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# Variations in the rate of accumulation and chemical structure of soil organic matter in a coastal peatland in Sarawak, Malaysia

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## ABSTRACT

To determine the variation in the rate of accumulation and chemical structure of soil organic matter (SOM) in a tropical coastal peatland, series of soil cores samples were collected in the Maludam National Park, Sarawak, Malaysia and analyzed. Duplicate soil core samples were collected from three phasic communities in the peat swamp forests, Mixed Peat Swamp (MPS; depth of 50-450 cm), Alan Batu (ABt; depth of 200-700 cm), and Alan Bunga (ABg; depth of 200-800 cm), which were located at outer, middle, and inner sites on the peat dome. The <sup>14</sup>C age of the SOM was determined at depths of every 50 or 100 cm and 4-6 samples from each profile were subjected to ramp cross polarization/magic angle spinning <sup>13</sup>C nuclear magnetic resonance (NMR) analysis. The <sup>14</sup>C age of SOM ranged from 1602 to 5162 years before the present (yBP), 1134–4043 yBP, and 928–3376 yBP in the cases of the MPS, ABt, and ABg forests, respectively. Those after calibration were in the ranges of 1482–5958 (MPS), 1322-4603 (ABt), and 837-3629 (ABg) yBP. The relationship between calibrated <sup>14</sup>C age and soil depth, when regressed to a linear function with the constraint that the age of surface sediment is 0 year, showed that the rate of peat accumulation was 0.60  $\pm$  0.01 (MPS), 1.5  $\pm$  0.0 (ABt), and 1.9  $\pm$  0.1 (ABg) mm y<sup>-1</sup>. However, in the case of the MPS forest, this rate was not constant but decreased toward the present. The <sup>13</sup>C NMR results showed the decrease in the relative abundance of alkyl C and an increase in that of carbohydrate C with soil age/ depth in the MPS forest soil profiles, suggesting an acceleration in the decomposition of SOM as the cause of the decrease in the rate of accumulation of the peat. Variations in the C composition of the other two forest soils with soil age were small. The relative content of aromatic C remained relatively constant, indicating that aromatic C is consistently a major component of the C pool throughout the peat soil layers.

#### 1. Introduction

Tropical peatland covers 441,025 km<sup>2</sup>, of which 247,778 km<sup>2</sup> is distributed in Southeast Asia (Page et al., 2011). Total peatland area in Malaysia is 27,000 km<sup>2</sup> and 17,000 km<sup>2</sup> is located in Sarawak (Mutalib et al., 1992). It is estimated that the total amount of peat carbon (C) in Southeast Asia and Malaysia are 68.5 and 8.9 Gt C, respectively (Page et al., 2011). This vast accumulation of soil organic C is attributed to the high productivity of tropical peat swamp forests and the slow rate of SOM decomposition under saturated conditions (Chimner and Ewel, 2004). The decomposition of SOM in tropical peatland is also impeded by high acidity and poor nutrient status (Dommain et al., 2011). However, a peatland ecosystem can turn from a C sink to a C source

depending on the land use and management (Inubushi et al., 1998; Melling et al., 2005; Hooijer et al., 2012; Dommain et al., 2018), in which environmental conditions, such as temperature, groundwater table, pH, and nutrient contents, likely play key roles in regulating the rate of SOC decomposition (Jungkunst and Fiedler, 2007).

The amount and composition of organic matter supplied by vegetation and the catenary sequence of phasic communities from the edge to the peat dome center are another important factor that determines the C stock in tropical peatland (Belyea and Malmer, 2004; Heller and Zeitz, 2012; Dommain et al., 2015). Hodgkins et al. (2018) showed that the relative abundance of aromatic components in peat was larger in low latitudes than high latitudes, which was postulated to be due to the difference in chemical composition of parent plants, e.g., lignin content,

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Fig. 1. Map of Borneo Island and Maludam National Park (Modified from Sangok et al., 2017).

with rapid loss of carbohydrates and progression of humification under warm climate. To understand the stability of the SOM that accumulates in tropical peatland, information about the variations in chemical structures of SOM derived from different vegetation communities is required.

The history of peat accumulation in tropical peatlands is reflected in the <sup>14</sup>C age profile of SOM (Dommain et al., 2015). In the inland area of west Kalimantan and central and east Kalimantan, Indonesia, peatlands have been present since approximately 30,000 calibrated years before the present (cal yBP; Anshari et al., 2004) and since 15,000–6300 cal yBP (Hope et al., 2005; Wüst et al., 2008), respectively, while coastal peatlands in Indonesia and Malaysia started to form after 7300 cal yBP (Staub and Esterle, 1994; Dommain et al., 2011). According to a review by Dommain et al. (2011), the rate of peat accumulation also varies between inland and coastal peatlands in Malaysia and Indonesia, for which inland peatlands show a lower rate of accumulation, because of the lower precipitation and increased El Niño intensity in inland areas over the past millennia and their sensitivity to late Holocene sea-level fall.

