

Effects of ion exchange resins in different mobile ion forms on semi-aerobic landfill leachate treatment

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ABSTRACT

Landfill leachate is one of the major contamination sources. In this study, the ability of synthetic ion exchange resins which carry different mobile ion for removing color, chemical oxygen demand (COD), and ammonia nitrogen (NH₃-N) from stabilized leachate was investigated. The synthetic resin INDION 225 Na as a cationic exchanger and INDION FFIP MB as an anionic exchanger were used in this study. INDION 225 Na was used in hydrogen form (H⁺) and in sodium form (Na⁺), while INDION FFIP MB resin was used in hydroxide form (OH⁻) and in calcium form (Cl⁻) form. The results indicated better removal of color, COD and NH₃-N by using INDION 225 Na in H⁺ as compared with Na⁺ form, while no performance differences were observed by using INDION FFIP MB in OH⁻ or Cl⁻ form. Applying cationic resin followed by anionic resin achieved 97, 88 and 94, percent removal of color, COD and NH₃-N. The residual amounts were 160 Pt-Co, 290 mg/L and 110 mg/L of color, COD and NH₃-N respectively.

Key words | anionic exchanger, cationic exchanger, ion exchange, semi-aerobic landfill leachate, stabilized leachate

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INTRODUCTION

Currently, Malaysia generates about 6.2 million tonnes of solid waste per year, or approximately 17,000 tonnes daily. This amount is expected to increase to more than 30,000 tonnes per day by 2020 due to the increase in population and per capita waste generation. Therefore solid waste management is a main challenge for Malaysia to address in the light of 2020, when Malaysia plans to become a fully developed nation by 2020 (Abdul Rahman *et al.* 2009). About 95 percent of the collected solid wastes in Malaysia are disposed in landfills. Generally sanitary landfill is one of the most common methods for municipal solid waste disposal. It is widely accepted and used in several countries due to its operation and maintenance cost. Landfills generate a liquid leachate which is a complicated wastewater that contains high concentration of pollutants which adversely affects the environment (Li *et al.* 1995; Aziz *et al.* 2007). These pollutants include

organic contaminants such as COD, BOD, ammonia, suspended solids, heavy metals and inorganic salts etc. Untreated disposal of landfill leachate cause a severe contamination of surface and groundwater through different mechanism as well as deep percolation into soils and subsoils (Tatsi *et al.* 2003). Landfill leachate characteristics are strongly depend on the disposed solid waste composition, land fill age, site operation and weather variation (Kurniawan *et al.* 2006).

Generally, during the early settlement phase (acidogenic phase) of the disposed municipal solid waste, young landfill leachate contains large amount of biodegradable and non-biodegradable materials, particularly in terms of volatile fatty acids (Kurniawan *et al.* 2006; Renou *et al.* 2008). At this stage, leachate would be characterized with high BOD (4,000–40,000 mg/L), high COD (6,000–60,000 mg/L), high NH₃-N (500–2,000 mg/L), BOD/COD

ratio typically ≤ 1.0 and pH range from 4.5 to 7.5 (Christensen *et al.* 2001; Alvarez-Vazquez *et al.* 2004). At later stages (methanogenic phase), the biodegradable fraction of organic compounds are decomposed over a long time period, resulting in the production of stabilized leachate which has large amount of non-biodegradable organic compounds with high molecular weight such as humic and fulvic substances. Therefore, stabilized leachate cannot be treated using biological processes (Aziz *et al.* 2007; Renou *et al.* 2008). Stabilized landfill leachate is normally characterized by high concentrations of COD (5,000–20,000 mg/L), low BOD (20–550 mg/L), pH range 7.5 to 9.0 and BOD/COD ratio of less than 0.1 (Christensen *et al.* 2001; Alvarez-Vazquez *et al.* 2004).

