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A low-cost optode-array measuring system based on 1 mm plastic optical fibers — new technique for in situ detection and quantification of pyrite weathering processes

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Abstract

Optical oxygen sensors and a sensor array were developed on the basis of 1 mm plastic optical fibers (POF). They can be adapted to a commercially available single-channel optical fluorescence lifetime measuring device. The sensors are inexpensive and show high mechanical stability. The developed sensor array for 1 mm POF shows very good reproducibility even in a low-cost prototype version. The measuring system allows long-term in situ measurement of oxygen concentrations. In a field test, the measuring system could be used successfully for the in situ measurement in the oxygen-consuming environment of a brown coal dump body. In laboratory experiments, the system was used for the observation and quantification of pyrite oxidation processes in column experiments. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Optode; Array; Plastic optical fiber; Diffusion; Oxygen; Pyrite weathering

1. Introduction

1.1. Optical oxygen measurement

Optodes are optical sensors based on optical fibers, with sensitive substances or systems attached to the fiber tip. A change in concentration results in a modification of its optical characteristics [1]. This is transmitted by the optical fiber and detected with a suitable device. For the optical oxygen detection different measuring systems are described. Some are commercially available. The measuring systems used here (Microx 1/FIBOX, PreSens, Regensburg, Germany) are based on the fluorescence lifetime measurement [2]. For the analysis, the oxygen-dependent change in fluorescence lifetime of a fluorescent dye is monitored. The instrument internally evaluates the phase shift between the modulated blue excitation light and the red fluorescence response from the detector foil [3]. The commercially available measuring system (Microx 1) is based on 100/140 μm multimode graded index silica fiber cables. The micro-optode tips are tapered to a diameter of approximately 30 μm and coated with an oxygen sensitive layer [4]. The optical oxygen measurement is temperature-dependent. The temperature can also be measured with optodes [4].

1.2. Sensor arrays

The automated operation of several optical fiber sensors at one measuring instrument is difficult due to signal attenuation at every fiber-to-fiber coupling. Depending upon switching technique the attenuation may vary widely. Fiber switches used in data processing technology are expensive and they are optimized for switching speed rather than for low attenuation. They show very short switching times within the range of milliseconds while the attenuation is usually >1 dB [5]. A cost-intensive solution with no extra attenuation is an array that provides one optical module for each sensor [6]. In this case, there is no optical, but only electronic switching between the individual optical modules, which does not introduce measurable signal attenuation. In the literature, different linear stage switches for glass fibers are described. In these systems, the fibers are either switched directly [5] or the coupling area is optically increased with the help of lenses in order to enhance switching accuracy [7].

1.3. Pyrite weathering

Pyrite (FeS_2) is thermodynamically unstable under oxic conditions. Weathering processes take place and enormously

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affect the ecosystem. Systems in which pyrite decomposition occurs are characterized by extreme geochemical conditions. The degradation of pyrite with oxygen causes the release of very large quantities of iron and sulfuric acid. Secondary reactions induced by pyrite weathering result in heavy metal release. The basic mechanisms associated with pyrite weathering are reviewed, e.g. by Evangelou [8], Appelo and Postma [9] and Nordstrom [10]. The initial cause of complex processes is the contact of pyrite with oxygen when pyrite-bearing material is being exposed to the atmosphere. The relevant processes are convective and diffusive oxygen transport as well as the pyrite reactivity (specific mineral surface, turnover rates). Therefore, it is of great interest to detect the zone of reaction (oxygen consumption) and observe its change over time. Data can be used to quantify oxygen transport into the pyrite-bearing material, and thus, evaluate resulting processes. In situ measurements of oxygen profiles, e.g. brown coal open mining dumps can be used as basic data to predict the development of the systems [11]. Column experiments help parameterizing the complex primary and secondary processes during the pyrite oxidation. One master variable associated with pyrite weathering is the development of oxygen concentrations over time and depth [12].

