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Iron and Hydrogen Peroxidation-Induced Post-Treatment Improvement of Municipal Mesophilic Digestate in an Alkaline Environment and Its Impact on Biosolids Quality

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Abstract: Challenges associated with mesophilic digestate (MD) involve volume, odor, and pathogens, which effective post-digestion treatments can address. The efficiency of MD post-treatment can be enhanced by conditioning with ferric chloride ($FeCl_3$), hydrogen peroxide (H_2O_2), and polymer. This study aimed to observe the effect of combined chemical conditioning on volume reduction, phosphorus (P) release, odor, and pathogen reduction potential for MD. MD was conditioned with polymer only, polymer and $FeCl_3$ at pH adjusted to 8.0 with lime ($Ca(OH)_2$), and a blend of polymer, $FeCl_3$, and hydrogen peroxide (H_2O_2) at pH 8.0. The results show that adding all three chemicals improved post-treatment efficiency at 2.1 kg/t DS $FeCl_3$, 2.1 kg/t DS polymer, and 600 mg/L H_2O_2 at pH 8.0, compared with polymer or dual conditioning. At the combined dose, cake solid content, centrate P removal, and odor reduction capability improved compared with raw MD by 20%, 99%, and 66%, respectively. Combined chemical treatment reduced fecal coliform by 98% but does not fulfil class A requirements and showed 50% regrowth potential. The synergic effect of polymer, $FeCl_3$, H_2O_2, and alkaline pH breakdown EPS, reduced water holding capacity and formed compacted flocs for better water removal and settling. This combination also precipitated P through $FeCl_3$ while H_2O_2 oxidation curbs odor, enhancing further P removal from centrate.

Keywords: mesophilic digestate; ferric chloride; hydrogen peroxide; biosolids post-treatment; biosolids quality analysis; centrate phosphorous removal

1. Introduction

As wastewater treatment practices become more sustainable and resource-efficient, the post-treatment of digested sludge is drawing more research interest due to regulatory pressures and environmental concerns, along with expanding reuse and resource recovery options [1]. Challenges related to the reuse and disposal of digested sludge include high water and nutrient content, unpleasant odor, heavy metals, etc. High volume causes logistic and transportation problems; pathogen concentrations might contaminate land and pose health risks to the public if disposed of or reused. High P content might cause eutrophication in aquatic environments.

Digested sludges often undergo post-treatment, where polymers are added to increase solid–liquid separation and reduce the volume. Traditional polymer conditioning incurs extra costs, as polymers are relatively expensive [2]. The high viscosity of polymers contributes to clogging and fouling issues in centrifuges and belt filter presses, thus reducing operational efficiency and increasing maintenance costs [3]. Residual polymers remain in biosolids, potentially impact soil, and can cause aquatic toxicity if treated biosolids are applied to land [4].

MD, the residual material left after mesophilic anaerobic digestion (MAD) between 35 and 40 °C, contains 98% water [5]. Wastewater treatment plants mostly operate their digestors in mesophilic temperature mode because of the simplicity of operation, low operations and energy cost, less sensitivity to feedstock variation, and lower greenhouse gas emissions. MD has a lower reduction of volatile solids, lower pathogen reduction capability, and a lower degree of odor reduction than thermophilic anaerobic digestion [6]. Due to volatile organic sulfur compounds, MD can generate an unpleasant odor [7]. MD also has total solids (TS) of 1.8% and high viscosity, making it challenging to dewater [8]. High water content increases its volume, making dewatering, handling, and transportation difficult. MD has an elevated level of nutrient content, including Nitrogen (N) and P, compared with thermophilic or other types of digested sludge. These nutrients are essential for plant growth and soil conditioning, but if N and P remain in biosolids and the liquid portion of dewatered digestate, they can cause algal blooms and kill fish, and eutrophication can lead to degraded water quality [9].

Polymers are susceptible to microbial consumption, which lowers their efficiency and generates undesirable odor [10]. Furthermore, microbial activity may result in the development of EPS, improving the MD's ability to retain water and lessen dewatering efficiency [11]. As EPS, particularly PN, has a high water-holding capacity, conventional methods require more polymer to separate water from solids [12,13]. Therefore, researchers have attempted to improve digestate dewatering by modifying or destroying hydrophilic EPS properties or the physicochemical forces linking digestate particles and bound water [14]. EPS, bound water, particle charge, and floc size are crucial controlling variables that act as barriers to the dewatering of digestate [15].

$FeCl_3$ acts as a coagulant, producing iron hydroxides with low solubility that act as bridges between particles, forming compact and larger flocs that settle quickly [16]. Trivalent cation Fe^{3+} added to digestate releases free hydrogen, decreasing solution pH. The iron hydroxide produced is highly soluble at very low or high pH, but forms a precipitate at optimum pH [17]. The digestate's pH can be adjusted by adding lime $(Ca(OH)_2)$. $Ca(OH)_2$ raises the number of multivalent cations in the solution along with Fe^{3+}. Multivalent cations Fe^{3+} and Ca^{2+} cause negatively charged phosphate ions to bind with them and form an insoluble precipitate [18]. Increased multivalent cations promote high-density floc formation facilitating settling and dewatering [19]. Alkaline pH makes organic matter more soluble, lowering the surface tension of water [20]. The reduced surface tension of water helps it flow more quickly, making it easier to remove.

Fe^{3+} and Ca^{2+} also help P recover through chemical P precipitation, producing insoluble P compounds and significantly decreasing P concentration from the liquid portion of treated digestate [21]. Pretreatment with ferric oxide might improve the P removal rate by 80% to 90% [22]. Regulatory requirement set levels of P concentration in wastewater effluent at 1 to 2 mg/L [23]. This stringent requirement requires a more efficient conditioning process for P recovery, which has prompted research into alternative chemicals that might improve P recovery and contribute to enhanced digestate dewatering.

H_2O_2 produces highly reactive hydroxyl radicals ($^{\cdot}OH$), potent oxidizing substances in the presence of iron ions [24]. An advanced oxidation process combining H_2O_2, and microwave heating was used to solubilize phosphate from secondary sludge to enhance biological P removal. Over 99% P removal was achieved from wastewater using manganese oxide nanoparticles and iron oxide catalyst, $FeCl_3$ coagulant, and 0.5 M H_2O_2 at pH 5.0 to 7.0 [25]. Some studies have also shown the possibility of higher P removal from industrial wastewater through H_2O_2 oxidation at lower pH 3.5 [26]. However, the effect of the trivalent Fe^{3+}-based oxidation process remains unknown, and study of its mechanism to improve the dewaterability of digested sludge in an alkaline environment is yet to be completed. Additionally, the feasibility of reusing treated biosolids is also unknown.

Moreover, traditional polymer conditioning focuses only on increased solid–liquid separation and volume reduction [27]. Other potential challenges related to pathogens, odor, nutrient recovery, and metal concentration remain unaddressed. Recent research

emphasizes converting waste to energy and resource recovery from waste sludge. Post-treatment processes such as chemical conditioning, thermal drying, composting, alkaline stabilization, etc., are considered effective for digesting digestate. Among these processes, the iron-based oxidation process is a promising alternative to the traditional polymer flocculation process as it may cause deep digestate dewatering, reducing moisture content by up to 60% [28,29]. While there are some review publications on iron-based oxidation, experiments have not been conducted on iron-based advanced oxidation processes, such as Fenton-like processes which combine $FeCl_3$ and H_2O_2 to improve post-treatment efficiency. Further research is required to comprehend the mechanism and application of iron-based advanced oxidation processes for enhancing post-treatment efficiency.

The efficient post-treatment of digested sludge is important to convert it from waste to resource and increase the options for its careful reuse. Chemical treatment is considered an essential conditioning method. The current research aims to study the impact of the combined application of polymer flocculant, iron based coagulant $FeCl_3$, and oxidant H_2O_2 at alkaline pH to improve the post-treatment efficiency of digestate while analyzing its mechanism. The lab-scale experiment compared raw MD and polymer alone with the dual application of polymer and $FeCl_3$, and the combined application of polymer, $FeCl_3$, and H_2O_2, adjusting the pH of MD. This study also assessed several dewatering parameters to determine which combination was most effective and how their effect enhanced the efficiency of MD post-treatment, including volume reduction, P recovery, odor reduction, and pathogen destruction.

2. Materials and Method

2.1. Sampling of MD

MD was acquired from Ashbridges Bay wastewater treatment plant (WWTP) in Toronto, Canada. Twelve solid bowl centrifuges were used to dewater the MD, having 1.8% TS with polymer.

The percentage of total suspended solids (TSS) in the centrate ranged from 635 to 863 mg/L, and TS in dewatered cake was 24%. Table 1 reports the MD characteristics.

Table 1. Characteristics of MD.

