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Ca/Al and Ca/Fe as Indicators of Terrigenous and Marine Origin in East Coast Peninsular Malaysia During Holocene

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ABSTRACT

A coastal area is an area that is very exposed to sea level changes for a long period, especially during the Holocene. Three marine sediment cores (TRC 3 and TER16-GC13C) in the offshore area were analysed for radiocarbon (^{14}C) and some geochemical elements (Na, Al, Ca, Fe, Li, Pb, Zn, and Ba) in response to the terrigenous-and marine-originated sediment changes of in East Coast Peninsula Malaysia (ECPM) during the Holocene epoch. Across three Holocene stages, geochemical elements show a distinct temporal pattern. The average concentrations of the studied metals were in decreasing order as follows: Al > Na > Ca > Fe > Pb > Zn > Li > Ba. Dominant terrigenous/marine origin was captured by the ratio Ca/Al and Ca/Fe in the early Holocene/late Holocene. The present study suggests that sea level transgression is the primary factor controlling the shifting of terrigenous- and marine-originated sediments in the ECPM.

Keywords: South china sea, Sunda shelf, Geochemical elements, Radiocarbon dating, Sea level.

INTRODUCTION

The Holocene epoch is the geological timescale period from 11,700 years ago to the present, divided into early, mid, and late Holocene[1,2,3,4]. A gradual increase in sea level after the Last Glacial Maximum marked the early Holocene period[5]. In the mid-Holocene period, there was a significant cooling trend caused by alterations in ocean circulation resulting from glacial melting and a large amount of freshwater entering the system. The late Holocene ranges from 4200 Cal yrs BP to the present day. Paleoenvironmental studies have focused on understanding the Holocene environment because of the period's importance for grasping the current climate system and predicting future global changes[1,2]. Thus, marine sediment geochemistry can offer valuable information about climate and ocean changes[3].

Sediment records from the ocean floor have allowed scientists to analyse and reconstruct information about past climate patterns and types of sediment in area[6,7]. Aluminium (Al) and Iron (Fe) in marine sediments are primarily linked to terrigenous detritus[8]. Both elements are called terrigenous elements because they mainly originate from land. Thus, fluctuations in the input of land-derived material can be assessed by examining the composition of those elements[9,10]. The Ca/Al and Ca/Fe ratios in marine sediments offer information on the balance between marine- and terrigenous-originated materials[11,12].

According to Tam *et al.,* (2018), the sea level in the East Coast Peninsula Malaysia (ECPM) rose rapidly during the early Holocene with an average rate of 16.2 ± 4.5mm. Between 9000 and 5000 cal a BP, the rate of rise gradually slowed until it attained zero. Since the mid-Holocene, the relative sea level (RSL) dropped to -0.60 ± 0.12m by 800 years. The current phase is marked by a reduced sedimentation rate and increased amounts of carbonate[14]. The Previous studies indicate that the RSL in Peninsular Malaysia peaked around 5000 to 6000 cal yr BP at ca. +5 metres before gradually decreasing to its current level[13,15].

Therefore, the main goal of this study is to understand the response of Ca/Al and Ca/Fe ratios on the terrigenous- and marine-originated sediments in the ECPM during the Holocene period. Analyzing the distribution of major and trace elements in marine sediments has increased the utilization of inorganic indicators for studying past environments. Our study's results enable us to evaluate the response of elemental composition to sea level changes in the ECPM during the Holocene. This information sheds light on the historical patterns of environmental changes.

MATERIALS AND METHODS

Sample Collection

In the present study, two sediment cores, TER16-GC13C (5°39.99' N, 104°15.63' E, 180 cm long at 73m water depth) and TRC3 (5°30.19'N, 103°32.94'E, water depth 58.3m, 158 cm in length) were collected by UMT Research Vessel RV Discovery in Terengganu during the 2016 and 2017 cruise in the East Coast Peninsular Malaysia (Fig. 1). The core samples were brought to the Institute of Oceanography and Environment (INOS) for further treatment and stored at a low temperature to decrease chemical and biological reactions.

