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Integrated chemical coagulation with natural base coagulant – electro-proxone process and ultrafiltration membrane for treatment of compost leachate

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ABSTRACT

Guilan Province compost leachate was treated utilizing a hybrid process including chemical treatment followed by electro-proxone and ultra filtration (UF). The effects of different parameters including ratio of natural / chemical coagulant (1:5, 1:15, 1:25), pH (5-8), time (10-90 min), ozone dose (10-90 mg L-1), current intensity (50-500 mA), TMP (0.5-3.5 bar) and temperature (20-50 °C) on COD and turbidity removal were investigated. At optimum conditions of the coagulation process (Salvia hispanica mucilage/FeCl3 = 1:15 and pH = 6.5), 36.19 % and 56.9 % removal efficiencies were obtained for COD and turbidity, respectively. Effluent from the chemical treatment went through. Results from the electro-proxone process showed that by increasing the current intensity from 50 to 350 mA, COD removal efficiency reaches form 14 % to 79 %. By changing O3 concentration from 10 mg L-1 to 90 mg L-1, COD reduction increased and reached from 29 % to 84 %. UF reactor was used for final polishment of the treated wastewater at the optimum TMP of about 1.5 bar and pH = 7.5. By increasing the temperature from 20 °C to 40 °C, permeation flux increased from 76.7 Lm-2 h-1 to 104.1 Lm-2 h-1. However, following the increase of the permeation flux, the removal efficiency of COD and turbidity decreased slightly to 54 % and 90 %, respectively. This study showed that the employed hybrid method is capable of treating the hardly biodegradable compost leachate and obtaining the environmental standards.

Keywords: Compost leachate, E- proxone, *Salvia hispanica* mucilage, Wastewater treatment.
Article type: Research Article.

INTRODUCTION

Composting is one of the most cost-effective methods for treatment of municipal or agriculture solid waste in human societies. However, the management and treatment of leachate produced in composting facilities is a major challenge (Mokhtarani *et al.* 2012; Shu *et al.* 2016). Some various parameters such as temperature, waste composition, climate condition, moisture content and so on, can affect leachate characteristics. The leachate has a high concentration of organic and inorganic matters, heavy metals, xenobiotic and aromatic compounds (Hasar *et al.* 2009; Dolar *et al.* 2016) and its discharge into the receiving resources without proper treatment will lead to severe environmental damages (Wang *et al.* 2012). A variety of common physicochemical methods, such as, coagulation-flocculation process (Smaoui *et al.* 2016; Smaoui *et al.* 2019), chemical oxidation (Amr & Aziz 2012; Zhang *et al.* 2014), chemical precipitation (Kurniawan *et al.* 2006), air stripping[3], adsorption with activated carbon (Li *et al.* 2010), membrane (Pirbazari *et al.* 1996; Renou *et al.* 2009; Dolar *et al.* 2014) and biological processes (Kennedy & Lentz 2000; Im *et al.* 2001) have been used for leachate treatment. Biological processes are cost-effective alternatives for the treatment of wastewater, among which treatment methods based on suspended biomass have shown high potential for organic carbon and nutrient removal (Laitinen *et al.* 2006). However, some problems such as sludge settleability, the need for large reactors, biomass recycling and settling tanks make operation of the process difficult. Furthermore, in high strength and industrial wastewater with high concentration of non-biodegradable and inorganic compounds, the biological process alone is not efficient enough (Lin & Chang 2000; Abood *et al.* 2014). The coagulation-flocculation process is widely used in the treatment of

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municipal solid waste landfills leachates and pretreatment, because of its simplicity and cost-effective benefits (Liu *et al.* 2012; Rui *et al.* 2012). In this process negatively-charged colloids are neutralized by cationic hydrolysis products. Afterwards, these impurities accumulate in the form of amorphous hydroxides in the flocculation process and precipitate (Yoo *et al.* 2001; Ishak *et al.* 2018). Some coagulants including ferrous sulfate, poly-aluminum chloride (PAC), ferric chloride and aluminum sulfate (alum) are generally used in coagulation–flocculation process (Amor *et al.* 2015). Despite the advantages of the coagulation-flocculation process, it has some drawbacks such as adverse effects on human health, relatively high costs, and the large volume of sludge production (Rasool *et al.* 2016). The idea of using natural-based coagulants was immerged to overcome some of these limitations (Aziz *et al.* 2018). The advantages of coagulants include: cost-effectiveness, non-toxicity, biodegradability and low sludge generation (Awang & Aziz 2012; Adewuyi & Adewumi 2018). These kinds of coagulants can be produced from microorganisms, animals or plant.

Furthermore, due to the economic situation, using local productions is important for developing countries (Freitas *et al.* 2015). Utilization of these bio-based coagulants for treatment of wastewater is a significant progress in environmentally friendly technologies (Al-Hamadani *et al.* 2011). In various studies the application of natural coagulants for the treatment of various types of wastewater such as landfill leachate (Rasool *et al.* 2016; Adewuyi & Adewumi 2018), dye wastewater (Freitas *et al.* 2015; Shamsnejati *et al.* 2015), pulp and paper wastewater (Subramonian *et al.* 2014) and heavy metals effluent (Shan *et al.* 2017) has been reported. Membrane technology is becoming increasingly important in wastewater treatment. With the help of ultra/microfiltration it is possible to remove particles, colloids and macromolecules, so that wastewater can be disinfected in this way. Based on the pressure driving force of the operation, membrane process can be classified into: microfiltration, ultrafiltration, nanofiltration, and reverse osmosis (Bohdziewicz *et al.* 2001). For industrial applications, membranes should provide enough mass transfer area to process large amounts of feed stream. The selected membrane requires to have high selectivity (rejection) properties for certain particles. It should resist fouling and have high mechanical stability. It also needs to be reproducible and to have low manufacturing costs. The main modeling equation for the dead-end filtration at constant pressure drop is represented by Darcy's law (Judd 2010):

$$\frac{dV_P}{dt} = Q = \frac{\Delta p}{\mu} A \left(\frac{1}{R_m + R} \right) \quad (1)$$

