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Embeddable sensors for in-situ monitoring of chloride and pH in concrete

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Embeddable sensors for in-situ monitoring of chloride and pH in concrete

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SUMMARY

This report summarises the development of embeddable sensors for monitoring the chloride content and the pH in concrete.

First, a basic introduction to sensor technology (electrochemistry and design) is given. Then a summary of publications dealing with this topic is presented. The report also offers a principal guideline on how to manufacture such embeddable sensors (Appendix 1 and 2).

Appendix 3 gives a comprehensive and chronological overview of open literature (published 2024→1996) covering embeddable sensors for monitoring chloride and pH in concrete.

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Table of contents

1	Introduction	3
2	Sensor types in general.....	3
2.1	Ion-selective electrochemical sensors	3
2.2	Non-electrochemical sensors.....	4
3	Chloride sensors for concrete.....	5
4	pH sensors for concrete	5
4.1	Electrochemical pH sensors	6
4.2	Fiber optical pH sensors.....	6
5	Commercial chloride and pH sensors for concrete	7
6	Conclusions	7
7	References	7
8	Appendix	9
A	Embeddable chloride electrode for concrete	9
B	Embeddable pH electrode for concrete.....	10
C	Literature overview	11

1 Introduction

It is well established that the presence of chloride and/or low pH of in concrete structures will initiate corrosion of the steel reinforcement if the levels are within a certain range. Therefore, it would be good to monitor the level of these two initiators over time (years) at various concrete cover depths.

After a brief introduction to relevant sensor types in general, this report summarises what has been published about chloride and pH sensors for in-situ monitoring of concrete structures. It also offers guidelines on how to manufacture such embeddable sensors (Appendix 1 and 2). Appendix 3 gives a comprehensive and chronological overview of open literature (published 2024→1996) covering embeddable sensors for monitoring chloride and pH in concrete.

2 Sensor types in general

Almost all reported/documented embeddable sensors for monitoring chloride in concrete are electrochemical in nature and are based on an ion-selective electrode (ISE); more specifically a Cl^- -selective electrode. There are only a few exceptions which are based on non-electrochemical techniques, like optical fiber sensors.

Most pH sensors are also based on an ISE; in this case a H^+ -selective electrode. However, non-electrochemical pH sensors are also reported, typically optical fiber pH sensors.

Before describing the specific sensors used for monitoring chloride and pH in concrete, the basics of this sensor technology is briefly described.

2.1 Ion-selective electrochemical sensors

An ion-selective sensor consists of an ion-selective electrode (ISE) connected to a reference electrode through a voltmeter. It can also be termed ‘a potentiometric sensor’ since the electrochemical potential measured is dependent on the ion activity on the ISE surface [1]. This combination of an ion-selective electrode and a reference electrode is termed an ISE cell (Figure 1).

Note that the term ‘selective’ is used – not the term ‘specific’ – because the ISE can also be affected by other ions (interfering ions) present in the electrolytic solution in the concrete pores.

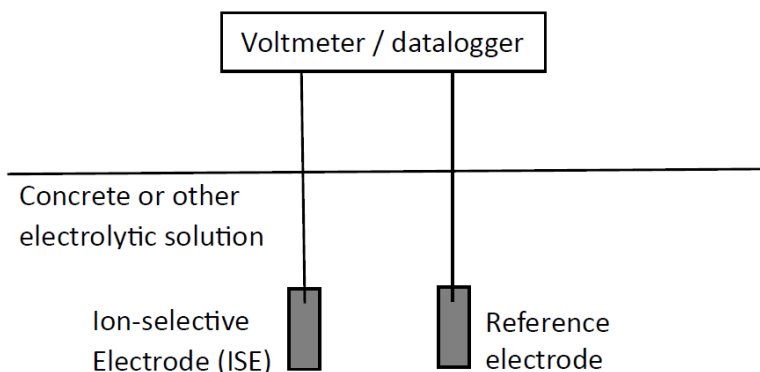


Figure 1 A schematic setup of an electrochemical sensor of type ISE cell.

The electrode material of an ISE is made of a metal and a chemical compound formed by this metal:



where M is the metal, X is a chemical element bound to the metal, and y and z are whole numbers.

The activity of the ion to be measured (dissolved, not chemically bound) in contact with the ion-selective electrode (ISE) will affect the electrochemical response of the ISE. This response varies with the concentration of the ion which can be detected as a change in the electrochemical potential difference between the reference electrode (with a known/fixed potential) and the potential of the ISE.

There are various ISEs tailormade for different ions. Type of ISE material will affect how the ISE responds to the ion. For example, to measure the concentration of dissolved Cl^- ions (for instance in the moisture inside concrete pores), silver is the most used, whilst iridium is typically used to measure the concentration of H^+ (pH).

2.2 Non-electrochemical sensors

Probably, the most studied non-electrochemical sensor type for measuring environmental factors affecting corrosion in concrete is based on optical fiber technology. An optical fiber is a very thin, flexible, transparent, and cylindrical waveguide (to “guide” the light) made of plastic or silica [2]. The sensor contains an indicator dye that changes its optical properties in the presence of the substance to be investigated. These changes comprise colour and/or colour intensity, luminescence properties (fluorescence or phosphorescence) or other photo-physical properties [3].

There is a variety of chemicals, materials, and designs used to manufacture such optical sensors. One design is to measure light of a specific wavelength by converting the light rays into electric signals. Another design is to use a fluorescent dye which changes colour depending on the chemistry of the measured medium, a technique used for decades in many areas of biotechnology, medical technology, and life sciences [3]. The principal set-up of an optical fiber sensor in general is shown in Figure 2.

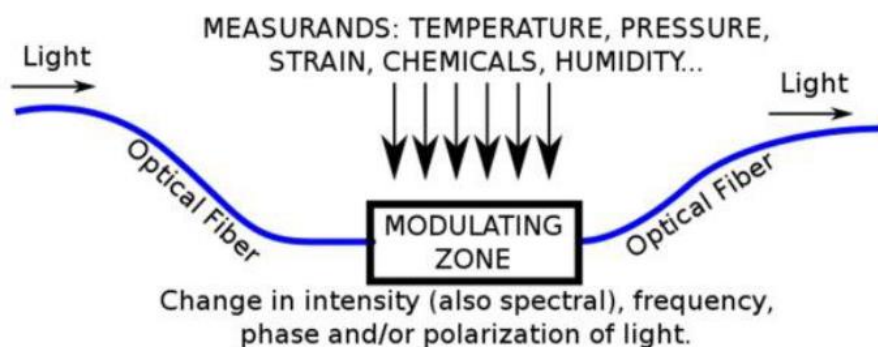


Figure 2 The basic schematic set-up of optical fiber sensor. The light that is fed into an optical fiber reach a zone where the light is being modulated due to the interaction with the parameter of interest. Then, the modulated light propagates, via an optical fiber, to a monitor [4].