Parameters	Values
Temperature (°C)	25 ± 1.9
Viscosity (cp)	46 ± 1.2
Turbidity (NTU)	1209 ± 2.9
CST (s)	234 ± 10
SRF (Tm/kg)	204.2 ± 5.8
Cake solid content (%)	24 ± 0
Centrate P (mg/L)	111 ± 6.6
Cake P (mg/L)	328 ± 2
Soluble chemical oxygen demand (sCOD) (mg/L)	1247 ± 106.7
NH_3-N (mg/L)	509.1 ± 0.9
PN/PS ratio	44 ± 4
Alkalinity (mg/L Ca CO_3)	2116.5 ± 12.5
Centrate total iron (TFe) (mg/L)	1.8 ± 0
Centrate total sulfide (TS^{2-}) (µg/L)	86.7 ± 1.7

2.2. Methodology

A bench-scale batch experiment was carried out at room temperature. Experiments were conducted by conditioning MD without and with pH adjustment. pH was adjusted using Ca $(OH)_2$.

MD was collected from Ashbridges Bay wastewater treatment plant (ABWWTP), Ontario, Canada. Evaluation of MD post-treatment efficiency was calculated measuring various indices for conditioned digestate, conditioned digestate supernatant, dewatered cake, and centrate. Cationic polymer was collected from ABWWTP. Ferric chloride ($FeCl_3$)

as coagulant and hydrogen peroxide (H_2O_2) as oxidant were used along with the polymer. Chemicals and reagents were purchased from VWR Scientific, Toronto, Canada.

Municipal MD was conditioned with only polymer, the dual application of polymer and $FeCl_3$, and the combined application of polymer, $FeCl_3$, and H_2O_2. MD was first conditioned with different polymer doses without pH adjustment to find the optimum polymer dose. MD pH was then adjusted to 8.0, and various $FeCl_3$ doses were applied at the optimum polymer dose. The best dose of $FeCl_3$ from the experiment was then used with various polymer doses at pH 8.0 to find the best combination of $FeCl_3$ and polymer dose. In the last step, MD was conditioned with the best polymer and $FeCl_3$ dose at 8.0 and several H_2O_2 doses to find the best combination of the three chemicals. A conditional sample was centrifuged at 5000 rpm for 10 min, and then the centrate was filtered through a 0.45 mm syringe filter with Supor® membrane, used afterward for analyses.

2.3. Experimental Set-Up

A jar test apparatus with five or six beakers containing 500 mL of MD was used to blend the polymer and chemicals and determine the optimal dosage. The chemicals were added to the MD in the necessary quantities, mixed for 2 min at 180 to 200 rpm, gently stirred for 2 min at 30 rpm, and left to stand for 10 min before further examination.

Polymers and chemicals were combined and optimized using a Phillips & Bird 7790-400 jar test device purchased from Express Lab Werks, Summerfield, FL, USA. Dewatering was carried out with a Heraeus Multifuge X1 bench-top centrifuge from Thermo Fisher Scientific, Toronto, Canada.

The digestate's dewaterability was assessed by measuring CST, SRF, turbidity, viscosity, centrate P concentration, centrate PN/PS ratio, centrate TS^{2-} concentration, etc. A Nalgene® Buchner funnel apparatus, which included a 9 cm Buchner funnel from Thermo Fisher Scientific, graduated cylinders with the adapter, Whatman filter paper 4, and a mini vacuum pump purchased from VWR scientific, Toronto, ON, Canada, were used to determine SRF. The Triton 304B CST time device and compatible filter paper were obtained from Geneq Corporation, Quebec, ON, Canada. A Heraeus Multifuge X1 bench-top centrifuge from Thermo Scientific was used for dewatering. A DRB 200 digital reactor block was employed to digest samples, while a DR 3900 spectrophotometer from HACH, Toronto, ON, Canada was used to read the samples.

2.4. Analytical Methods

The SRF test determines how easily water can pass through Whatman filter paper 4 when 100 mL of conditioned digestate is subjected to a 0.5 bar vacuum using the Buchner funnel apparatus [30]. The method of determining CST involved using a 1.8 cm diameter funnel to pour 10 mL of treated sludge onto a standard thick filter paper until the liquid reached a height of 1 cm [31]. The capillary suction of a standard thick filter paper removed the water from the conditioned MD sample [32].

Standard procedures for examining water and wastewater were used to analyze the TS of MD and dewatered cake [33]. A VWR Symphony bench-top pH meter and Micro 100 turbidity meter were used to measure pH and turbidity. A HACH DR 3900 spectrophotometer was also used, and a Fungilab viscometer was used to measure viscosity. Using HACH procedure 10127 HR Molybdovanadate with Acid Persulfate Digestion, total phosphorous (TP) was determined. TFe and TS^{2-} in the centrate were determined using HACH 8131 Methylene Blue and 8008 FerroVer® methods, respectively. CST and SRF measurements were made to ascertain the conditioned MD's water-release rate. Method 2710G was used to measure CST [33].

Using the Bradford Coomassie reagent, a colorimetric approach was used to measure centrate PN [34]. The colorimetric approach was used to quantify PS using the phenol sulfuric acid reaction. Test tubes were used to conduct the reaction between PN and PS, and the DR 3900 spectrophotometer from HACH, Toronto, Canada was used to measure absorbance at wavelengths of 595 nm for PN and 490 nm for PS [35].

Fecal coliform was found in the dewatered cake using method 8001A, a multiple-tube fermentation technique created by the USEPA and modified from EPA method 1680 for biosolids [36]. Fecal coliform was measured as MPN per g of DS. In addition, the American Society of Microbiology-approved MAC methodology was used to measure *Escherichia coli (E. coli)* in this study's MAC media, using the unit CFU of *E. coli*/g of DS [37].

3. Results and Discussion

Post-treatment of MD optimizes resource recovery and efficiency in biosolids management. Beyond CST, our study explored diverse dewatering indices such as SRF, cake P concentration, and centrate quality (P, PN/PS ratio, S^{2-}, Fe) to evaluate chemical post-treatment efficiency. Phosphorus content, fecal coliform, and *E. coli* counts in the dewatered cake were also analyzed to assess the biosolid's quality. TS^{2-} uniquely indicates odor reduction potential. Polymer $FeCl_3$ blends, pH optimization, and precise dosing of combined polymer, $FeCl_3$, and H_2O_2 are the components that shape the core of the chemical treatment. The dewatering aptitude of conditioned MD, cake, and centrate was calculated and determined through meticulous analysis.

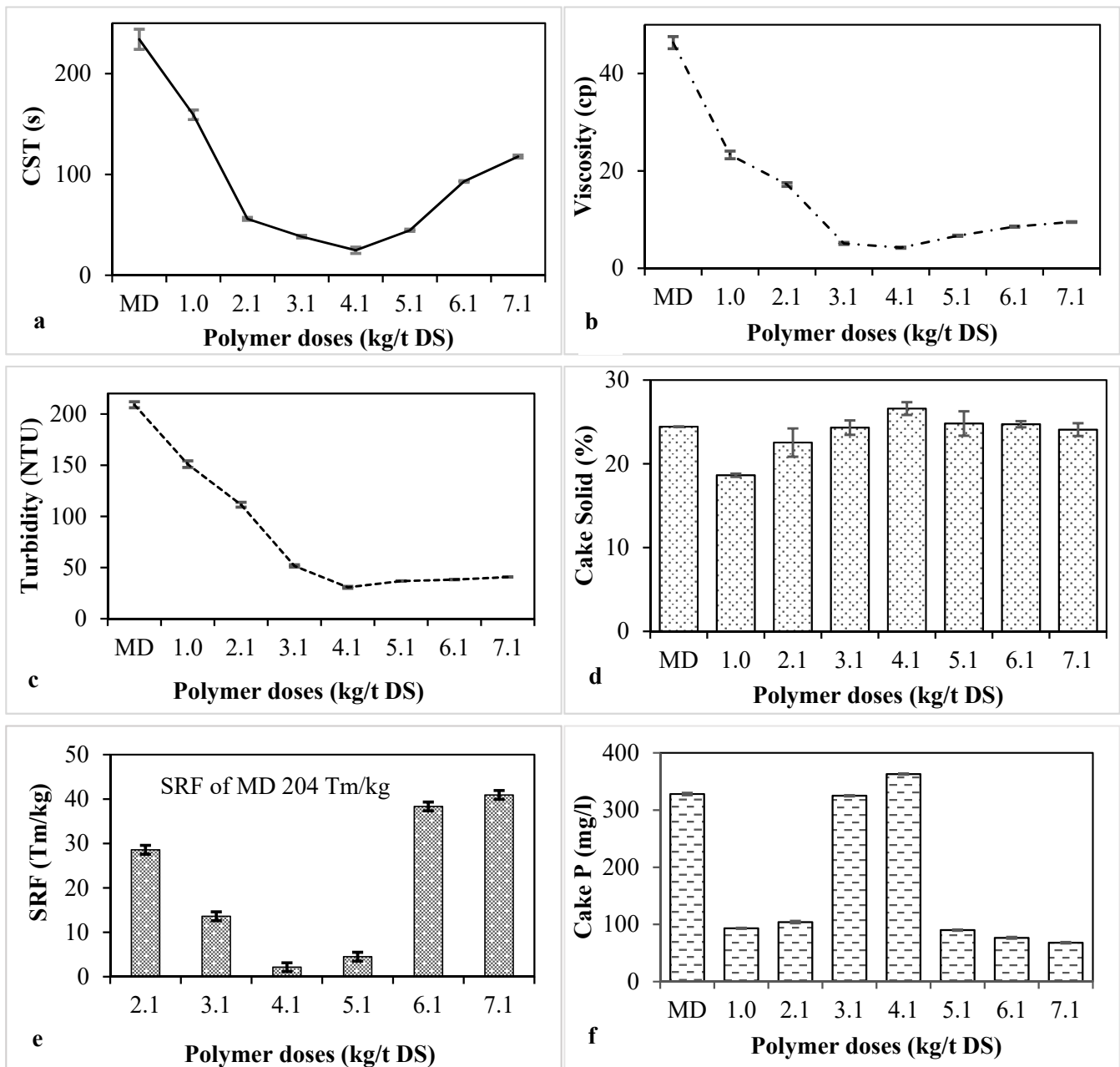### 3.1. Determination of the Optimum Polymer Dose for MD

