

A Comparative Study on the Use of Seawater and Sea Salt in Nutrient Elimination

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영양염 제거에서 해수 및 해수염에 관한 비교연구

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Abstract : An excess in the nutrients such as nitrogen and phosphate leads to a phenomenon called eutrophication. In order to avoid this, numerous methods have been used to remove excess nutrients in the water. In this study, the use of a chemical method was assessed through the formation of magnesium ammonium phosphate. The difference in the removal efficiency of seawater and sea salt solution as primary sources of Mg^{2+} ions and Ca^{2+} ions for the formation of magnesium ammonium phosphate (MAP) and hydroxyapatite (HAP) respectively, were observed, taking into account the changes in pH and concentration. The results showed that seawater removed about 90% phosphate and less than 50% ammonia in sewage water condition, whereas the sea salt solution removed almost 90% phosphate and 70% ammonia in solution at pH 9 and 10 mM concentration of sea salt which further increases as the optimum Mg/PO_4^{3-} , NH_4^+ ratio reaches 2. The difference in the removal efficiency of seawater and sea salt was due to the fact that the set-ups were prepared in different conditions. This study suggests that both seawater and sea salt can be used to remove nutrients from the water. The relatively higher removal of phosphate can be explained by the formation of HAP from free Ca^{2+} ions initially present in seawater and sea salt solution.

Key Words : Eutrophication, Hydroxyapatite, Struvite, Seawater, Sea salt, Magnesium ammonium phosphate

요 약 : 부영양화를 일으키는 대표적인 영양물질인 질소와 인을 제거하기 위하여 많은 연구들이 진행되어오고 있다. 본 연구에서는 질소와 인을 제거하기 위하여 해수 및 해수염에 존재하는 마그네슘과 칼슘을 사용하여 스트루바이트와 수산화인회석을 만들어 침전을 시켰다. 실험의 목적은 해수와 해수염을 사용하여 pH와 농도의 변화에 따른 영양염의 제거율을 비교평가 하였다. 하수의 실험조건에서 해수를 사용한 결과 인의 제거율은 90%, 질소의 제거율은 50%로 나타났다. 또한 pH 9, 질소와 인의 농도 10 mM, Mg/PO_4^{3-} , NH_4^+ 의 비율 2의 조건에서 해수염을 사용하여 실험한 결과 질소의 제거율은 90%, 인의 제거율은 70%로 나타났다. 상대적으로 인의 제거율이 높은 이유는 해수를 사용한 경우 질소와 인의 몰 농도의 차이에서 비롯되었으며, 해수염을 사용한 경우 해수에 포함된 칼슘이 인과 반응하여 수산화인회석으로 침전 제거되었다고 할 수 있다. 수중의 질소와 인을 제거를 위하여 해수와 해수염을 사용한 결과 높은 제거율을 나타내었다.

핵심용어 : 부영양화, 수산화인회석, 스트루바이트, 해수, 해수염

1. Introduction

Excessive amount of nutrients in the water leads to further environmental problems primarily caused by eutrophication. Eutrophication can induce high biochemical oxygen demand that

can reduce the dissolved oxygen levels in the receiving water significantly, and in turn may result to fish kills and other ecological and aesthetic concerns(Vohra and Selimuzzaman, 2010). Nutrients such as nitrogen and phosphorous are the main drivers for this phenomenon, thus, reduction, if not complete removal of these nutrients is needed to prevent the incidence of eutrophication. Several methods have been found to be effective in removing such

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nutrients including chemical and physical approaches. In this study however, the use of a chemical method was utilized.