3 Chloride sensors for concrete

Embeddable chloride sensors for concrete are – with hardly any exceptions – made of the ion-selective electrode silver/silver chloride (Ag/AgCl). This is essentially a version of the well-known Ag/AgCl/KCl reference electrode where the integrated fixed concentration of chloride (saturated potassium chloride solution) is removed so that the Ag/AgCl material is ‘naked’, that is directly (physically/chemically) exposed to the concrete.

The chloride concentration is measured as an electrochemical potential reflecting the concentration of chloride ions (solved, not bound) in the concrete in contact with the ISE. The concentration of chloride ions (or more precisely the chemical activity) is given by the Nernst law, simplified as [5, 6]:

$$E(\text{Ag/AgCl}) = E^0(\text{Ag/AgCl}) + k \cdot \log[\text{Cl}^-]$$

where E is the measured potential against the reference electrode, E^0 is the standard potential of the ISE (known value), k is a constant at a given temperature, and $[\text{Cl}^-]$ is the molar concentration of chloride (mol/L).

The first papers describing ion-selective Ag/AgCl electrodes for in-situ chloride monitoring in concrete were published almost 30 years ago. In 1996 two papers presented the results from laboratory studies of self-made Ag/AgCl electrodes embedded in cement paste [7] and cement mortar [8] containing different amounts of chloride salts. The electrochemical potentials were not measured using embedded reference electrodes, but by using external reference electrodes in physical contact with the cement paste/mortar. Typical external reference electrodes used were saturated calomel electrode (SCE) and saturated Hg_2SO_4 electrode. These reference electrodes are unsuitable for in-situ monitoring because the liquid electrolytes inside them may leak or become invalid if exposed to cold temperatures [9].

Since then, several papers on this topic have been published with the same Ag/AgCl electrode, but with a new type of embeddable reference electrode for concrete, mostly the alkaline manganese dioxide type (Mn/MnO_2). In a recent paper, this electrode combination has been described in detail [6].

Ion-selective electrodes are chemically stable and quite easy to manufacture. However, in-situ monitoring (ISE embedded in concrete) over several years can be challenging due to lack of long-term stability of the reference electrode, not the ISE [10]. Other factors like temperature, pH and interfering ions may also affect the electrochemical potential of the ISE and cause inaccurate measurements [6, 10]. The electrochemical potential response of today’s Ag/AgCl electrodes for a given concentration of chlorides is reported to have an accuracy of approximately ± 0.05 mol/L [11].

Comprehensive recent studies on Ag/AgCl electrodes for use in concrete is given by Wang et al in 2024 [21] and Zhang et al in 2020 [10].

A detailed procedure for manufacturing an embeddable Ag/AgCl electrode for concrete is presented in Appendix 1.

4 pH sensors for concrete

Most pH sensors are also based on an ISE; in this case a H^+ -selective electrode. However, non-electrochemical pH sensors are also reported, typically optical pH sensors (Chapter 4.2).

4.1 Electrochemical pH sensors

An electrochemical pH sensor for in-situ monitoring in concrete is typically based on the ISE design with iridium/iridium oxide (Ir/IrO_x) as the 'sensing' material. This electrode material – like many other metal/metal oxides – responds to changes in the concentration of H⁺ ions at the electrode/concrete interphase [12].

The pH (or more precisely the chemical activity of H⁺ ions) is given by the Nernst law, simplified as [13]:

$$E(\text{Ir/IrO}_x) = E^0(\text{Ir/IrO}_x) - k \cdot \text{pH}$$

where E is the measured potential against the reference electrode, E⁰ is the standard potential of the ISE (known value), and k is a constant at a given temperature.

Often, the Ir/IrO_x electrode is termed Ir/IrO₂ assuming that exactly two oxygen atoms are bonded to one iridium atom, which is probably almost correct in most cases. However, in this report the term Ir/IrO_x will be used as a general formula.

The first paper describing an Ir/IrO_x electrode for in-situ pH chloride monitoring in concrete was published almost 20 years ago [14]. Since then, several researchers have studied and further developed this sensor technology.

The Ir/IrO_x electrode is – like the Ag/AgCl electrode – chemically stable in concrete and easy to manufacture. Like ISEs in general, the Ir/IrO_x sensor technology might suffer from lack of long-term stability of the reference electrode used to measure the potential, and the presence of diffusion potentials arising from gradients in the concentration of solvated ions like H⁺ (pH) and Cl⁻ between the ISE and the reference electrode [11]. The Ir/IrO_x electrode responds to pH in concrete with a maximum error of 0.5 pH units over a range of at least pH 9–13.5 [15].

The Ir/IrO_x electrode can be manufactured in many ways including preparation techniques like electrochemical deposition, sputtering deposition, sol–gel processes, electrochemical oxidation, and thermal oxidation [15]. The thermal oxidation technique is probably the most used today [16].

A detailed thermal oxidation procedure for manufacturing an embeddable Ir/IrO_x electrode for concrete is presented in Appendix 2.

4.2 Fiber optical pH sensors

Compared to ISE sensors, one might state that there is an advantage to use a fiber optical sensor to detect changes in pH because a fiber optical sensor does not require a separate reference electrode. As described above, lack of long-term stability of an ISE sensor is often caused by stability issues related to the reference electrode. Besides, fiber optical sensors are normally robust, and with less electrical components and wiring [17].

Various optical pH sensors have been developed over the last decades. However, their suitability for in-situ pH monitoring in concrete has been questioned, for instance possible leaching of the pH indicator [17]. To avoid this issue, the pH indicator has been chemically bonded to a polymer substrate [18]. Further, it seems that the measurable range of pH response is quite narrow (pH=10-13) [19]. A recent attempt has been made to counteract some of these shortcomings and to design a special version for use in concrete [20].

However, neither experiences from field applications, nor long term stability of such sensors, have been documented. Currently, it seems that most – if not all – R&D activities related to fiber optical pH sensors for concrete can be characterised as rather basic lab studies. Obviously, for monitoring pH in concrete this is still a technology with a low TRL (Technology Readiness Level).