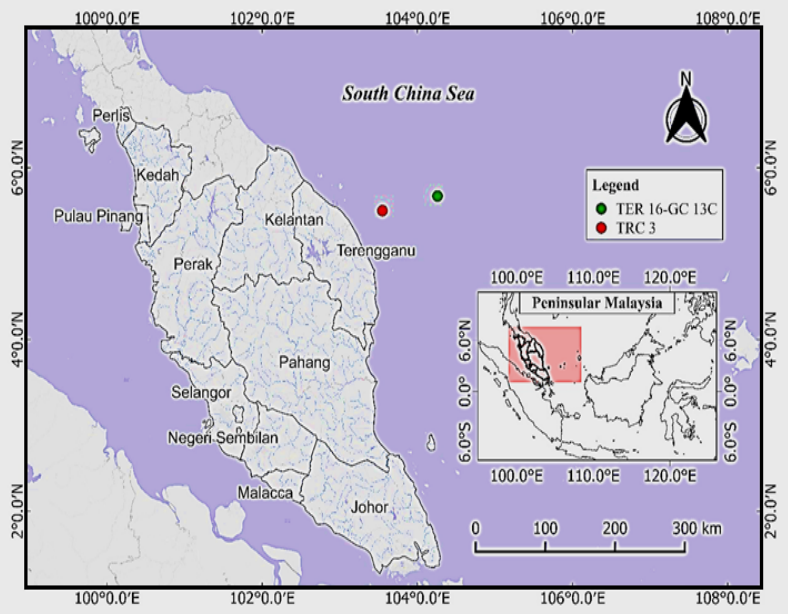

Fig. 1. Map showing the Sampling Locations for TRC3 and TER16-GC13C on the East Coast of Peninsular Malaysia

Radiocarbon dating

The complete shell fragments obtained from different depths of the cores TER16GC13C and TRC3 were used for radiocarbon dating. The shells were identified using the Natural History Museum Rotterdam online database and then sent to International Chemical Analysis (ICA) in Miami, Florida, the United States of America, for radiocarbon dating. The conventional ages obtained were converted into cal years BP using the software Calib 8.1[16] and updated MARINE 20 dataset[17] with reservoir effect ΔR = -155, based on the location of the study area (Table 1). All ages are given in calendar year BP (cal yr BP). Linear interpolation is employed to approximate ages at other locations in AMS dating close by, assuming a consistent sedimentation rate[18]. This method entails graphing radiocarbon or calibrated ages versus depth and connecting adjacent dots with straight lines.

Table 1: The value of accuracy analysis for standard reference.

Metals	NBS1646a (ppm)	Measured Value (ppm)	Accuracy Test (%)
Al	2.297	2.2	95.78
Ca	0.519	0.510	98.26
Fe	2.008	1.66	82.67
Pb	11.7	10.02	85.63
Zn	48.9	48.62	99.42
Ba	210	181.61	86.48

Elemental Composition Analysis

The samples were digested and analysed using the modified methods of Kamaruzzaman *et al.,* (2008) and Noriki *et al.,* (1980). Approximately 0.25 g of powdered sediment samples were placed in a Teflon vessel and digested in mixed acid of 1 mL perchloric acid ($HClO_4$), 1 mL hydrogen peroxide (H_2O_2), and 6 mL nitric acid (HNO_3) in a microwave digester. The digestion process was set at the temperature of 100°C for 7 hours. After cooling, the clear digested sample was transferred into a 50 mL centrifuge tube, and added with milli-Q water to 50 mL. After an appropriate dilution, the samples were analysed for elemental composition by a Perkin Elmer ELAN 9000 Inductively Coupled Plasma Mass Spectrometer (ICP-MS) in the Institute of Oceanography and Environment (INOS), Universiti Malaysia Terengganu (UMT). The accuracy of the analytical procedure was examined by analysing a standard research material (SRM) NBS1646a estuarine sediment in duplicate and blank as control. The recovery test coincided with the certified values of NBS1646a (Table 2). The precision test of six elements in the certified value of NBS1646a ranged from 82.67% to 99.42%.

