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MINI REVIEW

CHEMICAL PRECIPITATION AND REDUCTION METHODS FOR THE RESTORATION OF WATER FROM AQUACULTURE OPERATION

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Abstract

Effort towards combating water shortages and environmental problems such as eutrophication, which is associated with the indiscriminate discharge of aquaculture wastewater into the environment has resulted in the quest for an efficient aquaculture wastewater treatment system. This paper reviewed the importance and efficiency of chemical precipitation method as well as chemical reduction method in the treatment of aquaculture wastewater. The percentage efficiency of chemical precipitating agents such as aluminium sulphate, calcium oxide and ferric sulphate is dependent on the precipitant dosage and the pH of the solution. On the other hand, chemical reducing reagents such as hydroxylamine hydrochloride and modified chitosan composites have been effective in removing nitrate from wastewater. Nevertheless the percentage efficiency of chemical reducing reagents increases with an increase in initial concentration and contact time but decreases with a decrease in pH and an increase in temperature. Chemical precipitation and reduction methods have been reported to have high removal efficiencies for phosphate and nitrate from aquaculture wastewater.

Key words: Precipitation, reduction, phosphate, aquaculture, wastewater, efficiency.

Introduction

Aquaculture, also known as fish farming, has a long imperative history in many part of the world and has currently gained prominence by becoming one of the fastest growing food production industries due to the significant increase in demand for fish and seafood throughout the world, thereby providing healthy human nutrition, generating employment and foreign exchange for the global economy (Jinzhou *et al.*, 2017; Ariel and Jutta, 2014). Unfortunately for the aquaculture industry, the level of fish production is directly proportional to the amount of aquaculture wastewater generated. Although, the composition of aquaculture wastewater is directly proportional to the type of operation system and the type of feed fed to the reared species, the major sources of waste from aquaculture consist primarily of untreated water with excreta, fecal matter and uneaten feed from fish (Sohail, 2003). However, the reports from previous studies revealed that the major contaminants in aquaculture wastewater can be broadly grouped into organic carbon and nutrients (nitrogen and phosphorus), which require necessary treatment prior to recirculation or disposal, as a result of their negative impact on the aquatic ecosystem (Jiang and Graham, 1998; Hesterberg and Beauchemin, 2000; Sohail, 2003; Ariel and Jutta, 2014; Sengupta *et al.*, 2015; Jinzhou *et al.*, 2017). The organic matter negatively impact the aquatic ecosystem by reducing the level of dissolved oxygen and increasing the buildup of bottom sediments, on the other hand, high concentration of nitrates and phosphates has been reported to stimulate the uncontrollable growth of algae and other photosynthetic aquatic life, which results to eutrophication, thereby creating undesirable changes in the aquatic ecosystems, which threatens the survival of aquatic organisms (Nora'Aini *et al.*, 2005; Chang-jun *et al.*, 2007; Banu *et al.*, 2008; Ariel and Jutta, 2014; Li *et al.*, 2013; Xie *et al.*, 2014; Khodadadi *et al.*, 2017; Yazdani *et al.*, 2019). Furthermore, nitrogenous compounds such as ammonium and nitrite has been reported to impact toxic effects on aquatic life, if presents in high concentration. Moreover, the high concentration of nitrate in water bodies has been reported to be a potential public health threat due to its associated with blue babe syndrome (Matosic *et al.*, 2000; Jang *et al.*, 2004; Jung *et al.*, 2015; Schwantes *et al.*, 2015; Takaya *et al.*, 2016; Jinzhou *et al.*, 2017; Bashir *et al.*, 2017; Nujić *et al.*, 2017). As a result of the adverse environmental problems associated with effluents from aquaculture systems, the aquaculture industry has been criticized by various environmental groups (Sohail, 2003). Several international organizations have exerted enormous pressure on the treatment of wastewater before discharge in order to protect the aquatic environment and reuse of water resources (Ariel and Jutta, 2014, Bunting, 2001; Bunting, 2007; Dunets and Zheng, 2015; Choi, 2016; Li *et al.*, 2016). This review paper is aimed at emphasizing the importance and efficiency of chemical precipitation and reduction methods in the treatment of aquaculture wastewater.

The Efficiency of Chemical Precipitation and Reduction Methods in Aquaculture Wastewater Treatment

Chemical Precipitation Method

The efficiency of using metallic oxides and salts such as calcium oxide (CaO), aluminium sulphate, and iron sulphate as precipitating reagents in the removal of phosphorus from wastewater has been observed to be dependent on the precipitant dosage and the pH of the solution as shown in Figure 1 (Nassef, 2012). It was observed that 90 % of phosphate was removed when CaO precipitant was used at a dosage of 40 mg/L and pH value of 8.5-10. Aluminium sulphate at a pH of 4 was observed to remove 85 % of phosphate while iron sulphate at a pH of 8.5-10 was able to remove 80 % of phosphate from wastewater. It was however observed that a composite of the two precipitants has no effect on increasing the percentage removal of phosphate from wastewater (Nassef, 2012).