5 Commercial chloride and pH sensors for concrete

Commercial chloride and pH sensors have been used for many decades within various technologies, but for the application in concrete it is primarily results from R&D studies that are known/published. It seems that only one commercial supplier (Duramon) can offer a system for long-term in-situ monitoring in concrete, incorporating sensors for both chloride and pH. Duramon is a spin-off company from ETH Zürich (<https://duramon.ch/>). Duramon offers only the sensor system (including all parts), and not the separate electrodes.

6 Conclusions

From this short state-of-the-art overview of embeddable chloride and pH sensors for concrete, the following conclusion can be made:

- Two different sensor technologies are under development: Ion-selective electrodes and fiber optical sensors.
- Ion-selective electrodes can be used for chloride and pH measurement.
- Fiber optical sensor can be used for pH measurement only.
- Ion-selective electrodes have the best documented characteristics for use in concrete.
- There is only one commercial supplier that can offer chloride and pH sensors specially designed for concrete.
- There are guidelines available for manufacturing ion-selective electrodes for chloride and pH measurements in concrete.

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8 Appendix

A Embeddable chloride electrode for concrete

A silver/silver chloride electrode (Ag/AgCl) – as described in Chapter 3 – is made by forming a AgCl film on a thin silver wire (99.99 % purity). The diameter of the wire should be in the range of 0.5 – 1.0 mm, and the wire length approx. 2 cm.

Before starting the coating process, the silver wire must be cleaned: Initially degreased with acetone, the cleaned with dilute nitric acid (approx. 5 %) by immersing the wire in the acid for a few minutes, and finally rinsed with de-ionized water before starting the film-forming procedure.

A very straightforward film-forming procedure is simply to immerse the cleaned wire into a FeCl₃ solution overnight. In this way a very thin and invisible film of AgCl will be attached onto the silver wire. However, the probably most effective way is to make the film by an electrochemical set-up where the wire is anodised galvanostatically in a 0.1 M hydrochloric acid solution for up to a couple of hours under a current density of 0.2 – 0.5 mA/cm². After the film-forming process, the electrode is gently rinsed in distilled water and stored in closed and out of direct sunlight environment until next step.

To have connection to a measurement device, the coated silver wire is welded to a copper wire. The welding junction is then sealed, typically with an epoxy resin. The electrode can now be mounted in a rigid tube (typically stainless steel) where the sensing part is sticking out (approx. 5 mm) from the tube as illustrated in Figure 3.

The electrochemical potential response of the Ag/AgCl electrode on exposure to chloride (dissolved, not chemically bound) is measured by a reference electrode. The reference electrode could easily be integrated as a part of the sensor, and not necessarily as separated parts as schematically shown in Figure 1. Before installing the sensor in concrete, the Ag/AgCl electrode needs to be calibrated in order to relate the electrochemical potential (measured against the reference electrode) to actual chloride values in a solution.

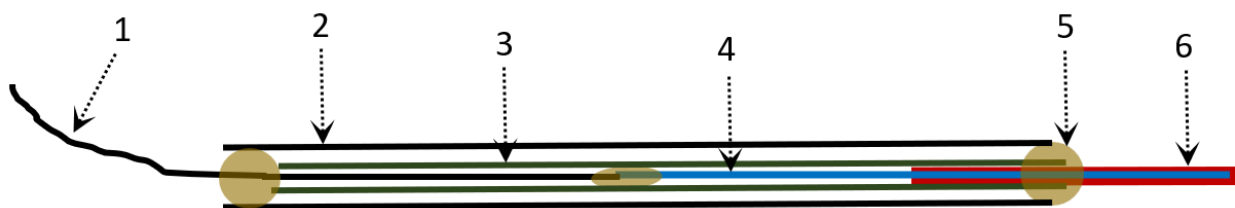


Figure 3 The basic construction of a chloride selective Ag/AgCl electrode to be used as part of an embeddable sensor for in-situ monitoring of chloride content in concrete.

- 1 – Copper wire
- 2 – Stainless steel tube
- 3 – Teflon tube
- 4 – Silver wire
- 5 – Junction sealed/glued with epoxy resin (for all three junctions shown in the figure)
- 6 – Silver wire part with the AgCl film (sensing part). The film thickness is schematically magnified in the figure. The actual film is almost invisible, has no colour, and is sticking out approx. 5 mm.

The apparently empty spaces between the wire and the Teflon tube, and between the Teflon tube and the stainless steel tube, are only schematically presented to illustrate the individual components.

B Embeddable pH electrode for concrete

A common technique for making a pH sensor is to thermally oxidise a thin iridium wire to form an iridium oxide (IrO_x) layer/film on the wire. The wire length and diameter are typically 0.5 mm and approx. 10-15 mm respectively, and the iridium purity should be $\geq 99.8\%$. Before starting the oxidation process, the wire needs to be cleaned: A few minutes in a 5-6 M HCl solution, and then washing in de-ionized water to remove impurities from the wire surface.

The cleaned and dry iridium wire is placed in a ceramic crucible (pot) lined with a thin gold foil. The wire is then covered all over with pure lithium carbonate powder ($> 99\% \text{Li}_2\text{CO}_3$) and heated gradually (approx. $10^\circ\text{C}/\text{min}$) in a furnace. After reaching approx. 850°C , the crucible is kept at that temperature for about 5 hours. After cooling to ambient temperature, the remaining solid carbonate can be dissolved with 1 molar HCl. Finally, the oxidised wire is washed with distilled water and dried at approx. 120°C for about 12 hours.

For electrical connection to a measurement device, one end of the oxidised wire needs to be scraped off until pure iridium is exposed (approx. 2 mm). This end is then connected to a metal wire (e.g. copper) by welding, and the connection point sealed with an epoxy coating. Sometimes a gold wire is used instead of copper wire [15], possibly due to less risk of contamination and/or unwanted side reactions. However, this is not clearly documented.

As for the Ag/AgCl electrode described in Appendix 1, the Ir/IrO_x electrode can be mounted in a rigid tube (typically stainless steel) where the sensing part is sticking out (approx. 5 mm). This is illustrated in Figure 4. This is a similar arrangement as for the Ag/AgCl electrode shown in Figure 3.

The electrochemical potential response of the Ir/IrO_x electrode on pH variations in a solution is measured by a reference electrode. The reference electrode could easily be integrated as a part of the sensor, and not necessarily as separated parts as schematically shown in Figure 1. Before installing the sensor in concrete, the Ir/IrO_x electrode needs to be calibrated in order to relate the electrochemical potential (measured against the reference electrode) to actual pH values in a solution.