Ammonia and phosphate are the most common forms of these nutrients which react with Mg^{2+} and Ca^{2+} ions (from seawater and sea salt solution) to form magnesium ammonium phosphate (MAP) or struvite crystals ($MgNH_4PO_4 \cdot 6H_2O$) and calcium phosphate in the form of hydroxyapatite (HAP) ($Ca_{10}(PO_4)_6(OH)_2$), respectively. This method of chemical precipitation can be applied to remove excess NH_4^+ and PO_4^{3-} in the water as the insoluble precipitate of MAP and HAP are produced. Since the raw materials for these chemical processes are often expensive, Shin and Lee (1997) suggested the use of seawater that initially contains Mg^{2+} of 44 mmol kg^{-1} concentration and $9.76 \text{ mmol kg}^{-1}$ of Ca^{2+} , as an excellent coagulant for nutrient removal from industrial wastewater. In another study by Matsumiya et al. (2000), seawater which contains 50 mmol kg^{-1} of Mg^{2+} removed phosphorous from the digester sludge dewatering liquor by crystallization of MAP. The use of sea salt for the formation of struvite crystal however, have not been thoroughly studied. Sea salt contains ions such as Na^+ , Cl^- , K^+ , Mg^{2+} , Ca^{2+} and SO_4^{2-} in larger amounts up to 472 mmol kg^{-1} , 537 mmol kg^{-1} , 9.9 mmol kg^{-1} , 55 mmol kg^{-1} , 9.9 mmol kg^{-1} and 25 mmol kg^{-1} , respectively (Atkinson and Bingman, 2000). These chemical components, therefore suggests that sea salt, can also be used to form struvite and calcium phosphate as Mg^{2+} and Ca^{2+} ions are present in ample amounts in order to form MAP and HAP. Additionally, sea salt was expected to remove a slightly higher amount of nutrients since its chemical components contains higher amount of the needed Mg^{2+} ions. This study aimed (i) to show the difference between the removal efficiency of seawater and sea salt solution as the source of magnesium and calcium ions. Moreover, (ii) the effects of the reaction time, pH and concentration of the materials used to remove the nutrients were also observed.

2. Materials and Method

Seawater was first obtained in the coastal area of Daebandong in Mokpo city, Korea. All seawater samples were filtered initially using a $200 \mu\text{m}$ mesh net to remove any suspended particulate matter. Samples were assumed to contain negligible amount of nutrients. In order to determine the effects of Mg^{2+} concentration, four volumes of seawater were tested; 25, 50, 75 and 100 mL in a 500 mL total sample volume. Experiments were carried out in an ambient laboratory temperature with a jar tester. Solutions (99.9% purity) of NH_4Cl and KH_2PO_4 were prepared at 1000 ppm. The

standard ammonium solution was mixed with standard phosphate solution to obtain a concentration of 100 ppm (7.1 mM) NH_4^+ -N and 50 ppm (1.61 mM) PO_4^{3-} -P nutrient, respectively. Concentrations of NH_4^+ -N and PO_4^{3-} -P nutrient were set to simulate sewage water condition. Several mixtures were made and the pH of each mixture was adjusted to pH 6, 7, 8, 9 and 10. The solutions were constantly agitated at 140 rev/min speed. Samples were drawn after 1, 10 and 30 minute/s of the reaction time and filtered through $0.45 \mu\text{m}$ pore size membrane filter. The filtrates were then analyzed for ammonia and phosphate using Phenate method for Ammonia and Ascorbic acid method for Phosphate under a UV-Vis Spectrophotometer at 630 nm and 880 nm, respectively. To test for the removal at equal molar ratio, stock solutions of NH_4Cl and KH_2PO_4 were prepared at 1000 mM NH_4^+ -N and 1000 mM PO_4^{3-} -P, correspondingly. Stock solution of $MgCl_2$ was also prepared at 1000 mM Mg^{2+} . Right amount of ammonia, phosphate and magnesium chloride solutions were obtained from the stock solutions and diluted to 500 mL. The unadjusted pH was 5. To test the effect of pH and Mg^{2+} , constant NH_4^+ (1 mM) and constant PO_4^{3-} (1 mM) were used. The pH of each of the solutions was adjusted from 5 to 7, 8 and 9 using NaOH and HCl solutions. Five concentrations of Mg^{2+} were examined: 1 mM, 2 mM, 4 mM, 10 mM, and 50 mM. Experiments were carried out in an ambient laboratory temperature with a jar tester at 140 rev/min. All samples were obtained after 30 minutes and later filtered through $0.45 \mu\text{m}$ pore size membrane filter and were subjected to Phenate and Ascorbic acid methods for ammonia and phosphate, respectively. For sea salt, standard solutions of NH_4Cl and KH_2PO_4 were prepared at 0.1 mM respectively. A seasalt solution was also prepared with 0.2 mM concentration. Analysis of samples were done at differing concentrations from 0.1 mM, 1 mM and 10 mM from the mixture of NH_4Cl and KH_2PO_4 solutions mixed with seasalt solution all in the same ratio of 1 at pH 9. Similarly, another set-up was made by preparing the same mixture of NH_4Cl and KH_2PO_4 solutions mixed with seasalt solution at 10 mM this time, with differing ratios of 0.5, 1, 2, and 5 at the same pH 9. The mixtures were agitated using a magnetic stirrer and were allowed to react for 24 hours before the decanted liquid was subjected to Phenate method and Ascorbic acid method under a UV-Vis Spectrophotometer for ammonia and phosphate, respectively.