Similarly, the use of alum and lime precipitation in removing nutrient from secondary effluents at an optimum pH of 5.5, have been experimented and posited that 259 mg/L dose of alum exhibited removal efficiencies of 90 % (total phosphorus), 60 % (organic nitrogen), 55 % (chemical oxygen demand) and 25 % (NO_2-N and NO_3-N), while the use of 600 mg/L dose of lime with NaOH adjustment of solution pH to beyond 11, exhibited a 99 % removal of total phosphorus as shown in Figure 2 (Malhotra *et al.*, 1964). Reduction in the level of phosphate in wastewater by precipitation via the employment of lime has been reported to achieve 99 % phosphorus removal at a lime-phosphorus ratio of 1.5 and a pH between 8.6

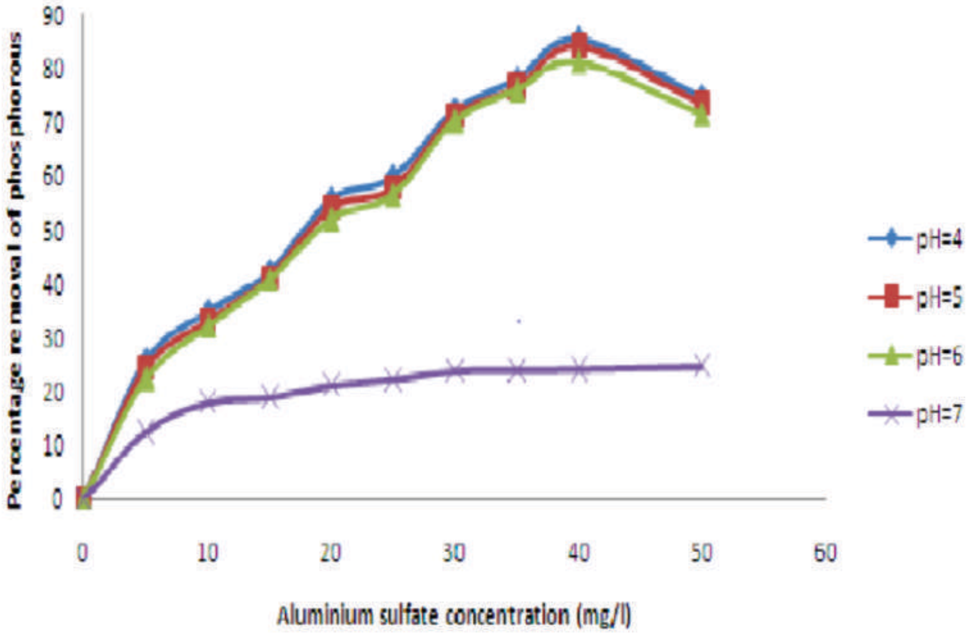

Figure 1: The effect of pH and Al_2SO_4 dosage in the removal of phosphorus from wastewater (Nassef, 2012).

to 9.0, It was however posited that the separation efficacy of the precipitate can be improved via flocculation by using guar gum or cationic starch (Dunets, 2014). The utilization of ferrous as well as ferric sulphate buffered with TRIS hydroxylmethyl amino methane at pH 7.3 to 7.6 has been experimentally proved to obtain 95 % removal efficiency of phosphorus from wastewater, it was observed that the ferrous phosphate precipitation reaction was completed in a period of 60 minutes while that of ferric phosphate precipitation required 100 minutes for completion, and a settling time of 100 minutes was reported for both precipitates (Rich, 2005). Likewise, the use of ferric chloride in the co-precipitation removal of phosphorus in sequencing batch reactor revealed 93, 87 and 64 % removal for total phosphorus, P-PO_4 and N-NO_3(mg/L) respectively and 93 % removal for COD as well as BOD_5, compared to biological method that displayed 42, 39 and 64 % removal for total phosphorus, P-PO_4 and N-NO_3(mg/L) respectively in addition to 79 and 80 % removal for COD and BOD_5 respectively. However, ferric chloride was observed to have inhibitory effect on microorganism activity (Costa *et al.*, 2019).

Research on the utilization of magnesium sulphate in the removal and recovery of nutrients as sturvite or magnesium ammonium phosphate (MAP) has been successfully carried out (Siciliano *et al.*, 2020), it was observed that a 97 % removal efficiency of nitrate and phosphate in a molar ratio form of NH^{4+}: Mg^{2+}:PO_4^{3-} of 1:1.5:1.5 can be achieved at an initial ammonium concentration of 600 mg/L and pH of 9 (Smet, 2000). Further studies by Jaffer *et al.*, (2001) on using magnesium hydroxide Mg (OH_2) and magnesium chloride ($MgCl_2$) at respective pH of 8.5 and 9 resulted in a corresponding average percentage phosphate removal of 68-91 % and 94-97%. In addition, Jia, (2014) reported the utilization of $MgCl_2$ as a precipitating agent and postulated that optimum pH of 9 to 9.5 is required for the

