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Variation characteristics of ocean sediment Fe levels and their relationship with grain sizes in culture areas over a long period

Baoyi Zhang[1], Manyi Li[1], Maoguo An[2], Chenglong Zhi[2], Qingcai Li[2], Yingmei Zhang[2], Shuangyuan Han[2], Haitao Zheng[2], Jun Li[2], Huaixin Chen[2] and Qiao Chen[3*]

Abstract

Iron (Fe) is an essential component for marine ecosystems, and it is related to the growth of phytoplankton communities and environmental evolution in coastal area. However, the effect of aquaculture activities on sediment Fe levels is not well studied. Fe levels and grain sizes are determined in two cores (respectively Core C in the culture area and Core A in the control area) in Sishili Bay to reveal the influence of cultivation on sediment Fe levels over an extended period. The sediment Fe levels are distinguished in the upper sections (culture period) but equal in the lower sections (non-culture period) of the two cores. The core C has the same Fe levels as Core A before 1950s (non-culture period). However, the sediment Fe levels of Core C increased during 1950s–1970s (the algae culture period) and decreased after the 1970s (shellfish culture period) compared with Core A, indicating the algae and shellfish culture impose opposite effects on sediment Fe levels. Similarly, sediment grain sizes are observed to be finer during the algae culture period but coarser during the shellfish culture period, and the variation of sediment grain sizes because of culture activities is the important factor affecting sediment Fe levels. The slowing down of ocean current due to algae culture causes finer particles and higher Fe levels in sediment. However, during the shellfish culture period, bio-deposition and re-suspension play major roles in coarsening sediment particles and decreasing sediment Fe levels.

Keywords: Sediment Fe levels, Sediment grain sizes, Culture activities, Sishili Bay

Background

Marine culture activities have seen an increase during the last several decades due to the increasing demands for aquaculture products and the need for seafood supplies [1, 2]. In China, almost 70% of marine products are supplied by marine culture, with the annual output of more than 2×10^7 tons in recent years [3]. These culture activities deteriorate marine environment by changing the geochemical balance of the ecological system. Therefore, the influence of culture on oceanic systems and its mechanisms have recently received great attention.

Iron (Fe) ranks as the fourth most abundant element in the earth's crust, and approximately half of the global surface Fe is delivered into the ocean by runoff [4]. Fe is an essential nutrient and energy source for the growth of microbial organisms as many Fe-containing enzymes facilitate the transfer of electrons used to generate chemical energy in cells [5]. Ocean Fe deficiency limits the phytoplankton to use the excess carbon, sulfate, nitrate and phosphate [6]. Therefore, bioavailable Fe has been recognized as an important element for phytoplankton to synthesize chlorophyll and metabolize, and as the crucial factor controlling ocean primary productivity [7, 8].

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*Correspondence: qchen5581@163.com
[3] Shandong Provincial Key Laboratory of Depositional Mineralization & Sedimentary Minerals, College of Earth Science & Engineering, Shandong University of Science and Technology, Qingdao 266590, Shandong, China
Full list of author information is available at the end of the article

Bioavailable Fe in coastal area is generally higher than that in ocean. However, the physiological character and Fe-absorption mechanism of phytoplankton communities in coastal area differ greatly from those in ocean, and Fe in coastal area also serves as the limitation factor of the growth of phytoplankton. Sunda and Huntsman [9] observed the Fe-demand of diatom in coastal area is more than 100 times of that in ocean. Primary productivity along the coastal area was also found to be limited because of Fe-deficiency, such as in California [10]. Moreover, Fe content has also been widely proved to be the inducer of red tide and eutrophication in coastal area [11, 12]. Thus, Fe content and its variation are deeply related to coastal environmental evolution.

Although a series of environmental indicators, such as heavy metals, benthic macrofaunal assemblages and bacterial diversity, is discussed in culture areas [13–15], the influence of marine culture on Fe geochemistry and its interactions with other elements remain poorly understood. Specifically, there is hardly any information about Fe variation for a long term culture period.

