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# Optical fiber sensors for monitoring cement paste carbonation

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**Abstract**. The use of concrete has been widespread in our society in housing and infrastructure, despite the environmental cost associated with its production. Its decay poses a social, economic, and environmental problem. Currently, the carbonation of cement paste is monitored through the measurement of its pH, with several optical fiber sensors (OFS) have been produced for this purpose. In the current work the focus is, also, on the carbonation monitoring of cement paste through an OFS, but not through pH measurements. Single fiber reflectance spectroscopy, previously employed to measure cement paste durability, is used to monitor the discoloration of cement paste caused by carbonation. As the carbonation front reaches the fiber tip embedded in the cement paste, the signal reflected onto the fiber increases. The accelerated carbonation of two limestone cement paste samples in an atmosphere of 100% $CO_2$ was successfully monitored. The applicability of the sensor for operational use with ambient $CO_2$ was confirmed through the measurement of carbonation at 3% $CO_2$. The cross interference from water ingress and egress was also evaluated, and it didn't hinder the measurements of carbonation. Therefore, a novel OFS capable of measuring cement paste carbonation and durability, was achieved.

## 1. Introduction

Concrete is one of the most used materials by mankind, second only to water [1]. Incidentally, it is by mixing with water that cement gains its binding and strength properties which make concrete a premium material for both housing and infrastructure. Without it, our society would be unrecognizable. As cement ages and reaches the limits of its service life, between 50-100 years [2], repairing and rebuilding infrastructure becomes an increasing necessity. Understanding and monitoring the causes behind cement paste decay is a pressing issue. An issue that allows for the prevention of catastrophic failure, the extension of concrete service life and optimization of repair costs. It is also environmentally friendly given that it will allow for the reduction in the demand of cement whose manufacturing accounts for a large amount of $CO_2$ emission and energy consumption [3].

Cement is usually combined with sand, aggregates, and water to form concrete. Concrete is capable of withstanding large compressive forces but is weak against tensile forces. For this reason, steel beams are added to produce reinforced concrete which is structurally capable of resisting both compressive and tensile forces [4].

There are several mechanisms, working concurrently, of reinforced concrete decay. Some of them are alkali-silica reactions [5], ion chloride ingress [6], freeze and thaw [7] and carbonation [8]. All these mechanisms are influenced or require water to occur, which denotes the importance of its monitoring in concrete [9]. And all these mechanisms produce cracks on concrete, which will further expose these structures to further deterioration.

The monitoring of infrastructures relies, predominantly, on visual inspections which can only address decay after there are signs of it. Visual inspections can't respond to the high spatial variability of concrete decay [10] nor are they up to current demands of the 4.0 industry [11]. Alternatively, electrochemical sensors have been developed to continuously monitor the health of both concrete and rebars. Unfortunately, these sensors are prone to electromagnetic interference and use reference electrodes with poor long-term stability [12]. On the other hand, optical fiber sensors, immune to electromagnetic interference, are small, stable, and resistant. Moreover, they were used to monitor temperature [13], humidity [14], strain [13], pH [12,15] and ion chloride ingress [16] in concrete.

Carbonation occurs from the reaction of $CO_2$ with hydrated cement paste provided the former is dissolved in water. $CO_2$ will react with water to form bicarbonate ion ($HCO_3^-$), equation 1, which will transform into carbonate ion ($CO_3^{2-}$), equation 2, and ultimately react with calcium ions ($Ca^{2+}$) of cement paste forming calcium carbonate ($CaCO_3$), equation 3 [17].

$$CO_2 + H_2O \rightleftharpoons HCO_3^- + H^+ \tag{1}$$

$$HCO_3^- \rightleftharpoons CO_3^{2-} + H^+ \tag{2}$$

$$Ca^{2+} + CO_3^{2-} \rightleftharpoons CaCO_3 \tag{3}$$

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O \tag{4}$$

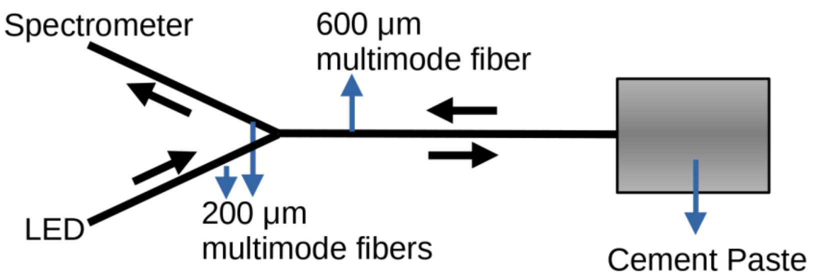
The dissolution of portlandite ($Ca(OH)_2$) gives cement paste its characteristic alkalinity, maintaining the pore solution >12 pH [18]. The carbonation of portlandite, equation 4, will acidify the pore solution of the cement paste. If the pH dips below 9.5 [19], the passivation of the steel beams embedded in concrete is no longer possible. This leaves the rebars exposed to corrosion and spalling which will in turn crack and expose concrete to further deterioration.

