Isotope and lignin signatures in tropical peat core (Rawa Danau, Indonesia): An approach to reconstruct past vegetation and climate changes

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ABSTRACT Peat samples from a 3.60 m core collected from Rawa Danau, West Java, Indonesia, were studied to reconstruct past vegetation and climate changes. The study carried out combining visual observations, elemental analyses, stable carbon and nitrogen isotope analyses as well as CuO oxidation of lignin residues. Variations of total organic carbon (TOC: 55.53–3.18 %, mean 23.31 %), total nitrogen (TN: 2.52–0.06 %, mean 0.94 %) and water content (WC: 46.8–93.2 %, mean 79.0 %) with core lithology reflected dry and wet cycles. Vertical profiles for TOC, TN, WC and core lithology also had signatures of heavy precipitations and consequent land sliding at the abrupt change of climate (wet cycles). Variations of $(C/N)_a$ (15–93, mean 27) and $\delta^{13}C$ (–27.0 to –30.1 ‰, mean –28.5 ‰) of organic matter (OM) in the peat core indicated that the organic material was comprised of terrestrial C3 plant material. The direct input of aquatic plant matter was small or overwhelmed by high terrestrial input, although changes in lake productivity due to decreasing/increasing nutrient availability were reflected in $\delta^{15}N$ profile. At low elevations in the tropical area, a large shift in vegetation zones was not likely observed. Most species might persist at the site when small-scale localized environmental changes and human disturbances or natural forest fires occurred, but the abundances of particular species might change, depending on the natural conditions in which a species could grow. However, vegetation changes associated with periodical sequence of dry and wet microenvironment were inferred from the total lignin phenol (TLP) profile.

Key words: isotope, lignin, peat, tropical, wetland, vegetation and environment

INTRODUCTION

Histosole (peat soil) contains a considerable amount of organic matter (OM) and provides a variety of indicators that can be used to reconstruct the past depositional environment and paleoclimate (Cerling et al. 1989; Aucour et al. 1999; Müller and Voss, 1999; Choi, 2001). The types of vegetation that existed at different times in the past can also be estimated from compositions of OM in different age's peat layers. Tropical lowland peat deposits are often dominated by trees and shrubs (Phillips and Bustin, 1998; Wüst and Bustin, 1999). Peats from Malaysia, Indonesia, Irian Jaya and Thailand have large amounts of woody material and amorphous matrix (Wüst et al. 2001). Tropical peats are therefore rich in lignin with significant amounts of cellulose, hemicelluloses, protein and water-soluble compounds which are partially lost during early microbial activities (Orem et al. 1996; Kuder and Krüge, 1998).

A wealth of environmental and ecological information preserved in peat can often be obtained from stable isotope analyses (Rundel et al. 1989; Bowen, 1991). The natural variations in stable isotope ratios of both carbon ($\delta^{13}C$) and nitrogen ($\delta^{15}N$) are extremely useful in tracing and quantifying sources, sinks and flux within the biogeochemical cycles (Filley et al. 2001).

In the present study, an attempt has been made to reconstruct the past vegetation and climate changes in tropical area, Rawa Danau, West Java, Indonesia, using $\delta^{13}C$, $\delta^{15}N$, atomic $(C/N)_a$ ratios of OM together with lignin phenol composition of a peat core. The principals behind using these parameters for this purpose are; (1) significant differences of $\delta^{13}C$ values in different plant types depend on photosynthetic pathway [C3 plant: –23 ‰ to –34 ‰ (–27 ‰) C4 plant: –9 ‰ to –17 ‰ (–13 ‰) and CAM plant: –10 ‰ to –30 ‰; Deines, 1980; O' Leary, 1988; Farquhar et al. 1989], (2) source plant can be

identified by lignin phenol compositions (gymnosperm plant: vanillyl phenols, angiosperm plants: vanillyl and syringyl phenols, nonwoody plant: vanillyl, syringyl and cinnamyl phenols; Hedges and Mann, 1979; Orem et al. 1997) as well as lignin enrichment of peat sample, and (3) the isotope and lignin composition of the OM in peat is similar to the isotope and lignin composition of the vegetation cover from which it derived.

