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RESEARCH ARTICLE

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Environmental bioindication of sulfur in tree rings of Masson pine (*Pinus massoniana* L.) in the Pearl River Delta of China

Abstract In order to identify the potential of sulfur (S) content in the rings of Masson pine (*Pinus massoniana*) in the Pearl River Delta as a bio-indicator of regional history of atmospheric pollution, dendrochemistry was used to determine the temporal distribution of S content in the xylem of Masson pines from Zhaoqing Dinghushan and Nanhai Xiqiaoshan, Guangdong Province, southern China. The results indicated that contents of xylem S increased temporally and peaked in the rings formed in the most recent years at both sites. In the rings formed during the same period before the 1980s, S contents were not significantly different between the two sites, while in the rings formed at the same period after the 1980s, S content at Xiqiaoshan were significantly higher than those at Dinghushan. The chronosequences of the S indices at both sites could be easily marked as three periods: before 1970, during 1971–1985, and during 1986–2002. Based on the temporal changes of the xylem S contents and certain social-economic indices after the 1980s in the Delta, the history of atmospheric pollution at the study sites could be reconstructed as follows: 1) before 1970, a period in which the air was relatively clear, 2) 1971–1985, a period in which the air was gradually polluted, and 3) 1986–2002, a period in which the air was most severely polluted in the Delta.

Keywords Pearl River Delta, *Pinus massoniana*, dendrochemistry, sulfur, environmental bioindication

1 Introduction

Environmental pollution caused by industrialization and urbanization has become one of the major problems all over the world. Relevant researches, particularly on the historical trends of atmospheric pollution, have been conducted globally. Dendrochemistry, a branch of dendrochronology, was successfully used for the first time to monitor the long-term changes of trace metals by Lepp (1975). Since then, dendrochemistry has been widely used in revealing regionally historical environmental pollution (Jiang, 1994; Eklund, 1995; Qian et al., 1998; Watmough et al., 1998; Edmands et al., 2001; Nie et al., 2001; Watmough and Hutchinson, 2002; Binlder et al., 2003) after more than 30 years of theoretically- and technically-based development (Wen et al., 2004), and has become one of the most important methods for reconstructing historical changes in the environment (Watmough, 1997).

Since China's reforming and opening-up policy was implemented in the late 1970s, the Pearl River Delta in south China has rapidly developed in terms of economy and society. However, the over-exploitation of natural resources, the deterioration of the environment and the degradation of natural ecosystem services that accompanied the development, have caused adverse impacts on the sustainability of the local society and economy, and also poses severe threats on human health. Due to the rapid growth of the ceramic industry and the sustained expansion of production capacity, increasing energy sources including coal and oil were consumed that resulted in continued rising concentrations of atmospheric SO_2, NO_x and heavy metals in dust depositions. Air pollution has become the most prominent environmental issue in this region (Yang, 1999; Zhang, 1999).

South China has been one of the most serious acid rain regions in China since the late 1980s. Acid pollution has caused base leaching of forest canopy and of soil, and decreased soil buffer capacity which subsequently resulted in forest decline and decreased biodiversity. In the case of severely polluted sites, forest health decline and even large-scale tree dieback were reported (Wen et al., 2006). Tremendous losses of crop production, forest volume and natural ecosystems were also estimated. In addition, heavy metals (Yan et al., 2000; Wong et al., 2002), organic pollutants (Mai et al., 2003) and atmo-

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spheric trace metal deposition (Zhu et al., 2001) have also become of increasing concern. Therefore, it is urgent to call for correct and reasonable measures to evaluate the impacts of environmental pollution and to reflect upon human activities and their threats on the natural environment.