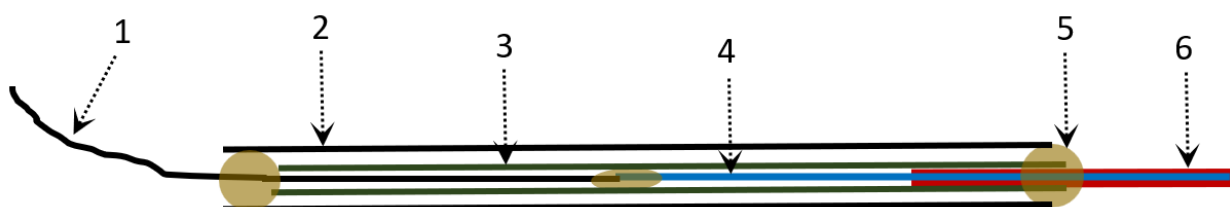


Figure 4 The basic construction of an Ir/IrO_x electrode to be used as part of an embeddable sensor for in-situ monitoring of pH in concrete.

- 1 – Copper wire
- 2 – Stainless steel tube
- 3 – Teflon tube
- 4 – Iridium wire
- 5 – Junction sealed/glued with epoxy resin (for all three junctions shown in the figure)
- 6 – Iridium wire part with the IrO_x layer (sensing part). The IrO_x layer thickness is schematically magnified in the figure. The actual IrO_x layer is very thin; has a black colour; and is sticking out approx. 5 mm.

The apparently empty spaces between the wire and the Teflon tube, and between the Teflon tube and the stainless steel tube, are only schematically presented to illustrate the individual components.

C Literature overview

The following literature overview lists published papers/articles/documents on embeddable sensors for chloride and pH monitoring in concrete. The list is arranged in chronological order from 2024→1996 (year marked **yellow**). The literature search has been carried out using Google Scholar, ResearchGate, and ScienceDirect with search terms: Concrete, monitoring, embeddable sensor, chloride, pH.

The quotes from abstracts (not full abstracts) give brief outlines of the topics covered in the documents. To distinguish between the documents describing the sensor types, those covering chloride sensors are marked with **◆**, whilst those covering pH sensors are marked with **◇**. This will hopefully ease the search for relevant documents. A few documents cover both sensor types.

Progress in in-situ electrochemical monitoring techniques for chloride ions in concrete structures **◆**

D. Wang et al, International Journal of Electrochemical Science 19 (2024) 100744, <https://doi.org/10.1016/j.ijeos.2024.100744>

Quote from abstract: This review provides a comprehensive overview of the progress in electrochemical monitoring techniques for chloride ions in concrete, focusing on the development and application of Ag/AgCl ion-selective electrodes, all-solid-state chloride sensors, electrochemical impedance spectroscopy (EIS) sensors, and multiparametric analysis approaches. ... Ag/AgCl electrodes have shown excellent stability and reproducibility, with various preparation methods and microstructural optimizations enhancing their long-term performance.

Embeddable Chloride Sensor for Monitoring Chloride Penetration into Cement Mortar **◆**

M. Zhang et al, Sensors 2024, 24, 2149. <https://doi.org/10.3390/s24072149>

Quote from abstract, A composite solid chloride sensor consisting of two single sensors, i.e., Ag/AgCl working electrode and Mn/MnO₂ reference electrode, was developed. The Ag/AgCl electrode was prepared by the anodic polarization method, while the Mn/MnO₂ reference electrode was prepared using the powder compaction technique. ... In every measurement, the time needed for the potential of a composite sensor to become stable was less than 30 s. The sensor enables non-destructive in situ monitoring of the chloride ion content in cement mortar, thus realizing early warning of deterioration of reinforcement and guaranteeing long service life of the structure.

pH sensor using aniline blue and functionalized nylon for monitoring the final stage of concrete carbonation **◇**

H. Jeong et al, Smart Mater. Struct. 32 (2023) 095013 (12pp), <https://doi.org/10.1088/1361-665X/ace8d9>

Quote from abstract: The sensor was manufactured via functionalization of nylon using formaldehyde and chloroacetyl chloride and then covalent bonding with aniline blue. The sensor showed a significant color change in the pH ranging from 5 to 10 and exhibited sufficient response within 30 min and reversibility. In addition, the sensor was stable even in high pH environments such as concrete, and its applicability was verified inside the concrete.

Optical sensors for the durability assessment of cement-based infrastructure ♦

C. Grengg *et al*, *ce/papers* 6 (2023), No. 5, pp 1033-1036, <https://doi.org/10.1002/cepa.2082>

Quote from abstract: ... a novel sensor technology is presented, based on luminescent pH sensitive dyes, to quantitatively determine the pH distribution in cement-based construction materials. Different sensor platforms were explored resulting in high resolution imaging techniques, as well as in miniaturized sensor probes for field application and in situ monitoring.

Progress in Sensors for Monitoring Reinforcement Corrosion in Reinforced Concrete Structures—A Review ♦ ♦

D. Shevtsov *et al*, *Sensors* 2022, 22, 3421, 24 pages, <https://www.mdpi.com/journal/sensors>

Quote from abstract: The purpose of our study was to summarise the data on the most common sensors and systems for the non-destructive monitoring of reinforced concrete structures developed over the past 20 years. We considered systems based on electrochemical (potentiometry, methods related to polarisation) and physical (electromagnetic and ultrasonic waves, piezoelectric effect, thermography) examination methods.

All-solid-state chloride sensor for in-situ monitoring of chloride penetration in concrete ♦

Z. Du *et al*, *Construction and Building Materials* 357 (2022) 129345, <https://doi.org/10.1016/j.conbuildmat.2022.129345>

Quote from abstract: In this study, an all-solid-state chloride sensor constituted with Ag/AgCl working electrode and Mn/MnO₂ reference electrode was developed. The Ag/AgCl electrode fabricated with galvanostatic anodization method and Mn/MnO₂ fabricated with powder compaction method exhibited homogeneous micro-structures. The results indicate that the chloride sensor can provide accurate chloride content variations of the mortar specimens, and it has great potential for non-destructively monitoring the chloride penetration in concrete structures.

All-solid-state, long term stable, and embedded pH sensor for corrosion monitoring of concrete ♦

Z. Du *et al*, *Journal of Building Engineering* 57 (2022) 104978, 13 pages, <https://doi.org/10.1016/j.jobbe.2022.104978>

Quote from abstract: In this study, an all-solid-state pH sensor constituted with iridium oxide (IrO_x) working electrode and manganese/manganese dioxide (Mn/MnO₂) reference electrode was developed. The IrO_x electrode fabricated with carbonate melt oxidation method and Mn/MnO₂ fabricated with powder compaction method exhibited a dense and homogeneous micro structure. The as prepared pH sensors showed remarkable long-term stability, ideal potential-pH response, low hysteresis, fast pH response time and strong resistance to ionic interference in solutions.

