Influence of Elevated Temperature and Pressure on Treatment of Landfill Leachate by Potassium Ferrate(VI)

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Abstract The significant problem is the treatment of landfill leachate characterized by high chemical oxygen demand (COD) and total organic carbon (TOC) and presence of biodegradation inhibitors such as heavy metals and oily substances. For the case of landfill leachate (pH 7.9, color 1550 mg Pt/L, COD 1880 mg O₂/L, TOC 620 mg/L, total nitrogen (TN) 220 mg/L, total phosphorus (TP) 6 mg/L and heavy metals), the effect of elevated pressure and temperature on the decrease COD value by potassium ferrate(VI) (K₂FeO₄) was investigated. Taguchi method (TM) was employed for optimizing the treatment process parameters (repetition, pH, K₂FeO₄ concentration, time, temperature, and pressure). Maximizing the criterion function of signal-to-noise (S/N), the optimal parameters were determined: pH 3.5, K₂FeO₄ 2.0 g/L, time 45 min, temperature

30 °C, and pressure of 1 MPa. At optimal conditions, a decrease of color, COD, TOC, and TP was above 91%. Additionally, the decrease of TN was 48.2%. Comparatively, at atmospheric pressure (0.1013 MPa) and at 19 °C, the decrease of color, COD, TOC, TP, and TN was 99.9%, 83.0%, 79.0%, 19.1%, and 100%, respectively. Addition of potassium ferrate(VI) may improve organic compound removal and decreases energy consumption in subcritical water oxidation.

Keywords Landfill leachate · Potassium ferrate(VI) · Taguchi's method · Green oxidizer · Optimization

1 Introduction

The treatment of wastewater characterized by high chemical oxygen demand (COD) and low biological oxygen demand (BOD₅) is a challenging problem (Andreozzi et al., 1999). Due to low biodegradability of organic pollutants and their frequently occurring high toxicity, biological treatment of such wastewater is often ineffective. Treatment by advanced oxidation processes (AOPs) such as Fenton reaction, ozone, or UV-based processes is also ineffective due to high concentration of the pollutants present in the wastewater and their oxidative recalcitrance (Andreozzi et al., 1999). Additionally, a presence of inorganic radical scavengers, mainly carbonate and chloride,

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additionally decreased pollutant removal in AOPs (Drzewicz et al., 1999; Jankowska et al., 2004). It seems that only highly energy-intensive oxidation processes such as ionizing radiation (γ -radiation, electron beam irradiation, EBI), electric discharge (electrohydraulic cavitation), hydraulic cavitation, and supercritical water oxidation (SCWO) processes are capable to remove organic pollutants from wastewater (Drzewicz et al., 2004; Locke et al., 2006; Marrone, 2013; Carpenter et al., 2017; Takeuchi & Yasuoka, 2021; Wei et al., 2021). During the course of these processes, the high concentrations of numerous reactive species that very effectively decompose organic pollutants are obtained in a very short time. Additionally, physical conditions (high temperature and pressure) associated with the processes favor decomposition of refractory compounds. However, in comparison to other AOP, SCWO processes seem to be economically feasible, mainly, due to the application of microwave radiation that allows to deliver effectively high amount of energy (heat) to the treated wastewater in a very short time (Marrone, 2013; Serpone et al., 2010; Wei et al., 2021). SCWO processes require temperature above 374 °C and pressure above 22.1 MPa; therefore, various catalysts are frequently used in SWCO in order to improve economical parameters by reducing the operating parameters, i.e., temperature, pressure, and reaction time. (Jing et al., 2016; Sushma & Saroha, 2018). However, application of catalysts triggers additional problem related to catalyst deterioration and its recovery after the process (Jing et al., 2016; Keav et al., 2011). Addition of green oxidant that decompose to non-harmful products, e.g., ozone, H_2O_2 , or magnetic nanoparticles of metallic iron or iron oxides, may also alleviate the operating conditions and improve pollutant removal efficiency (Drzewicz et al., 2012; Kirmizakis et al., 2014; Scandelai et al., 2018). Landfill leachate is dark colored aqueous effluent formed as a result of rain-water percolation through the landfill, during which the pollutants are transferred from the wastes to the percolating water (Kulikowska & Klimiuk, 2008). The leachate is characterized by low BOD_5/COD ratio, which decreases with the age, and fairly high ammonia nitrogen (N_{NH_4}) and heavy metal content (Kulikowska & Klimiuk, 2008; Renou et al., 2008). Conventional leachate treatment methods, such as air stripping, coagulation, flocculation, and settling, are often costly in terms of initial outlay of plant

