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Determination of pH in Powdered Concrete Samples or in Suspension

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Featured Application: Construction industry—application in determination of pH value of concrete.

Abstract: Concrete is a material that is widely used by mankind. Although different deterioration mechanisms can lead to degradation of the concrete itself, reinforcement corrosion is the biggest durability issue for reinforced concrete structures. One of the key parameters influencing the corrosion rate is pH value. Accordingly, this work presents two capacitive sensor platforms—one based on parallel plate electrodes and the other based on a planar interdigitated electrode structure. The first platform is used to determine whether the pH value is lower or higher than a predetermined limit (around 11) and this device was successfully tested using concrete suspensions. The second platform can determine the pH value by establishing a relationship between pH and measured capacitance from a powdered concrete specimen. Both multi-layered platforms were manufactured by means of a cost-effective xurography technique, which provides technically and mechanically robust structures very quickly.

Keywords: pH value; concrete; microfluidics; capacitive sensor; xurography

1. Introduction

The majority of urban infrastructure such as buildings, bridges, roads and tunnels are built from reinforced concrete. Reinforced concrete structures may deteriorate due to exposure too severe conditions related with the environment—carbonation, chloride ingress, freeze-thaw effect, abrasion/erosion or chemical action.

Although different deterioration mechanisms can lead to degradation of the concrete itself, reinforcement corrosion is the biggest durability issue for reinforced concrete structures, and can lead to the cracking, loss of bearing capacity and catastrophic failures. To maintain the structural integrity of our extensive urban concrete infrastructure and prevent catastrophic situations it is very important to implement regular inspection regimes that include the measurement of pH [1].

The pH value (defined as the negative logarithm of the hydrogen-ion concentration) in this domain represents the level of alkalinity of concrete under different conditions. Portland cement paste in fresh and hardened concrete has a pH value usually between 12.5 and 13.8 [2], and in this high alkalinity environment a thin passivation layer is formed above the steel surface that prevents the rebar from corrosion [3]. The carbonation process, in which calcium hydroxide in concrete is transformed into calcium carbonate, can decrease the pH of concrete and active corrosion will begin near the rebar once the alkalinity level (pH) is reduced to values 9.0–9.5. As one of the key parameters influencing the corrosion rate together with the temperature (T) and relative humidity (RH), the precise methods of measuring pH in concrete are of great interest to science and industry. The in situ leaching method for measuring the pH in concrete was based on inserting a glass micro pH electrode into a hole drilled into the concrete sample [4]. An Ag/AgCl electrode was used as the reference electrode. The potential difference between the working and the reference electrode was measured and translated to pH values. However, this method is time-consuming and destructive [5]. Hence, considerable effort has recently been invested in developing non-destructive techniques based on the application of different embedded sensors [6]. A multifunctional sensor consisting of pH and chloride sensors was reported in [7]. The sensor was embedded in the concrete at a depth of 10 mm and a drop in pH was noted in as little as 120 days. However, embedded sensors cannot be calibrated in situ and they can be broken inside the concrete. Elsewhere, fibre optic sensors for measuring pH in concrete were proposed [8]. Necessary components for performing this measurement are a light source, a coupler, a fibre switch, a spectrometer, a sensor and a PC [9]. A disadvantage of optics sensors is that they are chemically instable. Phenolphthalein is a commercial indicator that can be used for visual estimation of carbonation because it changes colour from red-violet (non-carbonated areas) to colourless (carbonated areas) within a pH range of 8.9 to 9.8 [10]. Alizarin yellow-R and indigo carmine were also applied as pH indicators in concrete [11]. These indicators are spread onto a cross-section of the specimen; thus, they are also able to detect the carbonation depth [12]. However, the information provided by these indicators regarding the actual carbonation status of concrete is limited by their very narrow pH range. In [13] the authors proposed the application of pH strips for verification of concrete carbonation status, mixing concrete powder with a few drops of water. Elsewhere, a halochromic method was applied for mapping pH values of Portland cement concrete [14]. The measuring system was composed of a white light source, a camera, and two optical filters. More accurate measurement of pH values in concrete have been achieved by various methods including: (a) a flat-surface electrode system in combination with filter paper [15], (b) a sensor based on a three-electrode electrochemical test [16] and (c) Ag/Ag_2O sensors manufactured using a screen-printing process, which demonstrated good response time and reproducibility [17]. Further, a technique for measuring the free chloride concentration and pH value of mortar specimens was reported in [18] based on several Ag/AgCl ion-selective and iridium/iridium oxide electrodes. X-ray diffraction and thermogravimetric analysis [19] and the electrochemical impedance spectroscopy method [20] have also been developed. The latter method was time efficient, and was applied to analyse concrete samples exposed to a marine environment at five different pH values [21].