New Sensors for Monitoring pH and Corrosion of Embedded Steel in Mortars during Sulfuric Acid Attack ♦

R. Sampaio *et al*, *Sensors* 2022, 22, 5356, 13 pages, <https://doi.org/10.3390/s22145356>

Quote from abstract: In this work, new pH-sensitive IrO_x electrodes were developed for monitoring the pH inside mortar or concrete. To test their ability, the pH sensors were embedded in mortar samples at different depths and the samples were exposed to sulfuric acid solution. In another set of experiments, iron wires were placed at the same depths inside similar mortar samples and their corrosion was

monitored as the acid attacked the mortar. Severe acid attack led to cement dissolution and formation of gypsum. The new pH sensors succeeded in measuring the pH changes inside the mortars.

Distributed fiber optic pH sensors using sol-gel silica based sensitive materials ◆

F. Lu et al, *Sensors & Actuators: B. Chemical* 340 (2021) 129853, <https://doi.org/10.1016/j.snb.2021.129853>

Quote from abstract: Fiber optic pH sensors using either silica (SiO₂) or gold nanoparticle incorporated silica (Au-SiO₂) as the sensitive layers for pH monitoring are presented.

A facile sol-gel dip-coating process was utilized to immobilize the SiO₂ based sensitive layers on the coreless fiber. In the high pH range of ~8-12 simulating the wellbore cement conditions, the transmission spectra at room temperature demonstrated notable sensitivity using the SiO₂ based coating.

Fluorescent molecular probe based optical fiber sensor dedicated to pH measurement of concrete ◆

A. Tariq et al, *Sensors & Actuators: B. Chemical* 327 (2021) 128906, <https://doi.org/10.1016/j.snb.2020.128906>

Quote from abstract: An optical fiber sensor based on a fluorescent molecular probe has been developed to measure the pH of cementitious materials, from the early stages of hydration to advanced ageing of the material... The developed optode has the required characteristics for the study of concrete, with a response time of 100 s and a precision of ± 0.1 pH units.

A state-of-the-art review on Ag/AgCl ion-selective electrode used for non-destructive chloride detection in concrete ◆

Z. Zhang et al, *Composites Part B* 200 (2020) 108289, <https://doi.org/10.1016/j.compositesb.2020.108289>

Quote from abstract: The accurate measurement of chloride content in concrete is of great importance, because chloride-initiated corrosion of the reinforcement significantly influences the durability of reinforced concrete. This paper summarizes the research status of Ag/AgCl ion-selective electrode (ISE) used for non-destructive chloride detection in concrete. In general, four main topics are reviewed, including preparation of Ag/AgCl-ISE, main factors influencing the potential of Ag/AgCl-ISE, detection limit/potential response of Ag/AgCl-ISE, and novel composites/electrochemical methods based on Ag/AgCl-ISE for chloride detection in concrete. Further, the existing problems and research strategies are also discussed in detail in this present review.

Fibre optic ratiometric fluorescence pH sensor for monitoring corrosion in concrete ◆

J. Bartelmess et al, *Analyst*, 2020, 145, pp 2111–2117, The Royal Society of Chemistry, <https://pubs.rsc.org/en/content/articlelanding/2020/an/c9an02348h>

Quote from abstract: In this communication a novel concept for pH sensing is introduced which is specifically adapted to monitor carbonation induced corrosion in concrete structures. The method is based on a ratiometric measurement principle, exploiting the pH sensitive colour switching of thymol blue in the basic pH regime and the emissive properties of two different (Zn)CdSe/ZnS core shell quantum dots. The transition point of thymol blue in a Hydrogel D4 matrix was determined to be at around pH 11.6, which fits ideally to the intended application. Next to the fundamental spectroscopic characterization of the ratiometric response, a new design for a sensor head, suitable for the incorporation into concrete matrices is presented.

Non-destructive measurement of chloride ions concentration in concrete – A comparative analysis of limitations and prospects ◆

Y. Abbas et al, *Construction and Building Materials* 174 (2018) 376–387, <https://doi.org/10.1016/j.conbuildmat.2018.04.135>

Quote from abstract: In this work, the different techniques for non-destructive in situ measurement of chloride ion concentration are presented. Non-destructive (ND) in situ measurement is crucial for reliable and continuous determination of chloride ion concentration in concrete. Over the last 20 years, several studies have been performed on ND measurements. These were mainly focused on the application of electrochemical and electromagnetic techniques. Each technique has its advantages and disadvantages.

Development of a Novel Methodology to Assess the Corrosion Threshold in Concrete Based on Simultaneous Monitoring of pH and Free Chloride Concentration ◆ ◆

Y. S. Femenias et al, *Sensors* 2018, 18, 3101; doi:10.3390/s18093101, 14 pages, www.mdpi.com/journal/sensors

Quote from abstract: The approach is based on a recently developed electrode system that consists of several different potentiometric sensors as well as a data interpretation procedure. Instrumented mortar specimens containing different amounts of admixed chlorides were exposed to accelerated carbonation, and changes in free chloride concentration and pH were monitored simultaneously over time. The results revealed the stepwise decrease in pH as well as corresponding increases in free chlorides, resulting from the release of bound chlorides. For a pH drop of about 1 unit (from pH 13.5 down to pH 12.5), the free chloride concentration increased up to 1.5-fold.

Monitoring pH in corrosion engineering by means of thermally produced iridium oxide electrodes ◆

Y. S. Femenias et al, *Materials and Corrosion*, 2018;69, pp 76–88, DOI: 10.1002/maco.201709715, www.matcorr.com

Quote from abstract: This work develops the production protocol and posterior conditioning of thermally oxidized iridium (IrO_x) electrodes to be used as potentiometric pH sensors embedded in highly alkaline environments such as concrete or cathodically protected steel in soil. The main investigated aspects for the desired applications are the potential-pH response, its reproducibility, accuracy, and oxygen dependency. Preliminary results show that the studied electrodes are promising sensors for monitoring pH changes in concrete.

pH-monitoring in mortar with thermally-oxidized iridium electrodes ◆

Y. S. Femenias et al, *RILEM Technical Letters* (2017) 2, pp 59-66, DOI: <http://dx.doi.org/10.21809/rilemtechlett.2017.37>

Quote from abstract: Here, we present results from embeddable pH sensors that permit the continuous, in-situ monitoring of the pH in the concrete pore solution. These are potentiometric sensors, based on thermally oxidized iridium/iridium oxide (IrO_x) electrodes. We propose an iterative calculation algorithm taking into account diffusion potentials arising from pH changes, thus permitting the reliable, non-destructive determination of the pore solution pH over time. This calculation algorithm forms an essential part of the method using IrO_x electrodes.

