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Application of psyllium husk as coagulant and coagulant aid in semi-aerobic landfill leachate treatment

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ABSTRACT

Landfill leachate is a heavily polluted and a likely hazardous liquid that is produced as a result of water infiltration through solid wastes generated industrially and domestically. This study investigates the potential of using psyllium husk as coagulant and coagulant aid for the treatment of landfill leachate. Psyllium husk has been tested as primary coagulant and as coagulant aid with poly-aluminum chloride (PACl) and aluminum sulfate (alum). As primary coagulant, the optimum dosage and pH for PACl were 7.2 and 7.5 g/L, respectively, with removal efficiencies of 55, 80 and 95% for COD, color and TSS, respectively. For alum, the optimum conditions were 11 g/L alum dosage and pH 6.5 with removal efficiencies of 58, 79 and 78% for COD, color and TSS, respectively. The maximum removal efficiencies of COD, color and TSS were 64, 90 and 96%, respectively, when psyllium husk was used as coagulant aid with PACl. Based on the results, psyllium husk was found to be more effective as coagulant aid with PACl in the removal of COD, color and TSS as compared to alum. Zeta potential test was carried out for leachate, PACl, alum and psyllium husk before and after running the jar test to enhance the results of the jar test experiments.

1. Introduction

Landfills are the ultimate solid waste disposal mechanism in countries worldwide because they fulfil the purpose of high quantities of solid waste disposal at relatively lower costs [1,2]. However, landfill leachate which is produced mainly by the percolation of rainwater through an open landfill or through the cap of a completed site is a serious drawback associated with this method of disposal [3,4]. Municipal solid waste (MSW) landfill leachate is often defined as hazardous and heavily polluted wastewater [5]. Leachate may contain large amounts of organic matter including both biodegradable and refractory compounds, where humic-type constituents form an important group, as well as ammonia–nitrogen, heavy metals, chlorinated organic and inorganic salts [6]. Leachate from different landfills vary considerably in its chemical composition depending on the type of solid wastes deposited, hydrogeology of the landfill site, specific climate conditions, moisture routing through the landfill and landfill age, design and operation [7,8]. The choice of treatment method is strictly based on the composition and the properties of landfill leachate [1]. Biological treatment processes are effective for young or freshly produced leachate but are ineffective for leachate from older landfills. In contrast, physico-chemical methods which are not favored for young leachate treatment are advised for older leachate [9–11]. It is sometimes difficult to predict short term leachate quantity since it heavily depends on precipitation; however, long term leachate quantity can be predicted relatively more precisely. Leachate quality is also difficult to predict because each landfill is unique and the nature of wastes varies widely.

Leachate produced at the initial stages of the waste decomposition is highly rich in biological oxygen demand (BOD_5) and contains large quantity of biodegradable and non-biodegradable materials, mainly in terms of volatile fatty acids [10]. On the other hand, leachate from old landfills (stabilized leachate) is usually highly contaminated with non-biodegradable organic substances, such as humic-like and fulvic substances (measured as COD). In addition, the stabilized leachate contains substantial amounts of inorganic substances, particularly NH_3–N [11] as a result of hydrolysis and fermentation of nitrogen containing fractions of biodegradable refuse substrates. As stabilization of the waste proceeds, the accumulating concentration of ammonia is also influenced by washout as leachate is collected and removed for off site treatment. However, in bioreactor landfills with leachate containment, collection and in situ recirculation to accelerate decomposition of readily available organic fractions of the wastes, leachate ammonia nitrogen concentrations may accumulate to much higher levels as compared to traditional landfills [12].

Coagulation–flocculation is a relatively simple physico-chemical technique commonly applied for the removal of non-biodegradable organic compounds and heavy metals from

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Table 1
Chemical composition of psyllium husk [22].