where V_p and Q are the volume of the permeate and its volumetric flow rate respectively, μ is dynamic viscosity of permeating fluid, A is membrane area, R_m and R are the respective resistances of membrane and growing deposit of the foulants. R_m can be interpreted as a membrane resistance to the solvent (water) permeation. This resistance is a membrane intrinsic property and is expected to be fairly constant and independent of the driving force, Δ_p. R is related to the type of membrane foulant, its concentration in the filtering solution, and the nature of foulant-membrane interactions (Mulder 2012). Darcy's law allows for calculation of the membrane area for a targeted separation at given conditions (Judd 2010; Mulder 2012). Membrane technologies such as nanofiltration (NF) and reverse osmosis (RO) were extensively used in the last decade for treatment of landfill leachate (Bohdziewicz *et al.* 2001; Di Palma *et al.* 2002; Dolar *et al.* 2016). Nevertheless, some operation problems, such as the flux decline due to fouling and as a result concentration polarization, redused its suitability as a single process for landfill leachate treatment (Bohdziewicz *et al.* 2001; Cui *et al.* 2018). Electro-oxidation (EO), also known as anodic oxidation, is a technique used for wastewater treatment, mainly for industrial effluents, and is a type of advanced oxidation process (AOP; Lin & Chang 2000). The most general layout comprises two electrodes, operating as anode and cathode, connected to a power source. When an energy input and sufficient supporting electrolyte are provided to the system, strong oxidizing species are formed, which interact with the contaminants and degrade them. The refractory compounds are thus converted into reaction intermediates and, ultimately, into water and CO_2 by complete mineralization (Li *et al.* 2013; Wang *et al.* 2015).

Electro-oxidation has been applied to treat a wide variety of harmful and non-biodegradable contaminants, including pesticides (Jaafarzadeh *et al.* 2017), drugs (Wang *et al.* 2018; Zheng *et al.* 2018), dyes (Bakheet *et al.* 2013) and landfill leachate (Cui *et al.* 2018). Due to its relatively high operating costs, it is often combined with other technologies, such as biological remediation (Li *et al.* 2013). Organic pollutants during electrochemical oxidation can be degraded by two ways: indirect oxidation by mediators (e.g., HClO, $H_2S_2O_8$, and H_2O_2) that are generated in situ in solutions based on the following reactions and direct oxidation at the anode surface (Barrera-Díaz *et al.* 2014):

$$Cl_2 + H_2O \leftrightarrow HOCl + H^+ + Cl^- \quad \textbf{(2)}$$

$$HOCl \leftrightarrow H^+ + OCl^- \quad \textbf{(3)}$$

$$6HOCl + 3H_2O \rightarrow 2ClO_3 + 4Cl^- + 12H^+ + 1.5O_2 + 6e^- \quad \textbf{(4)}$$

$$2H_2SO_4 \leftrightarrow H_2S_2O_8 + 2e^- + 2H^+ \quad \textbf{(5)}$$

$$HSO_4^- + {}^{\circ}OH \rightarrow SO_4^{-\bullet} + H_2O \quad \textbf{(6)}$$

$$SO_4^{2-} + {}^{\bullet}OH \rightarrow SO_4^{-\bullet} + OH^- \quad \textbf{(7)}$$

$$SO_4^{-\bullet} + SO_4^{-\bullet} \rightarrow S_2O_8^{2-} \quad \textbf{(8)}$$

$$H_2O_2 + e^- + H^+ \leftrightarrow H_2O + {}^{\bullet}OH \quad \textbf{(9)}$$

$$RH + {}^{\bullet}OH \leftrightarrow RH + H_2O \quad \textbf{(10)}$$

$$R^{\bullet} + O_2 \leftrightarrow ROO^{\bullet} \quad \textbf{(11)}$$

$$ROO^{\bullet} + RH \leftrightarrow ROOH + R^{\bullet} \quad \textbf{(12)}$$

In this study, for treatment of Guilan Province compost leachate a hybrid processes consisting of plant-derived coagulant (*Salvia hispanica*)/ electro-peroxone process/ membrane reactor were used.

MATERIALS AND METHODS

Landfill leachate

The leachate was collected from Guilan municipal waste compost facilities in Rasht (north of Iran). General characteristics of the raw leachate are shown in Table 1.

Table 1. The general characteristics of the raw leachate produced in Guilan compost plant.

Parameter	Value (mg L^{-1})	Parameter	Value (mg L^{-1})
pH	5 – 6.5	Mn	1.42 ± 0.05
COD	6000 - 33000	Cu	0.335 ± 0.02
BOD_5	2100 - 17000	Zn	2.12 ± 0.1
TDS	4200 ± 200	Pd	0.09
TSS	3650 ± 150	Cd	1.02 ± 0.03
SS	5300 ± 200	Cr	1.16 ± 0.2
Oil &Grease	2460 ± 100	Ni	0.573 ± 0.1
NH_4^+	180 ± 15	Ca	146 ± 10
PO_4^{-3}	57 ± 5	Na	1750 ± 30
SO_4^{-2}	450 ± 15	K	1292 ± 25
Fe	37 ± 3		

The samples were transferred to the laboratory in 20-L plastic containers and then kept at 4 °C prior to analysis. The amounts of COD, BOD_5, ammonia (NH_3), pPhosphate (PO_4^{3-}), turbidity, and color in samples, were determined according to the standard methods for the examination of water and wastewater (Rice *et al.* 2012). For COD measuring, potassium dichromate analysis using AL125-AQUALYTIC-reactor and AL100-AQUALYTIC

COD-meter were employed by AL125-AQUALYTIC-reactor and AL100-AQUALYTIC COD-meter. The BOD was determined using the BOD-meter BD600-AQUALYTIC. The turbidity was measured by a portable turbidity meter (Hanna Instruments, Italy). The Nessler method (SPECTRONIC GENESYS5 spectrophotometer, USA) was used to measure the ammonia concentration. pH was monitored using a portable device (Aqualytic AL15) and the intervals of pH were set up by adding 0.1 N of NaOH and HCl. The color was determined by measuring the absorbance at the wavelength of 465 nm using a UV–visible spectrophotometer (DR 5000, Hach, Germany) and comparing the obtained results with platinum-cobalt standards. Eq. 13 was applied for calculation of the removal efficiency. In the following equation, C_0 and C_i are the initial and final values of COD, turbidity, ammonia, phosphate, respectively.

