ON DETERMINING THE CHEMICAL STABILITY OF BASALT FIBRES IN AN ARTIFICIAL ALKALINE MEDIUM FOR TEXTILE REINFORCED CONCRETE (TRC) APPLICATIONS

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Abstract

Textile Reinforced Concrete (TRC) is currently entering the growth stage of market maturity in Europe. It is a composite made of cement based matrix and a textile reinforcement. AR-Glass fibres are used as reinforcement in concrete, due to its excellent chemical stability in alkali medium. Alternatively carbon fibres are used for high value and complex engineering projects because of its much better alkali performance in comparison to AR-Glass however at extortionate costs. Nonetheless, neither AR-Glass fibre nor carbon fibres can reach the required target ratio between price and performance. Basalt fibres are a valuable alternative to AR-Glass and carbon fibres. Basalt fibre has very good mechanical properties and thermal resistance. The potential of basalt fibres as an alternative to AR-Glass or Carbon fibre in TRC applications is very high; nevertheless this product is still underperforming in high alkaline environment. Aim of this paper is to present a combination of methods developed to determine at a micro scale the chemical stability of fibres and the results related to basalt fibres in an artificial environment similar to the concrete but free from external conditions such as handling damage, scratches, micro stress and friction due to solidification. The tests have be carried out combining optical, mechanical and chemical analysis.

1. Introduction

Basalt fibers are mostly produced from natural basalt rocks, which are rocks formed from solidification of volcanic magma that came out on the surface from the earth’s mantel. The chemical composition of basalt fiber depends on the composure of the raw material.
Basalt is an inorganic material, a silicate composed by a mix of different oxides (SiO2 45 % - 52 %, Al2O3 12 % - 16 %, FexOy 6 % - 18 %, alkaline earth oxides 10 % - 20 % and alkaline oxides 2 % - 8 %).

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Even if their commercialization began in the 90's, the fiberization process of basalt was invented in the 60's. These fibers are nowadays manufactured by melting process.Due to the natural variability of the chemical composition of rocks, basalt fiber manufacturing is very difficult to control. The melting process strongly depends on the temperature range and basalt has a very short temperature range fitting the viscosity requirements. Because of that, there are several technical issues to fiberize, unlike similar fibrous material like E-glass.

Basalt fibers, if compared to the benchmark, the E-Glass fibers, have a very high thermal and chemical stability, strength (up to 4,84 GPa) and stiffness (up to 110 GPa). Basalt fibers, like glass fibers, find application in different textile products. The main applications are in fiber reinforced concrete (mostly as rebars) and fiber reinforced plastics.

The global volume of basalt fiber production is around 30,000 tons, making them a niche product if compared to glass fibers (more than 4 million tons in 2011 [1]). Around the world there are only few basalt fiber producers such as: Kamenny Vek (Russia), DBF – Deutsche Basalt Faser GmbH (Germany), Technobasalt (Ukraine), GBF Basalt Fiber Co., Hebei Tong Hui Science Technology Co. (China), Isomatex (Belgium) and Mafic (Ireland). [2]

Basalt fiber have a good chemical stability resulting in adoption of products such as rebars in concrete. Rebars are composite materials made of a fibrous reinforcement and a matrix able to protect the fibers from an alkaline environment like concrete so that the fibers preserve their original mechanical properties. Several developments have been done in order to improve the alkali resitence of basalt fibers in alkali medium in order to use the fibres also in TRC.

TRC reinforcement is usually an open grid type textile with the chief function to take the tensile loads. Several projects with TRC had been already realised in countries such as Germany and Switzerland. These projects have traditionally used AR-Glass or Carbon fibre as a reinforcement.

Due to the cost implications of carbon fibres, AR-Glass fibres are in concrete the most common fibrous material used nowadays. AR-Glass fibres consist of a glass containing expensive oxides such as zirconium dioxide and are coated with an epoxy or styrene butadiene rubber to ensure required durability and performance. Carbon fibres are only preferred in complex engineering projects because of their extortionate costs. However, both of them cannot reach the required comfort ratio between price and performance, something that is essential in the construction sector.

In addition to glass and carbon fibres, basalt fibres have a potential to be a valuable alternative. They are neither widespread like glass fibres nor popular like carbon fibres. However, basalt fibre has very good mechanical properties,thermal resistance and their potential as an alternative to AR-Glass or Carbon fibre in TRC applications is very high. Nevertheless, this product is still underperforming in high alkaline environment such as concrete (PH value between 12 – 14).

