International Journal of EngineeringScienceInvention (IJESI)
ISSN (Online): 2319 – 6734, ISSN (Print): 2319 – 6726
www.ijesi.org ||Volume 8 Issue 11 Series. II || November 2019 || PP 53-58

Pre-treatment of Sludge using Fenton Process

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ABSTRACT:*The anaerobic digestion process is an extremely common process for stabilization, with the advantage of producing CH_4. However, the rate limiting step in the anaerobic digestion process is the hydrolysis step. Researchers use different disintegration methods of sludge to accelerate the anaerobic digestion and increase the degree of stabilization.The aim of this study is to investigate the effect of Fenton Process on waste activated sludge disintegration. As a result of Fenton Process carried out by adding iron and hydrogen peroxide in different doses, the degree of disintegration, total solids content, supernatant and sludge characteristics were investigated.Fenton pre-treatment provides disintegration of treatment sludge when used as a pretreatment process before anaerobic digestion. The highest disintegration degree value (31%) was obtained by application of 8 gFe /gTS and 120 gH_2O_2 / gTS.The disintegration of the flocks in the sludge with the effect of Fe and H_2O_2 caused an increase in COD concentration in the liquid phase.It was found that the application of Fenton Process had a positive effect on the filterability of sludge.*

KEYWORDS: *Fenton Process, disintegration, sludge pre-treatment, anaerobic digestion*

Date of Submission: 15-12-2019 Date of acceptance: 27-12-2019

I. INTRODUCTION

The activated sludge process, which is widely used in domestic and industrial wastewater treatment, produces a large amount of waste activated sludge. Due to the high quantity and potential environmental risk, sludge treatment and disposal has become a major problem for wastewater treatment plants[1]. Sludge treatment and disposal accounts for 60% of the total cost of wastewater treatment. On the other hand, sludge is a potential source because it contains a large amount of organic matter which can be converted to biogas by anaerobic digestion [2]. Anaerobic digestion also provides a 40-50% reduction in sludge [3]. However, hydrolysis of large organic molecules is the rate-limiting step of anaerobic sludge digestion. In order to increase the digestibility of sludge, a pretreatment process called disintegration method which accelerates low biodegradation has been developed [4,5]. Degradation of sludge leads to disruption of the cell wall and release of extracellular and intracellular matter [2,5,6]. The efficiency of sludge digesters can be improved by incorporating a pretreatment process to accelerate biochemical hydrolysis reactions before anaerobic digestion. [7]. As a result of effective disintegration, most of the organic materials in the sludge go into the liquid phase, the solid sludge particles that do not pass into the liquid phase contain mostly inorganic substances and therefore the disintegrated treatment sludges reach higher solids content after dewatering [5,8]. The anaerobic digestion of the disintegrated sludge allows for a high degree of stabilization due to the advanced degradation of the organic material, so that the amount of waste sludge can be reduced by 30-40 % compared to the conventional anaerobic decay process. Mechanical, acoustic, chemical, physical, thermal and biological processes are used as disintegration method. In addition, combinations of these methods have been applied for synergistic effects [5,9]. Fenton Process is one of these methods. During Fenton oxidation, waste is destroyed and water and organic substances are released. Fenton reaction using Fe (II) to catalyze H_2O_2 to form the OH radical at high acidic pH has proven to be a promising advanced oxidation process in wastewater treatment [10,11]. The use of this process, which is widely used in wastewater treatment, for sludge treatment, has also been discussed to improve the properties of treatment sludges[12,13].

The aim of this study is to investigate the effect of Fenton Process on waste activated sludge disintegration. As a result of Fenton Process carried out by adding iron and hydrogen peroxide in different doses, the degree of disintegration, total solids content, sludge supernatant and sludge characteristics were investigated.

II. MATERIAL AND METHODS

2.1.Sludge Characteristics

Sludge was taken from a municipal wastewater treatment plant. It was sampled from the return sludge line. Sludge used in the study was characterized in terms of total solids (TS), volatile solids (VS), soluble chemical oxygen demand (CODs), total nitrogen (TN) and total phopshorus (TP) of the supernatant [14].

