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Treatment of net washing wastewater by Fenton process

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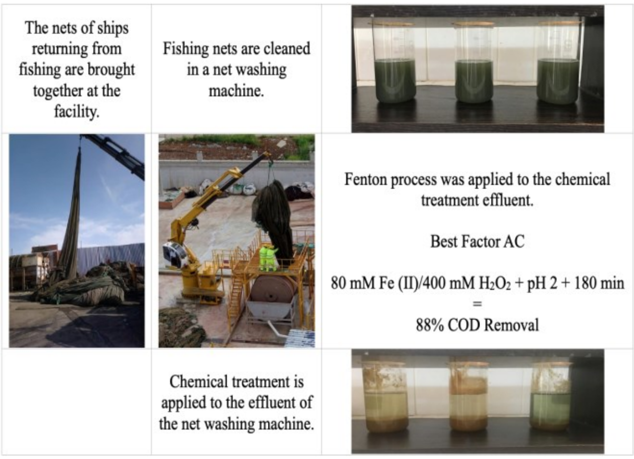
Graphical abstract



Abstract

The discharge of wastewater from net washing is one of the most significant problems in the marine ecosystem because of its high organic and saline content. Sea water quality is deteriorated every day due to the high amount of organic matter produced in the aquaculture sector. The aim of this study was to investigate the treatability of net washing wastewater by Fenton process. The efficiency of advanced oxidation processes (AOPs) in the treatability of net washing wastewater was evaluated through chemical oxygen demand (COD) and color removal. The effects of H_2O_2, and Fe (II) dosages, pH and reaction time were investigated, and the parameters were optimized by 2^3 full factorial design principles and variance analysis (ANOVA). The results showed that H_2O_2 dosage was the most effective parameter for Fenton process. The maximum COD removal was found at Fe (II)/H_2O_2 concentration ratio of 1/5 and the 180 minutes reaction time. The COD removal increased from 77 % to 88 % by increasing the peroxide dose from 80 mM to 400 mM. The total R^2 for the three factors was calculated to be 59.67%. According to the experimental results, in the factor analysis between the three variables and COD removal, only the variation in peroxide concentration was statistically significant. Since the efficiency of biological treatment is unstable in treating of high organic matter and saline wastewaters, the Fenton process has great potential in the treatment of net washing wastewater.

Keywords: ANOVA, factorial design, fenton, fishing net, wastewater, marine ecology

1. Introduction

One of the problems that the world is the unavoidable population growth. According to estimations for the middle of the twenty-first century, there are around 10 million people on earth (McMichael *et al*., 2020). The biggest problem in this respect, on a global basis, is food. Given that the oceans and seas make up 3/4 of the earth, the demand for fish will surely increase. In 2016, 171 million tons of seafood were thought to have been consumed (FAO, 2018). The consumption of seafood is expected to increase annually, which is expected to influence the ecology.

Traditional methods like tossing nets into the water have been used for sea fishing in deep waters since ancient time (Karadurmus *et al*., 2021). Fishing nets are commonly used by fishermen to catch fish, but over time they become dirty, clogged with debris, and can even develop odors (Galimany *et al*., 2019; Comas *et al*., 2021). After the fishing activity at sea, the nets need to be hauled ashore, separated, and cleaned. The nets must be carefully cleaned chemically and physically before being reused. The main reasons for cleaning the nets are the loss in oxygen transmission during fishing operations and the restriction of the nets' pores (Srithongouthai *et al*., 2006). It's typical to observe the production of solid wastes and wastewater after cleaning of the nets.

Wastewater from the marine aquaculture has high levels of nitrogen, phosphate, organic matter, salt, oil, and grease (Ching and Redzwan, 2017; Kamau *et al*., 2021). Shellfish, dead fish, and fish food are the sources of organic and inorganic materials and are captured in fishing nets (Reyes *et al*., 2020). Wastewater from fish nets causes an increase in dissolved and suspended organic matter in the aquatic environment, which also decrease the quantity of dissolved oxygen (Ariyunita and Listyawati, 2019). Cleaning fishing nets becomes necessary when considering marine environment and sustainable fisheries (Castillo-Carvajal *et al*., 2014). Solid waste from net cleaning can be burned or dumped in landfills, while treated wastewater can be sent back into the sea. If net washing wastewater is discharged into the sea without treatment, the high organic pollution load, may have an adverse effect on the ecosystem such as mucilage (Savun-Hekimoglu and Gazioglu, 2021). Therefore, it is important to investigate the treatability of non-biologically treated wastewater having a considerable organic content using advanced oxidation processes (AOPs) (Amor *et al*., 2019).

