Application of calcium-rich mineral under nonwoven fabric mats and sand armor as cap layer for interrupting N and P release from river sediments

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Abstract This work investigates the applicability of thermally treated calcium-rich minerals (CRMs), such as sepiolite (SPL), attapulgite (ATT), and dolomite (DLM) to hinder the nitrogen (N) and phosphorus (P) release from river sediments. A non-woven fabric mat (NWFM) or a sand layer were also capped as armor layers, i.e., placed over CRMs to investigate the capping impact on the N/P release. The capping efficiency was evaluated in a cylindrical reactor, consisting of CRMs, armor layers, sediments, and sampled water. We monitored N/P concentrations, dissolved oxygen (DO), oxidation reduction potential, pH, and electric conductivity in overlying water over 70 days. The DO concentrations in the uncapped and capped conditions were preserved for 30 days and 70 days (until the end of experiment duration), respectively. ATT showed higher efficiency for NH4-N and T-N than the other two materials, and the capping efficiency of NH4-N was measured as 96.4%, 93.7%, and 61.6% when capped with 2-cm sand layer, 1-cm sand layer, and NWFM layer, respectively. DLM showed a superior rejection capability of PO4-P to ATT and SPL, reported as 97.2% when capped with 2-cm sand armor. The content of weakly adsorbed-P was lower in the uncapped condition than in the capping condition. It can be concluded that ATT and DLM can be used as capping agents to deactivate N and P, respectively, to reduce water contamination from sediments of the eutrophic river.

Keywords In situ capping · Sediment · Calcium · Clay mineral · Phosphate · Nitrogen

Introduction

Phosphorus (P) and nitrogen (N), i.e., vital nutrients to living organisms in aquatic environments, often trigger eutrophication (Correll 1999; Zhu et al. 2019). Excessive P concentrations initiate eutrophication in freshwater resources such as lakes, reservoirs, streams, and the headwaters of estuaries, and eutrophication by excessive N is often observed in ocean environments (Correll 1999). These eutrophication phenomena degrade organic matter on sediment surfaces, deplete dissolved oxygen near the bottom of the water column, and accelerate the nutrient flux from sediments to the overlying water column (Holmboe et al. 2001). Once started, eutrophication can continue for several decades due to the internal nutrient loading from sediments (Yin et al. 2016; Zhan et al. 2019).

In situ capping is an effective method for mitigating potential water-body eutrophication by impeding the nutrient migration from polluted sediments to the water above them (Jiao et al. 2020). Compared to monitored natural recovery and dredging, this treatment method is less costly, less disruptive, and less time-consuming (Ghosh et al. 2011). In capping technology, a layer of material is placed on top of contaminated sediment as a physical barrier to prevent the contaminant release into the overlying layer and to sequester contaminants in sediments (Gu et al. 2017; Perelo 2010).

The choice of suitable capping material is, therefore, of particular importance to mitigate the contaminant release and other environmental impacts (Zhu et al. 2019). Capping materials used in laboratories and field studies include...
increase in hydraulic retention time by the impoundment will improve the overlying water quality (Lee et al. 2018). The accumulated sediments due to the altered flow patterns seem to deteriorate the overlying water quality (Lee et al. 2018). The increase in hydraulic retention time by the impoundment may influence sedimentation of inorganic/organic nutrients and also the gradual change of stratification patterns (Fantin-Cruz et al. 2016). To our best knowledge, there has been no study on the capping treatment for the sediments formed by the construction of artificial facilities in rivers.

In this work, we applied the CRMs (as a reactive layer) and sand and NWFM (as the armor layer) to investigate their remediation potential for contaminated sediments formed by artificial impoundments. Under their different combination uses, the capping efficiencies to interrupt the N and P release from sediments to the overlying water were compared. We compared the performance of sand and NWFM as arms of capping layers and monitored T-N, NH4-N, NO3-N, T-P, and PO4-P concentrations in overlying water and calculated nutrient fluxes from the sediment layer to the overlying water. Other environmental parameters were also observed, such as pH, oxidation reduction potential (ORP), electrical conductivity (EC), and dissolved oxygen (DO), to investigate any possible impacts or correlations to the capping-enhanced sediment remediation.

