Dissolved organic sulfur in streams draining forested catchments in southern China

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Abstract

Dissolved organic sulfur (DOS) is an important fraction for sulfur mobilization in ecosystem. In this work stream waters were sampled in 25 forested sites in southern China to study the dissolved sulfur fractions. Dissolved sulfur was fractionated into dissolved organic sulfur (DOS) and inorganic sulfate (SO_4^{2-}) for 95 stream water samples. The results showed that the concentration of DOS ranged from 0 to 13.1 mg/L (average 1.3 mg/L) in all the streams. High concentrations of DOS in stream waters were found in the sites with high concentrations of sulfate. DOS constituted less than 60.1% of dissolved sulfur (average 17.9%). Statistical analysis showed that DOS concentration was correlated with SO_4^{2-} in streams waters and total sulfur in surface layer soils. The results also showed that DOS concentration in stream waters had a seasonal variation, but no trends were found with it. The implication was that the long term sulfur deposition had led the increase of the concentration and fraction of DOS in stream waters in acid rain prevailing regions

Key words: dissolved organic sulfur; stream water; Chinese forests; sulfur deposition

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Introduction

Dissolved natural organic matter (DNOM) is a continuum of organic molecules of different sizes and structures that pass through a 0.45 μm -pore size filter (Kalbitz et al., 2000; Zsolnay, 2003). DNOM plays an important role in soil formation (Dawson et al., 1978), mineral weathering (Raulund-Rasmussen et al., 1998) and transport of pollutants (Kalbitz et al., 1997; Marschner, 1998; Temminghoff et al., 1997). Approximately half mass of DNOM is constituted by carbon. The concentration of dissolved organic carbon is therefore commonly used as a proxy for DNOM. The rest is mainly made up by oxygen and hydrogen, along with varying content of nitrogen and sulfur, and a minor content of phosphorous. DNOM is therefore also important in the transport of nutrients from soil to surface waters (Neff et al., 2003). There were many studies describing the content of the primary macronutrient nitrogen in soil and groundwater DNOM (Christou et al., 2006; Jones et al., 2004; Kroeger et al., 2006; Lorite-Herrera et al., 2009; Yue et al., 2008; Wu et al., 2010), but the literature regarding the content of the secondary macronutrient sulfur in DNOM, dissolved organic sulfur (DOS) was much less.

Sulfur in DNOM occurs as reduced (e.g., sulfide and thiol) or as oxidized species (e.g., sulfonate and sulfate).

Several studies on the transport of mercury (Hg) from soils to surface waters have focused on the amount and form of sulfur in DNOM since the reduced sulfur sites in DNOM are found to be important for mercury binding (Amirbahman et al., 2002; Haitzer et al., 2002; Skyllberg et al., 2000).

A few studies indicate that 10%–20% of dissolved sulfur content in soil and groundwater could be bound to DNOM (Nilsson et al., 2001; Mitchell et al., 1986, 1989). DOS may therefore in some cases constitute a significant part of the total flux of sulfur from terrestrial to aquatic environment. Nevertheless, the sulfur flux calculations are commonly based solely on sulfate (SO_4^{2-}) – assuming that DOS is present in only minor amounts. In a few cases the content of DOS is roughly estimated based on an assumed fixed ratio to the amount of dissolved organic carbon (DOC) (Raue et al., 1991). In fact, the content of DOS in DNOM may vary considerably with season and space (Peuravuori et al., 2005). A sound understanding of the factors governing the temporal and spatial variation in DOS is therefore a prerequisite in order to make sound catchment budgets of sulfur.

Houle et al. (1995) found that the content of DOS in lake water DNOM was affected by sulfur loading in the form of acid deposition. Regional sulfur deposition increases the accumulation of sulfate adsorbed to the soil (Galloway et al., 1983) and thereby the amount of sulfur incorporated

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into the soil organic matter (Prietz et al., 2004). There is therefore a specific need for a better understanding of the effects of sulfur deposition on the amount of DOS in stream water in the acid rain prevailing region in southern China, experiencing a changed sulfur deposition.

In this study, seasonal and spatial variation of the sulfur fractions, DOS and SO_4^{2-} were assessed based on sulfur fractionation results on 95 stream water samples from 25 small forested catchments in southern China, experiencing a span in sulfur loading.

1 Materials and methods

1.1 Sampling and analysis

Water samples were collected from 25 forested catchments in southern China (Fig. 1 and Table 1) during July to November in 2009 for a synoptic study of spatial variation

Fig. 1 Geographical distribution of sampling sites.

in sulfur fractionation. Two to six water samples were collected from each catchment depended on local condition. Soils were also sampled from the surface layers in the recharge areas close to the streams. At sites YLS and MJW stream water samples were collected weekly in 2009 to obtain the seasonal fluctuations in stream water chemistry.

