



## Investigating the effect of coagulants and nano-coagulants to reduce biological oxygen demands in industrial wastewater

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### Abstract

One of the environmental consequences of industrial estates is the creation of environmental pollution, including the production of industrial wastewater. In this research, the main objective is to investigate the effect of coagulants and nano-coagulants in different concentrations to reduce the BOD of industrial wastewater using the Taguchi statistical method. Two chemicals, ferric chloride and aluminum sulphate, and two nanoparticles of iron oxide and alumina, were used as coagulants in the wastewater flocculation process at Beshel industrial complex. A L16 orthogonal array with the four factors of control at four levels each and two interactions was employed for optimization of coagulants and nano-coagulants concentration to reduce biological oxygen demands (BOD) in industrial wastewater. The optimal conditions were obtained concentration of ferric chloride 0.8 g/l, concentration of iron oxide nanoparticles 3 g/l, concentration of aluminum sulphate 1 g/l and concentration of alumina nanoparticles 1.5 g/l to achieve the highest percentage of BOD reduction.

Keywords: Nano-coagulants; Industrial wastewater; BOD

### 1. Introduction

We One of the most common by-products of industrial or commercial activities is industrial wastewater. Once this process water has been used, it is considered waste and needs to be treated before it is discharged. Industrial wastewater is not just a by-product of oil and gas or mining and chemical manufacturing companies, but also a by-product of food and beverage processing industries, essential in the making of the clothes on your back, the shoes on your feet, the computer at your fingertips, and the car your drive [1-2-3]. Industrial wastewater treatment includes a wide range of methods such as physical, physico-chemical and biological methods. The use of each of these methods depends on different factors, the most important of factors are the pollutants in the wastewater and the desired level of reduction of these pollutants. Various works have been done in the field of wastewater treatment using coagulants [4-5]. In addition, nanotechnology involves tailoring of

materials at the atomic level to achieve unique properties, which can be suitably manipulated for some desired applications. These nanoparticles can incredibly change the physical, chemical and biological properties of materials and used as nano-coagulants [6-7-8].

The removal of COD, TSS and colour of black liquor by coagulation–flocculation process was studied by Irfana et al. in 2017. They used from this process to find out the performance of various coagulants and flocculants like alum, ferric chloride, aluminium chloride, ferrous sulphate, poly aluminium chloride (PAC), cationic and anionic polyacrylamide polymers in individual form as well as in different combinations. They studied the effects of dosing rate, settling time and pH to reduce COD, TSS and colour. Their results showed that it reduced 76% COD, 95% TSS and 95% dye at pH <3 using the cationic and anionic polyacrylamide combination with ferric chloride and aluminum chloride [9]. In other work,

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coagulation and flocculation treatment of wastewater in textile industry using chitosan was investigated. Abu Hassan et al. used from aluminum sulfate (alum), ferrous sulfate, ferric chloride and ferric chloro-sulfate as coagulants. Their experiments were carried out on textile industry wastewater by varying chitosan dosage, pH and mixing time to study their effect in flocculation process using chitosan. The results showed the reduce of COD and turbidity in textile industry wastewater. Finally, they found the chitosan is an effective coagulant, which can reduce the level of COD and turbidity in textile industry wastewater [10]. In 2019, the efficiency of the physicochemical treatment process for removal of hazardous refractory wastewater from carpet industry was investigated by Nasr et al. Their results showed that ferric chloride (1.2 g/L), flocculent (2 mg/L) at pH 8 achieved COD removal efficiency of 97% whereas using ferrous sulfate (1.8 g/L), flocculent (2 mg/L) at pH 7 achieved 98% COD removal [11].

In this work, the main objective is to investigate the effect of coagulants and nano-coagulants in different concentrations to reduce the BOD of industrial wastewater using the Taguchi statistical method. Two chemicals, ferric chloride and aluminum sulphate, and two nanoparticles of iron oxide and alumina, were used as coagulants in the wastewater flocculation process at Beshel industrial complex.

## 2. Experimental

**Materials:** In this research, iron oxide nanoparticles (20-30 nm) and alumina nanoparticles (20-30 nm) were purchased from Nanotech Company (India). Ferric chloride and aluminum sulphate were also purchased from Merk Company (Germany).