equipment, energy requirements, and frequent use of additional chemicals (Abbas et al., 2009; Renou et al., 2008; Wiszniowski et al., 2006). Other methods such as reverse osmosis and active carbon adsorption only transfer the pollution and do not solve the environmental problems. The biological processing of leachate needs, in most cases, large areas to run with the additional disadvantage of generating large amounts of sludge. AOPs, e.g., Fenton process ($\text{Fe(II)} + \text{H}_2\text{O}_2$), photo-Fenton process ($\text{Fe(II)} + \text{H}_2\text{O}_2 + \text{UV}$), $\text{UV}/\text{H}_2\text{O}_2$, UV/O_3 , and UV/TiO_2 , have been proposed in the recent years as an effective alternative for mineralization of recalcitrant organics in landfill leachate (Gautam et al., 2019). However, these methods in application for the treatment of large-scale effluents are not economically feasible (Abbas et al., 2009; Wiszniowski et al., 2006). Combination of physical, chemical, and biological processes is frequently proposed for treatment of landfill leachate; however, economic feasibility of such solution has not been proved yet (Keyikoglu et al., 2021; Luo et al., 2020a, 2020b). SCWO is another method proposed for treatment of landfill leachate (Wang et al., 2011). However, high temperature and pressure (significantly higher than 374 °C and 22.1 MPa) leads to gasification of organic matter and precipitation of inorganic salts (Ferreira Pinto et al., 2017). Moreover, corrosion is a key barrier that limits commercial applications of SCWO (Adar et al., 2019). Corrosion problems in SCWO are caused by high temperature and sharp pressure changes, high content of undissolved oxygen, the solubility of gases, high pH value, and formation of chemically aggressive decomposition products. Therefore, the oxidation process in landfill leachate is conducting below supercritical point of water (Kirmizakis et al., 2014; Zhai et al., 2016). So-called wet air oxidation (WAO) is conducted in temperature from above 100 °C and above atmospheric pressure to near below supercritical water condition (Kolaczowski et al., 1999). The green oxidant, e.g., H_2O_2 , potassium peroxysulfate ($2\text{KHSO}_5 \times \text{KHSO}_4 \times \text{K}_2\text{SO}_4$), potassium persulfate ($\text{K}_2\text{S}_2\text{O}_8$), and ozone (O_3), is added in order to promote oxidation of organic compounds in landfill leachate (Anglada et al., 2011; Rivas et al., 2005; Xu et al., 2012; Zhai et al., 2016). Potassium ferrate(VI) is an emerging green water treatment agent frequently proposed for wastewater treatment (Wang et al., 2021). The results of recent study has shown that potassium ferrate(VI) can be

successfully applied also for treatment of landfill leachate (Thomas et al., 2020). Potassium ferrate(VI) may oxidize organic compounds by electron or/and oxygen transfer mechanisms (Rush & Bielski, 1989; Chen et al., 2018; Luo et al., 2020a, 2020b; Wang et al., 2021). Thus, reactivity of potassium ferrate(VI) depends on pH value. The formation of many radical species was also observed during the oxidation reactions (Drzewicz et al., 2019). Therefore, one can assumed that increasing reaction temperature results in an increase of potassium ferrate(VI) reactivity. Increasing water temperature improves diffusion of radicals, miscibility of low polar compounds, and hydrolysis of the compounds whereas increasing pressure-solubility of gases such as oxygen (Akiya & Savage, 2002; Kruse & Dinjus, 2007; Weingärtner & Franck, 2005). It was found that, in the presence of oxygen, formed superoxide radicals are involved in oxidation of organic compounds by potassium ferrate(VI) (Drzewicz et al., 2019). As a result, at elevated pressure, an increase of oxygen concentration increases formation of superoxide radicals that are involved in oxidation of organic compounds (Hayyan et al., 2016). Superoxide radical and hydrogen peroxide are not reactive towards ferrate(VI) anions (Rush et al., 1996). Most studies (Sharma et al., 2000; Graham et al., 2004; Li et al., 2005; Jiang et al., 2009; Lan et al., 2014; Moradnia et al., 2016; Lv et al., 2018; Luo et al., 2019; Drzewicz et al., 2019) have only focused on the application of potassium ferrate(VI) under normal temperature and pressure. For this reason, the aim of presented study is to broaden current knowledge of application of K_2FeO_4 . Thus, it was examined an effect of elevated pressure and temperature on oxidation of organic pollutants in landfill leachate by K_2FeO_4 and following coagulation and flocculation process. Taguchi's method was used in planning the experiments and optimization of process conditions as well as determination of the most influential parameters.

2 Materials and Methods

2.1 Materials

The study used a leachate sample from old municipal waste landfill located in southern Poland. Two

municipal landfill leachate samples were collected manually from two drainage wells connected to vertical drainage system. The samples were transported to the laboratory, and then, combined and well mixed. Samples were transported and stored in cool conditions at 4 °C, without any preservation, until the tests were performed. The physicochemical parameters of the leachate sample are given in Table 1

The commercial Envifer® (Nano Iron, s.r.o., Czech Republic) was used as the source of K_2FeO_4 . Envifer® contained 40 wt.% of pure K_2FeO_4 . The detail description of the composition and physicochemical properties of Envifer® is reported elsewhere (Thomas et al., 2018). The pH value of the samples was adjusted by addition of 25 wt.% H_2SO_4 and 25 wt.% NaOH (analytical grade, Chempur, Poland). For flocculation, anionic flocculant (polyacrylamide), 0.05 wt.% solution (technical grade, Furoflock CW 277, Chemische Fabrik Wocklum GmbH & Co. KG, Germany) was applied. Double distilled water (conductivity < 2 $\mu S/cm$) was used in all experiments. Additionally, synthetic air containing 21% O_2 and 79% N_2 by volume (analytical grade, Air Liquide Polska Ltd., Poland) was used.

