

# Microscale Chemical Features of Sediment-Water Interface in Hongfeng Lake

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**ABSTRACT:** In situ microscale distributions of O<sub>2</sub>, H<sub>2</sub>S, pH and redox potential in sediments of Hongfeng Lake, SW China, were investigated using the powerful microsensors. Our results show that O<sub>2</sub> was depleted within the top 3.9 mm in surface sediments, and H<sub>2</sub>S was subsequently detected at ~6.0 mm depth, and reached its maximum concentrations at ~25 mm. The degradation of organic matter and reduction of sulfate might be the major pathways of producing H<sub>2</sub>S in sediments. pH rapidly reduced in surface layers mainly due to H<sup>+</sup> release in the oxidation of organic matter. Eh also decreased sharply in surface sediments, probably indicating the coexistence of Fe and Mn oxides with O<sub>2</sub> in aerobic region. Furthermore, the programme of PROFILE was applied to model the O<sub>2</sub> gradient, and good fit was obtained between the simulated values and the factual values both in sediments and in the diffusive boundary layer (DBL). The results indicate that the depth-integrated O<sub>2</sub> consumption rates within sediments were 0.083 and 0.134 nmol·m<sup>-3</sup>·s<sup>-1</sup> in site S1 and site S2, respectively. In addition, there were distinct DBL in two sediment profiles, with 1.2 mm thickness in S1 and 0.9 mm thickness in S2. The diffusive fluxes of O<sub>2</sub> within the DBL were 67.13 nmol·m<sup>-2</sup>·s<sup>-1</sup> in S1 and 88.54 nmol·m<sup>-2</sup>·s<sup>-1</sup> in S2.

**KEY WORDS:** microscale, chemical feature, DBL, sediment, Hongfeng Lake.

## 0 INTRODUCTION

Eutrophication is a global environmental problem, especially in developing countries (Zhang et al., 2008). Human activities impelled the emission of pollutants and accelerated the process of eutrophication (Conley et al., 2009). Consequences of this pollution could threaten the supply of drinking water for human beings directly. In comparison of the immense amounts of studies on freshwater system of natural lakes, few investigations have been carried out on the anthropogenic impacts to drinking waters. In general, as the conventional source of drinking water, reservoirs are set in surrounding areas of densely populated urban (Chen et al., 2010). A large quantity of wastewater containing heavy metal and other contaminants would be more easily discharged into the reservoirs from farmland and industries. The corresponding pollution results in different patterns of eutrophication from ecosystem of natural freshwaters, and it is imperative to analyze and evaluate the eutrophication in artificial water surrounding, especially reservoirs.

Nitrogen and phosphorus are critical limiting factors of eutrophication in aquatic ecosystem. The physicochemical properties of sediments not only indicate historic records, they

are also indexes of studying on the species, transport and transformation of nitrogen and phosphorus in sediment-water interface (Zhan et al., 2014; Zhang et al., 2014; de Beer et al., 2006; Paerl and Pinckney, 1996; Gundersen and Jørgensen, 1990). The research of physicochemical properties of sediments could be beneficial to understand the mechanisms and control of eutrophication for aquatic ecosystem. Especially, the availability and turnover of O<sub>2</sub> are related to the geochemical cycling of carbon, phosphorus, sulfur and metals directly (Kristensen and Holmer, 2001; Hulthe et al., 1998; Canfield, 1994). Whereas, previous studies figured out that oxygen could be depleted within a depth of a few millimetres in upper layer of sediments (Elberling and Damgaard, 2001; Rasmussen and Jørgensen, 1992; Gundersen and Jørgensen, 1990). Consequently, the microscale and in situ measurement techniques are widely applied in water environment.

