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Original Research

Seasonal Characterization of Landfill Leachate and Effect of Seasonal Variations on Treatment Processes of Coagulation/Flocculation and Adsorption

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Abstract

The aim of this study was to characterize landfill leachate of Çorlu that was heavily industrialized, and to examine the application of coagulation-flocculation and adsorption for the treatment of this leachate. This deposited area is non-regulated and municipal and industrial wastes are deposited uncontrollably. The experiments were conducted to investigate the combined process of coagulation-flocculation and granular activated carbon (GAC) and waste metal hydroxide (WMH) sludge. In this study, ferric chloride and aluminium sulphate were tested as conventional coagulants, and lime was used for pH adjustment. The optimum working pH for the tested coagulants was 9.5. The optimum dosages were 2.4 g/L for $Al_2(SO_4)_3$ and $FeCl_3$. Among two of the coagulants $FeCl_3$ showed the highest COD removal efficiency (40%) and showed small seasonal variability according to optimum conditions. The adsorption experiments suggested that the optimum dosage of GAC was 18 g/L and WMH was 13 g/L, and the optimum contact time was 12 h. Under the optimum conditions of coagulation/flocculation/GAC adsorption and coagulation/flocculation/WMH adsorption, COD removal efficiencies were respectively 56-63% and 45-48%. Color removal efficiencies were 77-91% and 81-92%, respectively. Results of the adsorption study showed that these adsorbents could be used for only color removal for this landfill leachate.

Keywords: landfill leachate, coagulation/flocculation, adsorption, waste metal hydroxide, seasonal variations

Introduction

The leachates from municipal solid waste landfill sites cause many environmental problems. These areas are often defined as hazardous and heavily polluted zones [1-3]. Leachate from municipal landfills sites are defined as the aqueous effluents generated as a consecuence of rainwater percolation through wastes, chemical and biological processes in waste cells, and their inherent water content [3, 4]. Leachate composition is site- and time-specific, based on the characteristcs of landfill age, moisture level, diversity of wastes, and physico-chemical characteristics [5].

In general, municipal landfill leachate composition is complex [6]. There are more than 200 kinds of organic substances in the leachates [7]. Leachates may contain large amounts of organic matter, amonia-nitrogen, pathogenic microorganisms, heavy metals, and chlorinated organic and inorganic salts [1, 8]. Characteristics of a land-

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Table 1. COD removal efficiencies by coagulation-flocculation obtained in some studies.

Coagulant	Parameter	COD removal efficiency (%)
$FeCl_3$ or Al_2SO_4 (0.01-0.07 M)	Turbidity and COD	40-50
$FeSO_4$ (0.3 g/L Fe)	COD and TOC	70
$Ca(OH)_2$ (6 kg/m³)	COD, BOD, metals etc.	57
$Ca(OH)_2+Al_2SO_4$ (1.5+1 kg/m³)	COD and BOD	42
$FeCl_3$ veya Al_2SO_4 (0.1-1 gr/L)	COD, BOD, TOC	53
$Ca(OH)_2+ FeSO_4$ (0.5-4 ve 0-0.2 gr/L)	COD, BOD	39
$FeCl_3+Al_2SO_4$ (1-5 gr/L)	COD, BOD, color	75
$FeCl_3 \cdot 6H_2O$ (0.1-1 gr/L)	COD, color	24
$FeCl_3$ (0.8-1 gr/L)	TOC	38-48
$FeCl_3$ (0.2-1.2 gr/L)	COD	39
$FeSO_4$, $FeCl_2$, $FeCl_3$, Al_2SO_4 (40-200 mg/L metal)	COD, BOD, TOC	50

fill leachate can be represented by chemical oxygen demand (COD), total organic carbon (TOC), biochemical oxygen demand (BOD), pH, BOD/COD ratio, ammonium nitrogen (NH_3-N), turbidity, and heavy metals [8-10]. The properties of a landfill leachate highly depend on the age of the landfill [4, 5]. Typically old landfills produce leachate catalogued as stabilized and characterized by a relatively low COD (500-5,000 mg/L), slightly basic pH (7.5-8.5), and low biodegradibility (BOD/COD below 0.1) [4].

Generally, the single method is not efficient for environmental and economical treatment of leachate [11]. So many physical-chemical and biological treatment methods and combinations of them are used to treat such wastewaters. Stabilized leachate effluents are especially difficult to deal with and biological processes are totally inefficient. Generally, alternative technologies based on physical-chemical stages are required [4] and there will be many studies concerning the best available technology providing both maximum treatment efficiency and optimum operation cost [11]. To achieve satisfactory removal of refractory pollutants from the leachate, several types of physico-chemical treatments have been employed worldwide. There is little research about the combined coagulation-flocculation and adsorption process applied in treating especially stabilized landfill leachate [2].