3. Results

3.1. Removal efficiency of seawater

A considerable increase in the removal of both nutrients has

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been observed since 1 minute of the reaction time. The significant reaction time to form struvite crystals, however, was found to be within 10~30 minutes. Fig. 1 shows the removal efficiency of seawater depending on the reaction time. Higher removal of PO_4^{3-} was observed compared to that of the NH_4^+ since the initial concentration of NH_4^+ is greater (7.1 mM) relative to that of the initial concentration of PO_4^{3-} (1.61 mM) and this is following the sewage water condition. In the figure, it was shown that further increase in the reaction time does not have a significant effect in the removal efficiency of seawater. In a related study by Stratful et al. (2001), only 4% more of Mg^{2+} and PO_4^{3-} ions were removed even after the reaction time was increased from 1 minute to 180 minutes.

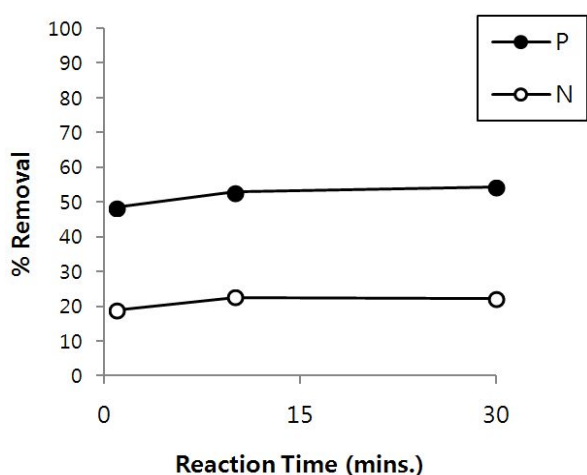


Fig. 1. Removal efficiency of 25 mL seawater depending on the reaction time in waste water condition.

Fig. 2 shows the effect of pH in the removal of nutrients. Increasing the pH from 6 to pH 9 was found to similarly, increase the removal efficiency by 80% for PO_4^{3-} and about 35% for NH_4^+ , further increase in the pH up to 10 also improved the removal for both nutrients, especially for phosphate reaching to about 90% elimination. The results can be explained by the fact that based on theoretical calculations, only 0.758 mg NH_4^+ can be removed per liter of seawater compared to that of 1.68 mg PO_4^{3-} removed per liter seawater following the reaction of MAP with a ratio of 1:1:1 (Doyle and Parsons, 2002) also, the formation of MAP was observed at pH 8 and beyond. The increasing removal of both nutrients can therefore be traced to the formation of MAP in the solution. MAP formation is a pH-dependent process in which as the pH increases, struvite occurrence also increases, hence, the larger removal of both nutrients. For PO_4^{3-} however, it can be seen

that a very drastic increase happened from pH 9 and above. This is because another ion, which is Ca^{2+} initially present in seawater, reacts with PO_4^{3-} in the solution. Formation of hydroxyapatite (HAP) is also a pH-dependent process. In recent studies, HAP can only occur at pH 9 and above which is favored by the addition of OH^- ions coming from the raise in the pH of the solution.