MATERIALS AND METHODS

Study Area

The Rawa Danau lies in an ancient caldera of Danau volcanic complex on the north-western tip of West Java, Indonesia, at an elevation of around 100 m above sea level (asl), at 6° 11' S and 105° 59' E (Fig. 1). The catchments include the steep hills and mountains forming the wall of the caldera. The surrounding caldera rim is at an altitude about 140 m asl, and the adjacent high volcanic terrain is mostly between 400–700 m asl. Two hundred years ago, there was a substantial lake in the caldera and it does not exist at present as its original form (Endert, 1932). Rawa Danau occupies a few km² and the deepest part is the crater bottom which is largely occupied by swamp. A survey of the swamp deposit by Indonesia Geological Survey (IGS) shows that deeper basin deposits (> 6 m depth) consist of fine-grains (organic silt-clay) with minor intercalations of peat and fluvial sands (Rimbaman, 1994). Peat deposits prevail in the central swamp area with sequences up to 4–5 m thickness just below the surface.

Climate of the Java Island in Indonesia is driven

by the Asian monsoon system. Van Steenis (1965) documented a pronounced floristic gradient across the island correlated with the change in climate. The main vegetation types of Rawa Danau are mixed forest swamp, *Ficus retusa* swamp forest, open herbaceous swamp and dry land largely deciduous (Stuijts, 1993; Kaars et al., 2001; references therein). The swamp vegetation in the northern part is partially replaced by rice field, but the signature of original swamp forest is still common at the edge of remaining fresh water swamp.

Sampling

After analyzing three test cores from Rawa Danau, the main core (RD-1), 360 cm deep and 10 cm diameter, was collected by piston coring in September 2001. Since piston coring disturbed upper portion of the main core, a gravity sub-core of 32 cm was collected to maintain sedimentary sequence. The peat cores were divided into slice approx. 2 cm thick, sealed in polyethylene bag and transported to laboratory. The material was freeze-dried, homogenized with mortar and pestle, passed through a 300 µm sieve and finally preserved for subsequent analysis.

Elemental and Isotopic Analyses

The water content (WC) was determined gravimetrically using weight differences between wet and dried samples and expressed in %. Weight percentage of total organic carbon (TOC) and total nitrogen (TN) were measured by Thermo Quest elemental analyzer of model NA2500 NCS. Samples were combusted at 1000°C to CO₂ and NO_x gas and a subsequent Cu-packed column reduced

Fig. 1. Map of the coring site. (after Tareq et al. 2004)

NO_x to N₂ gas at 750°C. Alanine was used as a standard for TOC and TN determination. A few selected samples run several times for estimating the reproducibility of the measurements and found that the deviation is less than $\pm 5\%$ for both TOC and TN.

Stable carbon and nitrogen isotope compositions were determined by the method involving automatic combustion in a elemental analyzer (Thermo Quest NA 2500 NCS) interfaced via a Finnigan CONFLOW II system to a Finnigan MAT 252 mass spectrometer (Finnigan, 1995). Half the samples were analyzed at least in duplicate with a maximum difference of $\pm 0.2\%$ between replicates. The performance of the automatic EA-CONFLOW II - MS system was evaluated by running the working standard alanine samples of known $\delta^{13}\text{C}$ (-21.56%) and $\delta^{15}\text{N}$ (-5.25%) values and the accepted values were obtained for both of carbon ($\pm 0.07\%$) and nitrogen ($\pm 0.21\%$) within samples size ranges 7–100 μg carbon and 100–300 μg nitrogen for the measurements.

Lignin Phenol Analysis

About 30 mg of bulk lipid free peat was oxidized with CuO at 170°C for 3 hours in 2N NaOH containing ferrous ammonium sulfate $[\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}]$ (Hedges and Ertel, 1982). After adding known amount of ethylvanillin as internal standards and adjusting the pH to 1 by adding 6N HCl, lignin phenols were extracted with peroxide-free diethylether. The quantification of lignin phenol monomers were performed by GC (Shimadzu 14B) equipped with DB-1 (60 m $\times$ 0.25 mm, i.d.). Details of the procedures were described elsewhere (Tareq et al.