Multiple methods, such as in situ samplers (Kuivila et al., 1989), dialysis cells (Xu et al., 2012), so-called peepers (Brandl and Hanselmann, 1991), slicing techniques (Thamdrup et al., 1994) and gel samplers (Ding et al., 2010a, b; Krom et al., 1994), were used to monitor macroscale profiles in the past years. However, these measurements can not obtain precise data in microscale (Berg et al., 1998; Glud et al., 1994). High resolution microelectrodes with small needle-shaped probes (a tip diameter of 1–20 μm), which disturb microenvironments minimally, have been developed and proved to be an available tool in microscale studies (Pedersen et al., 2011; Kuhl and Revsbech, 2001; Gundersen and Jørgensen, 1990). At present,

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microelectrodes have been widely used to evaluate  $O_2$  distributions in sediments (Rasmussen and Jørgensen, 1992; Revsbech et al., 1986). Simultaneously, numerical procedures for measured concentration microprofiles of solutes in sediment pore water have been developed to calculate the flux across the sediment-water interface (Berg et al., 1998).

Hongfeng Lake is one of the largest artificial freshwater lakes of the southwest plateau of China, and supplies approximately 3 million people for drinking water. Quantities of wastewater from surrounding areas are directly discharged into the lake every year. Eutrophication has become more serious to the water quality of the lake (Feng et al., 2011; Jiang et al., 2011). According to the previous documents, there has carried out amounts of researches on physicochemical characters of sediments and porewater in relative macroscale, But few projects implemented in this area are based on microtechniques.

In our study, in situ microscale measurements of  $O_2$ ,  $H_2S$ , pH and redox potential in sediment cores were obtained using a powerful and versatile four-channel microsensors multimeter collected from Hongfeng Lake. Our main objectives were to (1) evaluate the consumption rates of  $O_2$  in surface layer of sediments in the study area, and (2) to deeply understand microgradients of the chemical properties in sediment-water interface.

## 1 MATERIALS AND METHODS

### 1.1 Study Area

Hongfeng Lake (106.24°N, 26.30°E) is one of the largest man-made reservoirs of the southwest plateau of China (Fig. 1). It has a surface area of 57.2 km<sup>2</sup>, with a mean water depth of 10.5 m and water capacity of  $6.01 \times 10^8$  m<sup>3</sup>. Two major inflows bring approximately 530 million m<sup>3</sup> of industrial wastewater into the reservoir every year. The only outlet, the dam, is set in the northeastern corner of the whole lake. The area has a subtropical monsoon climate with the annual precipitation of 1 200 mm. Carbonate formations are widely distributed, and water present near neutral to slightly alkaline (pH 7.47–8.67). Compared of other lakes in Guizhou, eutrophication is more serious in Hongfeng Lake, with concentrations of total P, total N, CHLa are 0.03–0.10, 1.0–2.5, and 2–75 mg/L, respectively.

### 1.2 Sample Collection

Sediment cores were collected from two sites (S1 and S2) of Hongfeng Lake in February 2012 (Fig. 1), using a gravity sediment core sampler invented by Institute of Geochemistry, CAS. The cores would be accepted only if they had a visibly undisturbed sediment surface and clear overlying water. As soon as the cores were obtained, the sampler (polymethyl methacrylate glass tube) was wrapped by the tinfoil being preserved from light through the transportation and measurement process. In order to reduce the effect of oxygen from air to the DBL and sediments, the overlying water of sediment cores was continuously flushed with a mixed gas of  $N_2$  and air, and keep it in the anaerobic conditions (DO~2.0 mg/L, close to the DO concentration level of the bottom waters of the lake). In the lab, cores for in situ monitor were pre-incubated for 2 h prior to measurements to make sure steady-state  $O_2$  distributions in the sediments. Meanwhile, paratactic sediment cores in two sample sites were taken for determining porosity.

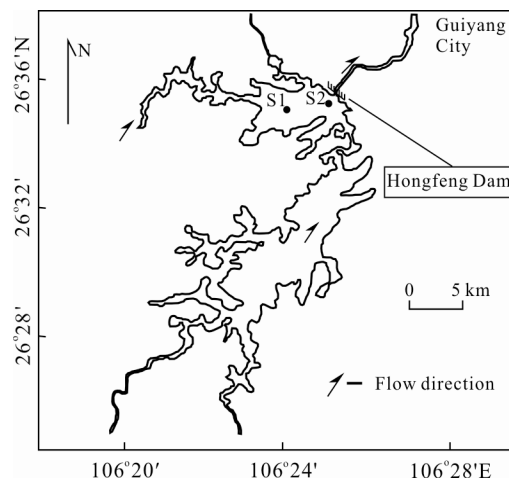


Figure 1. Description of the study area and sampling sites.

