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EFFECTS OF PERSULFATE, PEROXIDE ACTIVATED PERSULFATE AND PERMANGANATE OXIDATION ON TREATABILITY AND BIODEGRADABILITY OF LEACHATE NANOFILTRATION CONCENTRATE

The effects of persulfate (PS), peroxide activated persulfate (P-PS) and permanganate (PM) oxidation processes on leachate nanofiltration (NF) concentrate treatment have been activated. Additionally, COD fractions were determined and compared before and after each oxidation process. The total COD removal efficiencies by the oxidation processes using PS, P-PS, and PM as the oxidants were determined to be 24.5%, 32.9%, and 39.5%, respectively. The biodegradable COD fraction increased from 6.37% to 9.25%, 11.51%, and 13.64% after the PS, P-PS, and PM oxidation processes, respectively. About 25%, 34%, and 46% removal efficiencies of the inert COD content of the concentrate were obtained after the PS, P-PS and PM oxidation processes, and the soluble COD fraction increased from 71.1% to 87.2%, 89.0% and 84.6% by the PS, P-PS and PM oxidation processes, respectively. Although the highest removal efficiencies were achieved by PM oxidation with the lowest operational cost, all of the processes may be suggested as efficient methods for conversion of insoluble COD into the soluble COD fraction and for inert COD removal. The results of the study showed that the PS, P-PS, and PM oxidation processes may be effectively utilized as post-treatment techniques for leachate NF concentrate treatment.

1. INTRODUCTION

Sanitary landfilling is the most used method to dispose of the solid waste collected from cities, and it allows solid waste to decompose into stabilized materials under controlled conditions. During the landfilling process, high amounts of leachate form because of the interaction of waste with rainwater percolating through the landfill body,

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compaction of the waste landfilled, and biological degradation of the waste in the landfill body. Leachate is high-strength complex wastewater containing high concentrations of organic and inorganic compounds. Discharging untreated leachate causes severe environmental problems for receiving bodies [1].

Several treatment methods have been used for leachate treatment due to the composition of leachate depending on waste type, precipitation amount, site hydrology, compaction level of the waste, cover design, and the age, design, and operation of the landfill. Biological treatment is mostly preferred for young leachate with a high BOD/COD value, and nitrification/denitrification processes following biological treatment are applied for ammonium removal. Thereby, high BOD and ammonium removal is achieved. However, biologically treated leachate may contain various compounds as residual rapidly biodegradable, slowly biodegradable and non-biodegradable substrates, inter- and final products and soluble microbial products. Commonly used leachate treatment methods are limited for removal of the initially present inert COD in the influent and the inert COD produced by microorganisms during degradation of the waste in the landfill.

In recent years, to reach stringent effluent discharge standards, membrane treatment technologies have widely been applied after conventional biological treatment [1]. By application of membrane treatment methods, large amounts of membrane concentrate containing particularly refractory contaminants and salt compounds are formed [2]. Typically, membrane concentrate consists of approximately 15–50% of the feed volume in NF and RO membrane systems [3]. The constituents of the concentrate result in low biodegradability [4].

Primary treatment methods used for the treatment of concentrated leachate may be categorized as: recirculation to landfills, solidification/stabilization, evaporation/distillation, and advanced oxidation processes [2, 5–7]. Among these four categories, the most widely used method is recirculation to landfills but a high salt concentration of leachate membrane concentrate and the content of humic matter in the concentrate can inhibit the biological activity of microorganisms existing in the landfills [8]. Additionally, high salt and inorganic matter concentrations of the membrane concentrate lead to corrosion and fouling problems [5]. The evaporation/distillation method needs high land utilization and has high operating costs [7]. The disadvantage of the solidification and stabilization method is the disposal of solidified end-products [5, 9]. Among the aforementioned methods, advanced oxidation processes (AOPs) are more appropriate for the removal of recalcitrant organic compounds and improvement of the biodegradability of the concentrate.