**Materials and methods**

**Sampling for water and sediment**

We selected a sampling site at a location (36° 28′ 56″, 127° 17′ 15 ″), i.e., about 6 km upstream of the Sejong weir (constructed in 2009), where Geum River is curved, resulting in sediment deposition. The sediments were collected using a Van Veen grab sampler and mechanically mixed to be uniform before use. We also sampled 40 L of river water using a polyvinyl chloride airtight container and subsequently filtered it using a GF/C filter (1.2 μm pore size, Whatman, UK) to remove large debris and suspended particulates.

**Capping materials**

All the CRMs, ATT, DLM (Rexem Co., Pohang, Korea) and SPL (Sigma-Aldrich), were sieved with a standard sieve No. 200 (75 μm) to use upper-sized particles only. Thermal treatment under anoxic conditions was performed to improve the adsorption capacity of these materials using a tube furnace (CRFT 830S, Dongseo Science Co., Ltd., Korea), using optimal temperatures of ATT, DLM, and SPL, for the highest phosphate removal at 700 °C, 900 °C, and 950 °C, respectively, (Kim et al. 2018a, 2018b; Hong et al. 2020). Their physicochemical properties and phosphate adsorption capacity can be found elsewhere (Hong et al. 2020; Kim et al. 2018a, 2018b), as summarized in Table 1. The NWFM samples (E&H company, Pochon, Korea) have an average pore diameter of 15.64 μm and a thickness of 300 μm, which were synthesized from polypropylene via the melt-blown
method (E&H Company). The clean sand (>90% of SiO₂, Jumunjin silica sand Co., Korea) was washed with deionized water before use as armour for the cap layer.

**Lab-scale sediment incubation experiments**

Nine incubation experiments (three different capping adsorbents × three different armors) and two controls (uncapped condition) were performed to evaluate the efficiencies of ATT, DLM, and SPL to block the N and P release, as shown in Fig. 1. Four armoring conditions were applied to investigate the efficiency of capping materials: (1) no capping; (2) 1 cm capping of adsorbent under NWFM; (3) 1 cm capping of adsorbent under 1-cm sand armor; and (4) 1 cm capping of adsorbent under 2-cm sand armor. In the absence of capping materials or sand armor layers, sand was positioned below the sediments to make a total thickness of 9 cm and provide the same interval between the bottom of the water column and the sampling hole. Sediment incubation experiments were performed using a cylindrical tank with an inner
radius of 7.5 cm and a depth of 25 cm, and the sampling hole was positioned 10 cm above the base side. Through the sampling port, 10 mL of overlying water was obtained using a disposable syringe to analyze the concentrations of N and P. A DO meter (HI9146, Hanna Instruments, Romania) was implanted in the middle of the caps and sealed using silicone resin paste. The sampled river water of 3 L was injected into the cylindrical tank using a peristaltic pump (Model 7527–15, Cole-Parmer, USA) at a mild flow rate of 20 mL/min to minimize any potential erosions of sediments and capping materials. The cylindrical tanks containing sediments, capping materials, and mixed water were maintained in an incubator (IB3-15, JEIO Tech Co., Korea) at a constant temperature of 25 °C without lights for 70 days. Water was sampled at 1, 2, 3, 7, 14, 21, 28, 42, 54, and 70 days after the initiation of the incubation to analyze environmental parameters (pH, EC, DO, and ORP) and different N and P species concentrations (NH$_4$-N, NO$_3$-N, T-N, PO$_4$-P, and T-P). The procedures for analyzing the nutrient concentrations in sediments and overlying water were provided in Supplementary Information (Sect. 2.4). The flux of nutrients per unit area during a day was calculated using Eq. S1. The capping efficiency is the ratio of the flux in the different capping conditions to the flux in the uncapped condition, and it was calculated using Eq. S2. After the end of experiments, the four types of P fraction (Adsorbed-P, Non-apatite inorganic P, Apatite-P, and Residual-P) under capping materials were analyzed using methods reported by Hieltjeste and Lijklema (1980). The detailed procedure for analyzing each P fraction was described in Supplementary Information (Section 2.5). The sediments at a depth of 1–5 cm after 70 days of experiments were mechanically mixed, and 50 g of the homogenized sediment was centrifuged at 3500 rpm for 30 min to extract pore water. The separated pore water was filtered using a filter paper (0.45 μm, polypropylene, Whatman, USA) before analyzing T-N and T-P concentrations in the pore water.

