

Full Length Research Paper

Suppressing dissolved hydrogen sulfide in a sewer network using chemical methods

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Corrosion process and odor emission concerns caused by H₂S are a constant focus for many sewage system municipalities, usually from the first day of system operation. This study investigates the effect of several chemicals on prohibition of H₂S generation in sewage, where reagents were applied either individually or in combinations. Various concentrations of calcium peroxide, calcium hypochlorite, magnesium hydroxide, ferric chloride, and their combination with certain ratios were examined. Oxidation reduction potential (ORP) and pH were measured as the indicators for mitigation of H₂S. Although both methods (adding chemicals individually or in combination) raised the ORP and pH to the safe range (that is ORP ≥ 50 mV and 8.5 ≤ pH ≤ 9), experiments revealed that designed combination of chemicals resulted in more promising outcomes. The most cost effective combination was an admixture of 2 mg FeCl₃/L and 2.5 mg Mg(OH)₂/L with 30 min effective reaction time which could reduce the hydrogen sulfide greater than 95%. This combination is a blend for only 25% of the optimum dosage of these chemicals compared to when they were used individually. As a result, the aforementioned combination saved 12% on chemical costs compared to use of calcium peroxide as the most effective individual chemical.

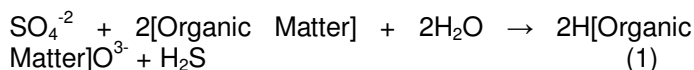
Key words: Hydrogen sulfide, chemical, corrosion, sewage.

INTRODUCTION

Environmental protection nowadays has become one of the most important initiatives around the world. In efforts to decrease environmental pollutants, hydrogen sulfide (H₂S) generation is important enough to make countries more concerned to prevent or remove such compound from sewage systems. Significant problems associated with hydrogen sulfide generation are corrosion of sewer concrete pipes, release of obnoxious odors to the atmosphere, toxicity to sewer workers or human health, water supply and environment pollution, influence on biological processes in sewage treatment plants, and cost escalation (US Environmental Protection Agency, 1974; Nielsen et al., 1998; WERF, 2003; Witherspoon et al., 2004). The presence of sulfide in wastewater is a result from physical, biological, and chemical processes

which mostly take place in the submerged portion of the sewer networks (Elmaleh et al., 1998). The highly toxic compound, H₂S, can be formed in any aqueous system containing both organic matter and sulfate (Poulton et al., 2002).

Production of sulfide in sewage collection networks is mainly a result of sulfate reducing bacteria (SRB) activity. Edwards et al. (2001) proposed the following reactions for describing hydrogen sulfide generation under SRB condition in wastewater streams:



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The rate of sulfide generation depends on several factors including pH, temperature, concentration of organic materials and nutrients, sulfate concentrations, collection

Table 1. The designed instruction of combined chemicals for addition to sewage.

Combination no.	Materials*
1	25% X_{FeCl_3} and 25% $Y_{\text{Mg(OH)}_2}$
2	25% X_{FeCl_3} and 37.5% $Y_{\text{Mg(OH)}_2}$
3	37.5% X_{FeCl_3} and 25% $Y_{\text{Mg(OH)}_2}$
4	37.5% X_{FeCl_3} and 37.5% $Y_{\text{Mg(OH)}_2}$
5	25% X_{FeCl_3} and 25% $Z_{\text{Ca(ClO)}_2}$
6	25% X_{FeCl_3} and 37.5% $Z_{\text{Ca(ClO)}_2}$
7	37.5% X_{FeCl_3} and 25% $Z_{\text{Ca(ClO)}_2}$
8	37.5% X_{FeCl_3} and 37.5% $Z_{\text{Ca(ClO)}_2}$

* X_{FeCl_3} , $Y_{\text{Mg(OH)}_2}$ and $Z_{\text{Ca(ClO)}_2}$ are the optimum concentrations of FeCl_3 , Mg(OH)_2 and Ca(ClO)_2 , respectively.

system parameters and performance, and ORP (ASCE, 1989; Delgado et al., 1999). Sulfide formation by sulfate reducing bacteria can be inhibited or eliminated through optimization of the environmental parameters that is ORP, pH, dissolved oxygen (DO), and temperature (Zhang et al., 2008). For sewage systems, one of the most common alternatives to protect the environment is by controlling the formation or generation of sulfides. Several techniques are currently used by the wastewater industries worldwide, to prevent or control sulfide generation in the sewage systems, such as increasing DO concentration, chemical oxidation, iron salts and nitrate salts addition, and increasing pH using effective chemicals (Hobson and Yang, 2000; De Lomas et al., 2005).

