Deliverable 2

Testing Protocol and Material Specifications for Basalt Fiber Reinforced Polymer Bars

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Contents

Research Plan and Tasks

1.1 Introduction

This deliverable describes the research methodology and lists all experiments that will be conducted on BFRP rebar constituent materials and BFRP rebar products. Several physical, mechanical, and chemical tests will be conducted on rebar samples, raw materials, and exposure solutions, both before and after exposure to various combinations of saline and alkaline environments. Accordingly, this deliverable focuses on four major aspects: 1) the general experimental concept, 2) the characterization of BFRP constituent materials, 3) the characterization of BFRP rebar specimens, and 4) the characterization of exposure solutions. It is the aim to analyze the characteristics and long-term chemical durability of raw materials used for the production of BFRP rebars and to quantify the material properties and limit states of the individual constituents as well as of the final rebar products.

Before studying the deterioration characteristics of BFRP rebars, the durability properties of the raw constituent materials will be studied because the resilience of BFRP rebars depends on the durability of the raw materials. Therefore, a study of the physical properties of basalt fibers will be conducted to qualify the fibers based on the chemical composition and purity, to provide minimum requirements for strength and durability related characteristics. Then, sizing and resins will be characterized based on chemical composition and in view of the durability properties. With the obtained durability results, this research aims to provide recommendations for suitable fiber-resin compositions to target high quality BFRP rebars.

FRP rebars are typically thought off as a resilient alternative in harsh environments, but several studies (c.f. previous deliverable) have shown that BFRP rebars are susceptible to degradation when exposed to a combination of alkaline and saline environments (Guo et al., 2018; Kochergin et al., 2013; Altalmas et al., 2015). Accordingly, the durability of FRP rebar is an important property, which needs to be further studied because degradation caused by chemical attacks may lead to strength reduction, which in-turn causes structure failure that may ultimately lead to personal and financial losses. To minimize the risk and to prevent failure due to actual degradation, strength reduction factors are applied to decrease the design strength of concrete structures in harsh environments, when designing according to AASHTO-LRFD Bridge Design Guide Specifications for GFRP Reinforced Concrete or ACI 440.1R Guide for the Design and Construction of Structural Concrete Reinforced with Fiber-Reinforced Polymer (FRP) Bars. For example, the guaranteed strength (f^*_{fu}) of FRP rebars has to be reduced by applying the environmental factor (C_E) (ACI Committee 440, 2015). Likewise, to avoid premature failure due to creep and fatigue, creep rupture factor (C_c) and fatigue reduction factor (C_f) are applied to the design strength for FRP rebars under sustained load and cyclic load (ACI Committee 440, 2015; du Béton, 2007). Such factors vary and depend on the actual fiber type (glass, carbon, basalt, etc.) and resin materials. Because the environmental factor, C_E, has not been fully developed for BFRP rebars and this factor is an important consideration for the design of infrastructure elements in Florida, it is the goal of this experimental program to systematically expose different BFRP rebar components to various harsh environments, such that the effective degradation can be quantified and data for the development of environmental factors under Florida conditions can be provided.

To standardize BFRP rebars with appropriate quality, the chemistry of raw materials and rebars in alkaline and saline environments has to be studied through systematic testing. To evaluate the potential material degradation of BFRP rebars and their components, a test matrix was developed to target various specimen characteristics in the virgin state and after exposure in harsh conditions after 300 day and 600 day. Mass transfer between the exposed materials (fibers, resin, and BFRP rebar) and the aggressive solution will be quantified. Specific experiments will be conducted to quantify the chemical compounds that are transferred between the exposed solids and the storage solution. The impact of chemical mass transfer on the physical and mechanical properties of conditioned specimens (fiber, resin, and rebars) and the effect of exposure conditions (salinity and alkalinity) on basalt based FRPs will be quantified.