### 1.3 Chemistry of Sediments

High-resolution vertical distributions of  $O_2$ ,  $H_2S$ , pH and redox potential (Eh) in Hongfeng Lake were determined by a powerful and versatile four-channel microsensors multimeter (Version 2.01, Unisense, Aarhus, Denmark) with a spatial resolution of 100  $\mu$ m.

(1) The applied  $O_2$  microsensor was of the micro-Clark-type described by Revsbech (1989), with a sensing-tip diameter of <10  $\mu$ m, a 90%-response time of 1 s, and a velocity sensitivity of <1%–2%. A two-point linearly calibration of the microsensor was done in air-saturated calibration solution and in the anoxic solution (~2 g sodium ascorbate in 100 mL of 0.1 M NaOH).

(2) The  $H_2S$  microsensor is a miniaturized amperometric sensor with an internal reference, a sensing and a guard anode (Jerosechewski et al., 1996). Calibration was done in a dilution series of a sulfide standard solution buffered to the same pH (7.2–7.3) as the synthetic biofilm medium and with the same salinity (flushed with nitrogen to avoid oxidation of the sulfide).

(3) The pH microelectrode is a miniaturized glass pH electrode with tip diameters down to 10  $\mu$ m. The electrodes were calibrated at room temperature (20 °C) in three standard pH buffers. It responds linearly between the three points with a slope of 52–60 mV/pH-unit and a 90% response time of <10–20 s.

(4) The redox microelectrode is a miniaturized platinum electrode, using in combination with a reference electrode (e.g., Unisense REF-RM). The offset of the reference electrode calibrated in quinhydrone redox buffer solution (pH 4 and 7) was related to the redox microelectrode.

### 1.4 Calculation of Diffusive $O_2$ Flux

Within the diffusion boundary layer (DBL), the water current velocity would be reduced by a thin film (<1 mm) of water in sediments. The DBL constitutes an important diffusion barrier to the exchange of  $O_2$  and other dissolved compounds, and molecular diffusion becomes the dominant transport mechanism for these materials along a near linear concentration gradient (Gundersen and Jørgensen, 1990). The diffusive flux ( $J$ , nmol·m<sup>-2</sup>·s<sup>-1</sup>) of  $O_2$  downwards across the sediment-water

interface was analyzed by Fick's first law, based on the measured  $O_2$  microgradients through the DBL

$$J = -D \frac{dC}{dx} \quad (1)$$

where  $D$  is molecular diffusion coefficient of oxygen ( $cm^2 \cdot s^{-1}$ ),  $C$  is  $O_2$  concentration ( $\mu M$ ),  $x$  is depth (mm,  $x=0$  at sediment-water interface), and  $\frac{dC}{dx}$  shows the concentration gradient.

Actually, the flux can also be realized as the finite shift in  $C$  across the DBL (Eq. (2)). This method was applied to study molecular diffusion in a coastal sediment by Rasmussen and Jørgensen (1992).

$$J = D(C_w - C_0) / x_\delta \quad (2)$$

where  $C_w$  is  $O_2$  concentration in the overlying water ( $\mu M$ ),  $C_0$  is  $O_2$  concentration at the sediment-water interface ( $\mu M$ ),  $x_\delta$  is the thickness of the effective DBL (mm).

Oxygen consumption rates within sediments were calculated by a program of PROFILE code (Berg et al., 1998) based on zero-order kinetics with the rate being independent of depth in the sediment. The designing principles of the program could be described by the following equation, assuming to be at steady state balanced by the supply due to diffusion (Berg et al., 1998)

$$\frac{d^2C}{dx^2} = \frac{R}{\phi_s D_s} \quad (3)$$

where  $R$  is the consumption rate of  $O_2$  per unit volume of pore water in sediments ( $nmol \cdot m^{-3} \cdot s^{-1}$ ),  $C$  is  $O_2$  concentration in the pore water ( $\mu M$ ),  $D_s$  is the sediment diffusion coefficient of  $O_2$  ( $cm^2 \cdot s^{-1}$ ),  $\phi_s$  is porosity.

