Sulfide, Phenols and Chromium (VI) Removal from Landfill Leachate and Domestic Wastewater by ZELIAC, Zeolite and Activated Carbon Augmented Sequencing Batch Reactor (SBR)

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ABSTRACT
Leachate is created, while water penetrates through the waste in a landfill, carrying some forms of pollutants. Sequencing Batch Reactor (SBR) is one of the biological methods for treating wastewater. The current research studied the removal of sulfide, phenols and hexavalent chromium from landfill leachate and domestic wastewater using Powdered ZELIAC (PZ), Powdered Activated Carbon (PAC) and powdered zeolite (PZE) augmented SBR process. The ZELIAC is a new adsorbent, which consists of zeolite, activated carbon, limestone, rice husk ash and Portland cement. Based on batch experiments and optimization experiments, powdered adsorbents dosage (PZ, PZE and PAC dosages = 3 g L⁻¹), settling time (90 min) and leachate-to-wastewater mixing ratio (20%; v/v) were fixed. The results indicated that the PZ-SBR showed higher performance in removing phenols, sulfide and hexavalent chromium compared with SBR, PZE-SBR and PAC-SBR. And also the PZE-SBR showed higher performance in removing Cr (VI) compared with SBR and PAC-SBR. In the PZ-SBR, the removal efficiencies for phenols, sulfide and Cr (VI) were 67.71, 74.13 and 79.24%, respectively.

Key words: Hexavalent chromium, landfill leachate, wastewater, phenols, sequencing batch reactor, ZELIAC

INTRODUCTION
Sanitary landfill leachate is a highly and complex polluted wastewater. Its quality is the result of biological, chemical and physical processes in landfills combined with the specific waste composition and the landfill water regime (Stegmann et al., 2005). Accumulated municipal solid wastes in landfills decompose by a combination of physical, chemical and biological processes (Kiayee, 2013). Landfill leachate could be a main foundation of water contamination (Mojiri et al., 2013). Landfill leachate is formed by the percolation of rainwater through domestic refuses. The most serious features of leachate are connected of the high concentrations of some contaminants. Some of these contaminants are metals, sulfide and phenols. Many different methods have been investigated to treat leachate generated from municipal sanitary wastes. Since, leachate contains both biodegradable and non-biodegradable components, the studied methods can be divided into...
two major groups: biological and physical/chemical treatments (Pouliot, 1999). Sequencing batch reactor is a kind of biological method for wastewater treatment. As it can be seen in the literature, in some researches, adsorbents, such as activated carbon were added to activate sludge and SBR to enhancement of the biological treatment of landfill leachate (Mojiri et al., 2014; Aziz et al., 2011a, b, 2012; Foo and Hameed, 2009).

The presence of heavy metals in wastewaters has become a serious environmental problem in the last decades. Chromium is a metal used in various industrial processes (e.g. textile dying, tanneries, metallurgy, metal electroplating and wood preserving); therefore, large quantities of chromium have been discharged into the environment, especially in the past (Gheju and Pode, 2010). In wastewater mostly the chromium is found in two forms, one is hexavalent and the other is trivalent, whereas the hexavalent form is more common and hazardous to biological activities (Talokar, 2011). To meet environmental regulations, effluents or heavy metals contaminated water must be treated before discharge. Chemical precipitation, oxidation/reduction, mechanical filtration, ion exchange, membrane separation and carbon adsorption are among the variety of treatment processes widely used for the removal of toxic heavy metals from the waste streams (Boddu et al., 2003).

Another contaminant in the landfill leachate is phenols. Phenol is the priority pollutant since it is toxic and harmful to organisms even at low concentrations (Dakhil, 2013).

Another pollutant is sulfide. The corrosive properties of sulfide are apparent in the damage done to concrete walls of reactors, sewer systems and steel pipelines. It also inhibits the methanogenesis process. Soluble sulfide ranging from 50-100 mg L\(^{-1}\) can be tolerated in anaerobic treatment with little or no acclimation. Sulfide has high oxygen demand of 2 mols O\(_2\)/mol sulfide and causes depletion of oxygen in water (Midha and Dey, 2008).

A number of studies (Talokar, 2011; Boddu et al., 2003; Aggarwal et al., 1999) have verified that using biological methods and adsorbents can remove a large amount of metals from wastewater and landfill leachate and also some studies were conducted for phenol removal by biological technique and using adsorbents (Dakhil, 2013; Hameed and Rahman, 2008; Jadhav and Vanjara, 2004; Roostaei and Tezel, 2004; Molva, 2004).