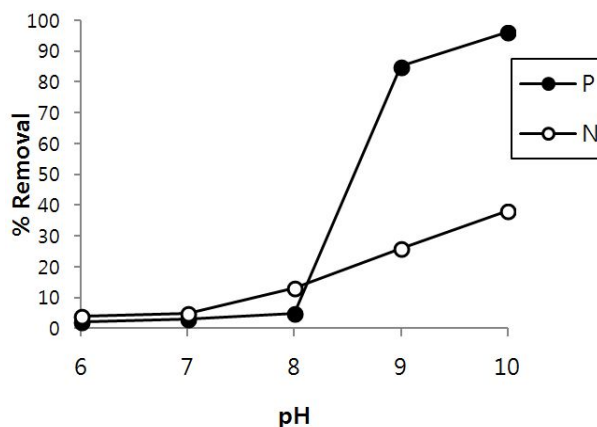


Fig. 2. Removal efficiency of 100 mL seawater depending on the pH in waste water condition.

The slightly higher removal of NH_4^+ from pH 6~8 can be explained by the fact that NH_4^+ is highly volatile, which means that through time, NH_4^+ can be converted to NH_3 gas and released in the atmosphere. This can be the reason for a slightly higher removal of NH_4^+ in lower pH. In a study by (Hao et al., 2008), MAP formation is optimum at pH ranges 7.0~7.5, this is lower compared to the studies previously made by Stratful et al. (2001). This study however, follows the results shown by the latter that MAP formation may have occurred at pH 8 and above. The removal of phosphate and ammonium is found to be optimum at pH 9~10. In order to further prove that indeed, MAP was formed at sewage water condition, the following graphs show the removal of both nutrients in several set-ups. Fig. 3 shows the removal efficiency at different seawater volumes. It can be observed that greater removal of PO_4^{3-} and NH_4^+ was achieved as the volume increases. Theoretically, following the stoichiometric reaction of struvite, removal efficiency should be the same for both nutrients as the ratio for MAP is 1:1:1. However, the succeeding data (Fig. 4) does not follow the said phenomenon. Instead, the removal efficiency did not increase when the concentration of Mg^{2+} is beyond 10 mM regardless of the pH. This disparity may be explained by some reasons including; the reaction of struvite may not necessarily follow a 1:1:1 molar ratio. Jaffer et al. (2002) proved

that the molar ratio of magnesium and phosphorous was at least 1.05:1 so that the struvite formation may proceed. If the ratio is decreased, the phosphorous removal is observed to be about 72 %.

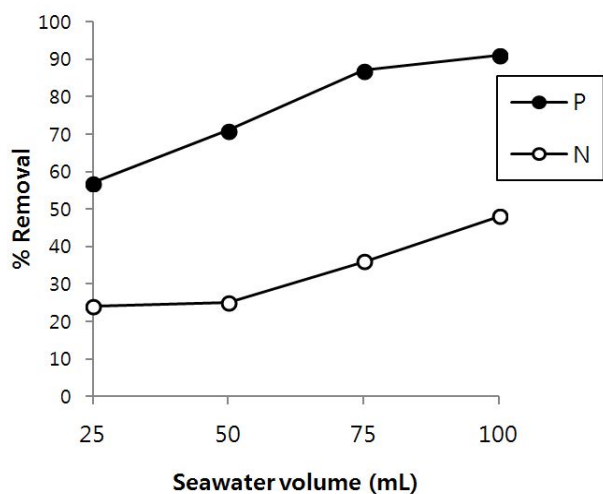


Fig. 3. Removal efficiency of different seawater volumes at pH 9 in sewage water condition.

