



Communication Influence of Oyster Shell Pyrolysis Temperature on Sediment Permeability and Remediation

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Abstract: Permeability is an important aspect of sediment remediation. It is well-known that oyster shells can be used for sediment remediation, however the influence of pyrolysis temperature on sediment permeability remains unknown. In this study, we examined sediment permeability and remediation using crushed oyster shells of less than 5 mm in size that were pyrolyzed at 350 °C (POS350) and 600 °C (POS600) for six hours. Based on the results of the variable head permeability test, POS600 has greater sediment permeability than POS350. In addition, POS600 has greater than POS350 to reduce dissolved inorganic nitrogen (DIN; NH₃-N, NO₂-N, and NO₃-N) and phosphate (PO₄-P) from organically enriched sediment because of its higher Ca²⁺ elution. In conclusion, pyrolysis of oyster shells at 600 °C is more effective than pyrolysis at 350 °C. This finding is true because the transformation of CaCO₃ to CaO, which is the source of Ca²⁺, stimulates pore water flow. Based on these findings, it can be concluded that pyrolyzed oyster shells are beneficial for increasing sediment permeability, thereby helping in the remediation of sediments.

Keywords: marine sediment; permeability; oyster shell; pyrolysis; phosphate; nitrogen; remediation

1. Introduction

Oyster shells are a common type of seafood waste in Republic of Korea, where approximately 300,000 tons of waste are generated annually, only half of which is recycled and the rest is deposited on the seashore or field-heaped on farmland [1]. This careless disposal of oyster shells causes socio-economic problems, such as the growth of insects and bad smells, as well as the production of toxic gases such as ammonia (NH₃) and hydrogen sulfide (H₂S) as a result of long-term microbial decomposition [2]. According to recent studies, oyster shells are a useful material for remediating organically enriched marine sediments by removing hydrogen sulfide (H₂S) from pore water or sediments via adsorption [3,4]; reducing the concentration of dissolved inorganic nitrogen (DIN), phosphate (PO₄-P), and acid volatile sulfides [5–9]; and increasing the oxidation-reduction potential (ORP), dissolved oxygen, pore water pH, and the shear strength of the sediment [8–12].

The water column and the sediment matrix are the two main components of the marine environment. The sediment layer hosts most of the biological activity, which is crucial for maintaining a balance between the burial and mineralization of organic materials. The accumulation of nutrients and organic material in sediment is well known, and these substances may continue to act as pollution sources. The flow of organic matter and gas exchange or transfer between the sediment and water column depends on the permeability of the sediment [13]. Low permeability not only causes the accumulation of contaminants but also reductive conditions; the ecosystem will be destroyed rapidly, resulting in the formation of odors caused by anaerobic decomposition processes.



Citation: Patil, M.P.; Woo, H.-E.; Yoon, S.; Kim, K. Influence of Oyster Shell Pyrolysis Temperature on Sediment Permeability and Remediation. J. Mar. Sci. Eng. 2023, 11, 934. https://doi.org/10.3390/ imse11050934

Academic Editors: Fred Wang Fat Lee, Steven Jingliang Xu, Wing Yin Mo and Sidney Man Ngai Chan

Received: 10 April 2023 Revised: 18 April 2023 Accepted: 25 April 2023 Published: 27 April 2023



Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). The cation absorption of clay mineral surfaces affects the hydraulic behavior of clayey particles. It is widely known that cations have a critical impact on soil permeability in the following order: $Na^+ > K^+ > Mg^{2+} > Ca^{2+}$ [14]. Monovalent cations reduce soil permeability, whereas divalent cations enhance soil permeability [15–17]. Oyster shells are composed of over 95% CaCO₃, an eco-friendly material that can supply Ca²⁺ to the ocean [18]. The chemical compositions of oyster shells change; the primary component of oyster shells, CaCO₃, transforms into calcium oxide (CaO) at temperatures above 500 °C [19]. Numerous studies have proven that the removal of nutrients and heavy metals from oyster shells calcined at 600 °C or above has a positive impact on the environment [20,21].

Oyster shells that have pyrolyzed at different temperatures have been shown to have an effect on both their chemical properties and sediment remediation [9–11,19–21]. However, its effect on permeability performance still has relevance, and further research is required for a better understanding of this phenomenon. Therefore, in this work, we constructed an experiment using oyster shells that were pyrolyzed at various temperatures and assessed their permeability performance, changes in cation concentrations, and impact on sediment remediation.