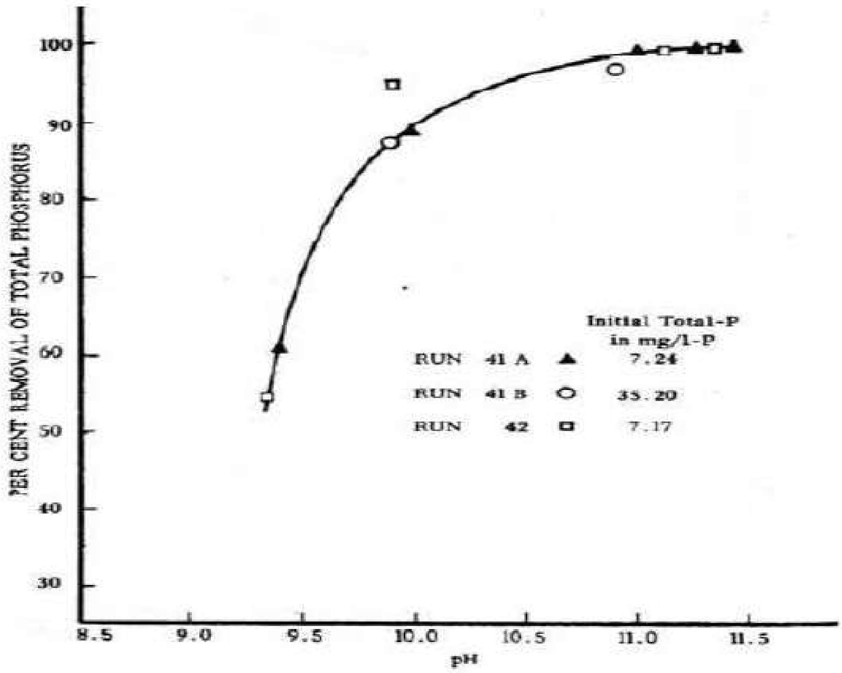

Figure 2: The removal of total phosphorus from wastewater using lime at pH adjustment (Malhotra *et al.*, 1964).

recovery and removal of ammonium-nitrogen and phosphate-phosphorus through struvite (MAP) precipitates formation from wastewater. The result of this experimental study is depicted in Figure 3 which revealed that the mass formation of crystal precipitate of stuvite (MAP) in a molar ratio form of NH^{4+}:Mg^{2+}:PO_4^{3-} of 1:1:1 from wastewater varies with the solution pH, while the percentage phosphate removal from wastewater increases from 96.7 to 99.9 % correspondingly with increase in solution pH from 8.0 to 11.0 (Jia, 2014). On the contrary, the ammonium-nitrogen removal from wastewater increases from 95.0 to 96.4 % with corresponding increase in solution pH from 8.0 to 9.0 but decline from 96.4 to 90.5 % when the solution pH increases from 9.0 to 11.0 (Jia, 2014). On the other hand, thermodynamic studies revealed that the mass and morphology of the struvite precipitated and the removal of ammonium and phosphate from wastewater via the precipitation of struvite crystal is not significantly influenced by a temperature range of 25 to 35 ^{0}C as depicted in Figure 4 (Jia, 2014).

A reduction of 18 % in the value of COD removal from wastewater has also been reported in precipitated struvite, while the presence of acidic medium (pH<7) has been reported to show lesser reduction in the removal of nutrients because of calcium ion interference which compete with magnesium ions to form an amorphous calcium phosphate changing the basic chemistry needed for MAP complex process and inhibiting struvite formation. However, the kinetics of the precipitation process was noted to occur via a first order approach due to its exhibition of a high correlation coefficient of 0.962 as shown in Table 1 (Suthar and Chokshi, 2011; Morales *et al.*, 2013).

Table 1: kinetic order for precipitation of phosphate as struvite from wastewater

Order of rea	R^2	*K*
1st order	0.962	0.4 h^{-1}
2nd order	0.958	5.64 1/mol h
3rd order	0.931	3.6 x 10^{-3} 1^2/mol^2 h

R^2= correlation coefficient, *K*= rate constant

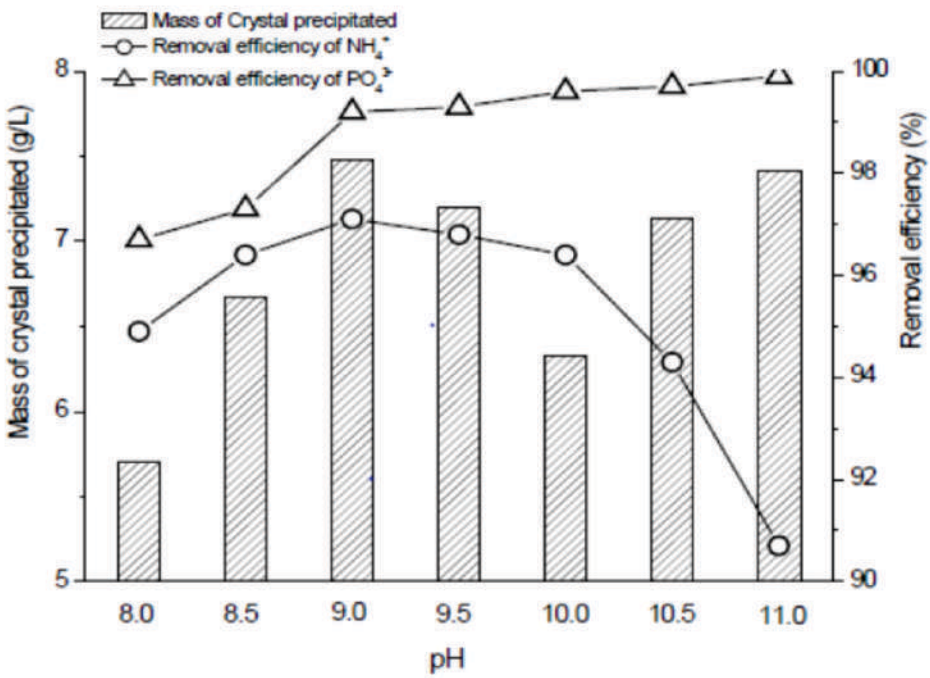

Figure 3: Removal of ammonium and phosphate in the form of mass crystal precipitate of struvite (MAP) (Jia, 2014).