Figure 1 shows graphs for various dewatering indices to determine the optimum polymer dose. MD was supplemented with 1 to 7.1 kg/t DS polymers to determine the optimum polymer dose. Dewatering indices, including CST, turbidity, SRF, viscosity, and cake solid content, were assessed to find the optimum polymer.

Figure 1a shows a decrease in CST value at a steeper slope as the polymer dose increases. CST reached the lowest value of 25 s at 4.1 kg/t DS, then increased gradually with polymer dose. Thus, 4.1 kg/t DS can be considered the optimum polymer dose for MD. At optimum polymer dose, the CST of conditioned MD was reduced by 89% compared with the CST of raw MD. Other dewatering parameters were also measured to observe the parameter trends with polymer dose to confirm the optimum polymer dose. Figure 1b,c,e shows a similar trend. However, Figure 1d shows that the cake solid content displayed an increasing pattern, rising to a maximum of 27% at 4.1 kg/t DS of polymer. Beyond the optimum polymer dose, no significant difference in cake solid % was observed for polymer doses at 3.1 kg/t or 5.1 to 7.1 kg/t DS. Polymer addition was able to raise the solid content of the cake by 12% from the raw cake.

Figure 1f shows a rising pattern in cake P content up to the optimum polymer dose, then a sharp decrease in cake P concentration. This P decrease is observed because an overdose of polymer cannot precipitate the remaining P in the MD. The rise in cake P is due to the binding of P through polymer floc formation. Maximum cake P concentration was 363 mg/L at 4.1 kg/t polymer, a 10% increase from the MD cake P content. MD viscosity was reduced by 91% and turbidity by 85% from those of raw MD after polymer conditioning.

The polymer added to the MD is used for flocculation, and the digestate solution becomes saturated at its optimum dose. As a result, the best value for MD's dewatering parameters is exerted by the optimal polymer dose. Beyond the optimum dose, however, the MD solution becomes supersaturated because of unused polymers in the digestate. Unused and overdosed polymer makes the supernatant viscous and increases filtering resistance. The reverse pattern of cake solid content, CST, and turbidity could be caused by the disaggregation and dispersion of flocs due to the excess polymer used [38]. In addition, dispersed colloid particles increase supernatant turbidity and blind the filtration media, raising turbidity and SRF. As the polymer dose increases, MD filterability increases, and filtrate viscosity is directly proportional to the polymer dose over the less-than-optimum dose region [39].

Observing the pattern of the graphs in Figure 1, it is evident that 4.1 kg/t DS is the optimum polymer dose for MD.

Figure 1. Optimum polymer dose determination for MD, based on (**a**) CST, (**b**) viscosity, (**c**) turbidity, (**d**) cake solid content, (**e**) SRF, and (**f**) cake P concentration.

3.2. Dual Conditioning of MD with FeCl₃ and Polymer with pH Adjustment

$FeCl_3$ is a trivalent metal coagulant added to polymer during the conditioning of MD to achieve better coagulation and flocculation. $FeCl_3$ reacts with P compounds and colloids, promoting particle aggregation, removal of suspended particles and P from the centrate, and facilitating digestate dewatering. $FeCl_3$ forms an insoluble ferric phosphate precipitate and is removed during centrifugal dewatering, resulting in P removal from the MD centrate [40]. Studies indicate that the alum precipitation region for the minimum solubility of aluminum hydroxide is at pH 6.0, with the pH range being 5.0 to 7.0 [41]. On the other hand, impurity removal during precipitation of ferric hydroxide through sweep flocculation occurs at pH 7.0 to 9.0, with minimum solubility at pH 8.0 [41].

$FeCl_3$ dosages ranging from 1, 1.5, 2.1, 2.5, to 3.1 kg/t DS were added to MD at pH 8.0 at the optimum polymer dose of 4.1 kg/t DS to determine the best combination.

Figure 2a shows that the minimum CST of conditioned MD was 27 s, and Figure 2b shows the lowest turbidity of 19 NTU at a dose of 4.1 kg/t DS polymer and 2.1 kg/t DS $FeCl_3$, adjusting the MD's pH to 8.0. With the addition of $FeCl_3$, values of CST started to decrease gradually, meeting the optimum point, beyond which parameter values started to increase due to overdose. When $FeCl_3$ is overdosed, it can cause charge reversal, causing particle floc to destabilize, thereby making dewatering more difficult [40].
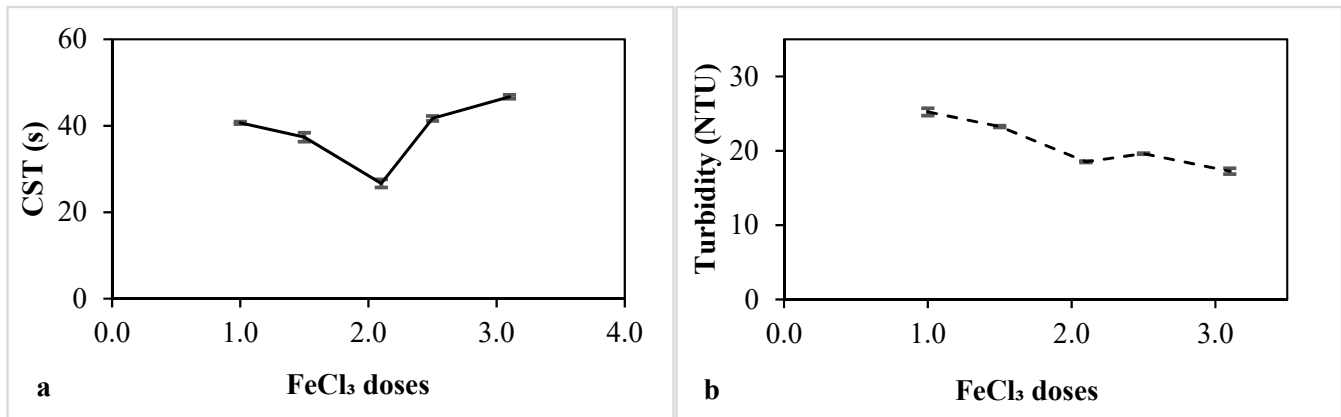
Figure 2. Finding the optimum $FeCl_3$ dose at 4.1 kg/t polymer for MD, based on (**a**) CST and (**b**) turbidity, with pH adjusted to 8.0.

A batch experiment was conducted at 2.1 kg/t DS $FeCl_3$, adjusting the pH to 8.0. Several polymer doses from 1 kg/t with an incremental value of 1 up to 6.1 kg/t DS were added to a fixed $FeCl_3$ dose of 2.1 kg/t DS. In addition, the MD's pH was adjusted with Ca $(OH)_2$ solution.

In Figure 3a, CST decreases with increasing polymer dose from 1 to 2.1 kg/t, as more polymer is added. Minimum CST of 15s was attained with 50% less polymer when $FeCl_3$ was applied. Compared to polymer conditioning alone, CST was reduced by 40% when we combined the polymer with $FeCl_3$ and changed the pH to 8.0. Figure 3c shows the same pattern with 3 cp viscosity at 2.1 kg/t DS polymer and 2.1 kg/t DS $FeCl_3$ added at alkaline pH 8.0. Dual application of chemicals reduced MD viscosity by 26% compared with only polymer addition. However, the graph in Figure 3b for turbidity and that in Figure 3c for viscosity show a similar pattern. Figure 3d shows an increase in cake solid content with polymer dose, but this decreases at a very much flatter rate as more polymer is added to the MD. Compared to conditioning with polymer alone, there was no apparent improvement in cake solid %.