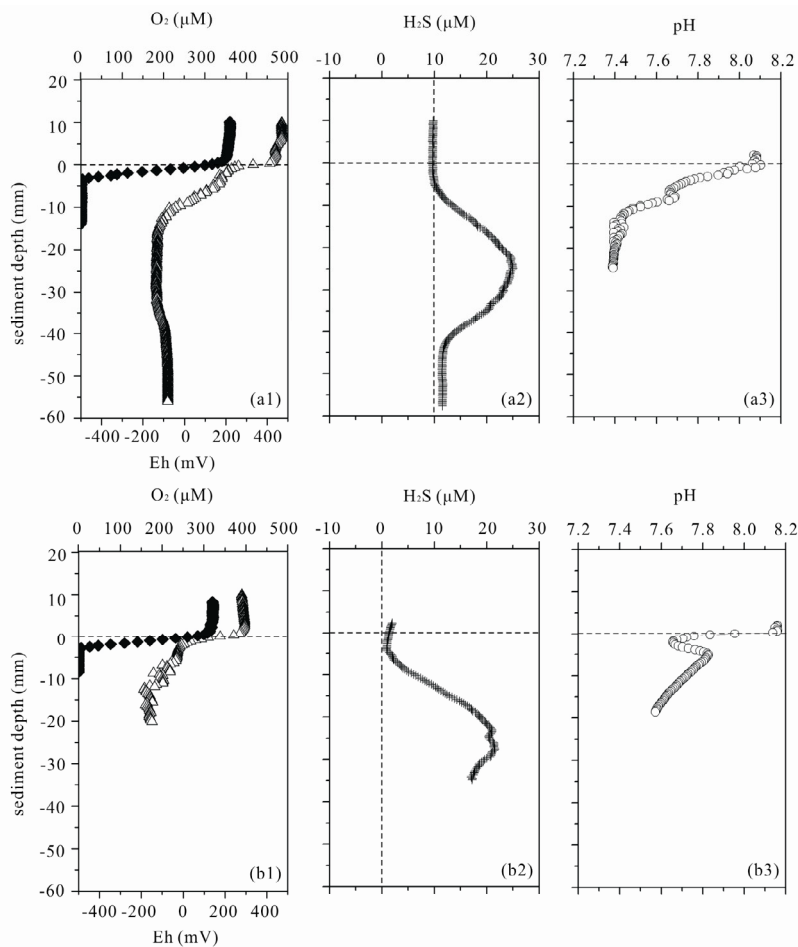
The porosity was determined from the weight loss upon drying at  $105^\circ C$  of sediment core segments. The weight and volume of the sediment segments were previously measured in lab. Values of  $D_s$  were calculated from the porosity and molecular diffusion coefficient of oxygen according to Eq. (4). Values of  $D$  were taken from Wu et al. (1997).

$$D_s = D / (1 + 3(1 - \phi_s)) \quad (4)$$

## 2 RESULTS AND DISCUSSION

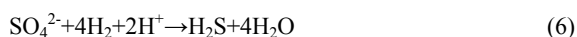
### 2.1 Microgradients of $H_2S$ , pH and Eh

Aside from the observation of microgradient of  $O_2$ , microprofiles of  $H_2S$ , pH and Eh were measured in two sediment cores collected from Hongfeng Lake. The results showed that similar distributions of  $H_2S$  and Eh in vertical profiles were monitored in two cores, as shown in Fig. 2. Oxygen was depleted within the top 3.9 mm of the sediment in two profiles. Subsequently,  $H_2S$  could be detected downwards to  $\sim 6.0$  mm



**Figure 2.** Distributions of  $O_2$  (black box),  $H_2S$  (cross), pH (circle) and redox potential (triangle), in two sediment profiles. (a1), (a2), (a3) mean the sediment collected from S1; (b1), (b2), (b3) mean the sediment collected from S2.