Coagulation-flocculation is an essential and simple process in water, wastewater, and landfill leachate treatment [2, 11]. Several studies have reported on this process for the treatment of landfill leachate [4, 12]. In this process aluminium sulfate ($Al_2(SO_4)_3$), ferrous sulfate ($FeSO_4$), and ferric chloride ($FeCl_3$) were commonly used as coagulants [1, 2, 4]. This method results in removal efficiency of COD, TOC, or color in the range of 24-70% (Table 1) [4].

Adsorption is a process by which a pollutant is transferred from the liquid phase to the surface of a solid. Coagulation-flocculation and adsorption processes have drawbacks when used as single process such as sludge production, regeneration, and high consumption of coagulants and adsorbents, etc. So their combined processes can prevent the drawbacks of each single process [2].

The aim of this study was to investigate the combined processes of coagulation-flocculation and adsorption of landfill leachate as pretreatment method. Treatment performance was evaluated in terms of organic matter removal expressed by COD and color. In the coagulation-flocculation experiments $FeCl_3$ and $Al_2(SO_4)_3$ were used as coagulants while granular activated carbon (GAC) and waste metal hydroxide (WMH) sludge were used as adsorbents in adsorption studies. WMH was generated by metal precipitation in the effluents of electroplating industries. Most of these sludges have surface areas over 100 m²/g and have a continuum of micro- and macro-pores [13]. In the literature there are few studies that use such waste metal hydroxide sludges as potential adsorbents [14-18]. The effect of pH, adsorbent dose, and contact time on the adsorption of COD and color onto WMH sludge and GAC were investigated using batch experiments. This study also discusses adsorption isotherms on WMH sludge and GAC.

Material and Methods

Site Description and Sampling Period

Leachate samples were collected from the sanitary landfill site of Çorlu, which has been in operation since 2004. It is located northwest of Çorlu and covers a surface of about 6 ha. The landfill receives approximately 110 tons of domestic and industrial solid wastes per day. Since the landfill was not properly lined with clay or other synthetic geomembrane, the exact quantity of produced leachate was not known. At the lowest point of the landfill, leachate exists on the surface, forming an artificial pond.

Samples used for characterization study were taken seasonally. The samples taken in spring and summer were used for coagulation-flocculation and adsorption study. The samples were collected from actual leachate streams in the Çorlu dumping site and analyzed for various physico-chemical characteristics. In another study conducted by Güneş et al. [19] BOD_5/COD, EC_{50} (to *Vibrio fischeri*), and TU (toxicity unit – calculated by the equation of 100/EC_{50}-) values were found as follows 0.19, 49.5, and 2.02 for the same landfill leachate [19].

Analytical Methods

Samples were collected in 20 L plastic carboys transported to the laboratory and stored at 4ºC. Chemical parameters for wastewater samples included pH, dissolved oxygen (DO, mg/L), total dissolved solids (TDS,mg/L), and temperature (T, ºC) determined in situ using appropriate sensors, biochemical oxygen demand (BOD_5, mg/L), chemical oxygen demand (COD, mg/L), total phosphate (T-P, mg/L), total sulfide (T-S, mg/L), ammonium nitrogen (NH_4-N, mg/L), and total suspended solids (TSS, mg/L) determined according to standard methods [20]. The color was measured at a wavelength corresponding to the maximum absorbance (µmax of 425 nm for leachate) using a thermospectronic AQUAMATE spectrometer.

Investigated Treatment Procedures

Coagulation-Flocculation

Coagulation-flocculation and sedimentation were carried out in an ISCO-JF4 conventional jar test apparatus at room temperature. The experimental process consisted of three stages: the initial rapid mixing stage took place for 1 min at 180 rpm, followed by slow mixing for 15 min at 50 rpm, and the sludge was left to settle for 30 min. After the settling period, the supernatant was withdrawn from the beakers and used for chemical analyses and for adsorption study. The treated leachates were characterized measuring the COD and color. The pH of the samples was adjusted to the desired levels by the addition of appropriate amounts of NaOH, lime ($Ca(OH)_2$), and H_2SO_4. But it was observed that when NaOH were used for pH adjustment the coagulation was not efficient so $Ca(OH)_2$ was chosen for pH adjustment instead of NaOH. As the use of coagulants such as $Al_2(SO_4)_3$ or $FeCl_3$ is a common practice to coagulate the suspended solids present in wastewater, these coagulants were used in this study. Samples of spring and summer periods were used in coagulation-flocculation experiments in order to evaluate the effect of seasonal variations on coagulant doses and optimum pH. COD and color of the raw and coagulated-flocculated samples were analyzed to evaluate the treatment performances.