Moisture (%)	Lipid (%)	Protein (%)	Total ash (%)	Soluble ash (%)	Total carbohydrate (%)
6.83 ± 0.04	0.00 ± 0.11	0.94 ± 0.00	0.00 ± 4.07	2.62 ± 0,03	84.98 ± 4.26

landfill leachate. The removal mechanism of this process mainly consists of charge neutralization of negatively charged colloids by cationic hydrolysis products followed by amalgamation of impurities in an amorphous hydroxide precipitate through flocculation [13]. The main function of this process is to remove total suspended solids (TSS) and colloid particles from a solution [14]. The colloid particles are mainly consisted of organic compounds. Coagulation is the destabilization of colloids by neutralizing the forces that keep them apart. Cationic coagulants provide positive electric charge to reduce the negative charge of the colloids in water and wastewater. As a result, the particles collide to form large particles (flocs). Rapid mixing is required to disperse the coagulant throughout the water. The coagulant overdose might cause a charge reversal and re-stabilization of the colloids [15].

The chemistry of coagulation and flocculation is primarily based on the electrical properties. Most particles present in leachate have a negative charge (−30 to −40 mV) so they tend to repel each other as a result. The purpose of most coagulant chemicals is to neutralize the negative charge on colloidal particles to prevent those particles repelling each other [6]. The amount of coagulant which should be added to the leachate will depend on the zeta potential which is defined as a measurement of the magnitude of electrical charge surrounding the colloidal particles. Hence, more coagulant is required when the zeta potential is large for negative values. The coagulants carry positive charge and are attracted to negative particles in water; hence the combination of positive and negative charges results in a neutral charge and the particles no longer repel each other [6].

Inorganic metal salts such as alum, ferrous sulfate, ferric chloride and ferric chloro-sulfate are generally used in coagulation–flocculation. Among these inorganic coagulants, iron salts are often more efficient than aluminum ones [16] and as a result, aluminum is removed from most of the water treatment plants and replaced by iron. In recent years, there has been a rise in the use of polymerized forms of metal coagulants such as polyaluminum chloride (PACl) for water treatment in Europe, Japan and North America due to their reduced cost and wider availability [17,18]. Such products are claimed to be more advantageous over conventional coagulants because of their higher removal of particulate and/or organic matter as well as natural advantages of lower alkalinity consumption and lesser sludge production [17]. Amokrane et al. [16] reported that conventional coagulants generally remove 10–25% COD from young leachate and 50–65% COD from stabilized or biologically pretreated leachate. A significant amount of research has been undertaken to investigate the effectiveness of inorganic coagulants for the treatment of mature landfill leachate. However, the use of natural coagulants is not well documented. Therefore, this study was performed to evaluate the coagulation potential of one of the natural coagulants, i.e., psyllium husk. The removal efficiency of COD, color, TSS was assessed by using psyllium husk alone and coagulant aid in the presence of PACl and alum as the main coagulants. The use of natural coagulants can not only reduce the requirements of conventional coagulants but can also reduce the cost of overall treatment process. Psyllium husk was assessed for its use as coagulant aid to assess its contribution in reducing the amount of PACl and alum. A rough cost estimate was also done to evaluate the cost reduction due to the use of psyllium husk as coagulant aid. Psyllium husk has a long history of being used in traditional and herbal medicine and is derived from the seed of the *Plantago Ovata* plant which is an annual herb native to Asia. Background knowledge on the basic physical and chemical properties of psyllium gum is helpful in understanding the binding mechanism and stability of psyllium gum in this study. There have only been a few researches done in exploring the structural and rheological properties of psyllium [19–21]. These studies focused on the health benefits related to structural and rheological properties of psyllium. The characteristics of the psyllium husk were explained by Qian et al. [22] as given in Tables 1 and 2.

Zeta potential test was carried out for raw leachate, PACl, alum and psyllium husk before and after treatment to enhance the findings of jar test. The objective of this study was to examine the efficiency of coagulation–flocculation processes for the removal of TSS, color and COD from a semi-aerobic landfill leachate using alum, PACl and psyllium husk as primary coagulants. The experiments involved the determination of the most appropriate coagulant types and dosages, the examination of the effect of pH on the removal capacity and assessment of the effectiveness of psyllium husk as coagulant aid with PACl and alum.