AOPs use oxidants solely or in a combination of two. In AOPs, various oxidants as sodium persulfate ($\text{Na}_2\text{S}_2\text{O}_8$), potassium permanganate (KMnO_4), and hydrogen peroxide (H_2O_2) with respective oxidation potentials have recently been popular in water and wastewater treatment [10, 11]. To increase the performance of persulfate oxidation by initiating sulfate radical formation, various activation methods are used. Sulfate radicals are potentially much more oxidative than persulfate. Many studies related to stabilized

leachate treatment by advanced oxidation processes are reported in the literature, but the effect of persulfate (PS), peroxide activated persulfate (P-PS), and permanganate (PM) oxidants on variation in the COD fractions of the leachate NF concentrate is deficient [10–12].

The total COD value cannot reflect the real data in leachate treatment. Total COD may be categorized as biodegradable COD (COD_{bi}), non-biodegradable COD (COD_{ubi}), or particulate COD (COD_p) soluble COD (COD_s). Soluble COD may be further divided into biodegradable soluble COD ($COD_{(sbi)}$) and non-biodegradable soluble COD ($COD_{(subi)}$).

The objectives of this study are to investigate the performance of persulfate (PS), peroxide activated persulfate (P-PS) and permanganate (PM) oxidation processes in leachate NF concentrate treatment, optimize process variables such as oxidant dosage, pH and reaction time for maximum COD and TOC removal and determine the effect of the three processes on the COD fractions of the concentrate.

2. EXPERIMENTAL

Leachate sampling and characteristics. Leachate samples were collected from the nanofiltration unit of the Komurcuoda Leachate Treatment Plant (41°8'41"N, 29°22'21"E) in Istanbul, Turkey. The schematic view of the treatment plant is given in Fig. 1.

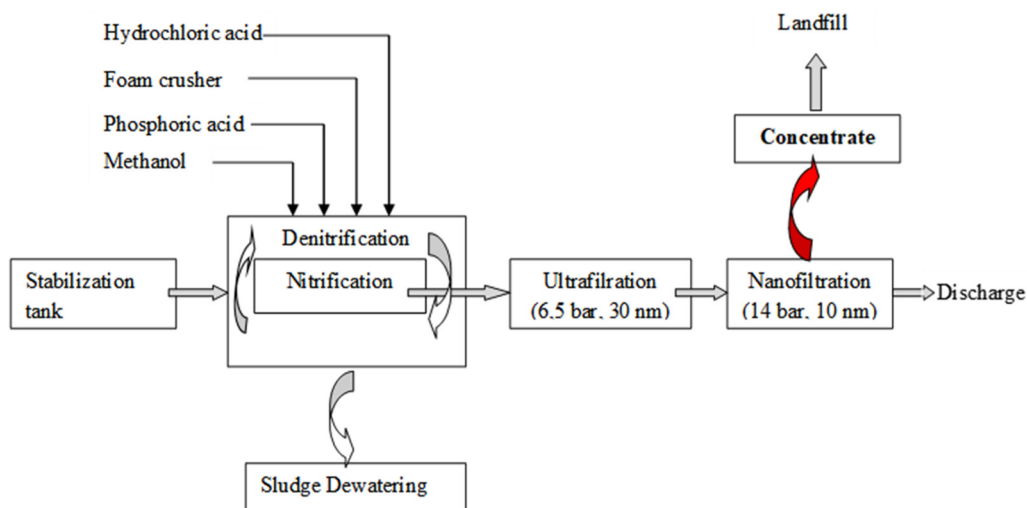


Fig. 1. Schematic view of a leachate treatment plant

Leachate NF concentrate samples were collected and preserved according to the standard methods recommended by APHA [13]. Table 1 presents the characterization of the leachate NF concentrate.