2.2 Analytical Methods

The concentration of K_2FeO_4 in Envifer® was determined before performing the tests by using two separated methods, i.e., spectrophotometrically and titrimetrically (Schreyer et al., 1950; Thomas et al., 2020; Wei et al., 2015). Determination of pH value was performed by the use of Inolab® pH/Ion/Cond 750 and pH combination electrode with built-in temperature sensor, SenTix®81 (WTW, Germany), according to ISO 10523 (ISO 10523, 2008). The determination of chemical oxygen demand (COD) was performed according to ISO 15705 (ISO 15705, 2002). The methods applied for determination of total organic carbon (TOC), total nitrogen (TN), and total phosphorus (TP) are described in detail elsewhere (Thomas et al., 2020). Chloride was determined by using Mohr's method according to ISO 9297 (ISO 9297, 1994) and the concentration of sulfate in the landfill leachate was determined by using gravimetric method according to ISO 9280 (ISO 9280, 2002). The determination of selected heavy metals: Cd, Cr, Cu,

Fe, Ni, Pb, and Zn were performed with ICP-OES in accordance with ISO 11885 (ISO 11885, 2007).

2.3 Optimization by Taguchi's Method

The operating parameters of the treatment process were optimized by using Taguchi's method. The software Statistica 13 (Tibco Software Inc., USA) was used to plan and analyze the experimental findings. Taguchi's method was used to find the parameters, which significantly influence the process and are independent from other process variables. The orthogonal array (OA) provides the vast amount of information from the minimum number of experiments. The process output was the decrease of COD in landfill leachate. In the Taguchi design, signal-to-noise (S/N) ratio is used for variation reduction of process output and optimization of process parameters. Signal-to-noise (S/N) ratio reflects the deviations of actual process output values from the target output values. "The smaller is better" and S/N ratio are the primary factors which help identify the optimal input parameters of the process. The S/N ratios were calculated with Eq. 1.

$$\eta = 10 \log_{10} \left(\frac{1}{n} \sum_{i=1}^n y_i^2 \right) \quad (1)$$

where i is a measurement number, η is a S/N factor, n is a number of experiments, and y is a response to the given factor level combination (Asghar et al., 2014; Thomas et al., 2018). The orthogonal array L_{48} ; five parameters: pH value (2.5, 3.5, 4.5, 5.5), K_2FeO_4 conc. (0.5, 1.0, 1.5, 2.0 g/L), time (15, 30, 45, 60 min), temperature (20, 30, 40, 50 °C), and pressure (1, 2, 3, 4 MPa) at four levels each; and the repetitions were designed (Tab. 2).

2.4 Experimental Set-up

All oxidation experiments were performed in a high-pressure reactor (Berghof HR-100 Hochdruck Autoklav, Berghof Products + Instruments GmbH, Germany) (see Fig. 1).

The capacity of the stainless steel reactor was 150 mL and its maximum operating parameters were 10 MPa and 250 °C. The reactor was heated using a dry block heater (BLH-800, Berghof Products + Instruments GmbH, Germany). The sample in the reactor was mixed with a magnetic stirrer.

Table 1 Physicochemical parameters of raw landfill leachate

Parameter	Unit	Raw landfill leachate $\pm$ uncertainty Coverage factor, $k=2$
pH	-	7.9 $\pm$ 0.1
Conductivity	μ S/cm	10 550 $\pm$ 530
Salinity	mg NaCl/L	7 210 $\pm$ 360
Color	mg Pt/L	1550 $\pm$ 310
Chemical oxygen demand, COD	mg O_2 /L	1880 $\pm$ 280
Total organic carbon, TOC	mg/L	620 $\pm$ 90
Total nitrogen (TN)	mg/L	220 $\pm$ 30
Total phosphorus (TP)	mg/L	6.0 $\pm$ 0.9
Chlorides	mg/L	790 $\pm$ 80
Sulfates	mg/L	150 $\pm$ 15
Cadmium	mg/L	0.020 $\pm$ 0.004
Chromium (total)	mg/L	0.10 $\pm$ 0.02
Copper	mg/L	0.50 $\pm$ 0.05
Iron	mg/L	38.0 $\pm$ 3.8
Nickel	mg/L	0.12 $\pm$ 0.02
Lead	mg/L	0.25 $\pm$ 0.05
Zinc	mg/L	14.0 $\pm$ 1.4