Since the 1980s, development of various methods for modeling peat accumulation has advanced our understanding of the growth of peat (Ingram, 1982; Clymo, 1984; Winston, 1994; Yu et al., 2001a, 2001b). Clymo (1984) proposed the use of a two component two-layer model, in which the growth rate of a peat bog was estimated separately for acrotelm, the surface thin oxic layer, and catotelm, the thick anoxic layer beneath the acrotelm (Ingram, 1978). His model was modified by Yu et al. (2001b) to accommodate boreal continental fens. The extended model produced a convex pattern of peat growth, suggesting either a continuous decrease in the rate of formation of peat or a continuous increase in decomposition rate. The other models (Ingram, 1982; Winston, 1994; Cobb et al., 2017) also suggest the importance of hydrological factors, such as groundwater table, recharge rate, and hydraulic conductivity, for the rate of accumulation of peat or C storage capacity of peatlands including those in the tropics. Hydrological conditions may also be associated with the type of vegetation, the source of peat SOM. Recently, Kurnianto et al. (2015) modified the Holocene

Peat Model, a one-dimensional and annual time step model for estimating the C cycle in northern temperate peatlands (Frolking et al., 2010), to estimate C accumulation in tropical peatlands. In their model, peat mass balance between vegetation inputs and decomposition is obtained on monthly scale and separately for leaves, woods and roots. This was due to their significant variations among plant parts. Litter C ratio among plant parts may vary depending on vegetation types and affect the rate of peat accumulation, which has not been extensively studied.

Solid state <sup>13</sup>C NMR spectroscopy has been applied to investigate the composition of SOC in peat soils with differences in particle size, soil depth, and land use type (Nordén et al., 1992; Krull et al., 2004; Grover and Baldock, 2012; Tfaily et al., 2014). Baldock et al. (1997) reviewed <sup>13</sup>C NMR data in the literature and proposed that the increase in the alkyl C to *O*-alkyl C ratio represents the extent of decomposition of peat. Normand et al. (2017) investigated the influence of the type of vegetation on the SOC composition of peat soils in Sweden and suggested that the Sphagnum and fen soil profiles with a higher *O*-alkyl C content decompose more readily than the peat soils at palsa sites that contain high levels of alkyl and aromatic C. Sangok et al. (2017) also suggested that SOC composition, as estimated by <sup>13</sup>C NMR, affects the rate of SOC decomposition in a 3-year field incubation that was conducted in an oil palm plantation using 3 soil samples from different phasic communities of tropical peat swamp forest.

The objective of the present study was to elucidate the variations in the chemical structure of SOM in tropical peat soil in relation to the rate of peat accumulation. For this purpose, peat soil core samples were collected from several depths in 3 major phasic communities of tropical peat swamp forests and their <sup>14</sup>C ages were determined. Chemical structure of SOM in those samples were analyzed using <sup>13</sup>C NMR spectroscopy.

#### 2. Materials and methods

#### 2.1. Location of soil sampling site

Soil samples (*Typic Haplofibrists* according to the USDA soil classification system; Soil Survey Staff, 2014) were collected in the Maludam National Park, northeast Sarawak, Malaysia in April 2013 (Fig. 1). The Maludam National Park is a domed peatland and contains ca 430 km<sup>2</sup> of a completely protected peatland forest (Melling, 2016). The principal tree species include Ramin (*Gonystylus bancanus*), Alan (*Shorea albida*), Jongkong (*Dactylocladus stenostachys*), and Sepetir paya (*Copaifera pallutris*) (Anderson, 1961). According to Anderson (1961), the Maludam National Park falls into the category of a true peat swamp, which is described as ombrogenous, uninfluenced by river flooding, a pH < 4, and a loss on ignition exceeding 75%. The water table is generally very high throughout the year and the forest conditions are shaded, damp, and humid. The forest floor has thick root mats and leaf litter. Annual mean precipitation and daily mean air temperature are 2770 mm and 26.9 °C, respectively (Sangok et al., 2017).

Duplicate peat soil profile samples were collected from Mixed Peat Swamp (MPS; 1°25'N 111°7'E), Alan Batu (ABt; 1°27'N 111°9'E), and Alan Bunga (ABg; 1°27'N 111°9'E) forests (Fig. 1). These types of forests correspond to phasic communities 1, 2, and 3 in Anderson (1983). The elevation was 3.7-3.8, 7.8-8.1, and 8.7-9.2 m and the distance from Lupar River was ca 1.0-1.1, 4.3-4.5, and 5.0-5.5 km at the MPS, ABt, and ABg forest sites, respectively, with a 500-1000 m distance between each 2 sampling points. Groundwater table during the dry season was -21 cm, -11.0 cm, and -8 cm on average in the MPS, ABt, and ABg forests, respectively, and these values increased to -13 cm, -9 cm, and -7 cm, respectively, in the wet season (Sangok et al., 2017). The average bulk density obtained for 0-150 cm layer soils (Melling, L., unpublished data) was 0.07 (MPS), 0.06 (ABt), and 0.02 (ABg) g cm $^{-3}$ . The smaller bulk density at the ABg forest site was attributed to the existence of abundant, large cavities. In the MPS forest, Gonstylus bancanus, Dactylocladus stenostachys, Copaifera palustris, and 4 species of Shorea make up the major vegetation, while both the ABt and ABg forests are dominated by Shorea albida (Sangok et al., 2017). Transect section through three types of forests in the Maludam peatland is shown in Supplementary Fig. 1.