2.2.Determination of the optimal dose

In this study, 6 different doses of iron and hydrogen peroxide were applied to sludge. The pH of the 500 mL sludge sample was brought to 3 with H_2SO_4. Fe (II), which was calculated according to the solids content, was then added. Since $FeSO_4.7H_2O$ was used as Fe (II) source, it was calculated how much $FeSO_4.7H_2O$ would be used in order to obtain the desired amount of Fe (II) from that compound. Then, different doses of H_2O_2 are added. The mixture is rotated at 100 rpm for 60 min. After the reaction, each sludge sample was neutralized with $Ca(OH)_2$. In this study, Fe (II) and H_2O_2 doses applied as Fenton Process are shown in Table 2.1. Each sample was numbered 1 to 6.

Table 1. Fe (II) and H_2O_2 doses applied to sludge

Fenton Dose	**Fe (II)**	**H_2O_2**
1	2 gFe/kgTS	30 gH_2O_2/kgTS
2	4 gFe/kgTS	60 gH_2O_2/kgTS
3	6 gFe/kgTS	90 gH_2O_2/kgTS
4	8 gFe/kgTS	120 gH_2O_2/kgTS
5	10 gFe/kgTS	150 gH_2O_2/kgTS
6	120 gFe/kgTS	180 gH_2O_2/kgTS

2.3.Analytic Methods

2.3.1.Soluble Chemical Oxygen Demand

Chemical Oxygen Demand (COD) analysis was carried out in both raw sludge and supernatant of the sludge disintegrated with Fenton Process. In addition, when calculating the degree of disintegration, COD analysis was performed in supernatant after chemical disintegration. Sludge was centrifuged at 4000 rpm for 10 min. All COD analyzes were performed according to the Standard Methods [14].

2.3.2.Determinantion of Disintegration Degree

The COD values obtained after centrifugation of the sludge obtained after the application of Fenton to each sample of sludge and after chemical disintegration were obtained using the formula (DD = [(COD_1 - COD_2) / (COD_3 - COD_2)] (COD_3 - COD_2)] previously given in equation (2.1). is used to calculate the degree of disintegration.

The degree of disintegration (DD) parameter is used to evaluate the reducibility of sludge after disintegration process. The degree of disintegration is a parameter that gives the highest solubility point in the sludge. Since the aim of the disintegration process is to convert the high content of organic matter in the sludge into a form that can be used more easily by bacteria, it is very important that the sludge passes into soluble form. This parameter is calculated in% using the following correlation.

DD = [(COD_1 - COD_2) / (COD_3 - COD_2)]. one hundred

Here;

COD_1 = COD concentration in sludge after disintegration

COD_2 = COD concentration in raw sludge

COD_3 = COD concentration in sludge after chemical disintegration

Chemical disintegration means that the sludge is treated at 90 °C for 10 minutes after the addition of NaOH. Sample is obtained by centrifugation of sludge at 15 000 rpm for 20 minutes at 4 °C [15].

2.3.3.Total solid and total organic matter analysis

Total and volatile solids content was determined before and after Fenton Process application. Total and volatile solids experiments were performed according to Standard Methods[14].

2.3.4.pH and temperature

pH measurement in raw sludge was measured using a Hach pH meter.