All water samples were filtered through 0.45 μm filters and frozen at -20°C prior to analysis. Dissolved sulfur (DOS plus sulfate) and major cations were analyzed according to ISO 11885 by inductively coupled plasma-optical emission spectroscopy (ICP-OES) (Prodigy, Leeman Labs, USA) with the optical system purged with argon. The line at 182.034 nm was selected for sulfur measurement (Prietz et al., 1996). SO_4^{2-} and other major anions were analyzed using ion chromatography (IC) (DX-120, Dionex corporation, USA) according to ISO 10304-1. The same standards were used for calibration of the measurements by both ICP-OES and IC to minimize error. Thus DOS concentration (dissolved sulfur minus the sulfate) was obtained (David and Mitchell, 1985; Homann et al., 1990; Mitchell et al., 1986).

DOC was determined by a TOC analyzer (Phoenix 8000 UV-persulfate TOC analyzer, Tekmar Dohrmann, USA). The pH value was determined by a pH combination electrode (Rex E-201-C-9, Shanghai Rex, China) according to ISO 10523. Total-sulfur, -carbon and -nitrogen in the soil samples was determined by a C\N\H\S analyzer (Vario Elementar III, Elementar Analysensysteme GmbH, Germany) (Wang et al., 2006).

1.2 Method for data analysis

Statistical methods were utilized to assess the parameters influencing the DOS concentration. These parameters included pH, DOC, major anions and cations in water, and total-carbon, -nitrogen and -sulfur in soils. The statistical

Table 1 List of the catchments in this study

Catchments (symbol)	Latitude/Longitude	Forest types
Daowushan (DWS)	28°12'N/113°34'E	Coniferous forest
Damaan (DMA)	21°59'N/108°36'E	Deciduous broadleaf forest
Dashipan (DSP)	25°48'N/115°02'E	Evergreen broadleaf forest
Dongjianghu (DJH)	25°52'N/113°18'E	Subtopic evergreen broadleaf forest
Fengzhan Reservoir (FZR)	27°22'N/117°39'E	Mixed coniferous-deciduous forest
Huanggang Reservoir (HGR)	25°01'N/116°54'E	Mixed coniferous-deciduous forest
Hongqi Reservoir (HQR)	25°56'N/114°50'E	Evergreen broadleaf forest
Juntan Reservoir (JTR)	28°13'N/118°16'E	Evergreen broadleaf forest
Laohuling (LHL)	22°56'N/108°20'E	Deciduous broadleaf forest
Longwantan (LWT)	28°20'N/120°52'E	Mixed coniferous-deciduous forest
Luchongguan (LCG)	26°26'N/106°39'E	Mixed coniferous-deciduous forest
Maweizhao (MWZ)	28°21'N/113°35'E	Mixed coniferous-deciduous forest
Meijiawu (MJW)	30°16'N/120°12'E	Mixed coniferous-deciduous forest
Sanmenjiang (SMJ)	24°21'N/109°29'E	Mixed coniferous-deciduous forest
Shuanglongdong (SLD)	29°12'N/119°37'E	Mixed coniferous-deciduous forest
36Qu Forest Park (36Q)	22°02'N/108°37'E	Mixed coniferous-deciduous forest
Tieshanping (TSP)	29°38'N/106°41'E	Mixed coniferous-deciduous forest
Tianmushan (TMS)	30°20'N/119°26'E	Mixed coniferous-deciduous forest
Xinfu Reservoir (XFR)	28°41'N/115°41'E	Evergreen broadleaf forest
Xixia Reservoir (XXR)	28°50'N/115°47'E	Evergreen broadleaf forest
Xiyunjiang (XYJ)	22°58'N/108°32'E	Deciduous broadleaf forest
XueDouShan (XDS)	29°40'N/121°13'E	Coniferous forest
Yuelushan (YLS)	28°11'N/112°55'E	Evergreen broadleaf
Yinpingshan (YPS)	22°52'N/114°14'E	Mixed coniferous-deciduous forest
Wangzai Reservoir (WZR)	28°42'N/118°04'E	Evergreen broadleaf forest

methods employed were a simple correlation analysis, stepwise multiple linear regression and partial least squares regression.

When co-linearity relationships exist between each independent variable, errors will occur in the results of linear regression. Therefore the partial least squares regression was utilized to reduce the errors (Guo et al., 2007).