2. Materials and methods

2.1. Leachate collection and characterization

Leachate samples were collected from Pulau Burung Landfill Site (PBLS) which is located within Byram Forest Reserve at 5°24′N and 00°24′E in Penang, Malaysia. The total operational area of the landfill is 33 ha and it is equipped with a leachate collection pond. This site has a natural marine clay liner and a semi aerobic system, making it one of the only three such sites in Malaysia. The landfill receives 2200 ton of solid waste daily [4]. PBSL was developed as a semi-aerobic system complying with a Level II sanitary landfill by establishing a controlled tipping technique in 1991. It was subsequently upgraded to a Level III sanitary landfill by employing controlled tipping with leachate recirculation in 2001 [9]. Samples were collected in 20 L plastic containers and transported to the laboratory within 30 min and stored at 4 °C. The samples were characterized in terms of temperature, pH, COD, BOD_5, color, ammonia nitrogen and TSS. The parameter measurements were conducted thrice according to the standard method of water and wastewater [23]. pH was measured using a portable pH meter (Hach, sens ion 1, USA). COD was determined using colorimetric method (5220-D). BOD_5 was determined using 5-Day BOD test (5210-B). Ammoniacal nitrogen was determined using Nesslerization method (4500-NH_3) while TSS was determined by using Method No. 2540D. Color measurements were reported as true color, filtered using GC-50 filter papers (Advantec Toyo Kaisha Ltd., Japan) with0.45 μm pore size, assayed at 455 nm using DR 2000 HACH spectrophotometer. Method No. 2120C reports color in platinum–cobalt (Pt–Co), the unit of color being produced by 1 mg platinum/L in the form of the chloroplatinate ion. The effect of filtration on color removal

Table 2
Mineral analysis of psyllium husk [22].

Contents	Psyllium husk (μg/g)
Calcium	1500
Magnesium	150
Phosphorous	140
Potassium	8500
Sodium	640
Sulphur	23

Table 3
Characteristics of semi-aerobic leachate.

Parameter	Unit	Range	Mean	[7]
Temperature	°C	23–29	25	27
pH	–	7.86–8.92	8.13	8.4
COD	mg/L	1640–2580	2130	1925
BOD_5	mg/L	156–219	192	–
BOD_5/COD	–	0.08–0.09	0.09	–
Color	Pt–Co	2980–3530	3140	3869
NH_3-N	mg/L	1670–2100	1950	1184
TSS	mg/L	201–398	380	80

was corrected by means of a control sample. The Malvern Zetasizer Nano ZS was used for the measurement of zeta potential.

2.2. Coagulation–flocculation

The current study investigated psyllium husk as coagulant and as coagulant aid with two common coagulants, i.e., alum as an aluminum salt and PACl as a pre-hydrolyzed metal salt. Alum used in this study was in powder form with the formula $Al_2(SO_4)_3 \cdot 18H_2O$ (M = 666.42 g/mol, 51–59% $Al_2(SO_4)_3$, pH 2.5–4) supplied by Merck, Germany. A hydrolyzed solution of PACl with the formula $Al(OH)_xCl_y$ (where x is in the range 1.35–1.65, and $y = 3 - x$) and pH 2.3–2.9 due to the presence of hydrochloric acid was supplied by Hasrat Bestari Sdn Bhd, Penang, Malaysia. An 18% solution of PACl was used as stock solution throughout the experiments. Psyllium husk was obtained from a local shop. Psyllium husk solution was prepared by adding 3 g dry psyllium husk in 1 L distilled water and mixing well for 30 min followed by settling for 10 min. Sticky or mucilage solution produced as a result was used as coagulant and coagulant aid. Coagulation–flocculation experiments were carried out using a conventional jar-test apparatus (VELP-Scientifica, Model: JLT6, Italy) with impellers equipped with 2.5 cm × 7.5 cm rectangular blades. The time and speed for rapid and slow mixing were set with an automatic controller.

