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RESEARCH ARTICLE

Geochemistry of Negombo Lagoon sediments in Sri Lanka: implications for environmental monitoring

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Abstract: Negombo Lagoon is one of the largest lagoons in Sri Lanka. This study focused on the geochemical evaluation of poorly understood tropical subtidal sediments of the western coast, Sri Lanka. Geochemical characteristics of sediments were examined using $\delta^{13}C$ and $\delta^{15}N$ stable isotopes, X-ray diffraction (XRD), and Fourier-transform infrared spectroscopy (FTIR) analyses. The chronology was determined using accelerated mass spectrometry (AMS) ^{14}C dates for undamaged two shells. Stratigraphic observations suggested grey to blackish fine-grained muddy clay sediments in the subtidal mudflat. The calculated organic matter and carbonate contents decrease slightly from bottom to surface sediments. Bulk C/N ratios and $\delta^{13}C$ values suggested terrestrial organic matter dominant sediments such as from mangrove swamps mixed with marine dissolved organic carbon. XRD identified the crystalline phases present in sediments and thereby chemical composition was identified by the software as illite, montmorillonite, and quartz. It indicates the deposition of limited chemical weathering materials from near distance sources. FTIR identified several functional groups such as carboxylic, polysaccharide, carboxylate ions, and aliphatic substances in the lagoon sediments. The variations of carboxylic groups/carboxylate ions indicate a slow rate of decomposition of organic matter during early diagenesis. Humification and decomposition proxies also indicate slow rates of decomposition under the anoxic condition. However, Negombo Lagoon still acts as a healthy ecosystem under the minor influence of sewage/agricultural nutrient contamination.

Keywords: Decomposition, diagenesis, humification, subtidal mudflat, tropical lagoon sediments.

INTRODUCTION

Tropical brackish ecosystems such as mangrove swamps, intertidal/subtidal mudflats, and coastal peatlands play an important role in the global biogeochemical cycles (Yu *et al.*, 2010; Page *et al.*, 2011; Gallego-Sala *et al.*, 2018). Tropical coastal aquatic systems are natural depositional archives for terrestrial materials over short to longer timescales (Kumaran *et al.*, 2013; Ratnayake *et al.*, 2017; Dubois *et al.*, 2018). Limnological studies provide proxy information to reconstruct the delivery and decomposition changes in the burial of sedimentary organic matter during early diagenesis. However, tropical subtidal sediments have received little attention in limnological studies on the modern time scale compared to mid- to high-latitude intertidal and subtidal sediments. The present study thus focuses to understand carbon sink potential, depositional, and decomposition characteristics of tropical subtidal sediments in Negombo Lagoon of Sri Lanka (Figure 1).

The island of Sri Lanka consists of about 1770 km long coastal tract with diverse geomorphological features such as lagoons, bays, lakes, wetlands, and peatlands. The formation of coastal lagoons has resulted in late Quaternary marine transgression (Katupotha, 1988; Katupotha & Fujiwara, 1988). Negombo Lagoon is one of the largest estuarine lagoons in Sri Lanka. The

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Muthurajawela peatland is the southern extension of Negombo Lagoon (Figure 1). Geochemical characteristics of Negombo Lagoon sediments can be influenced by the materials derived from the Muthurajawela peatland. Therefore, the lake sediments can be used to understand the sedimentary geochemistry as a function of erosion, transport, deposition, burial, and decomposition of organic matter during early diagenesis that is highly sensitive to environmental changes. However, geochemical characteristics of lagoon sediments have rarely been focused in Sri Lanka (Ratnayake *et al.*, 2017, 2018). In addition, understanding of geochemistry in lagoon sediments can be applied with aspects of environmental monitoring assessments and prediction of aquatic health (Owca *et al.*, 2020; Zerizghi *et al.*, 2020).

The objective of the current study is to provide reference data for quality assessment of Negombo Lagoon sediments. In this study, the authors investigated the geochemistry of Negombo Lagoon sediments using Fourier transform infrared spectroscopy (FTIR), X-Ray diffraction (XRD), stable isotope ($\delta^{13}C$, $\delta^{15}N$) analyses, along with accelerated mass spectrometry (AMS) ^{14}C dating. This study provides comprehensive coverage of geochemical characteristics of Negombo Lagoon sediments over the modern period of the Anthropocene.