A peat auger (Model 04.09, Eijkelkamp, Giesbeek, The Netherlands) consisting of an auger head (50 cm length) and metal extensions (100 cm length) was used to obtain the samples. The number of metal extensions was increased until contact was made with the mineral substratum. A 10-cm soil layer was collected every 50 cm (MPS forest) or 100 cm (ABt and ABg forests) depth from the center part of the auger head (10-cm thickness) to avoid contamination during boring. Soil sampling was terminated when mineral soil was found in the auger head. Mineral substratum appeared at a depth of 525 and 545 cm at MPS-1 and MPS-2, respectively. Those for ABt-1 and ABt-2 and for ABg-1 and ABg-2 were 750 and 735 cm and 925 and 840 cm, respectively. Since the 100 cm depth layers in the ABt and ABg forest soil profiles were empty and the surrounding layers were in danger of contamination, eventually soils from 50 to 450 cm depth, 200-700 cm depth, and 200-800 cm depth were collected in the MPS, ABt, and ABg forests, respectively. The soil samples were immediately double wrapped with aluminium foil, stored in a plastic bag, and transported to the laboratory, where the samples were freeze dried. Additional soil samples were collected for use in measuring fundamental properties. The total C and N contents and ash content were determined by dry combustion using a NC analyzer (TruMac CN, Leco, St. Joseph, MI, USA) and a thermogravimetry (TGA 701, Leco, St. Joseph, MI, USA). Soil pH was measured using the suspension of soil with distilled water mixed at 2:5 (w/w; Metrohm 827, Metrohm, Herisau, Switzerland).

## 2.2. Measurement of $^{14}C$ age

Approximately 500 mg freeze-dried soil core samples (10-cm thickness; 39 samples in total) were used for determining the  $^{14}$ C concentration after large plant debris was removed using tweezers and alkali- and acid-soluble materials were removed by the following procedures: The soil samples were allowed to stand overnight in 1 M hydrochloric acid (HCl) and the suspension was then boiled for 3 h. After cooling down, the supernatant was discarded by decantation. This treatment was repeated until no more floating material was observed. Then, 1 M sodium hydroxide (NaOH) was added to the residues and the samples were treated similar to the acid treatment until the supernatant became almost colorless. The residual samples were treated with 1 M HCl again, washed with purified water until the supernatant became neutral, and dried at 80 °C in an oven dryer.

Approximately 9 mg of each dried sample was placed in a quartz tube (ID 6 mm) with 800 mg of copper oxide. To convert all the organic C to carbon dioxide (CO<sub>2</sub>), the tubes were evacuated, sealed, and heated in a muffle furnace at 850 °C for 4 h. The CO<sub>2</sub> was purified in a glass vacuum line system and reduced to graphite by reacting them with reduced iron and hydrogen in an evacuated quartz tube at 650 °C for 6 h. The  ${}^{14}C/{}^{12}C$  and  ${}^{13}C/{}^{12}C$  ratios of the graphite targets were measured using a Tandetron accelerator mass spectrometer (Model 4130-AMS, High Voltage Engineering Europa B.V., Amersfoort, the Netherlands; Nakamura et al., 2004) at the Institute for Space-Earth Environmental Research, Nagoya University. A HOx-II standard (NIST new oxalic acid standard, SRM-4990C) and commercial dehydrated oxalic acid containing no 14C (Wako, Tokyo, Japan) were used as references for the C isotope ratios and for <sup>14</sup>C blank subtraction in the data analysis, respectively. Experimental one-sigma error was  $< \pm 50$ years. The obtained <sup>14</sup>C age data was calibrated using OxCal 4.3 (Ramsey, 2009).