A fishing net washing machine is a specialized type of washing system designed for cleaning nets (Dijkstra *et al*., 2021). Fishing net washing machines are an important tool for the fishing industry, helping to promote sustainability and reduce the environmental impact of fishing activities (Rowan, 2023). Because the water used to wash fishing nets is so salty, higher salinity reduces the effectiveness of biological treatment methods by restricting bacterial life (Srivastava *et al*., 2021). From an economic point of view, AOPs such as Fenton oxidation may offer alternative and promising treatment methods for net washing wastewater. Fenton oxidation is an efficient wastewater treatment processes that have been used to remove organic matter from various industrial wastewaters, such as cheese whey (Gurtekin, 2011), pesticides (Katip, 2019), pulp and paper mills (Irfan *et al*., 2017), pharmaceuticals (Argun, 2017), dyes (Ertugay and Acar, 2017), leachate (Ramirez-Sosa *et al*., 2013; Mohan and Arulmathi, 2022) and bilge water (Oz and Cetin, 2022). During Fenton oxidation, organic substances are degraded by hydroxyl radicals with high oxidation potential, which are formed by the catalytic cascade reaction of hydrogen peroxide with iron (II) salts under strongly acidic conditions (Kumar, 2011; Al Jabri *et al*., 2019).

To the best of our knowledge this is the first study for focusing on treatment of net washing wastewater. The goal of our study was to determine effectiveness of Fenton oxidation approach for removing organic components and color from fish net washing wastewater. The optimal conditions, which was created using 2^3 full factorial design principles, were determined by ANOVA with using peroxide concentration, pH, and reaction time as variables.

2. Materials and methods

2.1. Characteristics of the fish net washing wastewater

The tested wastewater sample was obtained from effluent of coagulation-flocculation processes in the washing facility for fishing nets, which is located about 60 kilometers from the Mugla City center of Turkey. The pH values of the water samples were measured using a pH-meter of the VWR brand and the electrometric technique (Standard Method 4500-H+) (APHA, 1998). The total organic carbon (TOC) content of the samples was determined using the TOC-L CPH (Shimadzu) apparatus using the high temperature combustion method (Standard Method 5310B). The LCK 1014 kit was used for COD analysis. The Hach LCK 1014 kit's COD measurement range is 100 to 2,000 mg/L, and it is suitable for wastewater with high COD concentrations. Since samples with chloride concentrations up to 4,000 mg/L may be evaluated with the LCK 1014 kit, it is generally utilized for this purpose.

The color measurements in this study were determined with two different scales, Platinum-Cobalt (Pt-Co) and Spectral absorption coefficient (SAC), by using a spectrophotometer (Hach Lange DR-5000). Pt-Co method is used as color parameter in the discharge of colored wastewater to the receiving environment in current regulations for Turkey. SAC measurement is preferred instead of Pt-Co in industrial wastewater because of misleading results in colored wastewater. Misleading results were obtained in colored industrial wastewater in that case the color tones move away from the Pt-Co standard (Tozum-Akgul, 2022). Pt-Co measurements were performed at 465 nm and the measured absorbance values at 436 (yellow), 525 (red) and 620 nm (blue) were converted to SAC values according to the following equation for the SAC parameters.

$$SAC = \frac{A_\lambda}{d} x1,000 \quad (1)$$

where A_λ is the absorbance at wavelength λ nm and d, the path length of the cell (mm) and the SAC unit is m^{-1}.

The wastewater sample showed color values of 163.78 m^{-1}, 119.69 m^{-1}, and 97.64 m^{-1} at wavelengths of 436 nm, 525 nm, and 620 nm, respectively, in three tests using the SAC method, whereas the color value at 465 nm was 6160 Pt-Co. The characteristics of the wastewater of the net washing were shown in Table 1.