$$Removal(\%) = \frac{(C_0 - C_i)}{C_0} \times 100 \quad \textbf{(13)}$$

Preparation of *Salvia hispanica* mucilage extract

The *Salvia hispanica* seeds were bought from a medicinal plant market in Rasht, Guilan Province. The seeds were cleaned and dried in an oven at 100 °C for 2 h (Rasool *et al.* 2016). The seeds were soaked in water and 0.9% NaCl solution at 30 °C. The concentrations of 30-50 g L^{-1} of coagulant were prepared according to the variable levels, e.g. 40 g L^{-1} mucilaginous was prepared by 4 g *S. hispanica* seeds in 100 mL 0.9% NaCl or 40 g L^{-1} seed extract. Then, the solution was stirred for 2 h at room temperature, and the seeds were completely swelled (Chaibakhsh *et al.* 2014; Shamsnejati *et al.* 2015). In order to extract the mucilage, the mixture was centrifuged (Sigma-301, Germany) at 2000 rpm in 15 min for solid/liquid separation (Rasool *et al.* 2016).

Design of experiments and statistical analysis for coagulation process

A response surface methodology (RSM) based on central composite design (CCD) was employed for optimization of the coagulation process. The variables levels selected for the coagulation-flocculation experiments including $FeCl_3$ (2 g L^{-1}), *S. hispanica* mucilage concentrations (10-50 g L^{-1}), reaction time (15-60 min), and pH (5-8) are depicted in Table 2. The COD and turbidity removal efficiency, shown in the Table, were evaluated as the responses. Design Expert Version 11.1.0.1 (Stat-Ease, Statistics Made Easy, Minneapolis, MN, USA) software package was used for statistical analysis of the experimental results.

Table 2. Composition of various experiments of the central composite design, COD turbidity removal responses for leachate treatment.

Entry	Variables			COD Removal (%)	Turbidity Removal (%)
	A: hybrid coagulant conc. -	B: Time (min)	C: pH -		
1	1:25	60	8	25.34	36.47
2	1:15	37.5	5	31.16	49.34
3	1:15	37.5	6.5	36.19	56.55
4	1:5	15	5	21.2	32.35
5	1:15	37.5	6.5	35.99	56.65
6	1:15	37.5	6.5	36.31	55.36
7	1:15	15	5	25.51	39.39
8	1:15	37.5	8	27.21	43.15
9	1:15	60	6.5	33.88	50.33
10	1:25	37.5	6.5	32.15	47.75
11	1:25	15	8	20.45	27.38
12	1:5	60	8	24.41	34.86
13	1:15	37.5	6.5	35.45	55.49
14	1:15	15	6.5	25.69	51.38
15	1:15	37.5	6.5	34.34	56.45
16	1:15	37.5	6.5	36.33	55.55
17	1:25	60	5	31.35	45.45
18	1:25	15	8	20.19	35.2
19	1:5	60	5	23.87	34.33
20	1:5	37.5	6.5	25.65	39.50

1.2. Electro-proxone process

For treatment of leachate with e-proxone process, a DC power supply (TP1303, Twintex Co., Shenzhen, China) was used. The anode was a 2 cm^2 BBD plate, while the cathode a 8 cm^2 (2 cm × 4 cm) carbon-PTFE electrode. In

order to increase electrical conductivity of solution, 0.05 M Na_2SO_4 was used. The capacity of ozone generator was 1 g h^{-1} and the flow rate of ozone injection to the solution was 200 $Lmin^{-1}$ (ozone concentrations ranged from 10 to 90 mg L^{-1}). The current intensity that was used in this study was varied between 50 and 500 mA. The E-proxone treatment were conducted for 90 min.

Membrane treatment

In the process of filtration, a plate and frame module of hydrophobic polyvinylidene fluoride UF membrane (Shanghai Sinap Membrane, China) with the effective area of 1,400 cm^2, pore diameter of 1 µm and molecular weight cut-off of 140 kDa was applied. The UF membrane was operated under TMPs of 0.5, 1.0, 1.5, 2.0, 2.5 and 3.5 bar and flow rate of 650 mL min^{-1}. After the treatment, an alkaline agent (NaOCl 1.5%) and demineralized water were used for washing the membranes. The cleaning agent (temperature between 35 °C) was circulated for 25 min followed by soaking the membrane in the agent (Shu *et al.* 2016).

Hybrid processes

Schematic representation of hybrid processes for treatment of landfill leachates is presented in Fig. 1. In this process, at first, in order to chemical treatment, leachates were entered the coagulation – flocculation reactor. After completing this step, the supernatant were collected and transferred to the electro-proxone process reactor and by adjusting different parameters, removal efficiency of pollutants was evaluated. Eventually, effluent of electrochemical process were fed to the membrane module for supplementary purification.