Adsorption

The supernatant from the coagulation-flocculation process with $Al_2(SO_4)_3$ and $FeCl_3$ was subjected to the adsorption test with GAC and WMH. The chemical composition of the WMH was measured using ICP spectroscopy. This WMH contained 45.36% Al, 0.31% Mg, 0.26% Na, 0.19% Fe, 0.0032% Cr, 0.002% Cu, 0.0034% Zn, and 0.0004% Mn. In the BET procedure, the surface area of GAC and WMH were found as 825.132 m^2/g and 130 m^2/g, respectively (Quantachrome Instruments Nova 4000E). The batch sorption studies were carried out by shaking a series of bottles containing different amounts of waste metal hydroxide sludge of Al(III) hydroxide (WMH) and activated carbon (GAC) dosage (1, 2, 3, 4, and 5 gr) in 300 mL of leachate samples. The samples were stirred at room temperature at 200 rpm for 12 h. Then samples were centrifuged (with CN180 Nüve fuge) at 3500 rpm for 5 min. The supernatant liquid was analyzed for color and COD.

The amount of color and COD adsorbed was calculated from the following equation:

$$q_e=V(C_0-C_e)/W \quad (1)$$

...where q_e is the amount adsorbed COD (mg/g); C_0 and C_e are the initial and equilibrium COD and concentrations and color in the solution (mg/L and abs), respectively; V is the volume (L); and W is the mass of WMH and GAC (g).

Adsorption equilibrium studies were employed to determine the adsorption capacity of the adsorbents using Langmuir and Freundlich isotherm models.

Results and Discussion

Seasonal Leachate Characteristics

The seasonal characteristics of raw leachate from Çorlu landfill were presented in Table 2. As seen from Table 2, during the rainy season in the winter several pollutants were diluted by rain water and so their concentrations decreased.

The main characteristics of this leachate were COD in the range of 1400-4845 mg/L, conductivity 25.1-36.1 mS/m, and pH 8.5-9. As can be seen from Table 2, pH was above 7, which indicated the partially stabilized nature of the leachate [21]. According to study of Güneş et al. [19], this leachate exhibited a BOD/COD ratio of 0.19, which indicated poor biodegradability [22, 23]. According to the BOD/COD ratio of <0.1, 0.1-0.5, and >0.5, leachate can be classified as stabilized, intermediate, or fresh, respectively [21].

Total phosphorus content in partially stabilized samples was lower than 20 mg/L [1]. As given in Table 2, these leachate samples had less than 20 mg/L total phosphorus content. It is also evident from Table 2 that NH_4^+ was present in high amounts and always between the reported ranges [22]. If NH_4^+ is present to a high degree, the leachate cannot be treated by conventional biological processes [22]. And it is also apparent that the collected samples may be classified as intermediate according to NH_4^+ value [10]. As seen from Table 2, this leachate also appeared to be saline because of high conductivity.

Table 2. Seasonal leachate characteristics of landfill leachate.

Pollutant	Autumn (November)	Winter (January)	Spring (April)	Summer (July)
pH	7.7	7.6	7.1	7.8
Temperature (°C)	13.5	1	15	25
Conductivity (mS/m)	36.1	25.4	33.2	29.1
Color (425 nm, abs)	2.101	0.332	2.14	1.696
COD (mg/)	2580	1400	4845	4400
Dissolved COD (mg/L)	2160	1060	4349	3950
Alkalinity (mg $CaCO_3$/L)	920	800	250	550
NH_4-N (mg/L)	15.5	25.2	52.7	129
NO_2-N (mg/L)	3.4	13.6	<0.1	<0.1
NO_3-N (mg/L)	324	235.5	28.8	<5
TSS (mg/L)	1580	1313	660	920
T-P (mg/L)	12.3	9.5	10.2	9.8
T-S (mg/L)	1.5	<0.01	0.9	0.9

Table 3. Results of optimum pH study.

pH	$FeCl_3$ (mg/L)	COD Removal Rate (%)	$Al_2(SO_4)_3$ (mg/L)	COD Removal Rate (%)
Spring				
6.5	200	6	400	5
7.5	200	9	400	10
8.5	200	14	400	13
9.5	200	20	400	18
10.5	200	20	400	18
Summer				
6.5	400	18	400	14
7.5	400	20	400	27
8.5	400	29	400	32
9.5	400	32	400	34
10.5	400	32	400	33

This deposited area is non-regulated and municipal and industrial wastes are deposited together. So obtained results were interpreted with some difficulty.

