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Turbidity using Magnetic Flocculation

A B S T R A C T

The use of conventional flocculants such as Aluminum sulphate (Alum) alone to treat the wastewater may be insufficient to get the required turbidity, suspended solids removal. It also requires relatively a long residence time. Magnetic flocculation is one of the techniques used for increasing the efficiency of the turbidity removal. In the present study, three sets of experiments are carried out in order to investigate the possibility of increasing the suspended solid removal efficiency from Al Doura oil refinery wastewater using iron oxide (Fe3O4), Nickel (Ni), and Cobalt (Co) ferromagnetic powders with alum. The following operating conditions namely, pH, alum dose, ferromagnetic powder dose, and initial turbidity are studied. The results revealed that an improvement in turbidity removal efficiency is satisfied, as well as, a reasonable reduction in the sedimentation period is achieved. The highest turbidity removal is 99.88% that obtained for 122NTU sample for alum dose 120 mg/L+ Nickel dose of 80mg/L and pH of 6.5.

1. INTRODUCTION

Earlier studies show that an average of 468 gallons of water were required to refine one barrel of crude oil [1]. However, recent studies show that in USA one barrel of crude oil requires 42–79.8 gallons of water to be refined, with a median of 63 gallons of water [2]. Taking into account that 18.9 million barrels per day of crude oil is refined in USA at 2013 [3], water reuse within an industrial plant is essential [4]. Wastewaters of the oil refineries contain a large quantities of solids, salts, crude oil, aromatic and cyclic hydrocarbons, surfactants, phenols, naphthalene acids, sulfides, heavy metals, and other chemical products. In primary purification of water and industrial wastewater treatment, a widely used process is coagulation–flocculation. This process is preferable in primary treatment due to its simplicity, high efficiency and

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cost-effectiveness [5]. However, this process exhibits several disadvantages, such as the need for high amounts of chemicals for neutralizing the charges of the suspended particles, the need for pH adjustment before and after treatment, the sensitivity to temperature change, in addition to the excessive sludge production [6]. Various improvements are introduced to the coagulation–flocculation process, such as using natural or synthetic polymers as a flocculant aids to strengthen flocs, employing another technology of separation with the coagulation–flocculation process, like magnetic flocculation. Its principle is adding particles of a higher magnetic susceptibility into a conventional coagulation–flocculation process to enhance the flocculation velocity and form flocs of high density to settle quickly [7]. It combines a traditional flocculation and a magnetic separation in one process exhibiting a quick, simple, energy–efficient, and cost-effective advantages [8].