METHODOLOGY

Study area and sampling locations

Negombo Lagoon and its southern extension of Muthurajawela peatland are located on the west coast of Sri Lanka (Figure 1). Negombo Lagoon covers an area of 32 km^2 with an average water depth of 1.2 m. The maximum length and width of the lagoon are about 12.0 km and 3.8 km, respectively. The northern part of the study area opens to the Indian Ocean through narrow channels due to having several islands at the estuary mouth.

The southward part of the study area is connected to the Muthurajawela peatlands which covers an area of approximately 21 km^2 with an average thickness of 3.7 m over the lateritic soil (Dissanayake *et al.*, 1982). This Quaternary formation is the largest peat deposit in Sri Lanka with having high sulfur content ranging from 3.0 to 5.5 % (Dissanayake *et al.*, 1982). The intertidal area of Negombo Lagoon is mainly composed of tropical wetland species such as fringe mangroves and mangrove associates. The study area can be recognised as the biodiversity-rich ecosystem. For example, Negombo Lagoon comprises 89 species of benthic invertebrates, 29 mangrove species, 7 seagrass species, and 140 species of fish (Dahanayake *et al.*, 2008).

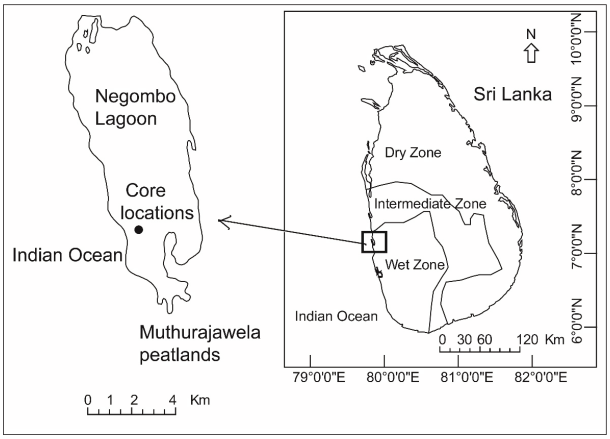

Figure 1: Sri Lanka map showing Negombo Lagoon and sampling locations.

The sampling sites were selected from the southern part of Negombo Lagoon which has greater vulnerability for the accumulation of tropical peaty organic matter in sediments. Two nearby sediment cores [(NLC 1, Global Positioning System (GPS) Coordinates: 7.07190 N, 79.50460 E and NLC2, GPS Coordinates: 7.122054 N, 79.846077 E)] were collected from the southern part of Negombo Lagoon using a hand auger sampler (Figure 1). The elevations of these coring sites are less than the mean sea-level. NCL 1 and NCL 2 show similar lithological variations. Therefore, NLC 1 (64 cm in depth) sediment core was used for geochemical analyses.

Methods

Moisture content and loss on ignition

Core logs were prepared in the field using visual inspection. Sediment core samples were sealed and placed in cooling boxes during transportation to the laboratory. After that, samples were stored in a refrigerator at 4 °C until further treatments. The core samples were sliced into 2 cm intervals. Moisture, organic matter, and carbonate contents were determined for 32 sediment samples.

First, 2 cm-interval sediment samples were ground into a fine powder (less than silt size) using agate mortar and pestle. The wet weight of powder samples was measured, and placed in the convection oven at 110 °C for 24 h to measure dry weight. Moisture content was expressed as a percentage of the difference between initial wet weight and dry weight (110 °C) to the initial wet weight [(American Society for Testing and Materials (ASTM) reference no: D2216 – 19)]. Loss on ignition (LOI) test ($LOI_{550°C}$ and $LOI_{950°C}$) was carried out for every 2 cm-interval sub samples to determine organic matter and carbonate contents, respectively (Dean, 1974; Byers *et al.*, 1978; Heiri *et al.*, 2001). Approximately 5.0 g of the same samples were reheated up to 550 °C for 6 h in a Muffle furnace to calculate organic matter content. Similarly, carbonate content was calculated using the weight of a baked sample up to 950 °C in a Muffle furnace for an additional 2 h.