In Figure 4a, the SRF graph indicates a gradual decrease with the combined chemical dose up to a specific polymer dose, then a gradual increase. When 2.1 kg/t DS $FeCl_3$ was added along with 2.1 kg/t DS polymer at pH 8.0 instead of adding only the polymer, the SRF of MD was lowered by an additional 80%. The same pattern is evident in the graphs in Figure 4b,c.

As shown in Figure 4b, the centrate TFe of MD decreased by 89%, and in Figure 4c, the centrate TS^{2-} concentration decreased by 56%. The graph in Figure 4d for PN/PS ratio shows a reduction of 95% with dual chemical addition compared with only polymer. The results show that $FeCl_3$ reduces the soluble PN content. Interstitial and bound water content decrease when PN content is reduced [42].

Excessive $FeCl_3$ and polymer dosage prevent contact with particle colloids, increasing turbidity and binding the filter media, resulting in higher filtering resistance. $FeCl_3$ can produce ferric hydroxide flocs in bicarbonate alkalinity, but it also releases H^+ if present in excess quantities, lowering pH [17,43]. Acidification may cause floc disintegration, particle dispersion, and turbidity to rise [44].
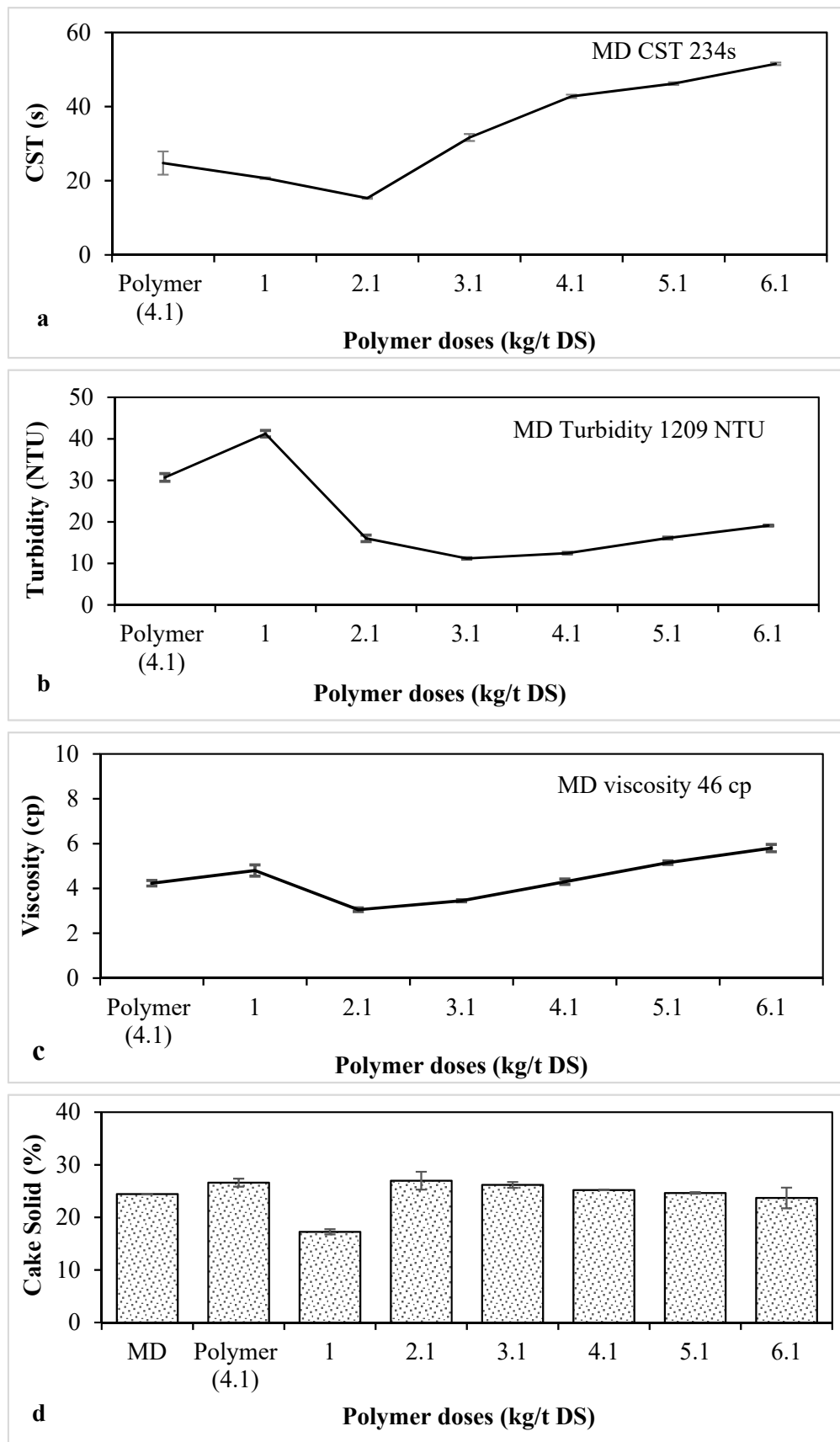
Figure 3. Polymer doses at 2.1 kg/t DS $FeCl_3$ for MD at pH 8.0, based on (**a**) CST, (**b**) turbidity, (**c**) viscosity, and (**d**) cake solid content.
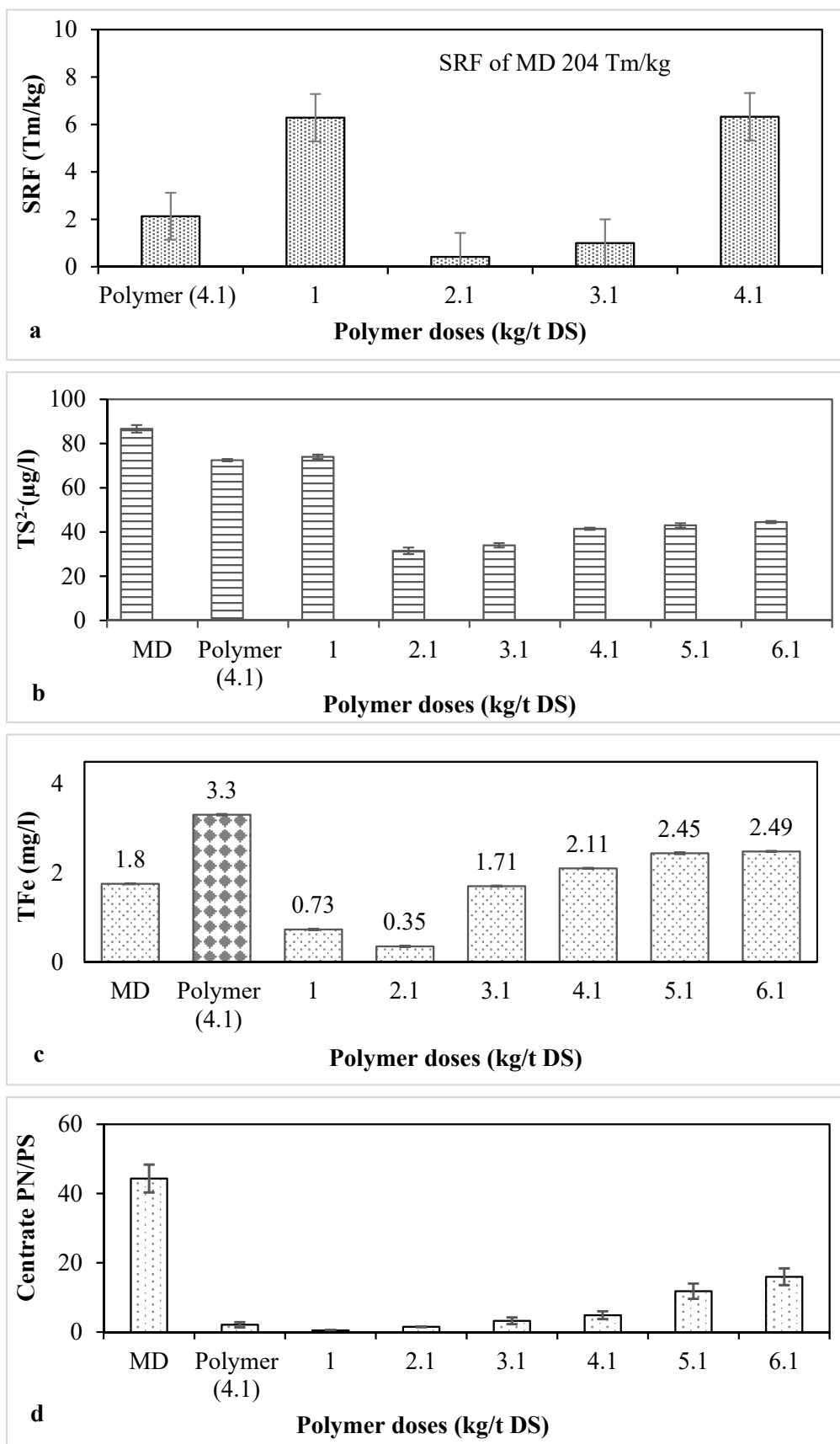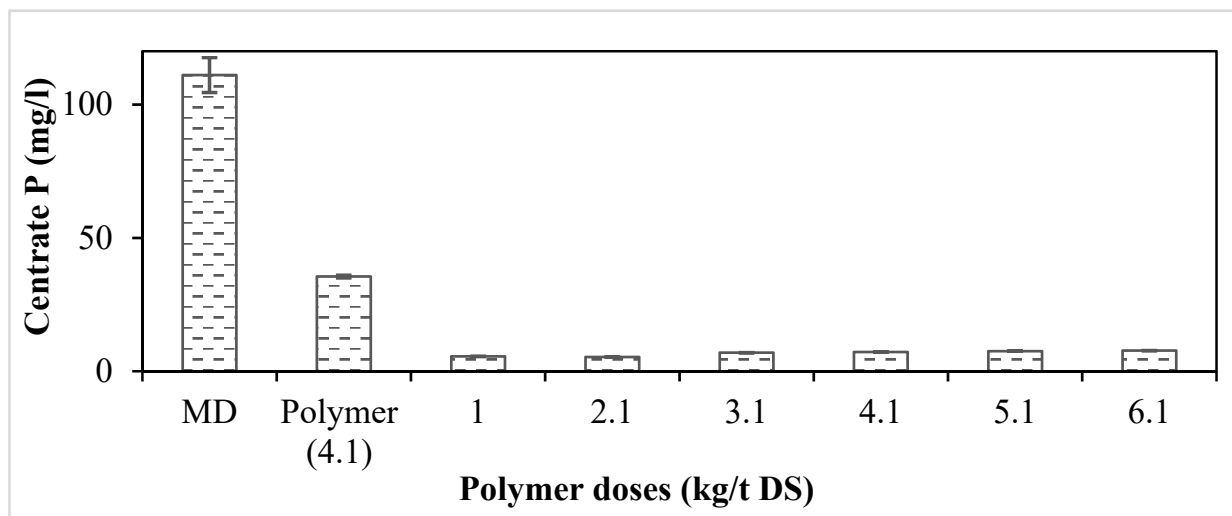
Figure 4. Polymer doses at 2.1 kg/t DS $FeCl_3$ for MD at pH 8.0, based on (**a**) SRF, (**b**) centrate TS^{2-}, (**c**) centrate TFe, and (**d**) PN/PS ratio.