In recent decades, the traditional methods of air pollution emission inventory and monitoring of concentrations of air pollutants have been constantly developed and improved at both local and national levels, and a large number of data has been collected for environmental quality evaluation. However, these monitoring data have failed to reflect the impact of air pollution on biota. Regionally, long-term and continuous information on air pollution is still insufficient. In the Pearl River Delta of China, several studies were conducted to show possible responses of Masson pine (*Pinus massoniana*) to air pollution and acid precipitation, as for example, the relationship between acid rain and concentrations of metal elements in xylem wood (Hou et al., 2002), the changes of ABA content (Ren et al., 2006) and photo-pigment contents (Yu et al., 2005) in the damaged needles, the alterations of bark pH and conductivity (Kuang et al., 2006), the nutrient loss and heavy metal accumulation in needles (Kuang et al., 2007a), and the temporal changes of heavy metals in xylem (Kuang et al., 2007b). Nevertheless, changes in S content, the acid pollution indicator, in the rings of Masson pine have not yet been reported. In this study, we determined the temporal changes of sulfur content in the xylem of Masson pine to evaluate the historical changes of sulfur pollution at the two sites (Zhaoqing and Nanhai) during the recent 50–60 years, particularly during the recent three decades when the China′s reformation and open-door policy was implemented. Results from this study can be expected to be strongly comparative and important in tracking the changes in air pollution in the Pearl River Delta, and can also be expected to highlight the potential in forecasting regional environment changes in the near future.

2 Site descriptions and methods

As areas having been severely polluted by acid rain in the Pearl River Delta for a long time, Zhaoqing City and Foshan City were selected for tree sampling. Masson pine trees were collected from forests mixed with broadleaf species at the Zhaoqing Dinghushan Biosphere Reserve (23°10′N, 112°34′E) and at the Foshan Xiqiaoshan Nature Reserve (23°55′N, 112°56′E), respectively. Dinghushan, located in the northwest edge of the Pearl River Delta economic zone, mainly received atmospheric pollutants transported via the southeast summer wind from the economic zone. Xiqiaoshan, located in the Nanhai District of Foshan City, is adversely affected by the industrial emissions from electroplating, aluminum and copper refining factories, and ceramic manufacturing industries, etc. The pollutants from the ever lasting inputs of industrial emissions have been reported to inflict significant damage on local forests (Wen et al., 2006).

Ten pine trees around 50 years old were randomly selected from the two sites, respectively, at the end of 2000 for dendrochronological study. The sample trees were healthy-looking and open-grown without suppression by shading. To minimize the impact of anthropogenic effect, all trees were selected at least 100 m away from the edge of forests and at least 50 m away from each other at similar locations and elevations. The selected trees were wholly harvested, and discs of about 30 cm thick were cut off from the base of each tree. The discs were then labeled as to the site, date and the environmental conditions, and brought to the laboratory for dendrochronological measurements. In the laboratory, all the discs were air-dried and polished using a sand-papering machine fitted up with a silicium-carbide band. Annual growth rings of each disc were dated with the WinDendro system (Canada) and ring widths were measured at an accuracy of 0.01 mm.

After the measurements of ring-widths, growth curvatures were fitted for each tree. Three pines from each site with the highest correlation coefficients were chosen for chemical analysis. Sample trees were No.3 (D-3, R^2 = 0.7207), No.7 (D-7, R^2 = 0.7510) and No.11 (D-11, R^2 = 0.7712) at Dinghushan, and No.2 (X-2, R^2 = 0.7308), No.3 (X-3, R^2 = 0.8758), and No.5 (X-5, R^2 = 0.8701) at Xiqiaoshan. Half-decade xylem representing 5-year growth intervals, from the pith to the outer part of each disc, was carefully split out in sequence with an electric micro-chisel. The wood chips were dried at 70°C to constant weight. Wood powder of about 0.080 g was placed into vessels and the S content determined according to the method presented by Yu (1993). Each sample had three replicates, and the xylem S content was averaged from the replicates. The quality of measurements was controlled by the national standard material (poplar leaf, GBV07604-GSV-3).

