin

		GREENPOXY 56 / GP 505 V2	GREENPOXY 56 / GP 505 V2	GREENPOXY 56 / GP 505 V2
<b>Curing cycle</b>		7 days 23 °C	24 h 23 °C + 24 h 40 °C	24 h à 23 °C + 8 h 60 °C
<b>Tension</b>				
Modulus of elasticity	N/mm <sup>2</sup>	3 100	3 100	2800
Maximum resistance	N/mm <sup>2</sup>	64	66	67
Resistance at break	N/mm <sup>2</sup>	64	66	67
Elongation at max. load	%	3.2	3.8	4.0
Elongation at break	%	3.2	3.8	4.1
<b>Flexion</b>				
Modulus of elasticity	N/mm <sup>2</sup>	2 900	2 800	2 700
Maximum resistance	N/mm <sup>2</sup>	99	96	95
Elongation at max. load	%	4,5	4,2	4,5
Elongation at break	%	6,5	4,9	5,2
<b>Charpy impact strength</b>				
Resilience	kJ/m <sup>2</sup>	14	21	13
<b>Glass transition</b>				
Tg1	°C	55	61	84 / 78
Tg1 maximum	°C	-	-	84

Tests carried out on samples of pure cast resin, without prior degassing, between steel plates.

Measures undertaken according to the following norms:

Tension: NF T 51-034

Flexion: NF T 51-001

Charpy impact strength: NF T 51-035

Water absorption: Internal. Polymerisation according to cycle, machining, weighing, time spent in distilled water at 70 °C / 48 hours, weighing 1 hour after emerging,

Glass transition DSC: ISO 11357-2: 1999 -5°C to 180°C under nitrogen gaz

Tg1 or Onset: 1st point at 20 °C/mn

Tg1 maximum or Onset: second passage

# Data sheet of SD 4771

## Hardeners SD 477x :

		<b>SD 4775</b>	<b>SD 4771</b>
Aspect / color		Clear Liquid	Clear to red Liquid
Gardner ASTM D 1544 Disc 4/30		5 maximum	12 maximum
Reactivity levels		Medium	Ultra slow
Viscosities (m.Pas ± 20 %)	@ 15 °C	285 ± 60	13 ± 3
	@ 20 °C	190 ± 40	11 ± 2.5
Rheometer CP 50 mm	@ 25 °C	130 ± 30	9 ± 2
Shear rate 10 s <sup>-1</sup>	@ 30 °C	95 ± 20	7 ± 1.5
	@ 40 °C	55 ± 10	5 ± 1
Density (g/cm <sup>3</sup> ± 0.005) Picnometer NF EN ISO 2811-1	@ 20 °C	1.01 ± 0.01	0.95 ± 0.01

## Epoxy systems SR 1138 / SD 477x

		<b>SR 1138 SD 4775</b>	<b>SR 1138 SD 4771</b>
Appearance cured		White solid	White solid
Weight ratio		100 g / 16 g	100 g / 16 g
Volume ratio		100 / 22 ml	100 / 23 ml
Viscosity (m.Pas)			
Rheometer	@ 20 °C	4 200 ± 840	1600 ± 320
CP 50 mm	@ 30 °C	2 300 ± 460	1 000 ± 200
Shear rate 10 s <sup>-1</sup>	@ 40 °C	1 750 ± 350	370 ± 75
Density	@ 20 °C	1.33 ± 0.01	1.33 ± 0.01

**Biotex Flax**



**Biotex Flax 275g/m<sup>2</sup> Unidirectional**

High performance unidirectional fabric for automotive, sporting goods and decorative applications



## **Biotex Flax** 275g/m<sup>2</sup> Unidirectional

Biotex Flax provides high levels of performance, coupled with the ease of processing normally associated with glass-reinforced materials. The materials use twistless technology to provide a combination of sustainability, performance and processability. Compared to glass fibre composites, Biotex Flax offers reduced weight, improved environmental impact, vibration damping, similar specific stiffness and safer handling.

Biotex Flax is available in a range of yarn weights and fabric constructions. The materials can be processed using standard composites manufacturing techniques and are suitable for semi-structural and decorative applications in a range of sectors, including automotive, sports & leisure, consumer goods and construction.

Biotex Flax 275g/m<sup>2</sup> Unidirectional fabric is typically used for high-performance applications in sporting goods, automotive and general purpose components.



The information provided here is believed to be accurate but should be considered indicative only. It is the responsibility of the customer to check the suitability of the product for their specific application prior to use.

## Specifications

Weave Style	Unidirectional
Fabric Weight	275 g/m <sup>2</sup>
Width	330 mm
Typical Ply Thickness	0.3-0.6 mm, depending on process

## Processing

Typical processes for Biotex Flax fabrics include vacuum infusion or resin transfer moulding using either standard resins or bio-based resins. The fabrics can also be pre-pregged, and processing is carried out in the same way as glass fibre.

## Mechanical Properties

Typical mechanical properties of moulded laminates

Fibre Volume Fraction	27%	
Density	1.28 g/cm <sup>3</sup>	
Tensile Modulus	18.8 GPa	ISO 527-4
Tensile Strength	174 MPa	ISO 527-4
Elongation	1.5%	ISO 527-4
Flexural Modulus	15.1 GPa	ISO 14125
Flexural Strength	196 MPa	ISO 14125

Data from vacuum infused laminates using unsaturated polyester resin, tested at ambient temperature.