## 2.3. Measurement of <sup>13</sup>C NMR spectra

The MPS soils at depths of 100, 200, 300, and 450 cm and the ABt and ABg soils at depths of 200, 400, 500, and 700 cm in duplicated profiles were used for the measurement of <sup>13</sup>C NMR spectra. Single samples from MPS-1 at a depth of 50 cm, ABt-1 at a depth of 300 cm, and ABg-1 at depths of 300 and 800 cm were also analyzed. A 30 mg aliquot of the peat soil samples was pulverized to < 0.5 mm placed in a 4 mm sample tube and the ramp cross polarization/magnetic angle spinning (CPMAS) <sup>13</sup>C NMR spectra were recorded at 176 MHz on an ECA 700 spectrometer (JEOL, Tokyo, Japan) installed at Research Center for Materials Science, Nagoya University. The operation conditions were as follows: contact time, 1.0 ms; spinning rate, 12.5 kHz; recycle delay, 1.0 s; and number of data accumulation, 3000-10,000. Chemical shifts were relative to tetramethylsilane (0 ppm) and adjusted with hexamethylbenzene (17.36 ppm). The spectra were divided into 4 regions: 0-45 (saturated alkyl C), 45-110 (oxygen or nitrogen substituted alkyl C; abbreviated to O-alkyl C), 110-160 (aromatic C), and 160–190 (carboxyl C). The O-alkyl C signals were further divided into methoxyl C, including Ca in amino acids (45-60 ppm), and carbohydrate C (60-110 ppm) regions and the aromatic C region was separated at 140 ppm into aromatic C-H/C-C (110-140 ppm) and aromatic C-O (140-160 ppm) regions. Cumulative signal intensity in each region relative to the total was regarded as the relative abundance of each C functional group. Since spinning side bands (SSBs) could not be removed from the chemical shift range for major signals because of too high magnetic field of the NMR spectrometer available, SSBs were corrected using the procedure described by Watanabe and Fujitake (2008). Assuming the signal intensity of SSBs at a higher magnetic field is equivalent to that of counter SSBs at a lower magnetic field, the cumulative signal intensities of the SSBs at 210, 230, and 250 ppm were subtracted from the cumulative signal intensity of O-alkyl C, while twice of them were added to the cumulative signal intensity of aromatic

Table 1									
Physico-chemical	properties and	<sup>14</sup> C age of	peat soil	profiles fro	n different	types of	forests in	Maludam j	peatland.

Forest type	Profile code	Soil depth (cm)	pH (H2O)	Ash (%)	Total C (g kg-1)	Total N (g kg-1)	C/N	$^{14}\text{C}$ age $\pm$ 1 $\sigma$ (yBP)	Cal $^{14}\mathrm{C}$ age 2 $\sigma$ range (cal yBP)
Mixed peat swamp	MPS-1	50	4.5	0.70	544	8	65	$1602 \pm 27$	1413–1550
		100	3.5	0.67	578	10	60	$2920 \pm 49$	2926-3212
		150	4.5	0.60	527	9	56	$3803 \pm 28$	4090-4288
		200	3.8	0.39	560	12	45	$4013 \pm 50$	4296–4798
		300	3.9	0.86	573	11	54	$4429 \pm 50$	4868–5284
		350	3.9	1.62	566	10	58	$4378 \pm 49$	4843–5268
		400	4.1	5.69	542	9	59	$4512 \pm 51$	4978–5314
		450	4.2	23.5	437	7	59	$5162 \pm 54$	5746-6170
	MPS-2	50	4.3	0.70	548	12	46	n.d.	n.d.
		100	3.3	0.72	580	12	48	$2783 \pm 27$	2795–2954
		150	4.4	0.63	505	8	63	$3331 \pm 29$	3478–3637
		200	3.5	0.68	579	11	54	4269 ± 29	4728–4874
		300	3.8	0.97	573	10	55	$4477 \pm 29$	4979–5288
		350	3.9	1.48	566	10	57	$4112 \pm 28$	4525-4814
		400	3.7	6.33	534	8	65	$4825 \pm 50$	5334–5657
		450	4.0	4.52	545	9	61	$4659 \pm 30$	5314–5468
Alan Batu	ABt-1	200	3.8	0.42	544	8	65	$1664 \pm 21$	1560–1613
		300	3.6	0.39	578	10	60	$2097 \pm 22$	1999–2129
		400	4.3	0.26	542	12	47	$2755 \pm 26$	2738-2920
		500	4.1	1.04	572	10	60	$3046 \pm 22$	3177–3343
		600	4.3	1.33	577	10	60	$3638 \pm 23$	3886-4075
		700	4.3	1.58	569	10	57	$4043 \pm 28$	4425-4781
	ABt-2	200	3.6	0.87	579	11	51	$1420 \pm 22$	1293–1351
		300	3.5	0.44	565	14	40	n.d.	n.d.
		400	3.6	0.78	575	12	50	$2622 \pm 21$	2738–2769
		500	3.7	0.74	577	11	53	$3266 \pm 23$	3447–3564
		600	4.1	1.08	582	10	57	$3360 \pm 28$	3509–3690
		700	4.5	1.97	570	12	47	$4020 \pm 23$	4423–4529
Alan Bunga	ABg-1	200	3.8	0.45	577	15	40	$928 \pm 43$	745–928
		300	3.8	0.44	584	14	43	$1771 \pm 47$	1570–1817
		400	3.9	0.83	585	13	44	$2165 \pm 45$	2011-2314
		500	4.2	1.28	584	12	47	$2746 \pm 50$	2760-2950
		600	4.4	1.43	592	11	54	$3277 \pm 22$	3454–3563
		700	4.3	1.39	593	12	52	3376 ± 29	3563–3694
		800	4.3	1.40	583	12	49	$3026 \pm 46$	3077–3357
	ABg-2	200	3.6	0.28	563	12	46	1366 ± 44	1183–1353
		300	3.7	0.43	568	12	47	$1726 \pm 26$	1565-1702
		400	3.7	0.45	569	11	51	$2161 \pm 27$	2060-2307
		500	3.8	0.59	575	12	49	$2362 \pm 26$	2337–2463
		600	3.7	0.60	570	11	52	n.d.	n.d.
		700	4.1	0.82	575	11	53	$2806 \pm 28$	2845–2993
		800	n.d.	0.60	571	11	52	2438 ± 27	2356-2700

n.d., Not determined.