Overdose of chemicals can cause the digestate's pH to drop, disintegrate EPS, and release intracellular material [45]. This raises turbidity and clogs the pores in the filtration media, leading to a rise in SRF [46]. Furthermore, Fe^{3+} binds with S^{2-} and PO_4^{3-} to precipitate them, which causes some S^{2-} to remain in the centrate.

Applying $FeCl_3$ and polymer at pH 8.0 adjusted with lime improved the P removal from the centrate. Dissoluble hydroxyapatite formation increased cake P content and caused P removal from the supernatant. P removal through adsorption in the floc is not robust as P is not tightly bound. Therefore, the floc adsorption mechanism requires a high dose of four molecules of Fe^{3+} to remove each P molecule [47].

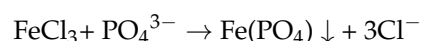If alkalinity is low, pH adjusted with $Ca(OH)_2$ can raise the pH of the digestate to above 5.0. Ca $(OH)_2$, once added to digestate, reacts with natural bicarbonate alkalinity to precipitate $CaCO_3$. Moreover, P that remains in the digestate as ortho-PO_4^{3-} can be easily removed by precipitation, adding $FeCl_3$ at pH higher than 6.5 to 10 [21].

Figure 5 shows a drastic drop in centrate P concentration with addition of polymer at a fixed $FeCl_3$ dose (2.1 kg/t DS) and alkaline pH. When 2.1 kg/t DS $FeCl_3$ was added with 2.1 kg/t DS polymer at pH 8.0, centrate P fell to a value of 5.35 mg/L, 85% lower than the centrate P with only polymer conditioning. $FeCl_3$, when added to the MD solution, dissolves in water and forms a complex hydroxide precipitate [48]. Moreover, low hydrolytic solubility product constant (pKsp) for Fe^{3+} means $Fe(OH)_3$ tends to form a precipitate and is challenging to dissolve or keep in solution [17,49]. Instead, it acts as an adsorption bridge between particles and forms flocs as per the reaction below [15].

$$Fe(OH)_3 \leftrightarrow Fe^{3+} + 3OH^- \quad \log pKsp = -38$$

Figure 5. Polymer doses at 2.1 kg/t DS $FeCl_3$ for MD at pH 8.0, based on centrate P concentration.

$Fe(OH)_3$ density is high and forms tight and stable iron floc with faster precipitation speed [50]. Fe^{3+} helps precipitation of P though adsorption or incorporation of PO_4^{3-} through formation of $Fe(PO_4)$ [49]:

$$FeCl_3 + PO_4^{3-} \rightarrow Fe(PO_4) \downarrow + 3Cl^-$$

$$nFe^{3+} + H_2(PO_4)^- + (3n - 1)OH^- \rightarrow Fe_nH_2(PO_4)(OH)_{3n-1} \text{ (s)}$$

Precipitated P can be separated through centrifugation and filtration of the suspended solids [51].

The dual application polymer and $FeCl_3$ stabilize the MD particles, causing improved flocculation and creating porous floc with a dense structure [52]. The porous structure creates a passage that helps to remove water from the conditioned MD. Dual chemical

addition thus improved the sedimentation and dewatering performance of MD. Thus, $FeCl_3$ maintains a strong and compact floc structure and forms dewatering channels more easily [52]. Thereby, combined $FeCl_3$ and polymer improve dewatering efficiency.

3.3. Combined Conditioning with Polymer, FeCl$_3$ and H$_2$O$_2$ for MD with pH Adjusted at 8.0

The addition of H_2O_2 with polymer and $FeCl_3$ can enhance floc formation. In addition to serving as an oxidizing agent, H_2O_2 can aid in the development of stronger, denser flocs. It can enhance particle bridging and binding in sludge, increasing floc formation and settling properties.

Moreover, H_2O_2 assists in the oxidation of P compounds, thus facilitating P removal. It can oxidize or break down odor-causing organic compounds, leading to improved odor control. H_2O_2-based pathogen inactivation can contribute to lowering the pathogen content in treated biosolids. Therefore, the effect of adding H_2O_2 along with polymer and $FeCl_3$ on the dewaterability improvement of MD was also analyzed.

The next run included the addition of $FeCl_3$, H_2O_2, and polymer at pH 8.0. The pH of the MD was adjusted with 2.7 mL of 1M Ca $(OH)_2$. Varying doses of H_2O_2 from 400 to 800 mg/L with an incremental value of 100 were added to the MD with 2.1 kg/t DS polymer and 2.1 kg/t DS $FeCl_3$ at pH 8.0.