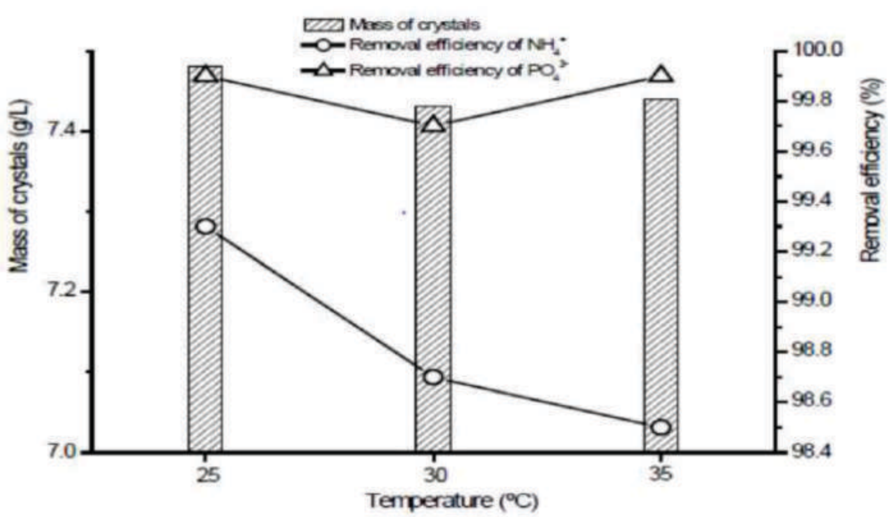

Figure 4: Influence of temperature on the formation mass struvite (MAP) crystals, removal of ammonium and phosphate in the form of (Jia, 2014).

The application of magnesium chloride ($MgCl_2$) in the removal of COD, nitrogen and phosphorus from wastewater has been reported to be technical to achieve, however 80 % reduction of COD has been reported when wastewater was treated with a dosage range of 35-40 mg/L of $MgCl_2$, while a maximum range reduction of 35-40 % (nitrogen) and 45-55 % (phosphorus) was observed to be removed from wastewater by using 30-35 mg/L and 25-30 mg/L of $MgCl_2$ respectively (Molahalli, 2011). The recovery of phosphorus from real wastewater sample by the use of electrochemically induce calcium phosphate precipitation has been reported to yield phosphorus concentration reduction from 8.0 to 4.3, 3.1 and 2.3 mg/L which correspond to a removal efficiency of 42.8, 62.1 and 71.5% for a period of 24, 48 and 72 hours respectively as depicted in Figure 5 and the phosphate removal percentage was attributed to the presence of calcium and magnesium ions in the real wastewater sample that removes phosphate in the form of calcium phosphate and magnesium phosphate (Lei *et al.*, 2017). Comparative study on electrochemical precipitation and conventional chemical precipitation of phosphorus revealed that chemical precipitation is more effective than electrochemical precipitation regarding removal efficiency, however a separation follow up is necessary for conventional chemical precipitation while removal and separation occurs simultaneously on the surface of the cathode in electrochemical precipitation system (Lei *et al.*, 2017).

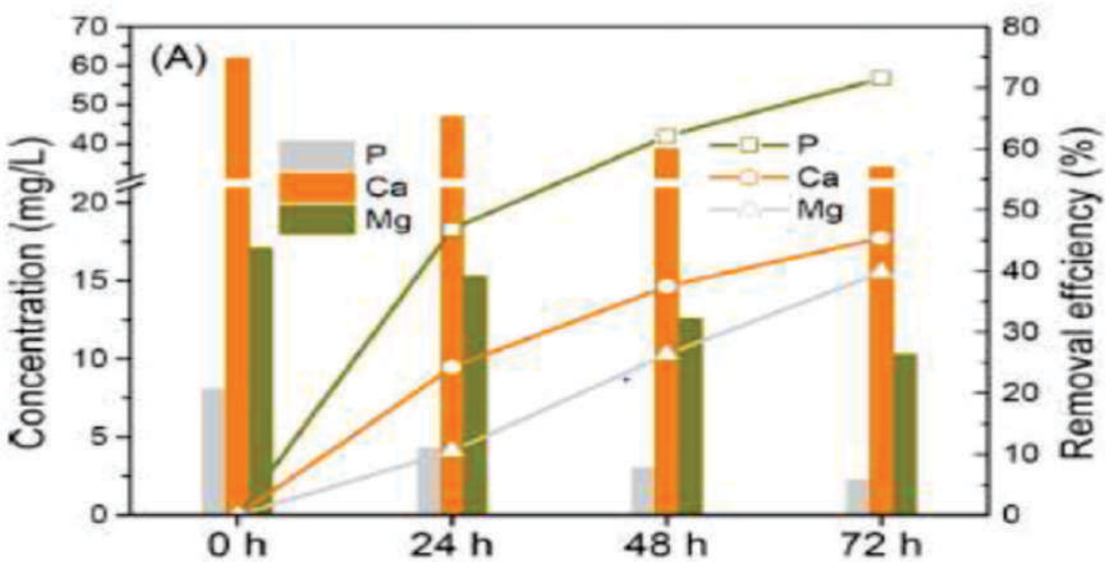

Figure 5: Electrochemical precipitation of P (phosphorus), Ca (calcium) and Mg (magnesium) in real wastewater (Lei *et al.*, 2017).