The Fe levels in ocean sediments are governed by pH, organic complexation, redox state, bacteria, sulfur and other parameters [16]. Sediment grain size is one of the most basic but important parameters in determining Fe contents. Total Fe ions are generally found to be concentrated within the finest fractions, and multiple researchers have argued the sediment Fe levels are significantly correlated with the grain size [17, 18]. In fact, some factors, which are responsible for Fe levels, are also reported to be co-correlated with sediment grain size. For example, Ma et al. [19] found that clay is viewed as the primary control in sediment Fe, Mn, and organic carbon. Karine et al. [20] found that Fe and organic carbon co-vary, and both Fe and organic carbon are commonly related to clay mineral surfaces. Thus, sediment grain size plays the most fundamental role in determining Fe levels. Therefore, the sediment grain size should be of concern when the mechanism of sediment Fe level is researched.

Sishili Bay is in the northeastern of Yantai City, Shandong province, China. And it is located in the northern Yellow Sea. Sishili Bay is one of the important fishery base and culture area in China. It has a culture history of over 70 years. Especially, it is famous for scallop culture, accounting for more than 70% of the production in Shandong province. In this regard, two cores (in culture area and non-culture area) in Sishili Bay were sampled. The sediment Fe levels and grain sizes were analyzed, aiming to: (1) clarify the differences of Fe and grain sizes in the sediment cores during culture period and non-culture period; (2) analyze the relationship between sediment Fe and grain sizes and their variation dynamics; and (3) discuss the effect mechanism of culture activities on sediment Fe levels.

Study area and methods

Study area and culture activities

The studied area, Sishili Bay, is in Yantai City in the northeastern Shandong province, China. It is connected to the northern Yellow Sea. The area is a typical semi-enclosed coastal bay surrounded by Yangma Island and Zifu Island (Fig. 1). It extends 20 km from the northwest to the southeast, and is about 6–7 km wide. The water depth varies from 0 to 15 m.

Sishili Bay is deeply influenced by human activities, such as population growth, industry, shipping, fertilizer use and sewage discharge. Eutrophication has significantly influenced the health of the marine ecosystem. Harmful algae and jellyfish in the bay have frequently bloomed over the recent years [21].

Sishili Bay has experienced a long history of cultivation, and is still the most important culture area in the north of China. The culture activities in Sishili Bay began early in the 1950s, dominated by *Laminaria* and *Wakame* culture [22]. Shellfish culture developed in the 1970s, and then intercropping or rotary culture of shellfish and algae was gradually popularized [23]. The *Laminaria* almost went extinct from white rot disease in 1998 [22, 24], and bivalve culture dominated since then, especially the *bay scallop* and *chlamys farreri*. The maximum culture area in Sishili Bay historically reached 2450 ha, of which the area for scallop, mussel and seaweed was 800, 400 and 250 ha, respectively [21].

Sampling and analyzing methods

Two cores were sampled using a gravity corer in 2009. Core A is from the offshore area without culture and human activities, at the geographical coordinate of N37°36′24.00″, E121°33′59.00″. The depth of Core A is 122 cm and the water depth is 10.5 m. Core C is from the culture area, at the geographical coordinate of N37°32′52.38″, E121°27′17.32″. Core C is in the center of culture area. The depth of Core C is 119 cm and the water depth is 11.2 m (Fig. 1). The core sediments were sent to laboratory and sectioned at 1 cm interval on the same day. Part of the sediments were freeze-dried for Fe, Ca level analysis, and chronology was done by ^{210}Pb and ^{137}Cs radiometric-dating techniques. Remaining samples were used to analyze grain size.

^{210}Pb and ^{137}Cs were determined by gamma-ray spectrometry using Ge detectors (Model Ortec HPGe GWL) at the Institute of Geography and Limnology, Chinese Academy of Sciences. The Constant Rate of ^{210}Pb Supply model was used to calculate the sedimentation rates. This work referred to Sun et al. [25].