2.3.5.Specific Resistance to Filtration (SRF)

The specific filter resistance (SRF) measurements of dewatering ability by vacuum filtration were used in Bucher Funnel test setup. The Bucher Funnel test was carried out in two steps: direct and after chemical treatment for each sample of sludge. In the filtration test, 100 mL sample volume was taken for each experiment. Whatman 2 filter paper was used in the filtration process and the adapter pump was adjusted to maintain a pressure of 49 cm Hg in the vacuum pump. For time control, the filtration time was accurately measured using a stopwatch. In order to calculate the sludge specific filter resistance, standard linear vacuum filtration lines were drawn and b slopes of these lines were calculated. This test allows the examination of the effects of changes in solids concentration and particle size to specific resistance. The Bucher Funnel test is performed for each sample of the sludge directly and in two steps after chemical treatment has been applied. In the tests performed without chemical conditioning process, after the pH, O_2, conductivity and temperature are measured, direct filtration test is applied to the sludge sample without adding any chemical dose. In the tests carried out after applying the chemical conditioning process, conditioning is applied by adding different doses of chemicals to the sludge. To calculate the sludge specific resistance, the slope (b) of the V - t / V standard linear vacuum filtration lines is calculated between the time t and the volume of the V filtrate recorded during the filtration. Specific resistance values of sludge samples by vacuum filtration,

It is calculated using the following mathematically formulated statement according to Poiseuille and Darcy's laws.

$$r = \frac{2 * A^\wedge 2 * P * b}{\mu * c} \qquad (2.1)$$

r: specific filter resistance, m / kg
P: filtration pressure, N / m^2
A: Filter area, m^2
b: time / volume - slope of the volume curve, s / $(m^3)^2$
μ: filtrate viscosity, N.s / m^2
c: the amount of solids deposited per unit filtrate volume (dry matter concentration in the cake), kg / m^3

The Buchner funnel test apparatus consists of a graduated cylinder, a Buchner funnel and a vacuum pump, as shown in Fig. 1.

Figure 1. Setup for SRF

III. RESULTS AND DISCUSSION

3.1. Sludge Characteristics

Table 2. Sludge characteristic of return sludge

Parametre	Return Sludge
pH	7,65
Temperature (^oC)	14,4
Total Solids (TS) (mg/L)	2910
VolatileSolids (VS) (mg/L)	2060
Total Nitrogen in thesupernatant (TN) (mg/L)	40
Total Phosphorus in thesupernatant (TP) (mg/L)	18,1
SolubleChemicalOxygenDemand (CODs) (mg/L)	112

3.2.Dissolved COD and Disintegration Degree (DD) and Optimum Dose

Fig 2 shows the DD that varies depending on the COD concentrations in the supernatant which varies with the dose of Fenton application. In Fenton pretreatment application, COD concentrations increased rapidly in sludge upper water due to increased Fe and H_2O_2 dose. There was no increase in doses above 8 gFe / gTS and 120 gH_2O_2 / gTS. It is possible to explain the increase in COD concentration in the supernatant water by the disintegration of the sludge solids by the effect of Fenton treatment. Because the disintegration process changes many properties of the sludge. When this process is applied, the sludge flock structure is disrupted and microbial cell walls are destroyed. By breaking down the cell wall, the substances protected by the cell wall pass into the liquid phase and turn into soluble form.

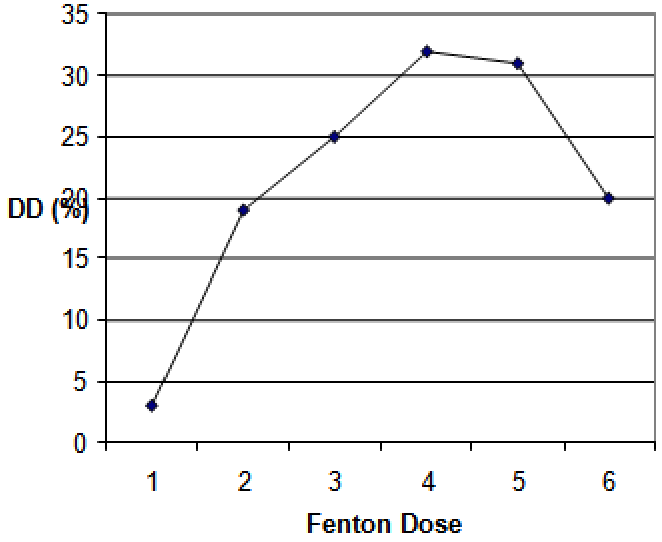

Figure 2.Disintegration Degree (DD %)

3.3. Change of Total Solid and Volatile Solid

Fenton pretreatment process resulted in the reduction of solids in the sludge (Fig. 3). The total solids content decreased significantly, especially after 4 g Fe / gTS and 60 gH_2O_2 / gkm. In the application of Fenton dose, which reached the highest DD value, TS content decreased from 2911 mg /L to 2536 mg/L and VS content decreased from 2696 mg / L to 1902 mg / L compared to raw sludge.