Table 1. Characterization of the leachate NF concentrate

Parameter	pH	Conductivity [mS/cm]	Chloride [mg/dm ³]	COD [mg/dm ³]	TOC [mg/dm ³]	TSS [mg/dm ³]	Color [mg/dm ³ Pt-Co]
Value	7.74±0.07	18.72±0.65	7200±25	10000±200	3719±42	33±3	14850±155

Experimental procedures. All experiments were performed at 20 °C, and an orbital shaker (Luckham R100/TW Rotatest Shaker) was used to shake 100 cm³ leachate NF samples in 250 cm³ glass bottles at 250 rpm. Na₂S₂O₈, KMnO₄ and H₂O₂ (37%) were used to prepare stock solutions for oxidation. The schematic view of the experimental setup is given in Fig. 2.

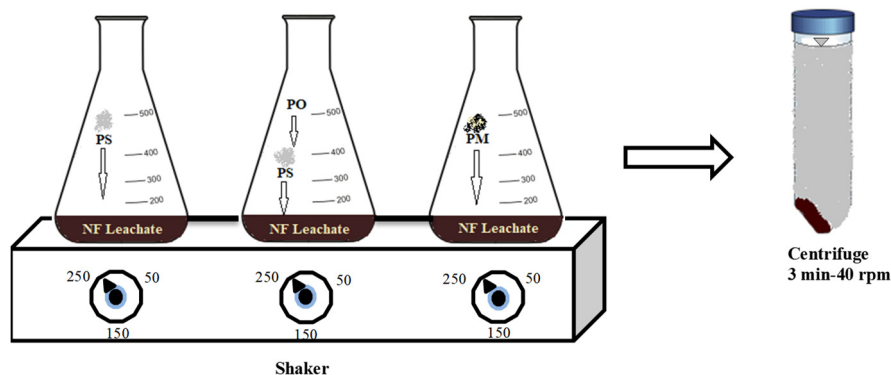


Fig. 2. Experimental set-up for PS, P-PS and PM oxidations

As seen in Fig. 2, PS (persulfate), PO (peroxide), and PM (permanganate) represent the oxidants added to leachate NF concentrate. The process variables were selected as oxidant dosage, pH, and reaction time. The ranges of the variables were determined by conducting preliminary experiments. To determine the optimum dosage of the oxidants, different doses (5–55 g PS/dm³, 0.5–6 g PM/dm³, 13.5–57.6 g PO/dm³) into the sample (100 cm³) of the oxidants were added before each run by keeping the other variables constant. All trials were conducted with the original leachate (pH 7.74) by shaking for 60 min, without pH adjustment for optimum dosage determination. In the second step, sodium hydroxide and sulfuric acid solutions were used to adjust the pH value of the samples in the range of 2–12 to determine the optimum pH value. The experiments were conducted with the optimum oxidant doses and 60 min of reaction time. At the last stage, the optimum reaction time was determined by the experiments carried out at the optimal pH value and with the optimum oxidant doses. H₂O₂ (37%) was used for the activation of the PS oxidant. For the best performance, the maximum removal ratio was obtained by the addition of the H₂O₂ reagent into the oxidation reactor gradually. PS and PO were added in varying ratios from 1:0.43 to 1:1.85 before each run. Oxidation by PS was optimized at its constant dosage of 25 g/dm³, according to the data obtained from the

preliminary experiments. All samples were centrifuged at 40 rpm for 3 min and then filtered through a 0.45 μm membrane before analysis. All experiments were performed in triplicates, and the averages are reported.

Analytical method. Biodegradable and non-biodegradable COD are subdivisions of total COD, which are in turn divided into the particulate and soluble portions [14]. The biodegradable and non-biodegradable fractions of COD were attained according to the method reported by Abu Amr et al. [14]. In the method described by Abu Amr et al. [14], the aeration of the samples was suggested till providing the lowest COD value which is determined as COD_f . The lowest COD value was determined by measuring the COD concentrations of the samples every 24 hours for 17 days. Water loss in the samples was compensated by the addition of distilled water.

Biodegradable COD fraction was calculated through the following equation:

$$\text{COD}_{\text{bi}} = \text{COD}_i - \text{COD}_f \quad (1)$$

In equation (1), COD_{bi} represents the biodegradable COD proportion, COD_i represents the initial COD concentration, and COD_f is the COD value after aeration. The COD_f value may be taken as the non-biodegradable COD proportion.