Embedded sensor system to detect chloride permeation in concrete: an overview ♦

H. S. Lee, Corrosion Engineering, Science and Technology, 2017, 52:5, 373-382,
<https://doi.org/10.1080/1478422X.2017.1300218>

Quote from abstract: This paper provides an overview of the latest development of chloride permeation measurement in concrete by evaluating the current techniques being used in industry and highlighting a new approach to monitor chloride ion progress in concrete structures wirelessly.

Determination of Chloride Content in Cementitious Materials: From Fundamental Aspects to Application of Ag/AgCl Chloride Sensors ♦

F. Pargar et al, Sensors 2017, 17, 2482, 22 pages, doi:10.3390/s17112482, www.mdpi.com/journal/sensors

Quote from abstract: This paper reports on the advantages and drawbacks of available test methods for the determination of chloride content in cementitious materials in general, and the application of Ag/AgCl chloride sensors in particular. The main factors that affect the reliability of a chloride sensor are presented. The thermodynamic behaviour of silver in the presence or absence of chloride ions is described and kinetic restrictions are addressed. The parameters that can affect the activity of chloride ions in the medium and/or the rate of ion exchange and dissolution/precipitation processes at the sensor's surface are also considered.

Methods for measuring pH in concrete: A review ♦

A. Behnood et al, Construction and Building Materials 105 (2016), 176–188,
<http://dx.doi.org/10.1016/j.conbuildmat.2015.12.032>

Quote from abstract: This paper critically reviews the methods that have been developed for measuring the pH of fresh and hardened concrete. These methods are categorized in two broad divisions including destructive and non-destructive methods. The expression, ex-situ and in-situ methods are explained in detail as destructive methods, while the use of embedded potentiometric electrodes (mainly metal/metal oxide electrodes) and fibre optic sensors are evaluated as non-destructive methods. Also, advantages and drawbacks of each method are investigated and they are compared based on different technical and practical aspects. Despite the broad range of used methods for measuring the pH of concrete, there is no standardized test procedure.

Potentiometric thick-film sensors for measuring the pH of concrete ♦

J. M. Gandía-Romero et al, Cement and Concrete Composites 68 (2016) 66-76,
<http://dx.doi.org/10.1016/j.cemconcomp.2016.02.006>

Quote from abstract: This paper includes the results of the research carried out to characterise the pH sensor [Ag/Ag₂O], including the materials, response time, sensitivity, detection limit, reproducibility, reversibility and the study of chloride interference. The sensors have been studied in pore solutions and concrete specimens. The study shows that the thick-film sensor has a potentiometric response to variations in pH and is capable of providing continuous, real-time information on the progress of the carbonation front.

Investigation on the performance characteristics of chloride selective electrode in concrete ♦

M. Jin *et al*, *Ionics* (2015) 21:2981-2992,

https://www.researchgate.net/publication/281369287_Investigation_on_the_performance_characteristics_of_chloride_selective_electrode_in_concrete

Quote from abstract: Results revealed that the electrode potential showed a good Nernst response with chloride concentration and was affected little by sulfate ion. The detection limit for the chloride concentration was 10^{-3} mol L⁻¹ at pH 13.5 and 10^{-4} at lower pH values. In addition, the electrode also had a high exchange current density and a high equivalent capacitance. The Ag/AgCl coating showed good long-term stability over 3 months in solutions containing chloride ions. Besides, there was a good agreement between the free chloride content determined by the electrode and by pore solution expression.

EVALUATION OF Ag/AgCl SENSORS FOR IN-SITU MONITORING OF FREE CHLORIDE CONCENTRATION IN REINFORCED CONCRETE STRUCTURES ♦

F. Pargar *et al*, Conference paper: Young Researchers" Forum II: Construction Materials, University College London, 2014, pp 153-158, <https://www.researchgate.net/publication/342926218>

Quote from abstract: In this paper the response of Ag/AgCl electrodes in simulated pore solutions having different chloride concentrations and different pH values was studied. The electrodes were calibrated in cement extract solution, distilled-water and simulated pore solution. The results show that at chloride concentrations of > 4 mM the effect of pH on the response of the sensors is insignificant which makes using Ag/AgCl sensors in concrete feasible i.e. this concentration is much lower than the generally reported thresholds for corrosion initiation.

Fluorescence based fibre optic pH sensor for the pH 10–13 range suitable for corrosion monitoring in concrete structures ♦

T. H. Nguyen *et al*, *Sensors and Actuators B* 191 (2014) 498– 507,

<http://dx.doi.org/10.1016/j.snb.2013.09.072>

Quote from abstract: The design, development and evaluation of an optical fibre pH sensor for monitoring pH in the alkaline region are discussed in detail in this paper. The design of this specific pH sensor is based on the pH induced change in fluorescence intensity of a coumarin imidazole dye which is covalently attached to a polymer network and then fixed to the distal end of an optical fibre. The sensor provides a response over a pH range of 10.0–13.2 with an acceptable response rate of around 50 min, having shown a very good stability over a period of longer than 20 months thus far.

CORROSION SENSOR ♦ ♦

H. Yu and L. Caseres, United States Patent No. US 8,833,146 B2, Sep. 16, 2014.

Quote from abstract: A corrosion sensor may include a sensor body, a chloride probe held in the sensor body, a pH probe held in the sensor body, a reference electrode for the chloride probe and the pH probe held in the sensor body, a multiple array sensor held in the sensor body and a resistivity probe held in the sensor body. A method of measuring corrosion in a reinforced concrete structure may include inserting a corrosion sensor into a reinforced concrete structure and monitoring chloride ions with the chloride probe, pH with the pH probe, localized concrete resistivity with the resistivity probe and corrosion current density with the multiple array sensor.

Potentiometric determination of the chloride ion activity in cement based materials ♦

U. Angst, et al, J. Appl. Electrochem. (2010) 40, pp 561–573, DOI: 10.1007/s10800-009-0029-6, <https://link.springer.com/article/10.1007/s10800-009-0029-6>

Quote from abstract: When using ISEs embedded in concrete, diffusion potentials between the reference electrode and the ISE, as arising e.g. from gradients in pH, significantly affect the potential measurement and present a most important error source for the application of direct potentiometry to concrete. To minimise such errors, the reference electrode has to be positioned as close to the ISE as possible.