3 Results and discussion

Mean contents and their standard deviation of S in the xylem formed in different periods at the two sites were presented in Table 1. The xylem S of Masson pine was found to increase over time at both sites. At Xiqiaoshan, the lowest S content in the xylem was 268.81 mg/kg for X-2, formed in the period of 1966–1970, 161.42 mg/kg for X-3, formed in the period of 1966–1970, and 190.61 mg/kg for X-5, formed in the period of 1956–1960. The maximum S content in the xylem was 1521.28 mg/kg for X-2, in the period of 1991–1995 (almost 6 times higher than the content in the period of 1966–1970), 824.35 mg/kg for X-3 in the period of 1991–1995 (5 times higher than the value in the period of 1966–1970), 954.45 mg/kg for X-5 in the period of 1996–2000 (4 times higher than the value in the period of 1956–1960). Comparing the historical changes of S content in xylem, a significantly increased trend was found

Table 1 Content of S ($mg \cdot kg^{-1}$) in the tree rings formed at different periods of *P. massoniana* from Dinghushan and Xiqiaoshan

year	sample trees from Dinghushan (D) and Xiqiaoshan (X)					
	D-3	D-7	D-11	X-2	X-3	X-5
1941–1945			294.87(16.24)			
1946–1950			345.87(88.28)			
1951–1955	256.17(14.97)		244.78(62.20)			
1956–1960	179.24(16.06)	121.74(20.54)	314.41(23.31)	303.78(74.91)	259.51(42.87)	190.61(28.18)
1961–1965	225.04(75.73)	239.14(39.77)	299.50(68.55)	490.53(60.44)	275.89(45.83)	212.16(37.30)
1966–1970	151.89(5.31)	169.85(36.45)	393.61(14.50)	268.81(18.24)	161.42(43.18)	310.24(17.57)
1971–1975	98.30(3.13)	266.26(106.23)	595.82(69.57)	615.55(95.41)	283.79(106.98)	411.43(43.21)
1976–1980	163.33(15.71)	234.13(12.96)	612.16(42.96)	627.45(28.54)	362.97(47.06)	422.60(64.09)
1981–1985	213.30(22.38)	634.17(46.52)	624.32(51.66)	1230.94(379.79)	813.71(23.25)	946.47(8.19)
1986–1990	252.17(40.66)	618.60(52.19)	666.10(9.30)	1243.88(274.30)	813.72(78.41)	924.11(19.34)
1991–1995	344.47(37.14)	636.06(217.93)	636.77(94.50)	1521.28(82.37)	824.35(36.75)	920.30(47.62)
1996–2002	447.32(24.93)	688.30(216.64)	610.81(39.91)	1519.65(273.79)	795.27(53.79)	954.45(27.74)

Note: Values in the bracket are the standard deviations.

in the period of 1976–1980. Before this period, the content of xylem S was at the level of 200–600 mg/kg, while after this period, it was at the level of 600–1500 mg/kg. In the xylem formed after 1975, S contents steadily increased, with a significant transition characteristic (Fig. 1).

At Dinghushan, S contents in the xylem formed in different periods in the three pine trees showed similar patterns with those at Xiqiaoshan. For example, in the tree of D-3, the maximum xylem S content (447.32 mg/kg, appeared in the period of 1996–2000) was five times the minimum value (98.30 mg/kg, appeared in the period of 1971–1975). For D-7, the minimum value (121.74 mg/kg) was found in the xylem formed in the period of 1956–1960, and the maximum value (688.30 mg/kg, 5 times higher than the minimum) was found in the xylem formed in the period of 1996–2000. For D-11, the minimum value was also found in the period of 1951–1956, the maximum value, in the period of 1996–2002. Highest values were generally found in the recently formed xylem (Fig. 1).

The significant increase of S in the rings of Masson pines at Dinghushan and Xiqiaoshan is non-synchronous despite of the S increase over time at both sites. Sulfur contents of the pine trees at Xiqiaoshan had minor fluctuations after an abrupt increase to the level of 1000 mg/kg in the xylem formed after 1980. On the contrary, S contents of the trees at Dinghushan remained at a steady level in the rings formed before 1980 and then increased continuously in the xylem formed after 1980. In the same period after 1980, the increase of xylem S at Dinghushan was smaller than that at Xiqiaoshan. However, in the latest 15 years, the contents of xylem S at both sites were at similar levels. Before 1980, the difference of S content in the xylem formed in the same period was small (at the level of 550–600 mg/kg) between the two sites; after 1980, S content in the xylem formed in the same period at Xiqiaoshan was twice higher than that at Dinghushan.