Stable isotopic composition

Powder samples were flash combusted at roughly 1800 °C to produce various carbon and nitrogen compounds. These compounds were carried by a continuous flow of helium (90 mL/min) and then passed through a series of catalysts to convert the remaining CO to CO_2. A reduction column consisted of elemental copper for reducing NO and NO_2 to elemental nitrogen (N_2). Nitrogen and carbon dioxide gasses were separated from the sample through a gas chromatography (GC) column and were then analysed for $\delta^{13}C$ and $\delta^{15}N$ using a Thermo Fisher Scientific Delta V Advantage Isotope Ratio Mass Spectrometer (IRMS) coupled with a Carlo Erba NC 2500 elemental analyser at Illinois State Geological Survey, USA.

Ammonium sulfate standards USGS-25 and USGS-26, L-Glutamic Acid USGS-40, as well as IAEA-600 Sucrose, and an internally calibrated amino acid (L-Serine) were analysed as nitrogen standards in duplicate and a calibration curve was generated. Samples were then calibrated and reported as $\delta^{15}N$ vs. Air. Atropine, IAEA-600 Caffeine, USGS-40, and Amino Acid L-Serine were analysed as carbon standards and a calibration curve was generated. Samples were then calibrated and reported as $\delta^{13}C$ Vienna Pee Dee Belemnite (VPDB). The calibration curves produced by the above-mentioned standards typically yields an R^2 value of 0.9995 or better. The compiled results show the $\delta^{15}N$ to be better than ± 0.22‰, and the $\delta^{13}C$ to be better than ± 0.11‰.

XRD analysis

The X-ray diffraction powder patterns were recorded for seven smooth and well-packed sediment samples at room temperature using Rigaku Ultima (IV) X-ray diffractometer (XRD) with a source of CuKα radiation and NaI scintillation counter detector. The narrow silt of 0.1 mm was used during the recording of diffractograms with a scanning speed of 0.02°/second. Diffraction data were collected in the Bragg-Brentano θ–2θ geometry with CuKα radiation. The estimated error in the lattice parameter is the order of 0.0005 A.

FTIR analysis

Sediment samples were finely powdered, and mixed with potassium bromide (KBr) at 1:10. In this study, potassium bromide (KBr) pellet method was used to prepare samples. FTIR grade (assay ≥ 99%) dried KBr was used as alkali halide. Approximately 0.10% powdered sample was well mixed with KBr powder using a clean dry agate mortar and pestle. A pellet of 1 mm in thickness and 7 mm in diameter was prepared using hand press pelletizer and 7 mm die. KBr/powder mixture was loaded into the die chamber and upper anvil was placed in the press. Then, hand pressure was applied to the lever to form clear KBr/powder disks. The die collar with the formed pellets was removed from the press and it was placed directly in the beam of the spectrometer for analysis. This procedure was followed for the preparation of every pellet. Before performing the measurements, the background was measured with a pellet of KBr that contains no sample.

FTIR analysis was carried out for 32 sediment samples using a Bruker Alpha spectrophotometer. The functional groups were identified over the range of 400–4000 cm^{-1} (the instrument resolution of $4cm^{-1}$ over 64 scans) after correction for the absorption of atmospheric water and CO_2. In this study, FTIR spectral data were used to determine the degree of humification and decomposition according to the literature (Broder *et al.*, 2012; Krumins *et al.*, 2012; Biester *et al.*, 2014).

Age dating

The ^{14}C radiometric dating was measured at the International Chemical Analysis Inc. Laboratory, USA for two undamaged shell samples at depths of 44–46 cm and 56–58 cm. The radiocarbon dates were calibrated into calendar ages using the Marine13 radiocarbon age calibration curves 0–50,000 year cal BP (Reimer *et al.*, 2013) and the calibration software Calib 7.10. Conventional ages are given in BP (1950 AD), and have been corrected for fractionation using the $\delta^{13}C$values.