2. Experimental

2.1. Low-cost POF oxygen-optodes

The developed optodes based on 1 mm plastic optical fibers (POF) have a very simple design. In contrast to micro-optodes the fiber tip is not directly coated with sensor chemistry. Instead, a piece of sensor foil [13] is fixed at the POF tip. The sensor foils are commercially available as 4 in. × 6 in. sheets coated with sensor dye material in an immobile matrix (Planar optode, PreSens, Regensburg, Germany). The matrix material is either gas-permeable (oxygen-optodes) or gastight (temperature optodes). From these sensor foils small wafers are punched out and fixed on the polished flat tip of the POF. Sensors of different design have been developed for different applications. Fig. 1 shows two sensor types. In type I, the sensor end of the optical fiber is built into a stainless steel needle, forming a durable sensor head. The POF is stripped and polished at the tip. The sensor foil wafer is attached to the tip with a layer of PTFE tape that forms a protection against liquid chemicals and increases the light intensity by reflection at the white surface. The used stainless steel needle has a massive tip and an aperture at the side. Both the tip and the aperture part of the needle are filled with PTFE. The stripped fiber with the sensor foil wafer is fed into the needle, such that the sensor foil gets placed right above the aperture. Finally, the optical fiber jacket and the needle are connected by heat shrink tubing.

The type II sensor is built without metal component (Fig. 1). The sensor foil is fixed on the flat fiber tip that has been polished without stripping off the jacket. A piece of PTFE tape is used to hold the sensor foil wafer in place. For mechanical protection of the sensor tip, a PTFE foil wafer (0.5 mm thickness) is attached above the sensor foil by two more layers of PTFE tape. The entire PTFE sequence forms a gas-permeable protective layer against mechanical stress and liquid chemicals. Heat shrink tubing fixes the entire sensor complex to the optical fiber tip.

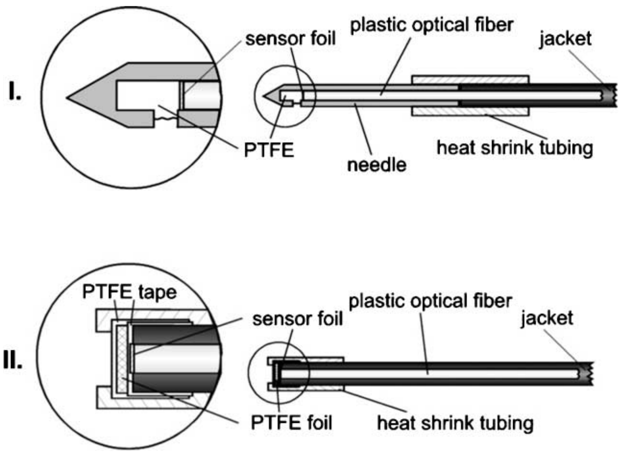

Fig. 1. Optodes based on 1 mm plastic optical fibers (POF). Type I: high mechanical stability, slow response time; type II: simple design, smaller mechanical stability, faster response time.

The sensors of the type I are suitable for the measurement in laboratory experiments. The needle connects well with luer-lock fittings, so that a tight installation in luer-lock sampling-ports is possible. Fig. 2 (left) shows oxygen-optodes of this design integrated into a column experiment setup (see Section 3.3). The type II sensor can be used as in situ sensor and may be permanently left in the system of interest. Fig. 2 (right) shows a batch of optodes of different lengths, which have been permanently installed in a borehole (see Section 3.4).

2.2. Low-cost array for plastic optical fiber optodes

An optical fiber switch was developed in order to attach several sensors to the one-channel measuring instrument. It allows an automated measurement during longer periods. Fig. 3 shows the schematic structure of the switch. In a one-channel instrument, the optode is connected to a coupler in the front plate. Inside the one-channel measuring instrument, the optical module is connected to the optode interface in the instrument front plate (optical fiber coupler) by a short optical fiber cable. The optical fiber switch replaces and automates this connection. The switch is based on a linear stage with an optical fiber fixed to the carriage. This optical fiber is directly connected to the optical module of the oxygen instrument. The carriage is moved using a stepper motor and positions the optical fiber in front of the optodes that are fixed in an aluminum block. The switch functions (positioning and switching times) are controlled by a micro-controller (BASIC Stamp II, PARALLAX, Rocklin,

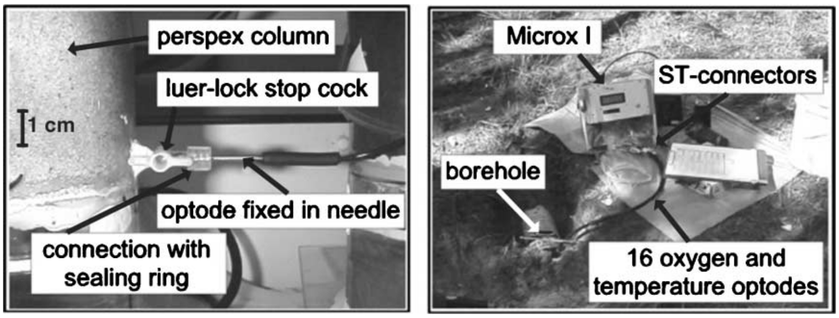

Fig. 2. Application possibilities of POF-optodes. Left: fixed installation in soil column experiment; right: field experiment — installation in borehole.