Reactions between chemicals and sulfide species in sewage are complex with some damages on the sewer network. This phenomenon compels costly renovation or repair of sewer networks after a while. Hence, using effective and economical chemicals in order to remove sulfide from sewage is a cure for this predicament which will result in two advantages:

- (I) Protection of networks against any corrosion and deterioration,
- (II) Provision of odorless and non-toxic environment.

As such, much attention has been given to chemical technologies for sulfide control in sewage systems. As ultimate treatment, SRB activity can be inhibited by pH elevation using inhibitors such as NaOH , Mg(OH)_2 , Ca(OH)_2 (Nemati et al., 2001; Jefferson et al., 2002; De Lomas et al., 2005). Oxidant materials (e.g. H_2O_2 , Ca(ClO)_2 , O_2) chemically oxidize sulfide, thereby decreasing the amount of sulfides will be observed (Zhang et al., 2008). Moreover, addition of iron salts (Fe^{2+} , Fe^{3+}) also removes hydrogen sulfide from the sewage which can convert H_2S in the sewage to insoluble metallic sulfides (Nielsen et al., 2005).

However, adding chemical materials is not free from disadvantages such as solid production, emission of

some toxics, and their transportation and storage in plants. The chemical material addition method was applied in this work to deplete hydrogen sulfide dissolved in sewage of Kuala Lumpur. The first objective of this study was to determine the effectiveness of the chemical addition on the environmental parameters namely ORP and pH. Secondly, the effective chemical dosages and the reaction time needed for hydrogen sulfide depletion was investigated for selected chemicals, which were applied individually or in combination. Thirdly, the cost analyses were also performed to find out the most economical chemical. The results reported herein may assist municipalities in using more effective and economical dosages of chemicals as corrosion and odor control alternatives.

MATERIALS AND METHODS

Materials

In regard to the efficiency of chemicals and previous researches, chemical materials such as Mg(OH)_2 (58.33% purity), Ca(ClO)_2 (70% purity), FeCl_3 (95% purity), and CaO_2 (50% purity) were selected to perform this work. After determining the optimum dosage of the mentioned chemicals, eight combinations of chemical materials (FeCl_3 with Mg(OH)_2 and FeCl_3 with Ca(ClO)_2) as listed in Table 1 were designed for addition to the sewage samples. The raw sewage was collected from a gravity sewer network outlet receiving 90% domestic and 10% low-toxic industrial wastewater in Kuala Lumpur. Sampling was carried out during daytime and collected samples were preserved at 4°C. Characterization was carried out after 8 to 12 h to provide samples with septic condition for analyses. The Thermo ECO 3-Star ORP and pH meter were used to measure ORP and pH for a duration of 90 min with 15 min intervals during the first hour followed by a single reading at 90 min after adding chemicals. Each sulfide test kit was used to investigate the hydrogen sulfide concentration changes at specific intervals.

Experimental methods

Laboratory experiments were carried out using a 1.2 L container filled by 1000 ml of the sample. The designed concentrations of the chemicals were then injected into the containers. The containers were sealed using rubber caps to prevent air from entering. To investigate the effect of FeCl_3 , it was mixed thoroughly in the sewage for 15 min when Mg(OH)_2 and Ca(ClO)_2 were added separately to the prepared mixed samples. Several concentrations of chemicals were examined in order to estimate their effective dosage. A 1.2 L container filled with untreated sewage was kept under the same condition of each experiment as the control sample during each step of tests. The mean values of a minimum of three measurements with less than 5% error were analyzed and presented in this study.

RESULTS AND DISCUSSION

Addition of chemicals individually

Initial experiments were conducted to determine the effective concentration of designed chemicals when they