## Safety

Biotex Flax reinforcements are based on renewable biomass and have fewer health and safety concerns than many conventional alternative materials. However, typical precautions should be taken when handling the material including using appropriate PPE and adequate ventilation.



# Appendix 8

Dimensions and design of the POT

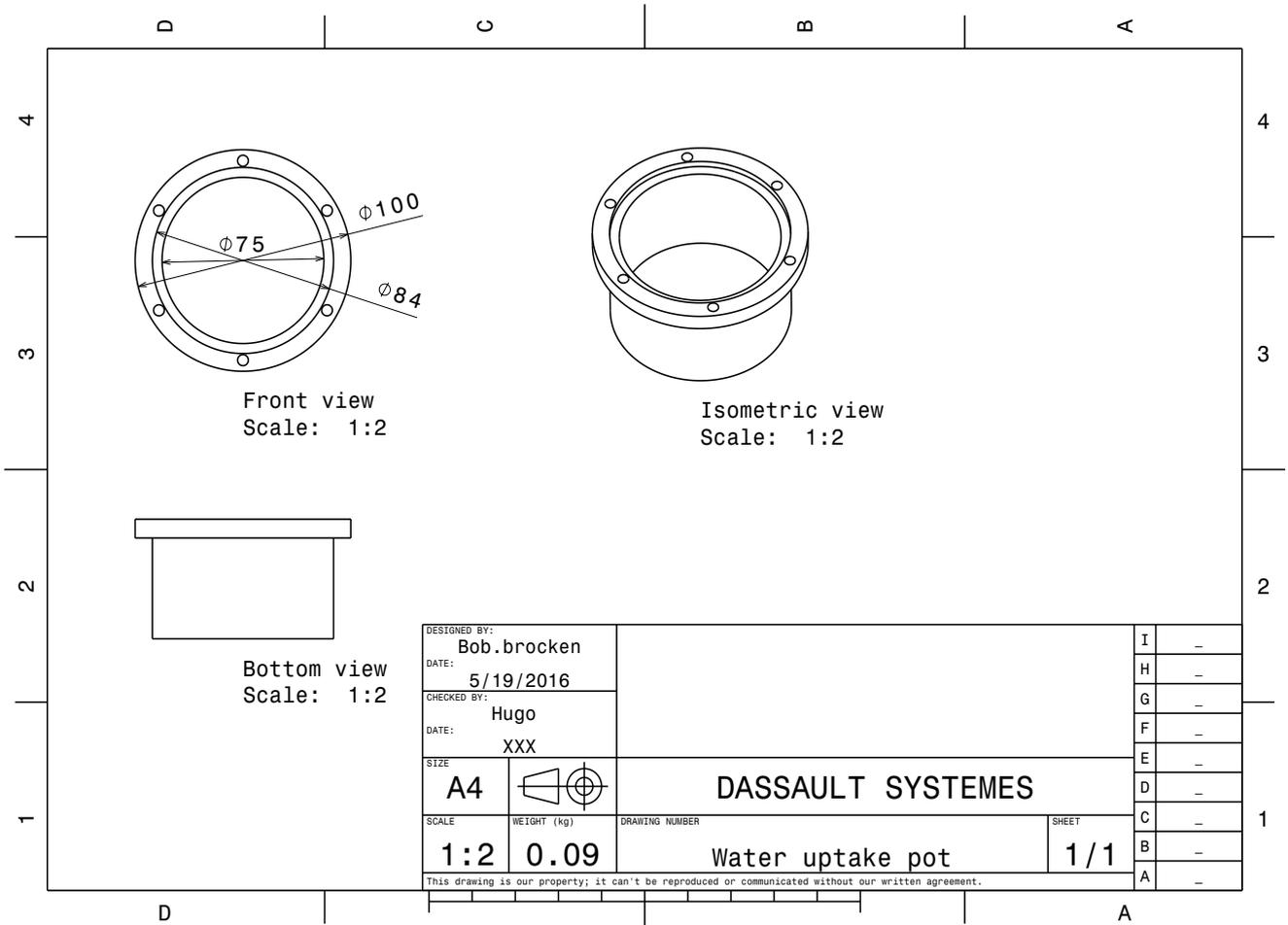
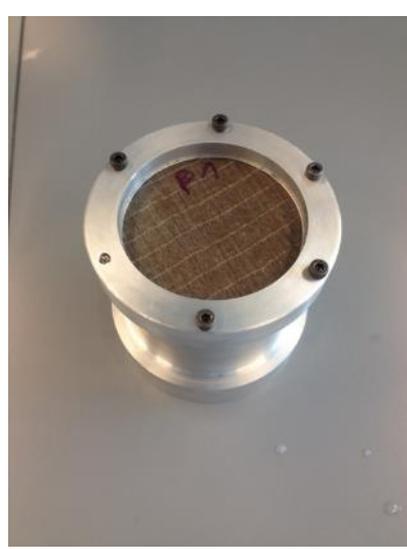


Photo of POT with specimen



# Appendix 9

## QLab UVA-340 Lamp Datasheet

TECHNICAL BULLETIN LU-8160



### A Choice of Lamps for the QUV Accelerated Weathering Tester

Your exposure application determines which type of UV lamps should be used. All of the QUV<sup>®</sup> accelerated weathering tester's lamps emit mainly ultraviolet rather than visible or infrared light. All are electrically equivalent to an ordinary 40-watt fluorescent. However, each lamp type differs in the total amount of UV energy emitted and in its wavelength spectrum. Fluorescent UV lamps are usually categorized as UVA or UVB lamps, depending on the region into which most of their output falls.