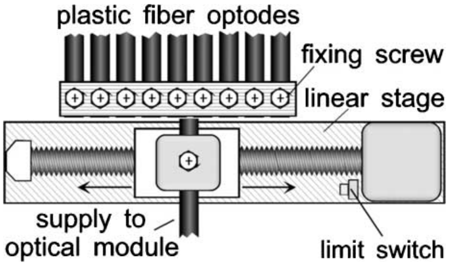

Fig. 3. General structure of optode-array based on 1 mm plastic optical fibers (POF).

CA, USA). The switch also activates and deactivates the oxygen meter. The micro-controller is programmed via a serial interface connected to a PC using a BASIC compiler for Win98 provided by PARALLAX. Nine optodes may be connected to the prototype version of the optical fiber switch. An expansion to more sensors depends only on the accuracy of the linear stage setup. The measured values are transmitted to a computer through a serial interface.

3. Results

3.1. Optodes

The sensors of different design show different characteristics. Type I sensors are characterized by good mechanical stability. In this sensor type, the tip of the steel needle is filled with PTFE in order to protect the sensor foil while allowing gas to penetrate. The PTFE holds a small gas volume, which is large enough to significantly increase the response time of this sensor type, since the gas has to equilibrate with the outer gas phase composition. Response times of this sensor type are in the range of minutes for gas phase measurements.

The gas volume captured in the PTFE protecting layer of type II sensors is smaller while the interface area between PTFE and environment is larger. The response time of this sensor type is below 1 min. The maximum mechanical stress is clearly smaller compared to type I sensors. The chemical characteristics of both sensor types are identical. It is responsible for the accuracy and long-term stability of the sensors. In several experiments or field situations, it is useful to install sensors permanently. In this case, the sensors may be calibrated only prior to installation. Calibrating an external reference-optode from the same batch and using the drift for correction may enhance subsequent uncalibrated measurements. With calibrated sensors of this design measurements show an absolute error in the range of <1% oxygen air saturation (C_{SA}).

Fig. 4 shows the oxygen concentrations from measurements with nine different 1 mm POF-optodes over a period of 280 days. The sensors were calibrated prior to the first measurement. Subsequent measurements were performed

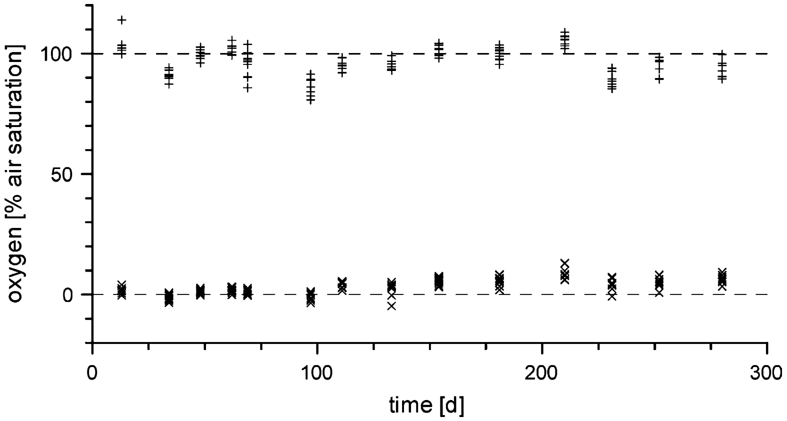

Fig. 4. Development of nine different POF-optodes over a time of 280 days. Results of measurement in ambient air (+) (20.7 vol.% O_2/100% O_2-saturation) and argon (×) (0 vol.% O_2/0% O_2-saturation). Single initial calibration of the sensors.