For this research, the operating parameters were adopted as rapid mixing speed 80 rpm, slow mixing speed 30 rpm, rapid mixing time 2 min, slow mixing time 30 min and settling time 120 min [7]. Different dosages and pH were investigated in this study for alum, PACl and psyllium husk as primary coagulant for removing COD, color and TSS. The examination of pH effect was performed by adjusting the pH value of leachate samples between 5 and 9 using solutions of 0.1 N sulphuric acid (H_2SO_4) and 0.1 N sodium hydroxide (NaOH). The removal efficiency was investigated by using psyllium husk as coagulant aid with alum and PACl. Zeta potential test was carried out to enhance the results of the jar test and give reasons to the removal mechanisms of the coagulation process. Zeta potential can present a measure of the net surface charge on the particle and potential distribution at the interface. Thus, zeta potential serves as a vital parameter in describing the electrostatic interaction between particles in dispersed systems and the properties of the dispersion as affected by this electrical phenomenon [24]. In the present study, the surface charge was evaluated by using Malvern Zetasizer Nano ZS. Measurements were taken at 25 °C with distilled water as the dispersal medium.

3. Results and discussion

3.1. Characteristics of semi-aerobic leachate

The characteristics of semi-aerobic leachate were investigated as given in Table 3. The leachate was highly dark in color (3140 Pt–Co) with pH between 7.86 and 8.92 (Table 3). In addition, leachate contained a huge amount of COD and ammonia. According to the literature, PBLS leachate was characterized as stabilized

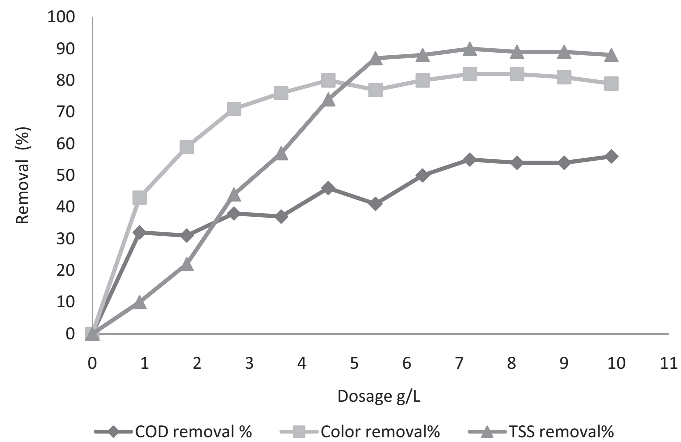

Fig. 1. Removal of COD, color and TSS at various dosages of PACl as primary coagulant at pH 7.5.

with low BOD_5/COD ratio <0.1 [7,10,12]. The BOD_5/COD ratio indicates the degree of biodegradation and landfill age. For young leachate, the BOD_5/COD ratio could reach to 0.83 during acidogenic phase and decrease to 0.05 for old landfills during methanogenic phase [25]. The low BOD_5 and BOD_5/COD values for stabilized leachate agreed with literature [9–11,26]. The concentration of TSS was 380 mg/L which indicates the presence of organic and inorganic solids. A considerable concentration of ammonia nitrogen was found which is attributed to the decomposition of nitrogenous substances in refuse and the release of soluble nitrogen from solid wastes [9]. A greater concentration of color was mainly contributed by the dissolved organics. These organic compounds may be present in the form of recalcitrant material mainly composed of humic-like substances. A low value of BOD_5 means low biodegradability while the presence of high concentration of NH_3-N indicates high leachate toxicity [27].