$CO_2$ will also react with other sources of $Ca^{2+}$, most notably calcium silicate hydrates (C-S-H). C-S-H is the main product of cement hydration, is responsible for its strength and adhesion properties and makes up to 50-60% of the hydrated cement paste [4]. C-S-H carbonation is associated with the increase in its porosity and the shrinkage and cracking of cement paste.

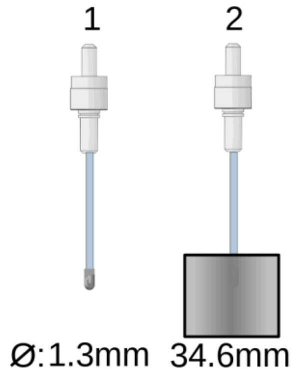
The monitoring of carbonation is usually done by spraying concrete with phenolphthalein, either on the exposed surfaces due to spalling and cracking or on retrieved samples from structures. It gives an accurate picture of the current state of the infrastructure and how at risk it is from steel corrosion. It won't, however, work on concrete surfaces that have been impregnated, nor will it actively help in preventing the decay of these structures. For this purpose, real-time and continuous monitoring is required. Electrochemical and optical fiber sensors have been used for the monitoring of cement paste carbonation, through the measurement of cement paste pH [15,20]. In the current work the focus is on measuring cement paste carbonation through a colorimetric optical fiber sensor that monitors cement paste color.

## 2. Materials and Methods

Single fiber reflectance spectroscopy [9] was used to monitor the changing color of cement paste due to the carbonation process. Figure 1 (a) displays the setup used for this purpose. Light from a light emitting diode (LED) is coupled into a 200 µm fiber and carried into a 600 µm sensing fiber with its polished distal tip embedded in cement paste. Light is reflected at the fiber tip, depending on the refractive index and color of the cement paste, and is transported into a spectrometer. Previously this setup was used to monitor the rate of water ingress to estimate the capillary coefficient of the cement paste and, hence, its durability [9]. This was possible, because the higher refractive index of water relative to air would decrease the reflectance of light at the fiber tip.




(a)




(b)

Figure 1. (a) Cement paste color optical fiber sensor setup. Light from an LED is reflected at the tip of a 600 µm multimode fiber embedded in a cement paste sample and measured by a spectrometer. (b) The two different types of cement paste samples used in the monitoring of cement paste carbonation.

Carbonation is a slow process which takes years to occur at ambient $CO_2$ concentrations. Raising the $\%CO_2$ increases the speed of carbonation, albeit it also changes the chemical composition of carbonated cement paste, if atmospheres with more than $3\%CO_2$ are used [21]. Thus, 100 $\%CO_2$ will be used throughout these measurements, and a $3\%CO_2$ measurement will be used to validate our results. Smaller sized samples are key in obtaining results in a timely manner and so is using the optimal conditions for carbonation, namely 60% RH. Three different samples were used, table 1, two of them were produced by dipping the optical fiber in a mixture of 0.5 w/c (water to cement ratio), and leaving the cement paste that adhered to the fiber to hydrate under 100% RH, samples A and B.

Table 1: Cement paste samples used, their type as seen in figure 1 (b) and its carbonation parameters

| Samples Name | Samples Type | $\%CO_2$ | RH % |
|---|---|---|---|
| A | 1 | 100 | 60 |
| B | 1 | 3 | 60 |
| C | 2 | 100 | - |

The diameter of the optical fiber embedded in the cement paste for these samples is around 1.3 mm. One of these thin samples will be carbonated at $100\%CO_2$, sample A, and the other will be carbonated at $3\%CO_2$, sample B. A third and larger sample will be used, sample C. This sample was produced by embedding the multimode sensing fiber into unhydrated cement paste (0.5 w/c), sealing the cement paste from the outside environment, and leaving it to cure. This sample will be carbonated with the goal of measuring how this process influences the wet/dry signal of the cement paste sample. Water ingress was previously used to monitor cement paste durability, but for the monitoring of cement paste carbonation this signal change could be a source of uncertainty and vice-versa.