All the above-mentioned methods have unique advantages and drawbacks and it is clear that there is a need for efficient and accurate devices for measuring the pH of concrete, including in the binary form to distinguish pH-High (in the secure zone) from pH-Low (in the dangerous zone), as a first step in development of a new measurement procedure. The portable sensor platform is of particular interest to professionals engaged in inspection jobs and can help experts to take appropriate decisions about interventions and remedial work on time.

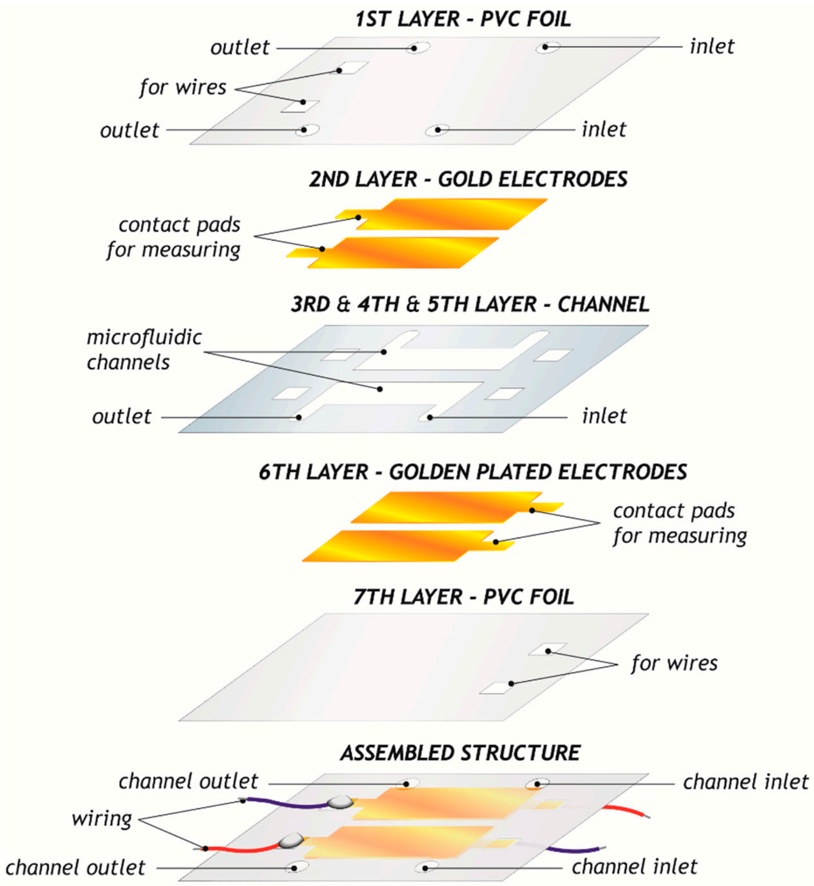
This article presents two sensors, one for measuring the pH of ground concrete powder in a suspension, and another one using the same powdered concrete, but dry, which are important from not only a scientific point of view, but also from a commercial point of view. Both sensor platforms were fabricated using cost-effective xurography technique. This method creates multi-layered structures, laminating separate transparent and mechanically flexible polyvinyl chloride (PVC) foils and using gold leaf as a conductive material. The first sensor platform is a microfluidic device which works on the principle of parallel capacitive electrodes. Concrete powder suspended in distilled water was injected into microfluidic channels between the two gold electrodes of a capacitor. The second platform is an interdigitated capacitive planar electrode system. Concrete powder was dispersed onto the upper side of the electrodes, changing the total capacitance of this structure in a pH-dependent manner. Electrical characterization of both sensor platforms was performed using an Impedance Analyzer.