2. Materials and Methods

2.1. Oyster Shells and Sediments

The oyster shells used in this study were obtained from Geoje, Republic of Korea, and had been naturally dried for over six months. Oyster shells were washed to remove the leftover substances, after which they were air-dried, crushed to obtain particles with a size \sim 5 mm, and pyrolyzed at 350 °C (POS350) and 600 °C (POS600) for 6 h in an electric furnace (SF-14, SciLab, Seoul, Republic of Korea). Oyster shells crushed to 5–35 mm have interior friction angles or permeability coefficients similar to sand soil [22]. Therefore, a oyster shell particle size of less than 5 mm was applied in this experiment.

Goseong Bay, Republic of Korea, is affected by high levels of terrigenous nutrients in sediments. Sediment samples were collected by dredging using a Van Veen Grab (CL-VG 351, HY Science, Pohang, Republic of Korea), and foreign substances such as shells and vinyl were removed as much as possible. Sediment samples were collected in plastic boxes and transported to the laboratory for testing.

2.2. Cations Analysis

For the analysis of the Ca²⁺ composition of oyster shells, 100 g of POS350 and POS600 particles were added to the Erlenmeyer flask (2 L) containing 1000 mL of distilled water, mixed well, and incubated at room temperature. After incubation for 6 h and 24 h, 15 mL samples were withdrawn and filtered through a 0.45 μ m membrane syringe filter (MILLEX-HV, Millipore, Burlington, MA, USA) and used for Ca²⁺ concentration determination via Inductively coupled mass spectrometry (ICP/MS, Perkin Elmer, Optima 730 V, Waltham, MA, USA).

2.3. Variable-Head Permeability Test

A variable-head permeability test instrument (Figure 1) was used in accordance with the ASTM standard method [23] to carry out the procedure for determining the sediment permeability coefficient. The sediment was placed a permeability mold (\emptyset 100 × 12.7 cm) up to a depth of 8 cm and the remaining 4.7 cm was then covered with either sand or oyster shells for the control and experimental cases, respectively. Crushed oyster shells (5–35 mm) have interior friction angles or permeability coefficients similar to sand soil [22]. Therefore, sand with a particle size of ~2 mm was used as a control. Deionized water was used as the permeating aqueous solution. Three sets of permeability molds, a control using sand, and an experiment using POS350 and POS600 were set and permeability tests were conducted for 200 h. The permeability coefficient (k) in cm/sec was determined using Equation (1) [23]:

$$k = 2.3 \frac{aL}{A(t2 - t1)} \log_{10}\left(\frac{h1}{h2}\right)$$
(1)

where *k* is the permeability coefficient in mm/s, *a* is the area of the standpipe in mm², and *L* and *A* are, respectively, the height in mm and area in mm² of the sand specimen. The time (t2 - t1) required for a water column to decrease from *h*1 to *h*2 was measured.



Figure 1. Schematic presentation of variable-head permeability test instrument.

2.4. Pore Water Analysis

Pore water samples were prepared via centrifugation; sediment samples were centrifuged at 3800 rpm for 15 min (LaboGene 416, Bio-Medical Science, Seoul, Republic of Korea) to collect the pore water and then filtered through a glass microfiber membrane filter (GF/B, Whatman, Maidstone, UK). Before the permeability test (initial) sediment samples and after the permeability test all sediment samples (control and experiment) were analyzed for pH, DIN (NH₃-N, NO₂-N, NO₃-N), and PO₄-P. The pH of pore water was measured using an Ag/AgCl glass electrode (Laqua F-73, Horiba, Kyoto, Japan), and the concentrations of NH₃-N, NO₂-N, NO₃-N, and PO₄-P were determined using standard procedures for the examination of water and wastewater [24]. All tests were repeated thrice and the results were reported as average with standard deviation.

2.5. Leachate Analysis

The permeating solution (leachate) was collected between for 10-20 h and 50-60 h of the experiment, the samples were then mixed well to create a homogenized solution and determined PO₄-P conc. was determined following the procedure used for pore water analysis.

