Coral sand from hydraulic reclamation for the remediation of acid sulfate soil

Jiachen Zeng1,a, Yuchi Hao1,b, Wenbo Sun1*, De Wei 2,c, Runli Tao1,d, Baolin Shi3,e, Jianfei Zheng3,f, Zhen Zhang3,g

1CCCC National Engineering Research Center of Dredging Technology and Equipment Co., Ltd, 1088 Yangshupu Road, Shanghai, 200082, PR China
2CCCC Shanghai Dreging Co., Ltd, 13, Zhongshan East 1st Road, Shanghai, 200002, PR China
3CHEC Dredging Co., Ltd, 92 Yuanshen Road, Shanghai, 200120, PR China

Abstract. For the first time, Coral sand, as the main geotechnical material in reclamation, has the characteristics of loose and porous structure, relatively small volume and mass, easy to break and high compression, and high calcium carbonate, which may be a natural material to control acid release of acid soil. In this paper, by studying the neutralization effect of coral sand under different sand ratio, particle size and adding methods, the optimal dosage and particle size of coral sand and the adding sequence were determined under typical acid soil conditions; The neutralization performance of different neutralizing materials was compared through internal structure characterization, and the structural advantages of coral sand were explored. The results show that the specific surface area of coral sand was 1.2361m²g⁻¹, second only to calcite and shell powder. The particles were evenly distributed and can fully react with sulfuric acid to produce CaSO₄ precipitation. When the addition of coral sand was 9% (Ca: S = 18:5), the PASS can be neutralized to pH > 6.5. The PASS neutralization ability of coral sand was related to particle size. The overall trend was that the smaller the particle size, the stronger the neutralization ability. The best effect was at 0.15mm, when the particle size exceeded 0.27mm, the neutralization ability began to decline.

1.Introduction

Potential acid sulphate soil (PASS) was often associated with reclamation projects in subtropical and tropical areas. Acid sulfate soil are naturally formed soils or sediments that are produced under waterlogged and poorly drained conditions. is developed in the parent material rich in reducing sulfide, mainly pyrite (FeS₂), which is an extremely bad soil containing reducing sulfur. The potential acid sulphate soil dredged ashore was exposed to the atmosphere[1]. After the oxidation of pyrite in the soil, a large number of strong acid substances such as sulfuric acid and sulfite are produced, which not only polluted the ecological environment, but also corroded the building materials. The sulfides can move to the surface water and into subsurface water and cause loss of nutrients in the soil and destroy the soil structure[2].

In the reclamation project, natural coral sand was mixed with dredged soil and then dredged to the shore[3]. Coral sand is the main geotechnical material in the reclamation project, which mainly composed of CaCO₃, MgCO₃ and other insoluble carbonates[4]. The interior of coral sand has the characteristics of loose structure, small volume mass, easy to break and high compression[5], which is expected to be a natural improvement material for potential acidic soil in land reclamation. Some studies[2] showed that CaCO₃ and polyacrylamide (PAM) as the soil ameliorants were applied to neutralize ASS and improve the permeability of the soil.

The main crystal forms of coral sand are aragonite, calcite and magnesium calcite. The results showed that calcium oxalate precipitates were formed by the reaction of high concentration oxalic acid and coral sand, which were bonded to the surface of coral sand[6]. Magnesium calcite, aragonite and calcite are preferentially dissolved
in the dissolution process\cite{7}. The particle size of coral sand affects the acid neutralization performance and the dissolution of Ca\(^{2+}\) and Mg\(^{2+}\). Akio Yosano et al.\cite{8} studied the neutralization ability of coral sand, and found that the neutralization curve of coral sand with particle size less than 0.5mm was similar to that of CaCO\(_3\) for fertilizer, and the neutralization ability is roughly the same, and decreases with the increase of particle size. Some studies\cite{9} have shown that coral calcareous sands with different particle sizes have potential effects on soil physical and chemical properties and enzyme activities. The application effect of calcareous sand with particle size of 0.075-0.25 mm was equivalent to that of CaCO\(_3\), while the application effect of calcareous sand with particle size < 0.075 mm was better than that of CaCO\(_3\). Li et al.\cite{10} found that dissolution efficiency of Ca\(^{2+}\), Mg\(^{2+}\) in small coral sand is high and can be dissolved by low molecular organic acids. However, at present, there are few reports on the treatment of PASS by coral sand in reclamation project area. It is of great significance to study the self-neutralization law of coral sand to acid soil for soil treatment and resource utilization of coral sand in reclamation project in harbor area.

