

## Article

# Treatment of Sewage Sludge and Phosphorus Removal Using Polyacrylamide and Calcium Chloride

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**Abstract:** The enhancement of the treatment of municipal wastewater treatment plants is limited by poor sludge settling qualities, and the excessive discharge of nitrogen and phosphorus exacerbate water eutrophication. The goal of the current work was to remove phosphorus from fresh sewage-activated sludge by developing a new conditioning and flocculation mechanism that included a coagulant and cationic polyelectrolytes in a dual conditioning system. The coagulant ( $\text{CaCl}_2$ ) and the high molecular weight polyacrylamide (CPAM-10) were chosen to be utilized singly or in pairs as cationic-coagulant combinations. The collected results showed that, in comparison to utilizing the coagulant ( $\text{CaCl}_2$ ), conditioning with the high molecular weight polymer (CPAM-10) produced improved settling and less turbidity. Only sludge with a lower solid content (TSS) exhibited better settling when pure  $\text{CaCl}_2$  was used for conditioning.  $\text{CaCl}_2$  conditioning enhanced settling by just 3%, while CPAM-10 improved the sludge setting by 60% for higher sludge TSSs. According to the results, conditioning settings using a dual mixture including 20 mL CPAM-10 and 50 mL  $\text{CaCl}_2$  improved settling by 80%. The amount of phosphorus in the supernatant was decreased by 15% and 9%, respectively, by using the coagulant ( $\text{CaCl}_2$ ) and 50 mL/L polyacrylamide (CPAM-10). As a result, there was a significant amount of phosphorus in the resultant supernatant. This suggested that the polymer had a significant impact on sludge settling because of its high positive charge, but had less of an impact on attracting phosphorus metal. Despite the lower positive charge of  $\text{CaCl}_2$ , it has a dual action of settling and removing phosphorus. A considerable amount of phosphorus was removed from the sludge and leached to the supernatant during treatment. This treatment was coupled with less sludge settling. However, 90% phosphorus removal was achieved when mixed conditioning agents (20 mL CPAM-10 and 50 mL  $\text{CaCl}_2$ ) were used. Furthermore, phosphorus was reduced by 33 and 39%, respectively, by adding 20 milliliters of  $\text{CaCl}_2$  to 100 milliliters of the pre-conditioned supernatant with pure CPAM-10 and  $\text{CaCl}_2$ . Using the CPAM-10 agent for sludge conditioning has a major impact on settling, because of the high positive charge, and because when a small amount of  $\text{Ca}^{++}$  is added to the polymer solution for conditioning to attract fine sludge particles and accelerate their combination, this results in flocculation and rapid dewatering. This mechanism allows for more phosphorus to be released to the supernatant, which has not been reported previously to the best of our knowledge.



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**Keywords:** sludge; polyacrylamide; calcium chloride

## 1. Introduction

The most efficient and cost-effective method for managing activated sludge and massive volumes of wastewater nowadays is biological treatment. Municipal wastewater treatment methods are tailored to remove organic compounds, phosphate, and nitrogen from sewage sludge [1]. Unfortunately, due to a lack of space, expanding any existing wastewater treatment plant is expensive and/or challenging [2].

One important factor for improving wastewater treatment plants' capacity is thought to be the floc settling of sewage-activated sludge. Additionally, sludge bulking may result in a decrease in the wastewater treatment efficiency [3]. The ability of bacteria to cause floc formation, which facilitates the separation of solid particles from treated wastewater, determines the efficacy of biological wastewater treatment. Additionally, a long period is needed for sludge separation and precipitation for a low sludge density [4]. The most expensive and challenging procedures in wastewater treatment facilities are those related to the management, disposal, and conditioning of activated sludge from treated sewage. Due to limitations in existing treatment technology, sewage sludge must be treated and disposed of using more effective and optimal procedures. More than 80% of the activated sludge that is produced after various separation techniques is water rich [5].

Extracellular polymers (EPS) adsorbed organic and inorganic materials, and both living and dead microorganisms form the composition of the floc. Since EPS, which are the main component of the microbial species, contain an anionic charge, cationic polymers and divalent metal ions are utilized to balance out the charges produced by the microbes. For at least forty years, polyelectrolytes have been used in coagulation/flocculation procedures to purify water. The use of polyelectrolytes can be beneficial to solve the problems of the flocs slow-settling, and for treating the soft colored waters to improve the settling ability and increase the flocs' toughness [6–9].

The impact of the digestion process on the dewatered sludge of many wastewater treatment plants in Japan was examined by Chen et al. [10]. The results of the study indicated that the digesting process had the greatest influence and that the conditioning process needed to be optimized in order to increase the effectiveness of sludge dewatering by choosing the right kinds of chemical agents and dosages. The choice of inappropriate chemicals for conditioning results in a decreased dewatering efficiency, which raises the operational cost significantly [11], as reported by Ghernaout and Ghernaout [12]. The agglomeration of the sludge particles is created during the chemical conditioning process as a result of bridging between the sludge particles and the neutralization of the negative surface charge. Consequently, this raises the efficiency of sludge dewatering [13]. Therefore, it is more effective to use high charge cationic polyelectrolyte density for floc formation.