Results and discussion

Properties of river water and sediments

The properties and concentrations of the river water and sediments obtained from the sampling sites described in the “Sampling for water and sediment” section are presented in Table S1. DO and T-P concentrations were 8.71 and 0.13 mg/L, qualified to be Level 1 and Level 3 standard, respectively, by the Korean water quality standards (Levels 1–6) for rivers. Both SS and TN concentrations were found to be highly contaminated as they are classified at Level 6. The COD concentration in water is at Level 5, and COD in sediments was within the moderate pollution range (40,000–80,000 mg/kg) by the US Environmental Protection Agency (US EPA) sediment criteria. In the sediments, T-N and T-P contents were 2478 and 839 mg/kg, respectively, which are below the sediment quality guidelines (T-N: < 5600 mg/kg and T-P: < 1600 mg/kg, respectively) of the Korean Ministry of Environment, which is less strict than those of US EPA guidelines (T-N: > 2000 mg/kg and T-P: > 650 mg/g, respectively). The organic matter content of the sampled sediments was measured as 9.98%, which was higher than the 8%, to be classified as seriously polluted sediment by US EPA guidelines. The high contents of organic matter, N, and P in the sediments indicate that the nutrients in the sediments may be high enough to lead to eutrophication.

Change of water environment parameters under different capping conditions

Figure S1 shows variations of DO, pH, EC, and ORP under the capping of CRMs and armoring materials along with those in the uncapped condition for 70 days. The DO concentrations of uncapped sediment decreased continuously below 0.5 mg/L within 16 days and reached zero on day 30, but those under capped conditions approached finite plateau values. The average concentrations of DO in the overlying water were in decreasing order (unit: mg/L): S1/SPL (4.8 ± 0.9) > S1/DLM (4.7 ± 0.9) > S2/DLM (4.5 ± 1.1) > S2/SPL (4.4 ± 1.2) > NWFM/SPL (4.1 ± 1.0) > S2/ATT (4.0 ± 1.6) > S1/ATT (3.6 ± 1.5) > NWFM/DLM (2.3 ± 1.7) > NWFM/ATT (1.6 ± 2.0) > UN-CAP (0.9 ± 1.8). Higher DO concentrations were observed in the overlying water above sand armor than NWFM because the thicker sand cap-layer retarded the DO depletion by interrupting organic matter release from sediments. The higher DO concentrations under capping conditions observed here indicate the valuable potential of capping materials for the remediation of eutrophic lake sediments. Our findings are supported by Yin and Kong (2015)’s work that managing higher DO concentrations in overlying water helps prevent the liberation of P from sediments, protect benthic animals, and restore submerged vegetation. Although the pH of UN-CAP was maintained at pH 7.7, the pHs of overlying-water under CRMs capping conditions were observed higher than that of UN-CAP. The pH increase under the capping condition was due to the alkaline nature of the capping materials: the dissolution of Ca$^{2+}$ from CRMs increases the pH of the overlying water as Ca is a base cation (Alvarado et al. 2020; McBride 1994). The decrease in pH under capping conditions from day 28 to day 70 indicates that the potential toxic effect associated with pH decreases over time. When the incubation experiments were finished, the pH under capping with SPL and DLM was measured about pH 11, much higher than that under ATT capping. The impact of ATT on the pH
PO4-P concentrations over time were also observed in the such as eutrophication (Correll 1998). Increases in T-P and ably high, and 0.02 mg/L of T-P often causes problems and estuaries, 0.1 mg/L of T-P concentration is unaccept - problems in water bodies. In most lakes, streams, reservoirs, 0.727 mg/g, which can cause eutrophication and severe 59454