2004).

RESULTS AND DISCUSSION

Core Lithology and Sedimentation Rate

The core (RD-1) of 360 cm in total length collected by 380 cm core tube insertion indicated only less than 5% compaction during coring operation. The upper 50 cm of the core was a mixture of grass-woody peat and partially decayed plant tissues except for the depths of 8–12 cm that were enriched by silty-sand-clay. Visual inspection during sample processing indicated that rest of the core (50–360 cm) was mainly composed of woody peat. A few numbers of silty-sand-clay enriched layers were detected at different depths (9, 115, 175, 225, and 280 cm). Perhaps, these silty-sand-clay layers are the signature of upper mountainous erosion due to heavy precipitation or changing water level of the lake. No decreasing trend with depth was observed in water content (WC) profile, rather it was correlated with core lithology, TOC and TN as shown in Fig. 2. It was also clear from the WC profile that compaction effect was not pronounced during sedimentation.

Nine conventional radiocarbon (^{14}C) dates on bulk sediment are available for the core RD-1 sequence. The ^{14}C analysis was carried out by Dr. Darwin Siregar (personal communication) in the Geological Research Development Center, Indonesia, according to the acetylene counting method using a gas proportional counter. We calibrated the ^{14}C dates by CALIB rev. 4.3 (Stuiver et al., 1998), and the results were listed in Table 1. The organic matter preserved at the bottom section of

Table 1. Calibrated radiocarbon ages* of the core RD-1.

Sample code	Depth (cm)	Uncalibrated ^{14}C age $\pm$ SD (BP)	Cal. Age in BP		Cal. Age in AD/BC	
			(1 σ)	(2 σ)	(1 σ)	(2 σ)
RDT2	22	410 $\pm$ 240	423 (0.78)	353	AD 1527 (0.78)	AD 1600
RDT3	55.5	880 $\pm$ 220	820 (0.94)	858 (0.97)	AD 1130 (0.94)	AD 1098 (0.97)
RDT4	66.9	1040 $\pm$ 280	954 (0.93)	974 (0.98)	AD 996 (0.93)	AD 976 (0.98)
RDT5	99	1400 $\pm$ 260	1305 (0.95)	1310	AD 645 (0.95)	AD 640
RDT6	123.2	1600 $\pm$ 290	1547 (0.97)	1548	AD 404 (0.97)	AD 402
RDT7	137.6	1740 $\pm$ 350	1672	1704	AD 278	AD 246
RDB1	175.3	3030 $\pm$ 260	3169	3287 (0.98)	BC 1220	BC 1338 (0.98)
RDB2	188.3	3500 $\pm$ 360	3813 (0.98)	3800 (0.98)	BC 1861 (0.98)	BC 1851 (0.98)
RDB3	207.7	4020 $\pm$ 360	4429 (0.96)	4455 (0.97)	BC 2480 (0.96)	BC 2506 (0.97)
Bottom	360	–	7428**	7428	BC 5479	BC 5479

*Ages are calibrated in both years B. P. and A.D./B.C., and expressed at one sigma (68.3 % confidence) and two sigma (95.4 % confidence) ranges. Where multiple intersections of the calibration curve occur, the largest relative area of the probability distribution in each case was chosen to represent the most probable age range (probability values shown in brackets and *italicized*). Age values are median age of the two sigma calibrated ranges.

**Age is estimated from the depth-age relationship found for upper sediment layers, assuming a constant accumulation rate.

the core represented Holocene materials (7.4 ka BP). The low accumulation rate observed after 1.7 ka BP made the interpolated ages somewhat uncertain in comparison to the much better resolution of the overlying sections.

Distribution of Organic Carbon and Nitrogen

The TOC content remained high throughout the all depths with some sharp peaks at different depths as usually observed for the accumulation of OM from terrestrial sources (Aucour et al. 1999; Bourdon et al. 2000, references therein). A vertical profile of TOC showed variation between 3.2 and 55.5 % with an average

Fig. 2. Vertical distributions of total organic carbon (TOC), total nitrogen (TN) and water content (WC) with calibrated ^{14}C -age. Shaded area indicates different hydrological regimes.