Fabrication and Performance of All-Solid-State Chloride Sensors in Synthetic Concrete Pore Solutions ♦

X. Gao et al, Sensors 2010, 10, pp 10226-10239; doi:10.3390/s101110226, www.mdpi.com/journal/sensors

Quote from abstract: One type of all-solid-state chloride sensor was fabricated using a MnO₂ electrode and a Ag/AgCl electrode. The potentiometric response of the sensor to chloride in synthetic concrete pore solutions was systematically studied, and the polarization performance was also evaluated. The results show a good linear relationship between the potential reading of the sensor and the logarithm of chloride activity (concentration ranges from 0.05 to 5.0 M), and the potential value remains stable with increasing immersion time. The existence of K⁺, Ca²⁺, Na⁺ and SO₄²⁻ ions have little influence on the potentiometric response of the sensor to chloride, but the pH has a significant influence on the potential value of the sensor at low chloride concentration. All of the results reveal that the developed sensor has a great potential for monitoring chloride ions in concrete environments.

A multifunctional sensor for monitoring corrosion of reinforced concrete structures ♦ ♦

Lin Changjian Dong Shigang Du Ronggui Li Lanqiang Li Sizhen Hu Ronggang, patent granted 2010-12-08, <https://patents.google.com/patent/CN101334353B/en>

Quote from abstract: The multifunctional sensor is provided with a metal shell, a Cl⁻ probe, a pH probe, a reinforcement metal electrode, a MnO₂ reference electrode and external guidelines. The Cl⁻ probe, the pH probe, the reinforcement metal electrode and the MnO₂ reference electrode are prepared separately, and then arranged in the shell.

Detecting critical chloride content in concrete using embedded ion selective electrodes – effect of liquid junction and membrane potentials ♦

U. Angst et al, Materials and Corrosion 60, No 8 (2009), 638–643, DOI:10.1002/maco.200905280.

Quote from abstract: ... In addition, liquid junction potentials at the interface of the concrete sample and the reference electrode contribute to the measured potential. Experimental observations in the present work illustrate the effect of liquid junction potentials on the application of ion selective electrodes in concrete. Moreover, the influence of internal membrane potentials has been estimated by a theoretical model.

Development of an embeddable sensor to monitor the corrosion process of new and existing reinforced concrete structures ♦

G. S. Duffó *et al*, Construction and Building Materials 23 (2009) pp 2746–2751, doi:10.1016/j.conbuildmat.2009.04.001.

Quote from abstract: An integrated and cost-effective sensor system to monitor the state of reinforced concrete structures from the corrosion point of view was developed. The sensor provides measurements of the open circuit potential of rebars, the corrosion current density of rebars, the electrical resistivity of concrete, the availability of oxygen, the chloride ions concentration in concrete, and the temperature inside the structure. The integrated system consists of different electrodes embedded in concrete and a software system that acquires and analyses the data. The results obtained so far show the capabilities of this type of sensor to determine the corrosion state of existing as well as new concrete structures.

A novel fabrication method of fiber-optical planar transmission sensors for monitoring pH in concrete structures ♦

M. Blumentritt *et al*, Sensors and Actuators B 131 (2008) 504–508, doi:10.1016/j.snb.2007.12.034.

Quote from abstract: Monitoring concrete structures requires robust, long-term stable, and low-cost sensors. We introduce a new technique for producing a fiber-optical planar transmission sensor setup using a wafer saw for cutting the sensitive zone, which can monitor in situ pH in concrete. The sensitive zone of the fiber-optical sensor setup is filled with a sensing material, which colour varies reversibly upon change of pH. Analyzing the characteristic changes in absorption of this material is done by a micro spectrometer. Our fabrication method is suited for industrial mass production because no cost-intensive adjustment of fibers is necessary. In this paper we will present beside our fabrication method the calibration of pH sensors produced with this method.

Multiprobe chloride sensor for in situ monitoring of reinforced concrete structures ♦

M. F. Montemor *et al*, Cement & Concrete Composites 28 (2006) 233–236, doi:10.1016/j.cemconcomp.2006.01.005.

Quote from abstract: The present work aims at developing and testing a sensor based on Ag/AgCl electrodes for in situ monitoring of chloride ions in reinforced concrete structures. Although these electrodes are widely used in analytical experiments due to their sensitivity to the chloride ion, little is known on their behaviour during exposure to alkaline environments, such as those existing in concrete. The multiprobe sensor presented in this work was tested in mortar and concrete specimens, revealing good stability. The results reveal that the Ag/AgCl sensor presents good sensitivity in a wide range of chloride concentrations.

Sensor Systems for Corrosion Monitoring in Concrete Structures ♦

K. Kumar *et al*, Sensors & Transducers Magazine (S&T e-Digest), Vol.67, Issue 5, May 2006, pp.553-560.

Quote from abstract: In this investigation we report the studies on the sensors systems based on the measurements of half-cell potential of rebars inside the concrete, resistivity of concrete, corrosion rate of rebars by eddy current measurements and sensing of chloride ions. An integrated system consists of above sensors are fabricated and embedded into concrete.

In Situ Measurement of Cl⁻ Concentrations and pH at the Reinforcing Steel/Concrete Interface by Combination Sensors ♦ ♦

R. G. Du *et al*, *Anal. Chem.*, 2006, 78, pp 3179-3185.

Quote from abstract: This paper presents an in situ, non-destructive method of monitoring Cl⁻ concentrations and pH values at the steel/concrete interface. The Ag/AgCl electrodes prepared by the electrochemical anodization and the Ir/IrO₂ electrodes prepared by thermal oxidation in carbonate served as Cl⁻ concentration and pH sensors, respectively. The results indicate that the combined sensor is robust and sensitive enough to in situ measure Cl⁻ concentrations and pH quantitatively at the steel/concrete interface,

Fiber optic pH sensor for early detection of danger of corrosion in steel-reinforced concrete structures ♦

N. Dantan *et al*, *Proc. SPIE 5758, Smart Structures and Materials* 2005: Smart Sensor Technology and Measurement Systems, (16 May 2005); doi:10.1117/12.600703.

Quote from abstract: At present, several reliable systems for determination of chemical parameters in aggressive environments are available on the market, but can not be used for long-term monitoring of pH in concrete structures. This paper describes the development of a fiber optic chemical sensor for this purpose. Particular attention is paid to the requirements on such a sensing system. ... Based on these results, a functional pH sensor has been configured. It shows good response behavior and works under strongly alkaline conditions for one year. Therefore, it represents a promising sensor type for in-situ long-term monitoring in concrete structures. Further work is in progress to test such sensors on-site under real application conditions, e.g. in ground anchors.