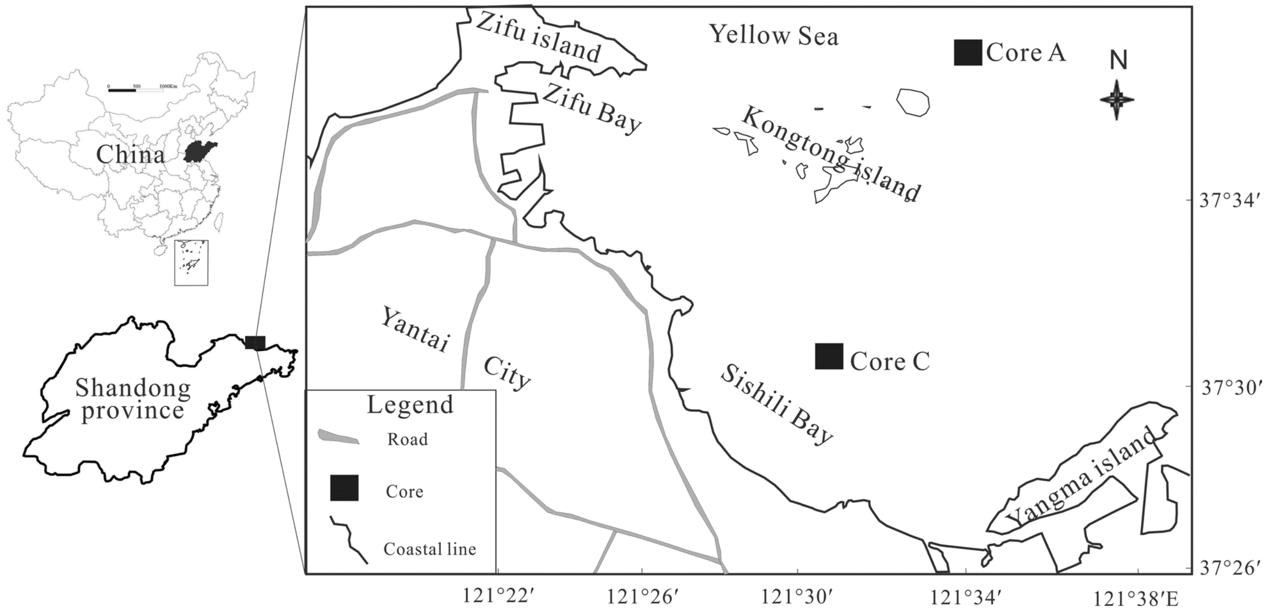

Fig. 1 The map of Sishili Bay and the sampling sites of sediment cores

The sediment sample was dried, slightly dis-aggregated, and sieved through a 200 mesh before element tests. Fe and Ca levels were analyzed using Axios-Advanced X-ray Fluorescence Spectrometry (PW 4400 model, with detection limit of 10^{-5}–10^{-6}, 1–10 ug/g). Fe and Ca levels are in weight percentage (%). The parallel samples and national reference samples (GSS-8 and GSD-12) were used to test the precision and accuracy, and the relative standard deviation was less than 5%. The recovery rate of this analysis is above 95%.

An appropriate amount of sediment was placed in tubes, and 2 ml HCl was used to remove carbonate. Subsequently, 10 ml of 10% H_2O_2 was added and the tubes were boiled at 60 ℃ for 2 h to remove organic matter. Some deionized water was added and the solution stood for 12 h and centrifuged. Finally, a 0.05% $(NaPO_3)_6$ solution was used to disperse the solution for 12 h and the solution was ultrasonicated for 10 min before the analysis. Guaranteed reagents (GR) are used for this analysis. The grain size was determined using a Mastersize 2000 Laser Particle Sizer. The grain sizes are in the unit of mass percentage (%). The grains were divided into four groups: clay (< 4 μm), fine silt (4–16 μm), coarse silt (16–64 μm) and sand (> 64 μm).

Result

The difference of sediment Fe levels in culture area and non-culture area

The sediment Fe levels are 3.00–3.89% in core A and 3.12–3.68% in core C respectively. The average Fe level of core A is 3.45%, while that of core C is 3.37%.