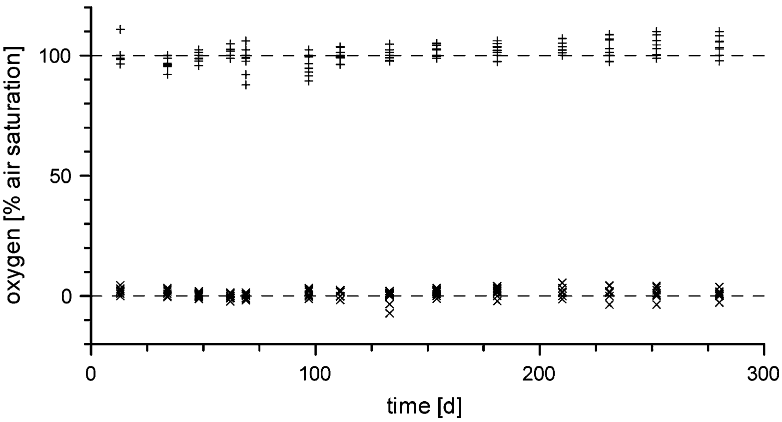

Fig. 5. Development of nine different POF-optodes over a time of 280 days. Results of measurement in ambient air (+) (20.7 vol.% O_2/100% O_2-saturation) and argon (×) (0 vol.% O_2/0% O_2-saturation). Initial calibration and simultaneous correction by a reference-optode.

without re-calibration in argon (0% oxygen air saturation) and ambient air (100% oxygen air saturation). We use argon instead of nitrogen as zero-oxygen standard because the residual oxygen content in welding-quality argon (99.96%) is lower than in nitrogen of the same quality. The measured values were analyzed and temperature-corrected based on the initial measurement of the sensor. The values are subject to quite large drift. The maximum deviations between measurement and correct value are within the range of 20% C_{SA}. The 90% of the measurements show an absolute error <10% C_{SA}. The shifts of the measured values show clear trends indicating either instrument drift or poor temperature correction. Using one sensor as reference sensor for drift correction, the deviation is reduced. In Fig. 5, this correction is included. In this case, 90% of the values show an absolute error <5% C_{SA}. The maximum deviation between measured and correct value is within 12% C_{SA}.

3.2. Optode-array

The fiber switch allows connecting several optodes to the instrument. In the prototype switch, the switching time between sequential channels amounts to <5 s. The maximum switching time amounts to <30 s. The positioning of the 1 mm fibers is sufficiently exact and well reproducible. The standard deviations of repetitive measurements of the different channels are smaller than the accuracy of the oxygen measurement (<1% C_{SA}). The quality of the measurement is hardly influenced by the switch. Due to the large diameter of the optical fiber the signal attenuation at the fiber–fiber interface is low, although there is a small gap (approximately 0.25 mm) between the two optical fibers. The signal attenuation at the interface is two-fold, since the intensities of both the blue excitation light from the instrument and the red fluorescence light from the sensor are decreased. Compared to the one-channel instrument with a single optode connected to a coupler at the instrument front plate, the total signal attenuation introduced by the switch is <2 dB.

3.3. Soil column experiments

Column experiments were performed in order to investigate pyrite oxidation in the unsaturated zone. Pyrite-bearing material was filled into perspex columns of 1 m length and a diameter of 10 cm. The columns were left open at the upper end to allow ambient air oxygen to penetrate. At different depths 1 mm plastic optodes were permanently installed in order to allow automated monitoring of the subsurface oxygen concentration changes using the optical fiber switch. The measured values were analyzed according to an initial calibration and corrected on the basis of a reference-optode from the same batch that was calibrated prior to each measurement. Additionally, the oxygen concentration could be measured using calibrated micro-optodes that were inserted through sampling-ports. Fig. 6 shows the oxygen concentration change over 100 days determined with the 1 mm optodes. Initially, most oxygen is consumed close to the surface. Subsequently, the oxygen

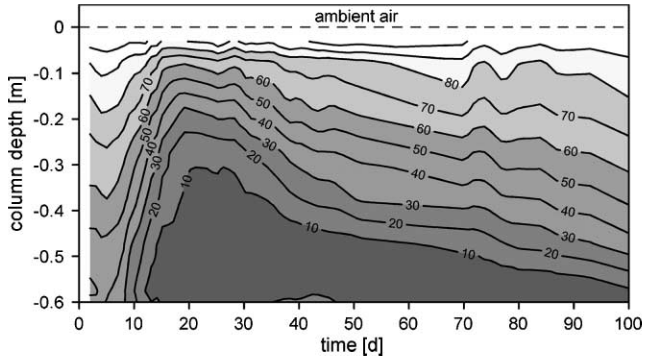

Fig. 6. Pyrite weathering in soil column experiment — development of oxygen concentrations (% air saturation) over time and depth. Measurements with initially calibrated POF-optodes corrected by reference-optode.