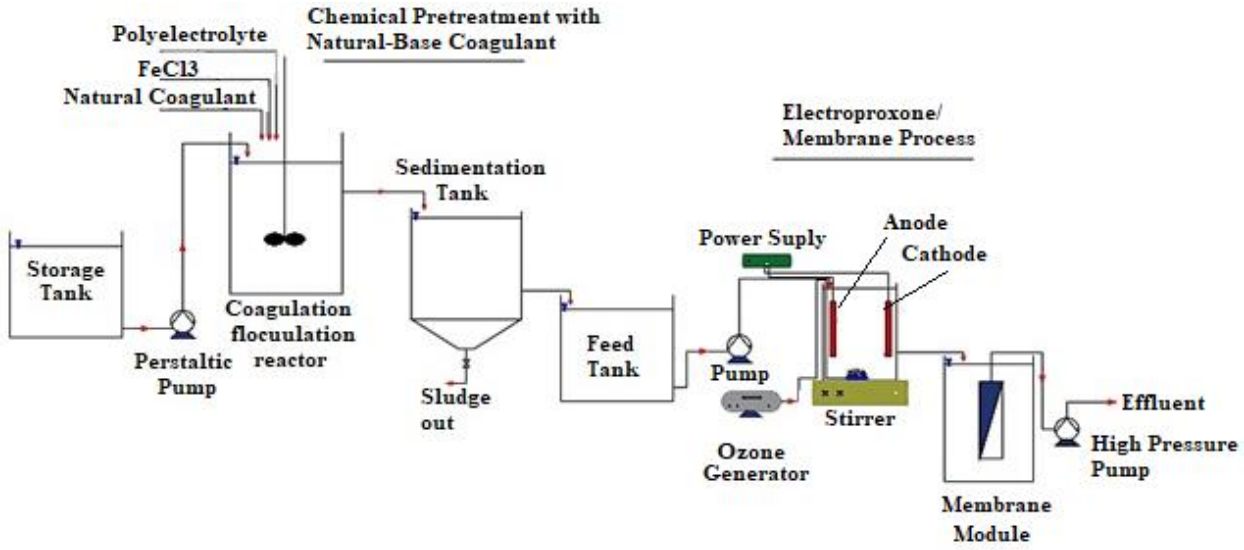

Fig. 1. Schematic diagram of hybrid process for treatment of landfill leachates.

RESULTS AND DISCUSSION

Model fitting and ANOVA for coagulation process

ANOVA analysis of the coagulation process results and the fitting quality of various models, including the linear, two factorial, quadratic, and cubic are shown in Table 3. According to the results, a quadratic polynomial model is the most suitable one for COD reduction. The F-value of the model for COD and turbidity were obtained 16.36 and 17.00 and p-value less than 0.05 (< 0.0001), respectively. The results showed that this model is significant at the 95% confidence level. Furthermore, a high coefficient of determination for COD (R^2 = 0.9364) and turbidity (R^2 = 0.9386) were achieved. The model equations in terms of the coded factors for COD and turbidity were obtained as follows:

$$\text{COD Removal} = 34.65 - 1.90\,A + 2.58\,B - 1.55\,C - 0.545\,AB + 1.39\,AC + 0.075\,BC - 4.07\,A^2 - 3.18B^2 - 3.78\,C^2 \quad \textbf{(14)}$$

$$\text{Turbidity Removal} = 56.15 - 1.5\,A + 1.90\,B - 2.78\,C + 0.76\,AB + 0.59\,AC$$

$$- 0.41\ BC - 4.30\ A^2 - 5.50\ B^2 - 10.11\ C^2 \quad \textbf{(15)}$$

where, A is the combined ratio of chemical and natural coagulants, B is the contact time (min), and C is pH. The effects of different parameters such of coagulant ratios, contact time and pH in COD and turbidity reduction were demonstrated using 3D response surface plots in Fig. 2, exhibiting that both COD and turbidity removal obeys quadratic equations for all three parameters and have optimum values in the examined ranges of the parameters. Optimum values of 36.3% and 56.6% were experimentally obtained for COD and turbidity removal, respectively. Given the interaction effects of the parameters on COD removal, the effects of pH and also Hybric coagulant concentration rise by elevating the operation time. In addition, upraising the solution pH, amplifies the effects of Hybric coagulant concentration on COD removal. Nevertheless, operating time and consequently, its interaction with the two other parameters, displayed lower effects of the turbidity removal in comparison with COD removal. The observe trends of the parameters are in accordance with previous studies (Rasool *et al.* 2016; Tawakkoly *et al.* 2019).

Table 3. ANOVA analysis of the quadratic polynomial model for COD turbidity removal of leachate.

Source	Sum of Squares	Degree of freedom	Mean Square	F-value	p-value
Model for COD reduction	591.16	9	65.68	16.36	< 0.0001
A-hybrid coagulant ratio.	35.95	1	35.95	8.95	0.0135
B-Time	66.62	1	66.62	16.59	0.0022
C-pH	23.99	1	23.99	5.98	0.0346
AB	2.38	1	2.38	0.5918	0.4595
AC	15.46	1	15.46	3.85	0.0782
BC	0.0450	1	0.0450	0.0112	0.9178
A^2	45.46	1	45.46	11.32	0.0072
B^2	27.83	1	27.83	6.93	0.0251
C^2	39.31	1	39.31	9.79	0.0107
Residual	40.15	10	4.02		
Lack of Fit	37.17	5	7.43	12.49	0.0075
Pure Error	2.98	5	0.5955		
Corrected Total	631.31	19			
Model for turbidity removal	1639.66	9	182.18	17.00	< 0.0001
A-hybrid coagulant ratio.	22.44	1	22.44	2.09	0.1785
B-Time	38.97	1	38.97	3.64	0.0857
C-pH	77.28	1	77.28	7.21	0.0229
AB	4.70	1	4.70	0.4382	0.5230
AC	2.80	1	2.80	0.2609	0.6206
BC	1.35	1	1.35	0.1262	0.7298
A^2	50.90	1	50.90	4.75	0.0543
B^2	83.26	1	83.26	7.77	0.0192
C^2	281.21	1	281.21	26.23	0.0004
Residual	107.19	10	10.72		
Lack of Fit	105.39	5	21.08	58.58	0.0002
Pure Error	1.80	5	0.3599		
Corrected Total	1746.86	19			

