Effect of pretreatment by coagulation on stabilized landfill leachate during anaerobic treatment

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Abstract: Landfill leachate contains both larger fractions of higher molecular weight organic materials and heavy metals. The study investigated the effect of coagulation as pretreatment during anaerobic treatment process on stabilized landfill leachate. Anaerobic studies were done on the raw stabilized leachate and on the pretreated leachate, where coagulation was used as a pre-treatment process for leachate. Landfill leachate samples were collected from India's largest and oldest landfill i.e. Deonar landfill, Mumbai. The leachate was characterized by high TS (10,910 mg/l), COD (2,300 mg/l), BOD (238 mg/l), TOC (716 mg/l) and low BOD/COD ratio (0.10). FeCl₃ and PACl were used as coagulants for the coagulation process. The highest removal efficiency obtained by FeCl₃ and PACl for COD was 53 and 59%, respectively. The highest removals of TOC were 52 and 57% by FeCl₃ and PACl, respectively. The optimum pH and dose for FeCl₃ was found to be 8 and 7 g/l, respectively. The best pH and dose for PACl was found to be 6 and 10 g/l, respectively. Anaerobic batch reactor treatment was applied as secondary treatment on pre-treated leachate and also on raw leachate. Anaerobic batch experiment was performed for batch time 72 h with solids retention time of 10 days. When anaerobic batch treatment
was done on raw leachate, it showed constant 35% TOC and 38% COD removal after 40 h. In the case of anaerobic batch experiment after coagulation, the overall TOC and COD removal was found to be 70 and 72%, respectively.

Subjects: Environmental Health; Pollution; Water Engineering; Water Science

Keywords: anaerobic biological process; coagulation; chemical oxygen demand; landfill leachate

1. Introduction

At present the population of world has reached nine billion. With the increasing population, solid waste generation is also increasing constantly. Economically, landfilling is one of the best methods for municipal solid waste disposal. Other volume reduction processes are also available, for e.g. incineration and composting, but they produce waste fractions (ashes and slag) which ultimately need landfilling. Despite the evolution of landfill technology, the generation of contaminated leachate remains an inevitable consequence of the practice of waste disposal in landfills (Tatyana, Bautista, Chairez, Cordova, & Rios, 2008). When the refuse decomposed in landfills gets moisture through rainwater or by other means, contaminants present in it get dissolved and flow out of the landfill in the form of leachate. Landfill leachate contains high BOD, COD, nitrogen compounds and many heavy metals with various types of organic matter. The composition of refuse has changed over the years due to changes in life styles which is now producing stronger landfill leachate. This high strength contaminated leachate contaminates the nearby groundwater and surface water. Generation of landfill leachate depends on design and operation of landfills. Before 1975, the design of landfills was not having any system to control or check the flow of landfill leachate as nobody thought about the contamination of the groundwater because of landfill leachate. Today the landfill leachate has taken the attention of researchers. Characteristics of landfill leachate vary from one to another, and they also change with space and time within the same landfill. It depends mainly on the variations in climate, hydrogeology and waste composition (Keenan, Steiner, & Fungaroli, 1984). Landfill engineering focuses on reducing leachate production, collection and treatment prior to discharge (Farquhar, 1989). So effective treatment technologies are required to treat landfill leachate for stopping the contamination of groundwater and to save the people living nearby from diseases. As the leachate quality varies from one landfill to other the treatment technology becomes site specific. So the general treatment chain cannot be followed on every landfill leachate. As the landfill leachate contains both larger fraction of higher molecular weight organic material and heavy metals, neither treatment by biological nor by physicochemical processes can give high removal efficiencies. Therefore, a combination of physicochemical and biological treatment is required to remove both organic matter and heavy metals. Recirculation, activated sludge, sequencing batch reactors, aerobic lagoons and constructed wetlands are biological treatment methods and oxidation, coagulation/flocculation, activated carbon, microfiltration, ultrafiltration and reverse osmosis are physical/chemical techniques used for the treatment of leachate (Bressi & Favali, 1997; Luning & Notenboom, 1997; Van & Roncken, 1997). Some of the adsorption processes also used polishing processes after the biological treatment to remove suspended solids, turbidity, dissolve metals and organics (Amosa, 2016; Amosa, Jami, Alkhatib, Jimat, & Muyibi, 2015; Amosa et al., 2016).