This research evaluates the performance of Sequencing Batch Reactor (SBR) with Powdered ZELIAC (PZ), powdered zeolite (PZE) and Powdered Activated Carbon (PAC) in removing phenols, sulfide and chromium from Sungai Petani landfill leachate and domestic wastewater from Bayan Baru Wastewater Treatment Plant in Malaysia. In addition, this research has introduced a novel inexpensive adsorbent, i.e., ZELIAC.

**MATERIALS AND METHODS**

**Landfill leachate sampling:** Leachate samples were collected from the Sungai Petani landfill site from June, 2012 to March, 2013. The landfill site (geographical coordinates, 05°43’ N and 100°29’ E) is located in Kedah, Malaysia. Sungai Petani landfill receives nearly 350-400 t of solid wastes daily, measured using a weight bridge. This open dumping site has been actively used since, 1990. The total landfill area of Sungai Petani is 11.24 ha (Mojiri et al., 2014). Samples were immediately transferred to the laboratory after collection and maintained in a cold room at 4°C to minimize the biological and chemical reactions (Aziz et al., 2011b). Table 1 shows the characteristics of the samples. To determine the risks of leachates to the environment, the obtained parameter values were compared with the 2009 Regulations of the Malaysia Environmental Quality Act of 1974 (Environmental Quality Council, 2009).
Table 1: Characteristics of landfill leachate, domestic wastewater and sludge

| Parameters               | Leachate | Wastewater | Standard discharge limit for leachate* |
|--------------------------|----------|------------|---------------------------------------|
| Temperature (°C)         | 28.5     | 27.9       | 40                                    |
| pH                       | 7.65     | 6.91       | 6-9                                   |
| EC (ms cm⁻¹)             | 3.73     | 1.13       | -                                     |
| Colour (Pt. Co)          | 1261     | 6.00       | 100                                   |
| BOD₅ (mg L⁻¹)            | 269.0    | 64.2       | 20                                    |
| COD (mg L⁻¹)             | 726      | 116        | 400                                   |
| Nitrite (mg L⁻¹ NO₂-N-HR) | 50.18   | 9.26       | -                                     |
| NH₃-N (mg L⁻¹)           | 417.0    | 149.0      | 5.00                                  |
| Sulphide (mg L⁻¹)        | 0.300    | 0.600      | 0.50                                  |
| Total iron (mg L⁻¹)      | 7.23     | 2.65       | 5.00                                  |
| Total manganese (mg L⁻¹) | 1.18     | 0.65       | 0.20                                  |
| Total nickel (mg L⁻¹)    | 5.44     | 0.31       | 0.20                                  |
| Chromium VI (mg L⁻¹)     | 0.21     | 0.17       | 0.20                                  |
| Phenols (mg L⁻¹)         | 1.84     | 0.10       | 0.001                                 |

*Environmental Quality (Control of Pollution from Solid Waste Transfer Station and Landfill) Regulations 2009, under the laws of Malaysia—Malaysia Environmental Quality Act 1974.

Domestic wastewater and activated sludge sampling: The activated sludge and domestic wastewater were collected from the Bayan Baru wastewater treatment plant in Penang, Malaysia. Table 1 shows the characteristics of the activated sludge and wastewater.

Reactor characteristics: Six beakers of 2000 mL each were used throughout the study. Each beaker had a working volume of 1200 mL, an inner diameter of 113 mm and a height of 200 mm. A magnetic stirrer placed at the bottom of the reactors was used to mix the media. The experiments were carried out at room temperature and an air pump (YASUNAGA, Air pump Inc.; voltage: 240 V, frequency: 50 Hz, input power: 61 W, model: LP-60A, pressure: 0.012 MPa, air volume: 60 L min⁻¹; Serial no.: 08110014, made in China) was used to provide the reactors with air. The air flow speed was manually regulated using an air flow meter (Dwyer Flow meter, Model: RMA-26-SSV).

Sludge acclimatization: According to the study of Aziz et al. (2011b), 120 mL (10%) of the collected landfill leachate was mixed with approximately 1080 mL of the activated sludge (90%). After the termination of the reaction and the settling phases, 120 mL of the supernatant was withdrawn. In another cycle, an additional 120 mL of the raw leachate was added to the reactor. This procedure was sustained for at least 10 day to allow the system to adapt to the experimental condition. The adjusted sludge was later used as seed in the SBRs.