Miura et al. [9] applied a ferromagnetic powder with aluminum sulfate or polyaluminum chloride in order to remove solids from the wastewater. They got a removal efficiency of 99%. It was noticed that the required time for separating flocculated suspended solids was only few seconds, while in conventional treatment, it takes about one hour. Slusarczuk and Brooks [10] added a magnetic ferric powder and polyethylene imine as a flocculant agent to treat the turbidity. It is found that ferrite powder exhibited synergism with the aqueous polyethylene imine solution. The results revealed that the sludge volume is about 80% less than the volume produced by using polyethyleneimine alone. The suspended solids removal efficiency is raised from 30% to 71% when 1000 ppm of ferrite powder and 10 ppm of polyethyleneimine are added. Kang et al. [11] used a magnetic ferric powder of about 5 μm average particle size after dealing with it with a solution of white alum (KAl(SO₄)₂), polyaluminum chloride or ferric chloride. Ferrite powder was added at stirring speed of (200 - 300 rpm) for (1.0 - 2.0 minutes). It was found that the flocculated particles settled rapidly at a rate of 5 cm/minute, whereas in conventional methods that use alum or polyaluminum chloride, a period of 2.0-4.0 hours is required for efficient settling. Magnetic seeding aggregation (MSA) of silica nanoparticles was studied by Ref. [12]. Influences of pH, salts addition, and type of magnetite seeding particles on the turbidity removal efficiency were examined. The turbidity of CMP treated wastewater is reduced from 110.0 NTU to 7.0 NTU at pH of 6. The results showed that the residual turbidity decreases with the increase of magnetic field intensity. When the magnetic field intensity is higher than 0.08 Tesla, the residual turbidity is about 1.0 NTU. High turbidity reduction during the storm period by magnetic aggregation and separation was obtained by Ref. [13]. High turbidity raw water was prepared by mixing a sludge sample that are taken from Shiemen reservoir’s tail water pond with deionized water. It was found that at magnetic field strength of 0.1 Tesla, the magnetic aggregation effects were not significant but at magnetic field strength of 0.15 Tesla, significant effects on the magnetic aggregation were observed. When the magnetic field strength raised to 0.2 Tesla, the effect on magnetic aggregation was stable. The results also showed that with increasing the magnetite dosage from 2880 mg/L to 3360 mg/L, the final turbidity is reduced from 130 NTU to 20 NTU, while raising the magnetite dosage from 3360 mg/L to 4800 mg/L, the final turbidity is decreased from 20 NTU to 18 NTU. It was found that the turbidity removal efficiency at pH of 8.0 was superior than that at pH of 6. Akbar et al. [14] proved that turbidity removal is affected by pH, coagulant dosage, as well as initial turbidity. They found that the highest turbidity removal fall within 82-99.4% for initial turbidity of 10-1000NTU at pH of 5-7 and coagulant dose of 10-20mg/L. Ching and Zhen [15] conducted a study on magnetic seeding aggregation of high turbid source water as a pretreatment process using magnetite nanoparticles. The effect of pH on turbidity removal efficiency was studied over pH range of 5.0-9.0 and magnetic field strength of 0.0 Tesla to 0.1 Tesla. It was found that the final turbidity is decreased with the increase of the magnetite dosages. They got a turbidity of 774, 240, 56,19 and 10 NTU when using 1.0, 3.0, 5.0, 7.0 and 9.0 g/L magnetic dose. Their results showed that at pH values of 5.0, 6.0, 7.0, and 8.0 give the residual turbidities of 80, 234, 36 and 128 NTU, respectively. Mann [16] treated North Saskatchewan River water with different concentration of combination magnetite nanoparticles, aluminum sulfate and polycrylamide. Turbidity test reported that, 300 mg/L magnetite nanoparticles has the highest removal efficiency of 98%. It was found that the required time for removing the turbidity using magnetite was 10 minutes, while by using aluminum sulfate and polycrylamide combination, it was 30 minutes. Basma and Hussein [17] found that turbidity removal depends mainly on the coagulant dose, pH, and settling time. They found that the turbidity could be reduced from 92 to 2.1NTU at pH of 6, coagulant dose of 80 mg/L, and 120 minutes settling time. The feasibility of turbidity removal using a high gradient superconducting magnetic separation was studied by Ref. [18]. The process variables are, polyaluminum chloride (PAC) and magnetic seeds dosages. The initial turbidity of wastewater was 110 NTU, and the applied magnetic field intensity was 5.0 Tesla. A study regarding the use of a flocculated magnetic separation technology for treating Iraqi oilfield co-produced water for injection purpose was accomplished by Al-Rubaie et al. [19]. Results revealed that effluent water with low suspended solids and oil content can be obtained by applying a flocculation magnetic separation. It was also found that the required time for settling, several times less than that of the conventional methods. Treating of the emulsified oil wastewaters using a modified Fe₃O₄ magnetic nanoparticles MNPs, was made by Ref. [20]. A chitosan grafted magnetic nanoparticles Fe₃O₄ @APFS MNPs was used. They found a good demulsification effect via electrostatic attraction. It was also found that the demulsification performance could be further more enhanced upon Chitosan grafting especially under alkaline condition.

In the present study, an investigation on applying magnetic flocculation to treat wastewater of Al-Doura oil refinery using iron oxide, Nickel, and Cobalt magnetic powders with alum is made. The main objectives of this study are: Increasing the removal efficiency of the suspended solids and reducing settling time and consequently treating large quantities of polluted water without a need for enlarging the treatment basin.


2. EXPERIMENTAL PROGRAM

2.1. Apparatus and Procedures

2.1.1. Jar test

The Jar test apparatus was used in this study, is pharma test PT-DT7, it was taken from Samarra’a Company for drug and medical implementations (SDI).

2.1.2. Turbidity meter

The turbidity of water samples were detected by HANNA turbidity meter.

2.1.3. pH-meter

The pH-meter that used in the present study is Jenway 3310.

2.1.4. Electrical Balance

Precisa XB 220A electrical balance. pH and turbidity were measured three times for each sample and the average values were registered.