1.2 Experimental Concept Overview

Because BFRP composites are potentially affected by saline and alkaline environments (Benmokrane et al., 2015), a major goal of the experimental program will be to simulate a combination of these two environments (factorial expeiments) with varying pH content and chloride ions to systematically study the impact of each chemical environment and the combined effect. Figure 1.1 depicts the various combinations of the exposure conditions, in which the alkalinity and the salinity are systematically increased. In the test matrix, gray filled

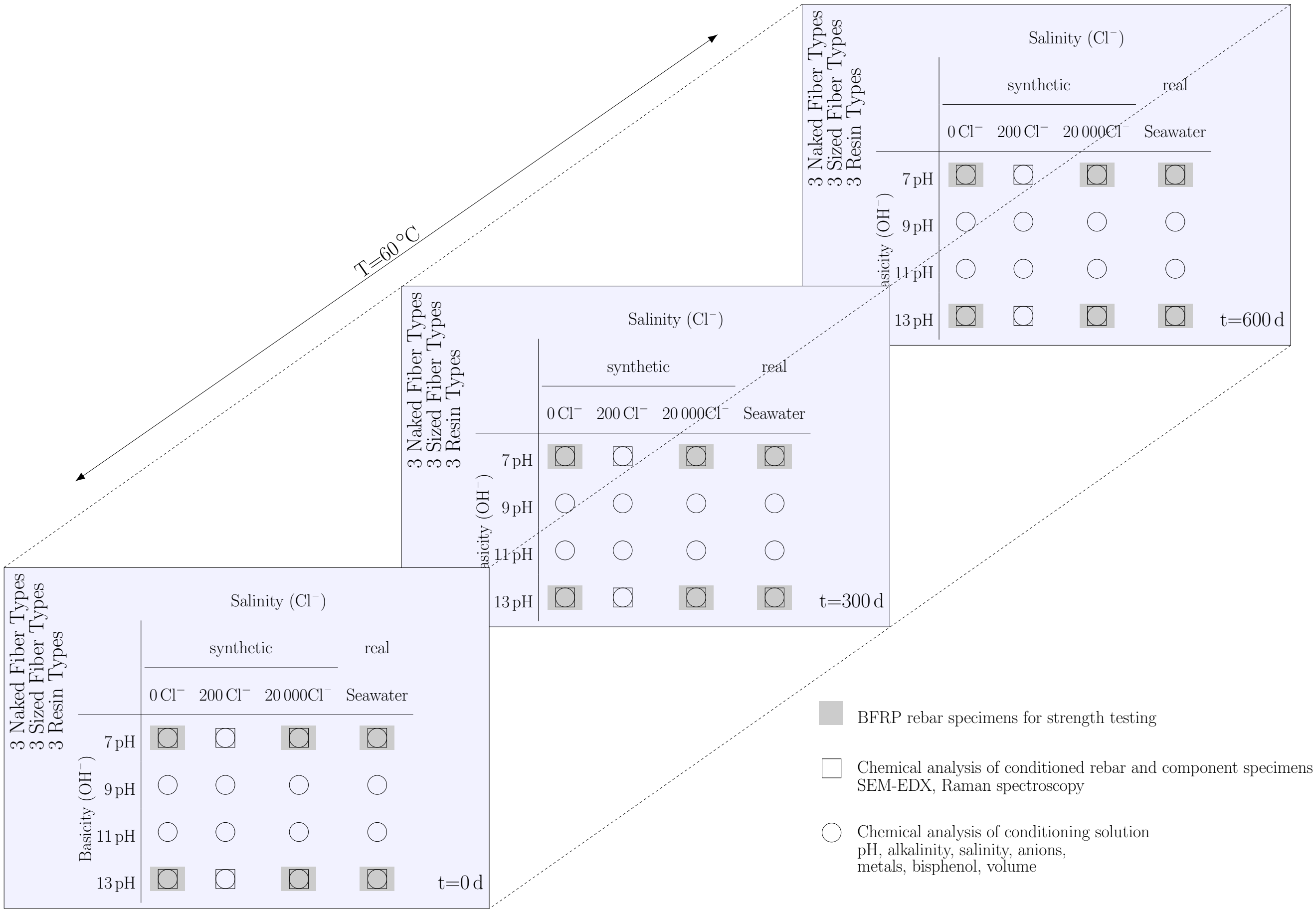

Figure 1.1: Test concept for chemical exposure

squares indicate BFRP rebar samples for mechanical strength tests, while wire squares identify raw material samples for chemical durability tests, and finally the wire circles represent chemical analysis conducted on the exposure solutions in which BFRP components are stored. As seen in the figure, the salinity of the exposure solutions will range from 0mgCl$^-$/L (deionized water), to 200mgCl$^-$/L (fresh water), and 20000mgCl$^-$/L (synthetic and real seawater), while the range of pH value will vary from 7pH (neutral water) to 13pH (high alkaline water). These exposure solutions will be developed synthetically to eliminate potential contamination and to precisely study the degradation caused by the main factors. Along with the synthetic solutions, control samples will be stored in real seawater before testing, to study the degradation properties under real world conditions. The exposure temperature for aging FRP in alkaline solutions according to ASTM International (2012) is 60 °C, and it is the most commonly used temperature suggested by Chen et al. (2007); Benmokrane et al. (2017). Therefore, a constant temperature of 60 °C will be maintained throughout the conditioning period to accelerate the chemical degradation process. Chemical baseline values for the virgin materials as well as for the virgin exposure solutions will be taken before aging (0 days) is initiated, and ultimately compared to the measurements after each exposure duration (300 days and 600 days). Table1.1 lists different types of tests that will be performed on the conditioned raw materials and BFRP rebar samples, before and after the exposure periods. To expand the general experimental concept and for additional clarification, the individual aspects of the test program (constituent materials, BFRP rebars, and exposure solutions) are separately discussed below.

Table 1.1: Test matrix

	Specimen type			
	Constituent Material			
Test type	Naked Fibers†	Sized Fibers	Resin	BFRP Rebar
Strength test	✗	✓	✓	✓
Chemical analysis of material solids	✓	✓	✗	✓
Chemical analysis of exposure solution	✓	✓	✓	✓

† Provided such fiber can be obtained/purchased

1.2.1 Characterization of BFRP Constituent Materials

FRP rebars are a product of composites made from sized fibers and resin matrices. The durability of these rebar products hugely depends on the resilience of the raw materials. Therefore, it is important to test the chemical durability of raw materials before analyzing chemical durability of rebars. For reliability of test results and to obtain representative values for the BFRP rebar product as a whole, the tests will be repeated five times for samples taken from different sections of the production lot — average values will be assigned and statistics (min, max, CV, etc.) for each sample will be documented. Specimens from three different production lots per manufacturer and three different manufacturers are intended to be tested. Accordingly, a total of 45 tests will be conducted — for virgin materials as well as after each aging period — for each test procedure that targets the characterization of constituent materials. Table 1.2 lists the tests that will be performed on fiber samples and resin samples. Basic material properties of basalt fibers, such as diameter