Several treatment processes have been applied to treat stabilized leachate such as aerobic and anaerobic biological degradation, chemical oxidation, precipitation, coagulation-flocculation, adsorption and ion exchange process (Bashir *et al.* 2009). The latter involved with a reversible interchange of ions between the solid and liquid phases. For example, the solid ion-exchange particles can exchange their mobile ions with similarly charged ions from the surrounding medium (Helfferich 1962). The solid ion-exchange particles can be classified as natural-inorganic particles (zeolites) and synthetic-organic resins, which were developed from high-molecular-weight polyelectrolytes (Cheremisinoff 2002). Natural zeolites (crystalline aluminosilicates) were the first ion exchangers used commercially, however, zeolites had been completely replaced with synthetic resins (consists of a cross linked polymer matrix where charged functional groups are attached by covalent bonding) in modern applications due to their faster exchange rates, longer life and higher capacity (Letterman 1999). Currently, there are no limitations for the commercialization of ion exchange resin varieties due to their polymer matrices, functional groups, capacity and porosity which are controllable during manufacturing (Letterman 1999).

Zagorodni (2006) indicated that ion exchange resins have been widely used in water and waste water treatment for extraction, separation, and purification of organic substances. Several studies have been conducted to remove metal ions from water and wastewater using ion exchange resins, such as (Cr) from industrial effluents (Cavaco

et al. 2007); $\text{NH}_3\text{-N}$ and nitrate from fertilizer factory wastewater (Leakovica *et al.* 2000); Cd (II) and Ni (II) from landfill leachate (Majone *et al.* 1998); $\text{NH}_3\text{-N}$ from wastewater (Jorgensen & Weatherley 2003); Cu^{2+} , Ni^{2+} , Cd^{2+} , Zn^{2+} , Ca^{2+} , Na^+ , and NH_4^+ ions from water (Kiefer *et al.* 2007); Zn^{2+} , Cr^{3+} and Cr^{6+} from mixed plating rinse wastewater (Sapari *et al.* 1996) etc.

Although the application of ion exchange is not commonly used for landfill leachate treatment, it has received significant attention for the removal of non-biodegradable substances particularly humic substance (Fettig 1999; Rodriguez *et al.* 2004). Many synthetic and natural organic compounds are classified as weak acids, which could be exchanged by using anionic exchanger so that humic substances could be exchanged (De Silva 1997; Gottlieb 1999; Li & Sengupta 2004; Mergen *et al.* 2008). The effectiveness of ion exchange resin to remove non-biodegradable organic substances (that significantly contribute to the COD and color in stabilized landfill leachate) has demonstrated the capability of ion exchange resin to remove these pollutants. Besides that, ion exchange technique has high capability to remove NH_4^+ ions in landfill leachate (Lin & Wu 1996). Jorgensen & Weatherley (2003) observed that high removal efficiency of ammonia from wastewater was achieved using ion exchangers i.e. Dowex 50w-x8 and Purolite MN500. The present study focused on the treatability of semi-aerobic stabilized landfill leachate using different ion exchange resins with different mobile ions form. Furthermore, the performance of anion and cation ion exchangers was investigated based on different sequence of treatment systems.

MATERIALS AND METHODS

Resins characteristics

We used two synthetic available ion-exchanger resins in this study: the anionic exchanger INDION FFIP MB and the cationic exchanger INDION 225 Na; the resins were supplied by “Ion Exchange (INDIA) Ltd”. The resins and their physicochemical properties are presented in Table 1.

Table 1 | The main physicochemical properties of the studied resins

Property	INDION FFIP MB	INDION 225 Na
Type	Strongly base anion exchange resin	Strongly acid cation exchange resin
Matrix	Cross-linked polystyrene, isoporous type	Cross-linked polystyrene, gel type
Functional group	Quaternary amine (-N+R ₃)	Sulphonic acid (-SO ₃)
Ionic form (as supplied)	Chloride	Sodium form
Maximum operating temperature	60°C (OH ⁻ form) 90°C (Cl ⁻ form)	120°C (H ⁺ form) 120°C (Na ⁺ form)
Operating pH range	0 to 14	0–14
Particles size range	0.45–0.55 mm	0.3–1.2 mm
Total exchange capacity	1.2 meq/mL	2.0 meq/mL
Appearance	Brown to dark brown	Yellow