Table 2: The established radiocarbon ages (cal year BP) of cores TRC3 and TER16-GC13C based on the intact shell fragments

Sediment core	Depth (cm)	Material	AMS ^{14}C age (year)	Calibrated age (cal age year BP)
TRC3	22.5	shell	2080+/-40	1508
TRC3	37.5	shell	3190+/-40	2869
TRC3	63.0	shell	3880+/-40	3687
TRC3	153.0	shell	9550+/-40	10296
TER16-GC13C	32.0	shell	2560+/-40	2249
TER16-GC13C	44.5	shell	3920+/-40	3927
TER16-GC13C	77.0	shell	6310+/-40	6734
TER16-GC13C	167.0	shell	7370+/-40	7799

RESULTS

Age of Sediment

Based on the results obtained, the studied core samples recorded the Holocene epoch (Table 2). Core TRC3 located in the waters of the nearshore area of Terengganu represents the entire Holocene epoch (1508 – 10296 cal yr BP), and TER16-GC13C located in the offshore area of Terengganu indicates the mid to late Holocene (2249 – 7799 cal yr BP). This implies that both core samples depict the environmental transition within the Holocene epoch.

Geochemical Elements Concentration

The elemental compositions were varied in the core sediments TRC3 (Fig. 2) and TER16-GC13C (Fig. 3). The major elements are Na, Al, Fe, and Ca, whereas minor elements consist of Li, Pb, Zn, and Ba. The mean concentrations of the studied metals were in decreasing order as follows: Al > Na > Ca > Fe > Pb > Zn > Li > Ba. In the TER16-GC13C and TRC3 cores, Al was the most abundant major element, with average compositions of 9.41 ± 2.82% and 5.23 ± 2.07%, respectively. Sodium had the lowest content, with percentages of 0.06 ± 0.05%, 0.86 ± 0.26%, and 1.30 ± 0.45% in TER16-GC13C and TRC3. Calcium content was most significant in the KELC17 sediment core compared to TER16-GC13C and TRC3. In TER16-GC13C, Ba is the predominant trace element with a mean concentration of 185.02 ± 42.49 ppm. In

TRC3, Ba has an average concentration of 131.09 ± 43.42ppm. The lowest composition was Pb, with average compositions of 22.00 ± 6.27 ppm and 17.70 ± 3.62ppm.