Table 1. Characterization of net washing wastewater

pH	7.20-7.71		
COD (mg/L)	1,400		
Conductivity (µs/cm)	28,000		
TOC (mg/L)	790		
Color 465 nm Pt-Co	6,160		
Color (SAC m^{-1})	λ_{436}	λ_{525}	λ_{620}
	163.78	119.69	97.64

2.2. Fenton experiments

In combination with a jar test system, batch glass reactors were used to conduct Fenton oxidation. pH, H_2O_2, and Fe (II) dosages are the variable parameters in Fenton process. A sample of wastewater of 500 mL was used for each experiment. All chemicals used in the study, including ferrous sulphate heptahydrate ($FeSO_4 \cdot 7H_2O_2$ content ≥ 99%), hydrogen peroxide (H_2O_2, 30% w/w), sodium hydroxide (NaOH) and sulfuric acid (H_2SO_4) and sodium sulfite (Na_2SO_3) were purchased from Merck. Prior to adding the necessary amounts of $FeSO_4.7H_2O$ (16, 40, 80 mM) and H_2O_2 (80, 200, 400 mM), the pH of the wastewater was first adjusted using 3 N NaOH and/or 3 N H_2SO_4. H_2O_2 was added to initiate the process. Different reaction periods were used for optimization. After reaction, pH of the solution was adjusted to 7.0 using 3 N NaOH to terminate the reaction and to allow the precipitation in the form of iron (III) hydroxide of the ferric ions. Residual H_2O_2 was quenched with Na_2SO_3 (Merck CAS: 7757-83-7) to prevent interference in COD measurements and to stop the reaction. Sodium sulfite react with H_2O_2, according to Eq. (2). The stoichiometric dose needed for sodium sulfite to accurately quench 1 mg/L H_2O_2 are 1 mg/L (Wang *et al*., 2013).

$$Na_2SO_3 + H_2O_2 \rightarrow Na_2SO_4 + H_2O \quad (2)$$

2.3. Full factorial design

The 2^3 full factorial designs were used to create the experimental design in the study. Table 2 shows the experimental variables that were employed in the study. Peroxide concentrations for component "A," pH for factor "B," and reaction time for factor "C" were the variables used for this investigation and total of 11 various test procedures were evaluated. By taking into account the lowest and maximum values of each variable, this design is accomplished. The variable's minimum and maximum values are indicated by the "-1" and "+1," respectively, that are displayed beneath the variable factors. The 2^3 full factorial design conditions are showed in Table 3.

Table 2. Minimum and maximum values of factors

Factor	Name	Lowest (-1)	Highest (+1)
A	Peroxide Concentrations (mM)	80	400
B	pH	2	4
C	Reaction Time (min)	60	180

Table 3. 2^3 full factorial design conditions

Run Order	Factors	Peroxide Concentrations (mM)	pH	Reaction Time (min)
1	1	80	2	60
2	A	400	2	60
3	B	80	4	60
4	AB	400	4	60
5	C	80	2	180
6	AC	400	2	180
7	BC	80	4	180
8	ABC	400	4	180
9	Central Point	200	3	120
10	Central Point	200	3	120
11	Central Point	200	3	120

3. Results and discussion

3.1. Treatability of net washing wastewater with Fenton Process

The conductivity of net washing wastewater was 28,000, µS/cm which was result of the seawater's salt content. The pH of the effluent fluctuates from 7.21 to 7.71 and average COD value was 1,400 mg/L. High conductivity in wastewater has a detrimental effect on the water's quality. TOC levels of wastewater were 790 mg/L. The color of wastewater sample tested in experiments was a vivid shade of green. In this study, wastewater color measurements were made using both Pt-Co and SAC methods.

The decrease in COD and color as 465 nm Pt-Co, SAC_{436}, SAC_{525} and SAC_{620} with reaction time for Fe (II)/H_2O_2 was shown in Figures 1 and 2. When the COD removal efficiencies were examined, it was determined that the constant pH (2) and the increasing peroxide concentration (from 80 mM to 400 mM) during the reaction (60 min) had a positive effect on the COD removal efficiency. The COD removal increased from 77 % to 88 % by increasing H_2O_2 dosage increasing from 80 to 400 mmol/L at pH 2 and Fe (II) dosage from 16 mM to 80 mM, respectively. H_2O_2 is a key component in the breakdown of organic molecules since it is the generator of hydroxyl radicals. The effectiveness of the treatment improves as the amount of hydrogen peroxide in the environment rises. But excessive use of H_2O_2 causes reaction with the hydroxyl radicals in the environment and inhibits the oxidation reaction, leading to a reduction of the yield (Kaya and Asci, 2019).