ZnSO_4 coagulant method was used to determine soluble COD (COD_s) and particulate COD (PCOD) fractions [12]. 1 cm^3 of 0.6 M ZnSO_4 solution was added in a 100 cm^3 sample. Preceding the coagulant addition, the pH level of the sample was adjusted as 10.5. The sample was mixed at a high rotation rate (about 200 rpm) for 1 min and a low rotation rate (30 rpm) for 5 min by the use of magnetic stirrer. After the sample was allowed to settle for an hour and 30 cm^3 supernatant was filtered, the COD value of the sample was analyzed. COD concentration obtained was expressed as soluble COD (COD_s). The subtracting COD_s value from the initial COD value, the particular COD (COD_p) value was determined. Soluble and particular COD values were calculated by using the following equations:

$$\text{COD}_s = \frac{\text{COD}_s}{\text{COD}} \times 100\% \quad (2)$$

$$\text{COD}_p = \frac{\text{COD} - \text{COD}_s}{\text{COD}} \times 100\% \quad (3)$$

where COD is the total COD, mg/dm^3 , COD_p represents the particular COD, mg/dm^3 , and COD_s represents the total soluble COD, mg/dm^3 .

Total soluble COD_s was also the sum of biodegradable soluble COD (COD_{sbi}) and non-biodegradable soluble COD (COD_{subi}). COD_{sbi} and COD_{subi} fractions were calculated using equations given by Abu Amr et al. [14]. The total soluble COD includes the

biodegradable and non-biodegradable fractions. COD_{sbi} is expressed as biodegradable soluble COD, and non-biodegradable soluble COD is obtained by subtracting the COD_{sbi} value from the COD_s value. The values of COD_{sbi} (biodegradable soluble COD) and COD_{subi} (non-biodegradable soluble COD) were calculated by equation (4).

$$COD_{sbi} = \frac{COD_{bi}}{COD_s} \times 100\% \quad (4)$$

$$COD_{subi} = \frac{COD_s - COD_{bi}}{COD_s} \times 100\% \quad (5)$$

In equation (5), COD_{sbi} represents biodegradable soluble COD, COD_{subi} represents non-biodegradable soluble COD, COD_{bi} represents biodegradable COD, and COD_s represents the total soluble COD value.

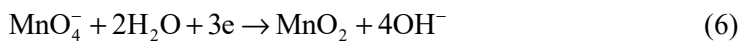
TOC concentrations in leachate NF concentrate before and after oxidation were measured using a Shimadzu TOC-Vcpn analyzer.

3. RESULTS AND DISCUSSION

3.1. EFFECT OF THE PS, P-PS AND PM OXIDANTS ON COD AND TOC REMOVAL

The effects of the oxidant doses (persulfate, persulfate-peroxide, and permanganate), pH, and reaction time on COD and TOC removal from the leachate NF concentrate are shown in Figs. 3–5. As can be seen in Fig. 3, the removal efficiencies increased as the oxidant doses increased. This may be explained by the higher quantity of the photons absorbed and reacted with oxidant at high concentrations. Though higher dosages of oxidants will provide more hydroxyl and sulfate radical formation, and these radicals that will be generated will react with organic and inorganic contaminants, the requirement of the optimum dosage of the oxidants is important for obtaining lower operational costs. Additionally, Wang et al. [15] and Abdullah et al. [11] concluded that excess dosages of oxidants tend to decrease removal efficiencies because of the scavenging effect.

The optimum PS and PM doses were determined to be 25 g/dm³ and 4 g/dm³, respectively. The difference between the removal efficiencies achieved by each oxidant may be explained by the type of the organic matter targeted by each of the oxidant and by the amount of the hydroxyl and sulfate radicals generated [11]. $KMnO_4$ is a highly reactive reagent used in the oxidation of various organic and inorganic substances. $KMnO_4$ is reduced to manganese dioxide (MnO_2) forming precipitates. The following equation gives the half-reaction of $KMnO_4$ at natural pH.