Sample of leachate

Leachate samples were collected from the aeration pond at Pulau Burung semi aerobic landfill site (PBLs) in Penang, Malaysia from February 2008 to January 2009. PBLs has an area of 62.4 ha, only 33 ha are currently operational, and this area receives 1,800 ton of municipal solid waste daily (Bashir *et al.* 2009). A semi-aerobic landfill is designed with piping system at the bottom for leachate collection. Collection pipes allowed the air to flow in and out from the solid waste. This system helps to make aerobic bacteria more active, increasing waste decomposition rate. The samples were transported to the laboratory and stored in a refrigerator at 4°C prior being used in experiments to minimize biological and chemical reactions. The initial concentration of pH, color, chemical oxygen demand (COD), ammonia nitrogen (NH₃-N), turbidity and suspended solid were measured according to the Standard Method of Water and Wastewater (APHA 2005). The samples were analyzed and the characteristics of the samples are presented in Table 2.

Experimental procedure

Studied cation and anion resins were rinsed with distilled water extensively to remove the adhering dirt followed by filtration using vacuum pump. According to the resin types the studied ion exchanger were pre-treated for experimental purpose as following:

- (i) *Cation Resin*: Removal of color, COD, and NH₃-N from PBLs stabilized leachate samples were studied by a batch experiment technique. The experiments were carried out after pre-treating the cationic exchange resin using 100 mL of different types of strong acid (HCl and H₂SO₄) concentration level at 0.2, 0.4, 0.6, 0.8, 1.0 and 1.5 normality (N), for converting cationic resin to H⁺ form, whereas, NaCl was used to reactivate Na⁺ form. The experiments were conducted to investigate the relationship between pollutants removal and acid concentrations that were used to regenerate resins.
- (ii) *Anion Resin*: Removal of color COD and NH₃-N from PBLs were also investigated by using anion exchange resin, which were pre-treated with 1N

Table 2 | Characteristics of stabilized landfill leachate from PBLs

Parameters	Units	Values
Age	Yrs	> 10
COD	mg/L	2,060–2,700
NH ₃ -N	mg/L	1,630–2,050
Apparent color	Pt-Co	4,250–5,700
Turbidity	FAU	180–330
SS	mg/L	140–360
pH	–	8.67–9.17
Alkalinity	(mg/L as CaCO ₃)	2,370
Iron	(mg/L Fe)	1.6–2.3
Zinc	(mg/L Zn)	0.2–0.4
Copper	(mg/L Cu)	0.1–0.55

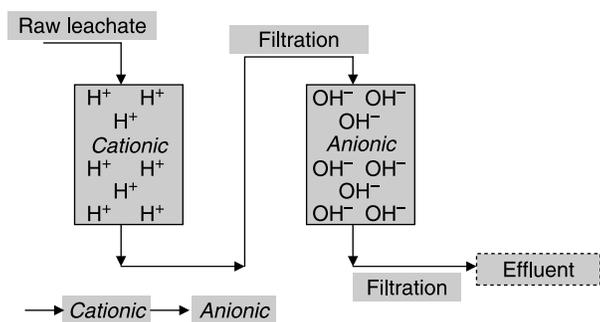


Figure 1 | Schematic of leachate treatment by using cationic resin followed by anionic resin.

concentration of NaCl and NaOH (solution volume, 100 mL). Then resins were washed with distilled water extensively followed by filtration and dried at surrounding environmental temperature.

A sequence treatment system was also investigated; where the leachate sample was treated using cationic followed by anionic exchanger resins (Figure 1). Similarly, a reverse series system also was investigated.

Condition of experiments

The batch experiments were carried out through shaking 100 mL of raw stabilized leachate sample in 250 mL size volumetric flask by using an orbital shaker. The raw leachate samples used in this study were not subjected to any pre-treatment process prior to ion exchange. The experimental conditions were the contact time, amount of exchange resin and shaking speed at 15 min, 38.0 gm and 200 rpm, respectively for cationic exchanger, while the conditions for anionic exchanger resin were 90 min, 33 gm and 200 rpm respectively. These optimum conditions of both cationic and anionic exchanger resin were chosen based on another preliminary experiment results. After each experiment, the effluent was filtered using glass fiber filter paper (GC-50) prior to examining the colour, COD, NH₃-N removal using spectrophotometer (HACH DR/2010).