Results of Coagulation/Flocculation Processes

Coagulation is acceptable as a successful pretreatment process in water and wastewater treatment. This process is used for removal of the waste materials in suspended or colloidal form, which doesn't settle out spontaneously or may settle over a very long period of time. Coagulation-flocculation involves the addition of chemicals to alter the physical state of dissolved and suspended solids, and then facilitates their removal by sedimentation [1, 24]. The coagulation process destabilizes colloidal particles by the addition of a coagulant. To increase particle size, coagulation is usually followed by flocculation of the unstable particles into bulky floccules so that they can settle more easily. The general approach for this technique includes pH adjustment and involves the addition of ferric/alum salts as the coagulant to overcome the repulsive forces between the particles [25, 26].

The results of optimum pH study can be seen from Table 3. The results shown for COD refer to the effluent after this treatment. As can be seen from Table 3, the optimum pH was 9.5 in terms of COD removal. As known, pH influences the nature of produced polymeric metal species that will be formed as soon as the metal coagulants are dissolved in water [27]. The influence of pH on chemical coagulation/flocculation may be considered as a balance of two competitive forces:

(1) between H and metal hydrolysis products for interaction with organic ligands

(2) between hydroxide ions and organic anions for interaction with metal hydrolysis products [27]

At low pH values, hydrogen ions compete metal hydrolysis products for organic ligands, hence poor removal rates occur and some of the generated organic acids will not precipitate. At higher pH values, such as those values examined in the current investigation with the addition of lime, hydroxide ions compete with organic compounds for metal adsorption sites, and the precipitation of metal-hydroxides mainly occurs by co-precipitation [1].

After the optimum pH study, the optimum coagulant dosages were studied. Dosages ranging 0.4-3.2 g $Al_2(SO_4)_3$/L and 0.8-3.6 g $FeCl_3$/L were tested. For two of the coagulants dosages of 2.4 g/L were obtained as optimum value. After the maximum dose value of 2.4 g/L, no appreciable improvement in the removal efficiency is observed by increasing the coagulant dosage. Results were given in Figs. 1 and 2.

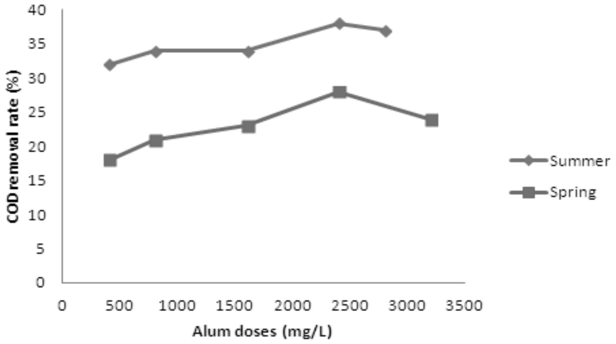

Fig. 1. COD removal rates according to alum doses and seasons.

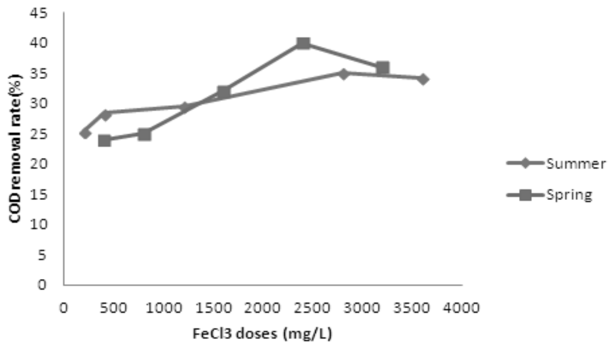

Fig. 2. COD removal rates according to ferric chloride doses and seasons.

Tablo 4. COD, color, and NH_3-N removal efficiencies after coagulation-flocculation studies.

Parameter	Removal efficiency (%)	
	Spring (after using of $FeCl_3$)	Summer (after using of $Al_2(SO_4)_3$)
COD (mg/L)	40	38
NH_3-N (mg/L)	80	57
Color (abs)	87	67

As can be seen from the figures, COD removal efficiencies changed depending on coagulant dosages and seasonal variations. The COD removal efficiencies, at the optimum dosage of alum, were found to be 28% in spring and 38% in summer. Considering optimum ferric chloride, COD removals were found to be 40% in spring and 35% in summer. According to these results it could be said that COD removal efficiencies show seasonal variations when $Al_2(SO_4)_3$ is used as coagulant. When $FeCl_3$ was used as coagulant in spring and summer almost similar results were obtained according to COD removal efficiencies. From these results it could be said that to control seasonal variations for COD removal efficiencies the use of $FeCl_3$ is more appropriate.