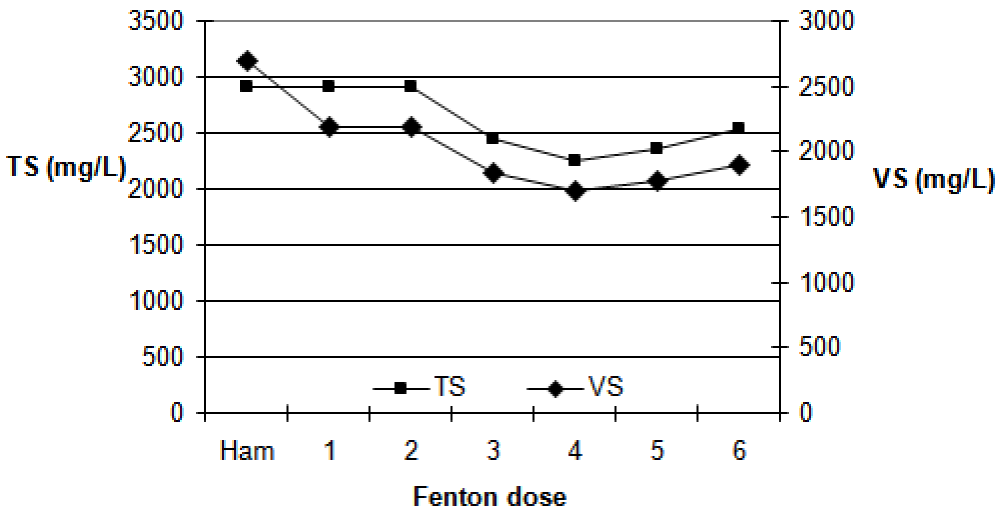

Figure 3..Change of Total Solid and Volatile Solid

3.4. Total Nitrogen (TN) and Total Phosphorus (TP)

The dissolution of the intracellular material by the Fenton process causes total nitrogen and total phosphorus increase in the liquid phase. Total nitrogen and total phosphorus concentrations in the sludge liquid phase are given in Figure 4.3. Total nitrogen and total phosphorus concentrations in the sludge liquid phase were increased due to increasing Fenton dose. Total nitrogen and total phosphorus concentrations were 40 mg /L and 18.1 mg /L in the raw sludge sample, respectively, and increased to 60 mg /L and 20 mg /L at the optimum dose of Fenton 4, where the highest degree of disintegration was achieved.