Several studies investigated the effectiveness of natural coagulants in removing contaminants (Awang & Aziz 2012; Aziz *et al.* 2018; Nithya & Abirami 2018). One of the key parameters in the coagulation-flocculation process playing significant role is pH. In coagulation process with chemical coagulant, alteration in pH has a significant effect in efficiency of coagulant, e.g., in pH above 7, with iron salt coagulants, due to the formation of hydroxide deposits and removal of active ions from solution, pollutant elimination efficiency is reduced (Adewuyi & Adewumi 2018). In the cases of COD and turbidity reduction in leachate, the optimum pH was found to be in the range of 5 to 6.5. Normally, the optimal pH range for coagulation by $FeCl_3$ is 5 to 7 (Amor *et al.* 2015). Surface charge of the colloidal particles in optimum pH is decreased or neutralized leading to unstable suspension. Leachate has high level of cations such as K^+, and consequently, positive charge, hence neutralization of positive charge in natural pH with the negatively-charged groups leads to destabilization and flocculation of colloids (Patel & Vashi 2012; Tawakkoly *et al.* 2019). In a study by Shamsnejati *et al.*, the optimum pH for the COD removal in

textile wastewater by *O. basilicum* was 6.5 (Shamsnejati *et al.* 2015). Noteworthy, the extracted mucilage from *S. hispanica* has polymeric characteristics, and removes contaminants by bridging, absorption and trapping particles in flocs (Freitas *et al.* 2015; Rasool *et al.* 2016). One of the disadvantages of natural coagulants for water and wastewater treatment, is the substantial increase in the COD and organic load of the treated effluent (Rasool *et al.* 2016). However, in this study, by using appropriate ratio of chemical and natural coagulant in treated leachate, a significant decrease in the COD and turbidity was obtained. However, the amount of reduction in turbidity and COD using the combination of $FeCl_3$ and *S. hispanica* mucilage was not still enough to meet the environmental standards.

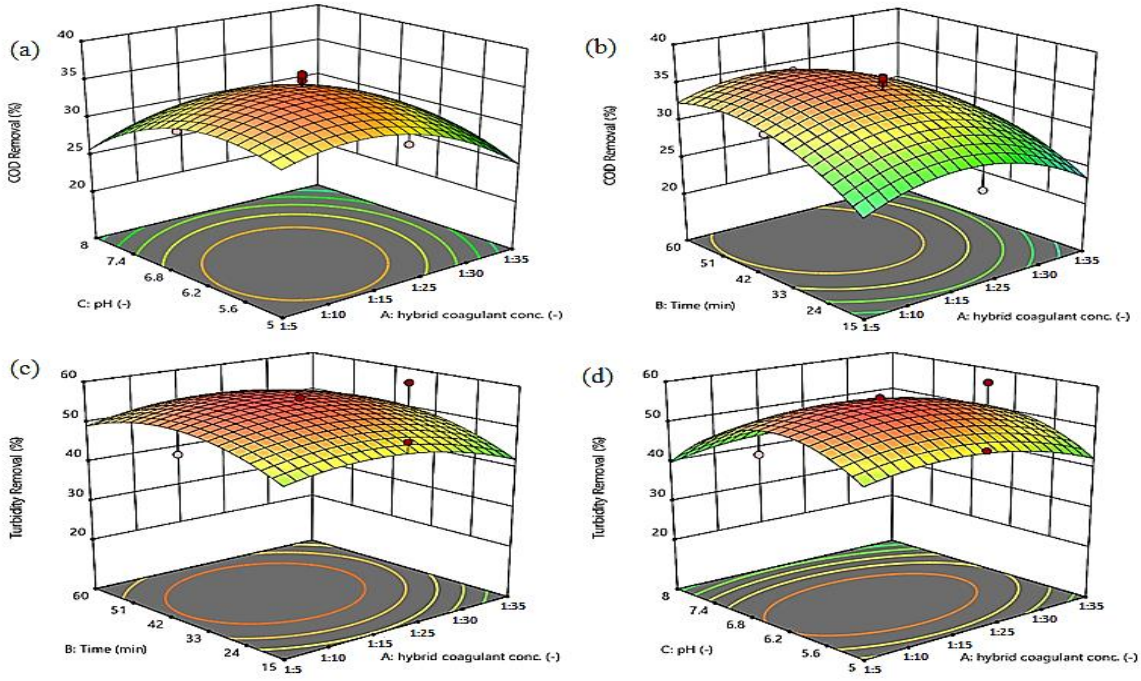

Fig. 2. a,b: Response surface plots showing the interaction between two parameters (contact time, coagulant dosage) in COD reduction (a,b); and in turbidity reduction (c,d).

Optimization of electro-proxone process

Effects of current intensity on removal efficiency

The experimental results of COD reduction with electro-proxone process are shown in Fig. 3 exhibiting that COD removal significantly grows by elevating the current intensity from 50 to 350 mA which may be due to more production of H_2O_2 at the carbon-PTFE cathode by upraising the current intensity. Consequently, by elevating reaction between O_3 and H_2O_2, more °OH can be produced (Li *et al.* 2013). However, as the current increased further from 350 to 500 mA, the rate of COD mineralization was slowed down.

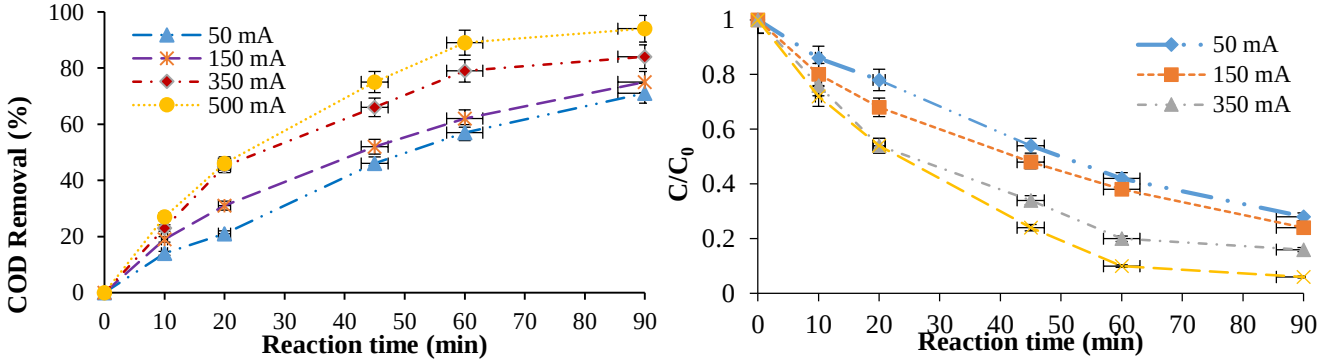

Fig. 3. Effects of applied current intensity COD degradation in the E-proxone process (initial COD = 10650 mg L^{-1}; pH = 6.5 (uncontrolled); gas flow rate = 200 L min^{-1}; inlet O_3 concentration = 45 mg L^{-1}).