Besides COD removal efficiencies, the NH_4-N and color removal efficiencies were determined at optimum conditions. The results are given in Table 4. As can be seen in Table 4, NH_3-N was also removed at pH 9.5 (optimum pH of the coagulation-flocculation experiments) due to the ammonia stripping. But it can be concluded that for more efficient ammonia removals, there must be better aeration conditions and longer contact time. Because color is planned to take part in discharge standards, color removal efficiency also was evaluated in the study. Color removal efficiencies were found as 87% for $FeCl_3$ and 67% for $Al_2(SO_4)_3$. It can be seen from Table 4 that, according to removal efficiencies of COD, NH_3-N, and color, $FeCl_3$ was more suitable.

Results of Adsorption Processes

The treated leachate with $FeCl_3$ (in spring) and $Al_2(SO_4)_3$ (in summer) were subjected to adsorption by GAC and WMH. In the adsorption study the pH was adjusted to 7 according to the preliminary experiments (data not shown). The results of the adsorption studies are delineated in Figs. 3 and 4 for GAC and WHM sludge, respectively. As can be seen from the figures, 6 h was sufficient for equilibrium [2].

Since equilibrium concentrations for adsorbents were highly variable for COD results they were calculated taking

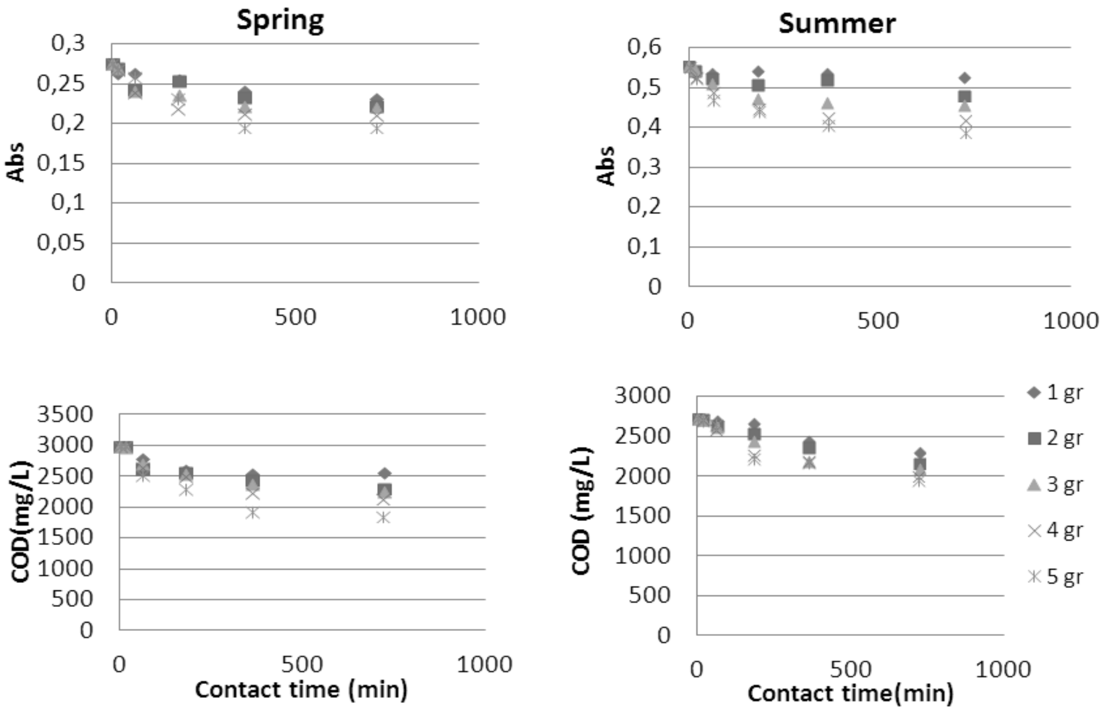

Fig. 3. COD and color abatements obtained for GAC at different doses and contact times.

Table 5. COD and color removal efficiencies of GAC and WMH for spring and summer samples.

	COD removal efficiency (%)	Color removal efficiency (%)
Spring samples		
After GAC	38	29
After WMH	13	36
Summer samples		
After GAC	29	30
After WMH	11	43

into account color removal. It was observed that the concentration of color decreased with an increase in the dosage of GAC and WMH, and it got stable when the dosage was more than 18 g/L for GAC and 13 g/L for WMH. At these dosages COD and color removal efficiencies are given in Table 5 for spring and summer samples. It can be seen from Table 5 that GAC is more efficient for COD removal, and WMH is more efficient for color removal. However, based on the obtained removal efficiencies it can be also concluded that these adsorbents are not very effective for the treatment of landfill leachate.