2. Materials and Methods

2.1. Microfluidics-Based Platform

Figure 1a shows the design of the first sensor platform, whereas Figure 1b depicts the complete fabricated structure. The structure was designed to have two parallel microfluidic channels, one containing pH-Low concrete solution injected via the relevant inlet hole and the other containing pH-High solution similarly injected. A comparison of these solutions is allowed by measuring output capacitance at the contact pads (terminals). The first layer, similarly to subsequent layers of this multi-layered device, used 80 µm-thick PVC foil as a substrate. One side (electrode) of the parallel plate capacitors was created from gold leaf with dimensions of 3 cm × 0.7 cm, each. In the middle, discrete, disconnected channels were created. Each channel was created from 3 PVC foil layers giving the thickness of the channel (distance between gold electrodes) equal to 240 µm. The next layer represents the second electrodes of the parallel plate capacitor. The thickness of the gold conductive material was 10 µm. In the last layer, holes for inlets and outlets for fluid injection were created. From the fabricated structure two short wires were connected, using silver paste, to create through-hole components and to enable measurement. The overall dimensions of this platform, shown in Figure 1b, are 5 cm × 3.5 cm.

The main fabrication steps of this platform were as follows: (1) putting the foil in a cutting plotter machine; (2) cutting the foil in the overall dimension of the chip; (3) engraving holes for contacts; (4) gluing gold conductive leaves for bottom and top electrodes; (5) engraving microfluidic channels; (6) cutting holes for inlets and outlets; and (7) laminating separate layers of PVC foils to create a multi-layered compact microfluidic sensor platform.



(**a**)

(**b**)

Figure 1. (**a**) Design of separate layers, and (**b**) the fabricated microfluidic-based structure.

The working principle of this platform is based on a parallel electrodes capacitive sensor. More specifically, when the suspension of the concrete sample is injected into inlets through the microfluidic channels and goes out of the outlet, the channel is full with the suspension, increasing the dielectric constant of the medium between the gold electrodes. If the dielectric constant is changed then the total capacitance is changed and this variation is correlated with the pH value of the concrete in suspension form, inside the microfluidic channels.

2.2. Interdigitated Electrodes-Based Platform

An interdigitated electrodes-based platform was fabricated on a PVC foil substrate using xurographically engraved gold (Ag) electrodes in the form of planar interdigitated capacitors (IDC), with either 4 or 5 fingers, as illustrated in Figure 2a. The width of these electrodes, including the gap between them, was 2 mm. The total dimensions of both IDC structures, with either 4 or 5 fingers, were 1.5 cm × 2.5 cm. Figure 2b displays this manufactured multi-layered structure after lamination.

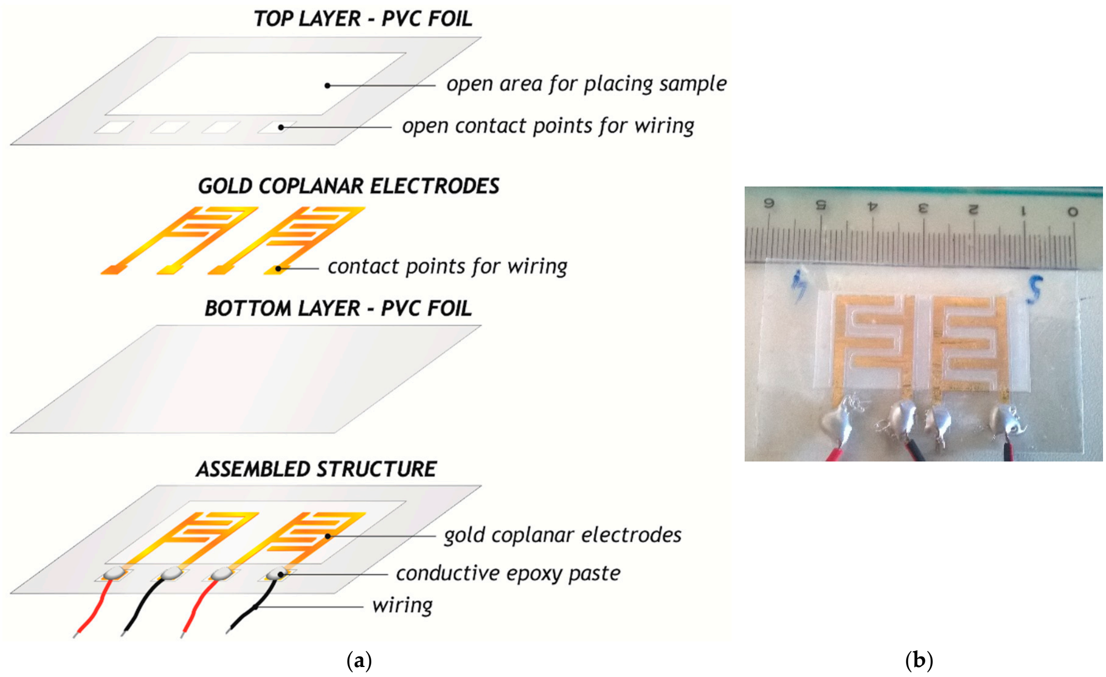


Figure 2. (**a**) Design of separate layers, and (**b**) the fabricated interdigitated capacitor (IDC)-based structure.