#### UVA Lamps

UVA lamps are especially useful for comparing different types of polymers. Because UVA lamps do not have any UV output below the normal solar cutoff of 295 nm, they usually do not degrade materials as fast as UVB lamps. However, they usually provide better correlation with actual outdoor weathering.

**UVA-340.** The UVA-340 provides the best possible simulation of sunlight in the critical short wavelength region from 365 nm down to the solar cutoff of 295 nm. Its peak emission is at 340 nm. UVA-340 lamps are especially useful for comparison tests of different formulations.

**UVA-351.** The UVA-351 simulates the UV portion of sunlight filtered through window glass. It is most useful for interior applications, testing of some inks and for polymer damage that can occur in an environment near a window.

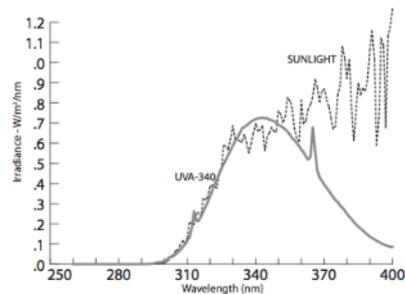
#### UVB Lamps

UV-B radiation includes the shortest wavelengths of sunlight found on the earth's surface. Consequently, fluorescent UVB lamps are widely used in QC and R&D for fast, cost-effective results. Because all UVB lamps emit unnatural, short-wavelengths of UV that are below the solar cutoff of 295 nm, anomalous results can occur. Two types of UVB lamps are available. They emit different amounts of total energy, but produce the same UV wavelengths in the same relative proportions.

**UVB-313EL.** The UVB-313EL gives fast results and it is particularly useful for QC and R&D applications and for durable materials. Compared to the QFS-40 lamp, the UVB-313EL produces substantially higher UV output, faster test results and is very stable.

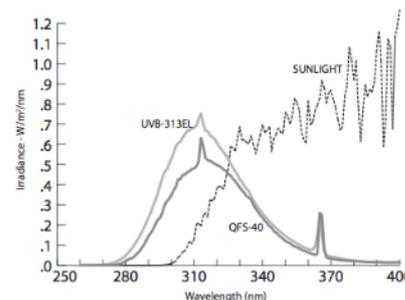
The UVB-313EL replaces the UVB-313, and offers more output and stability. The SOLAR EYE<sup>®</sup> irradiance controller can be used to decrease the output of the UVB-313EL to mimic the irradiance of a QFS-40. This allows longer lamp life and minimizes lamp replacement costs.

UVA-340 Lamps vs. Sunlight



UVA-340 lamps are the best available simulation of sunlight in the critical short-wave UV region.

UVB Lamps vs. Sunlight



UVB lamps produce mostly short-wave UV for maximum acceleration.

**QFS-40.** Also known as FS-40 or F40 UVB, this is the original QUV tester lamp. FS-40 lamps have been used for many years, and are still specified in many automotive test methods, particularly for coatings. QFS-40 should only be used in the QUV/basic model.

**Other Manufacturers.** Other UV lamps may be available from other manufacturers. These are often intended to be copies of Q-Lab's fluorescent lamps. Although the names may be the same (e.g., UVB-313 or UVA-340), other manufacturers' lamps may have very different irradiance, spectral power distribution or aging characteristics. Consequently, they may not give the same test results. For best results, use only Q-Lab lamps in your QUV weathering tester.

**General Lamp Recommendations**

<b>UVA-340</b>	Especially useful for comparison tests of different formulations. Recommended for correlation with outdoor results for most plastics, textiles, coatings, pigments and UV stabilizers.
<b>UVB-313EL</b>	Best for QC and R&D applications. Recommended for durable materials such as roofing, some exterior coatings etc.
<b>QFS-40 (F40 UVB)</b>	Automotive exterior coatings specifications.
<b>UVA-351</b>	Most useful for UV "sunlight through glass" simulations. Recommended for some automotive interiors, textiles and inks.

**IMPORTANT: DO NOT MIX DIFFERENT TYPES OF LAMPS**

Mixing different types of lamps in one QUV will produce major inconsistencies in the light falling on the samples, and may produce samples with "stripes" of greater and lesser degradation.

**SOLAR EYE Irradiance Control**

Models QUV/se and the QUV/spray are equipped with SOLAR EYE Irradiance Control. The controller continuously monitors the UV intensity using four sensors at the same plane. The feed-back loop systems allows it to auto-matically compensate for lamp aging or any other variability by adjusting power to the lamps. SOLAR EYE control allows better reproducibility and repeatability than manual irradiance control systems used in old-style QUV machines and the QUV/basic.

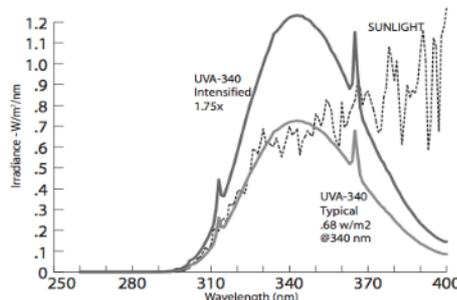
**High Irradiance**

With push-button irradiance setting, you can operate the SOLAR EYE control at various intensity levels for different applications and still maintain realistic test conditions. For example, with the UVA-340 lamps you could set the SOLAR EYE controller to simulate the following sunlight conditions:

Typical: For quick results without sacrificing correlation. With UVA 340 lamps, this irradiance level is equivalent to noon summer sunlight.