C-H/C-C, aromatic C-O, and carboxyl C, respectively.

#### 2.4. Statistical analysis

To estimate the rate of accumulation of peat, the relationship between the calibrated <sup>14</sup>C age and soil depth was regressed to a primary or exponential function. In the calculation, with the constraint that the age of surface sediment is 0 year, 63 years were added to the calibrated <sup>14</sup>C age in which AD1950 is regarded as 0 year, and data from each soil profile was regressed to y = ax exclusively. Correlation analysis was done to analyze the relationship between soil chemical properties or C composition of the SOC and soil depth or <sup>14</sup>C age.

## 3. Results

### 3.1. Characteristics of peat soil samples

Total C content, total N content, and C/N ratio for all of the soil samples were in the range of  $491-584 \, g \, kg^{-1}$ ,  $8-13 \, g \, kg^{-1}$ , and 43-67, respectively (Table 1). In the MPS-1, total C content was smaller and ash content was larger at a depth of  $450 \, cm$ , indicating the influence of mineral soil. The pH and ash content were in the range of 3.3-4.5 and 0.3-23.5%, respectively. The pH in the ABt forest soil profiles and the ash content in the ABg forest soil profiles increased with soil depth

(P < 0.01), respectively. Throughout the MPS, ABt, and ABg forest soil profiles, a higher C/N was attributed to a smaller N content (P < 0.005).

## 3.2. <sup>14</sup>C age of peat soil profiles

In the MPS, ABt, and ABg forest soil profiles, the <sup>14</sup>C age ranged from 1602 to 5162 yBP, 1134–4043 yBP, and 928–3277 yBP, respectively (Table 1). Fig. 2 shows the relationship between calibrated <sup>14</sup>C age and soil depth for those soil profiles. The calibrated <sup>14</sup>C ages of the ABt forest soils increased nearly constantly with increasing soil depth. The mean rate of peat accumulation was estimated to be 1.5 mm y<sup>-1</sup> for both profiles ( $R^2 = 0.97-0.98$ ; P < 0.005). A similar relationship between calibrated <sup>14</sup>C age and soil depth was also observed in the ABg forest soil profiles within a depth of 700 cm, and regression to a linear function indicated rate of peat accumulation of 1.8 mm y<sup>-1</sup> (ABg-1;  $R^2 = 0.947$ ; P < 0.005) or 2.0 mm y<sup>-1</sup> (ABg-2;  $R^2 = 0.86$ ; P < 0.005). However, the calibrated <sup>14</sup>C age of the ABg forest soils decreased from a depth of 700 cm to 800 cm.

Regression of the calibrated <sup>14</sup>C age and soil depth to a linear function indicated that the mean rate of accumulation of peat at the MPS forest was 0.61–0.62 mm y<sup>-1</sup> ( $R^2 = 0.60-0.69$ ; P < 0.01). However, their relationship was fitted better to the following exponential functions:



**Fig. 2.** Relationships between the calibrated <sup>14</sup>C age of bulk soil organic matter and soil depth in the Mixed Peat Swamp (MPS), Alan Batu (ABt), and Alan Bunga (ABg) forests in the Maludam National Park, Sarawak, Malaysia. Regression equations for the ABg soil profiles were obtained excluding the data from 800 cm depth. Error bars indicate  $2\sigma$  ranges. <sup>\*\*\*</sup> indicates P < 0.005.

$$MPS - 1: y = 20.9e^{0.000527x} (R^2 = 0.92; P < 0.005)$$
(1)

$$MPS - 2: y = 21.5e^{0.000529x} (R^2 = 0.87; P < 0.005)$$
(2)

According to these equations, the mean rate of accumulation of peat at the MPS forest was estimated to be  $0.74 \text{ mm y}^{-1}$  within a depth of 450 cm for both soil profiles.

## 3.3. Ramp CPMAS <sup>13</sup>C NMR spectra

Representative <sup>13</sup>C NMR spectra of the peat soil samples are shown in Figs. 3 and 4. The major signal peaks were observed at 27–33 (methylene C in long alkyl chain), 54–56 (methoxyl C), 61 (primary alcohol C), 72–74 (secondary alcohol C), 103–105 (acetal C), 129–132 (aromatic C-H/C-C), 151–154 (aromatic C–O), and 173–178 (carboxyl C) ppm for all samples. In the MPS forest soil profiles, the methylene C signals were the most intense for depth of 50 cm and decreased with depth (Fig. 3). On the contrary, the relative intensity of methoxyl, secondary alcohol, acetal, and aromatic C signals increased from an upper toward lower layers. Similar patterns were not observed in the ABt and ABg forest soil profiles, where the methylene C signals were generally small and tended to be larger in the upper layers in the ABg forest soil profiles (Fig. 4b).