Experiments made by Stratful et al. (2001) showed that the stoichiometric ratios for struvite formation should be increased to 3, 5, and 7 so that measurable amount of struvite can be produced. They had found out that whenever the molar ratio is 1:1:1, there was an excess of ammonium. Thus, can be observed in this study, in which ammonia was removed in low amount. This means that the precipitate was composed mostly of magnesium phosphate as compared to MAP (Bouropoulos and Koutsoukos, 1999). In this figure however, a difference in the removal for both nutrients can be explained by the fact that experiments were made in sewage water condition which has 100 ppm $\text{NH}_4^+\text{-N}$ and 50 ppm $\text{PO}_4^{3-}\text{-P}$ of total nutrient concentrations.

Additionally, the difference in the removal of NH_4^+ and PO_4^{3-} can be explained by the formation of HAP for PO_4^{3-} in which it can only occur at pH 9 and above. The removed NH_4^+ therefore, was only by the formation of MAP alone and PO_4^{3-} removal was due to MAP and HAP at higher pH. This graph also shows that in order to remove by seawater, almost 20% of the total sewage water volume is equivalent to the volume of seawater needed to attain the best results for removal of nutrients. Although this can remove PO_4^{3-} best and pH should be adjusted to 9 to attain the optimum removal efficiency. Fig. 4 shows the removal of phosphate and ammonia at equal molar concentrations in which it can be observed that removal of both nutrients is worst at pH

below 7. Stratful et al. (2001) explained in their study that struvite yield is affected when the solution is below pH 10 and that the addition of NaOH would stabilize the pH of the solution thereby making the formation of MAP to continue until the reactant becomes limiting. Furthermore, the removal of phosphate increased with increasing pH however, this is only true with the presence of excess magnesium ions. The optimum pH for both nutrients was found to be at pH 9. It had also been proven that ammonia exists in ionized form at pH below 7 regardless of the temperature (Lin and Chang, 1996).