This paper is the preliminary work carried out in a publically funded German project called Basflair. Basflair (2015-2017) aims to further improve the already existing, but not enough, alkali resistance of basalt fibres by means of a surface treatment (sizing). The sizing is applied during the fiberization process between the fibre formation and the winder. The sizing has different functions associated to the textil processes and also to the final application. It is used to preserve and protect the fibres, to keep them together in a roving and to allow the fibre to get processed into fabrics. Additionally, sizing for composites applications improve the adhesion between the fibres and the matrix. [3]
Considering the special application in TRC, a targeted adjustment of the sizing formulation is decisively critical for the fibre-matrix behavior and, in this case, for the alkali resistance of basalt fibre.

The aim of this paper is to present a combination of methods developed to determine at a micro scale the chemical stability of fibres. Additionally it discusses the results related to basalt fibres in an

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artificial environment similar to the concrete. In order to characterize the effect on basalt fibre in a concrete medium, tests were conducted on basalt fibres in comparable other alkaline solutions. These tests had been carried out combining optical, mechanical and chemical analysis. The solutions chosen will not affect the fibres in mechanical factors such as scratches, micro stress and friction due to external conditions (solidification of cement). The improved method to test fibres in alkaline solutions will help to streamline the fast testing of possible sizing solutions for fibres in alkali resistant applications such as TRC.

The composite behaviour is the main measure that decides whether a fiber / reinforcement structure is suitable for the manufacture of certain textile concrete elements. The analysis of the composite behaviour does not allow to determine or to exam the quality of the fibers, without these being affected by the concrete. The friction with particles in the medium, the chemical reactions and the volume expansion will have unknown effects on the fibers. Therefore, the properties of the fibers in the concrete had to be separed from the concrete itself. To make this possible, the fibers must be inserted into a concrete-like medium from which they can be removed after for examination.

The chemical resistance of the fibers is nowadays measured principally as loss of weight. They are immersed in an alkaline solution and its weight is measured before and after the experiment in order to determine the discrepancy. The weight loss is not an effective parameter. On one hand, the leaching is quantified as ion exchange and diffusion parameters. On the other hand, the properties of the fibers are mainly dependent on cracks and defects on the surface. These properties of the fibers are affected by how the fibers are attacked by alkalis. There is a relationship between strength and weight loss [4]. The relevant parameter by reinforcing fibers is the real tensile strength during the chemical leaching and not the indirect weight loss.

2. Materials and Methods

A leaching bath was developed in order to simulate the condition of the medium during the concrete hardening in its first phase. This leaching bath had to be able to work with several samples in the gram range but also large basalt fiber samples as textile structures in order to be characterizated. The main characteristics of the bath system are here listed:

- Continuous temperature (working temperature between 20 °C and 80 °C);
- Constant level of the water by reflux condenser;
- Homogeneous temperature distribution in the bath;
- Up to 20 experiments simultaneously;
- High reproducibility of the leaching;
- Material: PVDF (acid and alkali resistant);
- Possibility to leach entire textile structures.

The leaching bath is shown in Figure 1.

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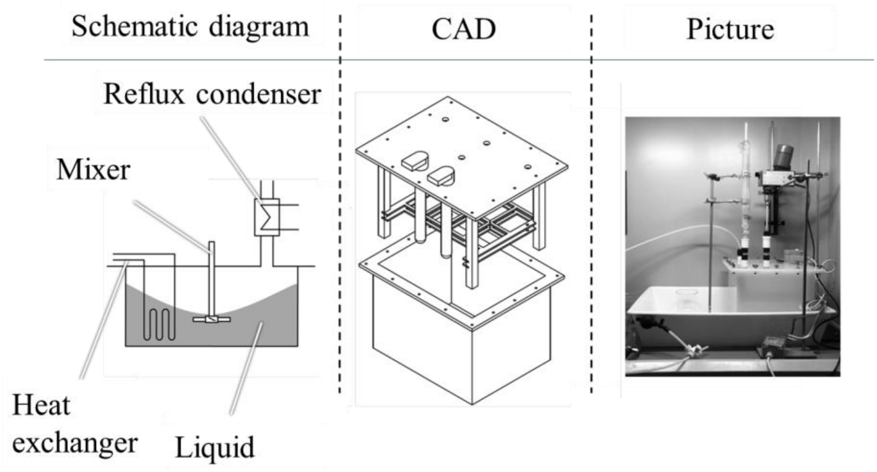

Figure 1. Leaching bath (from L to R) – schematic diagram, isometric CAD drawing and actual photograph of the equipment

The following tests were carried out on basalt fibers and on AR-glass:

- Standard method based on weight loss;
- Loss of strength;
- Material analysis / chemical composition SEM-EDX.