In order to show the temporal changes more intuitively, S contents in the xylem formed in different periods were standardized. The standardization, which is used to establish the chronology of xylem element contents in

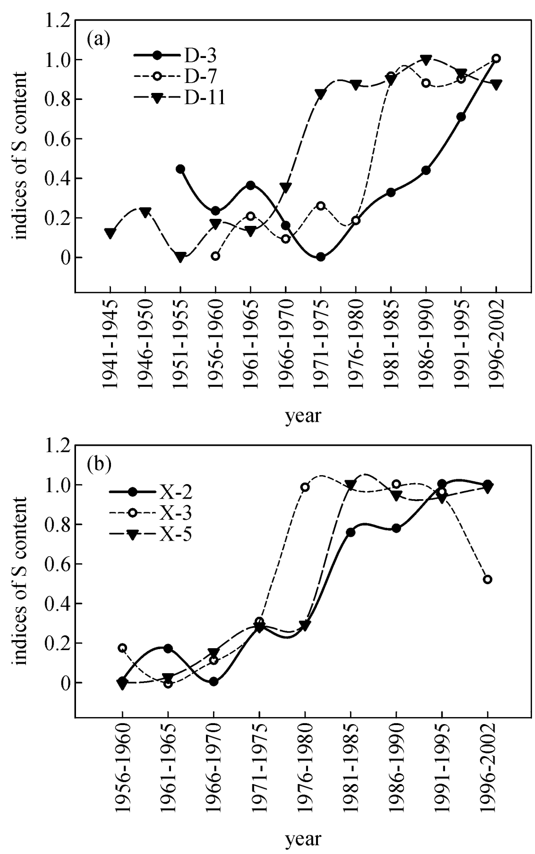

Fig. 1 Chrono-sequences index of S in the rings formed during1941–2000 of *P. massoniana* from Dinghushan (a) and formed during 1956–2002 from Xiqiaoshan (b)

trees of different ages, can make the data from different sources ordered into a uniform time series, avoid the neglect of young trees for different values of elements, while taking account of each tree equally. Thus, different tree-rings can reflect the same environmental information. The method of standardization not only retains the actual concentrations in the xylem formed in different periods, but also removes the difference in concentrations caused by non-environmental factors during the growth process. The standardized chronology will not exaggerate the influence of maximum values and will not reduce the impact of minimum values on the results during the measurements. The standardization and the calculation of chronology were followed as in Yu and Huang (1994):

$$C_i' = (C_i - C)/S_x$$

$$I_i = (C_i' - C_{\min}')/(C_{\max}' - C_{\min}')$$

where C_i' is the standardized concentration of element in the ring formed in year i, C_i is the concentration of element in the ring formed in year i, C is the average value in the rings, S_x is the standard deviation of C, I_i is the index of the xylem element, and $C_{\max}'$ and $C_{\min}'$ are the maximum and minimum values of the element in the tree-rings, respectively. Based on the xylem S contents measured, chronologies of S for each tree were attained (Fig. 1). Finally, the standardized chronologies of xylem S (I) of Masson pine at each site were calculated by using the formula: $I = \frac{1}{n}\sum_{n=1}^{n} I_n + M$, (Fig. 2), where I_n is the standardized chronology of the tree n, n is the number of trees measured, M is the average of the minimum concentration in the indices of all the tree-rings.