Under controlled conditions, waste is decomposed in layers and compaction is done to convert it into stabilized form. The decomposition, stabilization and extraction of pollutants from a landfill depends upon the following factors: composition of the wastes, degree of compaction, amount of moisture present, presence of inhibiting materials, rate of water movement, and temperature (Qasim & Chiang, 1994). Landfill leachate comes to the surface due to infiltration of water from waste which contains dissolved contaminated pollutants. Seepage of this leachate into the ground contaminates the groundwater with different types of recalcitrant compounds. There are different types of landfills depending on their design and infrastructure as mentioned in Table 1 and there are different criteria to classify leachate for e.g. old, medium and young as shown in Table 2.
The objective of this study was to pre-treat the landfill leachate by coagulation process and then perform anaerobic batch reactor as secondary treatment. The anaerobic batch reactor process was applied on the raw leachate as well. Leachate samples were collected from India’s largest and oldest landfill Deonar, Mumbai.

Coagulation is a physicochemical process which is used to remove fine suspended and colloidal particles from the wastewater. Removal of organic matter from wastewater by coagulation reduces...
BOD and COD levels. The addition of a coagulant causes the charge destabilization of the colloids followed by floc formation (Weber, 1972). Settlement of these flocs removes colloids. Charge reversal can occur if dose of coagulant is more than the optimum dose. It leads to the re-stabilization of colloids which increases the turbidity. First high speed stirring is done to increase the particle–particle interactions which are called perikinetic phase. For building the coarse flocs slow speed mixing is done which is called as orthokinetic phase. Then the mixing is stopped for the settlement of flocs. Comstock, Boyer, Graf, and Townsend (2010) collected stabilized leachate from four landfills for a total of seven leachate samples, and samples were coagulated using ferric sulphate. The optimum pH was found to be 8.01 and optimum dose of coagulant ferric sulphate as 0.06 g/l. 28% COD got removed at the optimum dose. Pi, Li, Wan, and Gao (2009) studied biodegradability enhancement of landfill leachate using air stripping followed by coagulation. The single coagulation process increased BOD/COD ratio by 0.089 with the FeCl₃ dosage of 570 mg/l at pH 7.0. Only 38% of COD removal efficiency was observed at the optimum pH of 6.3 and optimum dose of 0.7 g/l. Li, Hua, Zhou, Zhang, and Li (2010) performed coagulation/flocculation experiments, using coagulant ferric chloride to study the optimum conditions for the removal of COD, SS and turbidity. The COD was removed at optimum dose and pH. The optimum pH for the tested coagulants was 5.5–6.0. The optimum dose of the coagulant came out to be 1.7 g Fe³⁺/l.

Anaerobic batch reactors are now widely used in the treatment of wastewater because they have better retention of biological solids and improved process control than continuous reactors (Timur & Özturk, 1999). It has basic five operations (fill, react, settle, decant, idle) in the same reactor. Timur and Özturk (1999) treated raw leachate (initial COD 16,000–20,000 mg/l) in anaerobic sequencing batch reactor (ASBR). 64–85% COD removal was observed at different volumetric and specific loading rates (0.4–9.4 g COD/l/day and 0.17–1.85 g COD/g VSS/day, respectively). Kennedy and Lentz (2000) studied ASBR and upflow anaerobic sludge blanket (UASB) reactors by varying organic loading rates from 0.6 to 19.7. At low and intermediate organic loading rates both the treatment technologies, ASBR and UASB gave almost similar results. Removal efficiency of the ASBR was from 71 to 92% at hydraulic retention times (HRT) of 24, 18 and 12 h with dilute to concentrated feed. Laitinen, Luonsi, and Vilen (2006) compared sequencing batch reactor and submerged membrane bioreactor. Average BOD, and COD in influent were 1,240 and 2,300 mg/l, respectively. HRT for SBR and MBR varied from 4 to 9 days and 2 to 5 days, respectively. SRT for SBR and MBR varied from 10 to 40 days and 35 to over 60 days, respectively. 6.6 to 10 g/l and 7.3 to 9.3 g/l sludge concentration maintained in SBR and MBR, respectively. It was observed that SBR has COD of 500 mg/l and MBR has COD of 340 mg/l in effluent and it was concluded that both of the technology has almost similar results. COD concentration in effluent of SBR was high as some sludge escaped due to some disturbances in the process.

2. Materials and methods
Leachate was collected in 40 litres capacity cans and then stored in the cold store room at 4°C. Prior to its use for the experiments, it was taken out from the cold storage and allowed to attain the room temperature.