The application of extended aeration system aided with chemical precipitants for the treatment of wastewater has been successfully experimented and reported. The use of extended aeration system without the aid of chemical precipitants has been reported to achieve removal efficiencies of 23, 86, 82, 78, 72 and 71% for total phosphorus,

BOD_5, COD, suspended solids, turbidity and NH_3-N respectively (Martel *et al.*, 1977). It was also reported that the use of extended aeration system with the aid of sodium aluminate addition for wastewater treatment has been able to obtain removal efficiencies of 92, 94, 91, 86 and 97% for total phosphorus, BOD_5, suspended solids, turbidity and NH_3-N respectively. However, the use of extended aeration system with the aid of alum addition for wastewater treatment has been observed to achieve removal efficiencies of 92, 96, 92, 88, 90 and 93% for total phosphorus, BOD_5, COD, suspended solids, turbidity and NH_3-N respectively (Martel *et al.*, 1977). Experiment on the use of extended aeration system without chemical precipitant resulted to sludge bulking (sludge volume index of 62) and an effluent pH drop below 5.0, while the use of extended aeration system with sodium aluminate aid resulted to a system pH stabilized from 5.8 to 6.8 with a sludge volume index of 52. It was however reported that the use of alum to aid extended aeration system for wastewater treatment produce a sludge volume index of 54 and generated a disadvantage of pH value below 4.5 which is less than the desired pH range of 5.5 to 6.5 required for phosphate precipitation. Therefore, in order to control the pH to obtain the desired range for phosphate precipitation, the utilization of calcium hydroxide (lime) is required thereby leading to cost implication in the application of extended aeration system with the utilization of alum aid for wastewater treatment (Martel *et al.*, 1977).

A comparative study proposed by Aghapour *et al.*, (2016), on the efficiency of alum and ferric chloride in removing nitrate from wastewater revealed that at initial nitrate concentration in the range of 10-100 ml/L, and at varied dosage of alum and ferric chloride, it was posited that ferric chloride displayed a higher nitrate removal efficiency when compared to alum, also ferric chloride exhibited more than 90 % nitrate removal efficiency in a nitrate concentration range of 10-30 mg/L. Similarly, Adekoye *et al.*, (2014) reported a comparative outcome on using alum and ferrous sulphate at respective optimum pH of 7.90 and 8.20 for removing phosphate from wastewater. It was postulated that alum exhibited 30 % phosphate removal and was more effective than ferrous sulphate that exhibited 25 % in removing phosphate from wastewater.

The use of bauxite has been utilized in removing phosphate from natural stream water and effluent from wastewater treatment plant in Blantyre, Malawi (Altundogan and Tumen, 2001; Kamiyango *et al.*, 2011). It was posited that an increase in pH will lead to a decrease in phosphate removal as shown in Figure 6. However, a maximum phosphate removal of 99.75 % was reported but the presence of carbonate and sulphate ions were suggested to inhibit the removal of phosphate by competing for reactive sites.

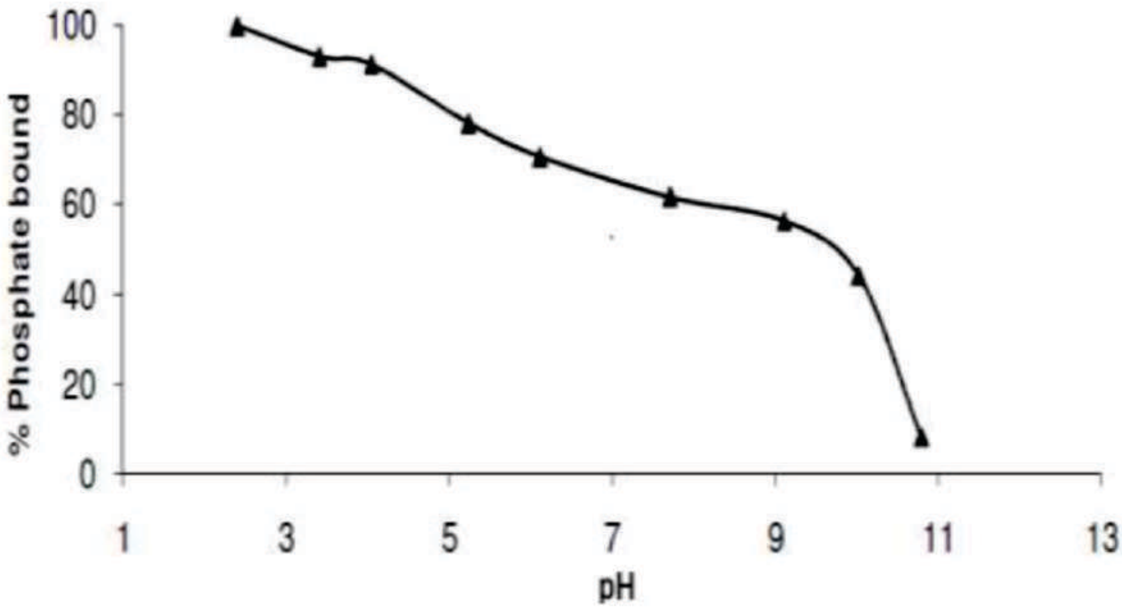

Figure 6: percentage phosphate bound (removal) against effect of pH (Kamiyango *et al.*, 2011)

The utilization of calcite at empty bed contact time (EBCT) ranging from 2.0 to 25.0 min has been reportedly effective in removing phosphate from wastewater. At optimum pH, the phosphate removal of above 96 % was observed as shown in Figure 7, however at pH 4.5, the percentage phosphate removal decreases with increase in sulphate ion concentration as shown in Figure 8 (Sperlich *et al.*, 2007; Liu *et al.*, 2012).