Early studies [14–18] discovered that raising the concentration of divalent cations, specifically  $\text{Ca}^{++}$  and  $\text{Mg}^{++}$ , enhanced the settling characteristics, sludge dewatering, and floc strength. Nonetheless, it was shown that the primary cause of floc formation was calcium. The floc characteristics deteriorated when  $\text{Ca}^{++}$  and  $\text{Mg}^{++}$  ions were removed from the floc structure, as demonstrated by Bruus et al. [19].

In order to sustain the floc matrix of the microbe-biopolymer, Higgins and Novak [20] proposed a bridging theory using divalent cations (such as  $\text{Ca}^{++}$  and  $\text{Mg}^{++}$ ) and negatively charged sites on exocellular biopolymers. Furthermore, it was shown by Langer et al. [21] that inorganic substances (such as  $\text{Ca}^{+2}$  and  $\text{Fe}^{+3}$ ) can be used to treat wastewater by adsorbing on dispersed sludge particles, creating hydroxide polymeric charged particles,

and then altering the surface charge of the particles. Further research revealed that because of the electrostatic interactions between the cation and the sludge particles, the cation valence state had a significant role in influencing the features of sludge strength, flocculation, settling, and dewatering.

Due to their higher positive charges, divalent and trivalent cations facilitate better dewatering and stronger flocculation between biological cells and the extracellular polysaccharide (EPS) matrix [22–24]. In a study conducted in Nizwa, Sultanate of Oman, Salam [25] examined the effects of the coagulants  $\text{Al}^{+3}$ ,  $\text{Ca}^{+2}$ , and  $\text{Fe}^{+3}$  on turbidity, SVI, and floc size. His findings revealed that these cations had less of an influence on settling and dewatering than those obtained using polyelectrolytes.  $\text{Ca}^{++}$  may have a beneficial influence on cell–EPS bridging in wastewater with a low solid content.

According to research by Erik Rumbaugh [26], employing a 2:1 ratio of monovalent to divalent cations (M/D) has a detrimental effect on floc formation. He found that the flocculation process may be improved by adding the cations trivalent iron and aluminum with the following correlation: monovalent cations/divalent cations/trivalent cations (M/D)/T  $\leq 1.0$ . Additionally, he noted that the floc formation was marginally enhanced by the addition of  $\text{Mg}^{++}$ .

Raghavendran Sivasubramanian [27] indicated that adding divalent cations enhanced settling and organic removal, whereas increasing the concentration of monovalent cations worsened wastewater treatment and sludge settling. Additionally, he studied the flocculation process's effects on the M/D and divalent concentration independently.

Nitrate in the water is used by fish and algae to make protein, but too much of it can be harmful to domestic animals and young children. The transformation of sunlight into energy that cells can use for growth and reproduction requires the nutrient phosphate. On the other hand, excessive phosphorus levels may be harmful to nearby fauna, upsetting the delicate balance of the ecosystem and reducing the oxygen concentration of the water as the algae break down. Wastewater contains phosphate and nitrogen from fertilizers, food, human waste, and some soaps and detergents [28].

Algal blooms can be avoided by de-phosphorating water to avoid eutrophication. Furthermore, too much phosphorus in water resources cause algae to grow too quickly, obstructing the water body's ability to receive sunlight, killing fish and aquatic plants, and disturbing the delicate balance of the aquatic system [29].

One of the treatments to minimize the eutrophication of water bodies through the employment of various procedures is the removal and recovery of nitrogen and phosphorus. The three main methods for removing phosphorus are chemical, biological, and physical. In a biological treatment facility, phosphate must be moved from the liquid phase to the sludge phase. Alternative techniques must be employed to remove any remaining phosphate because its removal efficacy usually drops below 30% [30].

Chemical precipitation is also frequently used for phosphate removal. The industry sees the precipitation of calcium phosphate from wastewater as a significantly more viable kind of phosphorus recovery than other forms, since it may be valorized in agriculture as a slightly soluble fertilizer. As a result, it is a crucial physiochemical process for the removal of phosphorus and is becoming increasingly significant for the recovery of phosphorus [3]. Agents used to precipitate dissolved phosphorus include metal salts, calcium ( $\text{Ca}^{+2}$ ), iron ( $\text{Fe}^{+2}$  or  $\text{Fe}^{+3}$ ), aluminum (either alum,  $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$ , or sodium aluminate,  $\text{Na}_2\text{Al}_2\text{O}_4$ ).  $\text{Fe}^{+2}$  and  $\text{Fe}^{+3}$  salts can be used as sulfate or chloride [3,31,32].