3.2. Experimental results of coagulation and flocculation

3.2.1. PACl as primary coagulant (optimum dosage and pH)

PACl showed its effectiveness when used as primary coagulant with the optimum dosage of 7.2 g/L. The removal efficiencies for COD, color and TSS were 55, 80 and 91%, respectively, as shown in Fig. 1 while the best pH for PACl as primary coagulant was 7.5 and the removal efficiencies for COD, color and TSS were 56, 83 and 84%, respectively, as shown in Fig. 2. The results agree with Ghafari et al. [7]. The removals of color, COD and TSS increased with an increase

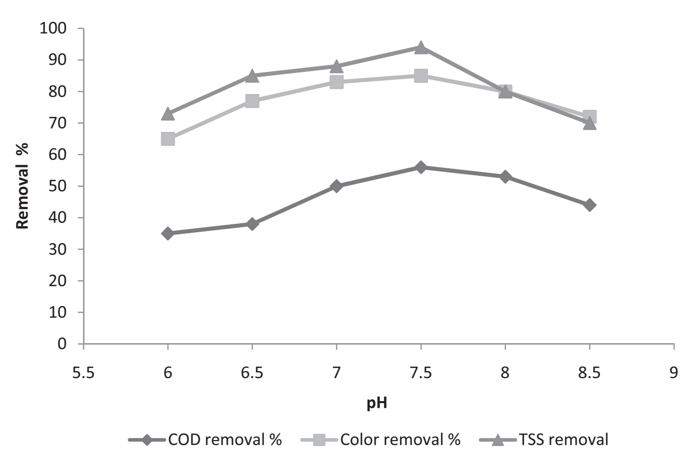

Fig. 2. Removal of COD, color and TSS at various pH values using 7.2 mg/L PACl as primary coagulant.

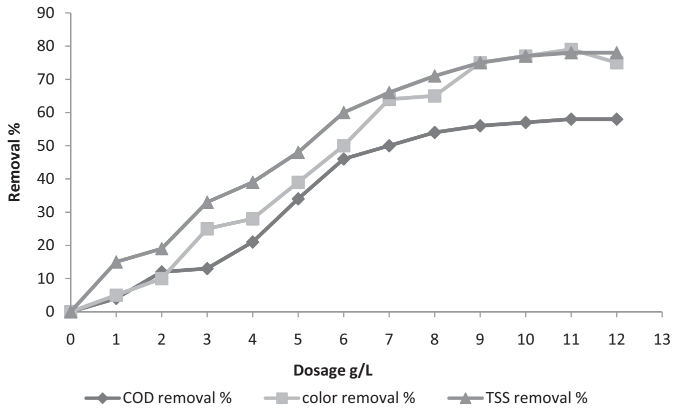

Fig. 3. Removal of COD, color and TSS at various dosages of alum as primary coagulant at pH 6.5.

in the concentration of PACl until the optimum coagulant dosage was reached. Excessive amount of coagulant causes re-stabilization of colloids and re-dispersion of the colloidal particles [9].

3.2.2. Alum as primary coagulant (optimum dosage and pH)

Alum showed effectiveness of being used as primary coagulant with the optimum dosage of 11 g/L and 58, 79 and 78% removals efficiencies for COD, color and TSS, respectively, as shown in Fig. 3. The optimum pH for alum as primary coagulant was 6.5 and the removal efficiencies for COD, color and TSS were 58%, 80% and 90%, respectively, as shown in Fig. 4. The results agree with Ghafari et al. [7].

3.2.3. Psyllium husk as primary coagulant aid (optimum dosage)

The result of the use of psyllium husk as primary coagulant aid was unsatisfactory with low removal efficiencies for COD, color and TSS due to the low zeta potential (−1.92 mV). The results of psyllium husk as primary coagulant aid are shown in Fig. 5. The optimum dosage of psyllium husk as primary coagulant aid was found to be 0.4 g/L and the removal efficiencies for COD, color and TSS were 17, 27 and 41%, respectively, which could be attributed to the mucilage component of the psyllium husk which attracted the particles to the gummy that was formed during the jar test. Color was found to have been removed due to the removal of the organic components of leachate and TSS were removed partially through the settlement of the SS and partially by the mucilage solution of the psyllium husk. Hence, the removal of COD, color and TSS was fairly low by only using psyllium husk when compared with using PACl and alum requiring it to be used as a coagulant aid.