ZELIAC preparation: To prepare the ZELIAC, zeolite, activated carbon, limestone, rice husk ash and Portland cement were ground, passed through a 300 µm mesh sieve and then mixed. The mixture was then evenly poured in the mold after the addition of water. After 24 h, the materials were removed from the mold and soaked in water for three days for the curing process. After allowing the materials to dry within two days, they were crushed and passed through a sieve. The features of the powdered ZELIAC with the autosorb (Quantachrome AS1wintm, version 2.02) test. The result of the XRF analysis of ZELIAC is shown in Table 2. The Zeolite and activated carbon are present in the ZELIAC; thus ZELIAC can act as both adsorbent and ion exchanger. In this study, Powdered ZELIAC (PZ) with a size ranging from 75-150 µm was used as adsorbent in the PZ-SBR (Aziz et al., 2011a).

Characteristics of zeolite and activated carbon: Characteristics of powdered zeolite and powdered activated carbon with the autosorb (Quantachrome AS1wintm, version 2.02) test have
Table 2: XRF results for ZELIAC

| Compounds/elements | Composition (%) |
|--------------------|-----------------|
| C                  | 14.350          |
| CaO                | 32.401          |
| SiO₂               | 42.002          |
| Al₂O₃              | 7.300           |
| Fe₂O₃              | 1.502           |
| K₂O                | 1.005           |
| MgO                | 1.000           |
| Na₂O               | 0.100           |
| P₂O₅               | 0.030           |
| SO₃                | 0.030           |
| Others             | 0.280           |

Table 3: Powdered ZELIAC, Zeolite and activated carbon characteristics

| Parameters                                      | Values       |
|------------------------------------------------|--------------|
| **Surface area data**                          |              |
| MultiPoint BET (m² g⁻¹)                        | 6.760×10¹    |
| Langmuir surface area (m² g⁻¹)                 | 9.328×10²    |
| BH method cumulative adsorption surface area (m² g⁻¹) |              |
| DH method cumulative adsorption surface area (m² g⁻¹) | 9.328×10² |
| t-method external surface area (m² g⁻¹)         | 3.421×10¹    |
| t-method micropore surface area (m² g⁻¹)        | 3.328×10¹    |
| DR method micropore area (m² g⁻¹)               | 1.153×10²    |
| **Pore volume data**                           |              |
| Total pore volume for pores with diameter less than 4.06 nm at P/P₀ = 0.501894 (cc g⁻¹) | 4.029×10⁻² |
| BH method cumulative adsorption pore volume (cc g⁻¹) | 9.393×10⁻³ |
| DH method cumulative adsorption pore volume (cc g⁻¹) | 1.011×10⁻² |
| t-method micropore volume (cc g⁻¹)              | 1.803×10⁻²   |
| DR method micropore volume (cc g⁻¹)             | 4.098×10⁻²   |
| HK method cumulative pore volume (cc g⁻¹)       | 3.172×10⁻²   |
| SF method cumulative pore volume (cc g⁻¹)       | 3.222×10⁻²   |
| **Pore size data**                              |              |
| Average pore diameter (nm)                      | 2.384×10⁰    |
| BH method adsorption pore diameter (Mode DV(d)) (nm) | 3.652×10⁰ |
| DH method adsorption pore diameter (Mode Dvφd) (nm) | 3.652×10⁰ |
| DA method pore diameter (Mode) (nm)             | 1.768×10⁰    |
| HK method pore diameter (Mode) (nm)             | 3.675×10⁻¹   |
| SF method pore diameter (Mode) (nm)             | 4.532×10⁻¹   |

been shown by Table 3. In this study, powdered zeolite (PZE) and Powdered Activated Carbon (PAC) with a size ranging from 75-150 μm were used as adsorbent in the PZE-SBR and PAC-SBR, respectively (Aziz et al., 2011a).

The aluminosilicate molecular structure with weak cationic bonding sites of zeolite is shown by Fig. 1. This structure is useful for ion exchange. Activated carbon is generally applied to adsorb natural organic compounds, taste and odor compounds and synthetic organic chemicals in drinking water treatment (Dvorak, 2013; Lin et al., 2010). Figure 1 shows the SEM image from surface of powdered activated carbon. This figure shows the pores on the surface of activated carbon.