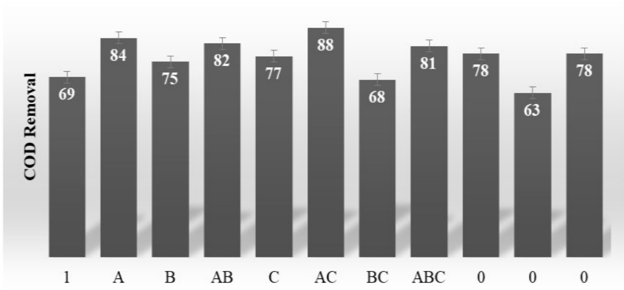
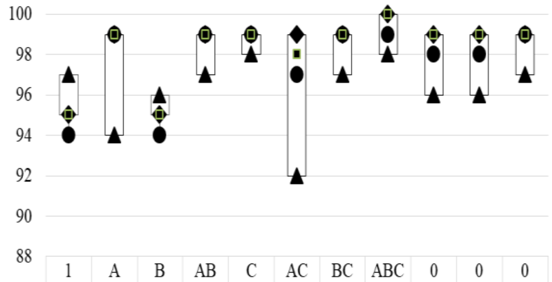


Figure 1. The COD removal efficiencies of the test matrices by factors (1: 80 mM H2O2 – pH 2 – 60 min; A: 400 mM H2O2 – pH 2 – 60 min; B: 80 mM H2O2 – pH 4 – 60 min; AB: 400 mM H2O2 – pH 4 – 60 min: C: 80 mM H2O2 – pH 2 – 180 min; AC: 400 mM H2O2 – pH 2 – 180 min; BC: 80 mM H2O2 – pH 4 – 180 min; ABC: 400 mM H2O2 – pH 4 – 180 min)

In addition, increasing the peroxide concentration (from 80 mM to 400 mM) during high pH (4) and long reaction time (180 min) resulted in increased COD removal efficiency. There are studies in which increasing the peroxide concentration has positive results on the Fenton process (Durgut *et al*., 2021; Oz and Cetin, 2022). The COD removal efficiencies and color removal efficiencies of the test matrices by factors were shown in Figures 1 and 2, respectively.



	1	A	B	AB	C	AC	BC	ABC	0	0	0
▲Pt-Co (%)	97	94	96	97	98	92	97	98	96	96	97
●SAC-436 (%)	94	99	94	99	99	97	99	99	98	98	99
◆SAC-620 (%)	95	99	95	99	99	99	99	100	99	99	99
■SAC-525 (%)	95	99	95	99	99	98	99	100	99	99	99

Figure 2. The color removal efficiencies of the test matrices by factors

To compare or gain insight, the wastewater used in the research is comparable to bilge waters in terms of conductivity and COD characteristics. According to scientific literature, the bilge wastewater's conductivity is in the range of 30,000 µS/cm (Ulucan *et al*., 2014) and COD values are between 1,000 and 2,500 mg/L (Oz and Cetin., 2022; Ulucan *et al*., 2014). Oz and Cetin (2022) obtained approximately 90% COD removal from bilge water.

Due to the complexity of the Fenton process, it is difficult to explain how Fenton's reagents degrade an organic substance (Tozum-Akgul, 2022). Although the Fenton system contains several reactions, the following steps might be given as a simplified, potential mechanism for the OH radicals' degradation of organic contaminants and color in net washing wastewater. The catalyst ferrous ions quickly break down H_2O_2 in the system's initial stage, creating the highly powerful oxidizing radical OH. The following step is target organic contaminants are attacked by the generated OH radicals, which additionally provide oxidation (Tozum-Akgul, 2022).