3. Results and Discussion

3.1. Cation Concentration

Depending on the pyrolysis temperature, different concentrations of Ca²⁺ ions were found on oyster shells. After 6 h, the amount of Ca²⁺ in POS350 was 35.3 μ g/g, while 24 h, it was only 32.7 μ g/g. Comparatively more Ca²⁺ was found in the POS600: 53.9 μ g/g after 6 h and 54.1 μ g/g after 24 h. Results indicate that the concentration of Ca²⁺ increased along with an increased temperature of pyrolysis. This result is due to the fact that pyrolysis of oyster shells below 350 °C does not generate CaO [9], whereas, above 500 °C, CaCO₃ is converted to CaO [19], which is the source of Ca²⁺ [18].

3.2. Changes in Sediment Permeability

Results of the sediment permeability test using variable-head permeability testing instrument are presented in Figure 2. The permeability coefficient of sediment covered with POS600 fluctuates more than that of sediment covered with POS350 and sand (as a

control) for the first 20 h of the experiment, which may be attributable to a sudden increase in permeability resulting from the various concentrations of ions and particulate sizes of POS350, POS600, and sand. The constant permeability coefficient was observed in all three sets of experiments after 110 h. No major differences in permeability coefficient results were observed between the POS350 and sand tests throughout the period of the experiment. Experiments ended after 200 h with permeability coefficients of 0.5×10^{-5} cm/sec for sediments covered with POS600 and nearly zero for sediments covered with POS350 and sand. Sediment permeability is affected by a number of factors including particle size, ionic composition, and shear strength. Even minute changes in the cation occupancy of clay surface can alter the permeability coefficient and alter clay particle behavior [14]. Monovalent cation (Na⁺) increases the thickness of clay particles due to dispersion or expansion in response to changing ionic strength and ultimately decreases soil permeability through increasing soil compactness and decreasing soil porosity. In contrast, divalent cation (Ca^{2+}) reduces the thickness of clay particles and enhances the aggregation of dispersed colloidal clay particles [15]. Recent studies have reported that calcium-based materials increase soil permeability because of the flocculation of clayey particles and prevent blockage of soil porosity [16,17]. Similar results were obtained in the present study; the permeability of POS600-covered sediments is greater than that of POS350-covered and sand-covered sediments, because of a higher percentage of CaO in oyster shells pyrolyzed at temperatures above 500 $^{\circ}$ C, which is the source of Ca²⁺ [19].



Figure 2. Variation in sediment permeability during variable head permeability testing with oyster shells pyrolyzed at 350 °C (POS350) and 600 °C (POS600) and sand as the covering material. The coefficient of permeability is presented as the mean of three replicate measurements.

3.3. Changes in Pore Water

The properties of sediments before (initial) and after (200 h) sediment permeability tests are shown in Table 1. The pH of pore water does not vary greatly between before and after the permeability test samples, with the exception of the POS600 sample pH, which was raised to 7.95 from the original sediment sample pH of 7.64. Pyrolysis changes the main chemical component of OS, CaCO₃, into the alkaline CaO [19]. This also helps increase the pH of the sediment. The increasing pH of pore water is due to decreasing concentrations of DIN and PO₄-P in the sediment treated with pyrolyzed oyster shells [5,6].

Test	Properties of Sediment (Pore Water)			
	Initial (Before-Permeability Test) —	After-Permeability Test (Sediment Covered with)		
		Sand	POS350	POS600
pН	7.64 ± 0.02	7.63 ± 0.01	7.76 ± 0.02	7.95 ± 0.01
PO_4 -P (mg-P/L)	2.20 ± 0.07	1.55 ± 0.04	1.65 ± 0.04	1.35 ± 0.04
NH ₃ -N (mg-N/L)	23.75 ± 0.18	11.00 ± 0.00	10.75 ± 0.18	5.50 ± 0.35
NO_2-N (mg-N/L)	0.07 ± 0.00	0.02 ± 0.00	0.02 ± 0.00	0.02 ± 0.00
NO ₃ -N (mg-N/L)	0.14 ± 0.00	0.18 ± 0.00	0.16 ± 0.00	0.18 ± 0.00

Table 1. Characteristics of properties of sediments before and after sediment permeability test. All results are given as the mean of measurements performed in triplicate with standard deviation. POS350 and POS600 are the acronyms of oyster shells pyrolyzed at 350 °C and 600 °C, respectively.