depth. It is worthy to note that there is a blank zone (a vertical distance of ~2.1 mm) during the process of O<sub>2</sub> exhausting and H<sub>2</sub>S presenting, which is different from the feature reported by de Beer et al. (2006) and Jørgensen and Revsbech (1983). In comparison of the phenomenon of existing overlap zone, chemolithotrophic bacteria competing successfully with non-biological H<sub>2</sub>S oxidation (Nelson et al., 1986; Møller et al., 1985), we supposed that oxidizing functions, such as NO<sub>3</sub><sup>-</sup>, Mn<sup>4+</sup>, Fe<sup>3+</sup>, acted as a prior electron acceptor to take part in redox reaction in the blank zone (Holmkvist et al., 2011; Berner, 1981; Froelich et al., 1979; Thauer et al., 1977). Our data also showed that the concentrations of H<sub>2</sub>S increased to the maximum at the depth of approximate 25 mm in two sediment cores. In sediments, the major pathways of producing H<sub>2</sub>S included degradation of organic matter (OM) and reduction of sulfate (Böttcher and Lepland, 2000). Bacteria obtained energy by oxidizing organic compounds or molecular hydrogen (H<sub>2</sub>) during reducing SO<sub>4</sub><sup>2-</sup> to H<sub>2</sub>S in anaerobic aqueous environments (Risgaard-Petersen et al., 2012; Eqs. (5), (6)). Besides, analogous distribution pattern of H<sub>2</sub>S in sediment profile was documented by Wang et al. (2003) sampled in A-ha Lake, where has a distance about 10 km from Hongfeng Lake. Sulfate-reducing bacteria (SRB) accounted for the distribution of H<sub>2</sub>S in this lake.



Compared with tendencies of pH in two sediment profiles, they declined rapidly in surface layers consistently. In the aerobic condition, the deposited organic matter in the reduction state was likely to be oxidized by O<sub>2</sub> to generate organic matter in the oxidation state, meanwhile, H<sup>+</sup> will be released into the pore water (Kamp et al., 2006; Sayama et al., 2005), as the probable approach shown in Eq. (7). It was probably the main reason of reduced pH in pore water in sediments. In addition, Eh decreased sharply in sediment-water interface in two sediment profiles. It probably indicated that other oxides, such as Mn and Fe oxides, coexisted with O<sub>2</sub> in aerobic region. Whereas, these oxides would be reduced across the sediment-water interface (Tankéré et al., 2002).



The distributions of H<sub>2</sub>S, pH and Eh observed that sediments exhibited a kind of small-scale dynamic environment in

Hongfeng Lake. Especially as a seasonal stratified lake, turbulent mixing followed by unexpected convection and material exchange occurred in Hongfeng Lake every year. Wang et al. (2012) figured out that the vertical mixing in this lake was turbulent not only in bottom water, but also in surface sediments during early autumn. This behaviour impelled H<sub>2</sub>S produced in sediments exchanging upwards to water, which would generate water stench, even lead to fish death. The microscale measurement of these chemical parameters of sediments and pore water were beneficial to analyze the probable geochemical process in aquatic environment. Meanwhile, they also could provide reliable data to reduce the negative impact from pollutants.