**Operation reactors:** The SBR consists of five steps, namely, load, react, settle, idle and draw. In all experiments, the loading (20 min), blending (20 min), settling (90 min), idle (10 min) and drawing (10 min) periods were present. Table 4 shows the operation parameters in SBR, PZ-SBR, PZE-SBR and PAC-SBR. The beakers were loaded with 120 mL (10%) of acclimated sludge and 1080 mL (90%) of household wastewater and Sungai Petani landfill leachate. The primary features of activated sludge, wastewater and leachate are shown in Table 1.
Table 4: Operation parameters in repeated experiments

| Parameters                  | Normal-SBR | PZ-SBR | PAC-SBR | PZE-SBR |
|-----------------------------|------------|--------|---------|---------|
| Settling time (min)         | 90         | 90     | 90      | 90      |
| Adsorbent dosage (g L⁻¹)    | 3          | 3      | 3       | 3       |
| Contact time (min)          | 769 (12.82 h) | 769 (12.82 h) | 756 (12.60 h) |
| Aeration rate (L min⁻¹)     | 4.32       | 2.41   | 3.07    | 1.82    |
| Leachate/wastewater ratio (v/v, %) | 20      | 20     | 20      | 20      |
| Cycle time (min)            | 930 (15.5 h) | 930 (15.5 h) | 930 (15.5 h) | 930 (15.5 h) |
| OLR (kg m⁻³ day⁻¹)          | 1.00       | 1.00   | 1.00    | 1.00    |
| HRT (day)                   | 0.72       | 0.72   | 0.72    | 0.72    |
| MLSS (mg L⁻¹)               | 1510       | 7613   | 6516    | 6105    |
| F/M                         | 0.25       | 0.05   | 0.06    | 0.06    |

The reactors were separated into four groups. Six reactors were used for SBR, six reactors were used for PZ-SBR, six reactors were used for PZE-SBR and six reactors were used for PAC-SBR. Based on preliminary experiments, 3.24 g of PZ, PZE and PAC (specifically, PZ, PZE and PAC dosages = 3 g L⁻¹) were added to each PZ-SBR, PZE-SBR and PAC-SBR prior to aeration. The
removal effectiveness of sulfide, phenols and chromium was experimentally verified by evaluating objective factors before and after treatment. The following Eq. 1 was used to measure removal effectiveness:

$$\text{Removal(\%)} = \frac{C_i - C_f}{C_i} \times 100$$

(1)

where, $C_i$ and $C_f$ are the first and last concentrations of the factors, respectively.

**Analytical methods:** All experiments were performed in compliance with the Standard Methods for the Examination of Water and Wastewater (APHA., 2005). YSI 556 MPS (YSI incorporated, USA) was used to record the rates of pH, temperature (°) and electrical conductivity (ms cm$^{-1}$). A spectrophotometer (DR/2800 HACH) was used to evaluate phenols (mg L$^{-1}$), sulfide (mg L$^{-1}$ S$^{2-}$), nitrite (mg L$^{-1}$), color (Pt. Co), Chemical Oxygen Demand (COD) (mg L$^{-1}$), total nitrogen (mg L$^{-1}$), total iron (mg L$^{-1}$ Fe), manganese (mg L$^{-1}$ Mn), chromium (mg L$^{-1}$ Cr) and nickel (mg L$^{-1}$ Ni).

**RESULTS AND DISCUSSION**

As Table 1 shows, Sungai Petani leachate contained high-intensity Cr (0.21 mg L$^{-1}$) and high concentration of phenols (1.84 mg L$^{-1}$) compared with Malaysian standard (Environmental Quality Council, 2009). Also the concentration of sulfide was 0.300. In this study, phenols, sulfide and Cr (VI) removal from landfill leachate and domestic wastewater using the PZ, PAC and PZE supplemented SBR process to decrease the environmental risks have been investigated. Table 5 is shown the Cr, phenols and sulfide removal efficiency by SBRs.

**Phenols removal:** Landfill leachate contains a large number of dangerous compounds, such as aromatics, halogenated compounds, heavy metals, phenols, pesticides and ammonium, which are considered dangerous even in small amounts. The harmful effects of these compounds are often caused by multiple and synergistic effects. Phenolic compounds released into the environment are particularly of high concern because of their potential toxicity. Kurata et al. (2008) measured 41 kinds of phenols in three landfill sites in Japan. The results achieved in the present study agree well with the literature (Aziz et al., 2012; Kurata et al., 2008). In this study, the 4-aminoantipyrine method was used to measure phenols and determine all ortho-substituted and meta-substituted phenols or naphthols, but not the para-substituted phenols.