For the adsorption isotherm study, two important models, Langmuir and Freundlich, were tested. It was found from the results that only the Freundlich isotherm model described and predicted COD and color adsorption on GAC and WMH. Freundlich isotherm assumes that the uptakes of adsorbate occur on a heterogeneous surface by multilayer adsorption and the amount of adsorbate adsorbed increases infinitely with an increase in concentration [28-30]. The Freundlich isotherm model can be expressed by Eq. (2) [2]:

$$log q_e = \frac{1}{n} log c_e + log k_f \tag{2}$$

...where q_e is the adsorbed amount (mg/g), c_e is the equilibrium organic concentration (mg/L), and n and k_f are the Freundlich constants. The regression Freundlich equation and constants which indicated the Freundlich model was able to apply for predicting the adsorption capacity of GAC and WMH were presented in Table 6 and Figs. 5 and 6. It can be observed that R^2 values for especially summer samples treated with alum of the plots are good.

As seen from Table, 6 k_f values that represent a specific capacity, an indicator of sorption capacity at a specific solution phase concentration [31, 32], are very low for COD removal for both adsorbents in spring and summer samples. Conversely for color removal, sorption capacities are higher and it can be concluded that these adsorbents can be used for only color removal. $1/n$ values show adsorption intensity and denote efficiency of adsorbents. Values of $1/n$ less than 1 show the favorable nature of adsorption on the adsorbents [32, 33]. In this study the adsorption intensities are found higher than 1. This result indicates concave isotherms [31]. There are similar results for leachates that have $1/n$ values ranging from 1 to 4 [31, 34]. If $1/n$ value is high it shows that the adsorbent used will lose its capacity more rapidly than the others [33]. So $1/n$ values found in this study show that these adsorbents will lose their capacity rapidly, especially when used for COD removal both in summer and in spring samples.

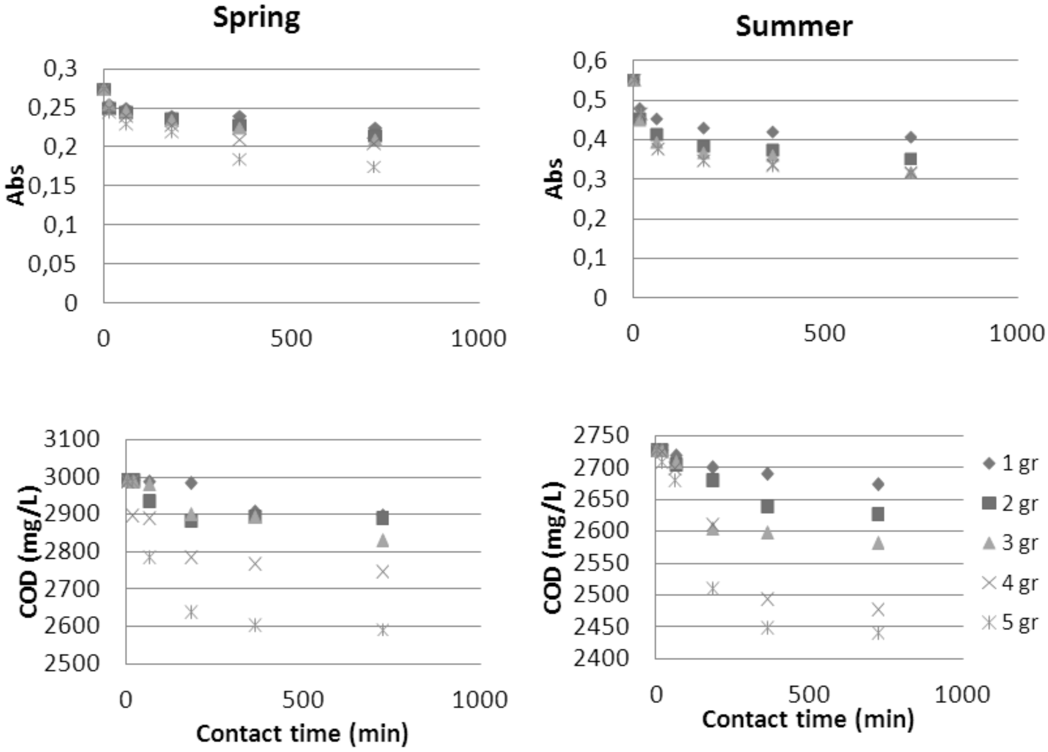

Fig. 4. COD and color abatements obtained for WMH at different doses and contact times.

Table 6. Isotherm constants for the Freundlich isotherms.