The working principle of this structure is based on changing permittivity among the fingers of the interdigitated capacitive structure when the concrete sample in powder form is placed on the top of the device. The pH value of powdered concrete has influence on permittivity and this will change the capacitance which is measured at the terminals. The size and dimensions of this structure were determined to be robust enough from one side and from another side to ensure the appropriate range of the capacitance variation which results in adequate sensitivity.

The reason for fabricating and using IDC-based structure is to cover both the possibility to analyse concrete samples in suspension (liquid form), using the already-presented microfluidics-based platform in Section 2.1, as well as to analyse concrete samples in powder form, which is more useful for applications on-site, where it is difficult to make suspensions, but it is easy to make powder from the concrete structure in some object.

2.3. Sample Preparations and Instruments

Two types of concrete samples were used in this investigation—carbonated and non-carbonated. They both originated from the same concrete specimen taken from the 30-year-old concrete structure exposed to an open environment during its whole service life. The specimen has been tested by a phenolphthalein test (Figure 3) and two zones can be obviously detected—a white layer of carbonated concrete (low pH: 10.79) and a pink layer of non-carbonated concrete (high pH: 12.24). From each of these two layers, samples were taken and prepared for pH-value testing using the following procedure: (1) concrete was chopped, milled and passed through a sieve with pores of 100 µm to obtain a fine powder; (2) four grams of sieved concrete was suspended in 50 mL distilled water; (3) the solution was mixed using a shaker; and (4) a small amount of solution was taken by syringe and the pH-Low suspension injected into one channel and the pH-High suspension injected into another channel of our microfluidic-based platform (Figure 1).

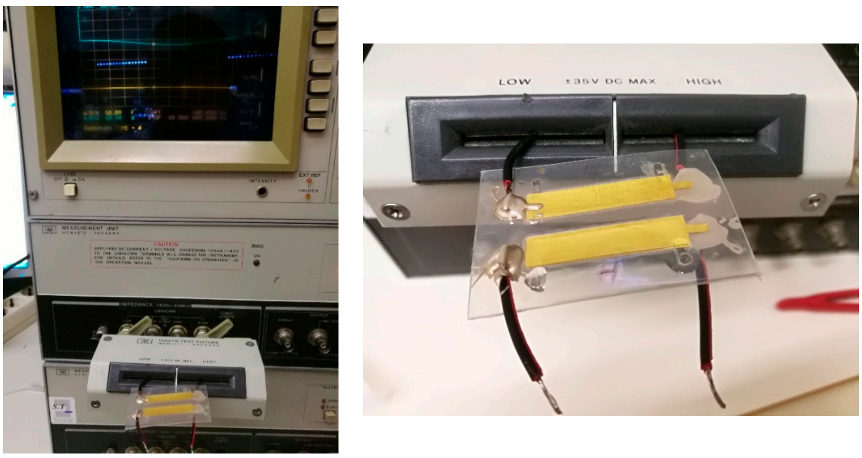
(**a**) (**b**) (**c**)

Figure 3. Studied concrete samples in (**a**) solid, (**b**) powder, and (**c**) suspension form.

In the IDC-based platform, the powder created in the above-mentioned step (1) was coated above the platform in the active area of the electrodes (also shown in Figure 2a). We used a cutting plotter (CE6000-60 PLUS, Graphtec America, Inc., USA) for cutting PVC foils and gold leaf in the desired pattern. Xurography technique used a cutting blade (CB09U) with a 45° angle. Separate layers were laminated by means of a thermal laminator, under elevated temperature (130 °C), with the aim of obtaining a compact, but technically robust structure.