Intensified (1.75x): 75% higher than noon summer sunlight for fast test results.

**UVA-340 Lamp Intensified 1.75x & Typical UVA-340 Irradiance**



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# Appendix 10

## GF specimens' dimensions

Sample name	mm								
	T1	T2	T3	Thickness	Length	W1	W2	W3	Width
GF B7 1	2,04	1,90	1,97	1,97	200,00	19,85	19,86	19,83	19,85
GF B7 2	1,79	1,87	1,80	1,82	200,00	19,95	19,97	19,90	19,94
GF B7 3	2,02	1,85	1,89	1,92	200,00	19,85	19,86	19,83	19,85
GF B7 4	1,95	1,80	1,85	1,87	200,00	19,78	19,80	19,81	19,80
GF B7 5	1,89	1,87	1,77	1,84	200,00	19,77	19,77	19,87	19,80
GF B7 6	1,86	1,86	1,91	1,88	200,00	19,80	19,84	19,90	19,85
GF B7 7	2,05	1,80	1,81	1,89	200,00	19,85	19,81	19,86	19,84
GF B7 8	1,96	1,90	1,81	1,89	200,00	19,84	19,89	19,83	19,85
GF B7 9	1,85	1,87	1,82	1,85	200,00	19,85	19,84	19,86	19,85
GF B7 10	1,93	1,90	1,83	1,89	200,00	19,86	19,88	19,89	19,88
GF B7 1	1,99	2,03	1,87	1,96	20,06	9,87	9,95	9,84	9,89
GF B7 2	1,92	1,79	1,84	1,85	19,99	9,91	9,90	9,92	9,91
GF B7 3	1,84	1,96	1,91	1,90	20,02	9,87	9,92	9,90	9,90
GF B7 4	1,80	1,76	1,73	1,76	19,96	10,01	10,01	10,01	10,01
GF B7 5	1,83	1,87	1,92	1,87	19,93	9,91	9,90	9,89	9,90
GF B7 6	2,15	2,02	2,01	2,06	19,96	9,97	9,98	9,98	9,98
GF B 1	2,05	1,96	1,93	1,98	200,00	19,81	19,88	19,80	19,83
GF B 2	1,78	1,71	1,81	1,77	200,00	19,84	19,89	19,88	19,87
GF B 3	1,90	1,78	1,94	1,87	200,00	19,79	19,89	19,83	19,84
GF B 4	1,98	1,98	1,92	1,96	200,00	19,83	19,78	19,89	19,83
GF B 5	1,96	1,84	1,97	1,92	200,00	19,89	19,84	19,92	19,88
GF B 6	1,96	1,91	2,02	1,96	200,00	19,81	19,85	19,81	19,82
GF B 7	1,78	1,99	1,84	1,87	200,00	19,92	19,93	20,01	19,95
GF B 8	2,02	1,81	1,91	1,91	200,00	20,01	20,08	20,01	20,03
GF B 9	1,81	1,84	1,79	1,81	200,00	19,87	19,83	19,80	19,83
GF B 10	1,75	1,90	1,85	1,83	200,00	19,84	19,91	19,77	19,84
GF B 1	1,94	1,78	1,82	1,85	20,06	9,94	9,93	9,90	9,92
GF B 2	1,81	1,73	1,87	1,80	20,00	9,95	9,96	9,92	9,94
GF B 3	1,75	1,87	1,87	1,83	20,01	9,96	10,01	10,00	9,99
GF B 4	1,75	1,72	1,79	1,75	19,92	9,98	10,02	10,00	10,00
GF B 5	1,77	1,77	1,70	1,75	20,05	9,94	9,99	9,97	9,97
GF B 6	1,84	1,88	1,80	1,84	19,98	9,96	10,00	10,00	9,99
GF U 1	1,90	1,86	2,03	1,93	200,00	20,03	20,01	19,93	19,99
GF U 2	1,90	1,89	1,89	1,89	200,00	19,82	19,81	19,83	19,82
GF U 3	1,86	1,90	1,85	1,87	200,00	19,88	19,89	19,94	19,90
GF U 4	1,88	1,84	1,88	1,87	200,00	19,86	19,92	19,97	19,92
GF U 5	2,16	2,34	2,05	2,18	200,00	19,88	19,85	19,95	19,89