Figure 2a shows the variation of sediment Fe levels over time. In core A, the Fe levels in the lower sections (> 57 cm, before 1949) remain almost stable with slight fluctuations, and Fe levels suddenly decrease in the 23–57 cm sections (1949–1982) and increase in the upper section (< 23 cm, after 1982). While in Core C, the Fe levels in the lower section of core C (> 37 cm, before 1970s) remain stable, and those in the upper section slightly decrease. Comparing the Fe levels in the two cores of the same year, the two cores almost have the same Fe levels in the sections before 1946 (non-culture period). However, core C shows higher Fe levels in the 31–49 cm sections (1949–1971, algae culture period), and lower ones in the 0–31 cm sections (after 1971, shellfish culture period) than core A. The relative Fe levels approximately show an increase by algae culture and a decrease by shellfish culture.

Sediment grain size characteristics and their relationship with Fe levels

Grain-size fractions of clay, fine silt, coarse silt and sand are in the ranges of 17.42–28.4%, 19.15–35.03%, 33.13–47.38%, 3.59–18.98% for core A, and 15.68–32.0%, 19.27–42.96%, 22.64–48.04%, 2.41–30.64% for core C, respectively. According to the classification of sediment fractions, both cores are categorized as clayed silt.

Figure 2b–e illustrates the variation of sediment grain sizes with depth or time. The coarse silt contents in the two cores remain almost equivalent, only with a slight deviation at the 0–15 cm section. The other three grain

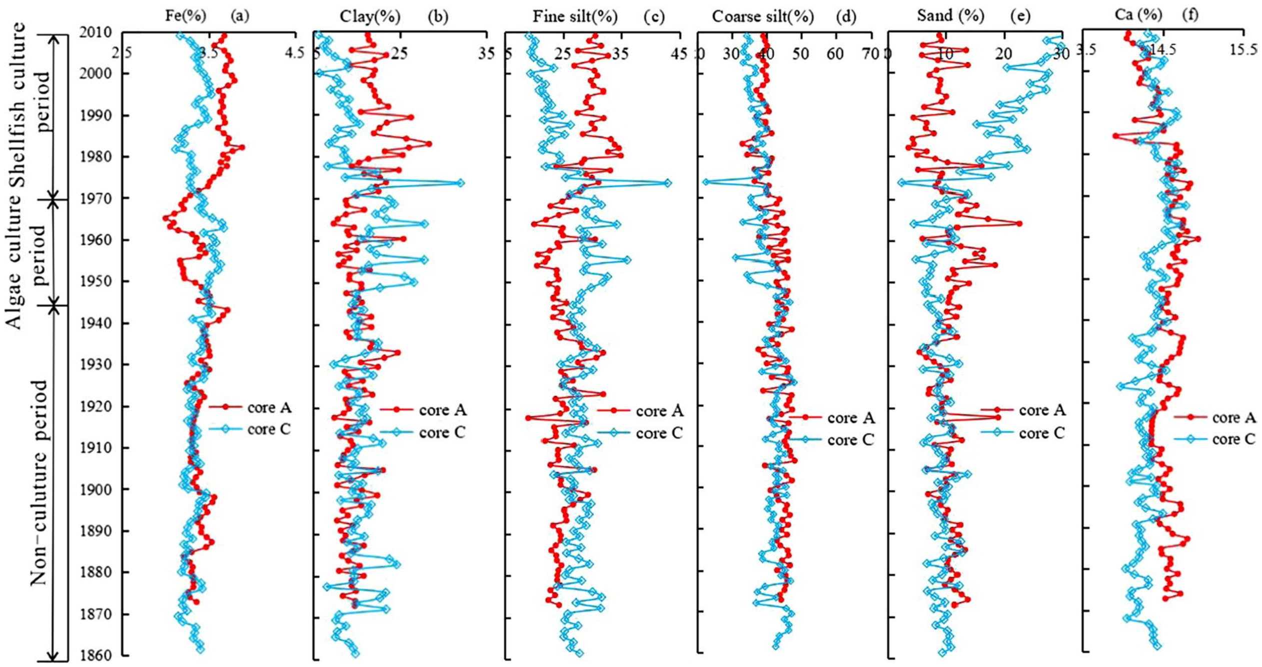

Fig. 2 The Fe, Ca levels ansd grain size characteristics of two sediment cores

size components in both cores show the same contents and variation trends in the lower sections during non-culture period (before 1950s). This means the two cores should have the same grain size components if there were no cultivation. Conversely, core C has significantly higher clay and fine silt amounts, and lower sand amounts than core A at the 31–54 cm sections (1946–1971, algae culture period). Similarly, in the < 23 cm section (after 1971, shellfish culture period), core C has lower contents of clay and fine silt, and higher ones of sand than core A.