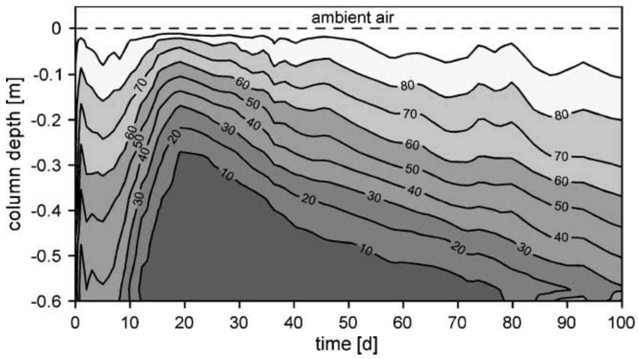

Fig. 7. Pyrite weathering in soil column experiment — development of oxygen concentrations (% air saturation) over time and depth. Measurements with calibrated micro-optodes.

penetration depth increases, indicating a proceeding depyritization of the sediment column. Fig. 7 shows the oxygen concentration change over depth and time measured with the calibrated micro-optodes. The differences between the two measurements are low. Both reflect the same processes. At low concentration levels the differences increase with time where the concentrations obtained with the calibrated micro-optodes are generally higher (up to 10% C_{SA}) than the concentrations obtained from the permanently installed 1 mm optodes.

The pyrite weathering processes on the column are reflected by the changes in oxygen distribution. In connection with soil-physical and chemical boundary conditions, the processes can be modeled and quantified [11].

3.4. Field experiment

In a dump body in the lower Lusatian brown coal district, the level of depyritization should be assessed and the residual acidification potential of the material should be predicted. The dump body has an age of about 43 years at the test site. Since this time pyrite decomposition processes have taken place. In a field experiment, a 7.8 m deep borehole was sunk using a percussion drill. In the borehole, eight oxygen-optodes were permanently installed at depths of 0.8, 1.8, 2.8, 3.8, 4.8, 5.8, 6.8 and 7.8 m. Additionally, optical temperature sensors were installed at the same depths (see Fig. 2). The type II sensors (see Fig. 1) were characterized and calibrated prior to installation. An external reference sensor from the same optode batch was used for drift correction. Measurements were carried out 0.5, 2 and 3.5 h, as well as 8 weeks after installation. The observation period is very short compared to the age of the system. The geochemical conditions can be regarded as steady state for this period. The results of the oxygen measurements are represented in Fig. 8. Already after 3.5 h quasi-stable conditions regarding the oxygen distribution have developed. The measurement after 8 weeks shows a similar oxygen distribution. The concentrations clearly decrease with depth. At a depth of 6–8 m below surface the measured oxygen concentration is below values of 15–20% air saturation.