RESULTS AND DISCUSSION

Lithology and geomorphology

The core observations indicate that the sedimentary succession is mainly composed of grey to blackish fine-grained muddy clay (Figure 2). Negombo Lagoon sediments record mollusk shells, wood fragments, and black carbon. This observation suggests that sedimentary organic matter can mainly derive from allochthonous sources. Field investigations indicated the herbaceous plants (e.g. grasses) in muddy sediment of the shallow area of Negombo Lagoon. However, the bottom of the lagoon mouth (Figure 1) is mainly composed of sandy sediments that are transported by longshore currents along the western coast of Sri Lanka (Amalan *et al.*, 2018; Ratnayake *et al.*, 2019b). Table 1 shows the results of the accelerated mass spectrometry (AMS) ^{14}C analysis. According to radiometric dating, the undamaged shells at 45 cm and 57 cm in depths were deposited after 1950, known as modern (Table 1).

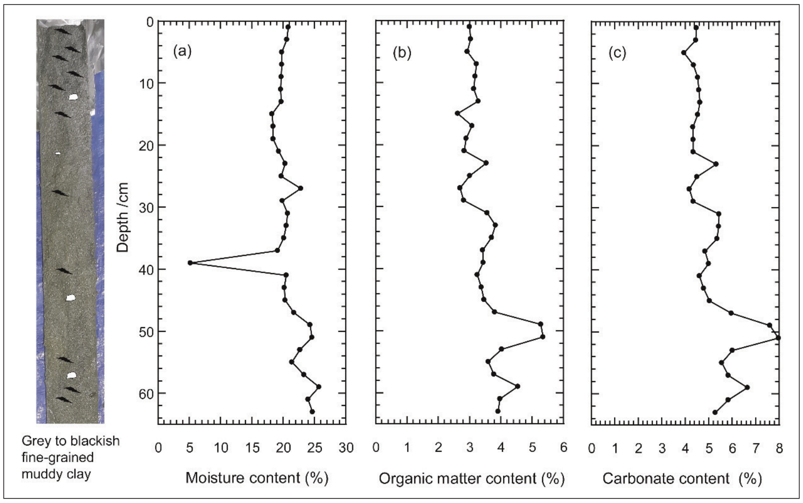

Figure 2: Vertical distributions of moisture, organic matter, and carbonate contents in Negombo Lagoon sediments, and simplified stratigraphic column of the sedimentary succession.

Table 1: AMS ^{14}C radiometric dating.

Depth/ cm	Type	Conventional 14C BP	Calendar age BP +/- 2 σ	Method	Reference no.
44-46	Shell	1.1920 +/- 0.0056 F14C	Modern	AMS	18S/1108
56-58	Shell	1.0810 +/- 0.0031 F14C	Modern	AMS	18S/1109

Moisture content and loss on ignition

The moisture content decreases from bottom to surface sediments (range from 18.2 to 25.7 %) with some outliers of 5.2 % at depth of 38–40 cm (Figure 2a). Figure 2b shows that sequential loss on ignition of $LOI_{550°C}$ also decreases towards surface sediments ranging from 2.6 to 5.3%. Sequential loss on ignition of $LOI_{550°C}$ is a widely used method to estimate organic matter content of sediments, as organic matters oxidise to carbon dioxide and ash at 500–550 °C (Dean, 1974; Heiri *et al.*, 2001). In this regard, the reduction of organic matter accumulation/ production or enhancement of bottom-water oxygenation can be expected from bottom to surface sediments in Negombo Lagoon. Therefore, it suggests that deterioration conditions of Negombo Lagoon gradually decrease compared to its past environment.

Carbonate content also decreases from bottom to surface sediments ranging from 4.0 to 8.0 % (Figure 2c). Sequential loss on ignition of $LOI_{950°C}$ is used to estimate carbonate content of sediments, as carbon dioxide releases from carbonate at 900–1000 °C (e.g. Dean, 1974; Heiri *et al.*, 2001). In this study, organic matter distribution shows strong positive correlation ($r > 0.9$) with the calculated carbonate distribution of sediments (not illustrated). Therefore, in this sedimentary succession, the similar trends of organic and carbonate (inorganic) carbon distributions (Figure 2) indicate minor influence for the anthropogenic/sewage contamination (Emerson & Hedges, 1988; Hartnett *et al.*, 1998; Stein & Fahl, 2000).