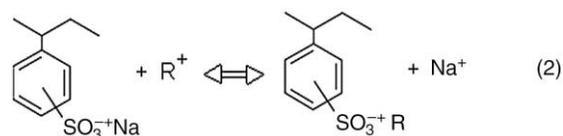
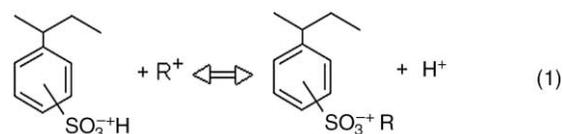
RESULTS AND DISCUSSION

The raw leachate characteristic showed in Table 2 indicates a high amount of organic matter presence and causing

high-strength dark color and COD amount. The results of treated leachate using ion exchange techniques are represented in the following sections.

The effect of cationic exchanger form on removal of parameters

According to Zagorodni (2006), cation ion exchange resins with sulfonate group (-SO₃⁻) as a stationary functional group and the mobile exchangeable cation i.e. hydrogen (Equation (1)) or sodium (Equation (2)) has great ability to remove cations from wastewater. Equations (1) and (2) illustrate the chemical interactions where counter ions such as hydrogen ion (H⁺) or sodium ion (Na⁺) leaving the ion exchanger are replaced by an equivalent amount of other positive counter ions (R⁺) from the solution such as; NH₄⁺, Ca⁺⁺, Mg⁺⁺ and heavy metals.



Synthetic cationic exchanger resin (INDION 225 Na) pre-treated with different concentration of acid regenerants i.e. HCl, H₂SO₄ and NaCl to convert cation exchanger resin to H⁺ or Na⁺ form which were used for treating the leachate samples at different reagents normality to remove color, COD and NH₃-N (Figures 2, 3 and 4).

Figures 2 and 3 show that using cationic exchange resin (in the form of H⁺ mobile ion) which was pre-treated with HCl and H₂SO₄ reagents have resulted in 74.3, 45.4 and 91.8 percent removal of color, COD and NH₃-N, respectively from the stabilized leachate in the case of using HCl, while 76.8 to 47.0 and 92.8 percent were removed in the case of H₂SO₄. Figure 4 shows the use of anionic resin in Na⁺ form resulted in 9.8, 4 and 72 percent removal of color, COD and NH₃-N. The result suggested that better performance of cationic resin in H⁺ form rather than Na⁺ form, may due to the high acidity of the solutions (pH < 3). Table 3 shows pH values of effluent using anionic resin in different form. High

acidity was resulted due to the high alkalinity removal and to the inter-exchanging between positive H^+ ions and the negative ions, such as chloride, sulfide, nitrite or weak organic compounds. The results indicated that pH has strongly affected the color and COD removals. This could be attributed by the presence of trace amount of ion Fe^{2+} and Fe^{3+} in the leachate which acts as an oxidant in oxidizing organic substance at low pH values (Wang *et al.* 2006). In acidic solution, free radical OH^{\cdot} can easily be produced in leachate sample and organic substances can simply be oxidized (Liu & Jiang 2005). Sheha & El-Zahhar (2008) reported that pH value of the solution played an important role on the percentage of pollutants removal due to its influence on the surface properties of the applied resins and the degree of ionization.