**Design of Experiments (DOE):** A L16 orthogonal array with the four factors of control at four levels each and two interactions was employed for coagulation process by adding iron oxide nanoparticles, alumina nanoparticles, ferric chloride and aluminum sulphate in various concentrations. The analysis of laboratory results using Taguchi method and signal-to-noise analysis finally led to the determination of the optimal conditions in terms of the mentioned factors. An orthogonal array was designed for experiments using Qualitek-4 software considering the four variables of iron oxide nanoparticles concentration, alumina nanoparticles concentration, ferric chloride concentration and aluminum sulphate concentration each at 4 different levels (Tables 1 and 2).

**Coagulation and flocculation process:** Experiments were performed according to the

orthogonal array that was designed to prepare the nanocomposite coating by considering 4 variables including iron oxide nanoparticles concentration, alumina nanoparticles concentration, ferric chloride concentration and aluminum sulphate concentration at 4 different levels (Table 2). It was prepared 16 containers containing 100 ml of wastewater. During stirring, ferric chloride, iron oxide nanoparticles, aluminum sulphate and alumina nanoparticles were added. The solution inside the 16 containers was first passed through filter paper separately and then the filtered solutions were used to measure the biological oxygen demand (BOD).

**BOD measurement:** In this study, the samples of wastewater were measured by BOD meter (Aqualytic Germany). In fact, the supernatant of solutions was used to measure the biological oxygen demand (BOD) by BOD meter after adding four materials of ferric chloride, iron oxide nanoparticles, aluminum sulphate and alumina nanoparticles to 16 wastewater containers according to the DOE.

## 3. Results and discussions

The biochemical oxygen demand (BOD) was measured in the wastewater from the Beshel industrial complex equal to 384 mg/l. In addition, the BOD testing was performed for 16 experimental samples in two replications and the percentage of BOD reduction was shown in Table 3. The output variable used in statistical analysis was the percentage of BOD reduction, which is a measurable physical quantity. The analysis considered in this research was signal-to-noise analysis using S/N ratio. The term "signal" represents the desirable control factors during the process and "noise" represents the undesirable effect. The S/N ratio can be calculated by the following equation [12]:

$$(S/N) = -10 \log(\text{MSD}) \quad (1)$$

Where MSD is the mean squared deviation, which was obtained using following equation [12]:

$$\text{MSD} = \frac{\sum_{i=1}^n (y_i)^2}{n} \quad (2)$$

Where n is the total number of repetitions of the experiment and  $y_i$  is the result of each experiment. Note that there are three main categories of the quality characteristics, they are: 1. smaller is better, 2. nominal is better and 3. bigger is better. To obtain high percentage of BOD reduction, the 'bigger is better' quality characteristic was chosen. The average of S/N ratio for different levels of each factor to reduce the BOD including ferric chloride concentration, iron

oxide nanoparticles concentration, aluminum sulphate concentration, and alumina nanoparticles concentration were presented in Figures (1) to (4).

Figure 1 showed the effect of ferric chloride concentration in flocculation process at four levels to reduce the BOD. As seen in Figure 1, the highest percentage of reduction of BOD was obtained at level 2 from the concentration of ferric chloride (0.8 gr/l). As it is known, the S/N ratio increases with increasing the concentration of ferric chloride from 0.4 to 0.8 g/l, about 48%, which indicates a further decrease in BOD

at this concentration (0.8 g/l). But, the S/N ratio decreases with increasing the concentration of ferric chloride to levels 3 and 4 (1.5 g/l and 3 g/l), which shows the percentage of BOD reduction decreases at concentrations higher than 0.8 g/l. The coagulation–flocculation mechanism is proposed based on zeta potential measurement as the criteria to define the electrostatic interaction between pollutants and coagulant–flocculant agents.