Several different samples of basalt fibers have been tested. The reference sample used was AR-Glass fiber (CemFil©).

3. Experiments

In order to determine the alkali resistance of the fibers in concrete, the samples have been simultaneously chemically and thermally treated. The temperature profile approximates to the hardening concrete profile (exothermic reaction). Thereafter, the mechanical properties of the individual filaments were measured and the surface was analyzed with an electron microscope.

To analyze the effect of the alkali medium on the surface of the fibers, the composition of the leached sheath and the core of the fibers had been examined. The quantification was done via SEM images.
Firstly, a suitable medium was researched with similar alkali behaviour of hardening concrete between three different liquids (sodium hydroxide, calcium hydroxide and a more complex solution of NaOH, KOH and CaOH). Due to the similarity of the concrete alkali medium, the last one was chosen.
The concentration of the alkaline solution was kept constant for all samples (Ca (OH) 2 3.6 g/l ; KOH 19.6 g/l ; NaOH 2.0 g/l)
The additional parameters of the experiments were temperature (50 ° C and 70 ° C), leaching (1 to 6 days), volume of solution (100 ml) and sample weight (approximately 0.2 g).
Following first results were reported in our research.

3.1. Morphology and characteristics of the samples

The list of samples that have been tested during the experiments are listed on
Table **1**.

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Table 1. Samples used for the experiments

Sample	*Diameter* *Ø ± σ* (µm)	*Weight-based specific area* (cm²/g)	*Weight-based specific area % rel. to AR-Glass* (%)
BF0069	17,46 ± 2,32	865	146%
BF0090	15,20 ± 2,37	993	168%
BF0103	17,72 ± 1,12	852	144%
BF103	18,41 ± 2,27	820	139%
BF0104	17,14 ± 1,53	881	149%
BF0112	17,46 ± 1,40	865	146%
AR	26,07 ± 2,63	590	100%

3.2. Weight loss method

As shown in
Table **1**, the samples had different fiber diameters. The leaching of the fibers is directly related to specific surface. The weight-based specific area of the samples depends on the fiber diameter. Therefore, an adaptation of the leaching to the weight-based specific area of the sample was required. The resulting corrections parameters are reported in
Table **1**. With respect to the weight-based specific area of 100 % for AR-Glass, the basalt fiber samples (BF) showed a higher specific surface related to the lowed fiber diameter. A consequent effect of the weight’s loss was taken in consideration.

Table 2. Weight loss of the sample after 2 days at 50 °C and after 2 days at 70 °C. Adaptation of leaching with regard to the weight-based surface.

Sample	*Loss of weight (50°C, 2 days)* (%)	*Loss of weight (70°C, 2 days)* (%)	*Weight-based specific area % rel. to AR-Glass* (%)	*Specific area based loss of weight*$^{(1)}$ *(50°C, 2 days)* (%)	*Specific area based loss of weight*$^{(1)}$ *(70°C, 2 days)* (%)
AR	0,00%	0,67%	100%	0,00%	0,67%
BF0112	0,42%	1,35%	146%	0,29%	0,92%
BF0090	0,90%	1,86%	168%	0,54%	1,11%
BF0104	1,69%	4,00%	149%	1,13%	2,68%
BF103	1,75%	3,05%	139%	1,26%	2,19%
BF0069	1,77%	3,55%	146%	1,21%	2,43%
BF0103	2,21%	2,70%	144%	1,53%	1,88%

3.3. Strength determination

The measurement of the fibers’ strength starts by determining its fineness. It is quite common in the textile industry to use the fiber denier to characterize this fineness [5, 6] that states the mass of a certain length of a filament, roving or yarn. In the case of the investigated fiber samples, which show a circular cross section, the diameter, d (µm) can be calculated starting from the fiber denier, Tt (dtex) and the fiber density ρ (kg/m³) according to the equation 1:

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$$d = \sqrt{\frac{4 \cdot 10^{-6} \cdot Tt}{\pi \cdot \rho}} \tag{1}$$

The measuring principle is based on a vibrational method. For each measurement a single filament is loaded with a pretension weight and placed in the bracket. An impulse puts the sample in a vibrational state and the oscillation frequency is detected by an optical sensor, in order to determine the fiber denier.