Release of nitrogen and phosphorus from uncapped/capped sediments to the overlying water

Figure 2 shows T-N, NH4-N, and NO3-N in the overlying water released from sediments during the experiment period. In Fig. 2a, the T-N concentration of UN-CAP continuously increased up to 22.7 mg/L; such an increase in T-N was also observed under the capping experiments except for S1/ATT and S2/ATT. In Fig. 2b, an increase in the NH4-N concentra - was also observed under uncapped conditions. The ratio of NH4-N/T-N under uncapped conditions ranged from 16.2 to 94.3%, and it was less than 50% at the beginning of incubation but higher than 75% after day 7. A high fraction of NH4-N in the N of overlying water was also observed in other studies (Gu et al. 2017), who reported that the portion of NH4-N increased from 64.0 to 91.8% as the incubation time increased. The release of N from sediments, mainly as NH4-N, may be attributed to the anaerobic condition after the depletion of DO ceased the nitrification of NH4 to NO3 (Beutel 2006; Lin et al. 2011). The higher amount of NH4-N released from uncapped sediments is consistent with the gradual decrease of NO3-N concentration that disappeared on day 21. The nitrification process is halted under anaerobic conditions, leading to the buildup of NH4 in the pore water occupied in the sediment layer and ensuing discharge of NH4 into the overlying water (Beutel 2006). In the capping condi - with DO remained, NO3 release from capped sediments was retarded during the incubation experiments.

The T-P and PO4-P concentrations in the overlying water during the incubation experiments are shown in Fig. 3. Both T-P and PO4-P increased continuously throughout the incubation experiments for the uncapped sediments. The T-P concentration under uncapped conditions increased up to 0.727 mg/g, which can cause eutrophication and severe problems in water bodies. In most lakes, streams, reservoirs, and estuaries, 0.1 mg/L of T-P concentration is unacceptably high, and 0.02 mg/L of T-P often causes problems such as eutrophication (Correll 1998). Increases in T-P and PO4-P concentrations over time were also observed in the capped conditions, but these were lower than those of UN-CAP. The ratio of PO4-P to T-P in the uncapped condition ranged from 54.2 to 93.0%, with an average of 69.5%. The effect of the capping treatment for the release of T-P and PO4-P is described in the “Flux and capping effi - of nitrogen and phosphorus under different capping conditions” section.

Flux and capping efficiency of nitrogen and phosphorus under different capping conditions