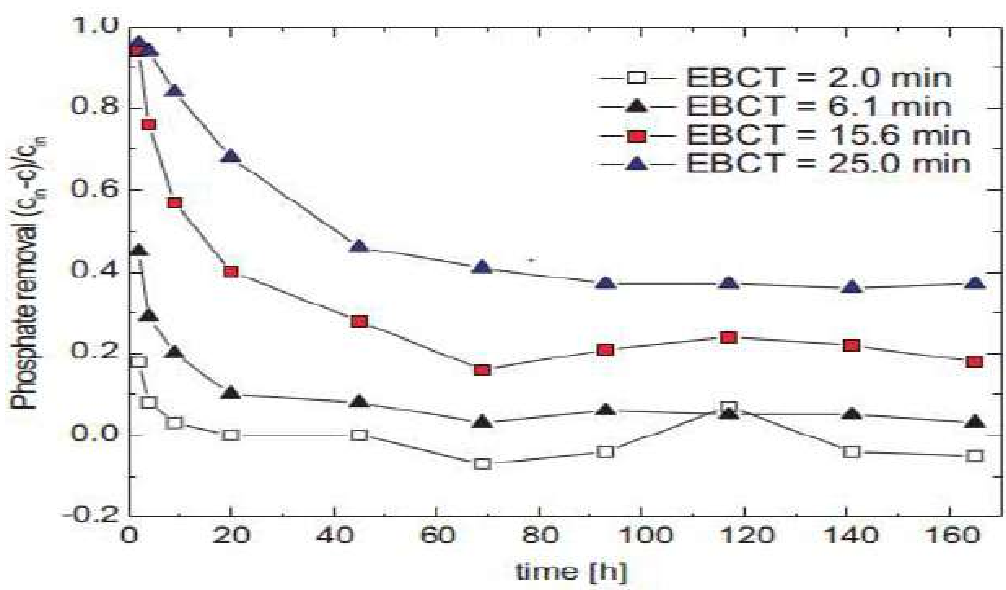

Figure 7: Percentage phosphate removal against time (Sperlich *et al.*, 2007)

Oladoja *et al.*, (2014, 2015) proposed the use of untreated and thermally treated gastropod shell in the precipitation removal of phosphate and turbidity from aqua system. It was stated that at initial phosphate concentrations (50-500 mg/L) and hydraulic resident time (6, 12 and 24 h), the untreated gastropod shell displayed 99.98 % removal of phosphate from wastewater. While, 99 and 60% of phosphate was respectively recovered from simulated and real aquaculture wastewater within the first two minutes by the thermally treated gastropod shell (Oladoja *et al.*, 2015). Also, 82.77 % turbidity was reportedly removed from wastewater for duration of 30 days, while nitrate-nitrogen and COD decrease with time as shown in Figure 9 (Oladoja *et al.*, 2017).

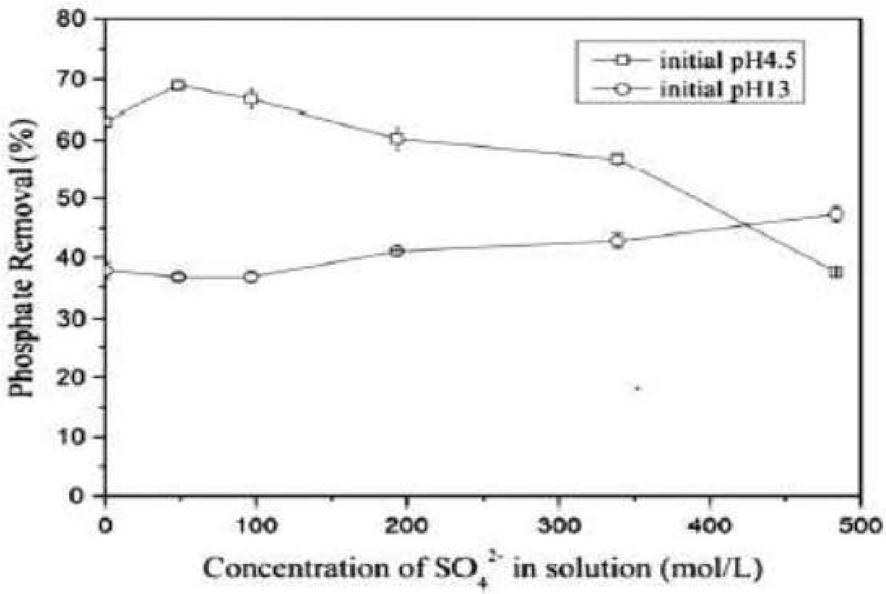

Figure 8: Effect of sulphate ion on percentage phosphate removal (Liu *et al.*, 2012)

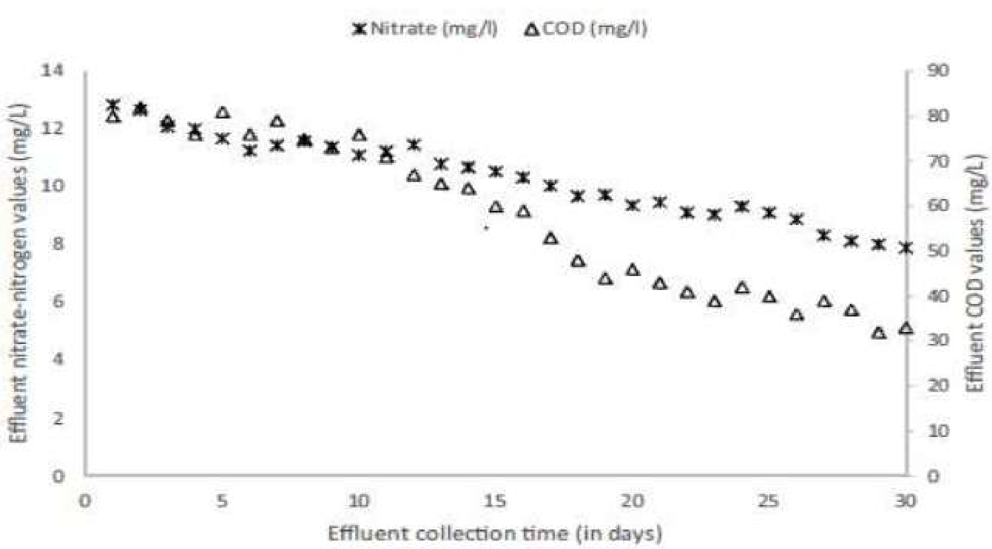

Figure 9: Effect of time on the removal of nitrate-nitrogen and COD (Oladoja *et al.*, 2017)