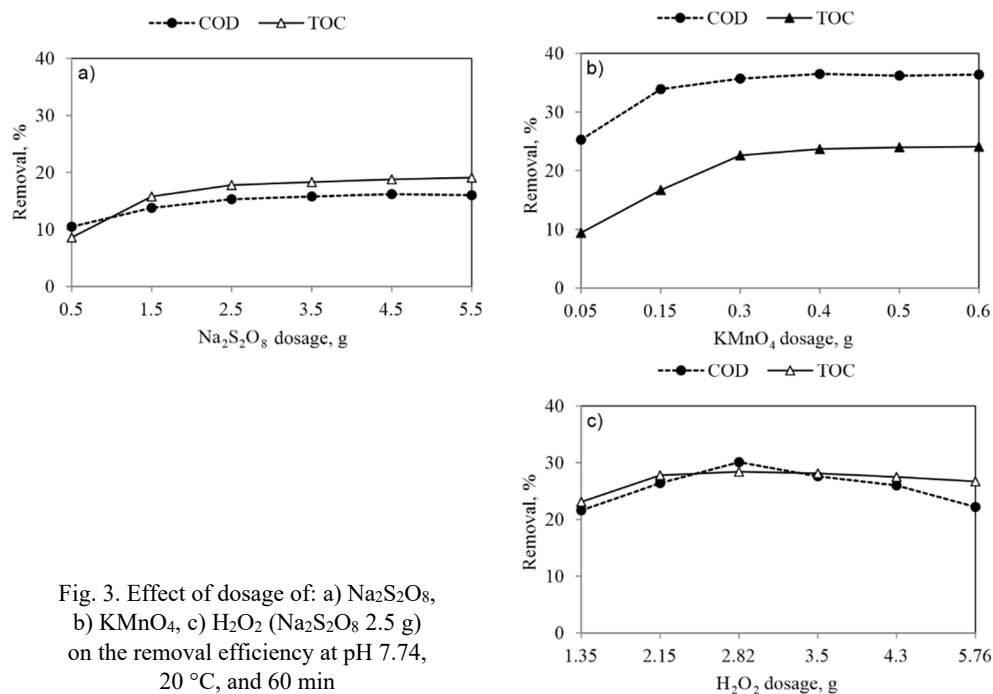
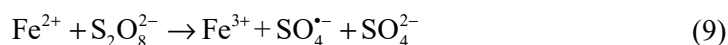
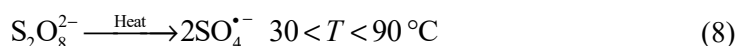


Fig. 3. Effect of dosage of: a) Na₂S₂O₈, b) KMnO₄, c) H₂O₂ (Na₂S₂O₈ 2.5 g) on the removal efficiency at pH 7.74, 20 °C, and 60 min

Although the PS reagent may have a role as a direct oxidant, it has a limited effect on degradation of organic matter at high concentrations [16]. Initiation of sulfate radicals enhance oxidation by PS, and generation of sulfate radicals during oxidation may be significantly increased by different factors (as iron ions, heat, H₂O₂ reagent, UV radiation) [10, 17, 18].



For persulfate activation and sulfate initiation, the H₂O₂ reagent was used. The performance of H₂O₂ in initiating sulfate radical formation from PS during the oxidation process was investigated by performing experiments at different PS/H₂O₂ ratios. The H₂O₂ doses added for PS activation were in the range of 13.5–57.6 g/dm³ whereas the PS dose was kept constant at the optimum value of 25 g/dm³. As may be seen in Fig. 3, the higher doses of H₂O₂ improved the removal efficiency of the targeted parameters.

In oxidation by P-PS, hydroxyl radicals (OH^\bullet) are formed by decomposition of H_2O_2 , and then, PS is activated to generate sulfate radicals [18, 19]. Both sulfate and hydroxyl radicals participate in COD and TOC degradation. The maximum COD removal ratio increased from 24.5% to 32.9% by H_2O_2 activation.