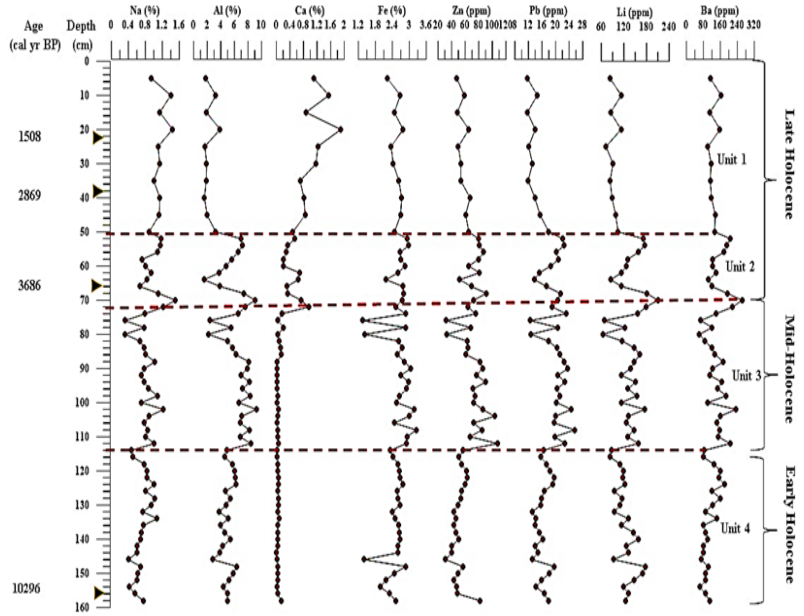

Fig. 2. Variation of Studied Elements in TRC3 Sediment Core during the Holocene

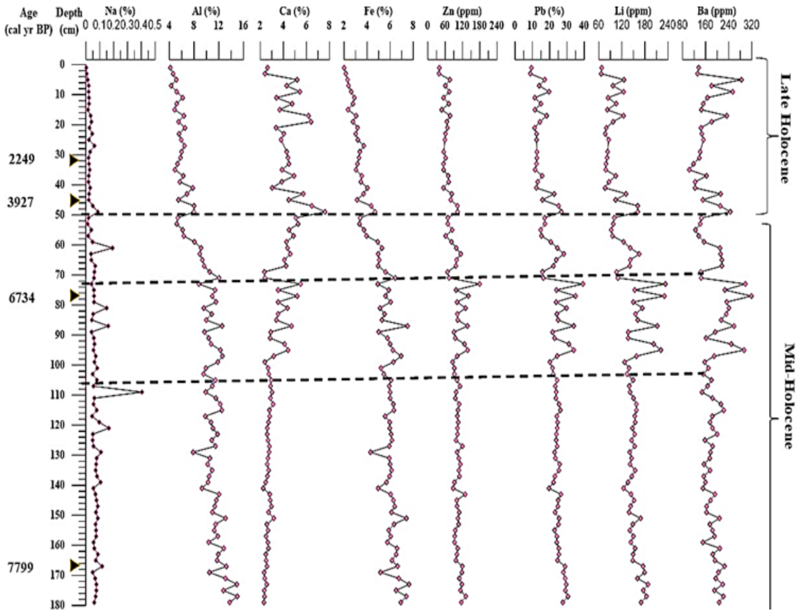

Fig. 3. Variation of Studied Elements in TER16-GC13C Sediment Core during the Holocene

Most elements had similar patterns during the Holocene, except Ca, which demonstrated an opposite pattern compared to the other elements. The TRC3 sediment core from the early Holocene is distinguished by a substantial presence of terrigenous elements, including Al, Fe, Pb, Li, Zn, and Ba. During the mid-Holocene, the concentration of these elements had a minor increase, followed by a decline in the late Holocene. The calcium content showed consistently low levels over the core, varying from 0.01 to 1.9%, with an average of 0.29%. The data showed an opposite relationship, with the lowest concentration observed during the early Holocene and the highest composition during the late Holocene. A comparable trend was noted in TER16-GC13C throughout the mid to late Holocene, as this sediment core represented only this period.

Elemental Ratios (Ca/Al and Ca/Fe)

The ratios of Ca/Al and Ca/Fe derived from cores TER16-GC13C and TRC3 showed a similar trend, rising from the early Holocene to the late Holocene (Fig. 4). The TRC3 sediment core showed a mean Ca/Al ratio of 0.010 ± 0.005 in the early Holocene, which rose to 0.012 ± 0.010 in the mid-Holocene and then increased significantly to 0.29 ± 0.24 in the late Holocene. The average Ca/Al ratio of core TER16-GC13C rose from 0.32 ± 0.15 during the mid-Holocene to 0.79 ± 0.19 in the late Holocene. In TRC3, the average Ca/Fe ratio was 0.020 ± 0.0099 in the early Holocene, increased to 0.026 ± 0.019 in the mid-Holocene, and peaked at 0.29 ± 0.18 in the late Holocene. In TER16-GC13C, the ratio rose from 0.58 ± 0.24 in the mid-Holocene to 1.49 ± 0.38 in the late Holocene.