2.1. Chemicals
All the chemicals used were of analytical grade. Chemicals such as concentrated sulphuric acid (98%), ferrous ammonium sulphate, potassium dichromate, nitric acid, ferric chloride, glycerol, sodium chloride, ammonium chloride, ammonium hydroxide, calcium chloride, sodium bicarbonate were purchased from Merck chemicals, Mumbai with 99.99% purity. Indicators such as phenolphthalein, ferroin and methyl orange used during the experiments were also purchased from the Merck chemicals, Mumbai.

2.2. Analytical methods
Most of the parameters such as COD, BOD₅, chlorides, total solids, total suspended solids and total dissolved solids were determined using the procedures given in American Public Health Association (APHA) handbook (American Public Health Association, American Water Works Association, and Water Environment Federation, 2005). COD was measured by closed reflux method using HACH COD...
Digestor (DRB 200, USA). Shimadzu total organic carbon analyser (TOC-VCSH, Japan) was used for TOC analysis. pH of the leachate was measured by the Orion digital pH meter (3 Star, Singapore).

3. Experimental study

3.1. Coagulation
Coagulation process was performed in a Jar test apparatus (Trishul jar apparatus, Mumbai). The assembly was equipped with a stirring mechanism for six beakers (1 l capacity) at the same time. Each beaker was filled with 500 ml of leachate and desired amount of coagulant was added. The jar contents were rapidly mixed at 150 rpm for 3 min to allow the maximum contact between the coagulant and the wastewater. After the rapid mixing, slow agitation at a stirring speed of 40 rpm was done for 20 min to generate coarse flocs. Once the stirring was stopped, the flocs were allowed to settle for 30 min. After settling 10 ml sample from the supernatant was withdrawn and filtered through Whatman (Grade = φ; pore size = 11 μm) filter paper before determining COD and other parameters.

4. Anaerobic batch reactor

4.1. Acclimation of mixed culture
Acclimation study of mixed culture from landfill leachate was done by taking 10 ml of landfill leachate and 90 ml of nutrient broth. It was done in a conical flask by making it air tight and then it was kept at a temperature of 35°C for 24 h. Then in the next step landfill leachate quantity was taken 20 ml, 80 ml nutrient broth and 10 ml culture from the previous conical flask. So by doing same procedure repeatedly, increasing the quantity of leachate and decreasing the nutrient broth, it was acclimatized up to 90% leachate and 10% nutrient broth. And then the culture was grown on large scale.

4.2. Acclimation of sludge with landfill leachate
Acclimation was done in the anaerobic batch reactor (Figure 1) which was fully air tight and nutrient was purged before every process. Sludge was taken from anaerobic food waste digester plant in

Figure 1. Set-up for acclimation of sludge.
Bhabha Atomic Research Centre Canteen, Mumbai. The mixed liquor suspended solids and volatile suspended solids were 23,000 and 12,000 mg/l, respectively. Sequential acclimation of mixed culture with landfill leachate was performed. Firstly 800 ml sludge, 100 ml leachate, 100 ml acclimatized mixed culture and adequate amount of nutrient solution with trace elements given in Table 3. The reactor was kept for 48 h and growth is continuously observed. Then in next step quantity of leachate was increased and sludge quantity was decreased. Growth of culture was seen maximum at a mix of 300 ml sludge and 700 ml leachate. Then the culture was grown on large scale for performing the experiments.

4.3. Batch study using anaerobic batch reactor

Anaerobic sequencing batch reactor (Figures 2 and 3) was made of acrylic sheet. The height of the reactor was 18 cm and diameter 15 cm which gave a total volume of 3.18 l. The working volume of

| Nutrients                  | g/l   |
|----------------------------|-------|
| K₂HPO₄                     | 0.06  |
| KH₂PO₄                     | 0.04  |
| MgSO₄·7H₂O                 | 0.5   |
| FeCl₃·6H₂O                 | 0.71  |
| ZnSO₄·7H₂O                 | 0.0001|
| CuSO₄·5H₂O                 | 0.0001|
| MnCl₂·2H₂O                 | 0.004 |
| (NH₄)₆MO₇O₂₄               | 0.00011|
| CaCl₂·H₂O                  | 0.1   |
| CoCl₂·6H₂O                 | 0.2   |
| Al₂(SO₄)₃·16H₂O            | 0.055 |
| H₃BO₃                     | 0.15  |

Source: Kundu et al. (2012).
2 l comprised of 600 ml sludge and 1,400 ml leachate. The reactor was made totally air tight with the stirrer in middle. It had three openings for taking out the effluent at 2, 4 and 6 cm from the bottom. Two openings were given on top, one for purging nitrogen gas and the other for removing biogas. Pipe used for removing biogas was submerged in the water so that air cannot enter in the reactor. Landfill leachate pH was adjusted to 6 for maximum bacterial growth. External stirrer machine was utilized with air tight mechanism for stirring. 10 ml samples were drawn at equal intervals and filtered with 0.2 μ membrane filter. Then it was analysed for total organic carbon.