One of the parameters limiting the •OH production rate is the low solubility of O_3 and rate of O_3 transfer from gas phase to liquid phase, which happens when the current intensity is increased beyond a critical value (e.g., 350 mA in this study; Li *et al.* 2015). At the reactor inlet and outlet, O_3 concentrations were calculated from the difference between the gas phase in order to find the O_3 consumption in the process (Cortez *et al.* 2010). It was observed that by elevating the current intensity, O_3 was more rapidly consumed in the e-proxone system. However, O_3 consumption did not significantly increase as the current was elevated from 350 to 500 mA. This is presumably because when the current increases beyond 350mA, the O_3 consumption limits by its mass transfer from the gas phase to the liquid (Kishimoto *et al.* 2005; Lin *et al.* 2016). In other words, when, there is not enough amount of O_3 to react with in-situ generated H_2O_2, the excess H_2O_2 has not high-efficiency in pollutant degradation, which is due to the low oxygen transfer capacity of H_2O_2 for mineralizing refractory organic pollutants (Brillas *et al.* 2000; Deng & Englehardt 2007). Furthermore, the excess H_2O_2 would undergo auto decomposition (Eq. 16) or act as •OH scavengers via reactions such as Eq. (17)[41, 43]:

$$2H_2O_2 \rightarrow 2H_2O + O_2 \uparrow \quad \textbf{(16)}$$

$$H_2O_2 + {}^{\bullet}OH + H_2O \quad \textbf{(17)}$$

According to these equations, the oxidizing efficiency by consumption of reactive •OH is decreased when the current intensity elevates beyond a critical value.

Effects of O_3 concentration on COD removal

The COD removal efficiency was shown in Fig. 4 exhibiting that by elevated O_3 concentration in the sparged gas, the COD mineralization efficiency rises. By upraising O_3 concentration from 10 mg L^{-1} to 90 mg L^{-1}, COD reduction was increased from 29 % to 84 % in 60 min, respectively. Likewise, by elevated reaction time from 60 min to 90 min, in 90 mg L^{-1} O_3 concentration, COD elimination rised slowly from 84% to 91%.

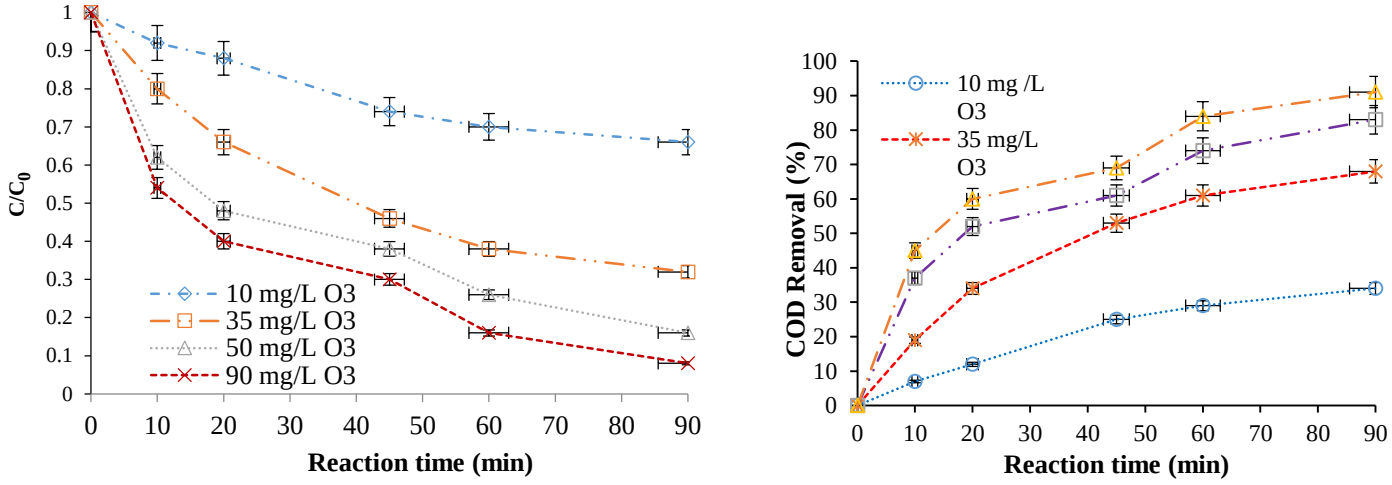

Fig. 4. Effects of O3 concentration in the sparged gas on COD degradation in the E-peroxone process [initial COD = 10650 mg L^{-1}; initial pH 6.5 (uncontrolled); gas flow rate = 200 L min^{-1}; current = 350 mA].

According to mass transfer theories, elevated O_3 concentration in the gas phase would increase the mass transfer rate of O_3 from the gas phase to liquid phase (Bakheet *et al.* 2013). As a result of the enhanced O_3 mass transfer, °OH can be produced more rapidly from the reaction between O_3 and H_2O_2 (Eq. 18) and from the reduction reaction of O_3 at the cathode (Eq. 19) (Kishimoto *et al.* 2011; Bakheet *et al.* 2013).

$$H_2O_2 + O_3 \rightarrow {}^{\bullet}OH + {}^{\bullet}O_2^- + O_2 \quad \textbf{(18)}$$

$$O_3 + H_2O + e^- \rightarrow {}^{\bullet}OH + O_2 + OH^- \quad \textbf{(19)}$$

The theoretical •OH yield for the reaction of O_3 with H_2O_2 is ~50% (Eq. 20) [57]:

$$2H_2O_2 + 2O_3 \rightarrow H_2O + 3O_2 + HO_2^{\bullet} + {}^{\bullet}OH \quad \textbf{(20)}$$