## 3. Results

### *3.1. Carbonation at 3 and 100 %$CO_2$*

The general signal response of monitoring cement paste carbonation through cement paste color is demonstrated in figure 2 (a) and (b), from samples A and B. In both sets of data there is an increase in the reflected light intensity, caused by the carbonation of cement paste which changes the cement paste color from gray to a lighter tone of gray.

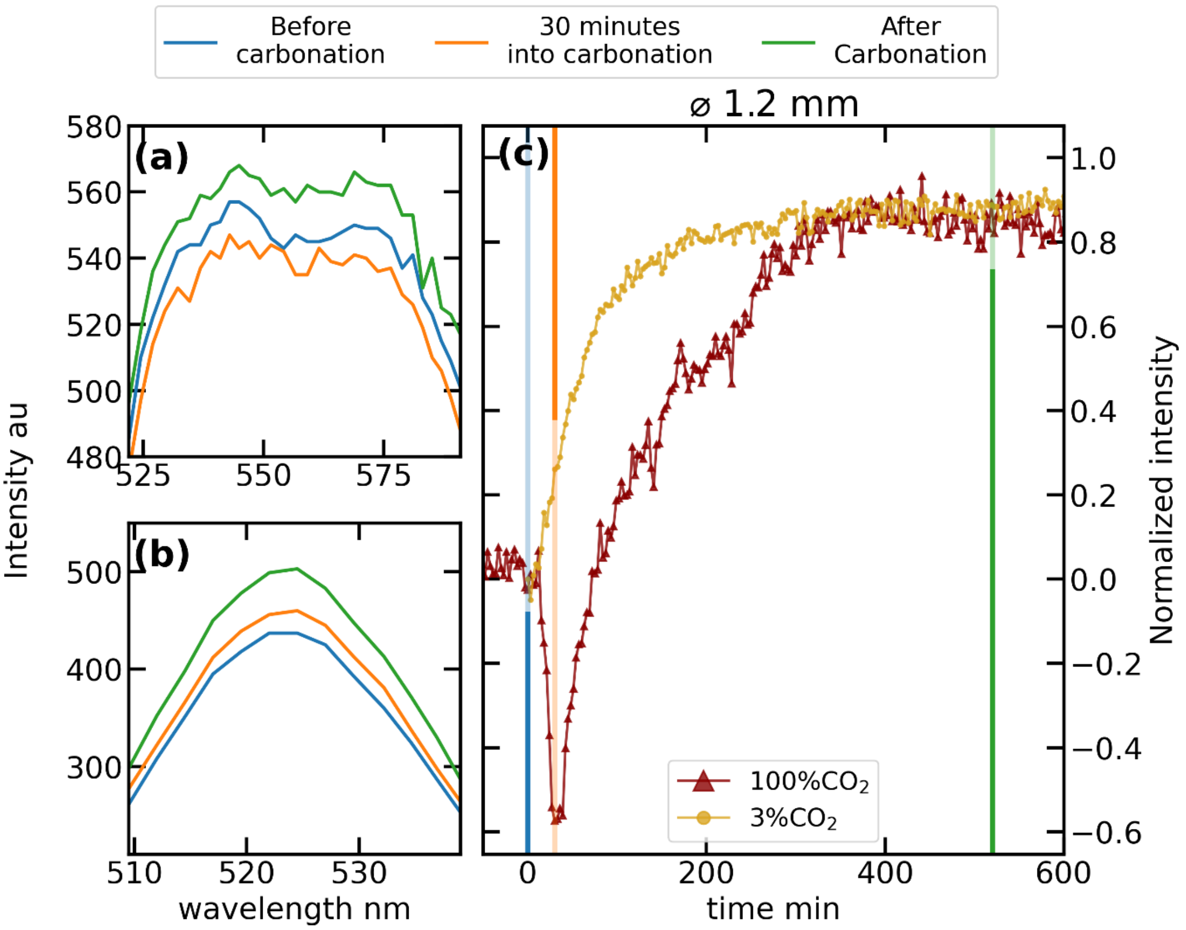



Figure 2 (a) Change in light intensity reflected at the fiber due to the change in color of the cement paste at 100%$CO_2$, sample A. White LED intensity measured before carbonation at blue, 30 min into carbonation at orange and after carbonation in green. (b) Reflected Light intensity change of a green LED for carbonation at 3%$CO_2$, sample B. (c) Comparison of carbonation of samples A and B.