2 Results and discussion

2.1 DOS and sulfate concentrations in stream water at different sites

The concentrations of dissolved sulfur (DOS plus sulfate) in the most of water samples were found to be less than 10 mg/L (Figs. 2 and 3). High concentrations of dissolved sulfur (> 10 mg/L) in water samples were found at LCG in Guizhou, at TSP in Chongqing and at SMJ in Guangxi. These three sites have all been subjected to a heavy loading of sulfur deposition during the past several decades (Chen

and Liu 1996; Larssen et al., 2006). At these sites the concentrations of DOS ranged from 4.4 to 13.1 mg/L and SO_4^{2-} from 6.2 to 33.6 mg/L (Fig. 3). The high concentrations of sulfate in these sites were linked with high concentrations of base cation leaching from soils. It may indicate that the soils in these sites had not capacity to adsorb extra amounts of sulfate. With the exception of the sites LCG, TSP and SMJ the DOS concentration were quite low with an average of 0.2 mg/L (Fig. 2), which were comparable to the concentrations (0.2 to 0.4 mg/L) for fifty-nine lakes in Québec, Canada (Houle et al., 1995).

DOS accounted for average 15.5% (0 to 42.2%) of the dissolved sulfur at the sites with low concentration (<10 mg/L) of dissolved sulfur in water samples. At the sites with high concentration of dissolved sulfur, DOS accounted for 12.8% to 60.1% of dissolved sulfur which was higher than the values (1% to 18%) in stream waters of the Adirondacks, USA (David and Mitchell, 1985) and the values (12% to 21%) in stream waters of the Rocky Mountains (Mitchell et al., 1986).

Fig. 2 Concentrations and fractions of DOS and sulfate at sites with dissolved sulfur level less than 10 mg/L (Gd denotes Guangdong).

Fig. 3 Concentrations (a) and fractions (b) of DOS and sulfate at sites with dissolved sulfur level higher than 10 mg/L.

2.2 Seasonal variations of DOS and sulfate concentrations in stream water

Monthly mean concentrations of DOS and sulfate in stream water at YLS and MJW sites were used to study the seasonal variation of dissolved sulfur. The sites YLS and MJW have sub-tropical monsoonal climate with a cold and dry winter and warm and wet summer.

The results showed clear seasonal variation in stream water at YLS (Fig. 4). The concentrations of sulfate remained high except during the fall, when the concentration dropped to less than 1.5 mg/L. The absolute and relative amounts of DOS in stream water were generally decreased along with decreasing amount of rain and temperature, with low values during the cold winter months (January and February) and high during the hot summer months

(Fig. 4). A maximum concentration of 2.4 mg/L for DOS was measured in May. The concentrations of dissolved sulfur was higher in summer than those in winter, which maybe caused by the persistent high temperature and drought in summer 2009 in Hu'nan Province (Luo et al., 2010).

Data were lacked for the winter months at MJW due to non-perennial flow. In the data available, the concentration of sulfate remained rather stable (basically from 5 to 6 mg/L), which may be caused by the adsorption of soil and absorption of biology. While the concentrations of DOS varied rather oppositely to what was found at YLS. The concentration of DOS was generally lower during early spring than that in the fall. The minimum and maximum concentrations of DOS were found in June (< 1 mg/L) and

Fig. 4 Seasonal variation of dissolved sulfur at YLS in Changsha and at MJW in Hangzhou.

in September (5.7 mg/L), respectively.

The differences of DOS concentrations in stream waters between catchments YLS and MJW might attributed to more influenced by heavy precipitation in MJW, and May to July is the rainy season with 250 to 300 mm/month rainfall in Hangzhou (Jin et al., 2010). The low DOS concentrations during these rainy seasons are therefore likely due to an overall dilution caused by the large amounts of rain water. In general, no consistent trends were found for the DOS concentrations in the streams at these two catchments. The seasonal variations of DOS in stream water still need to be further studied.

2.3 Factors influencing the DOS concentration

Stepwise multiple linear regressions were employed to empirically identify parameters governing the spatial variation in DOS concentrations. In the method, DOS was selected as dependent variable while DOC, SO_4^{2-} , NO_3^- , K^+ , Na^+ , Ca^{2+} , Mg^{2+} , NH_4^+ of surface waters and total carbon, total nitrogen, total sulfur of soils in the catchment were selected as possible independent variables. SO_4^{2-} along with Mg^{2+} in waters and total sulfur in the soils were found to be the significant factors in the model. Together they are able to explain 89% of the spatial variation in DOS (Table 2).

A significant co-variation between several independent variables in the correlation matrix of stepwise regression may have a confounding influence on the multiple regression results (Table 3). The data were therefore also analyzed using partial least squares regression. This anal-

ysis confirms that SO_4^{2-} in streams and total sulfur in surface layers soils play a governing role, and also suggests that NO_3^- and DOC also may play a significant role in governing the concentration of DOS, having variable importance in the projection greater than 1.0. Furthermore, Ca^{2+} was found to be more important by the partial least squares analysis and the index of Mg^{2+} changed into a positive relation (Fig. 5) comparing with the results from the stepwise multiple linear regressions. The indexes of SO_4^{2-} based on the two methods were similar. While the reason that Mg^{2+} and Ca^{2+} are positively correlated to DOS in the multiple regression is due to that they, as the major counter ions to the dominant anion sulfate, are inherently and positively correlated to SO_4^{2-} ($r = 0.76$ and 0.96 , respectively. As shown in Table 3). Both stepwise multiple linear and partial least square regressions showed the influence of stream sulfate and soil sulfur on DOS. Conceptually, sulfur deposition is the main source for these two sulfur pools in non-sulfate soil region. Therefore, sulfur deposition probably contributes to the leaching of DOS. This is the possible reason for high DOS concentrations at three sites (i.e. LCG, SMJ and TSP) receiving high sulfur deposition.