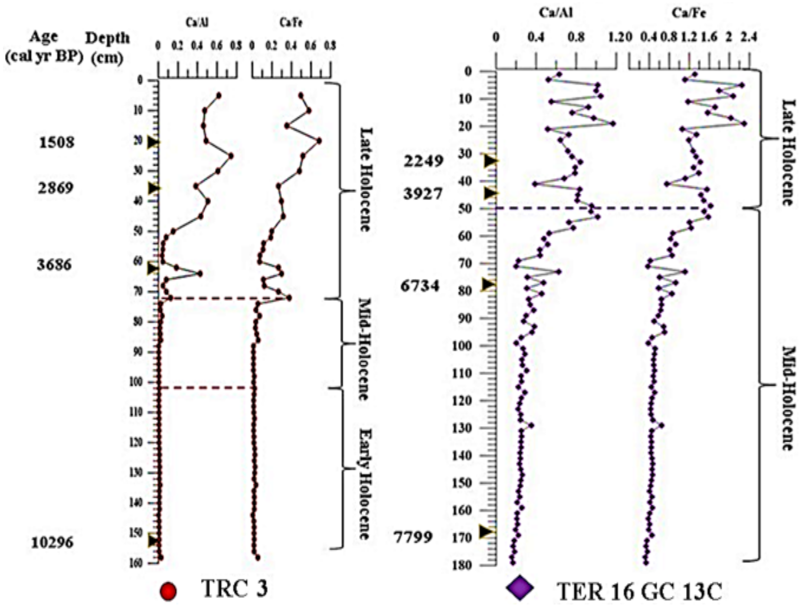

Fig. 4. Ca/Al and Ca/Fe Profile of TRC3 and TER16-GC13C sediment cores during the Holocene

DISCUSSION

Correlation between studied metals

SPSS version 28 statistical software was applied to study the correlation between the variables. In this study, the correlation and significance level of $p < 0.05$ was used to determine the magnitude and direction of the linear relation. The findings are presented in Tables 2, and 3. Statistical analysis was frequently used to determine the relationship between multiple variables to understand the factors and the sources of chemical components[21]. The magnitude of the correlation between the variables ranged from weakly negative to positively significant. Most elements, including Al and Pb, Na and Ba, Al and Li, Fe and Zn, Zn and Pb, and Pb and Li, exhibited significant positive correlations, indicating that they originated from comparable sources. The correlation among Al, Pb, and Li suggests a connection with aluminous clay minerals. Calcium which is a biogenic element or normally related to marine-originated material showed a weak or negative correlation with Al, Fe, Zn, Li, and Pb, suggesting they are derived from different sources[22,23].

Table 3: Pearson's Correlation Matrix for the Studied Elements in TRC3 Sediment Core

Element	Na	Al	Ca	Fe	Zn	Pb	Li	Ba
Na	1							
Al	.180	1						
Ca	.663	-.447	1					
Fe	.520	.599	.001	1				
Zn	.418	.676	-.028	.707	1			
Pb	.167	.901	-.379	.672	.792	1		
Li	.317	.808	-.205	.561	.516	.727	1	
Ba	.782	.652	.210	.594	.683	.616	.633	1

Correlation is significant at the $p<0.05$

Table 4: Pearson's Correlation Matrix for the Studied Elements in TER16-GC3C Sediment Core

Element	Na	Al	Ca	Fe	Zn	Pb	Li	Ba
Na	1							
Al	.425	1						
Ca	-.204	-.510	1					
Fe	.489	.979	-.483	1				
Zn	.398	.736	-.127	.743	1			
Pb	.422	.771	-.112	.778	.963	1		
Li	.380	.753	-.084	.748	.946	.982	1	
Ba	.136	.387	.302	.323	.687	.732	.791	1