## GG specimens' dimensions

Sample name	mm								
	T1	T2	T3	Thickness	Length	W1	W2	W3	Width
GG B7 1	1,61	1,58	1,55	1,58	200,00	19,74	19,64	19,72	19,70
GG B7 2	1,68	1,70	1,71	1,70	200,00	19,87	19,81	19,80	19,83
GG B7 3	1,59	1,62	1,64	1,62	200,00	19,86	19,93	19,93	19,91
GG B7 4	1,56	1,56	1,60	1,57	200,00	19,75	19,74	19,84	19,78
GG B7 5	1,58	1,62	1,57	1,59	200,00	19,81	19,68	19,78	19,76
GG B7 6	1,68	1,66	1,66	1,67	200,00	19,76	19,76	19,80	19,77
GG B7 7	1,59	1,62	1,55	1,59	200,00	20,72	20,31	19,93	20,32
GG B7 8	1,64	1,61	1,62	1,62	200,00	20,09	19,97	19,85	19,97
GG B7 9	1,62	1,67	1,71	1,67	200,00	19,76	19,78	19,80	19,78
GG B7 10	1,55	1,61	1,61	1,59	200,00	19,76	19,71	19,77	19,75
GG B7 1	1,59	1,59	1,57	1,58	19,78	9,77	9,77	9,80	9,78
GG B7 2	1,65	1,52	1,62	1,60	19,78	9,76	9,80	9,80	9,79
GG B7 3	1,70	1,65	1,69	1,68	19,81	9,76	9,80	9,80	9,79
GG B7 4	1,67	1,68	1,68	1,68	19,78	9,74	9,77	9,80	9,77
GG B7 5	1,54	1,55	1,52	1,54	19,79	9,71	9,72	9,77	9,73
GG B7 6	1,69	1,69	1,59	1,66	19,78	9,78	9,77	9,82	9,79
GG B 1	1,59	1,57	1,61	1,59	200,00	19,77	19,77	19,73	19,76
GG B 2	1,59	1,55	1,54	1,56	200,00	19,74	19,64	19,77	19,72
GG B 3	1,64	1,63	1,67	1,65	200,00	19,94	20,03	20,05	20,01
GG B 4	1,60	1,59	1,55	1,58	200,00	19,86	19,86	19,96	19,89
GG B 5	1,98	1,91	2,08	1,99	200,00	19,83	19,76	19,81	19,80
GG B 6	1,65	1,75	1,79	1,73	200,00	19,77	19,68	19,82	19,76
GG B 7	1,62	1,54	1,67	1,61	200,00	19,70	19,72	19,63	19,68
GG B 8	1,81	1,60	1,63	1,68	200,00	19,77	19,72	19,81	19,77
GG B 9	1,57	1,61	1,63	1,60	200,00	19,76	19,77	19,75	19,76
GG B 10	1,54	1,66	1,63	1,61	200,00	19,73	19,69	19,75	19,72
GG U 1	1,63	1,61	1,81	1,68	200,00	19,79	19,70	19,71	19,73
GG U 2	2,04	2,04	1,88	1,99	200,00	19,76	19,66	19,77	19,73
GG U 3	2,25	2,09	2,11	2,18	200,00	19,83	19,78	19,76	19,79
GG U 4	1,89	1,73	1,77	1,80	200,00	20,01	20,00	19,96	19,99
GG U 5	1,56	1,57	1,68	1,60	200,00	19,84	19,94	19,85	19,88

## GF specimens' dimensions after 500 hours exposure to water at RT

After water uptake Bath RT (mm)									
	T1	T2	T3	Thickness	Length	W1	W2	W3	Width
GF B 1	2,12	2,15	2,25	2,17	200,00	20,39	20,46	20,46	20,44
GF B 2	1,95	2,07	2,06	2,03	200,00	20,41	20,48	20,47	20,45
GF B 3	2,10	2,02	2,21	2,11	200,00	20,42	20,39	20,44	20,42
GF B 4	2,10	2,07	2,15	2,11	200,00	20,39	20,53	20,57	20,50
GF B 5	2,09	2,12	2,13	2,11	200,00	20,51	20,53	20,54	20,53
GF B 6	2,15	2,11	2,20	2,15	200,00	20,51	20,51	20,42	20,48
GF B 7	2,09	2,10	2,10	2,10	200,00	20,65	20,62	20,59	20,62
GF B 8	2,17	2,17	2,08	2,14	200,00	20,62	20,65	20,63	20,63
GF B 9	2,00	2,01	1,98	2,00	200,00	20,48	20,48	20,38	20,45
GF B 10	2,08	2,10	2,04	2,07	200,00	20,53	20,49	20,37	20,46