3.2. Variance analysis of Fenton process optimization

In the Fenton experiment, the Fe (II) dosage, H_2O_2 dosage, pH and time were investigated for optimization preliminary data (COD removal efficiency) were used to determine the levels of these factors used in the statistical model. The COD load of wastewater was 1,400 mg/L. In the experiments, an increase of about 8% in COD removal was observed when the duration in wastewater was increased from 60 min to 180 min. The best process optimization was determined at pH 2. Therefore, the pH range used in this study was set at 2-3. In the third part of the table, try to dose iron and peroxide together since there is a stoichiometric ratio between them. According to Fenton analysis results, highest COD and color removal could be observed in scenarios where Fe (II) dose was 80 mM and H_2O_2 dose was 400 mM and although the highest COD removal efficiency was 88% in all experiments. The matrix constructed from the obtained data was shown in Table 2. Based on the results in Figure 1, the ANOVA results for COD removal efficiency were shown in Table 4, indicating that only peroxide concentration efficiency may be significant. Therefore, by choosing the effective parameter $p > 0.05$ as the confidence interval, the following equation follows:

$$\begin{aligned} \textit{COD Removal} = {} & 43.2 + \\ & (0.002833 * \textit{Peroxide Concentration}) \\ & + (7.84 * \textit{pH}) \\ & + (0.1750 * \textit{Reaction Time}) \\ & - (0.000573 * \textit{Peroxide Concentration} * \textit{pH}) \\ & - (0.000010 * \textit{Peroxide Concentration} * \textit{Reaction Time}) \\ & - (0.0474 * \textit{pH} * \textit{Reaction Time}) \\ & + (0.000003 * \textit{Peroxide Concentration} * \textit{pH} * \textit{Reaction Time}) \end{aligned} \tag{3}$$

Table 4. Variance analysis results of COD removal efficiency

Source	**DF**	**Adj SS**	**Adj MS**	**F-Value**	**P-Value**
Model	7	559.93	79.99	7.48	0.005
Linear	3	466.18	155.39	14.54	0.001
Peroxide Concentration	1	451.56	451.56	42.25	0.000
pH	1	0.563	0.563	0.05	0.824
Reaction Time	1	14.06	14.06	1.32	0.284
2-Way Interactions	3	79.68	26.56	2.49	0.135
Peroxide Concentration*pH	1	39.06	39.06	3.65	0.092
Peroxide Concentration*Reaction Time	1	7.56	7.56	0.71	0.425
pH*Reaction Time	1	33.06	33.06	3.09	0.117
3-Way Interactions	1	14.06	14.06	1.32	0.284
Peroxide Concentration*pH*Reaction Time	1	14.06	14.06	1.32	0.284
Error	8	85.50	10.68		
Total	15	645.43			
S	R-sq	R-sq(adj)	R-sq(pred)	S	R-sq
3,577.89	59.67%	57.88%	54.11%	3,577.89	59.67%

The purpose of Pareto analysis is to identify and prioritize the most important or frequent problems or causes of problems in a particular process or system (Veli *et al*., 2018). In this study, Pareto analysis was used to determine which of the components affecting the Fenton process on COD removal. The results of Pareto analyze was also shown in Figure 3. Pareto analysis provides information on the validity of independent parameters. When comparing the results of the equation and the Pareto chart, it was found that the peroxide concentration was the most important parameter among the independent variables.

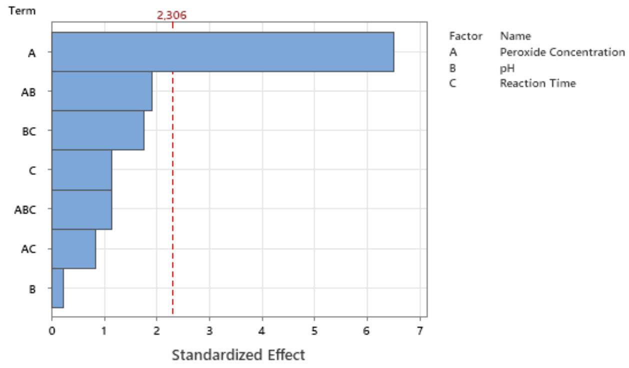


Figure 3. Pareto analysis of COD removal (A: 400 mM H2O2 – pH 2 – 60 min)

The peroxide concentration was found to be effective for COD removal, while the pH and reaction time were found to be less effective for COD removal. Based on these optimized values, 88% of COD can be removed. In the experiment performed under the optimal operating conditions obtained, Fe (II) dosage (80 mM), H_2O_2 dosage (400 mM), initial pH (2) and reaction time (180 min) were obtained to be 88% COD. These values are similar compared to literature data and according to literature studies, COD removal efficiencies of 90%-98%, 70%-85% and 60%-80% were achieved (Ilhan *et al*., 2019).