2. Materials and methods

2.1. Materials Preparation

Acid sulfate soils (ASS) were collected from mangrove area in Sanya City, Hainan Province, China. Table 1 lists the basic properties of ASS. After hydrogen oxidation, the pH value was decreased to 2.5. The pH of sulfide-containing soil was 6.33, which was decreased to 3.22 after hydrogen oxidation. Usually, soil with high organic substance and anaerobic condition (PASS) contain >5% total sulfur\cite{11}. Nevertheless, as the sample used in this study has been dried, the data has, therefore, yielded a lower value of total sulfur (<5%) with a disproportionate percentage of organic carbon(>3%)\cite{12}.

| Parameter                      | Value  |
|--------------------------------|--------|
| pH (1:2 water soil mass ratio) | 6.33   |
| pH-OX                         | 3.22   |
| EC (ds/m, 1:1water soil mass ratio) | 2.35  |
| Organic carbon (%)            | 0.36   |
| Total nitrogen (%)            | 0.037  |
| Cation exchange capacity (mg/kg)  | 126    |
| Available phosphorus (ppm)    | 2.8    |
| Available zinc (ppm)          | 0.75   |
| Exchangeable calcium (mg/kg)  | 8.1    |
| Substitutive magnesium (mg/kg) | 6.2    |
| Exchangeable potassium (mg/kg)| 7.5    |
| Alternative sodium (mg/kg)    | 4.9    |
| Fe (ppm)                      | 70.0   |
| Al (ppm)                      | 67.3   |
| K (ppm)                       | 54.7   |
| C (ppm)                       | 57.9   |
| Na (ppm)                      | 12.4   |
| Ca (ppm)                      | 9.1    |
| S (ppm)                       | 8.5    |
| Mg (ppm)                      | 5.2    |

These values were similar to those of field identification. In addition, a pyrite sample (PS) was supplied by a polymetallic sulfide mine in Xiangshan District, Guilin City, Yunnan Province.

The coral sand samples were taken from a dredger fill reef in the South China Sea. Table 2 lists the basic properties of coral sand. The main element was Ca, accounting for 95.854%. In addition, trace elements such as Sr, Si, S, Mg are detected. The study\cite{13} shows that the higher the calcium content, the stronger the acid-base neutralization ability. The samples were light gray in color, rough in surface, low in strength and brittle in quality. Before the test, the particle size distribution of the samples was not uniform, the main particle size distribution was 12-30μm, there were coarse particles larger than 1.5 mm, but the content was less than 2%. In the dosage inquiry test, in order to avoid the influence of particle size effect on the test results of acid soil, the coarse particles in the
sample were removed, and the adjusted particle gradation is shown in Fig. 1.

![Grading curve of coral sand](image)

**Table 2 Basic components of coral sand**

| Element | Content   |
|---------|-----------|
| Ca      | 95.854%   |
| Sr      | 2.236%    |
| Si      | 0.537%    |
| S       | 0.352%    |
| Mg      | 0.371%    |
| Fe      | 0.268%    |
| K       | 0.096%    |
| Cu      | 0.046%    |
| Br      | 0.039%    |
| Zn      | 0.021%    |

Dolomite, shell, calcite, montmorillonite, hydrated lime was supplied by a quarry. They were milled to 0.15 mm before being used and denoted as DMP, SHP, CAP, MLP and HLP. Coral sand was ground to 0.15 mm, denoted as CSP.

### 2.2. Characterizations

The solid of CSP was analyzed by X-ray diffraction and electron microscope analyzer. Inductively coupled plasma atomic emission spectrometry (ICP) was used to determine the contents of Ca, Mg and Fe in coral sand solution after digestion. X-ray diffraction (XRD) was recorded on a D/max-2500V+/PC diffractometer (Rigaku Industrial Co., Ltd.), which was equipped with an monochromatized Cu K radiation. The scanning speed was 8°/min and the scanning range was 5-80°. Micro-morphology was characterized with scanning electron microscopy (HTREM, JEOL JEM2010).

### 2.3. Dynamic analysis of coral sand self neutralization

3g (acid soil & CAP), (PS & CAP) and (CAP & 2ml 1mol / L sulfuric acid) were mixed with each other in 20ml of deionized for 30 min. Then, H₂O₂ (pH = 7) was added to the mixture, which was then stirred for 30 min. Afterward, the mixture was centrifugated, dried at 105°C, and milled to 100 mesh. The sample was recorded as S1~S3.

X-ray diffractometer was used to characterize S1 ~ S3. The pore size data of CAP, SHP, DMP, MLP, CAp and HLP were obtained by BET test.