Phosphorus concentration must be kept in check because it frequently acts as a barrier to eutrophication in inland receiving waters. In order to ascertain the optimal pH, dosage, and performance standards for the coagulants calcium chloride and alum for the removal of phosphorus, a series of jar tests were employed in this work. It was discovered that the

optimal pH range for alum use was 5.76. The dosage of calcium chloride and the pH of the solution were critical for the removal of phosphorus [30,33].

To improve the efficiency of the phosphorus removal process in pervious concrete, flocculant foamed lightweight aggregate (FFLA) containing polymeric ferric sulfate (PFS) as a flocculant was proposed to substitute the conventional aggregate. The effects and operation of PFS were investigated with respect to the basic physical properties, the evaluation of the phosphorus removal efficiency, and adsorption kinetics. The results showed that a decrease in the bulk density, cylinder compressive strength, softening coefficient, and water absorption of FFLAs gradually increased with an increase in PFS content. This was because PFS promoted the foaming of hydrogen peroxide, and the connection between loose bulk density and the softening coefficient and compressive strength of cylinders was greater than 0.93. Total phosphorus was removed at the highest rate of 87.71% by FFLAs [34].

This work was aimed at studying the utilization of  $\text{CaCl}_2$  for improving phosphorus removal concurrently with the use of positively charged polyacrylamide (CPAM-10) for a better settling process, and hence, lowered energy and operational costs and improved the sustainability of environmental waste.

The purpose of this work was to investigate the viability of using polyacrylamide (CPAM-10) and calcium chloride ( $\text{CaCl}_2$ ) to improve the settling and flocculation processes and to remove phosphorus from sewage effluent. The procedure was carried out using various doses of CPAM-10 and pure and mixed  $\text{Ca}^{++}$ .

## 2. Methodology

### 2.1. Collection and Characterization of Sludge Samples

Different sludge samples were taken from the aeration tank of the Izki (Sultanate of Oman) wastewater treatment facility. The total suspended solids (TSS), volatile suspended solids (VSS), and fixed suspended solids (FSS) are displayed in Table 1 for the sludge samples that were collected.

**Table 1.** Solid content in sludge samples.

| Sludge Sample | TSS (g/L) | FSS (g/L) | VSS (g/L) |
|---------------|-----------|-----------|-----------|
| 1             | 19.000    | 0.560     | 18.44     |
| 2             | 24.800    | 1.074     | 23.726    |
| 3             | 33.200    | 0.954     | 32.246    |
| 4             | 36.000    | 1.260     | 34.740    |
| 5             | 39.400    | 1.000     | 38.400    |
| 6             | 106.600   | 2.140     | 104.460   |
| 7             | 4.082     | 0.976     | 3.106     |
| 8             | 4.598     | 1.296     | 3.302     |
| 9             | 3.862     | 1.200     | 2.662     |
| 10            | 4.164     | 1.413     | 2.751     |
| 11            | 4.086     | 1.112     | 2.974     |
| 12            | 3.379     | 1.000     | 2.379     |
| 13            | 3.046     | 0.989     | 2.057     |

Using method 2540D, the total suspended solids for the sludge samples were determined in compliance with the 1998 APHA standard [35]. These findings showed that more than 80% of the samples' compositions were organic, as indicated by the values of VSS.

## 2.2. Conditioning Solutions

$\text{CaCl}_2$  and polyacrylamide (CPAM-10) were the two distinct cationic agents that were used to condition each sample, both singly and in combination. The conditioning solutions were prepared by mixing 1 g of each with one liter of deionized water and then stirring using a magnetic stirrer for at least 24 h. The jar test was conducted using six distinct conditioning solution concentrations, ranging from 5 to 50 ppm.

## 2.3. Flocculation Tests

For the flocculation testing, the jar test Armfield unit was utilized. Amount of conditioning solution were added; one milliliter is equivalent to one milligram of cation. The flocculation test was conducted in the jar test equipment using two distinct sequential mixing rates: first, a fast 200 rpm mixing rate for two minutes to facilitate the binding of the cation molecules with the dispersed sludge particles, and then a slower 30 rpm mixing rate for thirty minutes to encourage flocculation.

After three hours of settling, the supernatant was separated to measure phosphorus and turbidity. A turbidity meter (CL 52D Nephelometer, Elico Ltd., Hyderabad, India) and HANNA chemical reagents in conjunction with a colorimeter were used to quantify the turbidity and phosphorus, respectively. According to the 1998 APHA standard [35], the Sludge Volume Index (SVI), as established by method 2710D, was used to characterize the sludge settling properties of the samples.

## 2.4. Microscopic Analysis

Microscopic analysis using an optical microscope was performed using HORNET Micro Zoom 1280 (Wien, Austria) to photograph the fresh sludge samples; untreated and treated with polyacrylamide. The adopted magnification was 0.6–100 $\times$ . The photos were taken with the digital camera MICROS CAM 320 “Advanced”.