In the electrolysis-O_3 process, on the basis of Eq. 22, while some O_3 may be reduced to $O_3^{-\bullet}$, leading to the formation of $^{\bullet}$OH, the major cathodic reaction is O_3 reduction to O_2 (Eq. 21; Kishimoto *et al.* 2011).

$$O_3 + H_2O + 2e^- \rightarrow O_2 + 2OH^- \quad \textbf{(21)}$$

$$O_3 + e^- \rightarrow O_3^{-\bullet} \quad \textbf{(22)}$$

With the cathodic reduction of O_3 to O_2, the O_3 concentration for the oxidation of pollution is decreased (Bakheet *et al.* 2013; Wang *et al.* 2018). Moreover, the cathodic reduction of O_3 to O_2 can cause excessive consumption and wasted energy. At first, electric energy is consumed to convert the O_2 feed gas to O_3 in ozone generators, then energy is absorbed again to reduce O_3 back to O_2 at the cathode. Therefore, in order to achieve optimal removal of pollutants, cathodic O_3 reduction should be prevented in the e-peroxone process (Wang *et al.* 2018).

Membrane treatment

Effects of operation conditions

Effect of TMP

The effects of different parameters such as operation pressure and temperature on the separation performance and removal of pollutant from leachate were evaluated. As shown in Fig. 5, by increased TMP from 0.5 to 3.5 bar, the permeate flux also enhanced from 35.3 L m^{-2} h^{-1} to 108.4 L m^{-2} h^{-1}, while the COD removal was relatively decreased. By increasing TMP, according to the Darcy's law, permeation flux is upraised, however, fouling restricts this basic law (Salahi *et al.* 2009; Shu *et al.* 2016). By elevating TMP, the sediments compact on the surface of membrane and block the membrane pores. Thus, by reaching to optimum TMP, the maximum permeation flux is obtained, while the formation of cake/gel layer can be decreased (Salahi *et al.* 2009; Dolar *et al.* 2016). According to Fig. 5, by rising TMP up to 2.5 bar, permeation flux increases linearly, which may be due to formation of cake/gel layer at high pressures on the membrane surface. As shown in this Fig., by increasing TMP to 2.5 bar, amount of cake/gel layer formation on the membrane surface (R_f) enhances slightly, while by elevated TMP to 2.5 bar, R_f increases severely, followed by reduced permeate flux. One of the important reasons for this phenomenon is low tendency to the formation of cake/gel layer at TMP up to 2.5 bar and lower elevation of R_f in membrane. However, be upraising TMP from 2.5 bar to 3.5 bar, resistance in surface of membrane is sharply elevated, hence, the cake/gel layer becomes denser leading to dropped COD and turbidity removal (Hoek & Elimelech 2003; Salahi *et al.* 2009). Operation of membrane in optimum condition, can lead to the maximum outlet flow along with reduced investment and operation costs (Salahi *et al.* 2009; Alavijeh *et al.* 2017).

Accumulation of solutes on the membrane surface can cause concentration polarization(CP) and increase fouling as a result of formation of a sticky or an irreversible cake/gel layer on the membrane, which decreases the water flux and apparent solute rejection (Mulder 2012). CP can be controlled by operating at a lower water flux, and enhancing the mass transfer at membrane surface via increasing the cross-flow velocity (Hoek & Elimelech 2003). Thus, optimum TMP of about 1.5 bar was considered for the separation process.

Effect of temperature on COD removal

The effect of temperature on COD removal by membrane process is shown in Fig. 6 revealing that by the elevated solution temperature, permeation flux through the membrane was upraised. At optimum TMP about of 1.5 bar and pH = 7.5, by raised temperature from 20 °C to 50 °C, permeation flux was increased from 76.7 L m^{-2} h^{-1} to 104.1 L m^{-2} h^{-1}. However, following the increased permeation flux, the removal efficiency of COD and turbidity was decreased slightly to 54 % and 90 %, respectively. Osmotic pressure is required to decrease or stop net flow of liquid across the semi - permeable membrane in different conditions. In order to explain changes in state functions in a thermodynamic system, the van't Hoff equation is used. The van't Hoff plot, which is derived from this equation, is especially effective in estimating the change in enthalpy, or total energy, and entropy, or number of accessible microstates, of a chemical reaction. Under standard conditions, the van 't Hoff equation can be expressed as (Eq. 23; Stamatialis *et al.* 2006):

$$\frac{d}{dT} lnK_{eq} = \frac{\Delta H^{\theta}}{RT^2} \quad \textbf{(23)}$$

where *ln* is natural logarithm and *R* is the ideal gas constant. This equation provides the correct calculation for any temperature. In practice, by assuming that in a reaction, reaction enthalpy*(ΔH*) is constant, the equation is often integrated between two temperatures. A major use of the integrated equation is to estimate a new equilibrium constant at a new absolute temperature assuming a constant standard enthalpy change over the temperature range. In order to obtain the integrated equation, it is convenient to rewrite the van 't Hoff equation as Eq. 24 (Chen *et al.* 2012):

$$\frac{dlnK_{eq}}{d\frac{1}{T}} = -\frac{\Delta H^{\theta}}{R} \quad \textbf{(24)}$$

The definite integral between temperatures T_1 and T_2 is then Eq. 25:

$$ln\frac{K_2}{K_1} = \frac{-\Delta H^{\theta}}{R}\left(\frac{1}{T_2} - \frac{1}{T_1}\right) \quad \textbf{(25)}$$