DOC is conceptually expected to be an explanatory parameter for DOS as both parameters are inherently related to the amount of DNOM. A study by Homann et al. (1990) found a strong relationship between DOC and DOS in various water compartments of temperate forests and that DOS was leached out together with DOC into stream water.

Table 2 Stepwise multiple linear regressions results

Model	Predictor in model	<i>B</i>	SE	<i>P</i>	<i>R</i>	Adjusted <i>R</i> ²	<i>R</i> ² change
1	(Constant)	0.29	0.62	0.64	0.64	0.41	0.41
	SO_4^{2-}	0.12	0.03	0.00			
2	(Constant)	0.88	0.46	0.07	0.85	0.72	0.31
	SO_4^{2-}	0.44	0.08	0.00			
	Mg^{2+}	-2.21	0.48	0.00			
3	(Constant)	-0.86	0.78	0.29	0.89	0.80	0.08
	SO_4^{2-}	0.38	0.07	0.00			
	Mg^{2+}	-1.88	0.44	0.00			
	Total sulfur	53.10	20.43	0.02			

Predictor in model: variables selected into the model; *B*: partial regression coefficient; SE: standard errors.

Table 3 Correlation matrixes between DOS and anion, cation, DOC in streams or total carbon (TC), total nitrogen (TN), total sulfur (TS) in soil

	pH	Na^+	NH_4^+	K^+	Mg^{2+}	Ca^{2+}	F^-	Cl^-	NO_3^-	SO_4^{2-}	DOC	TC	TN	TS
DOS	-0.01	0.07	0.06	0.25	0.45	0.63	-0.05	0.14	0.48	0.64**	-0.10	0.59**	0.46*	0.057**
pH	1	0.04	0.33	0.04*	0.50**	0.56*	0.15	-0.12	0.05	0.38*	0.14	-0.31	-0.23	-0.15
Na^+		1	0.11	0.57**	0.20	0.15	0.30	0.93***	0.17	0.20	0.36	-0.14	-0.23	-0.16
NH_4^+			1	0.36*	0.52**	0.21	-0.10	-0.01	-0.22	0.44*	0.25	0.09	0.02	0.17
K^+				1	0.66***	0.38**	0.41*	0.42*	0.17	0.59**	0.58**	-0.04	-0.20	0.04
Mg^{2+}					1	0.68***	0.09	0.15	0.22	0.96***	0.30	0.15	0.03	0.16
Ca^{2+}						1	0.02	0.16	0.62**	0.76***	0.08	0.03	0.01	0.05
F^-							1	0.12	0.06	0.03**	0.43*	-0.21	-0.22	-0.09
Cl^-								1	0.24	0.20	0.25	-0.06	-0.15	-0.16
NO_3^-									1	0.39*	-0.06	0.04	0.04	-0.08
SO_4^{2-}										1	0.22	0.29	0.14	0.25
DOC											1	-0.24	-0.43*	-0.46*
TC												1	0.93***	0.77***
TN													1	0.77***
TS														1

***, **, * Significance at level $p < 0.001$, $p < 0.01$, $p < 0.05$, respectively.

Fig. 5 Variable importance and coefficients in the projection of partial least squares regression.

3 Conclusions

Dissolved organic sulfur and sulfate in stream waters from forested catchments were studied in southern China. It was found that DOS concentration in all the streams ranged from 0 to 13.1 mg/L (average 1.3 mg/L), while SO₄-S ranged from 0.2 to 33.6 mg/L (average 3.9 mg/L). The DOS concentrations of water samples in most sites were below 1 mg/L. High concentrations of DOS were found in the sites that are exposed to a heavy acid rain loading and therefore had high SO₄²⁻ concentrations in streams. The DOS constituted less than 60.1% of dissolved sulfur (average 17.9%) in all the streams. The concentration of DOS in streams water varied with seasons, and wasn't found consistent trend at two studied catchments. Multivariate statistics showed that the DOS concentration was correlated to SO₄²⁻ in streams and total sulfur in surface layers soils. These implied that high long-term load of sulfate deposition facilitated the sulfate accumulation in forest soil and then increased the concentration and fraction of DOS in stream waters in acid rain prevailing regions.

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