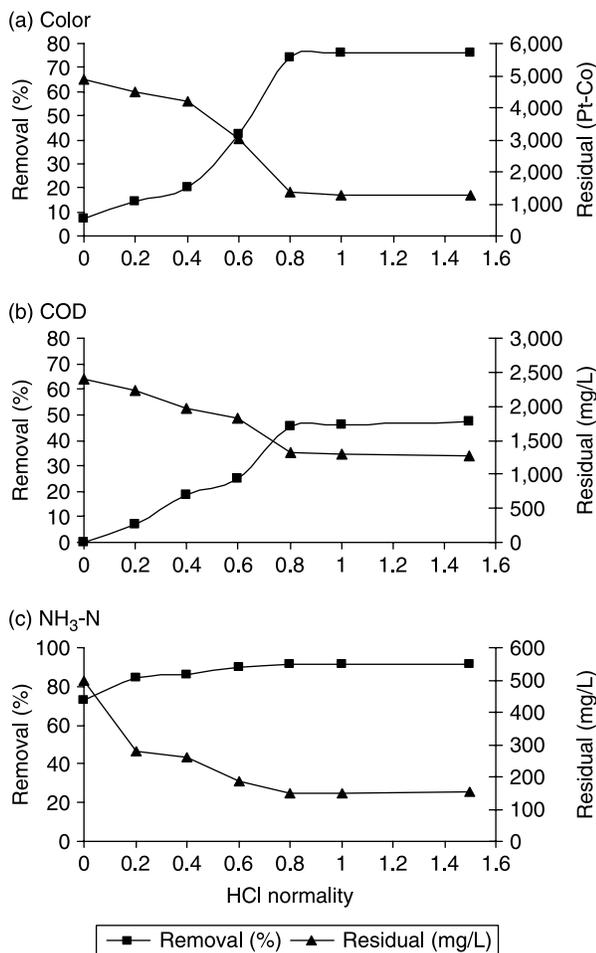


Figure 2 | Effect of treating cationic resin using different HCl (strong acid) concentration on color, COD and NH_3-N removal.

No removal performance difference was observed in cationic exchange resins which were pre-treated with HCl and H_2SO_4 . However, pollutant removal was increased by increasing the acids concentration level up to 0.8 N and rather it remained constant. The effects of pre-treating cation on the color, COD, NH_3-N removal and pH were statistically analyzed using single factor one way ANOVA. Table 3 shows that P-value for experimental parameters is small < 0.05 (the results were statistically significant), thus the null hypothesis are rejected, indicating that there are evidence against all populations mean being equal.

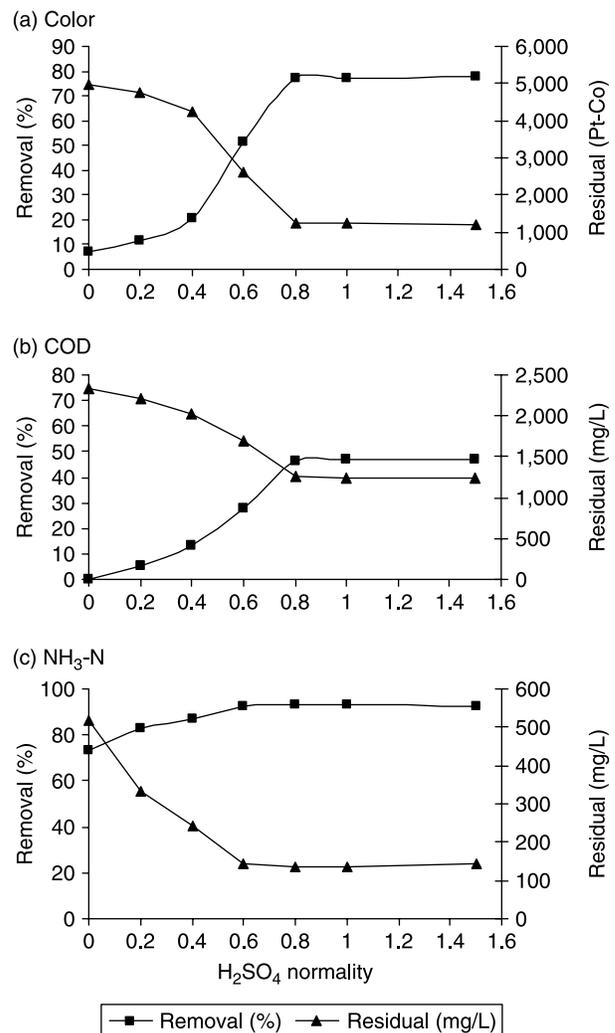


Figure 3 | Effect of treating cationic resin using different H_2SO_4 (strong acid) concentration on color, COD and NH_3-N removal.