	Freundlich constants of adsorbents					
	GAC			WMH		
Samples	k_f	$1/n$	R^2	k_f	$1/n$	R^2
Spring COD	1.17.10-10	3.62	0.80	7.4.10-31	9.32	0.61
Summer COD	3.5.10-25	7.99	0.98	1.10-42	12.89	0.85
Spring color	21.7	4.76	0.69	16	4.47	0.60
Summer color	2.33	3.65	0.92	4.94	4.66	0.89

Table 7. COD and color removal efficiencies of coagulation/flocculation and adsorption for spring and summer samples.

Seasonal samples and treatment alternatives	COD removal efficiency (%)	Color removal efficiency (%)
Spring samples		
$FeCl_3$ + GAC	63	91
$FeCl_3$ + WMH	48	92
Summer samples		
$Al_2(SO_4)_3$ + GAC	56	77
$Al_2(SO_4)_3$ + WMH	45	81

Total Removal Efficiencies after Coagulation/Flocculation and Adsorption

The effects of seasonal variations, types of coagulants, and adsorbents on overall treatment efficiency are given in Table 7. COD removal efficiencies were between 56-63% for coagulation/flocculation + GAC and 45-48% for coagulation/flocculation + WMH. As it can be seen from Table 7 in spring and in summer samples, COD and color removal efficiencies were the highest after using $FeCl_3$+GAC. For only color removal GAC and WMH could be used after $FeCl_3$ treatment. It could be concluded that $FeCl_3$+GAC is the most suitable coagulant and adsorbent for this leachate treatment.

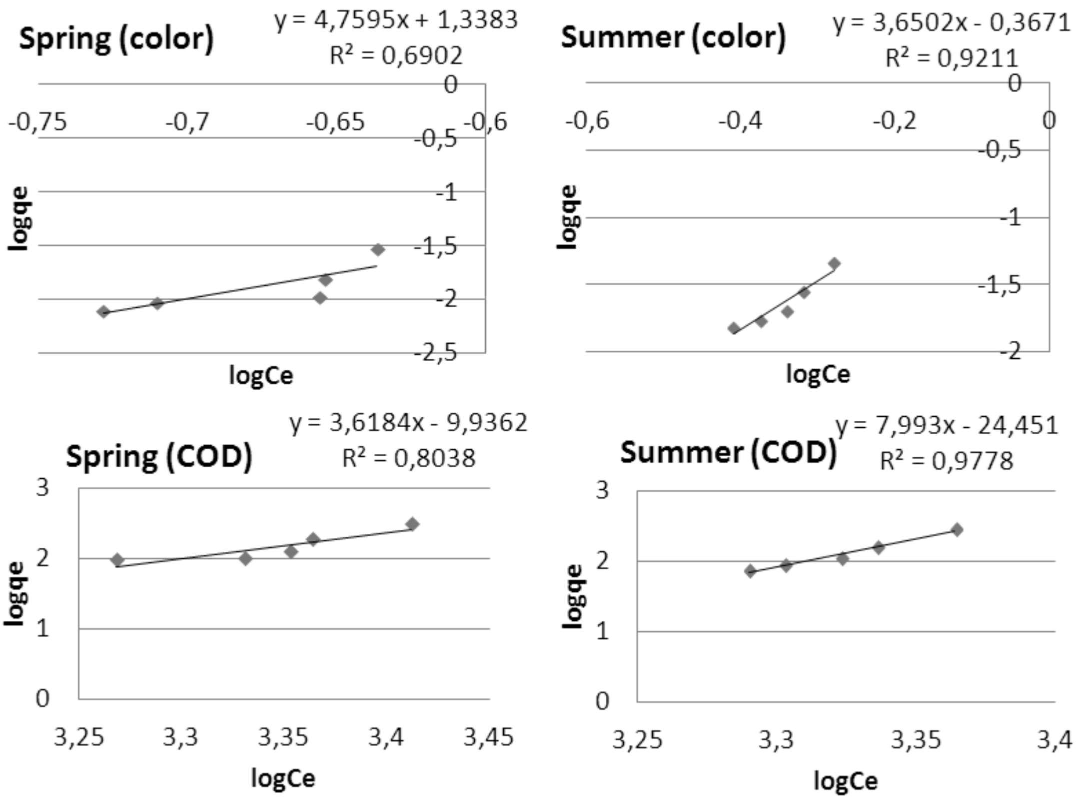

Fig. 5. Freundlich isotherm plots for the adsorption of COD and color on GAC.