Table 2 shows the C composition of the soil samples, as estimated from the <sup>13</sup>C NMR spectra. In the MPS soil profiles, the relative abundance of alkyl C (alkyl C %) decreased from 40% at a depth of 50 cm to 20 or 24% at 450 cm depth. There was a significant negative correlation between the alkyl C % and calibrated <sup>14</sup>C age (r = -0.87; P < 0.005; Fig. 5a). On the contrary, the relative abundance of *O*-alkyl C increased from 15% to 31 or 34%. Positive correlations were observed between calibrated <sup>14</sup>C age and both the methoxyl C % (r = 0.81; P < 0.01) and carbohydrate C % (r = 0.87; P < 0.005). The relative abundance of aromatic C remained high, 34–42%, including aromatic C-H/C-C (19–27%) and phenolic C (13–16%), although their signal intensity varied relative to the intensity of alkyl C signals.



Fig. 3. Ramp CPMAS  $^{13}\mathrm{C}$  NMR spectra of peat soil samples from the MPS-1 soil core.

In the ABt soil samples, alkyl C, 24–39%, and aromatic C, 26–43%, were consistently the major C groups. The decrease in the relative abundance of *O*-alkyl C with increasing soil depth or age was observed between 200 and 500 cm depths or ca 1300–3500 cal yBP (P < 0.05), which was attributed to the variation in carbohydrate C % (Fig. 5b). In the ABg soil samples, the relative abundance of carbonyl C decreased to a half with increasing soil age (P < 0.01; Fig. 5c), and no other chronological transitions were observed as is shown by the narrow range of alkyl C (25–37%) and aromatic C (31–41%).

## 4. Discussion

In the present study, <sup>14</sup>C age at the bottom of peat layer in each forest site was not determined. When the depth of peat layer bottom was extrapolated to the regression equations of exponential (MPS) or primary (ABt) functions, the results suggested that peat began to accumulate at ca 6000 cal yBP at the MPS forest site and ca 4700–4800 cal yBP at the ABt forest site. In the ABg forest site, <sup>14</sup>C age at a depth of 800 cm was younger than that at a depth of 700 cm in both profiles (Table 1). An upside down <sup>14</sup>C age with soil depth has often been observed in tropical peatlands (Page et al., 2004; Wüst et al., 2008; Cole et al., 2015). Penetration of plant roots from an upper layer, followed by decay and humification, may be a possible cause of this observation (Page et al., 2004). It is interesting that two ABg profiles, ca 1 km apart from each other, showed similar trend at the same depth. Assuming that



Fig. 4. Ramp CPMAS <sup>13</sup>C NMR spectra of peat soil samples from the ABt-1 (a) and ABg-1 (b) soil cores.

the rate of peat accumulation was similar between above and below 700 cm, peat formation at the ABg forest site started between 4000 and 5000 cal yBP. These values are uncertain to compare the period when peat formation started among the three forest sites, and direct determination of the <sup>14</sup>C age of the bottom layers is required to confirm them. Nevertheless, the period of the Maludam peatland history may be consistent with that of other lowland peat domes in Sarawak reported in Dommain et al. (2011), 1058-7309 cal yBP, which includes the calibrated basal dates from original data in Tie and Esterle (1992) and Staub and Esterle (1994). It has been reported that peat accumulates in coastal peatlands in Indonesia and Malaysia (Dommain et al., 2011) at an average of  $1.8 \text{ mm y}^{-1}$  and in inland peatlands in Central Kalimantan (Page et al., 2004) and in Eastern Kalimantan (Hope et al., 2005), accumulation is in the range from 0.2 to 0.4 and 1.4-4.1 mm  $y^{-1}$ , respectively. Thus, the rates of peat accumulation in our study sites appear to be moderate in comparison.

To estimate the rate of C accumulation in a peatland, the data of soil bulk density is required in addition to  $^{14}$ C age and soil C content. Although we do not have exact data of bulk density, according to the detailed record in a Brunei peatland (Dommain et al., 2011) bulk density of soil with a high C content was similar throughout peat layers, 0–340 cm depth (0.072 g cm<sup>-3</sup> on average). Assuming this trend is applicable to Maludam peatland, the long-term apparent rate of C accumulation in each forest site was estimated using the average bulk density obtained for 0–150 cm layer soils for each forest site (Melling, L., unpublished data). The estimated values for the MPS, ABt, and ABg

forest soils were 64 (with the range of 12-111), 55, and 21 (by the depth of 600 cm) g C  $m^{-2}\ y^{-1},$  respectively. These values were comparable with previously reported data for coastal peatlands in Indonesia and Malaysia, 55–107 g C m<sup>-2</sup> y<sup>-1</sup> with an average of 77 g C m<sup>-2</sup> y<sup>-1</sup> (Dommain et al., 2011) and inland peatlands in Kalimantan, 10-73 g C  $m^{-2}\ y^{-1}$  (Page et al., 2004; Dommain et al., 2015). The rates of C accumulation reported for northern peatlands are also generally distributed in the similar range, e.g., 12–80 g C m $^{-2}$  y $^{-1}$  in former Soviet Union (Botch et al., 1995), 31–34 g C m<sup>-2</sup> y<sup>-1</sup> in China (Zhao et al., 2011; Xing et al., 2015), and 14–22 g C m<sup>-2</sup> y<sup>-1</sup> in Canada (Roulet et al., 2007). As such, the geology and/or vegetation type is the likely cause of the wide range of C accumulation rates in tropical peatlands, while the function of ABg site and recent MPS site as C storage would still be invaluable in C cycle. It should be noted that the rate for the ABg forest soil could be underestimated because of very low data of bulk density  $(0.02 \, \text{g cm}^{-3})$ .