2.4. Experimental Setup

Figure 4 presents the experimental setup for measurement of electrical parameters using an Impedance Analyzer HP4194A (Keysight technologies former Hewlett-Packard, Palo Alto, CA, USA) governed by a PC Dell Intel Pentium (Dell Technologies, Austin, TX, USA). The microfluidic platform was filled with suspension by means of syringe and short tube, which are shown in Figure 3c as well. One microfluidic channel was filled with pH-Low solution and another channel with pH-High solution. On the Impedance Analyzer, short wires from the platforms were used for connecting with the holder for the device under testing.



(**a**) (**b**)

Figure 4. Experimental setup for the microfluidic-based platform (**a**) Impedance Analyzer HP4194A with the sample holder, (**b**) Developed microfluidics-based device under testing phase.

3. Results and Discussion

We analysed two concrete solutions, one denoted with pH-Low and another with pH-High. Capacitance between parallel plate electrodes was measured with these two suspensions in separate channels. The measured results are shown in Figure 5.

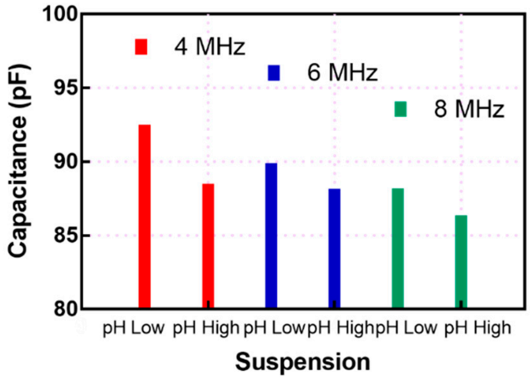
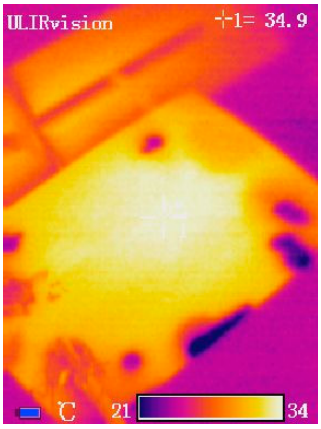


Figure 5. Measured capacitance for two different concrete solutions at room temperature.

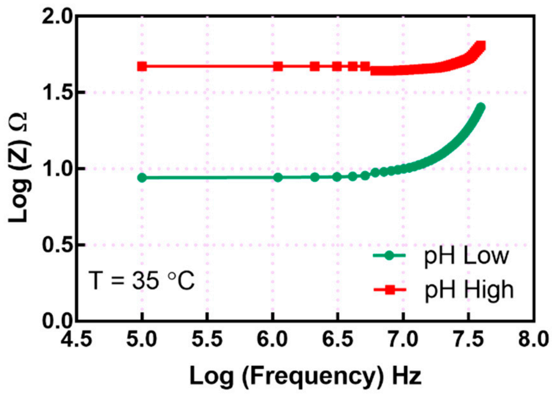
We selected three frequency points (Figure 5), and the results demonstrate that for each frequency, capacitance of pH-Low concrete solution was higher than that of the pH-High concrete solution. The obtained capacitance was in the range from 86.3 pF to 92.5 pF. It can be noticed that capacitance values decreased with increasing frequency, due to the same behaviour expressed by the dielectric constant of the fluid. It is known that the value of the dielectric constant decreases from the static state (low frequency range) through all four polarizations, up to the highest frequencies. The capacitance of the parallel plate electrodes capacitive structure can be calculated by

$$C = \frac{\varepsilon_0 \varepsilon_r A}{d}, \tag{1}$$

where $\varepsilon_0 = 8.85 \times 10^{-12} \frac{F}{m}$, ε_r is the relative permittivity of the fluid inside the channel, A is the surface area of the parallel plate electrodes and d is the distance between them. If we apply the dimensions of our structure in the mentioned equation, the total capacitance is 92.93 pF (for a relative dielectric constant equal to 1, which is well in agreement with results of measurements presented in Figure 5). It can be concluded that a less alkaline solution (pH-Low) will have higher capacitance, taking into account that the solution has a higher relative dielectric constant. Namely, the concrete belongs to dielectric materials which are characterized by the fact that valence electron shells are nearly full [22]. The relative permittivity of concrete is larger when it is easier for the material to polarize, meaning that the ions are mobile and there is little crystallization. The dielectric constant can be increased by adding chlorides as well as additional amounts of water. The relative permittivity of concrete is also dependent on its aggregates. Using the proposed microfluidic device it is possible to quickly provide a binary response: (a) this is a pH-Low concrete solution, which would indicate an alarm for intervention in that construction, or (b) this is a pH-High concrete solution, an indication that there is low risk of pH-related corrosion in the concrete and the concrete structure should have appropriate integrity. In order to explore the influence of temperature, the presented platform was heated at 35 °C, as can be seen in the infrared image shown in Figure 6a. Figure 6b presents the so-called Bode plot, i.e., measured impedance as a function of frequency (both in logarithmic scale).