The standardized chronologies directly reflect the historical changes of S contents in xylem of Masson pines at the two sites (Fig. 2). For example, significant changes in xylem S content at Dinghushan occurred in 1966–1970; before this period, indices of S fluctuated slightly. Since the period of 1976–1980, the indices climbed rapidly and the upward trend was maintained until the period of 1996–2002. The index of xylem S at Xiqiaoshan increased since the period of 1966–1970 and was followed by a sharp increase later. Before 1975, indices of xylem S at Xiqiaoshan were smaller than those at Dinghushan, implying that the environmental quality at Xiqiaoshan was better than that at Dinghushan. Since the period of 1976–1980, indices of xylem S at Xiqiaoshan were higher than those at Dinghushan, suggesting that serious environmental changes at Xiqiaoshan occurred. Biological characteristics of trees were removed during the standardization, thus, we inferred that the steady increase of S indices might have resulted from the increase in plant available S in the external environment. The index of xylem S was more effective than xylem S contents alone in reflecting the response of trees to environmental changes.

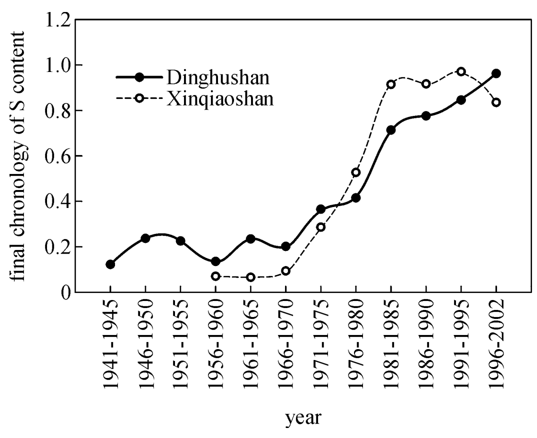

Fig. 2 Comparisons of the chrono-sequences index of S in the same period from Dinghushan and Xiqiaoshan

As is well known, S enters into the terrestrial ecosystem mainly through wet and dry deposition, and returns to the forest ecosystem through liquid, vapor (SO_2) and solid form ($(NH_4)_2SO_4$ or $CaSO_4$) (Cappellato et al., 1998). Dry deposition of sulfur (mainly SO_2) is a dominant component of acid deposition (Freedman, 1995) and is also a major approach of S deposition in many ecosystems (Johnson and Lindberg, 1992). As a necessary element for plant growth, S is mainly absorbed from the soil in the form of SO_4^{2-}, which meets the normal growth of plants. Sulfur content in plant tissues is highly influenced by atmospheric SO_2 concentration and total suspended particulates (Jiang, 1995) and is little influenced by the S content in soil (Chen and Yan, 1987), which makes it possible to monitor S pollution by plant analysis. Content of S in plants has been widely used to monitor air pollution (Jiang, 1994,1995; Nie et al., 2001).

Guangdong was listed as one of the most severely polluted areas in terms of acid deposition by the State Environmental Protection Bureau in 1998. Since China's opening-up and reforming policy was implemented in the late 1970s, Guangdong has experienced massive industrialization and urbanization over the past decades. The total production of industries kept steadily increasing and the total economy stayed at the forefront of the country for many years. As for energy consumption, coal is the dominant energy in Guangdong. The industrial consumption of coal has hugely increased since the economical reformation. The annual consumption of coal and fuel oil in Guangdong Province is shown in Fig. 3 (Bureau of Statistics of Guangdong, 1995–2001). From 1980 to 1997, the annual consumption of coal increased from 10.2 million tons to 36.5 million tons, with an average rate of 7.8 percent, and the annual consumption of fuel oil (mainly heavy oil) increased from 1.87 million tons to 6.23 million tons, with an average rate of 7.3 percent. SO_2 emission from power plants held the biggest proportion with a percentage of 59.19 in Guangdong Province (Xie et al., 1998). Moreover, exhaust from automobiles was also an important source of air pollutants in the Pearl River Delta, because gasoline without Pb had not been widely used till the early 1980s, and diesel with high S content also directly contributed to the rapid increase in atmospheric SO_2 concentration (Huang, 2003).