# 3. Results and Discussion

## 3.1. Microscopic Analysis of Fresh Sludge Sample

Figure 1 shows the non-dense morphology of the sample of sludge without conditioning. Figure 2 shows clearly how the polyelectrolyte carries out the function of separating solid particles of sludge and looks denser in comparison to a normal sludge. Also, these photos show the complexity of the sludge and that most of the sludge particles are heterogeneous in shape. Figure 3, using  $\text{CaCl}_2$ , shows not much difference to a fresh sludge sample.

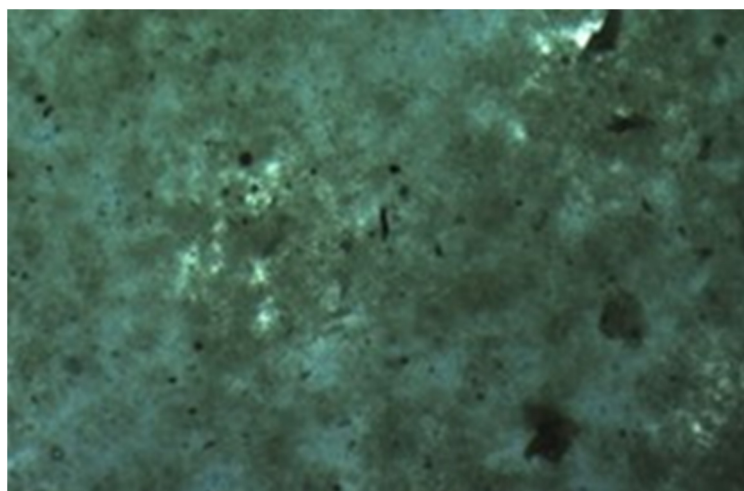


**Figure 1.** Shows a sample of sludge without conditioning.

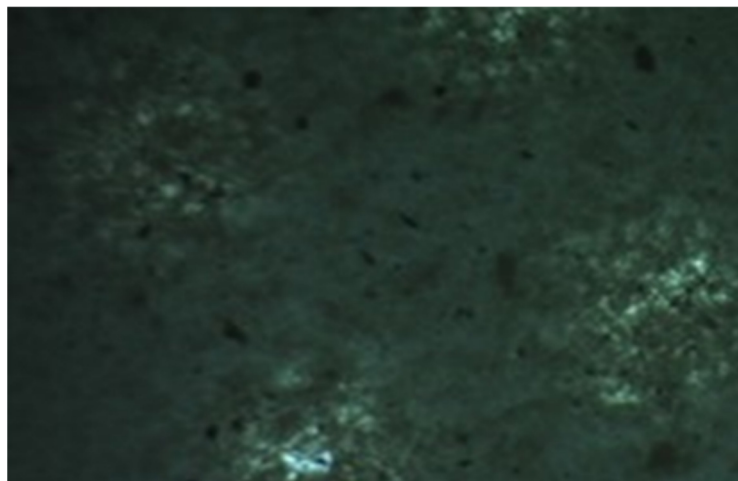


**Figure 2.** Sample treated with 100 mL CPAM-10 of a concentration of 1000 ppm.

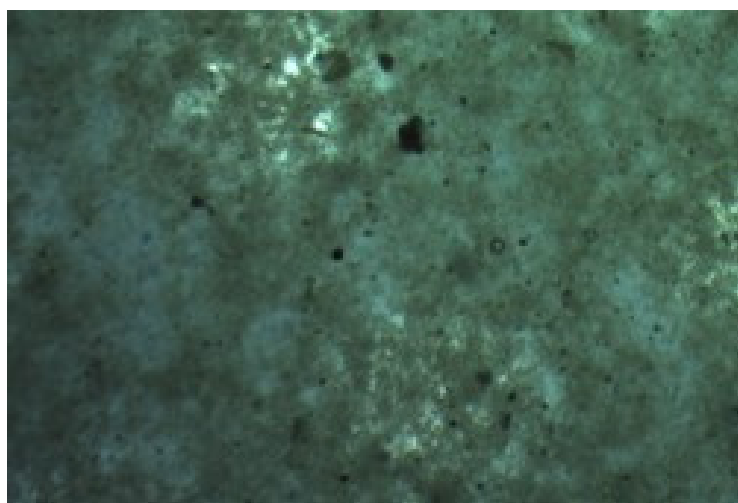
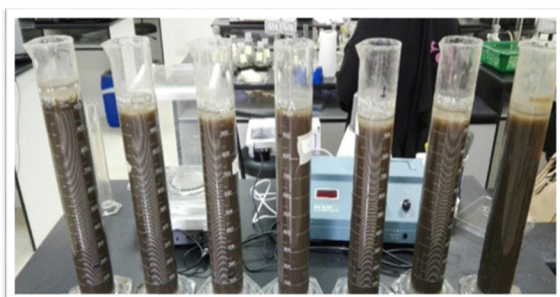
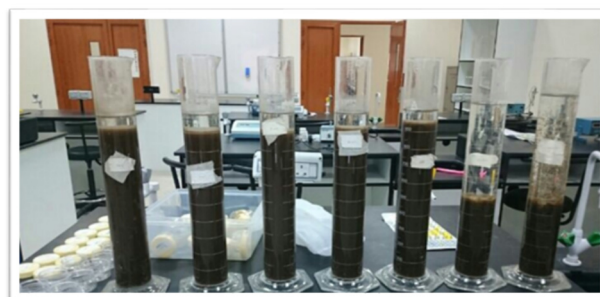