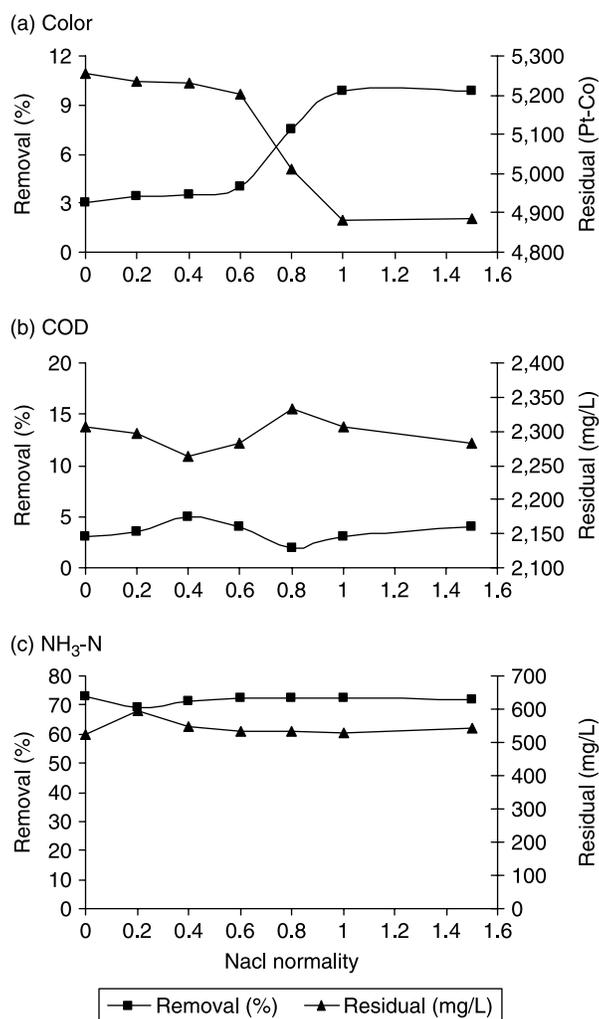


Figure 4 | Effect of treating cationic resin using different NaCl (salt) concentration on color, COD and $\text{NH}_3\text{-N}$ removal.

The effect of anionic exchanger form on removal of parameters

The performance of anionic exchanger (INDION FFIP MB) in Cl^- and OH^- form was investigated. It was

observed that the removal of color, COD and $\text{NH}_3\text{-N}$ were similar, where 68, 57 and 12 percent of color, COD and $\text{NH}_3\text{-N}$ were removed using anionic exchanger in Cl^- , while 71, 60 and 4 percent were removed by anionic exchanger in OH^- , respectively.

The use of anion resin pre-treated by NaCl or NaOH resulted good removal of color and COD from leachate which indicated good removal performance of synthetic and natural dissolved organic compounds. Many synthetic organic compounds contain weak and strong acid functional groups, therefore, these compounds are soluble and exist as ions in the aqueous phase (Li & Sengupta 2004). Moreover, most of the natural organic components are weak acids containing carboxylic acid groups (Gottlieb 1999). The removal of organic compound occurred in two ways: (i) Ion exchange (polar attractions); (ii) Physical adsorption (non-polar attractions), as illustrated in Figure 5 (Fu & Symons 1990; Li & Sengupta 2004; Tan & Kilduff 2007).

The treatment effective using cationic and anionic exchangers as a sequence

As per experimental procedure and above results for treating raw leachate, Figure 6 demonstrates leachate treatment efficiency via different cases i.e. (1) Cationic resin in H^+ form; (2) Anionic resin in OH^- form; (3) Anionic resin in OH^- form followed by cationic resin with H^+ form; and (4) Cationic resin in H^+ form followed by anionic resin in OH^- form.