Figure 6a shows that adding H_2O_2 with $FeCl_3$ and polymer lowered the CST value but at a flatter slope. H_2O_2 at a dose of 500 to 700 mg/L showed the optimum range of CST values from 8 to 9 s. According to Figure 6a, the lowest CST was observed at 2.1 kg/t DS polymer, 2.1 kg/t DS $FeCl_3$, and 600 mg/L of H_2O_2 at pH 8.0. The CST of MD was reduced by approximately 50% more with addition of H_2O_2 compared with the dual addition of polymer and $FeCl_3$. The turbidity graph in Figure 6b shows the same pattern.
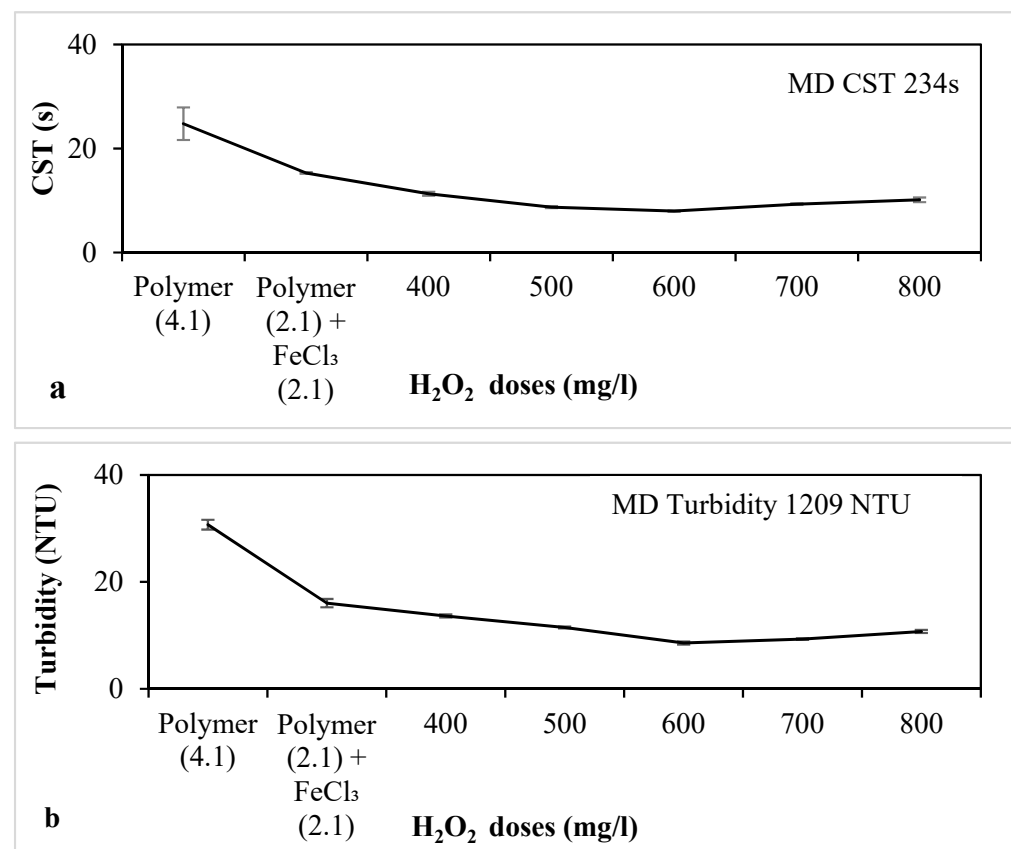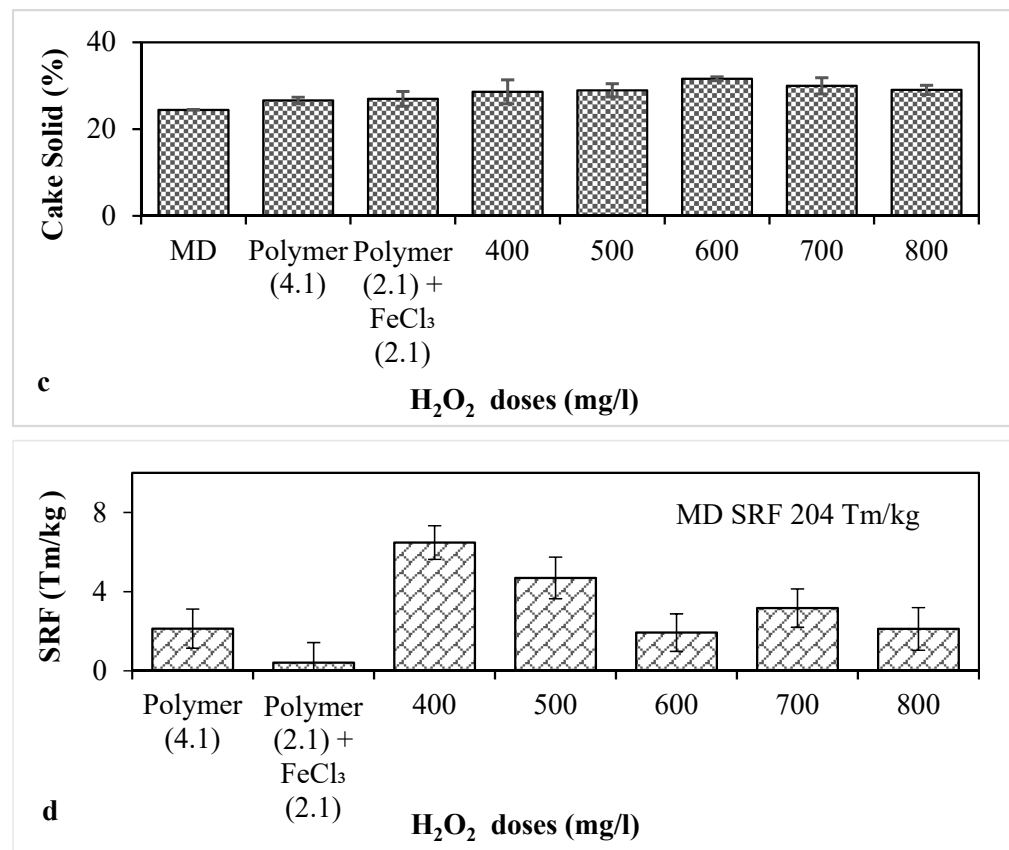
Figure 6. *Cont.*

Figure 6. Determination of the optimum H_2O_2 dose at 2.1 kg/t DS polymer and 2.1 kg/t DS FeCl$_3$ for MD at pH 8.0, based on (**a**) CST, (**b**) turbidity, (**c**) cake solid content, and (**d**) SRF.

The graph for cake solid content in Figure 6c shows an increasing pattern but does not vary significantly as doses of H_2O_2 are altered. Average cake solid content with H_2O_2 dose variation improved only by 11% compared with the addition of only polymer and FeCl$_3$. A 17% increase in cake solid content after polymer and FeCl$_3$ conditioning with H_2O_2 addition at the best combined chemical dose might be ascribed to increased precipitation of complex P and S compounds.

Figure 6d shows that the SRF value decreased with H_2O_2 addition but increased after a certain H_2O_2 dose. However, SRF rose from 0.42 Tm/kg with dual conditioning to 1.93 Tm/kg after combined conditioning with 2.1 kg/t DS polymer, FeCl$_3$, and H_2O_2 at alkaline pH. A possible explanation could be the creation of pin floc and breaking flocs into smaller molecules due to the H_2O_2 addition. Smaller flocs can cause binding of the filtration media, and pin floc makes filtration difficult, causing increased SRF values.