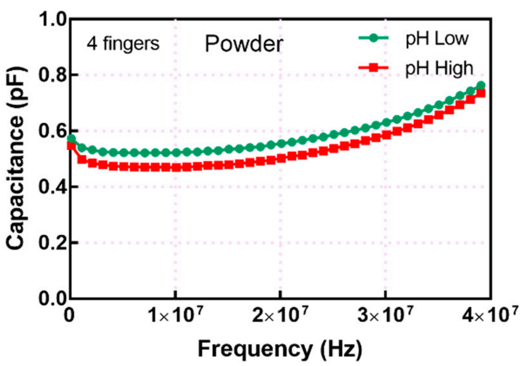
(a)



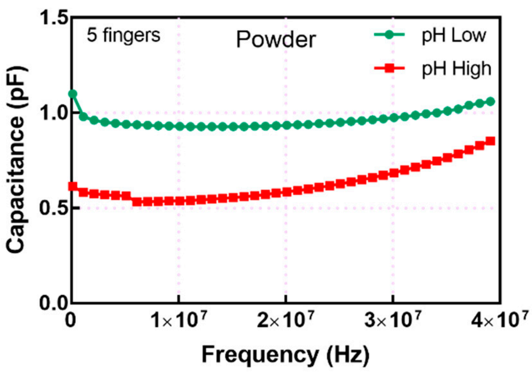
(b)

Figure 6. (**a**) Infrared image of the microfluidics-based platform at an elevated temperature, and (**b**) Bode plots for pH-Low and pH-High concrete suspensions.

It can be concluded that the pH-High sample has higher impedance due to the inverse dependence between capacitance and impedance. It is important to note that relative permittivity of concrete decreases with increasing temperature, because the amount of water is reduced during this time. This will result in a slight decrease in the output capacitance with increasing temperature. For example, when the temperature was increased from 25 °C to 35 °C, the capacitance decreased for approximately 3%. For determination of a more precise pH value we propose an IDC-based structure. This platform can also be exposed to liquid medium, but we successfully applied concrete in powder form to the upper side of the gold interdigitated electrodes system. The capacitance as a function of frequency was experimentally measured at room temperature for both 4- and 5-fingered gold electrode designs, and results are presented in Figure 7.



(a)



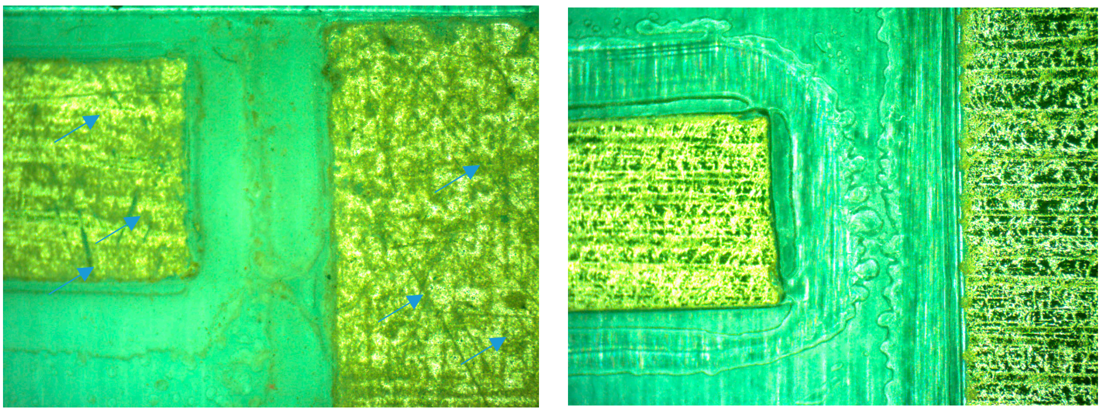
(b)

Figure 7. Capacitance as a function of frequency for pH-Low and pH-High concrete powder with (**a**) four and (**b**) five interdigitated fingers/electrodes.