Table 1.2: Tests on individual components of BFRP rebar

			Specimen count	
	Test type	Test method	Per sample	Total
Naked and sized fibers	Diameter of fibers	ASTM D6466	5	45
	Moisture absorption	Weight change observation	5	45
	Micro-structure observation	SEM and Raman Spectroscopy	5	45
	Tensile strength	ASTM C1557	5	45
	Mass transfer between solution and fibers	Based on chemical analysis results	5	45
Resin	Moisture absorption	ASTM D5229 / D5229M	5	45
	Glass transition temperature	Differential scanning calorimetry	5	45
	Micro-structure observation	SEM and Raman Spectroscopy	5	45
	Tensile strength	ASTM D638	5	45
	Shear strength	ASTM D732	5	45
	Mass transfer between solution and resin	Based on chemical analysis results	5	45

and unit weight, will be studied before measuring the tensile strength. The fibers will then be exposed to harsh environments as shown above. After each individual conditioning period, a battery of tests will be conducted on the specimens as listed in table 1.2. Likewise, the virgin material properties of resin will be tested and characterized before the resin specimens will be exposed to harsh environments. Change in physical properties will be quantified by performing an array of test procedures as shown in table 1.2 to evaluate the retention properties. Chemical mass transfer between the fiber/resin specimen and the exposure solutions will be studied through chemical analysis of the individual components and the exposure solutions.

1.2.2 Characterization of BFRP Rebar Specimens

To use FRP rebars in infrastructure projects, they have to meet or exceed specific test criteria (e.g.: Florida Specifications Section 932). Typical physical properties such as the cross-sectional area, fiber content, moisture absorption, mechanical properties such as tensile strength, horizontal shear strength, transverse shear strength, and bond-to-concrete strength, and chemical durability properties have to be studied. This section provides a general overview of these tests and details how these tests will be superimposed with the test concept shown above in Figure 1.1.

All physical and mechanical tests that are intended to be conducted on BFRP rebars are listed in Table 1.3. Two commonly used rebar sizes — # 3 and # 5 — will be tested in this project. For reliability of test results and to obtain representative values for the BFRP rebar product as a whole, the tests will be repeated five times for specimens taken from different sections of the production lot and the average values will be assigned (while monitoring statistics of each sample). Three different rebar products (each produced by a different manufacturer) will be chosen and three different production lots per manufacturer are intended to be tested. The physical and mechanical tests will be conducted on virgin rebars or on rebar specimens that have not been exposed to harsh environments. In addition, the mechanical tests will be conducted on conditioned rebar specimens after multiple exposure durations to evaluate and quantify the strength retention properties in harsh environments.