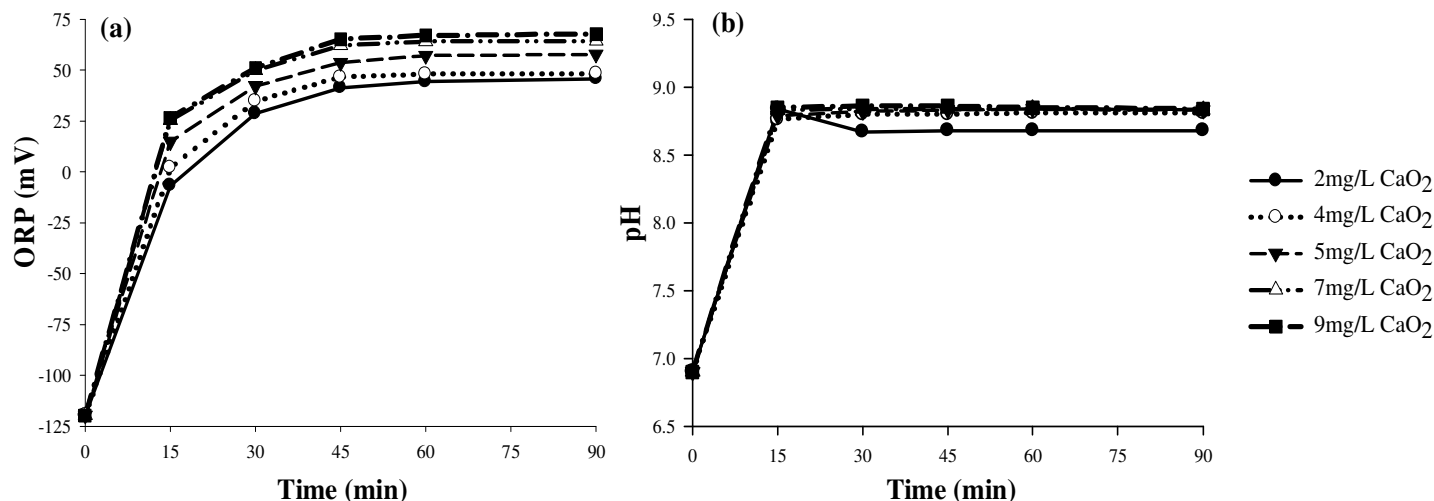
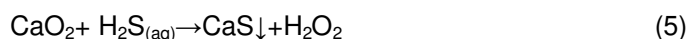
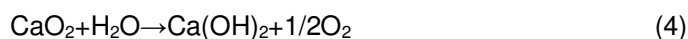


Figure 1. (a) ORP values and (b) pH values at different reaction times for different concentrations of added CaO_2 .

were added individually. The ORP was analyzed as an indicator whose relative changes can be used to interpret the effect of the applied SRB inhibition technique. In other words, increase in ORP is strongly related to the inhibition of SRB numbers; thus, ORP increment caused by chemical addition indicates a decrement in SRB activity and consequently cease on H_2S generation. This effort is agreed by the fact that ORP always rises after the SRB become extinct (Chang et al., 2008). The typically favorite range of ORP for SRB is defined by -50 to -300 mV (Edwards et al., 2001), furthermore, ORP above 50 mV was reported to circumvent hydrogen sulfide generation due to SRB (Derek, 1995). Besides, the proportion of H_2S in sewage at a pH higher than 8 decreases to less than 10% of dissolved sulfides (Thistlethwayte, 1972). However, Jefferson et al. (2002) proposed that pH raise to levels above 9 leads to some problems such as sludge generation, ammonia gas evolution, and calcium carbonate precipitation.

Therefore, $\text{ORP} > 50$ mV and $8.5 \leq \text{pH} \leq 9$ were picked as the safe range of response to evaluate the experimental results for each designed value of additional chemical. Calcium peroxide (CaO_2) as inhibitor, will form hydrogen peroxide if in contact with acid while with water, it will immediately begin to decompose and releasing oxygen producing calcium hydroxide (Zhang et al., 2008). Chang et al. (2007) stated that CaO_2 (solid-phase) is a slow oxygen releaser able to deplete the sulfide concentration in water. Reactions (4) and (5) depict production of base, generation of oxygen, and sulfide oxidation which can cause ORP and pH increment and SRB inhibition.