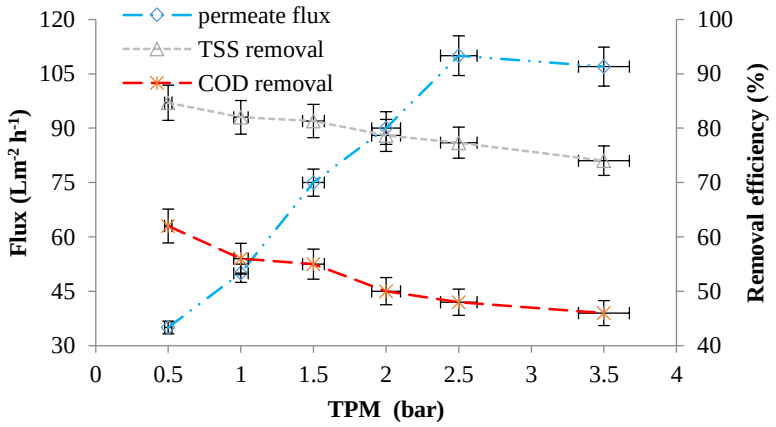

Fig. 5. Effects of TMP on permeation flux, COD and turbidity removal (T = 35 °C, pH = 7.5, CFV = 1).

where K_1 is the equilibrium constant at absolute temperature T_1, and K_2 is the equilibrium constant at absolute temperature T_2. The value of *K* is independent of pressure, although the composition of a system at equilibrium may dependent on pressure. Temperature dependence is another matter. Because the value of ΔG° is dependent on temperature, which is also true for *K* value. The form of the temperature dependence can be taken from the definition of the Gibbs function at constant temperature and pressure (Curcio *et al.* 2010; Chen *et al.* 2012):

$$\frac{\Delta G^{\circ}_{T_2}}{T_2} - \frac{\Delta G^{\circ}_{T_1}}{T_1} = \Delta H^{\circ}\left(\frac{1}{T_2} - \frac{1}{T_1}\right) \quad \textbf{(26)}$$

Because of the assumptions made in the derivation of the Gibbs-Helmholtz equation, this relationship is only valid if $\Delta H°$ is independent of temperature over the range being considered. This expression also suggests that a plot of $ln(K)$ as a function of $1/T$ should produce a straight line with a slope equal to $-\Delta H°/R$ (Curcio *et al.* 2010).
As shown in Fig. 6, by elevating temperature, osmotic pressure was upraised. According to Darcy's Law (Norouzbahari *et al.* 2009):

$$Q = \frac{-kA\Delta P}{\mu L} \quad \textbf{(27)}$$

where *k* is soil coefficient of permeability (m^2), *A* is cross section area of flow (m^2), ΔP is the differential pressure across the medium (Pa), μ is the kinematic viscosity of the fluid (Pa) and *L* , the depth or thickness of the bed or medium (m). According to this equation, by elevating osmotic pressure, permeation flux is dropped. On the other hand, increased temperature can decrease the liquid viscosity, and as a result, permeation flux is upraised (Šostar-Turk *et al.* 2005; Hua *et al.* 2007; Curcio *et al.* 2010).

Fig. 6. demonstrates that permeation flux was increased by elevating the temperature up to 40 ˚C, however, it dropped by a further increase is temperature to 50°C. Therefore, it can be concluded that temperature has a significant effect on permeation flux. At high temperatures, the viscosity effect diminishes, while the osmotic

pressure enhances. However, these two effects are finally balanced. By upraised temperature, membrane fouling was reduced, which is due to increased solubility of the components in the leachate. The results show that the optimum temperature of 30°C can be recommended to achieve high permeation flux with high elimination of pollution and low operating costs (Salahi *et al.* 2009).

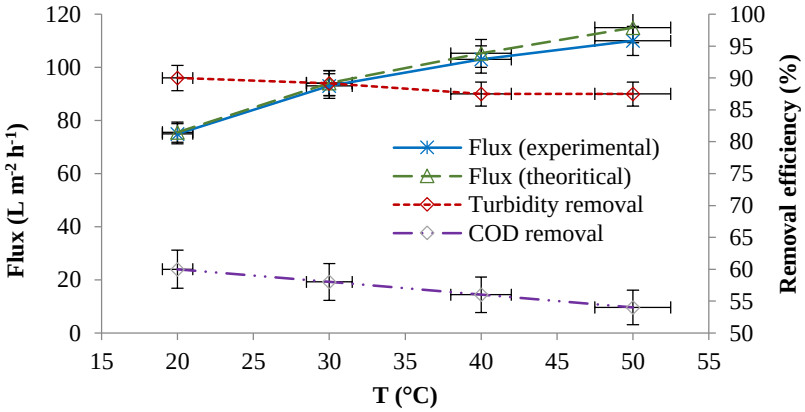

Fig. 6. Effects of temperature on permeation flux, COD and turbidity removal (TPM = 1.5 bar, pH = 7.5, CFV = 1).

CONCLUSION

A combined method including three processes were employed for experimental treatment of landfill leachate. At the first step, response surface methodology (RSM) based on a central composite design (CCD) was used for optimization of coagulation using *Salvia hispanica* mucilage extract as a natural coagulant. The maximum COD and turbidity removal in optimum conditions with coagulant ratio of 1:15, pH = 6.5, and contact time of 40 min, were 36.19 % and 56.9 %, respectively. Electro-proxone was untilized as the second treatment process. The results revealed that elevated applied current intensity, can upraise the rate of H_2O_2 electro generation and as a result increase O_2 formation at the carbon-PTFE cathode. Presumably, more $^{\bullet}OH$ can be generated at higher applied currents because of more rapidly generation of H_2O_2, that is due to subsequent reaction of the electro-generated H_2O_2 with the sparged ozone in the e-proxone process. By increased O_3 concentration from 10 mg L^{-1} to 90 mg L^{-1}, COD reduction was increased from 29 % to 84 % in 60 min. According to mass transfer theories, increasing O_3 concentration in the gas phase would elevate the mass transfer rate of O_3 from the gas phase to liquid phase. Ultrafiltration using a polyvinylidene fluoride membrane was used as the final treatment stage and the effects of temprature and transmembrane pressure on the process were investigated. It was observed that by upraising temperature from 20 °C to 50 °C, permeation flux was increased and reached from 76.7 L m^{-2} h^{-1} to 104.1 L m^{-2} h^{-1}. This can be due to the increased viscosity, solubility of the solution and thus reduced membrane fouling. By reaching to optimum TMP, permeation flux was enhanced, while the formation of cake/gel layer was reduced.

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