5. Results and discussion

5.1. Characteristics of landfill leachate

Leachate samples were tested for various parameters (like pH, TOC, COD, BOD, TS, TSS, TDS, alkalinity and turbidity) given in Table 4. The COD of leachate sample was found to be 2,300 mg/l which suggests that it is old/stabilized leachate as it exceeds the landfill leachate discharge standards given by CPCB. The BOD/COD ratio was also found to be very less which signifies that it has very less biodegradable organics and may have high concentration of heavy metals. So ICP-AES of leachate sample was performed to estimate the concentrations of heavy metals in leachate. Total solids (TS) concentration was found to be around 10,910 mg/l out of which almost 70% were in suspended form while rest (3,400 mg/l) were in form of total dissolved solids. BOD/COD ratio was around 0.1 initially and was tried to increase by using Fenton process but it also gave similar removal efficiency after anaerobic treatment. BOD/COD ratio was increased from 0.1 to 0.18 after Fenton process.
6. Coagulation of landfill leachate using PACl and FeCl3

6.1. Variation of pH for best removal
Poly-aluminium chloride and ferric chloride was used as coagulants for performing coagulation for pre-treatment of leachate. pH was varied from 2 to 12 with increment of 2 as shown in Figures 4 and 5. The pH was adjusted by NaOH and H2SO4. Fixed dose of 4 g/l was added in each beaker which contained 500 ml leachate sample with adjusted pH. The highest COD reduction of about 53% was obtained at a pH of 6.0 by PACl as shown in Figure 4. The isoelectric point of PACl is at about neutral pH. The maximum COD reduction by ferric chloride was 53% at a pH of 8.0 (Figure 5). The isoelectric point of amorphous ferric hydroxide is about pH 8. Positively charged polymers dominate below the isoelectric point which destabilizes the negatively charged colloids by charge neutralization (Weber, 1972).

Table 4. Physical and chemical characteristics of landfill leachate

| Parameter          | Method                              | Concentration |
|--------------------|-------------------------------------|---------------|
| Ph                 | Digital pH meter                    | 8.0–8.5       |
| Colour             |                                     | Dark brown    |
| Turbidity          | Turbidity meter                     | 177 NTU       |
| BOD                | Modified Winkler’s method           | 238 ± 30 mg/l |
| COD                | Standard closed reflux method       | 2,304 ± 152 mg/l |
| TOC                | TOC analyser                        | 680 ± 45 mg/l |
| BOD/COD            |                                     | 0.10          |
| Total solids       | Filtration and evaporation method   | 10,910 ± 152 mg/l |
| Total dissolved solids | Filtration and evaporation method   | 3,400 ± 61 mg/l |
| Total suspended solids | Filtration and evaporation method   | 7,510 ± 105 mg/l |
| Chlorides          | Argentometric method                | 349.53 mg/l   |
| Total hardness     | EDTA titrimetric method             | 1,051.52 mg/l |
| Total alkalinity   | Titration method                    | 12,450 mg/l   |
| Fe                 | ICP-AES                             | 1.06 mg/l     |
| Na                 | ICP-AES                             | 1,412.43 mg/l |
| Cd                 | ICP-AES                             | 0.09 mg/l     |
| Ag                 | ICP-AES                             | 0.346 mg/l    |
| Al                 | ICP-AES                             | 6.64 mg/l     |
| Mn                 | ICP-AES                             | 0.035 mg/l    |
| Ba                 | ICP-AES                             | 0.480 mg/l    |
| B                  | ICP-AES                             | 7.555 mg/l    |
| K                  | ICP-AES                             | 1,107.2 mg/l  |
| Ca                 | ICP-AES                             | 167.8 mg/l    |
| Cr                 | ICP-AES                             | 0.024 mg/l    |
| Sr                 | ICP-AES                             | 2.313 mg/l    |
| Mg                 | ICP-AES                             | 118.52 mg/l   |
| Zn                 | ICP-AES                             | ND            |
| Li                 | ICP-AES                             | ND            |
| Co                 | ICP-AES                             | ND            |
| Ni                 | ICP-AES                             | ND            |
| Cu                 | ICP-AES                             | ND            |
| Pb                 | ICP-AES                             | ND            |

Note: ND means not detected.
COD removal decreases after the isoelectric point because at points greater than pH 8 there is formation of less positively charged neutral colloids like Fe(OH)$_2^+$ and Fe(OH)$_3$.