Generally, the variation regularity of sediment Fe level is similar to that of sediment grain size. That is, comparing with core A, the grain sizes become finer and sediment Fe levels become higher in core C during 1946–1971 (algae culture period). While after 1971 (shellfish culture period), the grain sizes become coarser and sediment Fe levels correspondingly become lower in core C. Therefore, the variation of sediment grain size seems to determine that of sediment Fe level during culture period, and the finer sediment grain size due to culture activities caused the higher sediment Fe levels, and vice versa.

The Ca, TOC (Total Organic Carbon) levels of sediment cores and their relationship with Fe levels

The Ca levels of the two cores are depicted in Fig. 2f. Core A and Core C have Ca levels of 14–14.93% and 14.07–14.79%, with an average of 14.5% and 14.36%, respectively. The two cores have almost the same Ca levels. Significantly, the two cores have no obvious deviation of Ca levels during culture periods (the upper section). Chen et al. [26] reported two cores (also one in culture area and one in non-culture area) in Sishili Bay, and their locations were near our observed cores. The TOC levels of the two cores are presented in Fig. 3 according to his results. Approximately but not accurately, the obvious deviation of sediment TOC levels in upper sections between two cores also occurs. The core A nearby seems to have higher TOC contents in the < 20 cm section and lower ones during the 30–70 cm section than core C nearby. The reason may be that Fe and TOC are both governed by the same factor, grain sizes. While, Ca levels in the two cores have no such variation tendencies because Ca levels are hardly influenced by grain sizes.

Discussion

The influencing factors of marine culture on Fe levels

Marine culture has developed rapidly during the last several decades, and the effect of culture on marine ecosystems has also been widely investigated. The general method is to compare it with a control area [21, 27]. However, the contribution of other factors, such as source, ocean current, sediment transport and environments, cannot be easily excluded. Moreover, only surface sediments are studied, which cannot provide enough information for a long culture period [28, 29].

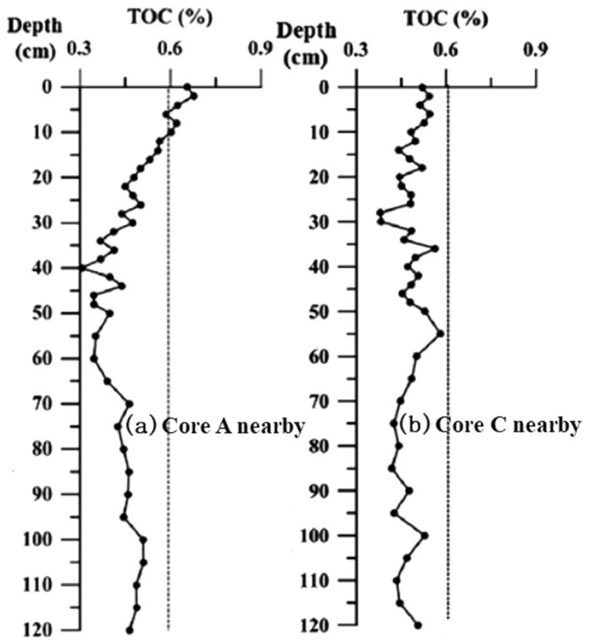

Fig. 3 The TOC of sediment cores near our studied stations (modified after Chen et al. [26])

The two cores for this research have the same grain sizes during the non-cultural period although they are almost 7 km away. Therefore, Core A can be considered as a control to detect the effect of culture activities on sediment Fe levels and grain sizes.

It was observed that sediment Fe levels in culture area (Core C) increased during the algae culture period and decreased during the shellfish culture period if compared with those in the control area (Core A) of the same year. Also, algae culture resulted in fine particles but shellfish culture coarsened sediment grain sizes.