The temperature was adjusted and controlled with an electronic temperature controller. The manometer was used for pressure monitoring. The inlet and outlet valves were used to control the pressure inside the reactor. The pressure was adjusted with synthetic air 21% O₂ and 79% N₂ by volume (analytical grade, Air Liquide Polska Ltd.). Specified amount of solid K₂FeO₄ (± 0.001 g) was placed in the reactor's Teflon vessel, and after that, 100 mL of landfill leachate was added. The solution was stirred at 200 rpm until the added salt dissolved completely. After adjusting pH value using 25 wt.% H₂SO₄ or 25 wt.% NaOH solution (if a further pH value correction after addition of H₂SO₄ was needed), the reactor was tightly closed and the pressure was adjusted with analytical grade synthetic air from a high-pressure gas cylinder. After that, the reactor was heated to the specified temperature. The increase of pressure during heating was carefully compensated. After the oxidation process, the solution was transferred to a beaker and left for 15 min to cool down to room temperature. In the next step, the solution was stirred at 200 rpm and the pH value of the solution was adjusted to 9.5 ± 0.1 . At these conditions, all amount of iron hydroxide precipitated. Then, 0.2 mL of 0.05 wt.% anionic flocculant solution was added and the stirring rate was reduced to 50 rpm. The stirring was stopped after 10 s and the solution was left for 5 min, during which time a formed sediment settled at the bottom of the beaker. The sample for analysis of the landfill leachate parameters was taken from the solution above the sediment and filtered through the 0.45- μ m PTFE syringe filter. Comparatively, additional experiments were carried out at ambient temperature (19 °C) and atmospheric pressure (0.1013 MPa).

3 Results and Discussion

It has been shown that the leachates examined were characterized by a slightly alkaline pH 7.9, high electrical conductivity 10 555 μ S/cm, and salinity 7210 mg NaCl/L as well as an intense brown color 1550 mg Pt/L (Tab. 1). The determined values of COD, TOC, and TN indicated a presence of a large concentration of various substances in the analyzed leachates (mainly organic compounds). As it reported earlier, pH value may change with leachate age. The pH for young leachate is below 6.5 whereas for mid

age is in the range from 6.5 to 7.5, and for old one is higher than 7.5 (Aziz, 2012; Yadav & Dikshit, 2017; Tejera et al., 2019). The COD value below 5000 mg O₂/L and pH 7.9 suggest that samples were collected from an old landfill. The leachates from old landfill are usually characterized by high production of methane and high fraction of organic compounds, mostly humic and fulvic acid fractions (Aziz, 2012; Yadav & Dikshit, 2017; Tejera et al., 2019). Humic and fulvic acid fraction is hardly removed in biodegradation and AOP processes (Andreozzi et al., 1999). Additionally, the concentration ammonium nitrogen (N_{NH4}) in such leachate is usually above 400 mg/L. However, TN for investigated leachate samples was 220 mg/L, and nitrate or nitrite nitrogen anions were not detected (detection limit was 0.1 mg/L). This value is characteristic for landfill from a young landfill. However, it is important to note that the physicochemical properties of leachate strongly depend also on kind of dumped waste and landfill location (Wang et al., 2018a, 2018b). For example, similar results, pH 7.7, and 290 mg/L ammonia have been reported for leachates from a landfill located in Jhiri village, Ranchi City, Jharkhand, India (Verma & Kumar, 2016). Due to high toxicity, ammonium cation hampers biodegradation process (Drzewicz et al., 2004). Moreover, excess amount of oxidant is needed for oxidation of ammonium cation to nitrate anion. Nitrate anion is not toxic and helps in biodegradation of organic matter; however, it causes eutrophication of natural water bodies.

The presence of high concentration of metal is also observed in the investigated leachate samples. The concentrations of metals in the leachates from landfills located in different parts of the world vary for iron in the range from 3.41 (in Matuanil landfill Bangladesh) up to 167.61 mg/L (in Riyadh City, Saudi Arabia) and for zinc from 0.05 (in Sivas, Turkey) to 17.80 mg/L (in MSW incineration plants, China). Moreover, the concentration of nickel and cadmium in investigated samples were 0.12 and 0.02 mg/L, respectively. In comparison, for example, the concentrations of nickel and cadmium were from 0.17 (in Matuanil landfill, Bangladesh) to 3.70 mg/L (in MSW incineration plants, China) (Mojiri et al., 2021). The heavy metals have detrimental effect on environment; thus, the landfill leachate cannot be released to environment without the treatment. Moreover, the

Table 2 Experimental conditions (factors and levels of the orthogonal array) and results of experiments-concentration COD, mg O₂/L landfill leachate after the treatment