Fibre Optic Chemical Sensor Systems for Monitoring pH Changes in Concrete ♦

P. A. M. Basheer *et al*, *Proc. SPIE 5586, Advanced Environmental, Chemical, and Biological Sensing Technologies II*, (7 December 2004); doi: 10.1117/12.601198.

Quote from abstract: This paper describes the development, testing and evaluation of two types of fibre optic sensors for the pH monitoring. One of these used a sol-gel based probe tip, into which an indicator dye has been introduced and the second used a disc containing an indicator operating over a narrower range of pH with shorter lifetime. The two types of sensors have been found to be sensitive to the changes in pH due to carbonation, but the response time depended on the thickness of the coating material in the case of the sol-gel sensor. The durability of the sensors is still under investigation. The disc type sensor has a life span of approximately 1 month and, hence, it is not suitable for embedding in concrete for long-term monitoring of pH changes.

Non destructive determination of the free chloride content in cement based materials ♦

B. Elsener *et al*, *Materials and Corrosion* 54, 440–446 (2003), DOI: [10.1002/maco.200390095](https://doi.org/10.1002/maco.200390095)

Quote from abstract: The sensor element determines the activity of the free chloride ions in solutions and in porous cement based materials such as cement paste, mortar or concrete. The calibration in synthetic pore solution showed a response according to Nernst law over three decades of chloride concentration. The sensor element has shown excellent reproducibility and long term stability. The sensor element has been used to monitor the chloride uptake into mortar specimens. The results show a good agreement between the free chloride content determined by the sensor and by pore water expression.

A long-term stable iridium oxide pH electrode ◆

M. Wang et al, *Sensors and Actuators B* 81 (2002), 313-315,
<https://www.sciencedirect.com/science/article/pii/S0925400501009728>

Quote from abstract: In this work, a long-term stable pH electrode based on iridium oxide film is reported. A new method, i.e., a carbonate melt oxidation method, has been developed to fabricate iridium oxide pH electrode. In this method, a uniform iridium oxide film is coated on the surface of an iridium metal wire through oxidation of the wire in a carbonate melt. The electrode made this way shows a “drift-free” behavior in pH buffer solutions. Calibration curves for the same electrode implied that the electrode is very stable over a long period of 2.5 years. Therefore, the present electrode is very suitable for continuous pH measurement without the need of frequent calibration.

A pH Electrode Based on Melt-Oxidized Iridium Oxide ◆

S. Yao et al, *Journal of The Electrochemical Society*, 148 (4) H29-H36 (2001),
<https://iopscience.iop.org/article/10.1149/1.1353582/pdf>

Quote from abstract: Fabrication and characterization of a novel potentiometric pH electrode based on melt-oxidized iridium oxide film is presented. The oxide film produced in a lithium carbonate melt has the composition of $\text{Li}_x\text{IrO}_y \cdot n\text{H}_2\text{O}$, and shows high chemical stability. The electrode based on this oxide film exhibits very promising pH sensing performance, with an ideal Nernstian response in the tested pH range of 1 to 13. The potential response is fast, with a 90 % response time obtained in less than 1 s for all pH changes. The open-circuit potential of the electrode is almost drift-free, with an average variation over time in a pH 6.6 solution as small as 0.1 mV/day.

Embedded micro-sensor for monitoring pH in concrete structures ◆

R. Srinivasan et al, proceedings from conference on Smart Systems for Bridges, Structures, and Highways, (20 April 2000), pp 40-44, <https://www.spiedigitallibrary.org/conference-proceedings-of-spie>

Quote from abstract: In this paper, we describe an inexpensive solid state pH sensor that can be embedded in concrete, to detect pH changes at the early stages. It employs a chemical reagent, trinitrobenzenesulfonic acid (TNBS) that exhibits changes in optical properties in the 12-14 pH range, and is held in a film of a sol-gel/TNBS composite on an optically transparent surface. A simple LED/filter/photodiode transducer monitors pH induced changes in TNBS. Such a device needs no periodic calibration or maintenance. The optical window, the light source and sensor can be easily housed and encapsulated in a chemically inert structure, and embedded in concrete.

Embeddable Ag/AgCl sensors for in-situ monitoring chloride contents in concrete ◆

M. A. Climent-Llorca et al, *Cement and Concrete Research*, 1996, Vol. 26, No. 8, pp. 1157-1161,
[https://doi.org/10.1016/0008-8846\(96\)00104-4](https://doi.org/10.1016/0008-8846(96)00104-4)

Quote from abstract: The possibility of using Ag/AgCl wire electrodes as in-situ sensors of chloride concentration in concrete was studied by embedding them in a series of mortar specimens with different admixed NaCl contents. They show a sensitive potentiometric response to overall Cl^- concentrations. The stability of the potential readings depends on the Cl^- concentration and allows these electrodes to be used as Cl^- content sensors in short-term tests.

MONITORING CHLORIDE CONCENTRATIONS IN HARDENED CEMENT PASTES USING ION SELECTIVE ELECTRODES ♦

C. P. Atkins *et al*, Cement and Concrete Research, 1996, Vol. 26, No. 2, pp. 319-324.

Quote from abstract: In a previous paper (1) it has been shown that silver/silver chloride ion selective electrodes are stable in a simulated pore solution and that the potential of these electrodes is a function of chloride ion activity. This has now been compared with chloride concentrations in the pore water, found using a pressure extraction technique, with chlorides added as sodium chloride to the mix water in concentrations ranging from 0.046 Mdm^{-3} to 0.46 Mdm^{-3} to ordinary portland cement pastes with a water cement ratio of 0.45. The results show good correlation between concentration added and activity calculated from electromotive force data, with an activity coefficient of 0.46 ± 0.07 , which agrees with values calculated in the previous paper.

Measurement of Environmental Parameters in Concrete ♦ ♦

R. Myrdal *et al*, Int. Appl. No. PCT/NO96/00064 - WO 96/30741; Int. filing date 22.03.1996, <https://patents.google.com/patent/WO1996030741A1/tr>

Quote from abstract: A combination of electrodes to be moulded into concrete with purpose of measuring environmental parameters in concrete. The combination comprises a plurality of sensor electrodes (1), each of which being comprised of substantially one material. The materials are chosen in such a way that the electrode potentials of the sensor electrodes as measured in relation to a reversible reference electrode (2), vary in a mutually divergent manner in relation to the environmental changes in concrete. By utilising this electrode combination values of the environmental parameters are calculated, and thus the corrosive environment for the reinforcement in the concrete is determined.

EXCON

<https://sintef/excon>



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