After determining the fineness and fiber diameter, the instrument determined the strength and the elongation. A single filament was fixed between two clamps. During measurement the lower clamp moved down stretching the fiber whilst, the upper clamp measured the resulting force. The test was carried out until the fiber's breakage. The strength was determined by combining the force with the respective denier and density of the fiber. Each value was the arithmetical average of 50 measurements. Figure 2 illustrates the mechanical properties of the samples before and after the leaching process (2 days at 50 °C).

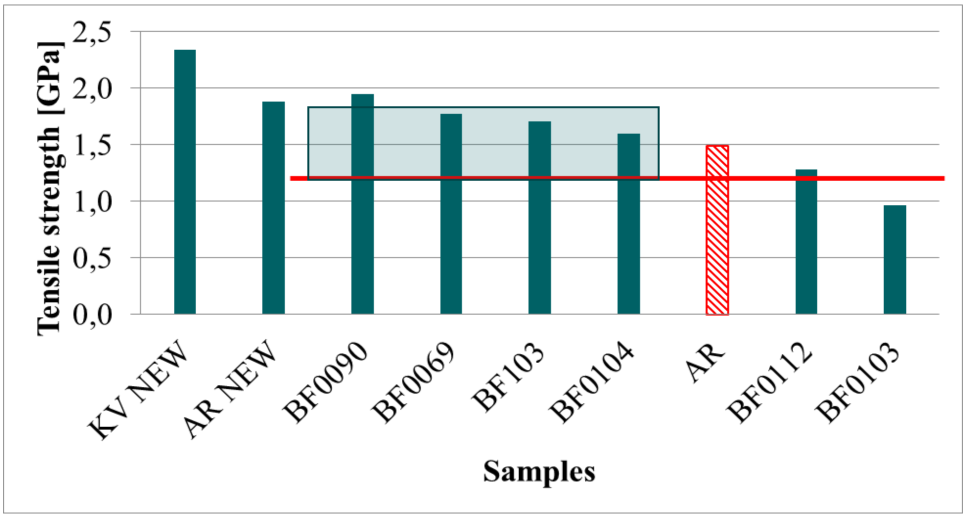

Figure 2. Tensile strength between different samples: virgin basalt fiber (KV NEW), virgin AR-Glass fiber (AR NEW), leached basalt (BF) and leached AR fibers at 50°C for 2 days.

All samples leached for 2 days at 70°C including AR-Glass, were not measurable anymore. That is due to the strong loss of strength of the leaching.

3.4. Chemical composition / SEM-EDX analysis

After the leaching process, fibres were analysed by SED EDX in order to detect the leaching effect on the chemical composition of the fibers. Figure 3 illustrates the EDX results of a basalt fiber (left) and of a AR-Glass fiber (right). Furthermore, Figure 4 illustrates the SEM pictures of a basalt fiber (left) and of a AR-Glass fiber (right). Both figures were not conclusive in order to determine the effect of the leaching on the fibers because the figures didn't show any surface decomposition.

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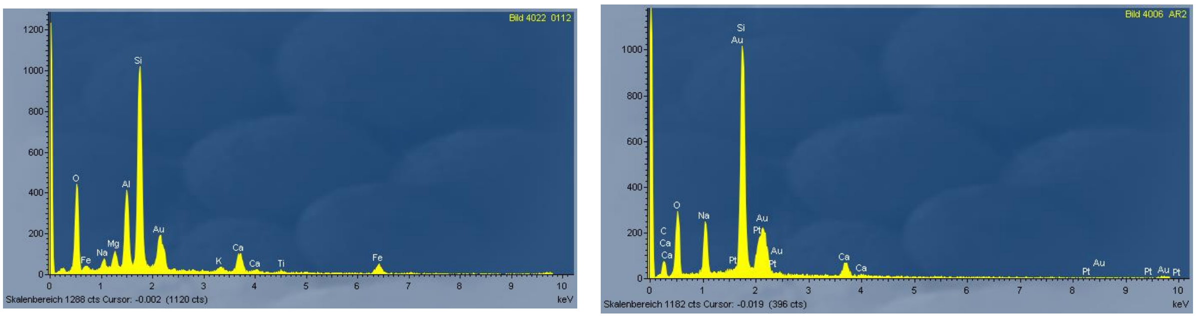