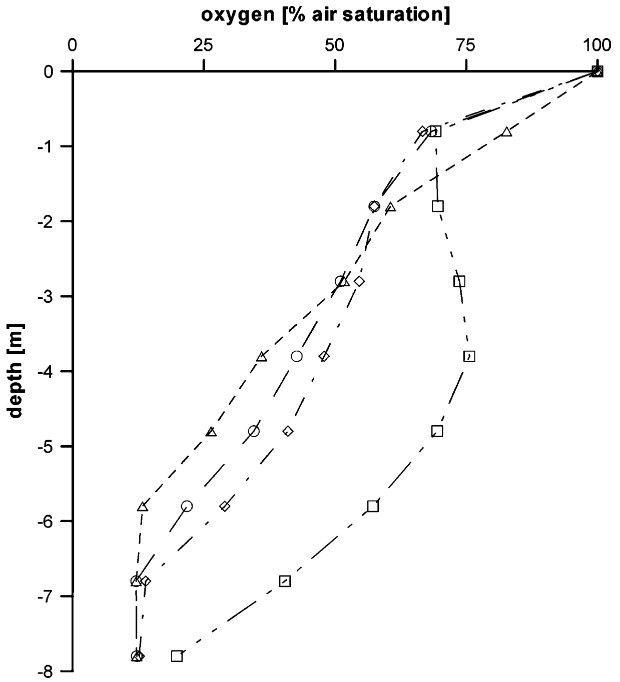

Fig. 8. Field experiment with POF-optodes: measured oxygen distribution in approximately 43 years old pyrite-bearing dump body. Measurements 0.5 h (squares), 2 h (rhombuses), 3.5 h (circles) and 8 weeks (triangles) after installation of the sensors (initial calibration and correction with reference-optodes).

4. Discussion

The 1 mm POF-optodes show response times within the range of minutes when measurements are performed in the gaseous phase. They are much slower than micro-optodes, which use same sensor chemistry (response time: in seconds). The optodes introduced here are intended primarily for long-term applications. They are meant to remain in the systems of interest such that the equilibration time is of minor importance if temporal concentration changes in the range of minutes are not expected. For measurements, in the aqueous phase, the response time additionally increases by another order of magnitude, since the equilibration time between liquid and gas phase enclosed in the sensor head increases.

The accuracy of calibrated individual measurements is within the range of <1% saturation concentration (C_{SA}) [6] for both micro-optodes and plastic fiber optodes. The accuracy of measurement strongly depends on a good temperature correction [4]. The temperature must be determined simultaneously. This may be done using either conventional temperature sensors (k-type, Pt100) or temperature optodes. The temperature optodes used here are almost identical to the oxygen sensors. The only difference is the matrix material of the sensor foil, which is gastight such that only the temperature response of the sensor may be measured with the same measuring instrument [4]. For long-term measurements, the use of reference sensors is also recommended here. The measuring accuracy decreases strongly if the sensors are used for long-term in situ measurements. Using a reference sensor for correction, the absolute error introduced by missing sensor re-calibration is within 10% C_{SA} over a 300 day period. In long-term use, the accuracy of the measurements (long-term stability) strongly depends on the stability of both the fluorescent dye and the immobilization matrix. Novel types of sensor foils promise higher long-term stability (personal communication, A. Stangelmeyer, University Regensburg, Germany), and thus, larger accuracy.

The accuracy level presented for the long-term use of in situ optodes cannot be attained with conventional oxygen sensors. Other methods for in situ measurement of oxygen over such long periods (gas sampling or multilevel wells in connection with gas chromatographic detection or oxygen probes) usually are cumbersome and expensive. The manufacturing cost of 1 mm POF-optodes with a length of 10 m is in the range of US$ 15 each. The sensors can be built with very good reproducibility and they may be easily adapted to the conditions in the system of interest. Depending on intended purpose, the emphasis can be put on mechanical stability or smaller response times. If optodes are to be used with a single-channel measuring instrument, sensors with an optical fiber length of at least 25 m can be manufactured. At greater lengths, the signal attenuation of the plastic fiber itself becomes too important. Newly developed measuring devices (FIBOX, PreSens, Regensburg, Germany) and new sensor foils promise larger flexibility in this point.

The sensor array allows connecting several sensors to a one-channel measuring instrument. With this device, data from a large number of sensors may be automatically collected over long periods (weeks to months). The switching time between the individual channels of the array is within the range of seconds compared to optical fiber switches known from data processing technology with switching times within the range of milliseconds. Since the presented measuring system is intended particularly for automated long-term measurements switching time is insignificant. The good and reproducible transfer of the measuring signal is more crucial. Fiber optical switches known from the data processing technology show a signal attenuation of 1–3 dB. The signal attenuation of the switch for 1 mm plastic optical fibers presented here is in the same range (<2 dB). The transmitted signal strength is sufficient for a reproducible measurement. The fiber coupling is simple, and thus, well reproducible due to the large diameter and a durable technique. The standard deviation of $<1\%$ C_{SA} is very small. The switch does not affect the quality of the measurement if it is directly connected to the instruments optical module. The cost of the sensor array setup presented is clearly smaller than other known sensor array solutions.