As can be seen in Figure 7, both IDC-based capacitive sensors showed similar behaviour: The capacitance increased with increased alkalinity. Note also that the sensor with 5 fingers had higher values of capacitance, the result of a larger active area. The IDC-based structure with 4 fingers has a capacitance equal to 0.52 pF for pH-Low concrete powder and 0.47 pF for pH-High concrete powder, at 8 MHz. We tested two concrete samples, one with a pH value equal to 10.79 (pH-Low, below boundary value = 11) and another one with a pH value equal to 12.24 (pH-High, higher than boundary value = 11). These precise pH values were measured by means of HQ440d Benchtop Multimeter (Hach, CO, USA). This small difference in pH values of tested samples caused the small difference in the capacitance value, but even in this case the difference is enough to make decisions if it is a pH-Low or pH-High sample, which has huge practical importance. The difference in capacitance was even higher when the IDC-structure with 5 fingers was used. These values are 0.93 pF for pH-Low, and 0.54 pF for pH-High concrete powder (at 8 MHz). This difference is a consequence of an additional "mini" capacitor created between the fourth and fifth fingers in the structure with five gold electrodes, but also the interaction of this fifth finger with all others, bearing in mind that electrical field lines are closed among all fingers in this coplanar structure through the sample of concrete from the top side. When this IDC-based platform was covered with concrete samples in powder form, the relative permittivity between the electrodes of the planar capacitive structure was increased. The real part of the complex relative permittivity of concrete is in the range from 4 to 12 [23], compared to the dielectric constant values in air, which is equal to 1. Therefore, when the concrete powder particles enter between electrodes, it causes the capacitance to increase. It can be concluded from Figure 7 that in the high frequency range capacitance starts to increase, which means that resonant frequency will occur at frequencies which are out of the measuring range of the applied Impedance Analyzer HP4194 (≤40 MHz).

The proposed platforms can be used on site, near to the construction structure, using our in-house developed portable and low-power microprocessor system for remote measuring of environmental parameters [24], including pH value. That means instead of a bulky Impedance Analyzer (in the laboratory), on site we can use our small, portable, impedance/capacitance meter, along with the presented sensor platforms in this paper.

Thanks to the selected fabrication technology, the proposed platforms are reliable and have long-term stability. Related to the reusability, for the microfluidics-based platform it is necessary to remove the previous suspension and to fill the channel with another one, whereas the IDC-based platform needs to be cleaned with a brush and covered with new powdered concrete samples. The presented sensor lifespan is mainly determined by the endurance of the gold layer. The interdigitated sensors platform is more prone to the appearance of scratches in the gold electrodes (Figure 8) due to using powdered concrete which limits its usage to approximately hundred times.



(**a**) (**b**)

Figure 8. Magnified gold electrode segment of an interdigitated capacitive structure for (**a**) a sensor used more than 60 times, degraded with many scratches (labelled with arrows), and (**b**) for a new fabricated sensor.

The microfluidics-based structure does not have this limitation bearing in mind that the suspension is used and the applied pressure on the syringe during the filling channel is not high.

4. Conclusions and Future Work

This work has presented two sensor platforms for quick evaluation of the pH of powdered concrete samples. The first platform is a microfluidic-based platform for binary decision-making based on whether the solution has a pH value lower or higher than the critical value, around 11, when corrosion is likely to begin. The second developed sensor platform is based on an interdigitated coplanar capacitive structure that changes dielectric constant values and consequently capacitance when the surface is covered with a concrete sample in powder form. The convenient, very tiny and light platforms presented can strongly support decision-making procedures in engineering practice and will significantly improve condition assessments of reinforced concrete structures. Their application will be particularly useful in a multi-aggressive environment. It is important to note that the same method can be applied for a new generation of "green" concretes with supplementary cementitious material, such as fly ash, slag or concrete with recycled aggregate. Future work will be focused on: (a) experimental testing extension on the concrete samples taken at different depths of a concrete specimen; (b) fabrication of an inductive–capacitive (LC) resonant circuit structure in one gold metal layer and achieving in that way a wireless detection of pH value through determination of resonant frequency changes; and (c) realization of an LC structure on flexible Kapton film foil with a carbon nanotubes coating and rolled around the rebar in the concrete structure.

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Conflicts of Interest: The authors declare no conflict of interest.

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