Based on the Table 5 and Fig. 2, the phenols removal was 32.56, 67.71, 45.29 and 33.31% in Normal-SBR, PZ-SBR, PAC-SBR and PZE-SBR, respectively. The PZ-SBR is more efficiency in phenols removal. Hameed and Rahman (2008) removed phenols from aqueous solutions by activated carbon. Aziz et al. (2012) removed 55% of phenols from landfill leachate by activated carbon augmented SBR process.

| Types      | Phenols (mg L$^{-1}$) | Sulfide (mg L$^{-1}$) | Cr (VI) (mg L$^{-1}$) | Removed concentration (mg L$^{-1}$) |
|------------|-----------------------|-----------------------|-----------------------|--------------------------------------|
| Normal-SBR | 32.56                 | 39.28                 | 38.15                 | Phenols: 0.60 Sulfide: 0.117 Cr (VI): 0.080 |
| PZ-SBR     | 67.71                 | 74.13                 | 79.24                 | Phenols: 1.24 Sulfide: 0.222 Cr (VI): 0.166 |
| PAC-SBR    | 45.29                 | 61.32                 | 55.24                 | Phenols: 0.83 Sulfide: 0.183 Cr (VI): 0.116 |
| PZE-SBR    | 43.31                 | 56.39                 | 66.37                 | Phenols: 0.80 Sulfide: 0.169 Cr (VI): 0.139 |
Sulfide removal: High sulfate concentrations are common in wastewaters generated from paper and pulp industries, molasses based fermentation industries, edible oil refineries and in acidic leachates of pyritic waste rock and tailings (Rao et al., 2003). Based on the Table 5, the sulfide removal was 39.28, 74.13, 61.32 and 56.39% in Normal-SBR, PZ-SBR, PAC-SBR and PZE-SBR, respectively. The PZ-SBR is more efficiency in sulfide removal. It is so clear that PZ has a good performance to remove sulfide in comparing with SBR, PAC-SBR and PZE-SBR.

Chaung et al. (2014) and Sattler and Rosenberk (2006) studied sulfide removal from wastewater by adsorption method.

Chromium removal: Chromium (Cr) is an environmental pollutant element and ranks seventh in abundance in the earth crust. The major contributors of Cr contamination are the leather tanning, electroplating and stainless steel industries (Karimi, 2013). Hexavalent chromium is known to be toxic to humans, animals, plants and microorganisms. Because of its significant mobility in the subsurface environment, the potential risk of groundwater contamination is high (Gheju and Pode, 2010). Based on the Table 5 and Fig. 2, the Cr (VI) removal was 38.15, 79.24, 55.24 and 66.37% in Normal-SBR, PZ-SBR, PAC-SBR and PZE-SBR, respectively. The PZ-SBR is more efficiency in Cr removal.

Talokar (2011) studied chromium removal from wastewater by adsorption using low cost materials such as powdered activated carbon. Aggarwal et al. (1999) removed Cr (VI) from aqueous solution by granular activated carbon.

CONCLUSION

Cr (VI), phenols and sulfide from Sungai Petani landfill leachate and domestic wastewater were eliminated by performing PZ, PAC and PZE-supplemented SBR. The reactors were separated into four groups, six reactors were used for SBR (normal SBR), six reactors were used for PZ-SBR (powdered ZELIAC-supplemented SBR), six reactors were used for PAC-SBR (powdered activated carbon-supplemented SBR) and six reactors were used for PZE-SBR (powdered Zeolite-supplemented SBR). Based on batch experiments and optimization experiments, powdered
adsorbents dosage (PZ, PZE and PAC dosages = 3 g L⁻¹), settling time (90 min) and leachate to wastewater mixing ratio (20%; v/v), were fixed. The main conclusions of this study are presented below:

- SBR was able to remove 32.56, 39.28 and 38.15% of phenols, sulfide and Cr (VI), respectively
- PZ-SBR removed 67.71, 74.13 and 79.24% of phenols, sulfide and Cr (VI), respectively
- PAC-SBR was able to remove 45.29, 61.32 and 55.24% of phenols, sulfide and Cr (VI), respectively
- PZE-SBR was able to remove 43.31, 56.39 and 66.37% of phenols, sulfide and Cr (VI), respectively
- This result indicates that PZ-SBR can treat phenols, sulfide and Cr (VI) of leachate more efficiently than SBR, PAC-SBR and PZE-SBR

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