The finer-grained sediments have relatively higher specific surface, surface absorption and ionic attraction [19]. Multiple researchers have confirmed sediment Fe levels are positively correlated with fine particles, and sediment grain size has been considered to be an important parameter controlling sediment Fe levels [16, 17, 19]. We observed sediment Fe levels become higher when sediment particles become finer because of culture activities in this work, and vice versa. So, the variation of sediment grain size evoked by culture activities is the crucial factor affecting sediment Fe level. Besides, the variation laws of TOC in the two cores agree with those of Fe and grain sizes. The finer grain size, the higher TOC and Fe levels. While Ca does not. So, the comparison of sediment Ca and TOC levels also indicates the function of grain size on Fe levels.

The relationship between grain size and local rainfall in core A is illustrated in Fig. 4. During 1946–1971, the average precipitation increases, resulting in higher sand contents than before 1946. While after 1971, the sand contents decrease because of the lower average precipitation. Thus, the rainfall and grain size have the same variation patterns along time, indicating the rainfall determines the sediment grain sizes of the control core A to a great extent. This is in agreement with the previous observation that the higher the precipitation, the coarser particles the sediment has [30, 31]. However, the variation of grain sizes in Core C along time cannot be explained by precipitation because of the disturbance by culture activities.Thus, it can be deduced that the deviation of grain size in the upper section (since 1946) in core C solely results from the cultivation.

The sediment in core C records finer particles and higher Fe levels during the algae culture period (1950s–1970s). Ocean current velocity is frequently documented to be slowed down by algae culture. Johnson et al. [32] tested and found the current velocity in the culture area is just 1/3 of that in the exterior zone. Jackson and Winant [33] theoretically proved the normal current velocity should be $10^{1/(2-3)}$ times of that in culture area. An investigation into the algae culture area of Sungo Bay indicated the surface current velocity degrades by 40% on average [34]. The study in Harny Bay argued the current velocity decreases in winter (*Laminaria* culture season), resulting in less suspended solids when compared with those in summer (non-culture season) [35]. Series of researchers also mentioned the finer particles in algae culture area than in non-culture area [36]. Therefore, the lower current velocity can explain the sediment grain size and Fe levels during *Laminaria* culture.

Similarly, the current velocity is also widely reported to be lower due to shellfish culture [34, 37], even in Sishili Bay. However, our observations concluded the sediment are contrarily coarsened during shellfish culture period, which does not match with the hydrodynamic changes due to shellfish cultivation. The particles may be coarsened by the following reasons: (1) Filter-feeding bivalves, such as shellfish, have strong water-filtration ability, which differs from algae, finfish and crustaceans. The process is also called bio-deposition. Many fine particulate matters are filtered from the water column and ejected as pseudo feces. Kuang et al. [38] found that suspended particles become less in mass and amounts because of the scallop consumption. Since pseudo feces are voided as mucus-bound aggregates, the sediments become larger. (2) Recent studies have revealed the important role of sediment re-suspension as the dominant source of Fe and grain sizes [33, 39, 40]. Shellfish culture area is characterized by balder bed because the