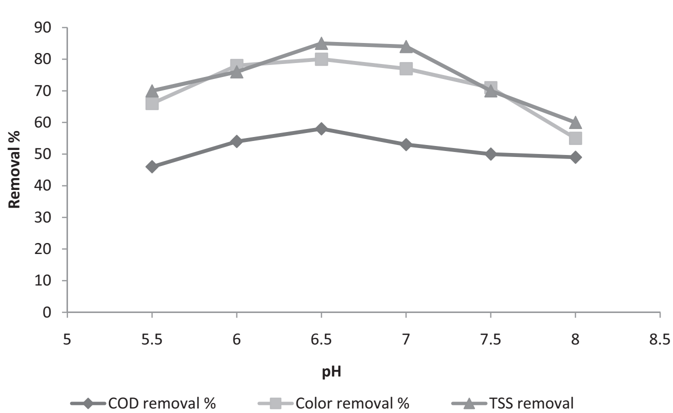

Fig. 4. Removal of COD, color and TSS at various pH values using 11 g/L alum as primary coagulant.

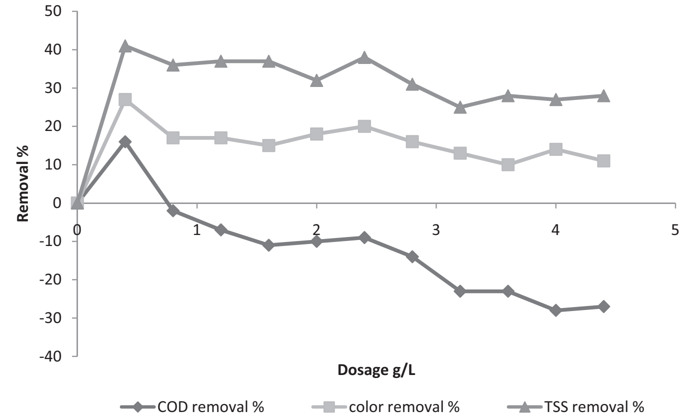

Fig. 5. Removal of COD, color and TSS at various dosages of psyllium husk as primary coagulant.

Table 4
Treated effluent characteristics.

Sample	COD (mg/L)	Color (Pt–Co)	TSS (mg/L)	Cost/L (RM/L)
A	890	880	25	0.030
B	950	500	5	0.003
C	17	1810	59	0.001
D	810	720	20	0.031
E	760	240	4	0.004

3.2.4. Alum as primary coagulant with psyllium husk as coagulant aid

Optimum dosage of psyllium husk (0.4 g/L) was added to different dosages of alum by fixing the same conditions of jar test for the rapid and slow mixing followed by settling for 2 h. The results of using psyllium husk with alum were better than the use of only alum in terms of reducing the dosage of alum and increase in the removal efficiencies. 10 g/L alum gave removals efficiencies of 63, 82 and 81%, for COD, color and TSS, respectively, as shown in Fig. 6.

3.2.5. PACl as primary coagulant and psyllium husk as coagulant aid

The optimum dosage (0.4 g/L) of psyllium husk was added to different dosages of PACl by fixing the same conditions of jar test for the rapid and slow mixing and the settling time. The results obtained by using the PACl with psyllium husk were better than using alum with psyllium husk. As seen in Fig. 7, using PACl with psyllium husk resulted in removal efficiencies of 64, 90 and 96% for COD, color and TSS, respectively, at an optimum dosage of 7.2 g/L.

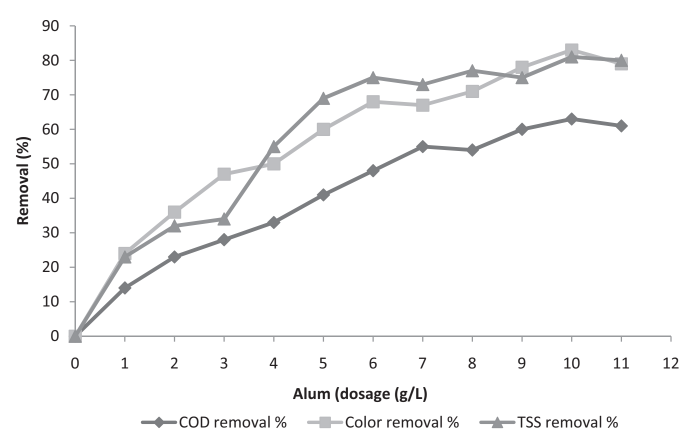

Fig. 6. Removal of COD, color and TSS at various alum and optimum psyllium husk dosage (0.4 g/L).