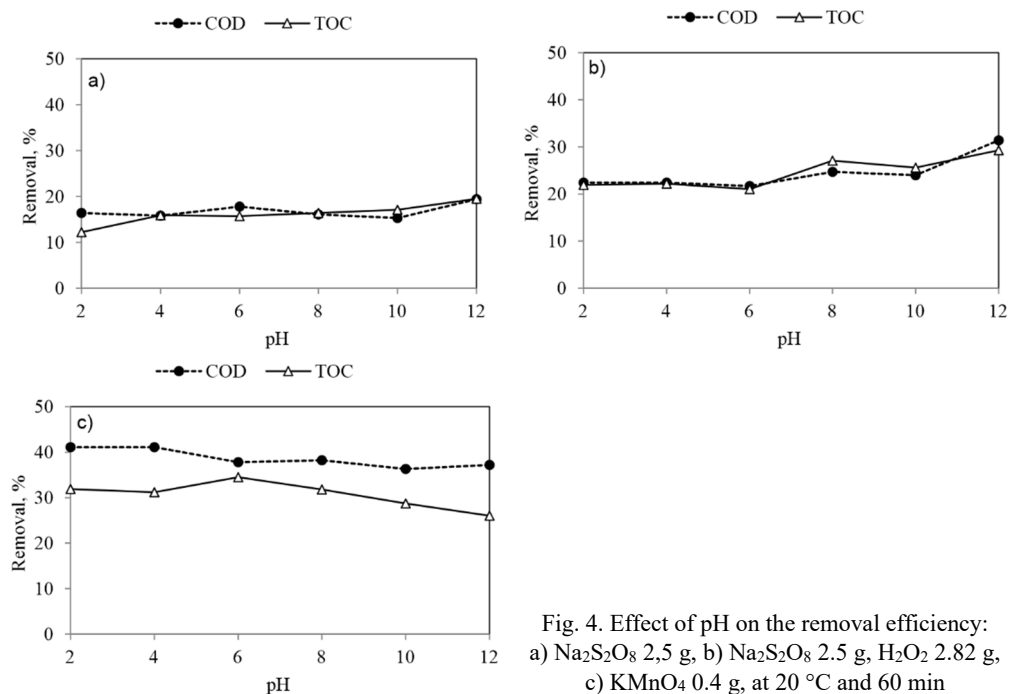


Fig. 4. Effect of pH on the removal efficiency: a) $\text{Na}_2\text{S}_2\text{O}_8$ 2,5 g, b) $\text{Na}_2\text{S}_2\text{O}_8$ 2.5 g, H_2O_2 2.82 g, c) KMnO_4 0.4 g, at 20 °C and 60 min

To determine the optimal pH for the oxidation processes by PS, P-PS, and PM experiments were carried out at pH between 2 and 12. The COD and TOC removal efficiencies increased as the pH increased (Fig. 4) in oxidation processes by PS and P-PS. At high pH values, hydroxyl radicals act to activate PS and initiate sulfate radicals. The results were consistent with the literature [12, 20, 21]. The optimum pH value determined for the PS and P-PS oxidants was 12 whereas optimum pH value determined for the PM oxidant was 4.

Figure 5 presents the effects of reaction time on COD and TOC removal by the oxidation processes of PS, P-PS, and PM. Different reaction times ranging from 15 min to 120 min were studied by keeping the other variables constant. The removal of organics increased with the increasing reaction time (Fig. 5). The optimum reaction time was found to be 60 min for the processes with PS and PM oxidants whereas optimum reaction time was determined as 90 min for the oxidation process by P-PS. At the optimized conditions, the maximum COD removal efficiencies were determined to be 24.5% and 39.1% for oxidation by the PS and PM oxidants, respectively after 60 min of oxidation.