The fluxes and capping efficiencies of T-N, NH4-N, and NO3-N under different capping conditions were calculated, and the results are illustrated in Fig. 4. The fluxes of T-N in decreasing order are as follows: NWFM/SPL > NWFM/ DLM > UN-CAP > S1/DLM > S1/SPL > S2/DLM > S2/ SPL > NWFM/ATT > S1/ATT > S2/ATT. The T-N fluxes of NWFM/SPL and NWFM/DLM were higher than that of UN-CAP, indicating that the capping treatment using NWFM/SPL and NWFM/DLM was ineffective for blocking the release of T-N. In Fig. 4b, the capping efficiencies for T-N under NWFM capping were less than two different sand armor depths under all CRM cases. These results indicate that the sand armor shows better performance for blocking the release of T-N than NWFM, an observation that has also been noted by other researchers (Jiao et al. 2020; Lampert et al. 2011). The thicker the capping layer, the greater the diffusion distance between sediments and overlying water, and more time is required for nutrients to travel from sediments to overlying water (Jiao et al. 2020). A higher capping efficiency for T-N under the 2-cm sand armor than the 1-cm sand armor was observed under all three different capping materials, and their difference ranged from 4.4 to 9.0%. The effect of capping treatment on NH4-N was similar to T-N, and the capping efficiencies for NH4-N in increasing order are as follows: NWFM/SPL > NWFM/DLM > S2/ DLM > S1/DLM > S1/SPL > S2/SPL > NWFM/ATT > S1/ ATT > S2/ATT. This result can be expected because the T-N released from sediment to overlying water was mainly composed of NH4-N, as described above. The NH4-N capping efficiencies of CRMs under NWFM were also less than that under sand armor, but the difference in NH4-N capping efficiencies between sand armor depths of 1 cm and 2 cm was not significant.

A lower flux of NH4-N was observed under all capping treatments than UN-CAP, and the NH4-N fluxes of ATT were lower than those of the other two capping materials. This result can be explained by higher adsorption capacity (11.74 mg/g) of ATT for NH4-N than the other two materials (SPL: 0.60 mg/g; DLM: 0.52 mg/g). Ammonium can be removed from water by ATT through electrostatic adsorption and ion exchange (Sun et al. 2017). The higher T-N flux of DLM capping is consistent with the higher T-N concentra - in pore water under DLM capping than the other two CRMs (Fig. S2(a)). Ca2+ and Mg2+ ions dissolved from the DLM surface can inhibit the adsorption of NH4+ to the ion exchange sites on the sediments (Alvarado et al. 2020).
Fig. 2 Changes in a T-N (mg/L), b NH₄-N (mg/L), and c NO₃-N (mg/L) concentration in the water overlying sediment (UN-CAP), different Ca-rich minerals (SPL, ATT, and DLM), and armor materials (S1, S2, and NWFM) during 70 days of laboratory incubations.
The fluxes and capping efficiencies of T-P and PO₄-P under uncapped and capped with different CRMs and armors are presented in Fig. 5. The lower T-P flux under capping conditions than UN-CAP indicates that all capping materials used in this study were useful for interrupting T-P release from sediments. Similar to T-P, the effect of capping was also observed in the reduction of the PO₄-P flux. This result was expected because the T-P in the overlying water accounted for approximately 70% of T-P, as described in the “Release of nitrogen and phosphorus from uncapped/capped sediments to the overlying water” section. For all capping materials, sand armor with a depth of 2 cm was more effective than 1 cm. In contrast to NH₄-N and T-N fluxes, the T-P and PO₄-P fluxes between the sand armor and NWFM did not show significant differences, and the PO₄-P and T-P fluxes of SPL with 1 cm of sand were higher than those of SPL with NWFM. The negatively charged surface of the sand was not adequate for adsorbing the anion PO₄³⁻, but the sand layer blocked the release of PO₄-P only by acting as a physical barrier interrupting PO₄-P diffusion. Compared to the flux of T-N and NH₄-N, the capping efficiencies for T-P and PO₄-P showed a significant difference in capping material. ATT was the most effective capping material for interrupting the release of T-N and NH₄-N; however, it was less effective for T-P and PO₄-P than SPL and DLM. This result is also consistent with the order of the PO₄-P adsorption capacity of CRMs, as shown in Table 1. The adsorption capacity for PO₄-P follows in decreasing order: DLM (385.5 mg/g) > SPL (172.3 mg/g) > ATT (54.5 mg/g), and this order is inverse proportional to the order of PO₄-P flux and proportional to the capping efficiencies for PO₄-P. The higher adsorption capacity of DLM than the other two CRMs is consistent with
more Ca\(^{2+}\) elution from DLM (613.5 mg/L) than SPL (299.6 mg/L) and ATT (231.5 mg/L). In addition, higher Ca\(^{2+}\) concentration under DLM capping (24.8 mg/L) was prolonged until the end of the experiment than SPL (6.5 mg/L) and ATT (6.4 mg/L). Ca\(^{2+}\) eluted from SPL and DLM can enhance the removal of PO\(_4^{3-}\) in the overlying water by forming calcium-bound P (Ca–P) precipitation (Hong et al. 2020; Kim et al. 2018b; Yin et al. 2013).