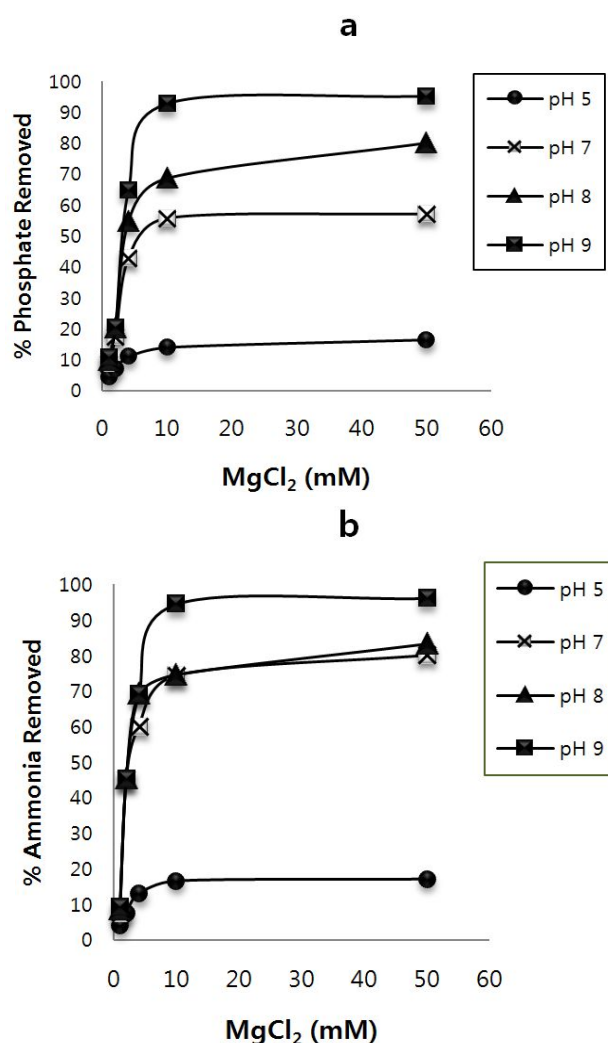


Fig. 4. Removal of (a) phosphate and (b) ammonia at equal molar concentrations: 1 mM NH_4^+ , 1 mM PO_4^{3-} , 1 mM Mg^{2+} .

The graph above shows that as the magnesium ion increases from 1 mM to 10 mM, removal efficiency increases for both nutrients, therefore MAP formation indeed follows a 1:1:1 ratio.

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However, no significant increase can be observed beyond 10 mM MgCl_2 . According to Lee et al. (2002), removal of nutrient using Mg^{2+} is also governed by a seed material regardless of the pH. The overall removal of NH_4^+ was enhanced by adding a pre-formed struvite as the seeding material. When the seeding material is low or absent, conversely, the precipitation may precede either by crystal nucleation or crystal growth. In the previous data however, low removal of NH_4^+ compared to PO_4^{3-} was observed and this is due to the fact that the set-up was simulated to be in a sewage water condition, other than that, if the ratio for PO_4^{3-} and NH_4^+ and Mg^{2+} are the same, then formation of MAP will most likely be the same.

3.2. Removal efficiency of sea salt

For sea salt solution, a preliminary assay was conducted to know the optimum concentration and pH in which the solution can remove both of the nutrients. It has been observed in the experiment that the best concentration for both solutions (NH_4Cl and KH_2PO_4) together with sea salt, is 10 mM at pH 9, for the removal efficiency of ammonium and phosphate. Fig. 5 and Fig. 6 below show the preliminary results for the removal of phosphate.

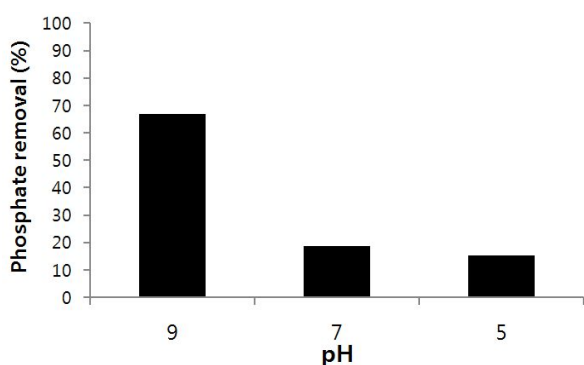


Fig. 5. Removed phosphate by 10 mM sea salt in varying pH.

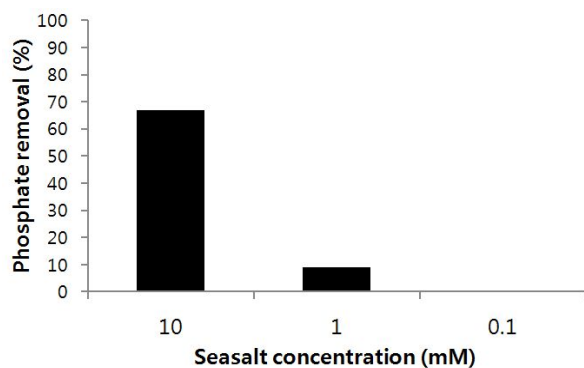


Fig. 6. Removed phosphate by sea salt at pH 9 with varying concentrations.