**Figure 3.** Sample treated with 100 mL  $\text{CaCl}_2$  of a concentration of 1000 ppm.

The effects of adding the two pure and mixed cationics,  $\text{CaCl}_2$  and polyacrylamide (CPAM-10), were investigated through several experimental tests. The settling after treatment with different concentrations (fresh, 5, 10, 20, 50, 100 mL) of  $\text{CaCl}_2$  and CPAM-10 each are shown in Figure 4.



**(a)**  $\text{CaCl}_2$



**(b)** CPAM

**Figure 4.** The settling after treatment with different concentrations.

### 3.2. Sludge Conditioning for Different (TSSs) Using 100 mL of Pure $\text{CaCl}_2$

Figure 5 indicates the effects of various total suspended solids (TSSs) on SVI for sludge conditioning using 100 mL of 1 ppm  $\text{CaCl}_2$ . It is evident that the amount of TSS present

had a significant impact on SVI, and the findings showed that increased TSS levels were associated with a sharp decline in the settling and flocculation processes.

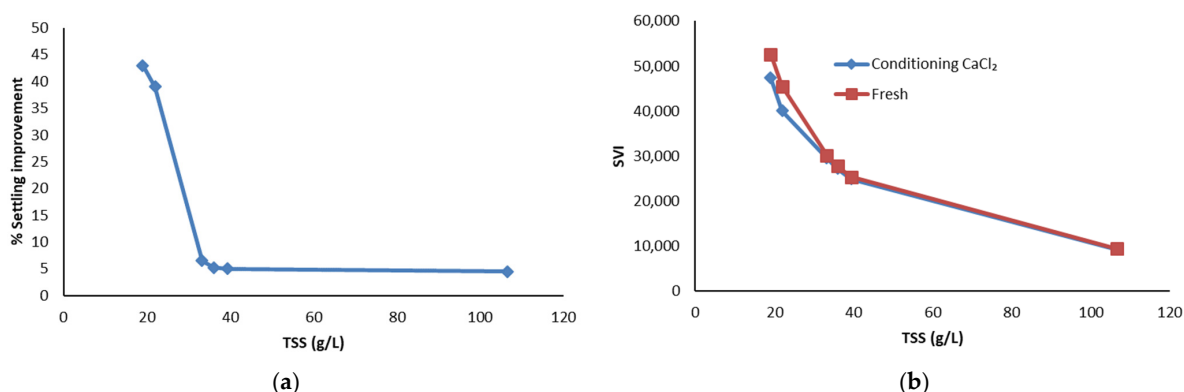


**Figure 5.** Sludge conditioning for different (TSSs) using 100 mL CaCl<sub>2</sub>; (a) percentage of settling improvement, (b) SVI.

When compared to fresh sludge samples, samples treated with less TSS, such as those with 19 g/L, showed a 45% improvement in settling efficiency and a 10% improvement in SVI due to the treatment using CaCl<sub>2</sub>. However, there was less than 5% improvement for samples with a greater TSS. The low improvement in settling referred to the weak interactions between the low cation charge and sludge particles, which can be referred to Van der Waals interactions, rather than the bridging mechanism. As a result, there was less dewatering and less effective flocculation promotion. These findings demonstrated how adding cations like Ca<sup>++</sup> could enhance the flocculation and settling for sludge with low solid contents. Following the jar test, the interface positions between the sediment and supernatant liquid were measured during settling. The findings are shown in Figure 6 as a function of time after the mixture was placed into cylindrical flasks with different Ca<sup>++</sup> concentrations. The outcomes demonstrated less settling, even when the Ca<sup>++</sup> dosage was increased to 100 ppm.

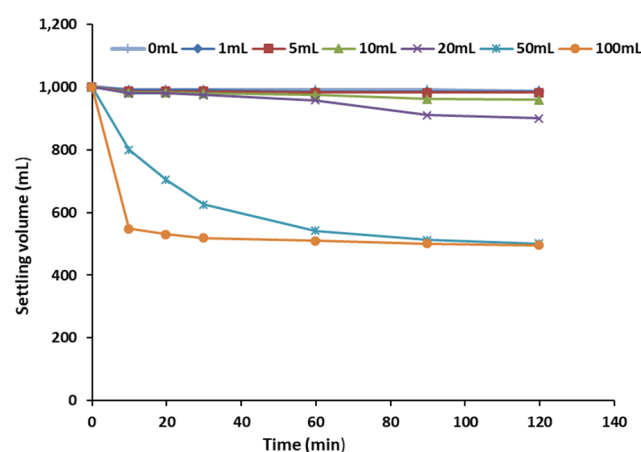


**Figure 6.** Sludge settling using pure CPAM-10.

### 3.3. Flocculation Process Using Pure CaCl<sub>2</sub> and CPAM-10

Several tests employed different cationics and polyacrylamide for improving sludge settling. The findings showed a 50% increase in sludge settling when using positive polyacrylamide with a high density. Various concentrations of polyacrylamide (CPAM-10) were employed, and the results are depicted in Figure 6. When compared to utilizing Ca<sup>++</sup> (Figure 7), the results indicated a considerable influence on settling with an increase