### 2.4. Neutralization ability

The neutralization test of PASS can be divided into two situations: the first is synchronous oxidation neutralization, and the relationship between oxidation rate and neutralization rate is explored according to the change of pH value in each period; the second is oxidation first and then neutralization, which aims to explore the neutralization rate of various neutralizing agents.

In order to investigate the influence of CSP dosage on pH, experiments were designed as follows: X g of CSP was mixed with 5 g of ASS and 10 mL of H₂O₂ (pH = 7). The weight ratio of Ca in CSP and S content in ASS was X:1 (X = 0.4, 0.8, 1.2, 1.6, 2.0, 2.4, 2.8, 3.2 and 3.6); the weight ratio of H₂O₂ and S content was 200:1. The reaction time was 45 min. Each experiment was denoted as PX. The pH value of soil suspension was tested every five minutes[14].

**Table 3** lists the specific experimental design.
### Table 3 PASS test method

| Type          | Particle size (mm) | M (n) | Soil | H₂O₂:S | Time | Methods           |
|---------------|-------------------|-------|------|--------|------|-------------------|
| 1 Coral sand  | 0.075             | 2:1   | 5g   | 200:1  | 45min | Simultaneous oxidation neutralization |
|               | 0.15              | 2:1   | 5g   | 200:1  | 45min |                   |
|               | 0.27              | 2:1   | 5g   | 200:1  | 45min |                   |
|               | 0.5               | 2:1   | 5g   | 200:1  | 45min |                   |
|               | 0.8-1.2           | 2:1   | 5g   | 200:1  | 45min |                   |
| 2 Montmorillonite | 0.15            | 2:1   | 5g   | 200:1  | 45min |                   |
| 3 Shell powder | 0.15              | 2:1   | 5g   | 200:1  | 45min |                   |
| 4 Hydrated lime powder | 0.15        | 2:1   | 5g   | 200:1  | 45min |                   |
| 5 Dolomite    | 0.15              | 2:1   | 5g   | 200:1  | 45min |                   |
| 6 Calcite     | 0.15              | 2:1   | 5g   | 200:1  | 45min |                   |
| 7 Coral sand  | 0.15              | 2:1   | 5g   | 200:1  | 45min |                   |
| 8 Hydrated lime powder | 0.15    | 2:1   | 5g   | 200:1  | 45min |                   |
| 9 Calcite     | 0.15              | 2:1   | 5g   | 200:1  | 45min |                   |
| 10 Shell powder | 0.15           | 2:1   | 5g   | 200:1  | 45min |                   |
| 11 Dolomite   | 0.15              | 2:1   | 5g   | 200:1  | 45min |                   |
| 12 Montmorillonite | 0.15        | 2:1   | 5g   | 200:1  | 45min |                   |

Note: Mn:S is the ratio of neutralizing materials to sulfur content in acid soil; H₂O₂:S is the ratio of hydrogen peroxide to sulfur in acid soil.

### 3. Results and discussion

#### 3.1. Neutralization performance

According to the standard for disposal of acid soil in actual dredging project, the PASS after neutralization material treatment must meet pHₕ ≥ 6.5, pHFOX ≥ 6.5 (pHF is the in-situ pH value of the dredged material to be treated, pHFOX is the pH value oxidized by hydrogen peroxide) to meet the land use standard. In order to explore the best proportion of coral sand and PASS for complete neutralization (pH ≥ 6.5 after complete oxidation), researchers used different proportions of coral sand to neutralize acid sulfate soil.

Fig. 2 shows the change of pH along with time when the ratio of CSP was increased from 0.4:1 to 3.6:1. During 0-5 min, the pH value was decreased significantly, which indicated that pyrite in the acid soil was oxidized to produce sulfuric acid. After 5 minutes, the decreasing trend of pH became slow. At this time, sulfuric acid produced by oxidation is neutralized by CSP, H⁺ reacted with CaCO₃ in CSP. As a result, the pH value of oxidation products reached stability after 30 minutes, and the pH value was stable around 6.3 (5.8–6.5), which indicated that most of the sulfuric acid is neutralized.

Fig. 2 also shows that the order of neutralization effect of each proportion followed the order of P0.4 < P0.8 < P1.2 < P1.6 < P2 < P2.4 < P2.8 < P3.2 < P3.6. When the ratio of Ca and S in acid soil was 0.4:1, the pH of oxidation products reached stability after 30 minutes, and the pH value was stable at 5.0, which was higher than that of undisturbed soil. In other words, when the Ca: S reached 18:5, the pH value of PASS was adjusted to 6.32, indicating the best dosage of CSP, which met the requirements of acid soil disposal.
In order to compare the advantages and disadvantages of coral sand with other neutralizing materials, it is necessary to explore the change of neutralization rate when neutralizing agent was added after oxidation of acid soil.