However, due to the increase in the salt content of the metal salts treated water as well as the cost implication of iron salt and calcium oxide precipitating agents in addition to the health effect of aluminum, plant-based extracts have been used in precipitation treatment of wastewater. For instance, organic extracts from plants have been recently used to remove 60 and 80 % of COD and TSS as well as 65-80% of organic matter from wastewater. However there has been paucity of information on the use of plant extracts in removing nutrients from wastewater (Czerwionka *et al.*, 2020).

Chemical Reduction Method

The application of hydroxylamine hydrochloride in the removal of nitrate from wastewater by chemical reduction of nitrate to nitrogen has been successfully reported to produce a desirable result. Experimental study on the application of hydroxylamine hydrochloride revealed that at an optimum dose of 0.08 g, the percentage reduction of nitrate increases with an increase in initial concentration and contact time but decreases with a decrease in solution pH and an increase in temperature (Islam, 2008). The utilization of five dosages (0, 0.075, 0.25, 0.50, and 1.0 g/L) of poly-(D) glucosamine (chitosan) adsorbed to ferruginous smectite (SWa-1) to form chitosan-SWa-1 composites (CSC) in the pH range 4-5.8 under redox-active conditions in order to reduce nitrate to lower oxidation states has been successfully experimented. It was reported that using chitosan-SWa-1 composites can remove 95 % of nitrate from an aqueous solution by reduction of nitrate to a lower oxidation state (Pentrak *et al.*, 2014). Electrochemically induced reduction of nitrate has been reported to be effective in wastewater treatment (Mook *et al.*, 2012). It was posited that the utilization of monometallic iron can increase the removal rate of nitrate from 7.6 to 25.0 %, while monometallic copper yielded a similar result when the current intensity was increased from 60 to 120 mA, however when the current intensity exceeded 120 mA, there was a decrease in the removal rate of nitrate as a result of excessive gas formation at the electrodes as shown in Figure 10. On the other hand, the utilization of nickel and carbon monometallic electrodes respectively resulted in 6.57 and 5.29 % removal of nitrate from simulated aquaculture wastewater thereby indicating their unsuitability as electrode in the removal of nitrate from aquaculture effluents as depicted in Figure 10 (Rajic *et al.*, 2017). Also, it was observed that the utilization of palladium as catalyst in electrochemically induced reduction by monometallic iron foam cathode further increased the removal rate of nitrate from 25 to 39.8% (Rajic *et al.*, 2017).

Further findings on using bimetallic electrodes along with catalyst revealed a better-quality performance for the removal of nitrate from wastewater when compared to monometallic electrodes. The employment of copper/palladium electrode displayed an increase in 25 % removal of nitrate from wastewater however the over-all percentage performance of copper/palladium electrode in nitrate removal is reportedly dependent on the composition ratio of the metals in the bimetallic electrode (Rajic *et al.*, 2017).

On the other hand, the use of bimetallic electrode of iron such as iron/palladium and iron/silver electrodes have been studied, it was reported that when compared to monometallic iron electrode that resulted in 25 % nitrate removal, the use of iron/palladium electrode yielded an increase from 25 to 39.8 %, while iron/silver electrode yielded 11 % removal of nitrate from wastewater as depicted in Figure 11 (Rajic *et al.*, 2017). Finally, the influence of flow modes was investigated in the removal of nitrate from wastewater, it was argued that a 36 % decrease in nitrate removal was noted when the flow rate was increase from 3 to 10 mL min^{-1}, however, there was an increase in the percentage nitrate removal from 39.8 to 53.2 % when the flow rate operated at 3 mL min^{-1} was changed from the flow-through mode to the circulation mode as depicted in Figure 12 (Rajic *et al.*, 2017).

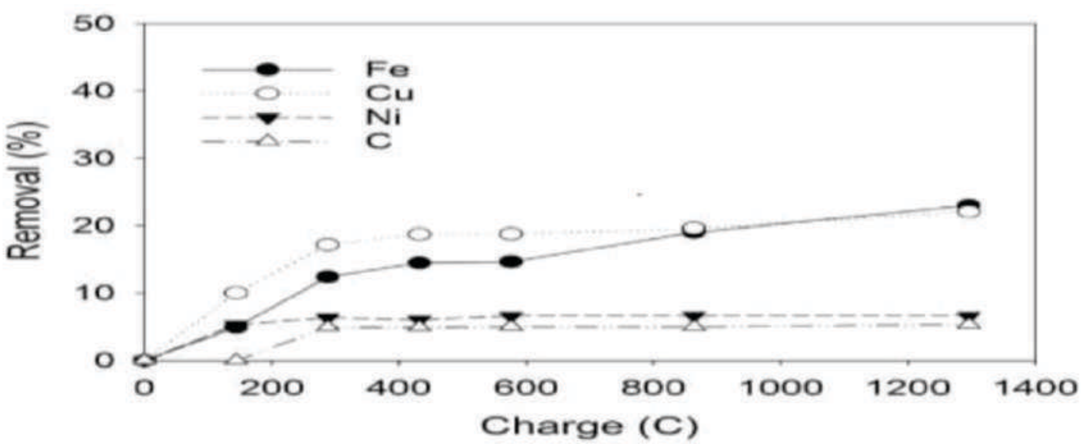

Figure 10: Percentage nitrate removal by means of different cathode equipment (current intensity (120 mA), flow rate (3 mL min^{-1}), 22.5 mg L^{-1} [NO^{3-}–N]) (Rajic *et al.*, 2017).