Table 5
Zeta potential reading before and after treatment.

Sample	Details	Zeta potential (mV)
A	Raw leachate	−46.1
B	After treatment at optimum pH and alum dosage	−39.1
C	After treatment at optimum pH and PACl dosage	−22.1
D	After treatment at optimum pH and psyllium husk dosage	−46.8
E	After treatment at optimum pH and alum + psyllium husk dosage	−30.2
F	After treatment at optimum pH and PACl + psyllium husk dosage	−16.2
G	Alum	+12.5
H	PACl	+34.8
I	Psyllium husk	−1.92

Theoretically, the optimal conditions of coagulation flocculation correspond in theory to a value of zeta potential equal to zero and PACl with highly positive zeta potential substantially neutralized the negative zeta potential of raw leachate while the mucilage solution of the psyllium husk as coagulant aid further assisted in the removal of COD, color and TSS. Since the charge neutralization is not the only mechanism governing the removal of pollutants, the theoretical range could be misleading in some cases. A variety of zeta potential range could exist depending on the type of pollutant.

3.3. Effect of dosage

The influence of different dosages of coagulants at different pH values for the removal of COD and color was further investigated. Results showed that the COD and color removal increased with an increase in coagulant dosage until it reached an optimum value. This could be attributed to the re-stabilization of colloidal particulates when coagulants were used at dosages in excess of the optimum value. The removal of COD and color was due to the removal of the particles that happened because of their attraction to the coagulants (PACl or alum) due to the positive zeta potential of these coagulants. Decolorization of leachate increased by increasing the dosages at all pH ranges studied. The addition of psyllium husk to PACl and alum increased the removal efficiency. For PACl in particular, the removal of COD and color were considerably increased (10%).

3.4. Effect of pH

The influence of pH (pH ranges between 5 and 9) on the reduction of COD, color and TSS was investigated without adding any coagulant. Chemical coagulation and precipitation is highly pH dependent. The addition of metal coagulants such as PACl and alum depressed the pH from being slightly alkaline for the untreated leachate to acidic level. Figs. 2 and 4 present the effect of pH on the performance of PACl for COD and color removals.

Experiments were subsequently conducted at different pH values employing the optimum dosage of PACl (7.2 g/L) and alum (1.1 g/L) with the aim of determining the optimum pH range. It was observed that the optimum pH range for PACl and alum was between 6.5 and 7.5 while better removals were obtained by using PACl which agrees with Aziz et al. [9]. The removals of COD, color and TSS by using PACl were 56, 83 and 94%, respectively, at pH 7.5 9 (Fig. 3) while the respective removals for COD, color and TSS using alum were 58, 80 and 84% as shown in Fig. 4. The results are in agreement with Ghafari et al. [7]. The pH of leachate remained unchanged after the addition of psyllium husk primarily due to its organic nature. Hence, pH adjustment was not required during the treatment process when psyllium husk was utilized as a primarily coagulant.

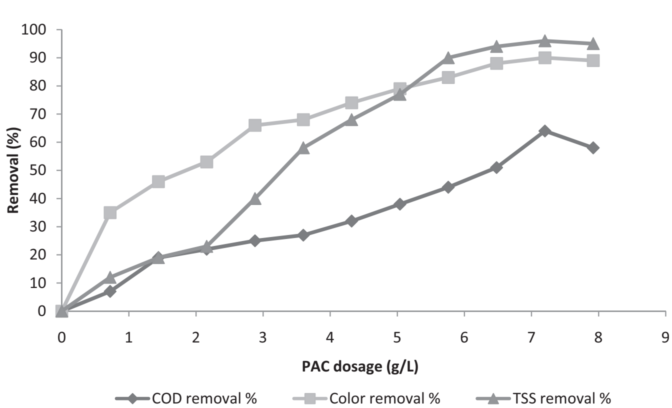

Fig. 7. Removal of COD, color and TSS at various PACl and optimum psyllium husk dosage.