The relationship between T-P flux and its concentration in pore water (Fig. S2(b)) was not linearly correlated, inferring that most of the P released from sediments was adsorbed and sequestrated in the capping layer.

**Effect of capping treatment on the P fractions in sediments**

The P fractions, including Adsorbed-P, NAI-P, Apatite-P, and Residual-P of sediment under different capping treatments, are presented in Fig. 6a. Under all experimental conditions, most of the P in the sediment was present in the form of NAI-P and Residual-P, and the tiny quantity of Apatite-P in sediments can be ascribed to the dissolution of Ca-associated P as river sediments turn into floodplains (Zehetner et al. 2009). The Adsorbed-P, a weakly adsorbed state, accounts for less than 1% of the total amount of P in sediments. The content of Adsorbed-P in UN-CAP was 5.5 mg/kg (Fig. 6b), which is equal to 0.5% of the T-P. The lower fraction of Adsorbed-P in UN-CAP compared to other conditions can be explained by a more incredible amount of P released from sediments into the overlying water, as shown in Fig. 3. A lower Adsorbed-P fraction was also observed under ATT capping than under SPL and DLM capping. This result is also consistent with the higher release of P to the overlying water under ATT capping than the other two CRMs. Under all capping conditions, the fraction of NAI-P, bound to Al/Fe and released from sediments under anaerobic conditions (Hong et al. 2020; Kaiserli et al. 2002), was less than uncapped condition
The Residual-P fraction, the most stable form of P and not readily released to overlying water, increased up to >80% under all capping conditions, indicating that the less stable P was converted to Residual-P through the CRM capping treatment. The transformation of mobile P to residual P fraction was also reported by other studies (Yin and Kong 2015), which reported that the transformation of P by CRMs is due to the P incorporation into the crystal structure of Ca minerals. The P fraction showed a distinct difference according to the CRMs used, but not the armor used. The 2 cm of sand armor showed the highest Residual-P fraction for SPL and DLM, but the lowest Residual-P fraction was observed under ATT.

### Conclusions

The results suggest that the application of Ca-rich minerals can remediate sediments deposited by the construction of artificial impoundments. The effectiveness of the sand layer and NWFM was also evaluated as armor materials. While DO was depleted under the uncapped condition on day 30, the DO under all capping conditions was preserved during the 70 days of the incubation experiment. The pH under the use of SPL and DLM significantly increased, and the pH of ATT was slightly higher than that of UN-CAP. The ATT effectively blocked the liberation of T-N and NH$_4$-N from sediments by adsorbing ammonia via ion exchange. Less PO$_4$-P and T-P from sediments were released from sediment capped with DLM than the other materials, which is attributed to the higher adsorption capacity of DLM for PO$_4$$^{3-}$. A thicker sand layer retarded the release of N and P from sediments by increasing the diffusion distance between the sediments and the overlying water. Higher Residual-P and lower Adsorbed-P fractions were obtained using CRMs capping than under uncapped conditions, indicating that P sequestration by capping was feasible. ATT is a useful capping material for interrupting N release while not having a significant influence on the pH. Owing to its low blocking efficiency of the P release, however, ATT...
should be mixed with a material such as DLM, which has excellent P adsorption capacity.

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Data Availability All data generated or analyzed during this study are included in this published article. 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 Not applicable.

Consent for publication Not applicable.

Conflict of interest The authors declare no competing interests.

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