The relationship between calibrated <sup>14</sup>C age and soil depth in the MPS forest soil profiles, a convex curve, was opposite to that reported for some temperate peatlands, a concave curve (Belyea and Malmer, 2004; Grover et al., 2012; Tfaily et al., 2014). In the latter cases, the SOM in the shallower layers is nearly completely plant debris and its decomposition progress at deeper layers. Yu et al. (2003) reported that the convex growth pattern for peat from a boreal fen was attributed to the progressive isolation from the groundwater table and a reduction in plant productivity. The lower groundwater table at the MPS forest site than at the ABt and ABg forest sites in Maludam (Sangok et al., 2017)

#### Table 2

C composition of peat soils at different depths (%).

Profile code	Soil depth (cm)	Alkyl C <sup>a</sup> (0–45 ppm)	O-alkyl C (45–110 ppm)	Methoxyl C (45–60 ppm)	Carbohydrate C (60–110 ppm)	Aromatic C <sup>b</sup> (110–160 ppm)	Aromatic C-H/C-C (110–140 ppm)	Phenolic C (140–160 ppm)	Carbonyl C (160–190 pm)		
Mixed Deat Swamp forest											
MPS-1	50	39.6	15.1	5.8	9.3	36.5	23.7	12.9	8.7		
	100	28.6	27.7	10.4	17.3	35.5	21.3	14.2	8.2		
	200	29.5	24.6	8.6	16.0	35.0	19.5	15.5	11.0		
	300	25.1	33.2	11.8	21.4	36.3	22.6	13.7	5.4		
	450	23.6	34.3	11.9	22.4	37.5	22.2	15.2	4.6		
MPS-2	100	34.5	17.2	6.1	11.1	37.6	23.7	13.9	10.7		
	200	31.3	25.2	9.0	16.3	34.1	19.1	14.9	9.4		
	300	27.1	28.2	10.0	18.2	37.7	24.9	12.8	7.0		
	450	19.6	31.2	10.3	20.9	42.0	26.5	15.5	7.1		
Alex Detry Co											
Alan batu io	and	22.2	22.0	7.0	16.1	05.1	00.1	12.0	7.0		
ABI-1	200	33.Z	23.9	/.8	10.1	33.1	23.1	12.0	7.8		
	300 400	30.1	21.0	0.0 7.0	12.0	30.8	18.5	12.3	12.0		
	400 E00	29.4	20.7	7.0 6.0	13.7	42.2	24.7	12.2	0.4		
	700	20.4	26.2	0.9	10.9	40.0	24.0	15.4	85		
A R+ 2	200	24.4	20.2	9.1 7.7	22.8	20.8	23.3	1J.4 8 5	5.6		
ADI-2	400	20.4	24.6	7./	17.2	25.6	18.0	7.6	10.5		
	500	25.6	16.5	6.2	17.2	29.0	21.0	16.2	0.6		
	700	21.0	27.5	0.2	10.5	34.5	21.9	10.5	5.0 6.1		
	/00	51.9	27.5	2.4	10.1	54.5	21.1	15.4	0.1		
Alan Bunga f	orest										
ABg-1	200	33.1	26.3	7.4	18.9	30.6	21.2	9.3	10.0		
	300	33.6	22.7	6.5	16.1	34.1	20.2	13.9	9.6		
	400	33.8	23.4	6.6	16.8	34.3	20.3	14	8.5		
	500	30.6	27.2	8.0	19.2	34.2	18.1	16.1	8.0		
	700	36.4	20.6	8.1	12.6	35.5	22.7	12.8	7.4		
	800	36.5	23.6	8.6	15.1	33.2	20.7	12.5	6.7		
ABg-2	200	27.8	19.2	6.5	12.7	40.9	24.4	16.5	12.1		
	400	26.6	22.9	7.4	15.5	39.7	24.3	15.4	10.8		
	500	29.2	31.7	8.1	23.5	31.7	18.4	13.2	7.5		
	700	25.1	32.2	10.7	21.5	36.2	23.3	12.9	6.5		

<sup>a</sup> Sum of methoxyl C and carbohydrate C.