Stable isotopic compositions

Table 2 and Figure 3 show $\delta^{13}C$ isotopic composition and bulk C/N ratio of Negombo Lagoon sediments. Bulk $\delta^{13}C$ stable isotopic composition is useful to distinguish between marine and continental plant sources, and to distinguish organic matter from different sources of land plants (e.g. $\delta^{13}C$ of C_3 plants = ca. –28‰: range from –32 to –21‰ and $\delta^{13}C$ of C_4 plants = ca. –14‰: range from –17 to –9‰). The $\delta^{13}C$ isotopic composition of plant biomass is thus primarily a function of the photosynthetic pathway (Bender, 1971; Lamb *et al.*, 2006). Therefore, stable isotope analyses of bulk lake sediments allow an estimation of the relative contributions from marine and C_3/C_4 plant sources (Meyers, 1997, 2003). Besides, C/N ratios of algae and land plants are between 4 and 10 and greater than 12, respectively (Meyers, 1997, 2003; Ratnayake *et al.*, 2019a). Therefore, in this study, $\delta^{13}C$ and C/N values (range from 12.9 to 21.4) indicate the mixing of C_3 land plants and marine sources as marine dissolved organic carbon (Table 2 and Figure 3). Similarly, $\delta^{15}N$ values can be generally used to distinguish marine and continental organic matter (Peters *et al.*, 1978; Lamb *et al.*, 2006), and aspects of nitrogen biogeochemical cycling during the environmental changes. For example, the increases in $\delta^{15}N$ values indicate the accumulation of isotopically heavy nitrate ($\delta^{15}N$ = 10–25‰) from agricultural runoff and human sewage (Teranes & Bernasconi, 2000; Meyers, 2003). Therefore, $\delta^{15}N$ values (range from 3.61 to 8.01‰ in Table 2) suggest that agricultural runoff and sewage contamination is low in Negombo Lagoon sediments.

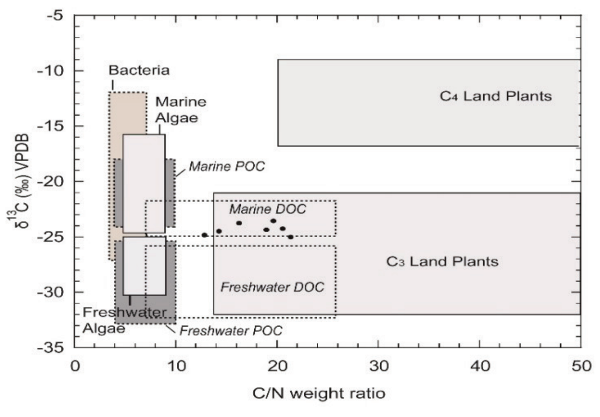

Figure 3: Generalised isotopic $\delta^{13}C$ and elemental atomic C/N ratio shows different sources of organic matter in the lake sediments (where, VPDB: Vienna Pee Dee Belemnite, POM: particulate organic matter and DOC: dissolved organic carbon, modified after Meyers 2003 and Lamb *et al.*, 2006).

Table 2: Bulk organic geochemical and stable isotopic compositions (VPDB: Vienna Pee Dee Belemnite).

Sample Name	Depth range/cm	δ13C value	Organic C%	δ 15N value	N %	C/N ratio
NLC1 1	0-10	-24.41	0.32	4.59	0.02	19.0
NLC1 2	10-16	-24.53	0.27	4.10	0.02	14.3
NLC1 3	20-26	-24.30	0.46	3.61	0.02	20.6
NLC1 4	30-34	-23.78	0.46	4.19	0.03	16.3
NLC1 5	40-44	-25.05	0.73	4.53	0.03	21.4
NLC1 6	50-56	-23.60	0.75	3.66	0.04	19.7
NLC1 7	56-64	-24.87	0.71	4.63	0.06	12.9

XRD observations

Figure 4 shows clay minerals such as montmorillonite (two theta/2θ values = 36.66°, 75.76°) and illite (two theta/2θ values = 20.10°, 39.58°, 67.82º) with different intensities, based on crystalline phases and discovered chemical composition by software. Non-clay minerals such as quartz (two theta/2θ values = 20.94°, 26.72°, 42.38°, 50.24°, 54.98°) were also observed in X-ray diffractograms with different peak intensities (Figure 4).