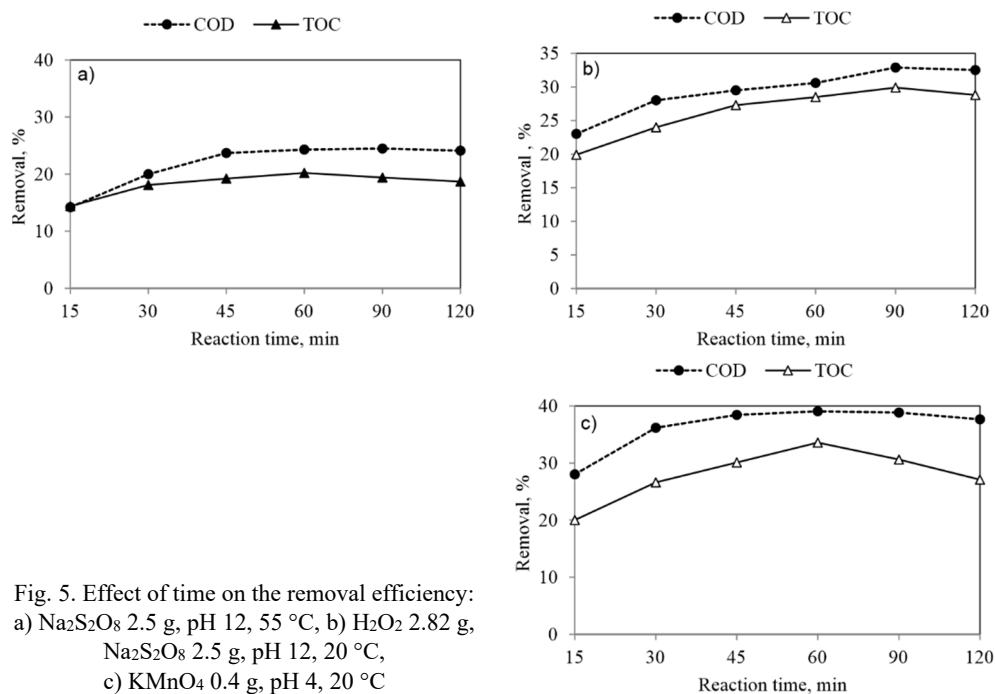


Fig. 5. Effect of time on the removal efficiency:
 a) $\text{Na}_2\text{S}_2\text{O}_8$ 2.5 g, pH 12, 55 °C, b) H_2O_2 2.82 g,
 $\text{Na}_2\text{S}_2\text{O}_8$ 2.5 g, pH 12, 20 °C,
 c) KMnO_4 0.4 g, pH 4, 20 °C

3.2. EFFECT OF PS, P-PS AND PM OXIDANTS ON COD FRACTIONS

The biodegradability of stabilized leachate may be improved by advanced treatment processes [12, 14, 22, 23]. The changes in the COD fractions of leachate NF concentrate before, and after the advanced treatment processes using PS, P-PS, and PM oxidants are presented in Table 2, and Fig. 6. The biodegradable COD fraction rose from 6.37% to 9.25%, 11.51%, and 13.64%, whereas the non-biodegradable COD fraction decreased from 93.63% to 90.75%, 88.49%, and 86.36% by the advanced oxidation processes using the PS, P-PS, and PM oxidants, respectively. With the use of PS, P-PS, and PM oxidants, respectively, about 25%, 34%, and 46% elimination of non-biodegradable COD was achieved.

The soluble, and particulate COD fractions of the raw leachate NF concentrate were found to be 71.14%, and 28.86%, respectively. Hilles et al. [12], and Abu Amr et al. [14] determined the soluble COD fraction of stabilized leachate as 70%, and 59%, respectively, whereas they found the particulate COD fraction as 30%, and 41%, respectively. Researchers concluded that insoluble organics can be converted to soluble organics by advanced treatment processes [18, 19, 22]. In this study, the soluble COD fraction increased to 87.23%, 89.03%, and 84.58% after oxidation by PS, P-PS, and PM, respectively. The results were consistent with the literature [18, 22].