<sup>b</sup> Sum of aromatic C-H/C-C and phenolic C.

may be related to the rise of dome center over time due to peat accumulation, which accelerates drainage at slope edges and therefore saturation that allows for constant rate of peat accumulation could not be maintained. The decrease in the rate of peat accumulation at the MPS site corresponded to the enrichment of alkyl C with the depression of *O*alkyl C (Fig. 3 and Table 2). Hence, during the initial stage of the peat soil development at the MPS site, the accumulation of peat deposit advanced fast without intensive SOM decomposition, and then it became slower due not to a decreased plant productivity but to an enhanced SOM decomposition resulting from the lowering of groundwater table relative to soil surface (Table 2). This mechanism may be applicable to other tropical peat domes as a common formation process (Cobb et al., 2017). High ash content in the 450 cm depth layer sample from the MPS-1 profile was possibly due to the inflow of river sediment during a temporal high tide, because many white sand particles were left after the acid-alkali-acid treatment. Their influence on the  $^{14}$ C age and C composition was not decisive.

The rate of peat accumulation in the ABt and ABg forest sites was almost constant, except for the 600–800 cm depth layer in the ABg forest soil samples (Fig. 2). Similar observation was also reported for some peatlands in coastal areas of Indonesia and Malaysia (Page et al., 2004; Dommain et al., 2011). On the contrary, inland peat domes have been characterized by a reduction in peat accumulation over time (Dommain et al., 2014). Thus, the almost constant peat accumulation could be typical for coastal peatlands. In the ABt forest soil samples, the gradual reduction in the proportion of SOC that is present as



**Fig. 5.** Relationships between <sup>14</sup>C age and relative content of C groups. (a) vs alkyl C, *O*-alkyl C, and carbohydrate C in the MPS soil profiles, (b) vs *O*-alkyl C and carbohydrate C in the ABt soil profiles, and (c) vs carbonyl C in the ABg soil profiles. The symbols  $\Delta$ ,  $\oplus$ ,  $\bigcirc$ , and  $\blacksquare$  indicate alkyl C, *O*-alkyl C, carbohydrate C, and carbohydrate C, respectively. \*, \*\*, and \*\*\*\* indicate P < 0.05, < 0.01, and < 0.005, respectively.

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carbohydrate C with a passage of time was also suggested. The higher carbohydrate C % at a depth of 700 cm, 17–18%, than at a depth of 500 cm, 10–11%, might have come from the different plant species or environmental conditions during the initial stage of peat accumulation there, since the carbohydrate C % in the neighboring MPS forest soil with similar <sup>14</sup>C age was also 16% (Table 2).

In the ABg forest soil samples, the proportion of SOC that is present as *O*-alkyl C in the uppermost layer soils was smaller than that reported for the 20–40 cm depth soil, 36.5% (Sangok et al., 2017). Thus, after the readily decomposable polysaccharides were removed, further compositional changes are slow. The negative correlation between the carbonyl C % and <sup>14</sup>C age (Fig. 6c) may express the higher extent of SOC oxidation in the upper layers, which supported by the negative correlation between carbonyl C % and methoxyl C % (P < 0.05). Methoxyl C is considered to be derived mainly from lignin residues and decrease in soil. Although the methoxyl C % includes  $C_{\alpha}$  in amino acid, it does not affect their relationship because the variation in the number of C replaced by N was small throughout the soil samples, 1.5–2.5% of total C (Table 1).

The proportion of C that was present as aromatic C ranged from 30 to 43% for all soil cores with an exception (Table 2), which was frequently the largest among the 5 types of SOC. The low activity of fungi that degrade lignin (Freeman et al., 2004) may contribute to it. Since the relative abundance of aromatic C in boreal peat SOC has been reported to be < 20% (Tfaily et al., 2012; Normand et al., 2017), high aromatic C content could be regarded as a characteristics of tropical peat SOM, which mainly originates from woody materials (Hodgkins et al., 2018). High phenolic C % to aromatic C % ratio, 0.29-0.47, corresponds roughly to benzene rings substituted with 2-3 oxygen atoms, suggested that oxidation of lignin have frequently stopped after the conversion of methoxyl groups to hydroxyl groups and that of alkyl chains to carboxyl groups. Alternative is the loss of aromatic components could be due to their oxidation to fulvic acids, followed by leaching, which can explain the lack of the increase in aromatic C % when alkyl C was concentrated.

#### 5. Conclusions

The findings reported herein provide information regarding the unique characteristics of SOM transition, closely related to the difference in peat accumulation rates, among three forests in a coastal tropical peatland. The rate of peat accumulation is in the order: ABg  $(1.8 \text{ mm y}^{-1}) > \text{ABt} (1.5 \text{ mm y}^{-1}) > \text{MPS} (0.63 \text{ mm y}^{-1})$ . Although the rate of peat accumulation in the older layers of the MPS forest soil profiles was similar to or faster than those in the ABt and ABg forests, the overall peat layers were thicker in an inner site of the dome that developed later. The decrease in the peat accumulation rate in the MPS forest site resulted in distinct changes in SOC composition, notably, a relative increase in alkyl C with decrease in *O*-alkyl C. Such variation was not observed when the rate of peat accumulation was high and constant (sites of ABt and ABg forests). The aromatic C was recognizable as a major and stable form of C in a coastal tropical peatland.

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