in polymer dosage. When cationic polymers were added, the polymers' amino groups combined with the carboxyl groups of the suspended sludge particles, causing flocculation and rapid dewatering.

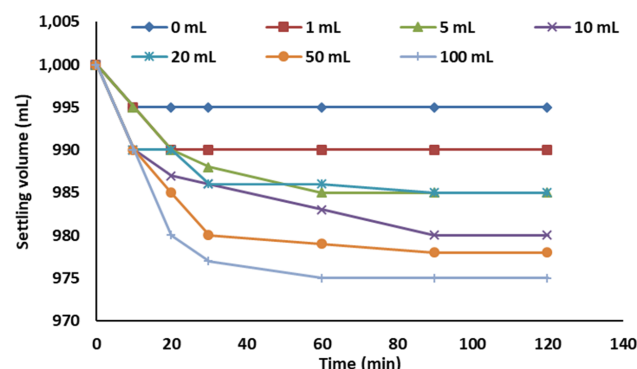


**Figure 7.** Sludge settling using pure  $\text{CaCl}_2$ .

### 3.4. Flocculation Process Using a Dual Mixture of $\text{CaCl}_2$ and CPAM-10

A dual mixture of CPAM-10 (10, 20, and 50 mL) at varying  $\text{CaCl}_2$  concentrations in the range of 5–100 mL was employed. Figure 8a, b, and c depict the result of the mixed polymer of dosages of 10, 20, and 50, respectively. The results obtained showed a significant improvement in settling for several combinations. The findings showed that adding more CPAM-10 to the dual agents for sludge conditioning had a major impact on settling, because the high positive charge drew in the negatively charged sludge surface, which caused flocculation. When a small amount of  $\text{Ca}^{++}$  (between 5 and 10 mL) is added to the polymer solution for conditioning, it attracts fine sludge particles and accelerates their combination, resulting in flocculation and rapid dewatering.

Nevertheless, despite the high concentration of positive charge, adding more  $\text{Ca}^{++}$  resulted in a lower improvement in settling. This was due to the repulsion between similar charges causing the dispersion of the sludge particles.

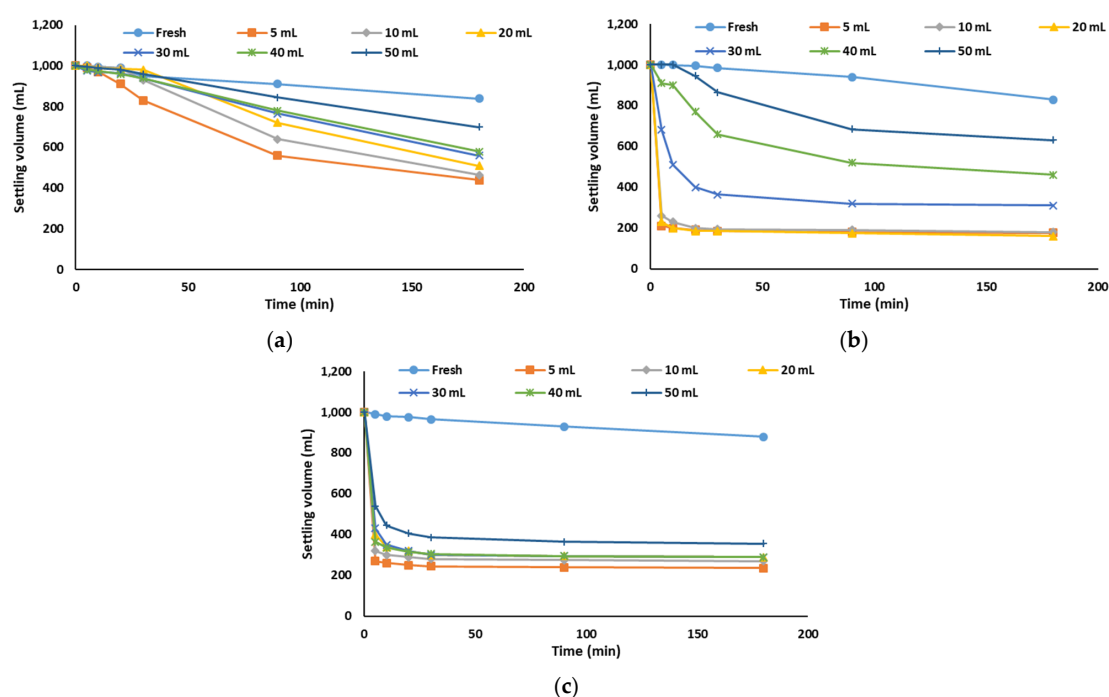


**Figure 8.** Settling using a dual mixture of  $\text{CaCl}_2$  and CPAM-10. (a)  $\text{CaCl}_2$  and 10 mL polymer, (b)  $\text{CaCl}_2$  and 20 mL polymer, (c)  $\text{CaCl}_2$  and 50 mL polymer.