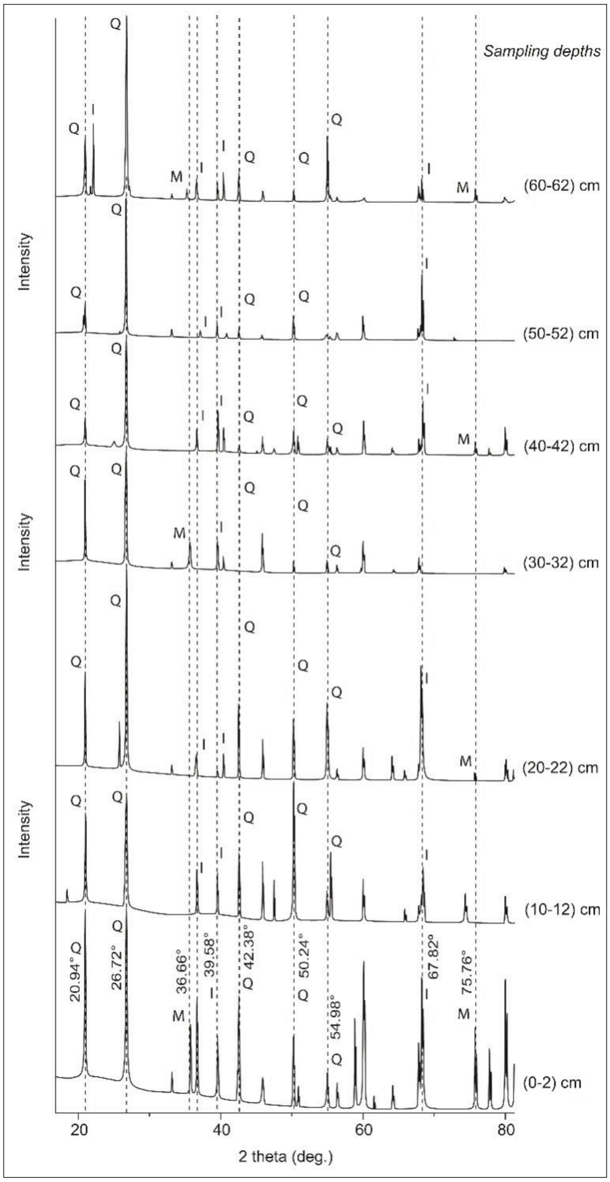

Figure 4: Characteristic XRD patterns of sediment samples in Negombo Lagoon (M: montmorillonite, I: illite and Q: quartz).

Clay minerals can be used as paleoclimatic indicators (Singer, 1980, 1984; Sedov *et al.*, 2003; Tabor & Myers, 2015). For example, montmorillonite and illite clay minerals assemblage can be considered as indicators for warm/dry paleoclimate and/or derived from near distance sources (e.g. Singer, 1980, 1984; Andrzejewski & Tabor, 2020). Therefore, an abundance of illite, montmorillonite, and quartz peaks (Figure 4) indicate the deposition of immature sediments (i.e. limited chemical weathering materials) from near distance sources.

FTIR observation

Figure 5 shows the representative FTIR spectrum of surface sediment samples (0–2 cm in depth) in Negombo Lagoon. FTIR spectrum in Figure 5 indicates main functional groups such as (a) C-O stretching of polysaccharide (1040-1090 cm^{-1}), (b) carboxylate ions (1720 cm^{-1}), (c) aliphatic substances (2850-2950 cm^{-1}), and (d) H bonded OH groups/amines (3200-3600 cm^{-1}). Consequently, the above-mentioned functional groups indicate a breakdown of complex sedimentary organic matter into long chains of hydrocarbon molecules (Meyers, 2003; Ratnayake *et al.*, 2019a). Similar functional groups (e.g. carboxylic groups, carboxylate ions, polysaccharide) can be identified in sediment core samples, but with different intensities (Figure 6). For example, the absorbance of carboxylic groups (about 0.1 absorbance units) and carboxylate ions (about 0.02 absorbance units) decrease with depth due to early diagenesis (Meyers & Ishiwatari, 1993; Sampei *et al.*, 1997; Chen *et al.*, 2017). Therefore, carboxylic groups/