Run	Experimental conditions*						
	Repetition	pH	K ₂ FeO ₄	Time	Temperature	Pressure	COD, mg O ₂ /L**
1	1	1	1	1	1	1	730 ± 110
2	1	1	2	2	2	2	405 ± 60
3	1	1	3	3	3	3	215 ± 30
4	1	1	4	4	4	4	200 ± 30
5	1	2	1	2	3	4	605 ± 90
6	1	2	2	1	4	3	380 ± 60
7	1	2	3	4	1	2	210 ± 30
8	1	2	4	3	2	1	150 ± 20
9	1	3	1	3	4	2	650 ± 100
10	1	3	2	4	3	1	405 ± 60
11	1	3	3	1	2	4	290 ± 40
12	1	3	4	2	1	3	210 ± 30
13	1	4	1	4	2	3	675 ± 100
14	1	4	2	3	1	4	420 ± 60
15	1	4	3	2	4	1	260 ± 40
16	1	4	4	1	3	2	250 ± 40
17	2	1	1	1	1	1	705 ± 105
18	2	1	2	2	2	2	395 ± 60
19	2	1	3	3	3	3	250 ± 40
20	2	1	4	4	4	4	205 ± 30
21	2	2	1	2	3	4	615 ± 90
22	2	2	2	1	4	3	395 ± 60
23	2	2	3	4	1	2	195 ± 30
24	2	2	4	3	2	1	150 ± 20
25	2	3	1	3	4	2	650 ± 100
26	2	3	2	4	3	1	395 ± 60
27	2	3	3	1	2	4	275 ± 40
28	2	3	4	2	1	3	210 ± 30
29	2	4	1	4	2	3	660 ± 100
30	2	4	2	3	1	4	420 ± 60
31	2	4	3	2	4	1	250 ± 40
32	2	4	4	1	3	2	240 ± 40
33	3	1	1	1	1	1	695 ± 100
34	3	1	2	2	2	2	395 ± 60
35	3	1	3	3	3	3	230 ± 35
36	3	1	4	4	4	4	185 ± 30
37	3	2	1	2	3	4	600 ± 90
38	3	2	2	1	4	3	380 ± 60
39	3	2	3	4	1	2	200 ± 30
40	3	2	4	3	2	1	150 ± 20
41	3	3	1	3	4	2	655 ± 100
42	3	3	2	4	3	1	405 ± 60
43	3	3	3	1	2	4	275 ± 40
44	3	3	4	2	1	3	205 ± 30
45	3	4	1	4	2	3	685 ± 100

Table 2 (continued)

Run	Experimental conditions*						
	Repetition	pH	K ₂ FeO ₄	Time	Temperature	Pressure	COD, mg O ₂ /L**
46	3	4	2	3	1	4	410 ± 60
47	3	4	3	2	4	1	240 ± 40
48	3	4	4	1	3	2	220 ± 30

*Experimental conditions for pH=2.5 (1), 3.5 (2), 4.5 (3), and 5.5 (4); K₂FeO₄=0.5 g/L (1), 1.0 g/L (2), 1.5 g/L (3), and 2.0 g/L (4); time=15 min (1), 30 min (2), 45 min (3), and 60 min (4); temperature=20 °C (1), 30 °C (2), 40 °C, and 50 °C (4); pressure=1 MPa (1), 2 MPa (2), 3 MPa (3), and 4 MPa (4)

** ± determination uncertainty

Fig. 1 High-pressure reactor (Berghof HR-100 Hochdruck Autoklav with heating block BLH-800 and temperature controller, Berghof Products + Instruments GmbH, Germany)

presence of heavy metals may affect the effectiveness of treatment by AOPs (Andreozzi et al., 1999).

The effect of various combinations of experimental conditions on COD of landfill leachate after the treatment is presented in Table 2. The investigated conditions were pH, concentration of K₂FeO₄, reaction time, temperature, and pressure. The lowest COD after the treatment, 150 mg O₂/L, was obtained for combination 8, 24, and 40, whereas the highest COD was observed for combination 1, 17, and 33 (730, 705, and 695 mg O₂/L, respectively). In combinations 8, 24, and 40, the highest dose of 2.0 g/L K₂FeO₄ has been applied at pH 3.5, 30 °C temperature, and 1 MPa pressure. The reaction time was 45 min. At these conditions, COD decreased by 92.0%, from 1880 to 150 mg O₂/L. It indicates that concentration of K₂FeO₄ and reaction time were the most influential parameters, and process is slightly affected by variation of temperature and pressure. In the case of combinations 1, 17, and 33, the lowest experimental conditions have been applied: pH 2.5, 0.5 g/L K₂FeO₄, 20 °C, and 1 MPa. The reaction time was 15 min. At these conditions, a decrease of COD value by 61.2, 62.5, and 63.0% was observed (from 1880 to 730, 705, and 695 mg O₂/L, respectively). The triplicate repetition of the experiments at lower and higher experimental conditions indicated that the treatment process is very reproducible and repeatable with very

low variability in output. In other words, the variability of the experimental results and experimental bias is very small at all combination of experimental conditions.

The analysis of variance (ANOVA) is frequently used in quantitative assessment whether there is a statistical association between experimental parameters and process outputs (Hsieh et al., 2009; Thomas et al., 2018, 2020). In this study, ANOVA was used to determine the significance of each parameter influence on the output of investigated process - a decrease of COD after the treatment. The variance ratio (F-ratio) was used to assess the significance of the parameter. The ANOVA results are presented in Table 3.