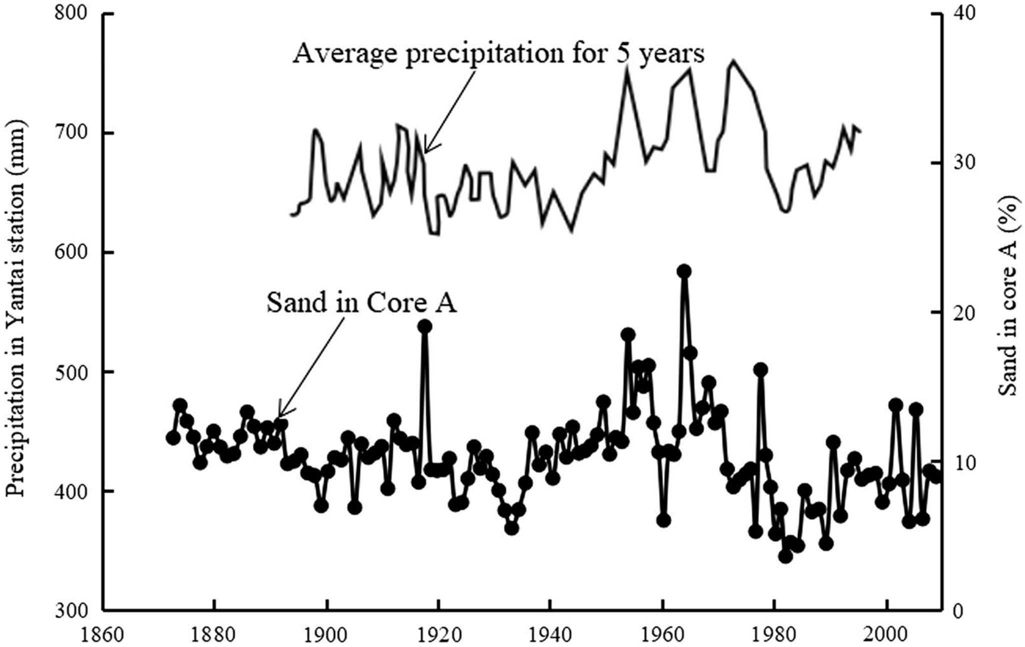

Fig. 4 The sediment grain sizes of core A and their relationship with the local average precipitation

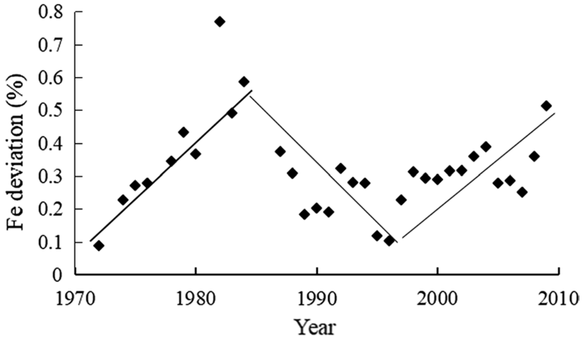

Fig. 5 The Fe deviation between the two cores during shellfish culture (since 1970s)

hydrophyte was destroyed, resulting in the strong re-suspension [41]. Moreover, Zhang [42] observed that the re-suspension was weakened in the *Laminaria* culture area. Obviously, these facts provide an explanation for the difference between the effect of algae culture and shellfish culture on grain size and Fe.

The Fe level deviations are estimated by subtracting the levels of core C from those of core A since the 1970s (Fig. 5). The deviations increase during 1970–1985 and 1996–2009, and decrease during 1985–1996. The increasing deviation during 1970–1985 may be caused by the prosperity of shellfish culture in Sishili Bay. While during 1996–2009, the intercropping and rotary culture of algae and shellfish were developed, and the algae culture output gradually occupied larger ratios [43], reasonably resulting in less deviation. The *Laminaria* were almost driven to extinction by white rot diseases in 1998 [22, 24], and the shellfish culture has dominated during 1985–1996, resulting in less deviation. Generally, the culture history in this area is closely correlated with sediment Fe levels.

Although there is little information about the effect of culture on sediment Fe levels, it should be noted that most researchers concluded that cultivation results in finer sediment and higher sediment rates [36, 44]. But the contrary is observed during the shellfish culture period by our investigation. In fact, some researches also documented the coarsening of sediments in culture area. Peng [44] reported the coarsened sediment since 1980s when long line culture was applied in a pearl oyster area along Cygnet Bay, Western Australia. Sheng et al. [14] recorded higher sand contents of surface sediments in a mariculture area than in the control area in Sishili Bay. Some geochemical properties related to finer particles are also found to be less in the culture area than in the control area in Sishili Bay, such as TOC, TN and heavy metal [14, 45]. These facts supported our observation from other aspects.

The potential significance of sediment Fe level variation in culture areas

Marine culture blooms because of the great demand for seafood, and its effect on marine ecosystem has raised concerns in recent decades. The full production capacity of global shellfish culture was 16.1 million tons in 2014. The area of shellfish culture in China was 12,411.1 km^2 and the production was 14.64 million tons in 2018 [46]. Such extensive activities surely affect sediment Fe levels according to our observation in this research.