### 3.5. Phosphorus Removal

To determine how well phosphorus could be removed from wastewater, several tests were conducted. The upcoming section contains these tests.

#### 3.5.1. Phosphorus Removal from Treated Sludge Using Pure $\text{CaCl}_2$ and CPAM-10

The phosphorus concentrations of supernatant samples obtained from the treated sludge employing CPAM-10 and  $\text{CaCl}_2$  are displayed in Figure 9. It can be observed that adding CPAM-10 and calcium chloride ( $\text{CaCl}_2$ ) to sewage sludge decreased its phosphorus concentration by 7% and 15%, respectively. The acquired results showed that the polymer had a significant effect on drawing the negatively charged sludge particles (causing dewatering); however, it had little attraction to the negatively charged phosphorus ions. But during sludge treatment,  $\text{CaCl}_2$  affected the settling of the fine sludge particles only, and largely the phosphorus ions. This is due to the coulombic attraction between the oppositely charged species, meaning that  $\text{Ca}^{++}$  tends to polarize the electron cloud of the phosphorus ions by pulling their electron density towards itself, while with the use of polyacrylamide, the weak Van der Waals force is dominant over the weak attraction between the polymer and phosphorus.

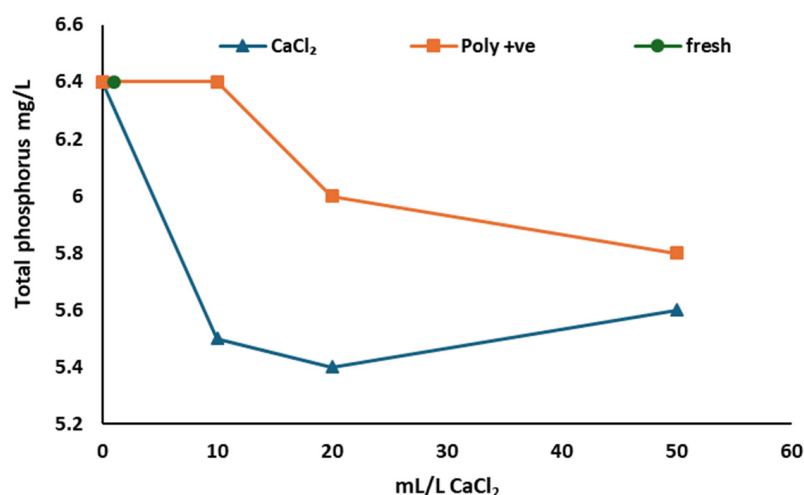


**Figure 9.** Phosphorus content in the supernatant after treatment with pure  $\text{CaCl}_2$  and the polymer.

#### 3.5.2. Phosphorus Removal from Conditioned Supernatant with Pure CPAM-10 and $\text{CaCl}_2$

Figure 10 displays the results of treating samples of the previously conditioned supernatant with 50 mL of pure CPAM-10 and  $\text{CaCl}_2$  each, followed by the addition of 20 mL of  $\text{Ca}^{++}$  to 100 mL of treated supernatant for the removal of phosphorus. The results show that phosphorus was reduced by 39% and 32% for the conditioned supernatants containing 50 mL of  $\text{CaCl}_2$  and 50 mL of CPAM-10, respectively. The availability of  $\text{Ca}^{++}$  in the previously treated supernatant was prompted by the addition of new  $\text{Ca}^{++}$  and caused a greater decrease in phosphorus content.