3.3. Full factorial design analysis of COD removal

Statistical analysis of the full factorial design was carried out with Minitab. The double interaction and triple interaction were not statistically significant (P- values > 0.05), the main effect was statistically significant (P- value 0.05). Only the peroxide concentration was statistically significant (P- value < 0.05) among the key impact components. The regression analysis's findings show that the variables utilized account for 54.11% of the system's variability. The increase in peroxide concentration and decrease in COD may be simply explained by the Eq. 3, according to the results of fitting the experimental data to the complete quadratic multiple regression model and then performing an ANOVA.

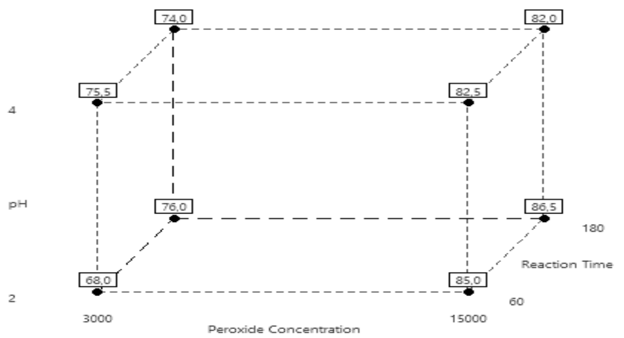


Figure 4. Cube Plot for COD removal

The trials were done twice, and Figure 4 displays the cube graph that was made based on the average test findings. Figure 4 illustrates how raising the pH from 2 to 4 enhanced the effectiveness of COD removal from 68% to 75.5%. The elimination of COD significantly increases from 68% to 85% with an increase in peroxide content from 80 mM to 400 mM. A relatively small effect on COD elimination from 85% to 86.5% was seen when the response time was increased from 60 to 180 minutes.

3.4. Variance analysis (ANOVA) of factors

ANOVA analysis was performed using Minitab statistical software. Peroxide concentration, pH, and reaction time form the three variables in the study. In terms of statistics, the confidence interval for each factor was set at 95%. Given that the P value is < 0.05, it is statistically significant. The three components were found to have an R^2 of 59.67%. The Tukey Method was used to group the data, and the 95% confidence interval was used to interpret the results. Table 5 displays the factor clustering and mean values. The Tukey technique emphasizes the recurrence of the letters in the grouping component when determining the significance of the elements (Ramasamy and Nagan, 2022). Group A contains A (Peroxide concentrations), and group B contains B (pH) and C (Reaction time). Differences between means that share a letter are not statistically significant. Peroxide concentration did not share the same letter as pH and reaction time. This indicates that the peroxide concentration had a significantly higher average than pH and reaction time. The difference between factor A and factor C groups is substantial since they are distinct from one another. The difference between the factor A and factor B groups is substantial since they differ as well. The difference between component B and factor C is not substantial, though, because they are in the same groupings.

Table 5. Tukey simultaneous comparisons

Factors	N	Mean	Grouping	
A: Peroxide concentrations	16	9,000	A	
C: Reaction time	16	120		B
B: pH	16	3,000		B