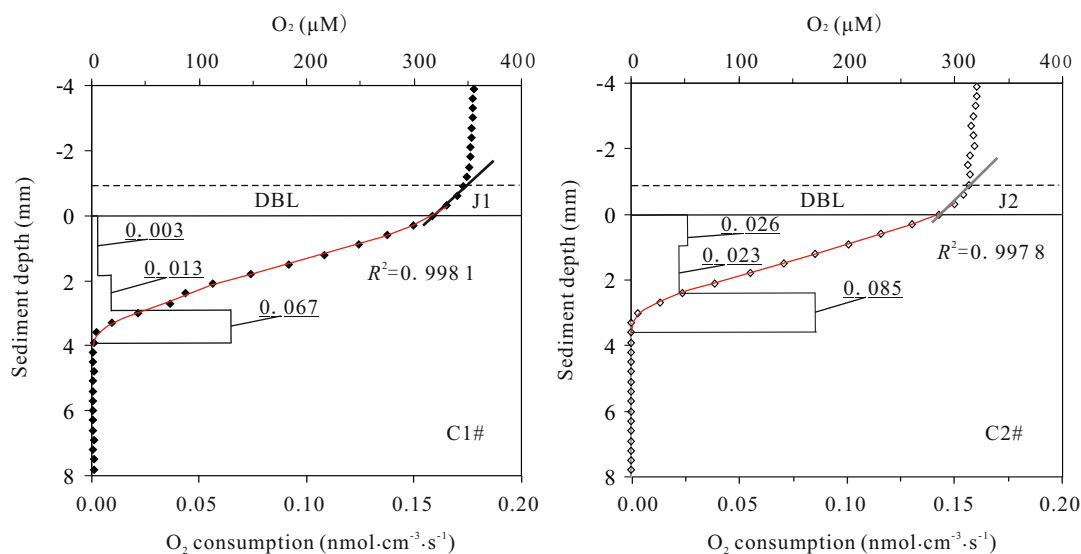
### 2.2 Modeling of Oxygen Consumption

During the sampling occasion, O<sub>2</sub> could penetrate 3.9 and 3.6 mm into the sediment in S1 and S2. Before calculating the O<sub>2</sub> consumption rates, sediment porosity was analyzed to obtain the value of D<sub>s</sub>, ranging from 0.90, 0.88 in the uppermost 0–2 mm to 0.82, 0.84 at the bottommost layer in S1 and S2, respectively (Table 1).

We used the program of PROFILE to model O<sub>2</sub> gradient, and the simulative value yielded a perfect fit with the corresponding factual value, both in the sediment and in the diffusive boundary layer (99.81% similitude for S1, and 99.78% similitude for S2). The microprofile and the corresponding consumption rate of O<sub>2</sub> within the oxic surface layer of two sediment profiles were showed in Fig. 3. At the in situ temperature of 13 °C, the O<sub>2</sub> consumption rate, R, was calculated through Eq. (1). Calculated results showed that the depth-integrated O<sub>2</sub> consumption rates within sediments were calculated to be 0.083 and 0.134 nmol·m<sup>-3</sup>·s<sup>-1</sup> in S1 and S2. Intense O<sub>2</sub> consumption rates were commonly exhibited through the whole profiles, but not below the DBL in S1. Generally, intense photo synthesis, freshly settling detritus, as well as microbial/iron respiration and nitrification activities impelled the O<sub>2</sub> consumption in the uppermost part of the oxic zone (Glud et al., 2005). And near the oxic-anoxic interface in the sediments, the elevated O<sub>2</sub> consumption rate was presumably due to oxidation of reduced compounds (NH<sub>4</sub><sup>+</sup>, Fe<sup>2+</sup>, H<sub>2</sub>S) diffusing up from below layers (Berg et al., 1998). Besides, the reoxidation of H<sub>2</sub>S might be another reason of the enhanced O<sub>2</sub> consumption. Reiners and Glud (2000) figured out that the DBL impedance would become almost negligible in certain environments,

**Table 1** Distributions of porosity and organic carbon in two sediment cores

	Depth (mm)	Porosity	Orgnic C (%)		Depth (mm)	Porosity	Orgnic C (%)
S1	0–2	0.90	4.907	S2	0–2	0.88	8.289
	2–4	0.90	4.521		2–4	0.87	7.535
	4–6	0.89	3.970		4–6	0.86	6.391
	6–8	0.87	4.113		6–8	0.86	6.846
	8–10	0.85	4.185		8–10	0.85	7.194
	10–30	0.83	4.244		10–30	0.84	7.352
	30–50	0.83	4.612		>30	0.84	7.833
	>50	0.82	4.044				



**Figure 3.** Measured  $O_2$  concentrations from sediment cores (C1#, sediment core from Site 1, C2# from Site 2). The calculated best-fitting concentration profile (Curve), and the connected consumption profile (folding line). Standard errors were calculated from the fits to each single concentration profile ( $R^2$ ). The sediment-water interface is located at depth 0 mm.

and the  $O_2$  consumption must be limited by internal diffusion through the pore space rather than the DBL thickness. It should be the major reason that it was the main reason of the relative lower consumption rate of  $O_2$  near the DBL in S1. However, there is no adequate evidence to elaborate this phenomenon.