3.5. Treated effluent characteristics

Optimization was carried out for 5 samples to compare the removal efficiencies of COD, color, ammonia nitrogen, TSS, BOD_5 and the treatment cost per liter of leachate. The samples were (A) leachate treated with alum (B) leachate treated with PACl (C) leachate treated with psyllium husk (D) leachate treated with alum and psyllium husk and (E) leachate treated with PACl and psyllium husk. The purpose was to find the significant coagulant and coagulant aid in terms COD, color, BOD_5, TSS, and ammonia nitrogen removal efficiency for each sample at optimum dosages and pH as shown in Table 4. The highest removals of COD, color and TSS were obtained when psyllium husk was added to the PACl. However, when PACl was used as coagulant, the removal of BOD_5 was 62% but it decreased to 38% when psyllium husk was added to PACl which could be attributed to the organic nature of psyllium husk which could increase the BOD_5 of the sample due to the release of carbohydrates from the solution of psyllium husk. The ammonia nitrogen removal was low as expected because coagulation–flocculation process are generally ineffective in ammonia nitrogen removal due to the positive surface charge of the ammonia nitrogen which prevents incident attraction between the coagulant and the ammonia nitrogen in the sample. The results agree with Makhtar et al. [28] who achieved merely 7% ammonia nitrogen removal by using PACl. Rough cost estimation was also carried out as given in Table 4. The cost estimation was based on the amount of coagulant used and it did not take into consideration the operational costs for the coagulation–flocculation process. The cost estimates were determined by determining the cost for each polymer per kg divided by the optimum coagulant dosage for each coagulant used. According to Table 4, the use of psyllium husk (C) reduced the cost while the cost of PACl in combination with psyllium husk as coagulant aid was comparable with PACl treatment only. Hence, natural coagulant could be used as coagulant aid to reduce the consumption of conventional main coagulant, i.e., PACl.

3.6. Zeta potential test

Analysis of zeta potential vs coagulant dosage results can be used to assess the effectiveness of alum and PACl and knowledge on zeta potential is important in terms of dosage adjustments to minimize the cost of treatment [29]. Zeta potential tests were carried out for the polymers before and after treatment in order to evaluate the effect of each polymer on the charge of particles. According to Table 5, the zeta potential for raw leachate was −46.1 mV while for the coagulant to be highly effective, the zeta potential is required to be more than +30 mV in order to neutralize and attract the particles to form flocs [30]. PACl was found to have higher zeta potential of +34.8 mV than alum and psyllium husk which might justify the higher removal of COD, color and TSS as primary coagulant. The surface charge of psyllium husk (−1.92 mV) indicates that the particles of psyllium husk were unsteady and could not make flocs when it was added as primary coagulant. However, when psyllium husk was added as coagulant aid with PACl, the zeta potential decreased from −22.1 mV for PACl to −16.2 mV for PACl and psyllium husk and when psyllium husk was added as coagulant aid with alum, the zeta potential reduced from −39.1 mV for alum to −30.2 mV for alum and psyllium husk.

4. Conclusions

The application of coagulation–flocculation process to semi-aerobic leachate collected from PBLS was examined in this study. Psyllium husk was not effective when used as primary coagulant because it had low surface charge and it had no effect on the pH. Compared to when PACl was used alone, an additional COD, color and TSS of 9, 10 and 2%, respectively, was recorded when PACl was used as primary coagulant and psyllium husk as coagulant aid. However, the percent removal using alum with psyllium husk was relatively lower and only 5, 4, 3% increment in COD, color and TSS removals was observed when compared to alum as the sole coagulant indicating that the psyllium husk was more effective as coagulant aid when used with PACl as coagulant.

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