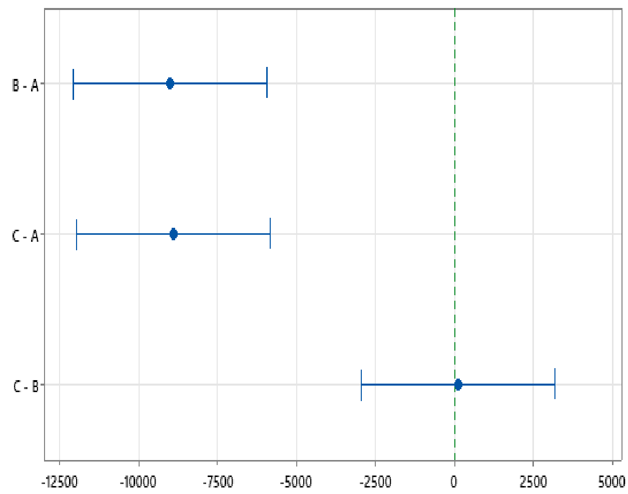


Figure 5. Tukey simultaneous 95 % of process factors

Figure 5 displays the Tukey Simultaneous (95% CI) of process factors. After the ANOVA, post hoc tests using Tukey's HSD test for significant factor were conducted to see whether there was a significant difference between the obtained findings among factors and the response. At $p < 0.05$, the difference was deemed significant (Al-Falahi *et al*., 2021). The C-B factor can ever reach zero. As a result, there is no statistically significant difference between the groups' means. At no time do factors B-A and C-A go close to zero. As a result, the means between groups show a statistically significant difference.

Figure 6 displays an interval plot of variables vs. factors (95% Cl). In these findings, the peroxide concentration, which is the A factor, has the greatest average while pH, which is the B factor, has the lowest average. This graph cannot tell us whether any change is statistically significant. Contrary to previous diagrams, this one emphasizes the significance of peroxide concentration.

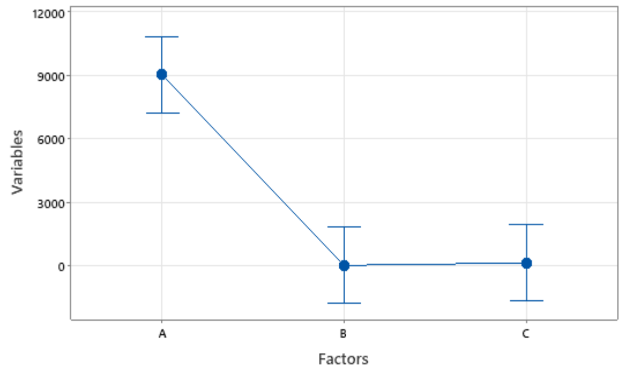


Figure 6. Interval plot of variables vs factors

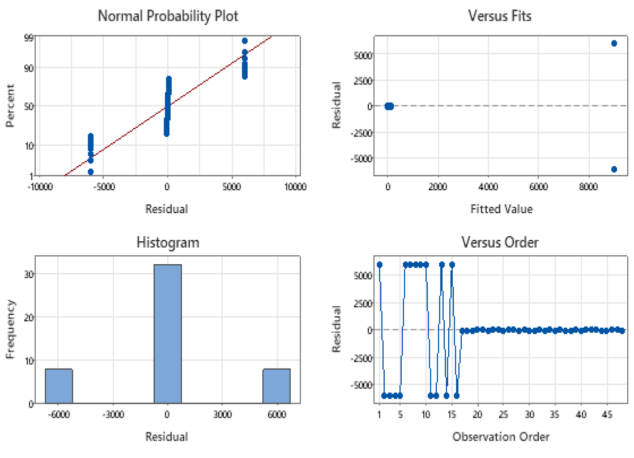


Figure 7. Residual plots for variables

Figure 7 graphs from the Minitab statistics program demonstrate the viability of the regression model assumption of zero mean error. The assumption of normality for the error component is tested using the normal probability plot (top left). The data points are grouped together along a straight line in the figure, which suggests that the error term is roughly typical. The normalcy assumption is therefore confirmed. The error terms are plotted against the fitted values in the Versus Fits graphic (top right). The top and bottom data points appear to be uniformly spaced, confirming the notion that the model has no mean error. The histogram's (bottom left) depiction of a bell-shaped curve further supports the notion of normalcy. Finally, the residuals move erratically about the zero line as shown in the versus sequence plot (bottom right). Typically, this behavior denotes the absence of serial correlation. Because there is no discernible pattern in the plots' random variability, the model appears to fulfill and satisfy the regression assumptions.

4. Conclusion

In this study, the treatability of net washing wastewater by Fenton oxidation was investigated. The optimum operational conditions for Fenton process, which resulted in a total COD removal efficiency of 88%, were identified to as follows: an equilibrium time of 180 min, pH is 2, the ratio of Fe (II)/H_2O_2: 1/5. Fenton oxidation has shown promising potential for treatment of the net washing wastewater.

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