Table 1 : Factors and their levels: the study of iron oxide nanoparticles concentration, alumina nanoparticles concentration, ferric chloride concentration and aluminum sulphate concentration in coagulation process

Factors	Level 1	Level 2	Level 3	Level 4
ferric chloride concentration (g/l)	0.4	0.8	1.5	3
Iron oxide nanoparticles concentration (g/l)	0.4	0.8	1.5	3
Aluminum sulphate concentration (g/l)	0.6	1	1.5	2
Alumina nanoparticles concentration (g/l)	0.6	1	1.5	2

Table 2 : Levels of each factor in experiments to optimize industrial wastewater treatment process by coagulation method

Experiment number	Ferric chloride concentration	Iron oxide nanoparticles concentration	Aluminum sulphate concentration	Alumina nanoparticles concentration
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	1	4	4	4
5	2	1	2	3
6	2	2	1	4
7	2	3	4	1
8	2	4	3	2
9	3	1	3	4
10	3	2	4	3
11	3	3	1	2
12	3	4	2	1
13	4	1	4	2
14	4	2	3	1
15	4	3	2	4
16	4	4	1	3

Table 3 : Levels of each factor in experiments to optimize industrial wastewater treatment process by coagulation method

Experiment number	BOD (iteration1)	BOD (iteration2)	BOD reduction%
1	349.66	349.65	8.94
2	345.41	345.40	10.05
3	354.03	354.04	7.80
4	348.09	348.10	9.35
5	227.63	227.63	40.72
6	301.82	301.81	21.40
7	312.18	312.19	18.70
8	293.08	293.07	23.68
9	354.11	354.10	7.78
10	295.62	295.63	23.01
11	307.92	307.92	19.81
12	315.69	315.68	17.79
13	328.73	328.73	14.39
14	335.87	335.88	12.53
15	304.33	304.34	20.75
16	322.74	322.73	15.95

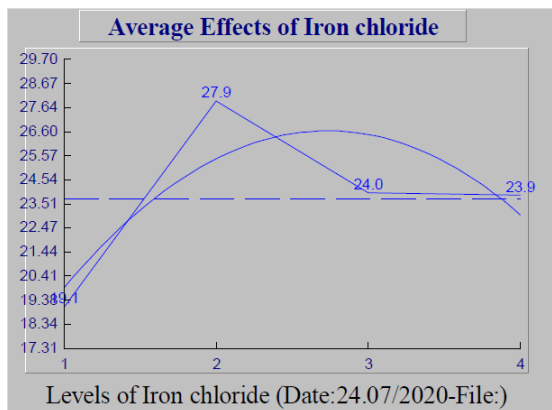


Fig. 1. Average of S/N ratio for different levels of ferric chloride concentration to reduce the BOD

As seen in Figure 2, iron oxide nanoparticles concentration changes have very little effect on the BOD reduction in wastewater. The difference between the lowest the percentage of BOD reduction and the highest the percentage of BOD reduction in the wastewater was about 3.75% for the iron oxide nanoparticles concentration factor, which is a very small amount and indicates the very low effect of this factor in flocculation process. It seems that iron oxide nanoparticles acted more as an adsorbent rather than as a coagulant and saturated at a concentration of 0.8 g/l, and at higher concentrations, there was little change in BOD reduction.

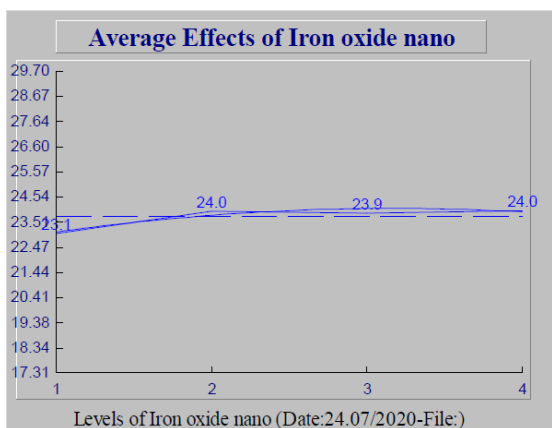


Fig. 2. Average of S/N ratio for different levels of iron oxide nanoparticles concentration to reduce the BOD

Figure 3 showed the percentage of BOD reduction in the wastewater was obtained at level 2 from the concentration of aluminum sulphate (1 g/l). In the flocculation process, aluminum sulphate acts similarly to ferric chloride. After adding aluminum sulphate (or ferric chloride) to wastewater, the aluminum (or ferric) reacted with the bicarbonates or hydroxides in the wastewater to form aluminum (or ferric) hydroxide or aluminum (or ferric) bicarbonate as a precipitate.