Applying cationic followed by anionic resin resulted in 97, 88 and 94 percent removal of color, COD and $\text{NH}_3\text{-N}$, respectively from initial concentration of 5250 Pt-Co, 2,430 mg/L and 1,870 mg/L. The residual amounts were 160 Pt-Co, 290 mg/L and 110 mg/L of color, COD and $\text{NH}_3\text{-N}$ respectively. As can be seen in Figure 6,

Table 3 | The effect of cationic resin pre-treated by 0.8N solution of different regenerants on color, COD, $\text{NH}_3\text{-N}$ removal and pH

Regenerants	Parameters							
	Color		COD		NH ₃ -N		pH	
	Removal (%)	P-value	Removal (%)	P-value	Removal (%)	P-value	pH	P-value
HCl	74.3	0.0032	45.4	0.0061	91.8	<0.0001	2.99	0.00027
H ₂ SO ₄	76.8	0.003	47	0.0059	92.8	<0.0001	3.12	0.0012
NaCl	7.5	<0.0001	4	<0.0001	72.4	<0.0001	8.95	<0.0001

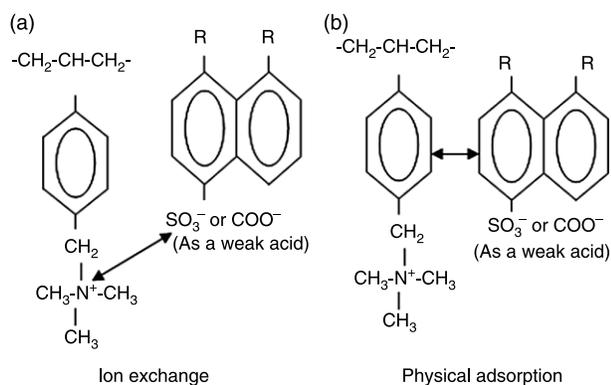


Figure 5 | A schematic illustrating adsorption mechanism of dissolved synthetics and natural organic compounds by anion exchange resin: (a) Ion exchange, involving counter ion displacement from the anion resin phase and electrostatic interaction between the positively charged quaternary ammonium functional group of exchange resins and the negatively charged of carboxylic or sulfonic; (b) physical adsorption, involving van der Waals interactions between non-ionic head and ion exchanger's hydrophobic polystyrene matrix.

performance of cationic followed by anionic resin process was better than applying anionic followed by cationic resin process. However anion followed by cation exchanger resulted in average 93, 86 and 92 percent removal of color, COD and $\text{NH}_3\text{-N}$ respectively. The performance difference between two various sequences may be due to the presence of high amount of different ions in leachate that can be demonstrated for example, by alkalinity of the raw leachate (2,370 mg/L as CaCO_3) that includes 1,900 mg/L as Ca-CaCO_3 and 470 mg/L as Mg-CaCO_3 . The application of cationic exchanger resin has eliminated the alkalinity to 860 mg/L as CaCO_3 which later enhanced the effectiveness of anionic resin. While applying anionic resin first and then followed by cationic resin, this has resulted in fouling and blockage of anionic resin pores and its surface which decrease in the exchange capacity of the resins and also reducing the ability of anion resin to remove non-ionized organic substances. The latter were in fact removed by resin adsorption and not by ion exchange.

The experimental procedure indicated a good removal performances were obtained via resin treatment in a sequence system. However high amount of COD was removed from studied leachate, still 290 mg/L of residual COD present in the effluent. Thus further treatment need to carry out before discharging, to meet with the standard B of the Environmental Quality (Sewage and Industrial Effluents) Regulations 1979, under the Environmental

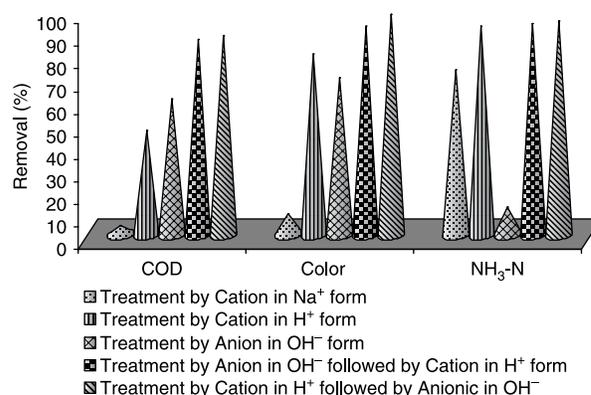


Figure 6 | The affectivity of applying anionic, cationic resin or both on COD, color and $\text{NH}_3\text{-N}$ removal.