GF data of the water uptake for the 70°C Bath

Weight (g) in function of exposure time (hours) 70°C																
Sample name	0 (dry)	1h	3h	5h	22h	27h	45h	52h	141h	165h	189h	213h	285h	309h	333h	
GF B7 1	8,6268	8,8620	9,0150	9,1271	9,6128	9,6791	9,7625	9,7863	/	/	/	/	9,7640	9,7712	9,7800	BS
GF B7 2	8,4745	8,6960	8,8540	8,9588	9,4336	9,4846	9,5596	9,5774	/	/	/	/	9,5602	9,5570	9,5545	
GF B7 3	8,7735	9,0190	9,1800	9,2880	9,7845	9,8512	9,9378	9,9594	/	/	/	/	9,9402	9,9441	9,9501	
GF B7 4	8,7511	8,9825	9,1291	9,2343	9,7371	9,7880	9,8773	9,8989	/	/	/	/	9,8904	9,8828	9,8985	
GF B7 5	8,4022	8,6420	8,7871	8,8976	9,3701	9,4216	9,5013	9,5170	/	/	/	/	9,4944	9,4970	9,4984	
GF B7 6	8,3811	8,6280	8,7745	8,8757	9,3416	9,3940	9,4641	9,4776	/	/	/	/	9,4558	9,4580	9,4563	
GF B7 7	8,6100	8,8496	8,9860	9,0913	9,5728	9,6260	9,7282	9,7312	/	/	/	/	9,7087	9,7125	9,7077	
GF B7 8	8,5258	8,7922	8,9386	9,0420	9,5290	9,5879	9,6618	9,6786	/	/	/	/	9,6550	9,6558	9,6606	
GF B7 9	8,2148	8,5002	8,6503	8,7566	9,2122	9,2744	9,3468	9,3690	/	/	/	/	9,3490	9,3568	9,3557	
GF B7 10	8,4741	8,7450	8,8947	8,9988	9,4812	9,5348	9,6134	9,6199	/	/	/	/	9,5961	9,6036	9,6025	
GF B7 1	0,4280	0,4472	/	0,4642	0,4849	0,4859	0,4878	0,4888	0,4878	0,4878	0,4887	0,4880	0,4891	0,4894	0,4896	ILS
GF B7 2	0,4183	0,4404	/	0,4592	0,4789	0,4790	0,4811	0,4823	0,4832	0,4819	0,4834	0,4832	0,4846	0,4833	0,4840	
GF B7 3	0,4131	0,4350	/	0,4436	0,4702	0,4710	0,4744	0,4745	0,4754	0,4747	0,4748	0,4744	0,4760	0,4754	0,4752	
GF B7 4	0,4128	0,4288	/	0,4520	0,4629	0,4639	0,4659	0,4661	0,4660	0,4647	0,4652	0,4652	0,4656	0,4657	0,4656	
GF B7 5	0,4157	0,4370	/	0,4539	0,4730	0,4747	0,4763	0,4783	0,4793	0,4792	0,4794	0,4794	0,4800	0,4805	0,4799	
GF B7 6	0,4367	0,4579	/	0,4761	0,4966	0,4977	0,5008	0,5010	0,5035	0,5022	0,5021	0,5025	0,5035	0,5029	0,5033	
Average	8,5234	8,7717	8,9209	9,0270	9,5075	9,5642	9,6453	9,6615	/	/	/	/	9,6414	9,6439	9,6464	70°C
	0,4208	0,4411	/	0,4582	0,4778	0,4787	0,4811	0,4818	0,4825	0,4818	0,4823	0,4821	0,4831	0,4829	0,4829	
W gain %	0,0000	2,9127	4,6641	5,9088	11,5459	12,2108	13,1625	13,3531	/	/	/	/	13,1167	13,1461	13,1760	
	0,0000	4,8206	/	8,8885	13,5427	13,7685	14,3270	14,5132	14,6796	14,4934	14,6162	14,5805	14,8222	14,7588	14,7746	

## GF data of the water uptake for the RT Bath

	Weight (g) in function of exposure time (hours) Room Temperature																			
Sample name	0 (dry)	5h	22h	24h	28h	46h	51h	70h	77h	166h	172h	190h	214h	238h	310h	334h	358h	382h	406h	484h
GF B 1	8,6927	8,7828	8,9736	8,9912	9,0245	9,1308	9,1602	9,2458	9,2726	9,5592	9,5780	9,6127	9,6586	9,6935	9,7623	9,7788	9,7952	9,8021	9,8066	9,8311
GF B 2	8,1734	8,2889	8,5068	8,5255	8,5673	8,6794	8,7097	8,8224	8,8503	9,1530	9,1596	9,1868	9,2266	9,2428	9,2957	9,3108	9,3285	9,3348	9,3389	9,3622
GF B 3	8,4008	8,4975	8,6900	8,7030	8,7391	8,8453	8,8684	8,9571	8,9850	9,2716	9,2855	9,3147	9,3563	9,3897	9,4553	9,4622	9,4852	9,4931	9,4997	9,5198
GF B 4	8,6393	8,7215	8,9005	8,9116	8,9401	9,0490	9,0730	9,1547	9,1843	9,4473	9,4668	9,5007	9,5467	9,5853	9,6638	9,6813	9,6927	9,7058	9,7088	9,7352
GF B 5	8,2874	8,3994	8,6370	8,6520	8,6947	8,8232	8,8507	8,9541	8,9956	9,3060	9,3162	9,3429	9,3767	9,3994	9,4541	9,4641	9,4839	9,4900	9,4993	9,5257
GF B 6	8,4684	8,5761	8,7900	8,8101	8,8515	8,9708	9,0036	9,0992	9,1294	9,4504	9,4668	9,4917	9,5319	9,5565	9,6179	9,6291	9,6501	9,6598	9,6646	9,6937
GF B 7	8,5306	8,6268	8,8097	8,8246	8,8574	8,9715	8,9932	9,0792	9,1096	9,4044	9,4150	9,4522	9,4942	9,5306	9,5963	9,6151	9,6354	9,6320	9,6406	9,6647
GF B 8	8,7775	8,8643	9,0361	9,0510	9,0796	9,1886	9,2154	9,2981	9,3275	9,6164	9,6249	9,6670	9,7138	9,7538	9,8552	9,8473	9,8664	9,8737	9,8817	9,9071
GF B 9	8,3782	8,4754	8,6484	8,6680	8,7000	8,8081	8,8341	8,9211	8,9508	9,2367	9,2480	9,2873	9,3298	9,3614	9,4249	9,4368	9,4505	9,4476	9,4558	9,4762
GF B 10	8,4949	8,5965	8,7945	8,8089	8,8386	8,9576	8,9817	9,0712	9,1026	9,4056	9,4268	9,4573	9,4956	9,5237	9,5809	9,5967	9,6085	9,6160	9,6204	9,6414
GF B 1	0,4235	0,4293	0,4422	0,4429	0,4457	0,4522	0,4539	0,4591	0,4605	0,4727	0,4729	0,4745	0,4752	0,4757	0,4774	0,4776	0,4781	0,4779	0,4779	0,4790
GF B 2	0,4204	0,4263	0,4386	0,4395	0,4416	0,4479	0,4495	0,4545	0,4555	0,4679	0,4684	0,4694	0,4706	0,4717	0,4720	0,4730	0,4734	0,4734	0,4731	0,4744
GF B 3	0,4193	0,4250	0,4378	0,4387	0,4410	0,4482	0,4499	0,4544	0,4558	0,4678	0,4681	0,4690	0,4701	0,4709	0,4718	0,4718	0,4726	0,4728	0,4735	0,4725
GF B 4	0,4006	0,4068	0,4188	0,4195	0,4212	0,4281	0,4294	0,4339	0,4353	0,4471	0,4475	0,4478	0,4490	0,4500	0,4510	0,4514	0,4522	0,4524	0,4517	0,4527
GF B 5	0,4110	0,4167	0,4294	0,4303	0,4326	0,4390	0,4398	0,4449	0,4462	0,4574	0,4566	0,4588	0,4596	0,4605	0,4616	0,4617	0,4623	0,4629	0,4622	0,4631
GF B 6	0,4080	0,4144	0,4271	0,4279	0,4302	0,4367	0,4375	0,4424	0,4439	0,4550	0,4555	0,4563	0,4569	0,4576	0,4590	0,4589	0,4600	0,4600	0,4598	0,4631
Average	8,4843	8,5829	8,7787	8,7946	8,8293	8,9424	8,9690	9,0603	9,0908	9,3851	9,3988	9,4313	9,4730	9,5037	9,5706	9,5822	9,5996	9,6055	9,6116	9,6357
	0,4138	0,4198	0,4323	0,4331	0,4354	0,4420	0,4433	0,4482	0,4495	0,4613	0,4615	0,4626	0,4636	0,4644	0,4655	0,4657	0,4664	0,4666	0,4664	0,4675
W gain %	0,0000	1,1621	3,4692	3,6570	4,0659	5,3995	5,7127	6,7886	7,1479	10,6165	10,7780	11,1619	11,6533	12,0145	12,8039	12,9403	13,1457	13,2146	13,2871	13,5708
	0,0000	1,4379	4,4748	4,6721	5,2159	6,8189	7,1371	8,3132	8,6354	11,4830	11,5273	11,8012	12,0267	12,2281	12,4859	12,5503	12,7195	12,7517	12,7034	12,9692
								BS		ILS										