Various concentrations (2, 4, 5, 7, 9 mg/L) of this

chemical were examined until less than 5% changes was observed in ORP. Figure 1a, b show the changes in ORP and pH versus time after the addition of CaO_2 by designed concentrations. It is revealed from Figure 1a that by the first 15 min the ORP had increased rapidly from -120 mV to a maximum value of 27 mV. Afterwards, the changes of ORP took place gently, and no significant change in ORP was observed after 45 min. This indicates that most reaction of CaO_2 happened for a period of 45 min. The efficient reaction time for CaO_2 in sewage samples was 45 min after addition. At this time, concentration between 4 to 5 mg/L of this material is enough to increase the ORP to more than 50 mV. In this case, the ORP rose to a maximum of 54 mV. However, the ORP value increased to more than 50 mV within the reaction time of 30 min when between 7 to 9 mg/L of CaO_2 was used. Figure 2 shows more specifically, ORP values obtained for each concentration of CaO_2 when reaction times were 45 (Equation 1) and 30 (Equation 2) min. Second order curves fitted data well and pertinent equations ($R^2 > 0.93$) are:

$$Y_1 = 3.6507X_1 + 34.086 \quad (1)$$

$$Y_2 = 3.4404X_2 + 22.742 \quad (2)$$

where Y_1 and Y_2 are ORP in mV and X_1 and X_2 are the CaO_2 concentration in mg/L.

Figure 1b shows that the pH changed quickly for the first 15 min (from pH 6.9 to about 8.9); while it remained at the safe range and there were no considerable changes afterwards. The results were similar for different concentrations of CaO_2 except for the case of adding 2 mg of CaO_2 /L, the pH increased to its maximum at 8.9,

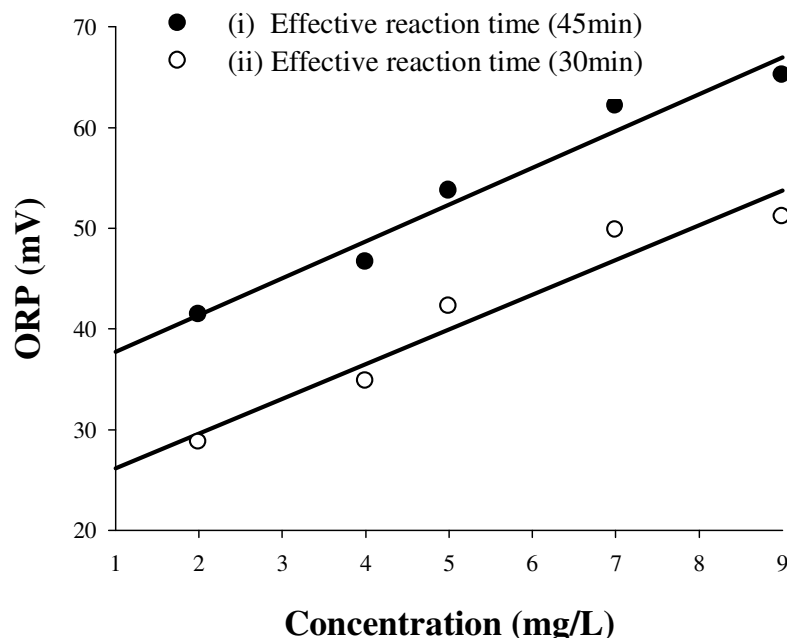


Figure 2. ORP achieved using effective CaO_2 concentration within reaction times (i) 30 min and (ii) 45 min.

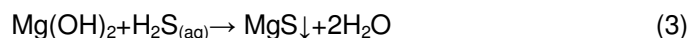
Table 2. Applied concentrations of the selected chemical and achieved optimum concentrations.

Chemical	Designed concentration (mg/L)	ORP changes Min – Max (mV)	pH changes Min - Max	Efficient reaction time (min)	Optimum concentration (mg/L)
CaO_2	2,4,5,7,9	-120 – 65	6.9 – 8.9	45	4.4
$\text{Mg}(\text{OH})_2$	4,6,8,10,12,14	-120 – 139	6.9 – 9.1	30	10.6
$\text{Ca}(\text{ClO})_2$	6,8,10,12,15,25	-120 – 132	6.9 – 8.8	30	13.3
FeCl_3	4,6,8,10,12,14	-120 – 143	6.9 – 6.7	30	8

but after 15 min of reaction it dropped to 8.7. The reason for the pH dropping after 15 min is difficult to explain as it has not been reported elsewhere. This may be due to the reaction of low concentration of CaO_2 with soluble compounds in sewage.