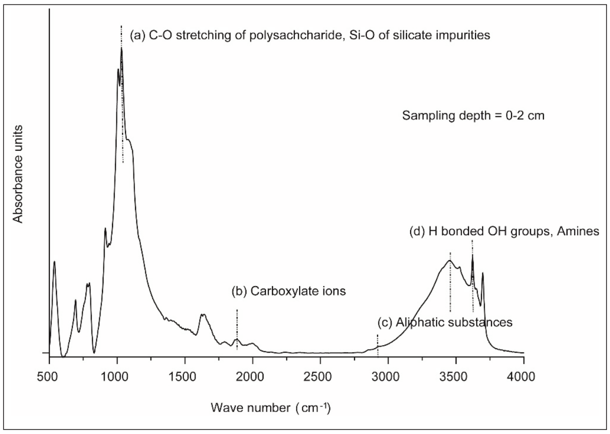

Figure 5: Representative FTIR spectrum of surface sediment samples (0–2 cm in depth) in Negombo Lagoon.

carboxylate ions can be decomposed preferentially compared to polysaccharide (Kruminset *al.*, 2012). In contrast, the lower sedimentary succession shows drastic enhancement of the absorbance of carboxylic groups/carboxylate ions between 48 cm to 50 cm in depth, may be due to sudden accumulation of highly decomposed organic matter from the terrestrial sources (Meyers, 1997, 2003).

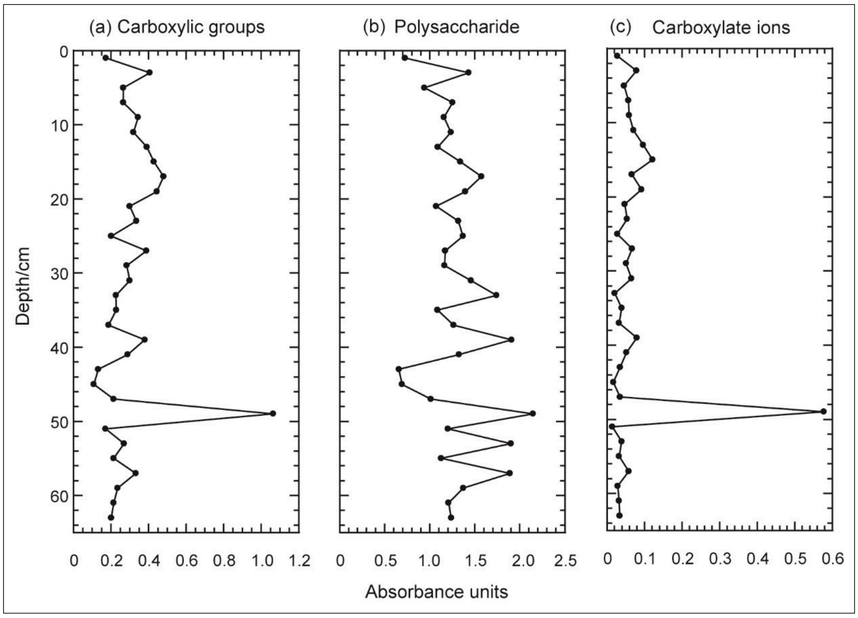

Figure 6: Vertical variations in absorbance of different functional groups based on FTIR spectra.