It is also seen that in the first 30 minutes of carbonation at 100%$CO_2$ the signal intensity drops as opposed to carbonation at 3%$CO_2$ which kept rising throughout. These results are better seen in figure 2 (c), where the response of both is compared by aligning the start of cement paste carbonation, at 250 minutes, and normalizing the data to the light intensity before carbonation had started. With the intensity in each data point being the sum of intensities of wavelengths displayed in figure 2 (a) and (b). In this figure carbonation at 3%$CO_2$ is faster than the one occurring at 100%$CO_2$. And, sample A has a distinct feature that does not occur at 3%$CO_2$ (sample B): a signal drop at the start of cement paste carbonation. This drop is likely caused by the exhaust of water from cement paste carbonation. At 100%$CO_2$ there is more water released from cement paste carbonation, then is or can be exhausted outwards. This leads to a buildup of water in the capillaries pores which reduces the refractive index of the cement paste previously filled with air. This in turn reduces the reflectance of light at the fiber tip, leading to the reduction of the measured signal. The slower rate of carbonation at 100%$CO_2$ is also explained with this buildup of water, which slows the diffusion rate of $CO_2$ in the capillary pores. The results recorded at 3%$CO_2$ can be used to conclude that this sensor can be used under natural carbonation conditions [21].

### 3.2. *Water Ingress and Carbonation*

Sample C, larger in size relative to the other samples, was submerged in water and left to dry to obtain its wet/dry signals. The sample is then carbonated at 100%$CO_2$ and finally submerged in water to achieve the new wet/dry signals. As it is seen in figure 3 the wet/dry signal travels upwards with carbonation, this is indicative that the setup, previously found to monitor water ingress/egress is measuring more than this phenomenon. Considering that the sample is being forcibly injected with 100%$CO_2$ it is concluded that the setup is also monitoring cement paste carbonation. The monitoring of cement paste durability is done by transforming the wet/dry signal into height of water at the fiber tip and measuring this value against the square root of time. Thus, it doesn't matter if the wet/dry signals drift, as much as both signals remain relatively stable. The ingress/egress of water denotes faster signal changes than forced carbonation of cement paste, which should hold in the case of natural carbonation which is far slower. And the carbonation dip at 100%$CO_2$ from the build-up of water, recorded for the smaller sized sample A, was also recorded for sample C. Hence, this setup also monitors water being released from cement paste due to forced carbonation.

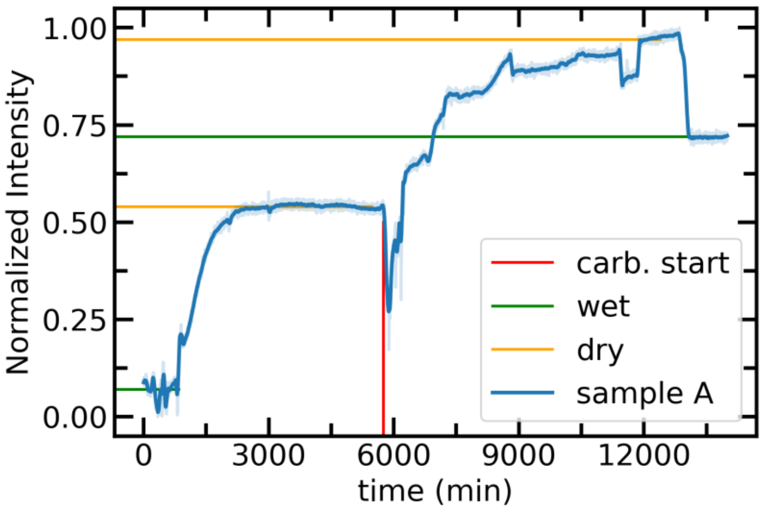



Figure 3. Influence of carbonation in the wet/dry signals previously used to monitor cement paste durability.

## 4. Conclusion

The color changes of cement paste were successfully used to produce a colorimetric optical fiber sensor that monitors the forced carbonation of cement paste samples with different sizes, figure 1. As carbonation changes the color of cement paste into a lighter tone of gray the optical system captures the increase in light intensity being reflected at the fiber tip. This sensor is also sensitive to changes in the refractive index that might occur in the vicinity of the fiber tip. For this reason, it could monitor the formation of water in the capillary pores from carbonation at 100% $CO_2$, figures 2 and 3. The possibility of this sensor being used for the monitoring of natural carbonation was positively evaluated through

3%$CO_2$ carbonation, which also induced an increase in reflected light intensity. This is an important result given that the carbonation products at 3% $CO_2$ are similar to those produced from natural occurring carbonation, where the concentration of $CO_2$ is 400 ppm. The measurement of light intensities from wet and dried cement paste and the upward intensity drift from cement paste carbonation suggest that this setup can be used to monitor both the durability of cement paste, as well as cement paste carbonation. All this in a simple optical fiber sensor setup, that can be upgraded into a low-power, low-cost optical fiber sensor if the spectrometer is replaced with a photodiode.

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