**6.2. Variation of dose for best removal**

At best pH, dose of coagulant PACl and FeCl$_3$ was varied from 1 to 12 g/l with increment of one in different beakers. Highest removal of COD was coming with 10 g/l with almost 59% removal efficiency by using PACl coagulant as shown in Figure 6. Figure 8 shows that pH decreased from 8 to around 4.0 as the coagulant dose increased from 1 to 12 g/l. Higher COD removal percentages were observed when the final pH of the solution was around 7. At best pH, dose of coagulant FeCl$_3$ was varied from the 1 to 11 g/l with increment of one in different beakers. Highest removal of COD was observed with 7 g/l with almost 51% removal efficiency shown in Figure 7. At lower doses ranging from 1 to 4 g/l the COD removal was less than 30%. Figure 9 shows that pH decreased from 8 to around 4 as the coagulant dose increased from 1 to 11 g/l. pH diagram of the coagulant suggests that ferrous ions present...
in the solution may be in form of $[\text{Fe}_2(\text{OH})_2]^{4+}$, $[\text{Fe}(\text{H}_2\text{O})_6]^{2+}$, $[\text{FeOH}^+]$ and $[\text{Fe(OH)}_3^{-}]$. $[\text{Fe}_2(\text{OH})_2]^{4+}$ ion species predominates compared to others at pH around 7 (Weber, 1972). The positively charged ions from the hydrolysis of the coagulant bring about the charge neutralization of the negatively charged particles, thus leading to the formation of flocs which settle down. The flocs formed get entrapped in the ferrous hydroxide precipitate and settle down leading to reduction of COD concentration.

Polyaluminium chloride gave more COD removal efficiency of leachate than ferric chloride. So it was used as a pretreatment for further studies of anaerobic treatment.
6.3. Anaerobic batch reactor

Batch study was done in AnBR on the (i) raw leachate, (ii) after coagulation treatment at the HRT of 72 h and SRT of 10 days. A fraction (1/10) of the culture was removed from the reactor every day to adjust the sludge age to 10 days (Neczaj, Kacprzak, Lach, & Okoniewska, 2007; Neczaj, Okoniewska, & Kacprzak, 2005).

Anaerobic batch reactor experiment was performed for hydraulic retention time of 72 h with sludge retention time of 10 days. When anaerobic batch treatment was done on raw leachate, it showed constant 38% removal after 56 h (Figure 10). In case of AnBR after coagulation (Figure 11), COD removal was 18% after 56 h. The less removal efficiency of COD in anaerobic batch reactor experiment after pre-treatment may be due to removal of organics after the coagulation. So there is less availability of degradable compounds in the leachate left. Actually the old leachate comes after the anaerobic degradation, hence, its COD has most of the non-biodegradable compounds and leads to less removal efficiency of anaerobic treatment.

7. Conclusions

The performance of coagulation was judged as a pretreatment of landfill leachate. Coagulant PACI showed 59% COD and 57% TOC removal with a dose of 10 g/l and pH of 6. So the COD and TOC of the effluent were 943 and 308 mg/l, respectively. Coagulant FeCl₃ showed 53% COD and 52% TOC removal with a dose of 7 g/l and pH of 8. Finally the effluent had 343 mg/l of TOC and 1,081 mg/l of COD. Anaerobic batch reactor showed 35 and 38% removal of TOC and COD, respectively, after 40 h in a batch study of 72 h and 10 days SRT on raw leachate. 70% of TOC and 72% of COD removed by combined treatment of coagulation from polyaluminium chloride followed by ASBR in 56 h. It is...
concluded that anaerobic treatment on stabilized leachate does not give high removal efficiency due to low BOD/COD ratio. It does not have any scope of biological treatment even after giving pretreatment. But there is scope to treat low BOD/COD ratio wastewater with chemical processes like adsorption process.

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