**GG** data of the water uptake for the **70°C Bath**

Weight (g) in function of exposure time (hours) 70°C								
Sample name	0 (dry)	1h	3h	5h	24h	72h	96h	166h
GG B7 1	11,3210	11,3616	11,3780	11,3895	11,4718	11,5356	11,5457	11,5873
GG B7 2	11,9749	12,0168	12,0360	12,0480	12,1337	12,2055	12,2189	12,2645
GG B7 3	11,7888	11,8315	11,8535	11,8614	11,9461	12,0130	12,0331	12,0754
GG B7 4	11,4724	11,5131	11,5311	11,5418	11,6256	11,6868	11,7080	11,7443
GG B7 5	11,5788	11,6216	11,6383	11,6487	11,7312	11,7949	11,8091	11,8507
GG B7 6	11,9465	11,9904	12,0091	12,0220	12,1078	12,1762	12,1884	12,2343
GG B7 7	12,1418	12,1829	12,2013	12,2136	12,2992	12,3671	12,3851	12,5270
GG B7 8	11,8440	11,8884	11,9059	11,9186	12,0026	12,0755	12,0859	12,1270
GG B7 9	11,5564	11,5992	11,6165	11,6288	11,7094	11,7734	11,7866	11,8273
GG B7 10	11,8995	11,9426	11,9595	11,9739	12,0550	12,1218	12,1384	12,1773
GG B7 1	0,5529	0,5563	0,5573	0,5579	0,5619	0,5648	0,5662	0,5668
GG B7 2	0,5507	0,5543	0,5552	0,5556	0,5600	0,5627	0,5632	0,5644
GG B7 3	0,5608	0,5645	0,5655	0,5663	0,5701	0,5726	0,5733	0,5747
GG B7 4	0,5620	0,5650	0,5665	0,5669	0,5708	0,5736	0,5743	0,5756
GG B7 5	0,5407	0,5440	0,5451	0,5455	0,5498	0,5528	0,5531	0,5539
GG B7 6	0,5696	0,5731	0,5736	0,5746	0,5787	0,5812	0,5820	0,5833
Average	11,7524	11,7948	11,8129	11,8246	11,9082	11,9750	11,9899	12,0415
	0,5561	0,5595	0,5605	0,5611	0,5652	0,5680	0,5687	0,5698
W gain %	0,0000	0,3608	0,5149	0,6145	1,3259	1,8938	2,0209	2,4599
	0,0000	0,6144	0,7942	0,9021	1,6363	2,1279	2,2597	2,4575
			BS	ILS				