However, this does not have a significant effect as the pH safe range is 8.5 and above. The optimum values predicted by Equations 1 and 2 were 4.4 and 8.0 mg/L for CaO_2 concentration which would result in $\text{ORP} \geq 50$ mV during 45 and 30 min reaction time. Subsequent experiments using the predicted values confirmed that the safe value of ORP (50, 51, 53 mV) and pH 8.8 ± 0.1 were obtained. A similar investigation was conducted to determine the effective concentration of $\text{Mg}(\text{OH})_2$, $\text{Ca}(\text{ClO})_2$, and FeCl_3 chemicals individually. The results obtained were compared with those achieved from using CaO_2 in Table 2. Table 2 lists the designed concentrations, the range of ORP and pH, and the effective doses and time for each chemical. Magnesium hydroxide ($\text{Mg}(\text{OH})_2$) is used industrially as a non-hazardous alkali to neutralize acidic wastewaters and

raise the pH up to 10.0. This pH level can deactivate the bacteria responsible for the acid generation, since they cannot tolerate the high pH conditions (Jefferson et al., 2002). Since $\text{Mg}(\text{OH})_2$ is a strong base, increase in pH was observed. This chemical also raised the ORP value. Due to its low solubility in water, it mostly react with $\text{H}_2\text{S}_{(\text{aq})}$. The precipitation of $\text{H}_2\text{S}_{(\text{aq})}$ is shown in the following reaction.

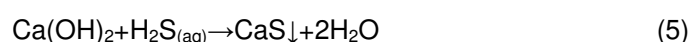
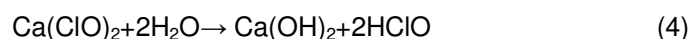


The ORP increased from minimum -120 mV to a maximum of 139 mV. The pH also raised from 6.9 to 9.1. The effective reaction time and obtained optimum concentration of $\text{Mg}(\text{OH})_2$ were estimated as 30 min and 10.6 mg/L, respectively. It is reported as a major disadvantage that $\text{Mg}(\text{OH})_2$ cannot remove the sulfide completely from the water. In such way, odor formation will occur if the pH is decreased in any consequent point in the sewage treatment process (Jefferson et al., 2002). Calcium hypochlorite ($\text{Ca}(\text{ClO})_2$) is identified as a strong

Table 3. The designed combined chemicals' concentrations.

Combination no.	Materials
1	2.0 mg/L FeCl ₃ and 2.5 mg/L Mg(OH) ₂
2	2.0 mg/L FeCl ₃ and 4.0 mg/L Mg(OH) ₂
3	3.0 mg/L FeCl ₃ and 2.5 mg/L Mg(OH) ₂
4	3.0 mg/L FeCl ₃ and 4.0 mg/L Mg(OH) ₂
5	2.0 mg/L FeCl ₃ and 3.0 mg/L Ca(ClO) ₂
6	2.0 mg/L FeCl ₃ and 5.0 mg/L Ca(ClO) ₂
7	3.0 mg/L FeCl ₃ and 3.0 mg/L Ca(ClO) ₂
8	3.0 mg/L FeCl ₃ and 5.0 mg/L Ca(ClO) ₂

oxidizer chemical that can be used for hydrogen sulfide oxidation (Zhang et al., 2008). Results also showed that Ca(ClO)₂ increased the ORP and pH values (Table 2). This chemical is soluble in water; thus, the chemical reaction (4) will take place in the mixture showing the production of a strong base (Ca(OH)₂). This strong base reacts with aqueous H₂S and oxidizes it (chemical reaction (5)). The reaction time was 30 min and the optimum concentration of this chemical was estimated as 13.3 mg/L. Researchers reported advantages for Ca(ClO)₂ as a strong oxidizer such as high removal efficiency for H₂S and other organic compounds as well as odor control (Tomar and Abdullah, 1994; Cadena and Petters, 1988); however, some drawbacks are associated with this reagent e.g. steel corrosion in concrete structures (Derek, 1995).



The wide use of iron salts mostly FeCl₃ in wastewater industries means that they have become an effective chemical in sulfide control (Tomar and Abdullah, 1994; Padival et al., 1995; Firer et al., 2008). FeCl₃ increased the ORP well (Table 2). However, results showed a little pH reduction which may be caused due to the acid phase production (chemical reaction (6)). Several pros and cons were reported for FeCl₃, some of which are: long-duration control, cost effectiveness, non-oxidation of any other odorous compounds apart from sulfides, effect on alkalinity, depletion of dissolved oxygen, and solids production (Poulton et al., 2002; Walton et al., 2003).