Fig. 3. Correlation between TOC and TN of the RD-1 core.

of 23.3 %.

The vertical distribution of total nitrogen (TN) with depth of peat core is shown in Fig. 2. The TN ranged from 0.06 to 2.52 % with an average value 0.94 % for overall the depths. In relation to TOC content, TN also showed peaks at the same depths. There was a significant correlation (Fig. 3; $r^2 = 0.66$) between TOC and TN which could be attributed to the possible unique source signature overall the accumulation period. Both TOC and TN contents were higher in woody peat layer than the clay-silt-sand enriched layers.

The atomic ratios of carbon to nitrogen (C/N_a) varied frequently (15–93) with an average value 27. The $(C/N)_a$ ratio is widely used to identify the source of OM (Meyers, 1994; Herczeg et al., 2001). Vascular land plant has C/N ratios higher than 20 ($C/N > 20$) due to comparatively high cellulose content whereas non-vascular sources like algae and phytoplankton have C/N ratios from 4 to 10 (Meyers, 1997). Most of the layers of core RD-1 had $(C/N)_a > 20$ indicating vascular plant sources, but few layers at different depths had $(C/N)_a$ 20–10 due to complex heterogeneous contributions of the terrestrial plant sources and in-situ developed aquatic species such as algae, phytoplankton, and other protein enriched sources under an optimized environmental condition.

The $\delta^{13}C$, $\delta^{15}N$ and $(C/N)_a$ values for the whole core are plotted versus depth in Fig. 4 along with calibrated time scale for the 7.4 ka BP (^{14}C age). The $\delta^{13}C$ displayed a small range of fluctuation between -27.0 and -30.1 ‰. This shift apparently should be caused at least by

selective loss of the isotopically heavy carbohydrate fraction (compare to lignin) of total OM (Spiker and Hatcher, 1984). Diagenetic effect usually induces minor $\delta^{13}C$ depletion in buried OM (>2 ‰; Macko et al., 1991; Meyers, 1994) and can be considered as a secondary factor for the observed variability.

The average values of $\delta^{13}C$ (-27.2 ‰) and C/N ratio (27) is a signature of the dominance of C_3 terrestrial plants though the isotopic signature of C_3 land plants can not be distinguished from that of fresh water algae (Meyers, 1994). The natural difference in stable carbon isotope ratio of different plants provides fine resolution spectrum of vegetation succession in tropical wetland (Chmura et al. 1987; DeLaune, 1986) and has the potential to provide valuable evidence of changing hydrological regime and climate. In Rawa Danau, the vegetation was not completely altered during peat development, but only relative abundances of different plant species (angiosperm, gymnosperm, nonwoody plants) changed as was shown by lignin phenol compositions (Tareq et al. 2004).

The irregular changes of $\delta^{13}C$ values at different depths were more likely due to isotopic fractionation in changing relevant climatic parameters including precipitation and air temperature. Stewart et al. (1995) suggested a co-efficient for eastern Australian climate covering a large range of precipitation (350–1500 mm) and open vegetation. The variation of $\delta^{13}C$ in the peat core reflects the changes water availability results in a complex balance among precipitation, annual distribution,

Fig. 4. Vertical profiles of $(C/N)_a$, $\delta^{13}C$, $\delta^{15}N$ with calibrated radiocarbon age.

an important role on shifting the $\delta^{15}\text{N}$ values. The chronological information derived from ^{14}C indicated that the enrichment of $\delta^{15}\text{N}$ in the peat core began in mid Holocene when the lake became shallow (From diatom records; Kaars et al. 2001) and $\delta^{15}\text{N}$ was depleted again from the late Holocene. Vertical profile of $\delta^{15}\text{N}$ should reflect changing in aquatic productivity of the wetland.