### 2.3 Calculating of the Diffusive Flux of Oxygen within the DBL

The upper DBL boundary was determined as the intersection between the extrapolated linear concentration gradient of the DBL and the constant  $O_2$  concentration of the overlying well-mixed water phase (Gundersen and Jørgensen, 1990). There were distinct DBL (1.2 mm in S1 and 0.9 mm in S2) which could be recognized from 2 shifts in the microgradient of  $O_2$  in every sediment core.  $O_2$  concentrations above DBL were relatively constant, which indicated homogeneous overlying water with average  $O_2$  concentrations of  $350.5 \pm 5.9 \mu\text{mol/L}$  in S1 and  $319.8 \pm 2.7 \mu\text{mol/L}$  in S2, respectively.

According to the Eq. (2), we obtained the diffusive flux of  $O_2$  within the DBL, with the flux of  $67.13 \text{ nmol}\cdot\text{m}^{-2}\cdot\text{s}^{-1}$  in S1 and  $88.54 \text{ nmol}\cdot\text{m}^{-2}\cdot\text{s}^{-1}$  in S2. Diffusion is an extremely fast transport mechanism on small spatial scales. Actually, the diffusion time relates to the diffusion distance, and meantime, the diffusion time across the DBL limits the benthic  $O_2$  consumption rate in enclosed water environment, especially in lakes and reservoirs (Glud, 2008). To some extent, it might be related to the lower  $O_2$  consumption rate in S1.

In addition, the thickness of the DBL regulates the distribution of  $O_2$  and microbial mat. In shallow  $O_2$  penetration depths, chemoautotrophic bacteria are most widely distributed, and the sulphur bacterium *Beggiatoa spp.* is the most typical study objective (Schulz and de Beer, 2002). The bacteria efficiently compete with the chemical oxidation of  $H_2S$  with  $O_2$  (Jørgensen and Revsbech, 1983). The DBL thickness can change the structure of the mat directly. Therefore, the infor-

mation of the DBL could contribute to better understanding of the distribution pattern of  $H_2S$  in two sediment cores discussed in the previous sections.

### 3 CONCLUSIONS

In our study, in situ microscale measurements of  $O_2$ ,  $H_2S$ , pH and redox potential in sediment cores were obtained using a powerful and versatile four-channel microsensors multimeter from Hongfeng Lake. Our results show that oxygen was depleted within the top 3.9 mm in sediments, and  $H_2S$  was subsequently detected at  $\sim 6.0$  mm depth, and reached its maximum concentrations at the depth of approximate 25 mm. Degradation of organic matter and reduction of sulfate was supposed to be the major pathways of producing  $H_2S$  in sediments. The rapid reduction of pH occurred in surface layers was likely due to  $H^+$  release in the oxidation of organic matter. Eh also decreased sharply in sediment-water interface, probable indicating that other oxides, such as Mn and Fe oxides, coexisted with  $O_2$  in aerobic region. Furthermore, a programme of PROFILE code was applied to model the  $O_2$  gradient. And the simulative value could perfectly fit the factual value, both in the sediment and in the diffusive boundary layer. The  $O_2$  consumption rate in sediment and diffusive flux in the DBL were calculated by a series of equations. The results indicate that the depth-integrated  $O_2$  consumption rates within sediments were  $0.083$  and  $0.134 \text{ nmol}\cdot\text{m}^{-3}\cdot\text{s}^{-1}$  in S1 and S2. In addition, there were distinct DBL in two sediment profiles, with 1.2 mm thickness in S1 and 0.9 mm thickness in S2. The diffusive fluxes of  $O_2$  within the DBL were  $67.13 \text{ nmol}\cdot\text{m}^{-2}\cdot\text{s}^{-1}$  in S1 and  $88.54 \text{ nmol}\cdot\text{m}^{-2}\cdot\text{s}^{-1}$  in S2.

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