Iron is an essential nutrient for all living organisms. Fe has been the important factor limiting the biological absorption of nitrite, phosphate and silicon in ocean, and thus ocean primary productivity and atmospheric CO_2 are directly affected when Fe is insufficient [8]. Observations in the Pacific subtropical zone, equatorial Pacific, Antarctic Ocean and upwelling region in California found low chlorophyll levels because of the Fe deficiency despite of the rich nitrite, phosphate [47, 48]. Iron fertilization were considered to be a future method of carbon fixing [48].

Fe levels in coastal area are relatively higher than those in ocean. However, the phytoplankton communities and their Fe-absorption mechanisms in coastal area differ greatly from those in ocean, and Fe-demand is different. The Fe-demand for the full growth of phytoplankton communities in coastal area are more than 100 times of those in ocean [9]. Zhang [49] observed a higher Fe-threshold levels in eutrophic water in Pearl River area. The limited primary productivity because of Fe deficiency along coastal area in California was documented [10]. The simulation experiments along coastal areas also confirmed the growth and chlorophyll of algae are improved when Fe is added, such as in Jiaozhou Bay [50, 51], Hainan Island [52]. Naturally, the variation of sediment Fe levels due to marine culture is significant in the coastal areas.

Red tide has been one of the most disasters along coastal area, and recent researchers have stated red tide is related to Fe levels along coastal area. Huang liangmin [12] argued Fe is the limiting factor for the red tide in Dapeng Bay. Wang [53] confirmed the blooming of red tide is related to Fe levels in East Sea. The occurrence of red tide in coastal area of California was found to be positively correlated with the discharged Fe from river [54]. The Fe levels reached 242 ug/dm^3 when red tide bloomed in Dalian Bay in 1986 [55]. The experiment in Xiamen found algae proliferated and caused red tide when Fe is added into seawater [56]. Therefore, the Fe levels in sediment and water change with culture activities, which may potentially cause red tide or eutrophication.

Conclusion

Two sediment cores in Sishili Bay (one in culture area and one in control area respectively) displayed variations of sediment Fe levels and grain sizes for a 100-year span. There were indications of sediment Fe deviation between the two areas since the 1970s. The result showed that the algae and shellfish culture impose opposite effects on sediment Fe levels. Comparing with the control area, algae culture causes an increasing while shellfish culture causes a decreasing of sediment Fe levels. Correspondingly, sediment grain sizes were observed to be finer due to algae culture and coarser due to shellfish culture. The affecting processes and mechanisms on sediment Fe levels differ greatly during algae and shellfish culture periods. The slowing down of ocean current velocity results in finer sediment particles and higher Fe levels in algae culture areas, but bio-deposition and re-suspension play key roles in governing the sediment particles and Fe levels in shellfish culture area. Fe levels in coastal areas are related to the growth of phytoplankton communities, red tide and eutrophication. Therefore, such processes are significant for the coastal environments in culture areas.

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Authors' contributions
BZ and ML did most of the writing, MA, CZ, QL, YZ developed idea and revised the manuscript. SH, HZ, JL and HC helped with the interpretation of the results, QC gathered the samples and did some of the writing. All authors read and approved the final manuscript.

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Availability of data and materials
The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Ethics approval and consent to participate
This study does not involve human participants, animals followed all applicable international and national guidelines for use of animals in research. All authors have consented to participate.

Consent for publication
Not applicable.

Competing interests
The authors declare that they have no competing interests.

Author details
[1] MOE Key Laboratory of Metallogenic Prediction of Nonferrous Metals and Geological Environment Monitoring, School of Geosciences & Info-Physics, Central South University, Changsha 410083, Hunan, China. [2] Shandong Provincial Lunan Geology and Exploration Institute (Shandong Provincial Bureau

of Geology and Mineral Resources No.2 Geological Brigade), Yanzhou 272100, Shandong, China. [3]Shandong Provincial Key Laboratory of Depositional Mineralization & Sedimentary Minerals, College of Earth Science & Engineering, Shandong University of Science and Technology, Qingdao 266590, Shandong, China.

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