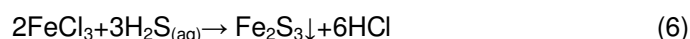
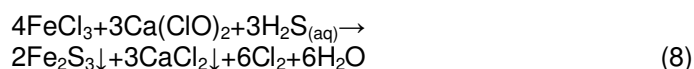
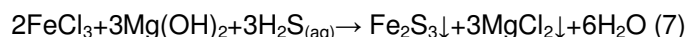


Table 2 summarizes the experimental results for the aforementioned chemicals. The safe range of ORP (≥50 mV) was experienced for all the chemicals, but at different concentrations. The lowest reagent concentration was achieved for CaO₂ but the sufficient reaction time was longer than others. However, higher concentrations of CaO₂ were examined aiming to achieve the same effective reaction time (30 min), and 8.0 mg/L

was found satisfactory, which is smaller than the optimum values for the other chemicals. Additionally, a poor pH increment by FeCl₃, and extremely high pH was observed when Mg(OH)₂ was utilized; however calcium compositions resulted in a satisfactory pH in the medium. The optimum concentration was predicted by the models generated (similar to Equations 1 and 2) from the experimental results and subsequent experiments approved their accuracy (as explained for CaO₂).

Addition of the combination of chemicals

A combination of chemical materials may result in lower dosage and higher efficiency in circumventing hydrogen sulfide concentration, and thus, alleviation in the drawbacks associated with chemicals is probable. As such, combination of FeCl₃ with Mg(OH)₂ or Ca(ClO)₂ to sewage was surveyed. As discussed, FeCl₃ reacts with sewage and produced HCl (acid compound) which acts as a pH decrement reagent. Mg(OH)₂ or Ca(ClO)₂ can react with the generated HCl and neutralize the solution (chemical reactions 7 and 8). Thus, it is expected that a proper combination of these chemicals regulate the resulted ORP and pH in the safe range.



Accordingly, the designed concentrations of these materials (25 and 37.5% of obtained optimum doses) were examined as shown in Table 3. For this experiment, FeCl₃ was added first, followed by the second chemical 15 min later. Figure 3a shows the ORP values versus time for the combination of FeCl₃ and Mg(OH)₂ with different combinations (1 to 4). It can be seen from the Figure that ORP increased sharply from -120 mV to the safe region within the first 15 min (to a maximum of 61 mV). After adding Mg(OH)₂, ORP rose more gently and reached its maximum at 90 min. It can be seen from Figure 3a that 30 min after adding Mg(OH)₂ (time point 45 min), no significant changes in ORP were observed. The pH vs. time is shown in Figure 3b and it can be concluded that adding FeCl₃ to the sewage samples caused a slight drop in the pH within the first 15 min. As mentioned previously, this little decrement might be due to production of HCl (acid phase).

However, addition of Mg(OH)₂ to the mixed samples within reaction time of 15 min (time point 30 min) increased the pH rapidly from about 6.8 to about 8.7. Results showed that combinations (1) and (2) resulted in pH increment to the safe range (that is 8.6 and 8.7, respectively). It can be concluded that the combination (1) is the most efficient combination resulting into ORP 78 mV and pH 8.6 within 30 min reaction time since it is using less chemicals. Similar outcomes resulted by