Fig. 3 shows the order of neutralization effect of each material followed the order of MLP < DMP < CSP < CAP = SHP < HLP. The neutralization rate of CSP was slower than that of HLP, CAP, SHP, DMP and MLP, reached stability at 50 minutes. At this time, the pH value was second only to SHP and CAP, and less than HLP. Because of its strong alkalinity, HLP was easy to cause soil salinization, lime was expected to improve the pH level of soil, which was at extreme acidity with a value of \( t < 3.5 \)\(^{[12]}\), the requirements in using modified lime on acid-sulfate lands or pyritic, which has been an aquaculture regime, are found to be inappropriate and unreliable\(^{[15]}\). Although the neutralization rate of CSP was relatively slow, it has the characteristics of high alkali content and high porosity. It has an outstanding effect on the prevention and control of PASS.

### 3.2 Particle size effect

Fig. 4 shows that the overall trend of pH decreased with the increase of particle size of coral sand when coral sand and PASS were mixed synchronously. When the particle size of coral sand was 0.15 mm, the best neutralization effect was obtained, and the pH value was finally stabilized around 5.95. When the particle size was reduced to 0.075 mm, the decrease rate of pH was faster than that of 0.15 mm coral sand, but the pH at the end point is slightly lower than the former. The neutralization effect of 0.15 mm coral sand is better than that of 0.075 mm coral sand. Generally speaking, the relationship between particle size and neutralization capacity was not simply that the smaller the particle size, the stronger the neutralization ability. For PASS, it may be related to whether the particle size of neutralizing materials matches the soil particles. When the particle size 0.5 mm was 0.5 mm, the pH value decreased significantly, and the final pH value was about 5.35. When the particle size changed to 3-4mm, the end point pH value was 4.27, which increased by 1 unit compared with the original soil after complete oxidation.
Fig. 4 Test of coral sand particle size (simultaneous oxidation neutralization)

Fig. 5 shows that the neutralization capacity and neutralization rate of coral sand decrease with the increase of particle size. The results show that the neutralizing acid content of coral sands with particle sizes of 0.075mm, 0.15mm and 0.27mm were similar, but the neutralization rate was different, and the pH value at the end point was around 6.5. The neutralization rate of 0.075mm and 0.15mm coral sand was significantly better than that of 0.27mm coral sand. When the particle size was larger than 0.27mm, the neutralization rate decreased significantly, and the terminal pH decreased to about 5.1. The neutralization rate at 0.5mm was slightly lower than that at 0.27mm, and the final pH was 6.03. Although the increase of particle size has a great impact on the neutralization effect of coral sand, the coral sand larger than 0.5mm still has neutralization ability. When the particle size was 3-4mm, compared with the undisturbed soil, the pH was still increased by 1.5 units.

Fig. 5 Test of coral sand particle size (preferential oxidation)

3.3 Properties and reaction mechanism

Fig. 6 shows the X-ray diffraction pattern. The characteristic peaks of 3.4085 (111), 3.2851 (021), 2.7089 (012), 2.4912 (102), 2.3791 (112), 1.9805 (221), 1.8849 (202), 1.8179 (132) and 1.7459 (113) were the characteristic spectral lines of aragonite, which match the powder diffraction card (PDF # 71-1663). After screening and comparison, the characteristic peak 2.9957 (104) was determined to be the characteristic peak of magnesium calcite. In addition, a small number of mixed peaks show that there was a small amount of calcite in the coral sand, which indicated that the contents of calcite, magnesium calcite and aragonite in coral sand are 10.2%, 21.5% and 68.3%, respectively.
In the SEM image of coral sand (Fig. 7), it can be clearly observed that the structural units of coral sand are long strip-shaped cubic deposits, with high angle and irregular shape of particles, many rod-shaped and flaky particles, and many internal pores. The structure has a large specific surface area, which is conducive to the full neutralization reaction with sulfuric acid.