Lignin Signature

A representative gas chromatogram of the CuO oxidation products of Rawa Danau peat shows in Fig. 5. Eleven lignin phenol monomers (p-hydroxybenzoic, vanillyl, syringyl and cinnamyl phenols) were determined among CuO oxidation products of 24 pre-selected samples. Vanillic phenols are derived from the coniferyl lignin and both vanillic and syringic phenols are from sinapyl moieties of lignin. They consist of aldehydes, ketones and acids. The p-coumaric and ferulic acids (cinnamic unit) linked to cellulose by ester bonds are generated from the cinnamyl alcohol-derived moieties. p-Hydroxybenzoic phenols were also determined, but this group can be partly derived from other sources than lignin (Hedges and Parker, 1976; Hedges and Ertel, 1982). Total lignin phenol (TLP) in mg/g dry peat is calculated as the sum of the four groups (p-hydroxybenzoic, vanillyl, syringyl and cinnamyl phenols).

Total lignin phenol (TLP) concentrations of peat core (RD-1) from Rawa Danau were generally higher than those from other sediments and more comparable to those observed in other tropical peat land (Bourdon et al., 2000). The high concentrations of TLP reflect high vascular plant contribution to the wetland in Rawa Danau as suggested by the high atomic (C/N)_a ratio.

The vertical distribution of TLP of the core RD-1 (Fig. 6) showed increasing vascular plant component in OM at the calibrated age of 1, 1.5, 3.5, 4.5 and 7.4 ka BP. However, this pattern can be influenced by rapid sedimentation and selective degradation of lignin under oxic conditions. It can be inferred from alternative variations of TLP that the terrestrial vegetation of Rawa Danau strongly influenced by the hydrological changed as indicated in Fig. 2. The detail of the vegetation change history is discussed elsewhere (Tareq et al. 2004) depending on composition of lignin monomers and newly defined lignin phenol vegetation index (LPVI).

We also analyzed CuO oxidation lignin monomers of the four most dominated plant species (*Polypodiaceae*, *Cyperaceae*, *Pandanus* and *Gramina*) around the coring site. The materials from the living plants showed high lignin contents comparable to the value previously obtained for tropical nonwoody plants (Bourdon et al., 2000). The cinnamic group composed of both p-coumaric and ferulic acid dominated (Table 2). By comparison with fresh plants, small fraction of cinnamic phenols was found in the surface of the wetland (Table 2) and the amount of lignin substantially decreased, indicating post-depositional diagenetic loss of cinnamic group is higher than that of syringic and vanillic groups. The loss of cinnamyl phenols can be attributed to early diagenetic nature of cellulose ester bonds and thus C/V ratio can be used as indicator of degree of early diagenesis of OM in wetland.

A comparison of average values of lignin phenol parameters between fresh plants and the surface peat is illustrated in Fig. 7. In every phenol family, peat yielded less aldehyde and proportionately more acid than fresh plant tissues did. These compositional differences are

Table 2. Lignin phenol composition (mg/g of dry samples) of the plant samples collected from coring site and relevant lignin parameters with mean values of surface peat.

Name of species	% OC	p-Hg	Vg	Sg	Cg	S/V	C/V	p-H/V	(Ad/Al) _v	(Ad/Al) _s	(Ad/Al) _{pH}	TLP	λ_s
<i>Polypodiaceae</i>	41.1	8.2	11.3	12.7	6.5	1.12	0.58	0.73	0.27	0.25	0.22	38.7	7.4
<i>Pandanus</i>	43.3	17.3	7.2	10.1	6.0	1.4	0.83	2.4	0.26	0.23	0.25	40.6	5.4
<i>Cyperaceae</i>	42.1	12.2	17.0	20.0	10.6	1.2	0.62	0.72	0.20	0.25	0.37	59.8	11.3
<i>Gramina</i>	41.5	10.5	10.3	18.2	8.6	1.8	0.83	1.02	0.26	0.20	0.38	47.6	9.0
Average (plants)	42.0	12.5	11.5	15.3	7.9	1.4	0.72	1.22	0.25	0.23	0.30	46.7	8.3
± SD	± 0.84	± 3.35	± 3.54	± 4.01	± 1.83	± 0.25	± 0.12	± 0.70	± 0.03	± 0.02	± 0.07	± 8.27	± 2.2
Average (surface peat)	26.4	11.3	16.3	15.8	2.1	1.03	0.13	0.73	0.52	1.28	0.37	45.5	14.0
± SD	± 7.73	± 1.70	± 4.73	± 1.72	± 0.42	± 0.24	± 0.03	± 0.15	± 0.18	± 0.40	± 0.15	± 8.11	± 4.6