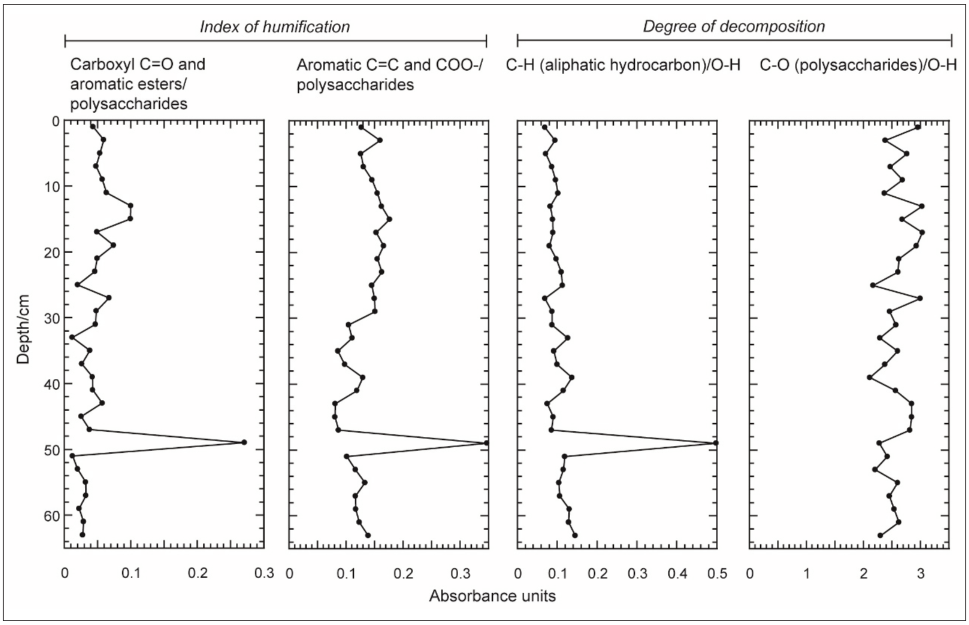

Figure 7: Humification index and degree of the decomposition based on FTIR results.

Degree of humification and decomposition

Figure 7 shows the ratio of 1630 cm^{-1}/1053 cm^{-1} (aromatic C=C and asymmetric COO-/polysaccharides) in Negombo Lagoon sediment. This ratio can be used as a humification index (i.e. the formation of humic acids) in sediments (Biester *et al.*, 2014 and references therein). Besides, the humification index is decreased slightly (about 0.04 absorbance units) with the depth suggesting a slow rate of transformation of complex organic matter into simple carbon substances. In this study, the ratios of 1720 cm^{-1}/1053 cm^{-1} (carboxyl C=O and aromatic ester group/polysaccharides) also indicate a parallel trend with the humification index (Figure 7), and thus can be proposed as possible novel humification index along with traditional proxies. According to the literature, several geochemical methods have been used to understand the degree of humification and decomposition of organic matter. For example, leachate's colour intensity based on UV absorption measurements (Bahnson, 1968), colourimetric method (Chambers *et al.*, 2011), Rock-Eval pyrolysis (Outridge & Sanei, 2010), C/N ratios (Kuhry & Vitt, 1996), stable isotopes (Jones *et al.*, 2010), and FTIR spectroscopy (Niemeyer *et al.*, 1992; Broder *et al.*, 2012) have been used to estimate the degree of humification and decomposition.

The ratios of 2850–2950 cm^{-1}/3200–3600 cm^{-1} (C-H/O-H: asymmetrical aliphatic hydrocarbon/O-H stretching of alcohols) and 1040–1090 cm^{-1}/ 3200–3600 cm^{-1} (C-O/O-H: C-O polysaccharides/O-H stretching of alcohols) can be used as indicators for the degree of decomposition (Krumins *et al.*, 2012). Therefore, Figure 7 suggests weak decomposition rates during the early diagenesis. However, decomposition and humification rates are drastically increased at 48–50 cm in depth (Figure 7), suggesting rapid accumulation of highly decomposed organic matter by short-lived anthropogenic or natural process (Meyers, 1997, 2003).

CONCLUSIONS

Negombo Lagoon is less susceptible to aquatic deterioration due to minor impacts of sewage/agricultural nutrient contamination based on δ^{15}N stable isotope composition and organic- and carbonate-carbon distributions. Negombo Lagoon sediments are characterised by the deposition of C_3 terrestrial plant dominant sources and marine sources as marine dissolved organic carbon. In addition, a mineral assemblage containing illite, montmorillonite, and quartz peaks can be interpreted as limited chemical weathering materials from near distance sources. This sedimentary succession indicated weak and nearly constant decomposition rates (i.e. weak microbial decomposition) under the anoxic/reducing depositional condition. In this study, the ratio of 1720 cm^{-1}/1053 cm^{-1} (i.e. carboxyl C=O and aromatic ester group vibrations to polysaccharides) was proposed as a novel humification index along with traditional proxy in the literature.

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