**GG** data of the water uptake for the **RT Bath**

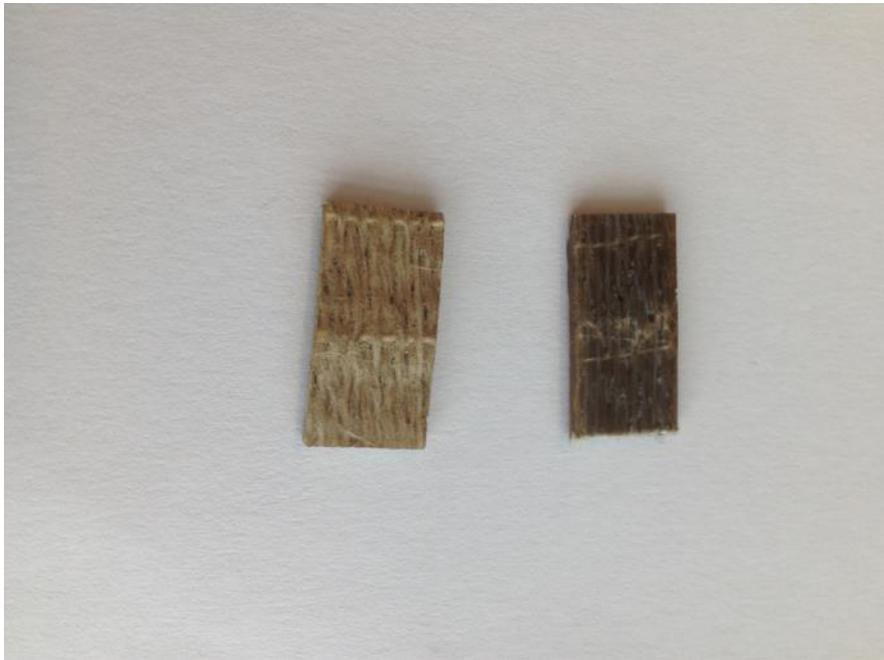
Weight (g) in function of exposure time (hours) Room Temperature							
Sample name	0 (dry)	5h	23h	71h	95h	165h	197h
GG B 1	11,5631	11,5822	11,5940	11,6089	11,6166	11,6256	11,6313
GG B 2	11,5209	11,5386	11,5505	11,5672	11,5711	11,5808	11,5903
GG B 3	11,8157	11,8384	11,8456	11,8608	11,8666	11,8771	11,8807
GG B 4	11,7919	11,8120	11,8237	11,8404	11,8470	11,8581	11,8622
GG B 5	12,4810	12,5020	12,5157	12,5312	12,5359	12,5474	12,5555
GG B 6	12,0412	12,0600	12,0728	12,0894	12,0929	12,1039	12,1094
GG B 7	11,6200	11,6388	11,6508	11,6667	11,6729	11,6927	11,6908
GG B 8	11,6487	11,6684	11,6820	11,6952	11,7014	11,7145	11,7201
GG B 9	11,4956	11,5162	11,5297	11,5445	11,5489	11,5621	11,5668
GG B 10	11,5832	11,6028	11,6181	11,6314	11,6353	11,6461	11,6510
Average	11,7561	11,7759	11,7883	11,8036	11,8089	11,8208	11,8258
W gain %	0,0000	0,1685	0,2736	0,4035	0,4485	0,5504	0,5927
			BS		ILS		

**GF** data of the water uptake for the **POT**

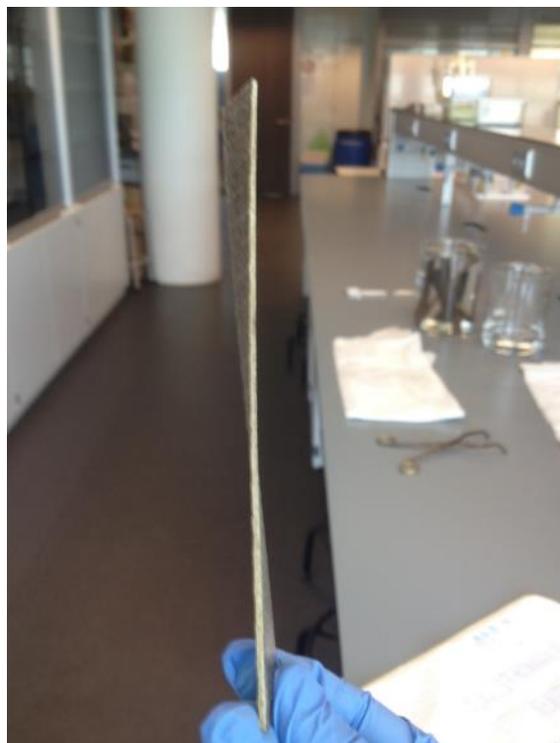
Weight (g) in function of Exposure time (hours)																				
0 dry	1h	3h	5h	7h	23h	28h	47h	54h	143h	149h	167h	191h	215h	287h	311h	335h	359h	383h	455h	479h
11,6099	11,6448	11,6843	11,7038	11,7282	11,8541	11,8927	11,985	12,0199	12,2187	12,2553	12,3001	12,3603	12,4009	12,5462	12,5539	12,6089	12,6385	12,6674	12,7645	12,7672

## Appendix 11

Difference between Standard Specimens (right) and Aged Specimens (left).



Twisted specimen because of swelling



# Appendix 12

Weigh change during UV exposure. The increase was only due to moisture and impurities like dust.