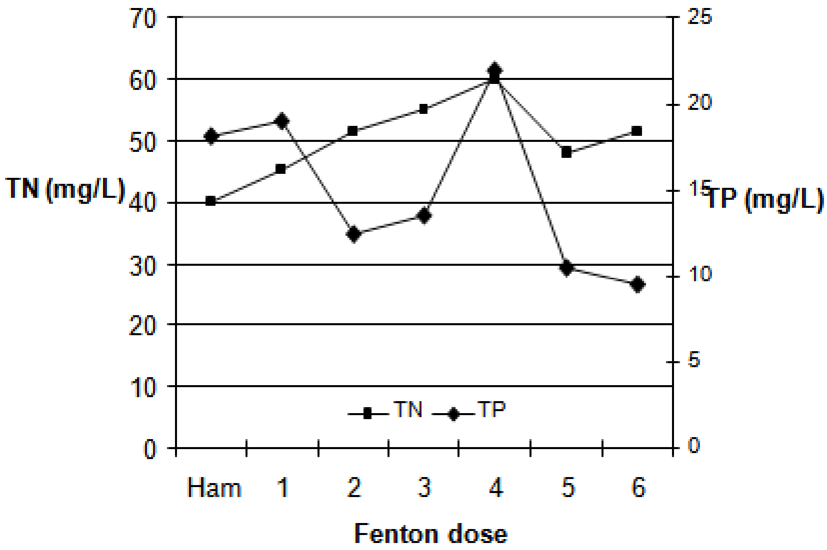

Figure 4.Total nitrogen and total phosphorus concentrations in the slugdesupernatant after Fenton Process

3.5. Changes of Sludge Characteristics in terms of Specific Resistance to Filtration (SRF)

The change in SRF after Fenton Process demonstrating the sludge filterability is shown in Table 2. According to the results of SRF tests, SRF values decreased after Fenton administration. Until the application of 8 gFe/gTS and 120 gH_2O_2/gTS which was determined as the optimum ozone dose, ÖFD values gradually decreased and it was observed that the optimum dose was $6{,}5.10^{14}$ m / kg and the same values were taken at higher doses. The decrease in SRF values after Fenton administration shows that Fenton Process has a positive effect on filterability.

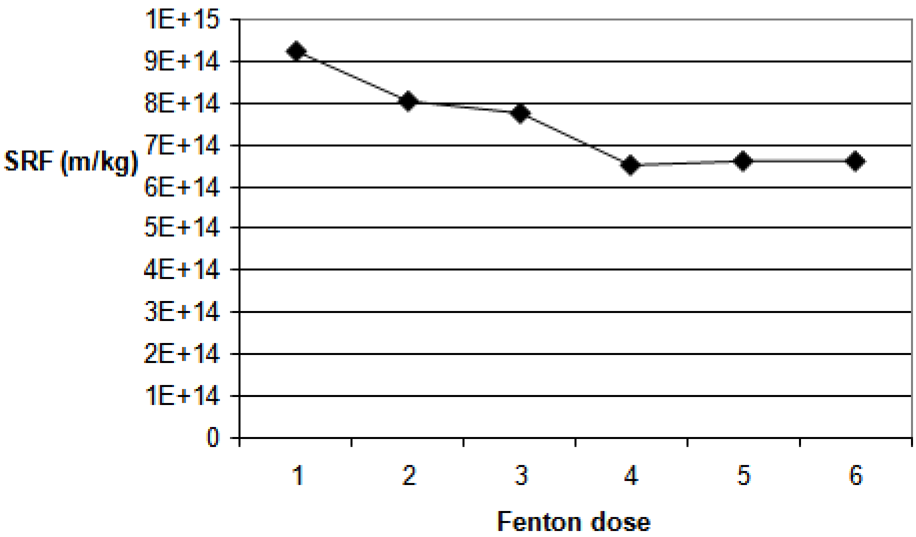

Figure 5. Change of SRF after Fenton Process

IV. Conclusion

As a result of this study, it was determined that Fenton pre-treatment provides disintegration of treatment sludge when used as a pretreatment process before anaerobic digestion. The highest DD value (31%) was obtained by application of 8 gFe/gTS and 120 gH_2O_2/gTS, and it was concluded that this value was sufficient for flock disintegration. The Fenton Process increased the solubility of sludge solids. Fenton application changed the supernatant properties. The disintegration of the flocks in the sludge with the effect of Fe^{+2} and H_2O_2 caused an increase in COD concentration in the liquid phase. The dissolution of intracellular material by Fenton process caused total nitrogen and total phosphorus increase in liquid phase. When the effect of Fenton Process on the filterability properties of sludge was examined, it was found that the application of Fenton Process had a positive effect on the filterability of sludge.

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Özlem Demir "Pre-treatment of SludgeusingFentonProcess"International Journal of EngineeringScienceInvention (IJESI), Vol. 08, No.11, 2019, PP 53-58