Fig. 8 shows the main crystal image composition of coral sand was CaCO$_3$ (PDF #41-1475) and calcium silicate (PDF # 27-0088), so it has strong neutralization performance. The main crystal images of S1 were CaCO$_3$, CaAl$_2$Si$_2$O$_8$·4H$_2$O (PDF # 20-0452) and SiO$_2$ (PDF # 46-1045). CaAl$_2$Si$_2$O$_8$·4H$_2$O is the main component of anorthite. Batch adsorption studies\cite{16} show that it is an effective adsorbent to remove CO$^{2+}$ and Sr$^{2+}$ from acidic aqueous medium. The final products of S3 are mainly CaSO$_4$ (PDF # 23-0128) and CaCO$_3$. Therefore, it can be inferred that the reaction process of coralline sand and pure sulfuric acid is the reaction of CaCO$_3$ and sulfuric acid, and the CaSO$_4$ produced successfully fixed SO$_4^{2-}$ in coralline sand. The crystal image of S2 product was similar to that of S3 product, which is CaSO$_4$ and CaCO$_3$. PS was completely oxidized to produce H$_2$SO$_4$, which reacted with CaCO$_3$ to form CaSO$_4$ slightly soluble in water. Hence, to some extent, CaCO$_3$ in coral sand can fix sulfur element in PS. A study shows\cite{18} that as a common neutralizer of ASS, CaCO$_3$ can not only neutralize the sulfuric acid produced by oxidation, but also inhibit the oxidation rate of ASS, there was minimal sulfide oxidation and no acidification after the addition of excess CaCO$_3$ over the 180 days of incubation.
3.4 Structural advantages

Fig. 9 compared the structural characteristics of several neutralizing materials. Montmorillonite shows IV type isotherm\(^{(19)}\) and H3 type hysteresis loop\(^{(20)}\), which indicated that it has complex mesoporous structure and irregular pore structure. Shell powder, coral sand, calcite and dolomite showed I-type isotherm\(^{(21)}\), and there was no hysteresis loop, which indicated that they had the characteristics of microporous adsorbent\(^{(22)}\), and the saturated adsorption value was equal to the filling volume of micropores. In addition, the order of BET surface area (Table 4): DM\(_P\) (0.16287m\(^2\)g\(^{-1}\)) < CS\(_P\) (1.2361m\(^2\)g\(^{-1}\)) < CA\(_P\) (2.5932m\(^2\)g\(^{-1}\)) < SH\(_P\) (3.297m\(^2\)g\(^{-1}\)) < ML\(_P\) (69.448m\(^2\) g\(^{-1}\)). The bet order of each material was different from that of the pore size. The order of pore size was ML\(_P\) < CA\(_P\) < DM\(_P\) < SH\(_P\) < CS\(_P\), which was different from the law of neutralization rate, but partly similar. Although montmorillonite has high specific surface area, its pore size was small and its neutralization rate was slow. DM\(_P\) and CA\(_P\) have similar pore size, but the specific surface area of CA\(_P\) was much higher than DM\(_P\), and its neutralization ability was better than dolomite.

Fig. 9 Adsorption isotherms of several neutralizing materials

| Table 4. Specific surface areas of several neutralizing materials |
|-----------------|-----------------|-----------------|
|                | BET (m\(^2\) g\(^{-1}\)) | Total pore volume (p/p0=0.99) | Average aperture (nm) |
| DM\(_P\)        | 0.16287          | 0.00077437      | 19.018                  |
| SH\(_P\)        | 3.297            | 0.020098        | 24.383                  |
| CA\(_P\)        | 2.5932           | 0.011594        | 17.883                  |
| ML\(_P\)        | 69.448           | 0.1121          | 6.4577                  |
| CS\(_P\)        | 1.2361           | 0.012523        | 40.524                  |
Therefore, the neutralization ability of materials was affected by specific surface area and pore size. CS indicates a small specific area, but its average pore size was the highest among several neutralizing materials, which was 0.524nm. Its large pore size can well react with acid and adsorb sulfate ion in the process of acid damage control of PASS, which can be used as a good PASS amendment.

4. Conclusion

In this work, coral sand was used to treat the PASS in the harbor area as a common calcium containing material in the reclamation of land. In general, the smaller the particle size was, the stronger the neutralization ability was. The best neutralization effect was at 0.15mm. When the particle size exceeded 0.27mm, coral sand still has neutralization effect on the PASS. When the median particle size was 0.5mm, the neutralization ability was 20% lower than that of coral sand below 0.27mm. When the particle size increased to 3-4mm, the neutralization ability was 50%. From the mixing mode, the final pH value of oxidation before neutralization was higher than that of synchronous mixing, which indicated that oxidation before neutralization can make sulfuric acid react with neutralizing materials better. After characterization, it was found that the coral sand has a large specific surface area (1.2361m²g⁻¹), second only to calcite and shell powder. The particles were evenly distributed and can fully react with sulfuric acid to produce CaSO₄ precipitation. When Ca: S = 18:5, the PASS can be neutralized to pH > 6.5. The neutralization rate of coral sand was lower than that of calcite and shell powder, but it can provide the same alkali. As the inevitable product of land reclamation, coral sand is much easier to obtain than calcite and shell powder, so it is an ideal neutralization material for PASS.

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