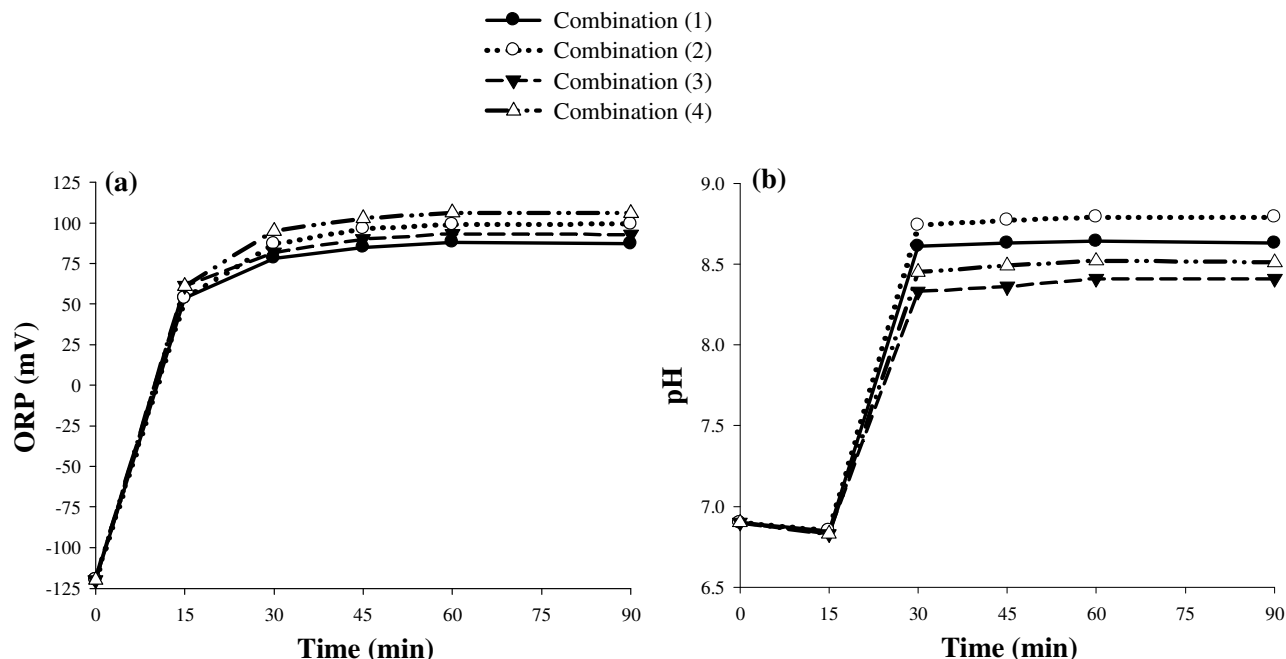


Figure 3. (a) ORP values and (b) pH values observed at different reaction times when different concentrations of FeCl_3 and $\text{Mg}(\text{OH})_2$ were added to sewage.

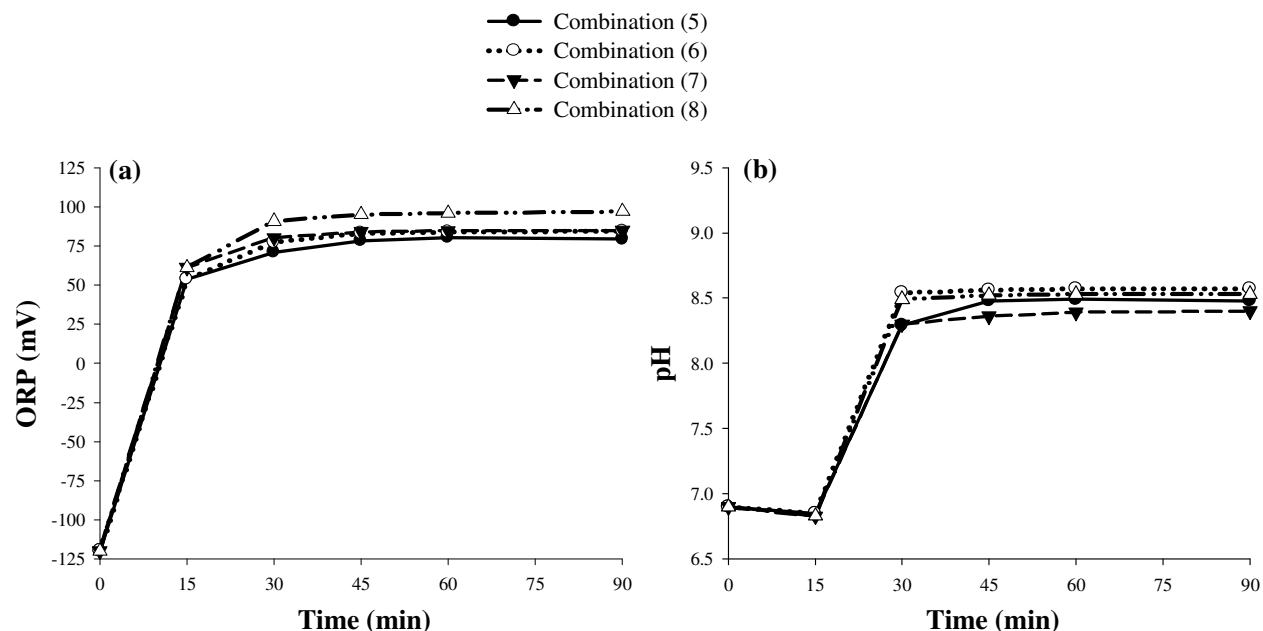


Figure 4. (a) ORP values and (b) pH values observed at different reaction times when different concentrations of FeCl_3 and $\text{Ca}(\text{ClO})_2$ were added to sewage.