Quality Act of Malaysia, 1974, where the effluent COD should be less than 100 mg/l.

Comparison of removal efficiency with other techniques for stabilized leachate treatment

In order to evaluate the color, COD and $\text{NH}_3\text{-N}$ removal efficiency from landfill leachate, the performance of different treatments was investigated in the present study. Electrochemical oxidation has been employed for stabilized landfill leachate treatment using graphite carbon electrodes (Bashir *et al.* 2009). This resulted in 68% COD and 84% color removals respectively. About 72% removal of COD and 90% removal of color were obtained by electrochemical treatment using cast iron electrodes (Atmaca 2009). Ghafari *et al.* (2009) reported that by using alum coagulant, the maximum removal of color and COD were 62.8% and 86.4% respectively. Struvite precipitation has achieved 50% and 90% removal efficiency of COD and $\text{NH}_3\text{-N}$ from initial concentration of 4,024 and 2,240 mg/L respectively (Ozturk *et al.* 2003). According to Calli *et al.* (2005), 94% of $\text{NH}_3\text{-N}$ was removed by ammonium stripping from initial concentration 3,260 mg/L while less than 15% of COD was removed under the same conditions. About 89% COD and 72% $\text{NH}_3\text{-N}$ were removed by nanofiltration from leachate with an initial COD concentration of 3,000 mg/L and $\text{NH}_3\text{-N}$ concentration of 950 mg/L (Ozturk *et al.* 2003). Reverse osmosis (RO) has achieved 95 and 82 percent removal of COD and $\text{NH}_3\text{-N}$ from stabilized

leachate, with initial concentration of 1,254 mg/L of COD and 541 mg/L of NH₃-N (Thorneby *et al.* 2003). Among the various stabilized leachate treatments, the present study has achieved 97, 88 and 94 percent of color, COD and NH₃-N removals respectively from initial concentrations of 5250 Pt-Co, 2,430 mg/L and 1,870 mg/L. Comparing with other treatment processes efficiency, the performance cation followed by anion ion exchange resin for the treatment of stabilized leachate can be considered satisfactory. However, the combination of two or more processes for stabilized leachate treatment can perform better. The combination of RO and activated sludge (with initial COD and NH₃-N concentrations of 6,440 mg/L and 1,153 mg/L, respectively) has achieved 99% removal for both COD and NH₃-N (Baumgarten & Seyfried 1996).

In this study, the application of ion exchange resin for the treatment of stabilized leachate has demonstrated some advantages i.e. good removal efficiency, low running cost and low energy consumption is desired. However, this application has some limitations due to the overall treatment cost related to the total amounts of resins required and the fouling problems. Besides that, this technology is not appropriate for young leachate treatment as biological treatment could be effectively used prior to ion exchange.

CONCLUSION

Stabilized leachate treatment using ion exchange technique was investigated in this study. Synthetic cation ion exchanger (INDION 225 Na) in H⁺ form showed better performance than same exchanger in Na⁺ with 76.8 to 47.0 and 92.8 percents removal of color, COD and NH₃-N, respectively. There were no performance differences in terms of the removals for all pollutants using anionic exchanger (INDION FFIP MB) in OH⁻ or Cl⁻ form. However, high removal efficiency was obtained using sequence treatment system, where applying of cationic exchangers followed by anionic exchangers achieved 97, 88 and 94 percent of color, COD and NH₃-N removals, respectively from initial concentration of 5250 Pt-Co, 2,430 mg/L and 1,870 mg/L as well. The residual amounts were 160 Pt-Co, 290 mg/L and 110 mg/L of color, COD and NH₃-N respectively. Employing of this technique could be

an effective alternative process for treatment of stabilized landfill leachate.

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