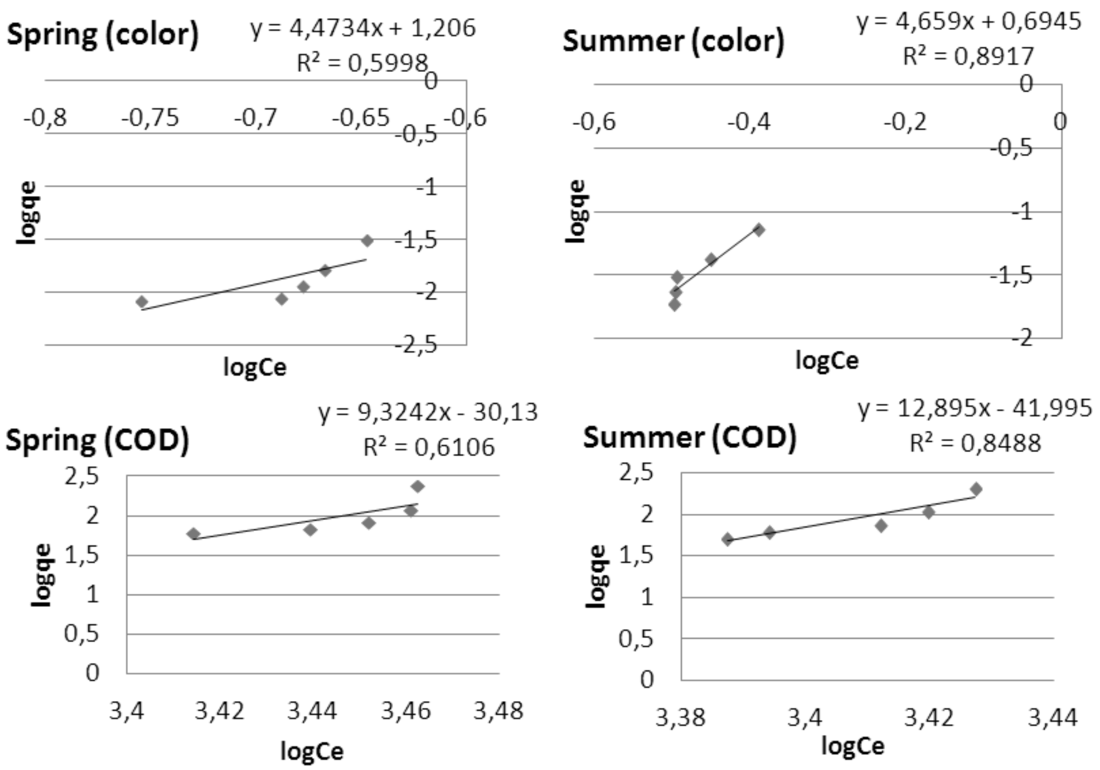

Fig. 6. Freundlich isotherm plots for the adsorption of COD and color on WMH.

Conclusion

In this work the main characteristics of a municipal landfill leachate and treatment alternatives depending on seasonal variations were studied. $FeCl_3$ and $Al_2(SO_4)_3$ were used as coagulants and granular activated carbon (GAC) and waste metal hydroxide (WMH) sludge from a factory in Corlu were used as adsorbents. The results of the study are summarized below:

- The main characteristics of this leachate are COD in the range of 1,400-4,845 mg/L, conductivity 25.1-36.1 mS/m, and pH 8.5-9, which indicates the partially stabilized nature of the leachate.
- In coagulation/flocculation study optimum pH was found as 9.5 and for two of the coagulants doses of 2.4 g/L were obtained as optimum value, and at this dosage treatment efficiencies were found as almost equal (38-40%).
- For spring and summer samples when $FeCl_3$ was used as coagulant almost similar results were obtained according to COD removal efficiencies. So controlling seasonal variability of COD removal efficiencies using $FeCl_3$ is more appropriate. It also was observed from the results that NH_3-N removal was also achieved because of ammonia stripping at the optimum pH of 9.5.
- The treated leachate with ferric chloride (in spring) and alum (in summer) were subjected to adsorption by GAC and WMH. Equilibrium concentrations were obtained as 18 g/L for GAC and 13 g/L for WMH. GAC was more efficient for COD removal, and WMH was more efficient for color removal.
- The overall treatment efficiency in terms of COD were between 56-63% for coagulation/flocculation + GAC and 45-48% for coagulation/flocculation + WMH whereas color removal efficiencies were 77-91% and 81-92%, respectively. Obtained results showed that both GAC and WMH could be used for only color removal.
- Equlibrium study showed that the Freundlich isotherm was described and predict the GAC and WMH adsorption for COD and color. 1/n values showed that adsorbents will lose their capacity rapidly, especially when used for COD removal both in summer and in spring samples. kf values were very low for COD removal for both adsorbents and higher for color removal in spring and summer samples. So it can be concluded that these adsorbents can be used for only color removal for this landfill leachate.

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