The graphs above show that the removal of phosphate through sea salt solution is affected by the increase in pH and concentration of the solution. High removal of phosphate was observed at pH 9 since the formation of MAP begins at pH 8 and above. The magnesium ions present in sea salt therefore reacts with phosphate and ammonium in the solution to form struvite crystals which leads to an almost 70% elimination of phosphate. Additionally, it has been proven that at pH 9 and above, HAP starts to form from the initial calcium ions present in the sea salt solution (Doyle and Parsons, 2002). Hence, the higher removal for phosphate relative to that of ammonia, as can be seen in the figures. Similarly, the same preliminary results were observed for the removal of ammonia in the solution in which the best pH and concentration of sea salt solution is at pH 9 and 10 mM respectively, as shown in Fig. 7 and Fig. 8.

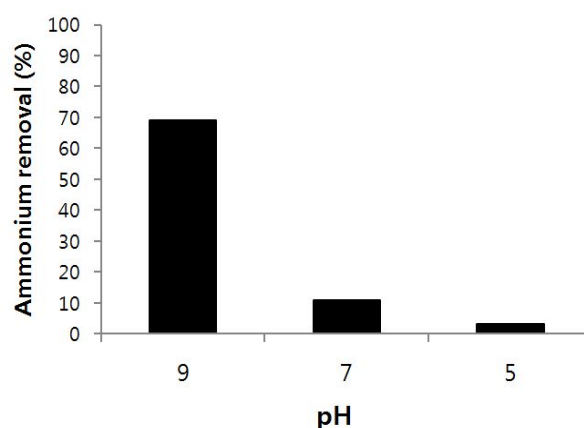


Fig. 7. Removed ammonia by 10 mM seasalt at varying pH.

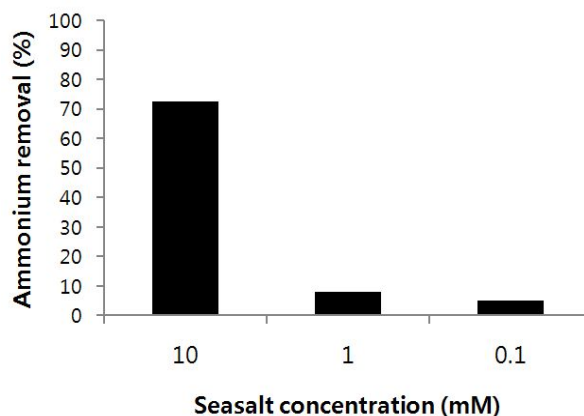


Fig. 8. Removed ammonia by seasalt at pH 9 with varying concentrations.

Both of the preliminary assays for phosphate and ammonia removal showed the best elimination at pH 9 and 10 mM of sea salt concentration. It can be observed that as the concentration greatly increases to 10 mM, the removal for both nutrients increases as well at pH 9. Reason for this is because, higher concentration of sea salt solution would mean greater amounts of Mg^{2+} ions initially present in the solution making more excess magnesium ions to react with phosphate and ammonium ions to form MAP (Fattah and Mavinic, 2011). Fig. 9 shows the experimental results for the removal efficiency of sea salt solution at 10 mM of concentration and pH 9 as the optimum condition for the removal of both nutrients. The graph below shows the removal efficiency of sea salt solution with regards to an increasing Mg/PO_4^{3-} , NH_4^+ ratio. It can be illustrated that there is a directly proportional relationship between the removal efficiency of both nutrients as the Mg/PO_4^{3-} , NH_4^+ ratio increase from 1 to 5. As the ratio increases however, there is a slow increase in the elimination of both nutrients and can be explained by the fact that excess Mg^{2+} ions may have reacted too fast to form MAP thus, as the ratio increases, more phosphate and ammonium ions have already reacted to magnesium ions to form struvite, making the unreacted excess Mg^{2+} ions to remain in the solution.