2.2. Experimental Procedure

The experimental procedures are listed below:
1. Beakers of 1000 ml are filled with 500 ml of wastewater after measuring its initial turbidity and adjusting the pH to the required value using 1.0 N HCl or 1.0 N NaOH.
2. The required magnetic powder dose was mixed with the wastewater at mixing speed of 250 rpm for 1.0 minute.

3. RESULTS AND DISCUSSION

3.1. Results of First Set

These results are listed in Tables 2-4 and samples of these results are shown graphically.

3.1.1. Effect of pH

Figures 1 and 2 represent the effect of pH on turbidity removal efficiency using alum alone and alum with 160 mg/L of iron oxide respectively. These Figures show that low turbidity removal efficiencies are obtained at pH=5.5, while a high turbidity removal efficiencies are gained at pH=6.5 and pH=7.5. These pH values which give the highest turbidity removal are within the range of operating conditions of the wastewater treatment plant for alum precipitation which is from 5.0 to 7.0 with minimum solubility occurring at pH equal to 6.0 [4]. Similar trend was obtained by [14, 17, 21]. Lo et al (2007) reported that the surface of the magnetite particles is positively charged at pH=6.0. Hence, at Fe₃O₄ doses equal to 160 mg/L and 200 mg/L the net charge of the wastewater will be positive so, a steric repulsion in the solution is occur, so high residual turbidity will remain, but at Fe₃O₄...
dose =240 mg/L the weighting effect predominates and overcomes the electrostatic repulsion forces. For aluminum-based coagulants, the best coagulation performance is generally observed at pH values that are as close as possible to the pH of minimum solubility of the coagulant [22].

The optimum pH value depends on the treated water properties, coagulant type, and coagulant concentration [23]. Similar trend was obtained for all magnetic powder, as it is clear from Tables 2 - 4 which indicate that the higher removal efficiency for all magnetic powders was obtained at pH 6.5 and 7.5. It is also clear that 200 mg/L of Nickel with 60 mg/L alum at pH of 7.5 gave the highest removal of 98.45% while the highest removal for 240 mg/L iron oxide (97.89%) was obtained at pH of 6.5 and 80mg/L alum and the highest removal for 240 mg/L of Cobalt was (97.22%) obtained at pH of 6.5 and 100 mg/L alum.

![Fig. 1](image1.png)

**Fig. 1.** Effect of pH on turbidity removal efficiency at different alum dose.

![Fig. 2](image2.png)

**Fig. 2.** Effect of pH on turbidity removal efficiency at different alum dose, Fe3O4 dose 160 mg/L.

### 3.1.2. Effect of Alum Dose

Figure 3 describes the influence of alum dose on turbidity removal efficiency at different pH values by applying alum only. It is clear that the removal efficiency increases with the increase of alum dose up to a certain limit then it drops. These results are in well agreement with that of [17, 24] who reported that colloidal particles are negatively charged and upon addition of aluminum sulfate, Al\(^{3+}\) ions are attracted to these particles. At the point of a complete charges neutralization, the colloids begin to agglomerate due to collisions between particles. If excess coagulant is added to the wastewater, the results are a reverse of the net charge on the colloidal particles (from negative to positive).

Particle re-stabilization by a reversal charge allowed greater amounts of smaller particles to remain in solution, thus increasing the total solids. Excess alum dose may exceed the saturation limit or produce excess aluminum hydroxide and thus will be a source of turbidity so the removal efficiency will be decreased [4]. The highest removal (96.12%) is obtained at 100 mg/L alum dose at pH of 6.5. The best removal (77.58%) at pH of 5.5 is obtained for alum dose of 80 mg/L, while at pH of 7.5 and alum dose of 120 mg/L the highest removal is 95.08%. These results show that the relationship between pH and alum dose is proportional. This may attribute to the alkalinity of the treated water. Metal coagulants are acidic, therefore, coagulant addition consumes alkalinity. In the case of pH =5.5 low dose of alum is required to get good results, since a high dose of alum will consume all the available alkalinity, lowering the pH too low values for efficient treatment. When pH=7.5, high dose of alum is required to depress the pH (reduce the alkalinity) to a favorable values for coagulation. At pH=6.5, an optimum alum dose and a best removal efficiency are obtained. This value is within the operating region range for alum precipitation which is from 5.0-7.0 with minimum solubility occurring at pH equal to 6.0 [4].