The variation of cross-sectional properties largely depends on the proprietary production methods and the surface enhancement features. Therefore, the cross-sectional area will be measured according to the ASTM D 792-13 to benchmark the physical property and to study the relative differences between the tested rebar products. The fiber content percentage of FRP rebar plays a crucial role for the load transfer, ductility,

Table 1.3: Tests on BFRP rebars

	Test type	Test method	Specimen count	
			Per sample	Total
Physical	Cross-sectional area	ASTM D792	5	90
	Fiber content	ASTM D2584	5	90
	Moisture absorption	ASTM D570	5	90
	Micro-structure observation	SEM and Raman Spectroscopy	5	90
	Glass transition temperature	Differential scanning calorimetry	5	90
Mechanical	Tensile strength	ASTM D7205	5	90
	Transverse shear strength	ASTM D7617	5	90
	Apparent horizontal shear strength	ASTM D4475	5	90
	Bond-to-concrete	ACI440.3R,B.3	5	90
	Mass transfer from solution to rebar	Based on chemical analysis results	5	90

elastic modulus, and the ultimate strength of a rebar product. Accordingly, the fiber-to-resin ratio of the BFRP rebars will be quantified in agreement with ASTM D 2584 (ASTM-International, 2011). Because FRP rebars are intended for use in bay areas and other harsh environments, moisture infiltration is a critical characteristic that relates to the durability properties of FRP rebars. Moisture can damage the rebar structure and deposit contaminates that decreases the strength of the composite material and compromise the overall rebar integrity. Therefore, the moisture absorption property of the BFRP rebar samples will be characterize according to ASTMD 5229/ D 5229M (ASTM, 2014). Chemical analysis on the rebars and porosity analysis will be conducted via SEM methods to study the deterioration of the BFRP rebars due to exposure. The glass transition temperature (T_g) of the rebar samples will be evaluated via differential scanning calorimetry according to ASTM E 1356 (ASTM International, 2014a). Because the T_g of a resin system defines when a thermoset polymer transitions from an amorphous rigid state to a more flexible state, it is an important rebar property that defines the nature (rigid and glassy or flexible and rubbery) of the polymer at its service temperature. ASTM D 7617 (ASTM-International, 2012b) will be used in the process of testing and analyzing the transverse shear properties. In addition, the BFRP rebar products will be tested for horizontal shear properties according to ASTM D 4475 (ASTM-International, 2012a) to evaluate the quality and strength of the resins when use for the production of BFRP rebars. The tensile properties (strength and elastic modulus) of the rebars will be evaluated according to the ASTM D 7205 (ASTM-International, 2015). Bond-to-concrete strength of the rebars will be tested in accordance with ASTM D 7913 (ASTM International, 2014b) to quantify the bond strength variations based on the different surface enhancement features.

1.2.3 Characterization of Exposure Solutions

To maintain the designed exposure conditions of the storage solutions, the conditioning environments will be monitored and analyzed at defined time interval. Different chemical characterization tests will be conducted to quantify and report the chemical properties. All chemical analysis tests — to be conducted on the exposure solutions — are listed in Table 1.4, along with the standard procedures that will be followed for each test. To monitor and maintain the designed exposure conditions, pH values, alkalinity, and salinity of the exposure solutions will be regularly measured. The anions and metals transferred from the fibers to the solutions and from the solution to fibers will be measured because these quantities are needed to determine the acidity modulus of basalt, which is an important property that characterizes the suitability of the raw materials

Table 1.4: Tests on exposure solutions

Test type	Test content	Test standard	Volume of specimen per test
Electrometric method	pH	SM4500-H+	No sampling needed[†]
Electrical conductivity	Salinity	SM2520-B	No sampling needed[†]
Titration method	Alkalinity	SM2320-B	100 mL
Ion chromatography	Anions	SM4100	1 mL
Atomic emission spectrometry	Metals	Agilent 4100 MP-AES	5 mL
Gas chromatographic/mass spectometric method	Biphenol A	SM6040	1 mL

[†] Probes will be placed in storage container

for fiber production. In addition, it is needed as a quality control indicator during basalt fiber production. Bisphenol A (BPA) will be measured because this organic synthetic compound is used in the manufacturing of resins and it may transfer from the resin to the exposure solution as a result of rebar degradation.

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