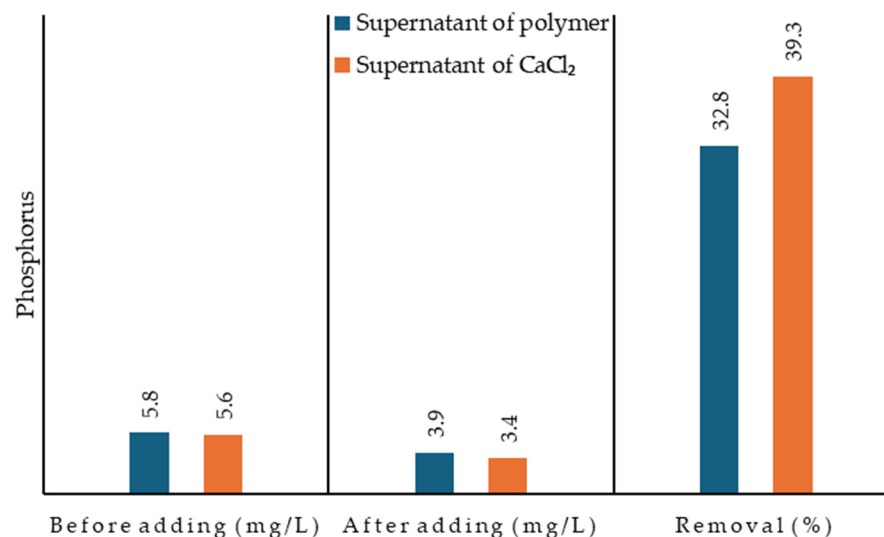


**Figure 10.** Phosphorus removal from conditioned supernatant with pure CaCl<sub>2</sub> and CPAM-10.

### 3.5.3. Phosphorus Removal from Supernatant from Treated Sludge Using Dual Mixture of CaCl<sub>2</sub> and CPAM-10

Supernatant samples were extracted from the treated sludge using the dual mixture of CPAM-10 and CaCl<sub>2</sub> concentrations. The supernatant samples were additionally treated for phosphorus removal using varying concentrations of CaCl<sub>2</sub>. The phosphorus content of each sample was evaluated independently, and the findings are displayed in Figure 11. The results showed that the removal of phosphorus ions increased with the amount of CaCl<sub>2</sub> employed; with 10, 20, and 50 mL of CPAM-10, the phosphorus removal was 78, 90, and 80% with 50 mL of CaCl<sub>2</sub>, respectively. Using CPAM-10 agents for sludge conditioning has a major impact on settling because of the high positive charge, and when a small amount of Ca<sup>++</sup> is added to the polymer solution for conditioning, it attracts fine sludge particles and accelerates their combination, resulting in flocculation and rapid dewatering. This mechanism allows for more phosphorus to be released to the supernatant, which has not been reported previously to the best of our knowledge. The high percentage of phosphorus removal was due to the coulombic attraction between the oppositely charged species.

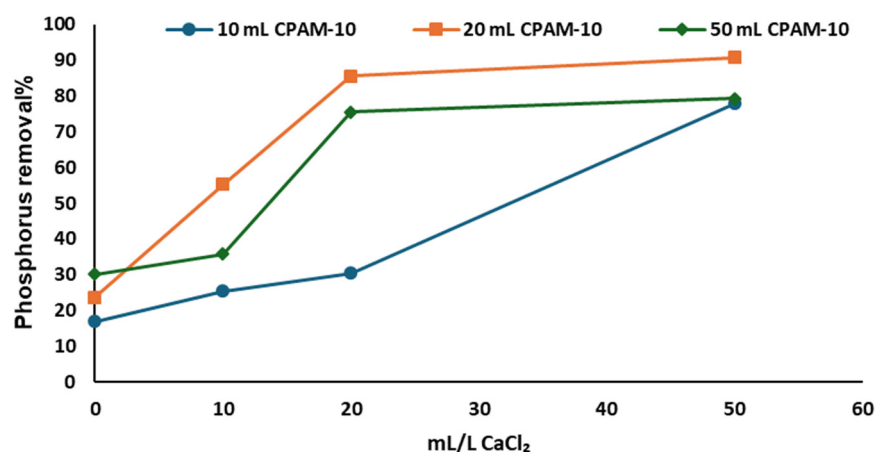


**Figure 11.** Phosphorus content in the supernatant after treatment using mixed CPAM-10 and different amounts of CaCl<sub>2</sub>.

## 4. Conclusions

Phosphates are derived from numerous sources such as agriculture, urban drainage systems, and home and industrial wastewater. In addition, detergents and domestic and commercial wastewater, including that from dry cleaners and laundry services, release large concentrations of phosphates into the land and aquatic environments. The treatment of sewage sludge and phosphorus removal using polyacrylamide and calcium chloride with conditioning with  $\text{CaCl}_2$  has less effect on the settling of the activated sludge with a large TSS content, due to its lower positive charge. CPAM-10 has a large impact on the settling of the activated sludge with all levels of TSS due to its large positive charge. The settling and flocculation processes can be enhanced by conditioning sludge with a dual mixture of CPAM-10 and  $\text{CaCl}_2$ . After conditioning with pure  $\text{CaCl}_2$  and pure CPAM-10, the amount of phosphorus was reduced by less than 20%. A dual mixture of CPAM-10 and  $\text{CaCl}_2$  could enhance phosphorus removal by up to 90% from the resultant supernatants. The optimum dosage to the treated sludge using 50 mL of pure CPAM-10 and  $\text{CaCl}_2$  was 20 mL of  $\text{Ca}^{++}$  to 100 mL of the supernatant solution. Adding more  $\text{CaCl}_2$  to the conditioned supernatant (made with pure CPAM-10 and  $\text{CaCl}_2$ ) can enhance phosphorus removal by 40% from the resultant supernatant.

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