The results of ANOVA confirmed that experimental bias is statistically insignificant, and process output is repeatable and reproducible at the same experimental conditions. However, pH, K_2FeO_4 dose, reaction time, and temperature were the most statistically significant parameters ($p < 0.05$). The results are in accordance with earlier studies. Lan et al. (2014) and Thomas et al. (2020) also indicated that pH value, concentration of K_2FeO_4 , and reaction time were statistically significant parameters for optimization of the treatment process. Additionally, Zhang et al. (2014) also indicated that temperature is important parameter for optimization of potassium ferrate(VI) treatments.

In Taguchi's method, notion of "Signal to Noise Ratio" (S/N ratio) is used that measure the contribution of each parameter to process output variability. The optimization procedure based on two steps: highly fractional factorial experiments (orthogonal array designs) at various levels of process' parameters (Tab. 1) and using the data to select those levels maximized S/N ratio (Cheng et al., 2010; Chowdhury et al., 2014; Thomas et al., 2018; Więkol-Ryk et al., 2020). Optimal experimental conditions and corresponding S/N ratio are presented in Table 4.

Application orthogonal array designs in TM allow to select independent parameters that do not interrelate with each other (Asghar et al., 2014; Srinu & Pydi, 2001; Thomas et al., 2018, 2020). Significance of the effects illustrates which experimental parameters are the most affecting. The process is governed (Tab. 4) by the K_2FeO_4 dose (4.6421) and pH (1.2063). The contribution of reaction time (0.5809), air pressure (0.3064), and temperature (0.1929) is significantly smaller. A graphical presentation of the

$\eta = S/N$ for each level of the parameters is shown in the Fig. 2. The combination pH 3.5 (level 2), 2.0 g/L K_2FeO_4 (level 4), duration time 45 min (level 3), temperature 30 °C (level 2), and pressure 1 MPa (level 1) gave the highest S/N ratio – 43.3819.

A wide range of η value suggests that the pH value and the K_2FeO_4 dose are considerably the most influential in comparison to other parameters. A very small range of η value for repetition parameter indicates that experimental bias related to conducting of the experiments is not significant. The ascending line for K_2FeO_4 dose shows that the efficiency of the process increases with dose. In the case of pH value, the shape of the line indicates that there is a certain optimum at which the process output reaches its maximum value. The same applied to time. Extension of the process duration (more than 45 min) and increasing the pressure value (more than 1 MPa) did not bring improvement in decreasing of COD. Thus, the optimal conditions were as follows: pH 3.5, K_2FeO_4 2.0 g/L, time 45 min, temperature 30 °C, pressure 1 MPa.

At optimal conditions, after additional flocculation with 0.2 mL of 0.05 wt.% anionic flocculant, the color of the effluent was reduced by 97.1% whereas the COD, TOC, and TN were reduced by 91.5%, 91.1%, and 48.2%, respectively (Tab. 5). Additionally, at the optimal condition, the COD of the treated effluent 160 ± 20 mg O_2/L was similar to the COD 150 ± 20 mg O_2/L after treatment at combination of experimental condition 8, 24, and 40 (Table 1). For comparison, the treatment of the landfill leachate was

Table 3 The results of ANOVA

Effect/factor	Analysis of variance, mean = - 50.450, sigma = 4.26646				
	SS	df	MS	F	p
Repetition	0.4864	2	0.2432	3.013	0.0642
pH	25.1881	3	8.3960	104.007	< < 0.05
K_2FeO_4 dose	811.1091	3	270.3697	3349.251	< < 0.05
Reaction time	13.5359	3	4.5120	55.893	< < 0.05
Temperature	0.8030	3	0.2677	3.316	0.0331
Pressure	1.9807	3	0.6602	8.179	0.0004
Residual	2.5264	30	0.0842	-	-

SS predicted residual error sum of squares, MS mean square error, F statistics, df number of degrees of freedom

Table 4 S/N ratio in optimal experimental conditions

Effect/factor	Predicted S/N ratio under optimal conditions, mean = -50.438, sigma = 4.27668		
	Parameter value	Significance of the effect	Standard error of the effect
Repetition	3	0.1399	0.0710
pH	2 (for pH = 3.5)	1.2063	0.0820
K ₂ FeO ₄	4 (for 2.0 g/L)	4.6421	0.0820
Time	3 (for 45 min)	0.5809	0.0820
Temperature	2 (for 30 °C)	0.1929	0.0820
Pressure	1 (for 1 MPa)	0.3064	0.0820
Expected S/N value	-	-43.3819	-

conducting at the same conditions but lower temperature 19 ± 1 °C and atmospheric pressure 0.1013 MPa. At these conditions, the obtained reduction of color, COD, TOC, and TN was 99.9%, 83.0%, 79.0%, and 19.1%, respectively (Tab. 5). The results suggest that the use of a combined coagulation and oxidation process at elevated pressure and temperature is more effective than the process conducted at ambient conditions. The efficiency of TP removal at condition cases was the same and reached 100%. The highest efficiency of color reduction and heavy metal removal

of leachate was obtained at ambient conditions. The decreased efficiency of metal removal after oxidation at elevated conditions (64.0 - 100%) was likely correlated with an increase of color after the process. At these conditions, an increased color of the leachate was also observed after the flocculation process. This can be explained by formation of colorful metal complexes with degradation products, or small particles of insoluble salts.