The soluble, and particulate COD fractions of the leachate NF concentrate were found to be similar to that of the stabilized leachate but the content of the biodegradable COD fraction was found to be lower, and the content of non-biodegradable COD fraction was determined to be higher in comparison to the stabilized leachate. This was because, in this study, membrane treatment was applied to biologically treated leachate, and the effect of advanced treatment processes on the concentrate was investigated, whereas, in studies in the literature, advanced oxidation processes were applied to mature landfill leachate. The effects of the PS, P-PS, and PM oxidants on the content of the sub-fractions of soluble COD, the soluble biodegradable COD, and soluble non-biodegradable COD fractions, are also shown in Fig. 6. It may be concluded that the PM oxidation process was the most effective in all of the advanced treatment processes applied to the concentrate in this study, and H_2O_2 activation increases the performance of the PS oxidation process.

Table 2

COD fractions of leachate NF concentrate
before and after advanced oxidation treatment [mg/dm^3]

Medium	COD _i	COD _s	COD _p	COD _{bi}	COD _{ubi}	COD _{sbi}	COD _{subi}
Raw NF leachate	10 000	7114	2886	637	9363	895	9105
PS	7738	6750	988	716	7022	821	6917
P-PS	6939	6178	761	799	6140	897	6042
PM	5791	4898	893	790	5001	934	4857

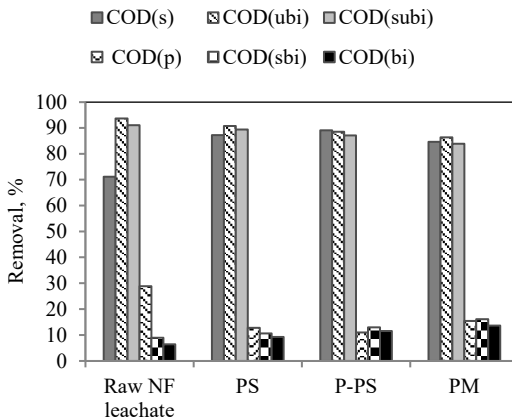


Fig. 6. The effect of PS, P-PS, and PM oxidations on COD fractions

4. CONCLUSION

The performance of persulfate (PS), peroxide activated persulfate (P-PS), and permanganate (PM) as a reagent in advanced oxidation of organic matter of leachate NF concentrate was investigated. The effects of the reagents on the biodegradability and

solubility of the leachate NF concentrate were also studied. The results of the study showed that 24.5%, 32.9%, and 39.5% total COD removal efficiencies were obtained respectively, by the advanced oxidation processes using PS, P-PS, and PM as the oxidants under the optimum conditions. The non-biodegradable COD fraction decreased dramatically, and the insoluble organic content was significantly converted into a soluble one. Thereby, the content of the soluble COD fraction increased. The initial COD concentration of the leachate nanofiltration concentrate was determined to be 10 000 mg/dm³ consisting of 9363 mg/dm³ non-biodegradable COD. At optimized conditions, the highest COD removal rates were obtained in the oxidation by PM. Since H₂O₂ activated the formation of sulfate radicals, the peroxide-activated PS process was found to be more efficient than the PS process. The biodegradable COD fraction increased from 6.37% to 9.25%, 11.51%, and 13.64% after the oxidation treatment processes by PS, P-PS, and PM, respectively. About 25%, 34%, and 46% removal efficiencies of inert COD were also obtained after PS, P-PS, and PM oxidation processes. Furthermore, it can be concluded that converting non-biodegradable COD into biodegradable COD was found to be 2.88%, 5.14%, and 7.27% using oxidation treatment by PS, P-PS, and PM, respectively. The soluble COD fraction increased from 71.1% to 87.2%, 89.0%, and 84.6%, respectively, after oxidation treatment processes by PS, P-PS, and PM, due to the conversion of the insoluble content of the concentrate into a soluble one. Although the highest removal efficiencies were achieved by PM oxidation system, it may be concluded that the three AOPs using different reagents or mechanisms were efficient methods to convert the insoluble COD fraction to the soluble COD fraction, and remove the inert COD fraction from the leachate NF concentrate. Consequently, the advanced oxidation processes using PS, P-PS, and PM as the reagents may be used as post-treatment processes for particularly inert COD removal.

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