adding combinations of FeCl_3 - $\text{Ca}(\text{ClO})_2$ to the sewage samples (combinations 5 to 8). Figure 4a demonstrates that the designed concentrations of FeCl_3 - $\text{Ca}(\text{ClO})_2$ increased the ORP from a minimum of -120 mV to a maximum of 91 mV at the first 30 min of addition (time point 45 min), and marginal changes in ORP were

observed onwards (ORP increased to maximum 97 mV). Figure 4b illustrates alteration in pH versus time, and it can be seen that only combinations (6) and (8) were successful in raising the pH to the safe range. Combination (6) raised the pH from 6.9 to a maximum value of 8.6, while combination (8) shifted the same initial

Table 4. Cost estimation for daily treatment of sewage using efficient dose of chemicals.

Chemical	Effective reaction time (min)	Obtained effective concentration (mg/L)	Price (US\$/g)	Cost (US\$/day)	Rate
CaO ₂	45	4.4	0.056	118	2
CaO ₂	30	8.0	0.056	215	5
Mg(OH) ₂	30	10.6	0.050	254	6
Ca(ClO) ₂	30	13.3	0.048	306	7
FeCl ₃	30	8	0.047	180	4
*FeCl ₃ and Mg(OH) ₂	30	2 and 2.5	0.047 - 0.050	105	1
**FeCl ₃ and Ca(ClO) ₂	30	2 and 5	0.047 - 0.048	160	3

*Combination (1), **Combination (6).

pH to 8.5. After the efficient reaction time (30 min) pH remained steady. Thus, combinations (6) and (8) could satisfy H₂S circumvention within 30 min.

However, considering less chemical consumption, the combination (6) was selected as the most efficient set leading to ORP 77 mV and pH 8.5. No considerable changes in ORP and pH were observed for the control sample with no chemical addition during the test procedure. Besides, as the temperature is quite significant in corrosion process and odor release in sewer networks, temperature was monitored throughout the experiments and no considerable change was detected due to adding chemicals. The average of initial hydrogen sulfide concentration in the sewage samples was 4 mg/L. Analyses revealed that combinations (1) and (6) decreased the hydrogen sulfide concentration higher than 95%. Using 4.4 and 8.0 mg CaO₂/L, hydrogen sulfide removal greater than 90% was measured for reaction times of 45 and 30 min, respectively.

Cost evaluation

A wide diversity in treatment costs reflects the unique aspects of each chemical that may be warranted for a special program in which economics is the dominant selection criterion. Therefore, chemical costs should be estimated for a real scale of wastewater system, and Equation (3) helped to analyze the estimations for each selected chemical with their effective doses;

$$C_{\text{chemical}} = \left(\sum_{i=1}^n (M_i \times P_i) \right) \times Q_N \quad (3)$$

Where C_{chemical} is cost of the chemicals (US\$/day), M_i represents dose of the chemical (mg/L), P_i is unit price of the chemical (US\$/g), and Q_N is the sewage discharge (field study network outlet discharge was estimated by approximately 480 m³/day). It is apparent from the equation that costs are dependent on dosage of chemicals and unit prices in the Malaysian market. Table

4 lists the estimated costs for the efficient dose of chemicals achieved in this work based on the market material prices provided by the chemical supplier (2008). It can be seen from Table 4 that the least cost was obtained when FeCl₃ and Mg(OH)₂ were used (combination 1). It is about 12 and 53% more cost effective than using CaO₂ alone and FeCl₃ and Ca(ClO)₂ (combination 6), respectively.

Conclusions

Different chemicals and their combinations were added to sewage at various concentrations for inhibition of dissolved H₂S in sewer systems. Experiments applying chemicals individually showed CaO₂ resulted in lower concentration of chemical rather than FeCl₃, Mg(OH)₂, and Ca(ClO)₂ to satisfy ORP and pH ranges in the medium. Addition of 4.4 mg/L CaO₂ with 45 min reaction time was the most economical case amongst individually added chemicals. However, results obtained from application of chemical combinations indicate that the 2 mg/L FeCl₃ plus 2.5 mg/L Mg(OH)₂ and also 2 mg/L FeCl₃ plus 5 mg/L Ca(ClO)₂, resulted in the safe ranges of ORP and pH with lower operational costs rather than utilizing chemicals individually within 30 min reaction time. Furthermore, reduction for dissolved H₂S in sewage at a rate higher than 95% was obtained through application of these chemical combinations.

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