It seems that decreasing of landfill leachate's pH from 7.9 ± 0.1 to 3.5 only slightly increase the cost of proposed treatment and consumption of chemicals. However, the application of K₂FeO₄ is more effective in acidic than in neutral and alkaline environment (Eqs. 2 and 3). The redox potential is higher in an acidic than in an alkaline environment ($E^0 = 2.2$ V and $E^0 = 0.7$ V, respectively)

Additionally, at acidic conditions, carboxylic acids, the main process by-products, are in less soluble in protonated. Other carboxylic acids may also formed insoluble salt with Fe³⁺. Potassium ferrate(VI) is stable in alkaline condition (Li et al., 2005). However, at strong alkaline conditions, above

Fig. 2 ETA (η) values versus input parameter values plot for landfill leachate (pH₁ = 2.5, pH₂ = 3.5, pH₃ = 4.5, pH₄ = 5.5, K₂FeO₄₍₁₎ = 0.5 g/L, K₂FeO₄₍₂₎ = 1.0 g/L, K₂FeO₄₍₃₎ = 1.5 g/L, K₂FeO₄₍₄₎ = 2.0 g/L, Time₍₁₎ = 15 min, Time₍₂₎ = 30 min, Time₍₃₎ = 45 min, Time₍₄₎ = 60 min, Temperature₍₁₎ = 20 °C, Temperature₍₂₎ = 30 °C, Temperature₍₃₎ = 40 °C, Temperature₍₄₎ = 50 °C, Pressure₍₁₎ = 1 MPa, Pressure₍₂₎ = 2 MPa, Pressure₍₃₎ = 3 MPa, Pressure₍₄₎ = 4 MPa). The black horizontal lines represent $\pm$ standard value

Table 5 The selected physicochemical parameters of raw and treated leachates

Parameter*	Unit	Leachate before treatment	Leachate after treatment (pH = 3.5, K ₂ FeO ₄ = 2.0 g/L, time = 45 min, temperature 30 °C, pressure 1 MPa)	Effect, %**	Leachate after treatment, pH = 3.5, K ₂ FeO ₄ = 2.0 g/L, Time = 45 min, Temperature 19 °C, Pressure 0.1013 MPa	Effect, %**
pH	-	7.9 ± 0.1	9.5 ± 0.1	-	9.5 ± 0.1	-
Color after filtering through the 0.45 µm syringe filter	mg Pt/L	1550 ± 310	45 ± 9 mg/L	97.1↓	2.0 ± 0.4	99.9↓
Chemical oxygen demand, COD	mg O ₂ /L	1880 ± 280	160 ± 20	91.5↓	320 ± 50	83.0↓
Total organic carbon, TOC	mg/L	620 ± 90	55 ± 8	91.1↓	130 ± 20	79.0↓
Total nitrogen (TN)	mg/L	220 ± 30	114 ± 17	48.2↓	178 ± 25	19.1↓
Total phosphorus (TP)	mg/L	6.0 ± 0.9	Not detected, < 0.3	100.0↓	Not detected, < 0.3	100.0↓
Heavy metals removal***						
Cadmium	mg/L	0.020 ± 0.004	< 0.01	100.0↓	< 0.01	100.0↓
Chromium (total)	mg/L	0.10 ± 0.02	0.05 ± 0.01	50.0↓	< 0.01	100.0↓
Copper	mg/L	0.50 ± 0.05	0.09 ± 0.02	82.0↓	0.009 ± 0.002	98.2↓
Iron	mg/L	38.0 ± 3.8	2.20 ± 0.4	94.2↓	0.28 ± 0.06	99.3↓
Nickel	mg/L	0.12 ± 0.02	0.04 ± 0.01	66.7↓	< 0.01	100.0↓
Lead	mg/L	0.25 ± 0.05	0.09 ± 0.02	64.0↓	< 0.01	100.0↓
Zinc	mg/L	14.0 ± 1.4	1.5 ± 0.3	89.3↓	< 0.01	100.0↓

*Parameter value ± the measurement uncertainty for an extension factor k = 2

**Wastewater treatment efficiency = $\frac{(C_1 - C_2) \times 100\%}{C_1}$, where C_1 is the concentration in the raw landfill leachate, C_2 is the concentration in the treated landfill leachate; does not apply to pH