Foshan is one of the 17 areas in Guangdong Province where acid rain should be urgently controlled. The atmospheric pollutants were predominated by SO_2, dust and NO_x. Investigations showed that the main fuels at the industrial and commercial centers in the urban district were petroleum (including heavy oil and diesel) and coals. A large amount of SO_2 is generated during the process of coal combustion because most of the boilers do not have any desulphurization equipment. It was reported that the total amount of SO_2 being put into the atmosphere in Foshan City and Nanhai District were 59700 t and 129300 t, respectively, during the "Eighth Five-Years" (Guan, 2001). The economic growth and the industrial scale of Foshan District were far above those of Zhaoqing District (Table 2). Emissions of SO_2 in Foshan were 9.78, 10.08 and 10.49 t in 1998, 1999 and 2000, respectively, while the emissions in Zhaoqing were 3.92, 3.32 and 31900 t in the same periods, respectively (Huang, 2003). However, Dinghushan has been adversely affected by the pollutants transported by the southeast wind from the Foshan industrial center in recent years, which was revealed by the increase in xylem S content in the pine trees.

Based on the temporal changes of xylem S and the historical increase of fuel consumption in the Pearl River Delta (Fig. 3), it may be inferred that combustion of coals is one of the important factors contributing to the sustained increase of S content in the xylem of Masson pines. The process of S increase in the xylem of Masson pines at the two sites coincided with the urbanization in the Pearl River Delta (Bureau of Statistics of Guangdong, 1995–2001).

In the present study, we found that the xylem S contents of Masson pine trees were far above those of *Pinus tabulaeformis* growing at Chengde city (44.4–420.7 mg/kg) (Jiang, 1994) and Xiangshan (24.53–1685 mg/kg) (Nie et al., 2001), respectively. The contents were also found to be much higher than those in the xylem of *P. taiwanensis* growing in the Huangshan Mountains in Anhui (0.43–13.5 mg/kg) (Wu et al., 2005) and in the xylem of *P. taeda* growing in Georgia Piedmont in USA (92.5–270.0 mg/kg) (Cappellato et al., 1998). The comparison of S contents in the xylem of trees growing at different sites implies that air pollution in the Delta might be more serious than in other areas.

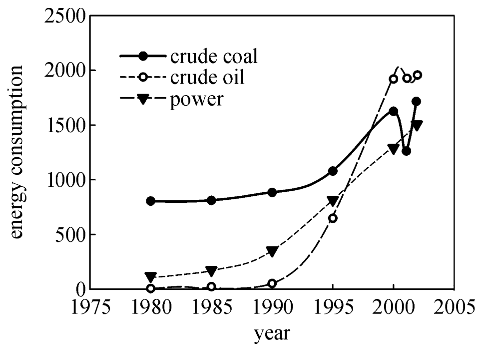

Fig. 3 Amount of energy consumption in Guangdong Province during 1980–2002. The units are ten thousands tons and a hundred million kW·h B for the coal, oil and power, respectively.

4 Conclusions

Based on the analysis of the indices of S content in the xylem of Masson pines at the two sites, we inferred that the period of 1976–1980 is a "threshold" period, since which the environment began to deteriorate. Coupled with temporal changes in xylem S content and certain social-economic indices after the 1980s in the Delta, the history of atmospheric pollution at the studied sites could be reconstructed as follows: 1) before 1970, a period in which the air was relatively clear, 2) during 1971–1985, a period in which the air was gradually polluted, and 3) during 1986–2002, a period in which the air was most severely polluted in the Delta. Temporal changes of the S chronologies in the tree-rings of Masson pines were consistent with the increase of fuel consumption in the past 20 years in the Delta. Our results confirmed that changes of S contents in the tree-rings of Masson pines could be used as an indicator of SO_2 pollution in the environment. The analysis of S in the tree-rings of Masson pines is one of the effective and prospective approaches to monitor the long-term changes in regional air pollution.

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Table 2 Comparisons of the amount of industrial fuel consumption and of atmospheric emission between Foshan and Zhaoqing cities in 1995

	coal consumption/10^8 kg	oil consumption/10^8 kg	atmospheric emissions/10^8 kg		
			SO_2	NO_x	dust
Foshan	316.40	232.84	10.28	5.04	3.77
Zhaoqing	97.37	7.77	1.31	0.97	2.12

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