Abbreviations: OC: organic carbon, p-Hg: p-hydroxybenzoic group, Vg: vanillyl group, Sg: syringyl group, Cg: cinnamyl group, (Ad/Al): acid/ aldehyde, TLP: total lignin phenols in mg/ g dry peat, λ_s (lamda): sum of vanillyl, syringyl and cinnamyl phenols per 100 mg organic carbon

Fig. 7. Bar chart of the mean of the lignin parameters of four living plants and surface peat of Rawa Danau (Vertical bars represents $\pm$ SD).

sensitively reflecting the post depositional oxidative degradation of lignin. Comparison of lignin phenol ratio parameters (S/V, C/V and p-H/V) between fresh plants and surface peat concurred with the results of earlier discussion on the rate of post-depositional diagenetic loss (cinnamic > syringic > vanillic). Average values of TLP and λ_s (Σ vanillic, syringic, and cinnamic in mg/100 mg OC) of peat are higher than those of fresh plants due to early diagenetic loss of non-lignin components of OM in the tropical wetland.

Human Dimension and Environment

Vegetation in a landscape can be disturbed by both of human activities and environmental changes. At a glance, the vegetation in Rawa Danau is apparently remained constant during Holocene, in which relative abundances of different species are established as a function of local topography, hydrology and climate. Anthropogenic disturbances are dominated by human social and economic processes and are more variable than natural disturbances. Charcoal is commonly present in tropical rainforest soils (Sanford et al. 1985; Saldarriaga and West, 1986). However, the implications of these data are unclear because we do not know whether the charcoal was produced by natural forest fires or by human activities.

People clearly had a large impact on the vegetation development in Rawa Danau area during the last

few hundred years as evidenced by increased burning, the appearance of food crops, the apparent opening of the vegetation with many grasses, sedges and ferns and presence of weeds (Kaars et al. 2001). Once the vegetation is disturbed by human impact and/or natural forest fire, the original forest is usually replaced by grasses and sedges with occasional shrub or trees. The pollen data suggested that the vegetation of Rawa Danau wetland dominated by angiosperms, and few sections have signatures of relatively high abundance of nonwoody plants (*e.g.*, grasses), especially at 0–50 cm and 150–200 cm depths (Yulianto et al. 2005). However, lignin phenols compositions reflected the presence of gymnosperm (S/V < 0.60) during forest fire events (Tareq et al. 2004). The gymnosperm lignin signature would most probably be attributed to the expansion of nonwoody gymnosperm

plants, *e.g.*, ferns which might predominate over grasses during vegetation succession in tropical wetlands, although Yulianto et al. (2005) did not describe in detail the distribution of fern spores.

It is quite difficult to distinguish between the human dimension and climate forcing on the vegetation changes at mid Holocene time (during 3.5–7.4 ka BP). Large scale vegetation changes/intensive forest fire occurred during 3.8–4.5 ka BP, but our data were unable to identify whether that changes caused by human dimension (through forest clearance or burning) or natural climatic changes (*i.e.*, drought). But it is clear from our records that the recent vegetation changes/forest fires were caused by human activities that enhanced the periodical ENSO events.

CONCLUSIONS

Variations of (C/N)_o, $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ of OM in the peat core indicated that the organic material was comprised of terrestrial C3 plant material, and the direct input of aquatic plant matter was small or overwhelmed by high terrestrial input. Excursions in the isotopic abundances of 5 ‰ in $\delta^{15}\text{N}$ and –3.1 ‰ in $\delta^{13}\text{C}$ are not artifacts of diagenetic processes but were caused by changes in sources of organic matter. Although, it was difficult from small scale excursion of stable isotope values to

identify paleovegetation and environmental changes, lignin phenols together with other geochemical data (like elemental carbon, TOC, $\delta^{13}\text{C}$) were able to indicate small scale shift of terrestrial vegetations. The TLP concentrations of core RD-1 varied with depth due to the changes in composition of terrestrial plant of Rawa Danau in response to climate changes.

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