The PO₄-P concentration varies remarkably; the highest concentration was observed in initial (2.20 mg-P/L) sediment samples compared to sand (1.55 mg-P/L), as well as POS350 (1.65 mg-P/L) and POS600-covered (1.35 mg-P/L) sediment pore water samples (Table 1). Significantly decreased concentration of PO₄-P in POS600 sediment due to alkaline condition of sediment (pH 7.95) and higher concentration of Ca^{2+} which possibly promoted the formation of a precipitate form of phosphate (CaPO₄) and decreased phosphate release rate. According to published findings, the formation of CaPO₄ precipitates, the development of alkaline conditions in the sediment, and the adsorption of PO₄-P onto oyster shells are all factors that contribute to the reduced concentration of phosphates in sediment treated with oyster shells [25]. While precipitates CaPO₄ tend to be transformed into stable mineral-bound phosphate with very low phosphate release rates in alkaline conditions of sediments, calcium-bounded phosphate is released from them in acidic conditions [26,27].

NO₂-N and NO₃-N exhibit no significant differences, whereas NH₃-N levels in POS-treated sediments are significantly lower than in sand-treated and untreated sediments (Table 1). The concentration of NH₃-N in POS600-treated sediment decreased to 5.50 mg-N/L from 23.75 mg-N/L (initial untreated sediment), most likely as a result of the active covering material-treated sediment permeability enhancement-led nitrification process [28]. Similar observations were made in this study; POS600-treated sediment shows higher permeability than the POS350 and sand-treated sediment (Figure 2). Increasing sediment permeability and supplementing of oxygen result in a decrease in the concentration of NH₃-N in sediment due to nitrification [29,30].

3.4. Changes in Leachate

Figure 3 shows the concentration of PO₄-P in leachate obtained from sand, POS350, and POS600-covered sediments during the permeability tests. The PO₄-P concentration in the leachate between 10 to 20 h and 50 to 60 h of experiment shows that POS600-covered sediment released less PO₄-P than POS350 and sand, and the amount of PO₄-P in the leachate also decreased with time. As POS600 has more Ca²⁺ ions, which react with PO₄-P to form a precipitate form of CaPO₄. Because the POS600-covered sediment turns alkaline (Table 1), possibly the elution rate of PO₄-P is decreased. Ca²⁺ ions from CaO react with PO₄-P and form a precipitate form of CaPO₄ that tends to transform into stable mineral-bound phosphate and, because of the alkaline condition in sediment, very low phosphate release [25–27]. The leachate of POS350 has a higher concentration of PO₄-P than POS600 because of the lower amount of Ca²⁺ ions. It is reported that when the pyrolysis temperature of oyster shells is below 350 °C, CaCO₃ does not transform to CaO [9].



Figure 3. PO_4 -P concentration in leachate from the sediment permeability test using sand, and pyrolyzed oyster shells at 350 °C (POS350) and 600 °C (POS600) respectively, as sediment covering agents. All tests were performed three times, and the results are presented as the mean with error bars indicating standard deviations.

4. Conclusions

The temperature of pyrolysis has a significant effect on the cation compositions of oyster shells; a higher concentration of Ca^{2+} was observed in oyster shells pyrolyzed at 600 °C than at 350 °C. The variable head sediment permeability test confirms that the high concentration of Ca^{2+} in POS600 enhanced the sediment permeability. On the other hand, a higher concentration of Ca^{2+} causes sediment pH to become alkaline, which promotes the formation of a precipitate form of phosphate and reduces the rate of phosphate in pore water and in leachate. Likewise, more remarkable decreases DIN concentration in sediment cover were recorded with oyster shells pyrolyzed at 600 °C than at 350 °C which is possibly due to enhanced sediment permeability and nitrification processes. Overall, pyrolysis of oyster shells at 600 °C is better than pyrolysis at 350 °C. This is due to the fact that the transformation of $CaCO_3$ to CaO, which is the source of Ca^{2+} , stimulates pore water flow. Based on these findings, it can be concluded that pyrolyzed oyster shells are beneficial for increasing sediment permeability, thereby helping in the remediation of sediments.

Author Contributions: Conceptualization, M.P.P. and H.-E.W.; methodology, M.P.P. and H.-E.W.; formal analysis, M.P.P., H.-E.W. and S.Y.; investigation, M.P.P. and S.Y.; data curation, M.P.P., S.Y. and H.-E.W.; writing—original draft preparation, M.P.P.; writing—review and editing, S.Y. and K.K.; visualization, M.P.P.; supervision, K.K.; funding acquisition, K.K. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by a grant from the National Institute of Fisheries Science, Republic of Korea (R2023005) and the Korea Institute of Marine Science and Technology Promotion (KIMST) funded by the Ministry of Oceans and Fisheries, Korea (G22202202522201).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

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