The centrate P concentration graph in Figure 7a illustrates the lowest amount of P as 1.55 mg/L for polymer (2.1 kg/t DS), FeCl$_3$ (2.1 kg/t DS), and 600 mg/L H_2O_2 at pH 8.0, equivalent to 99% P removal from raw MD. Combining the chemical treatment with polymer, FeCl$_3$, and H_2O_2 improved the P removal from the centrate by 20%. Degradation of H_2O_2 produces OH⋅ free radicals, and the Fe^{3+} in the solution is a catalyst for producing OH⋅ [53]. H_2O_2 removes P from the centrate [54].
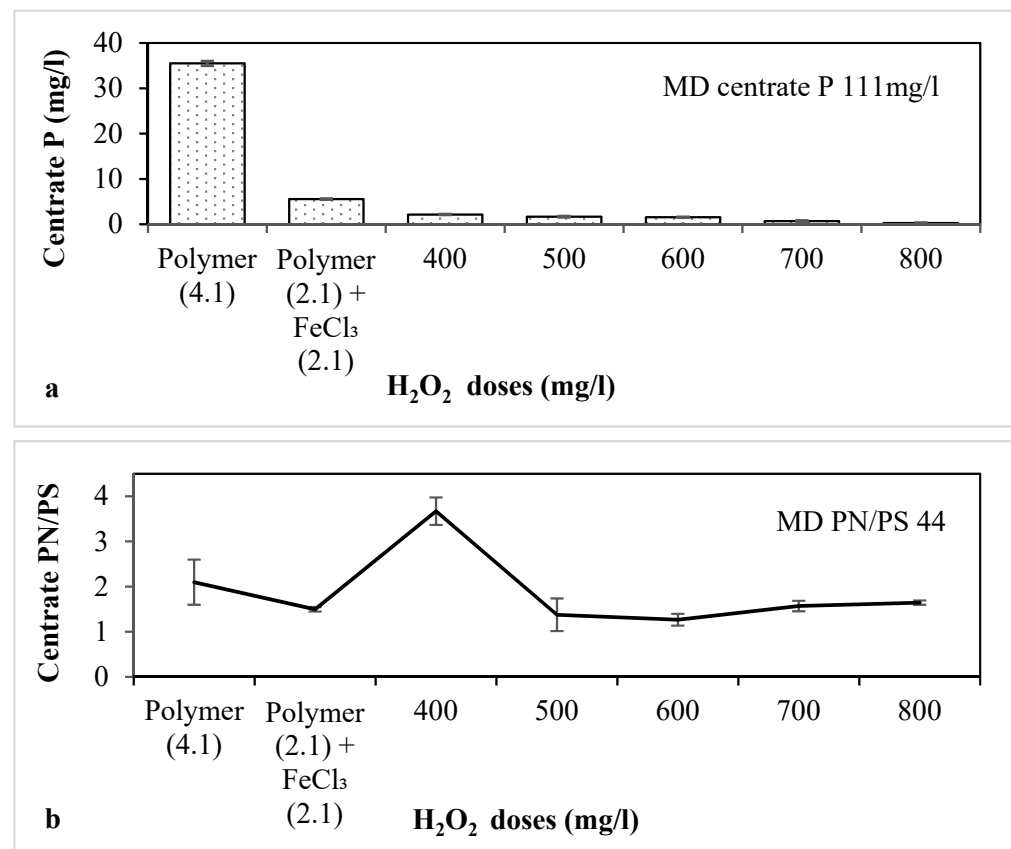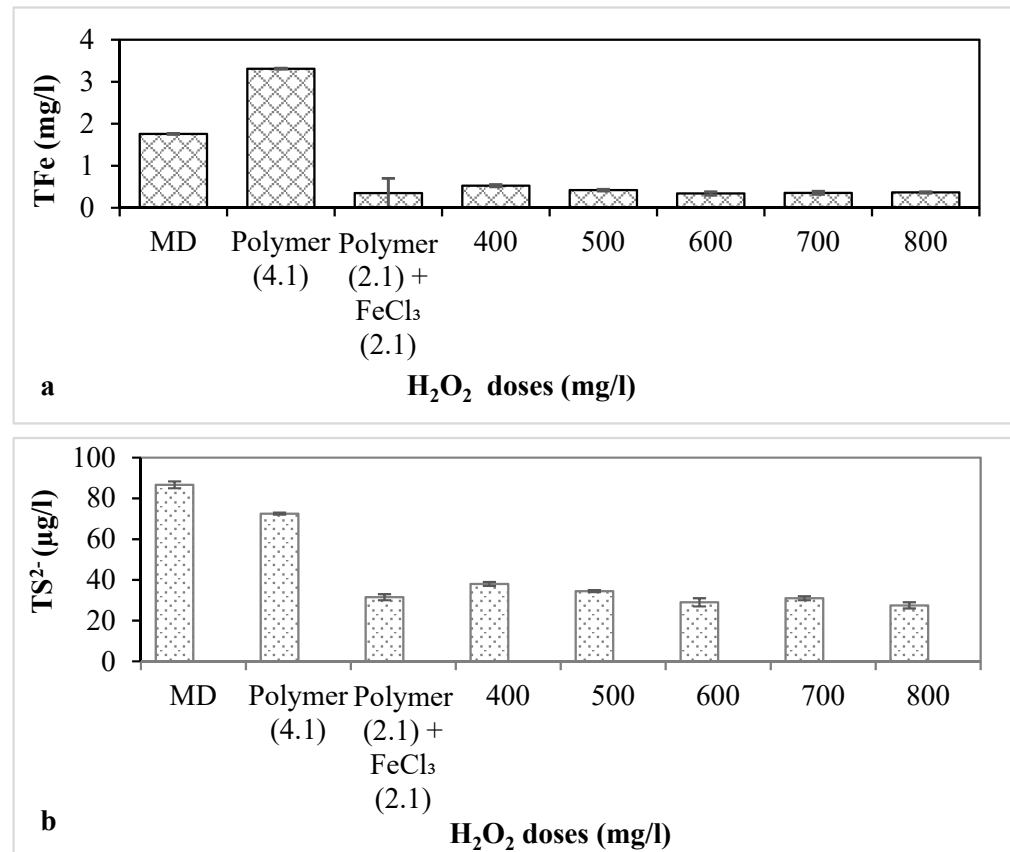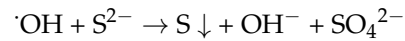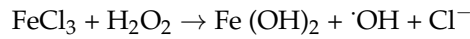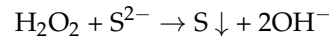
Figure 7. Effect of H_2O_2 addition with 2.1 kg/t DS polymer and 2.1 kg/t DS FeCl$_3$ for MD at pH 8.0 on (**a**) centrate P and (**b**) PN/PS ratio.

Figure 7b demonstrates a decrease in the centrate PN/PS ratio as the H_2O_2 dose increases. H_2O_2 doses of 500 and 600 mg/L did not significantly change the centrate PN/PS ratio. A slight PN/PS ratio increase can be seen as the H_2O_2 increased from 700 to 800 mg/L. The optimum number of decreases in PN/PS ratio were achieved at H_2O_2 doses ranging from 500 to 600 mg/L. Figure 7b shows a further 16% reduction in the PN/PS ratio with the addition of H_2O_2 compared with polymer and FeCl$_3$ conditioning. The reduced PN/PS ratio in the EPS can be attributed to the improved dewatering of the MD [55]. Alkalinity and H_2O_2 disrupt digestate floc, cells, and EPS, releasing the mechanically bound water and removing PN from the cell pellet to the outer slime portion [19]. H_2O_2 also oxidizes the functional groups in the EPS, penetrates the microbe's cell walls, improves cell membrane permeability, and causes cell wall lysis, releasing intracellular water and improving digestate dewaterability [24]. PN has water-holding capacity; particle surfaces become less hydrophobic due to reduced anion-binding PNs [55].

Figure 8a demonstrates no significant variation of centrate TFe concentration with changing H_2O_2 doses during combined chemical conditioning at alkaline pH. The amount of TFe and TS^{2-} in the centrate showed minimum values of 0.34 mg/L (Figure 8a) and 0.29 μg/L (Figure 8b), respectively.

Figure 8b shows a decreasing pattern in centrate TS^{2-} concentration with H_2O_2 addition, polymer, and FeCl$_3$. H_2O_2 addition lowered the centrate TS^{2-} concentration 10% more than polymer and FeCl$_3$. Fe^{3+} combined with S^{2-}, one of the primary sources of odor, forms odor-free FeSO$_4$. Thus, TFe supplemented centrate TS^{2-} concentration [24]. The odor emitted from MD is mainly attributed to sulfur compounds and ammonia or amine [56]. Sulfur compounds causing odour are mainly H_2S or volatile organic sulfur compounds [57]. The reduction of S^{2-} ions in the centrate by 10% with H_2O_2 conditioning is attributed to the fact that Fe^{3+} reduces sulfur compounds from dewatered biosolids.

Combined application of H_2O_2 and $FeCl_3$ to MD oxidizes S^{2-} ions into elemental sulfur (S) or sulfate (SO_4^{2-}) ions [58]. $FeCl_3$ enhances S^{2-} ion oxidation when added along with H_2O_2. The reaction is as below [56]:

$$H_2O_2 + S^{2-} \rightarrow S \downarrow + 2OH^-$$

$$FeCl_3 + H_2O_2 \rightarrow Fe(OH)_2 + {}^\cdot OH + Cl^-$$

$${}^\cdot OH + S^{2-} \rightarrow S \downarrow + OH^- + SO_4^{2-}$$

Figure 8. Determination of optimum H_2O_2 dose at 2.1 kg/t DS polymer and 2.1 kg/t DS $FeCl_3$ at pH 8.0, based on centrate (**a**) TFe, (**b**) TS^{2-}.

The produced S and sulfate compounds are less odorous than H_2S and can be removed from the conditioned MD by filtration or centrifugation [59].

Furthermore, the production of $OH^\cdot$ is crucial to the quantity of H_2O_2 and Fe^{3+} in the digestate and the solution pH. When there is an excess of H_2O_2 in the digestate, a lot of $OH^\cdot$ is produced during the breakdown of H_2O_2. The side reactions then consume the majority of the free $OH^\cdot$ radicals before they are used to remove contaminants like N and P [54].

The excess amount of Fe^{3+} or Fe^{2+} in the digestate consumes the free radicals that hinder the oxidation reaction. At higher doses, H_2O_2 has a scavenging effect on $OH^\cdot$ free radicals [54]. Since H_2O_2 exceeds the optimum dose, it auto-decomposes to produce oxygen plus water and recombines with $OH^\cdot$ radicals to produce hydroperoxyl ($OH^\cdot_2$) radicals [60]. The potential for oxidation of $^\cdot OH_2$ radicals is lower than $^\cdot OH$ free radicals, which explains the low P removal beyond optimum H_2O_2 doses [54]:

$$H_2O_2 + OH^\cdot \rightarrow H_2O + {}^\cdot OH_2$$
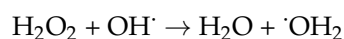
3.4. Biosolids Quality Analysis

Fecal coliform in untreated MD was found to be $>5.72 \times 10^7$ MPN/g DS. Table 2 shows that after conditioning MD with 2.1 kg/t DS $FeCl_3$, 2.1 kg/t DS polymer, and 600 mg/L H_2O_2 at pH 8.0, the fecal coliform count was 6728 MPN/g DS.

Table 2. Fecal coliform count in combined chemical-conditioned dewatered MD biosolids.