Correlation is significant at the $p<0.05$

The Response of Geochemical Elements to Holocene Sea Level Changes

The sedimentary records in this study exhibit significant changes over time, as shown by variations in elemental compositions. Sea level fluctuations significantly affect the transportation of land-derived sediments in coastal regions[24]. Mann *et al.,* (2019) observed an increase in sea level in the Malay-Thai Peninsula of 33m mean sea level (MSL) between 10,500 and 7000 cal yr BP. The mid-Holocene high stand occurred between 6000 and 4000 cal yr BP, reaching an estimated 2 to 4 metres above MSL. Tam et al. (2018) reported a significant increase in sea level from 10,500 cal yr BP at 35 ± 4.3 m to 9000 cal yr BP at -14.2 ± 1.6 m, with an average growth rate of 15.8 ± 3.9 mm/yr in the ECPM. From 9000 to 5000 cal yr BP, the rate of relative sea level decreased steadily until it stabilized at approximately 3.3 ± 0.2m. The sea level declined from 5000 cal yr BP to a minimum of -0.6 ± 0.1m. The present study results aligned with those of Mann *et al.,* (2019). These environmental conditions have significantly impacted the sediment pattern in the tropical waters of ECPM.

The significant rise in sea level led to a reduced amount of land-derived material reaching the TRC3 core, probably due to the greater distance from the coastline. However, the concentrations of terrigenous elements (such as Al, Fe, Pb, Li and Zn) in the TRC3 sediment core suggest a notable terrigenous input, possibly due to increased precipitation[24,26]. Consequently, the variation in terrigenous input during this period was influenced by fluctuations in sealevel[27,28]. The low Ca content and minima Ca/Al and Ca/Fe ratios indicate an increased influx of terrigenous materials, which dilute the marine-originated materials during this period[12,29].

The slightly higher concentration of terrigenous elements observed at this stage in the TRC3 core compared to the early Holocene (Fig. 2) indicates a higher volume discharge of terrigenous materials during this interval. This is due to the beginning of the mid-Holocene, the rate of sea level rise slowed down and reduced to zero at about 5000 cal yr BP in the ECPM13[,15,27]. Consequently, the terrigenous inflow was modulated by the remarkably stable sea level[26,28]. The low levels of terrigenous elements in this period were likely caused by a decrease in the influx of terrigenous materials. Conversely, the elevated Ca and the Ca/Al and Ca/Fe ratios indicate an increase in organic matter input, reducing terrigenous elements[29].

Higher Ca in the upper core may be due to the complete or partial dissolution of minerals during sediment diagenesis or variations in surface productivity in the past[28,30,31]. Sediment diagenesis is the physical and chemical changes that change the characteristics of sediment after deposition processes[32]. Furthermore, the different Ca patterns shown during the Early Holocene to Late Holocene period indicate high and low energy changes in the delivery of calcareous material between the soft and hard tissue microorganisms.

We suppose that in-situ production of marine products was more extensive in the late Holocene and this is in line with the increasing pattern of Ba. Barium is usually used as a proxy to determine paleoproductivity in the marine environment. Liguori *et al.,* (2016) stated that Ba is a trace element that occurs predominantly as a barite mineral ($BaSO_4$) in the marine environment. Therefore, the trend of selected geochemical elements in the marine sediment cores of ECPM is significantly related to environmental changes that occurred from the early to late Holocene, which is from terrigenous (early Holocene) to marine (late Holocene) dominated environment.

CONCLUSION

The investigation into sediment cores retrieved from the Terengganu waters in the East Coast Peninsula Malaysia (ECPM) has provided valuable insights into the changes of terrigenous-marine-originated sediment throughout the Holocene epoch. Based on the data of TRC3 and TER16-GC13 Ccores, we have effectively traced the evolution of studied geochemical elemental compositions which related to the environmental change. Two significant trends were observed: a gradual decrease in terrigenous origin and an increase in marine origin from the early to late Holocene. The geochemical element shifts bear witness to the intricate interplay of sea level transgression. The study provides insight, enhances understanding of past climatic and oceanography conditions in the ECPM, and contributes to the discussion surrounding Holocene climate dynamics in tropical waters.

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Conflict of interest

The authors declare no competing financial interest

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