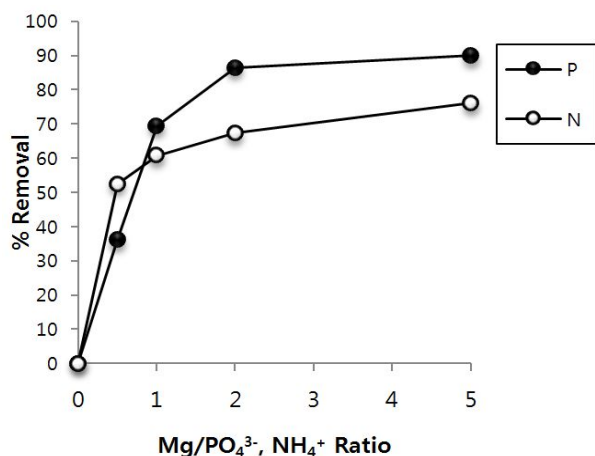


Fig. 9. Removal Efficiency of 10 mM seasalt at pH 9 in different Mg/PO_4^{3-} , NH_4^+ ratio.

The graph above shows that sea salt solution removal efficiency is higher compared to that of sea water for both nutrients; ammonia and phosphate, however, this is due to the fact that both set-ups were prepared differently. Previously, it was showed that phosphate removal reached to about 80 % elimination for seawater at sewage water condition, in this method, the removed phosphate

reached to about 90 %. In the same manner, a great increase in the removal of ammonia was observed in the study reaching to about 80% of removal compared to less than 20 % elimination using sea water. The small difference upon the removal of seawater and sea salt solution can be elucidated by the fact that, the composition of Mg^{2+} ions in sea water is relatively lower compared to pure sea salt which is about 44 mmol kg⁻¹ and 55 mmol kg⁻¹, respectively. A study by Yu et al. (2013) stated that the greater the concentration of excess Mg^{2+} ions, the greater the proportion of MAP forming in the solution. Hence, more precipitates will be formed leading to a greater removal of nutrients from the solution. Another difference in the removal can be observed in the graph is the slightly higher elimination of phosphate, again this can be accounted to by the formation of HAP as the Ca^{2+} ions are also present in the sea salt solution. HAP formation occurs at pH 9 and beyond, this experiment therefore, triggered the occurrence of HAP as the pH increases to 9.

4. Conclusion

The removal of nutrient via magnesium ammonium phosphate (MAP) formation is complete within 30 minutes of the reaction time regardless of the Mg^{2+} source. For seawater, the optimum pH is 10 and pH 9 when pure $MgCl_2$ was used. Since seawater also contain Ca^{2+} ions, phosphate was also removed by the formation of hydroxyapatite (HAP) which occur best at pH 9-10. Increasing the volume of seawater also increases its removal efficiency. This would mean that there would be more Mg^{2+} and Ca^{2+} available for reaction. Furthermore, the use of sea salt solution was observed to remove higher phosphate and ammonia in the solution at a certain condition. This condition was previously tested and resulted to the optimum pH and concentration being pH 9 and 10 mM, correspondingly. Higher removal efficiency was achieved as the Mg/PO_4^{3-} , NH_4^+ ratio also increases, as it would mean that more magnesium ions and calcium ions in the solution would react to the free phosphate and ammonium ions in the solution. MAP formation occurred favorably as the pH was at 9 and the concentration was at 10 mM. Similarly, the increase in the ratio would signify the increase in calcium ions which lead to the formation of HAP leading to a slightly higher removal for phosphate. Finally, the study therefore suggests that the use of sea water and sea salt solution can both be used in the removal of PO_4^{3-} and NH_4^+ ions in the water. There were only little differences in the removal however and that was because of the different initial concentrations of the solutions. Nonetheless, sea

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water can remove PO_4^{3-} and NH_4^+ in sewage water condition by up to 90% and 35%, respectively. Furthermore, sea salt was proven to remove PO_4^{3-} and NH_4^+ best at 10 mM concentration and at pH 9 of the solution which further increases as the ratio of $\text{Mg}/\text{PO}_4^{3-}$, NH_4^+ increases. Showing the optimum removal at $\text{Mg}/\text{PO}_4^{3-}$, NH_4^+ ratio of 2.

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