MD Cake Fecal Coliform Count	MPN Index	Fecal Coliform (MPN/mL)	Fecal Coliform (MPN/g DS)
MD fecal coliform			
5-5-5	>160.9	$>1.61 \times 10^7$	$>5.72 \times 10^7$
Chemically conditioned MD fecal coliform			
1-0-0	0.2	2000	6728
Chemically conditioned MD fecal coliform (Regrowth potential after two weeks)			
2-0-0	0.4	4000	13,455

The combined chemical treatment reduced the fecal coliform count by 98%, but class A biosolids could not be achieved. Therefore, fecal coliform regrowth potential was tested by keeping the chemically treated biosolids incubated for two weeks at ambient temperature. After two weeks, a 50% regrowth of fecal coliform was observed, with a count of 13,455 MPN/g DS, classifiable as class B biosolids.

E. coli was also measured in untreated and chemically treated biosolids. Chemically treated biosolids were heat treated at 40, 60, 80, and 100 °C. *E. coli* was found in untreated biosolids as 1.85×10^8 CFU/g DS. The *E. coli* count was reduced by 99.98% after combined chemical treatment with the best dose of 2.1 kg/t DS $FeCl_3$, 2.1 kg/t DS polymer, and 600 mg/L H_2O_2 at pH 8.0, as shown in Table 3.

Table 3. *E. coli* count in combined chemically conditioned dewatered MD biosolids.

Types of Heat Treatment of Combined Chemically Conditioned Biosolids	Chemically Conditioned	After Two Weeks Regrowth
	E. coli (CFU/g DS)	*E. coli* (CFU/g DS)
MD cake (Untreated)	1.85×10^8	1.85×10^8
Combined chemically conditioned at 20 °C	3.07×10^4	1.7×10^5
40 °C Heat treated	1.7×10^4	1.6×10^5
60 °C Heat treated	1.02×10^4	1.23×10^5
80 °C Heat treated	2.39×10^3	1.77×10^5
100 °C Heat treated	NG	NG

Fecal coliform and *E. coli* content of chemically treated MD were greatly reduced but could not meet class A criteria; heat treatment was carried out to observe to what extent heat could potentially reduce the coliform content to meet class A biosolids criteria. Therefore, the chemically treated cake of biosolids was heat treated under 40, 60, 80, and 100 °C. However, after the heat treatments at 40, 60, and 80 °C, *E. coli* was still found in the biosolids and was counted at 1.7×10^4, 1.02×10^4, 2.39×10^3 CFU/g DS. Results showed that heat treatment at 80 to 100 °C could produce class A biosolids.

Moreover, after two weeks, *E. coli* growth was observed following the heat treatment at 20, 40, 60, and 80 °C and reported as 1.7×10^5, 1.6×10^5, 1.23×10^5, 1.77×10^5 CFU/g DS. However, no *E. coli* growth was detected after conditioned biosolids were heat treated at 100 °C, and no regrowth was observed. According to the data in Table 3, heat treatment at 100 °C met the class A biosolids criteria, with no *E. coli* present and no regrowth potential observed.

3.5. Comparison of Different Doses

Comparative analysis was conducted of various dewatering parameters of MD conditioned with only polymer, dual application of polymer with $FeCl_3$, and combined addition of polymer, $FeCl_3$, and H_2O_2. The experimental data reveal that oxidation of MD with H_2O_2 in addition to polymer and $FeCl_3$ and digestate pH adjusted to 8.0 exhibited the best dewatering property.

The study results reveal 4.1 kg/t DS as the optimum polymer dose for MD, based on cake solid content, CST, SRF, viscosity, and centrate turbidity. Table 4 shows that polymer conditioning improved the CST of MD by 89%, turbidity by 85%, cake solid content by 6%, centrate P concentration by 67%, and centrate TS^{2-} concentration by 16% compared with raw MD.

Table 4. Comparison of various dewatering parameters on different chemical combinations.

Conditioned Chemical Doses, (Polymer & $FeCl_3$ in kg/t DS, H_2O_2 mg/L)	MD	Polymer (4.1)	Polymer (2.1) + $FeCl_3$ (2.1) at pH 8.0	Polymer (2.1) + $FeCl_3$ (2.1) + H_2O_2 (600) at pH 8.0
CST (s)	234 ± 10	25 ± 3.1	15 ± 0.2	8 ± 0.1
Turbidity (NTU)	1209 ± 2.9	31 ± 0.9	16 ± 0.8	9 ± 0.3
Cake solid (%)	25.6 ± 0	27 ± 0.8	27 ± 1.7	32 ± 0.5
Centrate P (mg/L)	111 ± 6.6	36 ± 0.5	7 ± 0	1.6 ± 1.6
TS^{2-} (μg/L)	87 ± 1.7	73 ± 0.5	32 ± 1.5	29 ± 2

When $FeCl_3$ was combined with the polymer at pH 8.0, the polymer dose was reduced by 50%. The CST and turbidity of dual-chemical-conditioned MD improved by 40 and 48%, respectively, TS^{2-} concentration by 56%, centrate P removal by 80% more than only polymer conditioning at a dose of 2.1 kg t DS and 2.1 kg/t DS $FeCl_3$ at pH 8.0. There was no change in cake solid content observed.

To improve the P recovery and hygienic effect of chemical treatment, MD was further treated with the combined addition of polymer, $FeCl_3$, and H_2O_2 at alkaline pH. Adding H_2O_2 further improved CST by 50%, turbidity by 44%, cake solid by 20%, centrate TS^{2-} concentration by 10%, and centrate P by 70% more than dual chemical application.

Improvement of 97 to 99% compared with raw MD in various dewatering parameters such as CST, turbidity, centrate P, and TS^{2-} was observed when MD was conditioned with combined chemicals at pH 8.0. Centrate TS^{2-} values were 29 μg/L. The highest cake solid content of 31.6% was observed when digestate was conditioned with the combined addition of polymer, $FeCl_3$, and H_2O_2.

The best combination of chemicals to achieve the best post-treatment of MD was 2.1 kg/t DS $FeCl_3$, 2.1 kg/t DS polymer, and 600 mg/L H_2O_2 at pH 8.0, in terms of dewatering enhancement, volume reduction, odor reduction, and P removal from centrate.

The combined chemical conditioning could reduce the fecal coliform count by 98% but the product remained as class B biosolids. A regrowth trend was also observed for fecal coliform and *E. coli* in the dewatered cake. Moreover, $FeCl_3$ and H_2O_2 oxidize S^{2-} ions to produce less odorous S or SO_4^{2-} compounds.

4. Conclusions

The challenge of post-treating MD arises from its high volume, P and pathogen content, and the presence of odorous substances. Traditional polymer treatment before dewatering is costly and ecotoxic due to residual polymer. This study offers insights into biosolids management and resource recovery through post-treatment for sustainable biosolids handling. The research explores a wide array of dewatering indices beyond conventional metrics, encompassing SRF, cake phosphorus concentration, centrate quality, PN/PS ratio, and phosphorus concentration. Chemical treatment involving polymer only, polymer with$FeCl_3$, and combined addition of polymer, $FeCl_3$ and H_2O_2 were examined for

their impacts on conditioned MD, dewatered cake, and centrate parameters. The optimum polymer dose was found to be 4.1 kg/t DS for the lab-scale study. The post-treatment efficiency was enhanced further by dual polymer and $FeCl_3$ addition at alkaline pH, with the most effective combination being 2.1 kg/t DS polymer and 2.1 kg/t DS $FeCl_3$ at pH 8.0, reducing polymer use by 50%. The addition of H_2O_2 amplified benefits, notably reducing pathogens and odor, and increasing cake solid content and P removal from the centrate, thereby enhancing post-treatment efficiency. A holistic approach combining all three chemicals proved most effective, boosting dewatering parameters by 4% and cake solid content by 20%, respectively, compared with the dual-chemical dose. Fecal coliform was reduced 98% with the combined chem, though biosolids remained class B. However, future work may focus on removing metals and other contaminants and converting MD to class A biosolids. By leveraging this synergistic approach, this study provides a sustainable and impactful method for MD sludge management, yielding insights for future biosolids post-treatment and resource recovery strategies.

Author Contributions: U.S.H. was responsible for the following tasks: conceptualization, methodology, equipment and chemical sourcing, sample collection, lab-scale investigations, formal analysis of samples, data analysis, calculation, writing journal drafts, and project administration. E.E. contributed through conceptualization, data curation, resource and funding procurement, draft review, editing, and supervision. A.A. was responsible for manuscript writing and review. *E. coli* testing materials and chemicals, *E. coli* calculations, and *E. coli* testing supervision were supported by J.M. R.M. helped analyze samples to measure the *E. coli* count. All authors have read and agreed to the published version of the manuscript.

Data Availability Statement: Data will be available upon request.

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

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