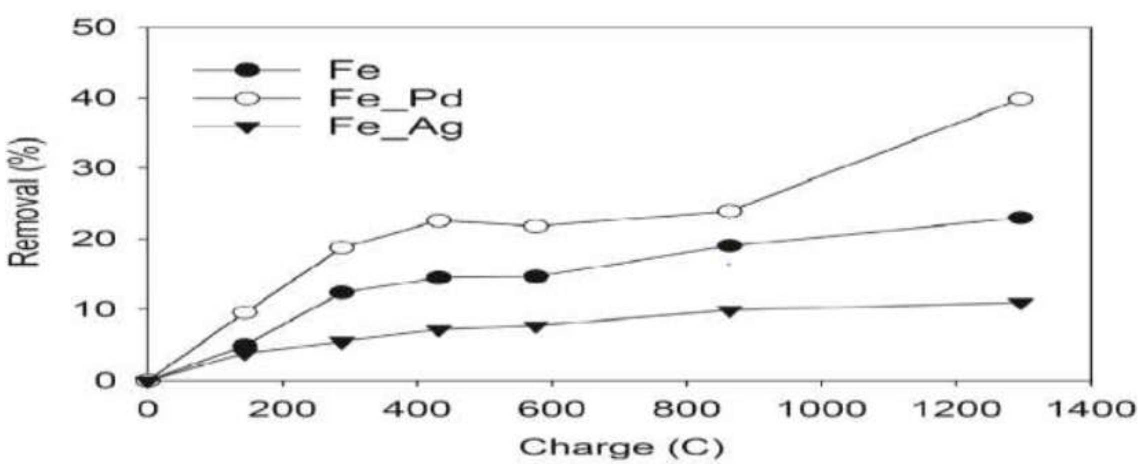

Figure 11: Percentage nitrate removal in the presence of different catalysts with support equipment (current intensity (120 mA), flow-through mode (3mL min^{-1}), 22.5 mg L^{-1} [NO^{3-}–N]) (Rajic *et al.*, 2017).

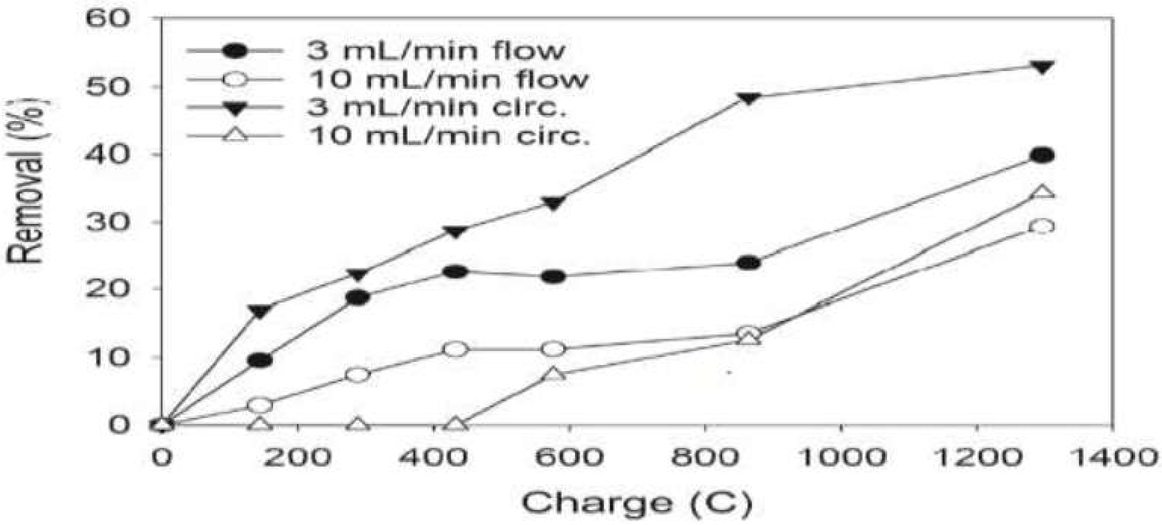

Figure 12: Percentage nitrate removal under flow modes and values (Fe/Pd bimetallic cathode, current intensity (120 mA), 22.5 mg L^{-1}[NO^{3-}–N]) (Rajic*et al.*, 2017).

Conclusion

The application of chemical precipitation and reduction methods have been proven to be effective in abating environmental problems such as eutrophication, which is associated with the indiscriminate release of nitrate and phosphate present in aquaculture wastewater. It has been postulated that at a high pH, chemical precipitating agents which include calcium oxide, aluminium sulphate and ferric sulphate have high percentage removal efficiencies for phosphate. On the other hand, chemical reducing agents which include chitosan-SWa-1 composites have 95% removal efficiency for nitrate from an aquaculture wastewater by reduction of nitrate to a lower oxidation state. There has been recent progress in the use of plant-based extracts in the precipitation treatment of wastewater such as in the removal of turbidiy, organic matter, COD and TSS but there is paucity of information on the use of plant-based extracts in the precipitation removal of nutrient from wastewater.

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