As shown in Figure 4, the S/N ratio for alumina nanoparticle concentration was 22.9 at the level 1, 24.2 at the level 2, 25.3 at the level 3, and 22.5 at the level 4. Therefore, the best performance occurred at the level 3 of this factor (concentration of 1.5 g/l) to reduce the BOD in wastewater.

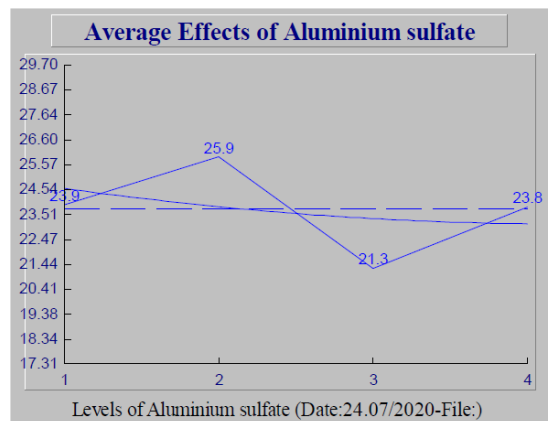


Fig. 3. Average of S/N ratio for different levels of aluminum sulphate concentration to reduce the BOD

The optimal conditions for achieving the highest efficiency of BOD reduction are presented in Table 4. The optimum concentrations of ferric chloride, iron oxide nanoparticles, aluminum sulphate, and alumina nanoparticles obtained 0.8 g/l, 3 g/l, 1 g/l, and 1.5 g/l, respectively. As illustrated, the optimal conditions did not occur in any of the 16 experiments. The BOD was measured equal to 42.6% under optimal conditions by BOD meter. This table also shows that the most effective factor in reducing the BOD was the ferric chloride concentration (51%). After ferric chloride concentration, the contribution of aluminum sulphate concentration (26.3%), alumina nanoparticle concentration (19.5%) and Iron oxide nanoparticles concentration (3.2%) have the greatest effect on reducing the BOD in the wastewater, respectively.

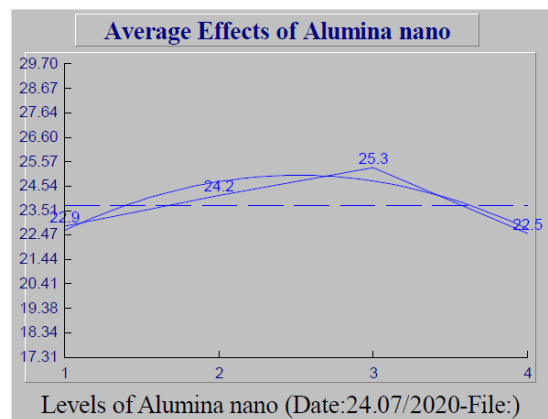


Fig. 4. Average of S/N ratio for different levels of alumina nanoparticles concentration to reduce the TSS

Table 4. Optimal levels and the contribution of factors in reducing the BOD

	Factors	Level Desc,	Level	Contribution
1	Ferric chloride concentration	0.8	2	4.208
2	Iron oxide nanoparticles concentration	3	4	0.267
3	Aluminum sulphate concentration	1	2	2.172
4	Alumina nanoparticles concentration	1.5	3	1.609

#### 4. Conclusions

In this work, it was studied the effect of coagulants and nano-coagulants in different concentrations to reduce the BOD of industrial wastewater using the Taguchi statistical method. The analysis of variance and signal-to-noise ratio showed the optimum concentrations of ferric chloride, iron oxide nanoparticles, aluminum sulphate, and alumina nanoparticles was 0.8 g/l, 3 g/l, 1 g/l, and 1.5 g/l to reduce the BOD in wastewater, respectively. In addition, the most effective factor to reduce the BOD in industrial wastewater was the ferric chloride concentration equal to 51%.

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