The results of the presented laboratory and field tests demonstrate the successful application of the measuring system in long-term measurement series. In the laboratory column experiments, the development of the oxygen distributions could automatically be recorded during a period of 100 days. The examination of the measured values with calibrated micro-optodes shows slightly deviating results. The oxygen concentrations measured with permanently installed optodes are always lower — especially, at low concentration levels. The long-term experiments showed that for a longer period of time oxygen concentrations may be determined within $\pm 10\%$ C_{SA} regardless of the time expired (no extra drift). A part of the difference may be explained by possible errors in the measurement with calibrated micro-optodes. When introducing these sensors into the sample ports of the experimental column, a small amount of ambient air may be introduced as well. This may distort the results with the highest deviations in the low concentration range. Yet, the processes revealed by the measurement with both sensor systems are similar.

The oxygen data collected in both field and lab experiments are the basis for the identification of processes and for the development of model conceptions on the pyrite decomposition in the water-unsaturated zone [11]. Using the optode-array presented here, the temporal and spatial resolution of such measurements can be increased depending on demand. Measurements with a spatial resolution of centimeters and a temporal resolution of minutes are easily feasible. With micro-optodes or 1 mm-sensors without PTFE protective layer the temporal resolution is in the range of 1 s [2]. With conventional measuring systems, a data collection at this temporal and spatial resolution would only be possible at very high technical and financial expenditure.

The field test shows that also in natural systems in situ measurements can be executed during longer periods. Already after a few hours equilibrium re-adjusted within the system examined. The sensors which remain in the system can be used for subsequent measurements. Since the optical measurement does not consume oxygen [14], the system is disturbed only on installation of the sensors. The measurement, which was executed 8 weeks after installation of the sensors, shows that the sensitivity does not noticeably decrease. The measurements indicate a continuous decrease of oxygen concentrations with depth indicating downward oxygen flux and oxygen consumption at a depth of 6–7 m [11]. In this area, the pyrite decomposition front is presently situated. Above this area, the material is pyrite-free. The determination of the pyrite content in sediment samples from this drilling showed the same results. Below a depth of 6 m, the material still contains pyrite in significant quantities (0.3–0.4 wt.%). The oxygen data can be used as data basis for the modeling as well as for prognostic purposes [11].

5. Conclusions

The presented measuring system is suitable for the automated or manual monitoring of oxygen distributions in gaseous and liquid phase. The sensors are mechanically stable and indicate a high long-term stability. After installation of the sensors oxygen concentrations can be measured in situ at high temporal and spatial resolution without affecting the processes by sampling. For long-term measurements (months), the errors of the measured values are below 10% saturation concentration. The optode-array automates the measurements without affecting the accuracy of the measured values. The costs and the technical expenditure of the measuring system are small in relation to other measuring systems. With this technique, percussion probes with integrated oxygen and temperature optode-arrays may be constructed. Such sensor arrays may be sunk directly such that reliable results may be obtained after a short time (<1 h). After measurement, these sensors may either permanently remain in the system or may be recovered for subsequent measurements at other locations. Such a measuring system enables large-area oxygen measurements in both soil and groundwater at a high-resolution. In systems that are affected by pyrite weathering, such data may serve as a basis for large-scale prognosis of processes and consequences.

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Biographies

Henrik Hecht born in 1972, studied geology at the University of Bremen, Germany and received the diploma in 1998 with a final work about use of fiber-optical sensors for the determination of diffusive oxygen transport in pyrite-bearing sediments. Since 1998, he has been working on his PhD in the geochemistry and hydrogeology group of Professor H.D. Schulz at the department of geosciences, University of Bremen, Germany about long-term stable fiber-optical oxygen sensors for monitoring of pyrite weathering processes.

Martin Kölling born in 1959, studied geology at the Christian-Albrechts-University in Kiel, Germany where he received the diploma in 1986 with a final work about the determination of redox potentials in natural waters. From 1986 to 1990, he completed his PhD in the hydrogeology and marine geochemistry group of Professor H.D. Schulz at the department of geosciences, University of Bremen, Germany about the application of geochemical models and the formation of acid mine drainage. In 1990, he became lab manager in the same group and continued his work on acid mine drainage and on redox systems. In 1996, he started a project in cooperation with the Micro-sensor Research Group of the Max-Planck-Institute for Marine Microbiology on the application of fiber-optical techniques to ground- and seepagewater systems affected by acid mine drainage formation. His scientific interests are in the conversion of fiber-optical sensor techniques originally developed for the micro-scale to inexpensive and long-term stable sensing systems for monitoring purposes.