Figure 3. EDX analysis of basalt fiber (left) and AR-glass fiber (right) after leaching at 50 °C – 2 days

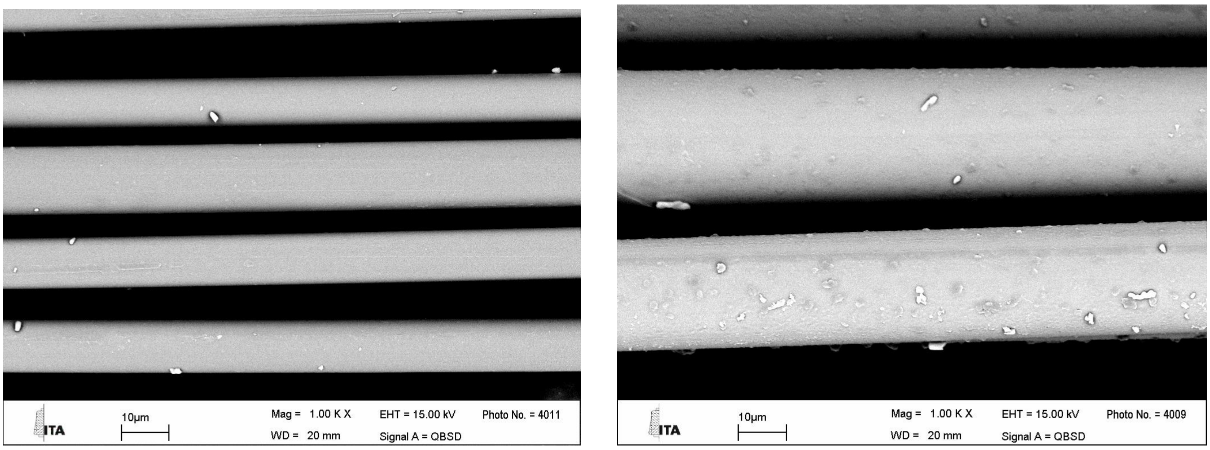

Figure 4. SEM picture of basalt fiber (left) and AR-glass fiber (right) after leaching at 50 °C – 2 days

4. Conclusions

Three different methods were compared in order to determine the chemical stability of basalt fibres in an artificial medium for TRC applications. All tests were made on different basalt fiber samples and on AR-Glass fiber which nowadays is the benchmark. The experiments evidenced a divergence between the results related to the loss of weight if compared to the degradation of the mechanical properties. AR-Glass especially showed a very low loss of weight combined with a complete loss of strength. Basalt fibers preserve in many samples higher mechanical properties when compared to the benchmark, even if this one shows lower loss of weight. Considering the loss of strength, it is also interesting to take into account that all the samples, included AR-glass, after 2 days at 70 °C couldn’t be measured anymore. The SEM-EDX was not a fruitfull instrument to measure the initial effects of the leaching on the fibers. On one hand, in order to be detected by SEM-EDX, the leaching should be more aggressive (higher temperature, longer time, stronger alkaline medium). On the other hand, the effect of the leaching would completly destroy the fibers and their mechanical properties.

In specific cases, SEM Pictures could help to detect the properly execution of the fiber cleaning. In some cases deposits of leachtates have been detected on the fiber’s surface. However, in order to determine by SEM-EDX a strong effect of the leaching to the fibers would be necessary to leach longer with a stronger alkali medium (NaOH).

Each method is actually able to cover a well defined sector of the leaching parameters. Strong leaching will require SEM-EDX combined with loss of weight. Loss of weight accomplish its function on average leaching and the determination of mechanical properties is usefull in case of low intensity leaching. It is evident that qualitative methods related to the mechanical properties priorize optical methods, that are related to a general loss of weigth or to a analysis of the chemical composition.

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