***The lower quantification limit of heavy metals was 0.01 mg/L, except Cu 0.005 mg/L

pH 10, Fe(OH)₃ is partially hydrolyzed (Graham et al., 2004; Sharma et al., 2000) and the removal of organic compounds and partially heavy metals by iron hydroxide flocs may be reduced. Lv et al. (2018) have shown that at pH 6.5 and 7.5, a lower iron doses were needed for FeO₄²⁻ coagulation than for direct Fe³⁺ coagulation. Moreover, the results of previous studies suggested that the reaction of potassium ferrate(VI) with organic compounds could be faster than self-decomposition reaction in acidic conditions (Moradnia et al., 2016; Thomas & Zdebik, 2019; Thomas et al., 2020). It can be explained by formation of various complexes of FeO₄²⁻ with organic matter and presence of carbonate that increase the stability of potassium ferrate(VI) in acidic solution (Luo et al., 2019). Addition of sulfuric acid for pH adjustment removes also scale-forming magnesium and calcium and decreases solubility of macromolecular carboxylic

acid due to protonation. The formation of peroxy radicals is involved mechanism of organic compound oxidation by K₂FeO₄ (Drzewicz et al., 2019). According to Kim et al. (2000), the amount of oxygen needed for oxidation organic compounds is calculated according to following equation:

where X represents the sum of all halogens and $b = n + \frac{m - k - 3j - 2i - 3h}{4} - \frac{e}{2} + 2i + 2h$.

Thus, such large amount of oxygen for oxidation of organic compounds in leachate cannot be delivered with ferrate anion. Moreover, solubility of oxygen in landfill leachate is very low due to high salinity and organic content. In such conditions, the high concentration of oxygen in solution can

be achieved only by application of high pressure. Thus, increasing the pressure improves performance of the process. However, it needs further studies to confirm this. Ferrates are well-known coagulation-flocculation agent (Lv et al., 2018). Part of organic compounds is removed from the solution with iron hydroxide flocs that is formed during the oxidation process. However, Fe(III) may be reduced to Fe(II) by organic compounds (Zhou et al., 2021). Thus, effectiveness of potassium ferrate(VI)-induced coagulation and flocculation may be reduced at elevated temperature and pressure. On the other hand, in Fenton reaction, Fe(II) reacts with H_2O_2 to form highly reactive hydroxyl radicals. Hydrogen peroxide may be formed during termination of peroxy radical during oxidation by potassium ferrate(VI). However, the elucidation of the multistep process mechanism of potassium ferrate(VI) oxidation at elevated temperature and pressure needs further studies.

Ferrate(VI) salts are unstable in both solid form and in the water solutions. However, the oxidant may be generated in situ by electrochemical processes (Jiang et al., 2009). According to Diaz et al (2021), the highest efficiency of ferrate(VI) generation was achieved using gray cast iron (GCI), in presence of 10 mol/L NaOH. The production rate was $0.979 \text{ mol FeO}_4 \text{ m}^{-3} \text{ day}^{-1}$ (including time for cleaning in between cycles) at the energy consumption 8 kWh kg^{-1} . GCI is widely available commercially material at a low cost, which is simple to shape into different electrode structures. It does not contained toxic metal like chromium. Comparatively, production of chlorine via electrolysis of NaCl has a typical energy consumption of $2.2\text{--}2.6 \text{ kWh kg}^{-1} \text{ Cl}_2$ whereas electrogeneration of ozone (depends on solution and electrode material composition) has energy consumption in the range from ~ 18 to $175 \text{ kWh kg}^{-1} \text{ O}_3$ (Diaz et al (2021)). However, Cl_2 and ozone have lower oxidation potential and lower reactivity than potassium ferrate(VI). As results, Cl_2 and O_3 require longer contact times which decrease effectiveness of the process.

In summary, increasing temperature and pressure is a viable option to improve effectiveness of landfill leachate treatment by potassium ferrate(VI). Solubility of oxygen needed in the process increases with a pressure increase whereas reactivity of the

compounds increases with temperature. Additionally, microwave irradiation applied in this process will increase efficiency of heat energy transfer to the solution and effectively break molecular bonds in organic compounds (Andreozzi et al., 1999; Serpone et al., 2010). However, further research at real scale is needed.

4 Conclusions

The impact of pressure and temperature on the effectiveness of landfill leachate treatment by potassium ferrate(VI) was investigated. The Taguchi's method of experiment design has been used in optimization of the process. Based on this method, it has been shown that the most influential parameters on the treatment process output were the following parameters: pH, K_2FeO_4 dose, reaction time, temperature, and pressure. Application of TM significantly reduced the number of experiments needed for determination of optimum conditions. The determined optimum conditions were as follows: pH 3.5, $2.0 \text{ g/L K}_2\text{FeO}_4$, 45 min of contact time, 30°C , 1 MPa. At optimal condition, a decrease of color by 97.1%, COD by 91.5%, TOC by 91.1%, TN by 48.2%, and TP by 100% was obtained. Comparatively, at the same condition, but lower the atmospheric pressure (0.1013 MPa) and temperature 19°C , a decrease of color by 99.9%, COD by 83.0%, TOC by 79.0%, TN by 19.1%, and TP by 100% was obtained. Application of elevated temperature and pressure is a viable option to improve effectiveness of landfill leachate treatment by potassium ferrate(VI). However, it needs further research at real-scale applications.

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Data Availability The datasets obtained during this study are available from the corresponding author on reasonable request.

Declarations

Ethics Approval and Consent to Participate Not applicable.

Consent for Publication Not applicable.

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