![Fig. 3](image3.png)

**Fig. 3.** Effect of alum dose on turbidity removal efficiency at different pH.

Figure 4 describes the influence of alum dose on turbidity removal efficiency at different pH values with the presence of 240 mg/L of Nickel. Inspection of this Figure and Tables 2 - 4 indicate that the general trend is nearly constant and the effect of alum dose with the presence of magnetic powder is little. Turbidity removal efficiency is increased slightly with the increase of alum dose at pH of 5.5 while it decreased slightly with the increase of alum dose at pH of 6.5 and 7.5. Moreover, the highest turbidity removal for alum alone or alum with any of the three magnetic materials is obtained at pH of 6.5 and 7.5 which are close to each other and the lowest removal was obtained at pH of 5.5. As mentioned previously, at low pH higher alum dose is required to get good results, since a high dose
of alum will consume all the available alkalinity, lowering the pH to too low values for efficient treatment. When pH=7.5, high dose of alum is required to depress the pH (reduce the alkalinity) to a favorable values for coagulation [4]. However, at pH of 6.5 and 7.5, there is a slight decrease of removal efficiency with the increase of alum dose. This is for two reasons; the first is excess alum dose may exceeds the saturation limit or produce excess aluminum hydroxide and thus will be a source of turbidity so the removal efficiency will be decreased [4] and the second is the fact that high magnetic powder dosage does not mean better efficiency, it becomes a source of turbidity that is extremely difficult to be removed without externally applied magnetic field. While at low dosage of magnetic powder, the effectiveness of magnetic aggregation will be poor [13]. This Figure show that the main effect is for pH and magnetic powder. Akbar et al. (2010) stated that turbidity removal was relatively stable at all selected dosages greater than 10 mg/L when pH was kept constant, whereas turbidity removal seemed to be more influenced by pH variation than coagulant dosage. It is also clear that alum dose can be reduced from 140 and 100 mg/L when it is used alone at pH 7.5 and 6.5 respectively to as low as 60 mg/L when magnetic powder is added. This can reduce the excess cost of this process and satisfy one of the purposes of this work.

is crucial, high dosage does not mean better efficiency, it becomes a source of turbidity that is extremely difficult to be removed without externally applied magnetic field, in addition to high amounts of sludge formation. While at low dosage of magnetic powder, the effectiveness of magnetic aggregation will be poor [13]. The highest turbidity removal of 98.45% is obtained when using 200mg/L of Nickel with 60 mg/L alum at pH of 7.5.

3.1.3. **Effect of Magnetic Powder Dose**

Three levels of magnetic powder dose were used namely; 160, 200, and 240 mg/L. Figures 5–7 represent samples of the results for iron oxide, Nickel, and Cobalt that gives the highest removal efficiency respectively. A careful inspection of these figures and Tables 2–4 clarify that turbidity removal is increased with the increase of the magnetic powder dose up to a certain limit then it drops slightly. These results are in agreement with that of [25]. The lowest removal takes place at pH of 5.5 and the highest removal takes place at pH 6.5 or 7.5. Moreover, the removal at pH 6.5 and 7.5 are close together for all magnetic powder except Cobalt. Also, it could be found that the magnetic powder value that gives the highest removal depends on both pH and alum dose. The optimum performance for turbidity removal depends on pH, treated water properties, coagulant type, and coagulant concentration [23]. Appropriate magnetic powder dosage
3.2. Second Set Results

Since the best pH is 6.5 and 7.5 according to the results of the first set, thus it was decided to take the average value (7) to determine the best alum dose at this average value of pH. It was found that 120 mg/L of alum gives the highest removal efficiency (Fig. 8). This alum dose is used to find the effect of pH on removal efficiency and it is found that the pH range 6.5-7.5 gives the highest removal. However at pH of 6.5, the highest removal is obtained (97.87%) (Fig. 9). This result is in agreement with that of [26]. Then, in a trial to test the possibility of reducing the magnetic powder dose, it is decided to use a range of 40 to 120 mg/L for each of the three magnetic powders. The results were graphed on Fig. 10. It is clear that at low doses of magnetic powders the removal efficiencies are low and it increases with the magnetic powder dose increase. At low dosage of magnetic powder, the effectiveness of magnetic aggregation will be poor [13]. It could be seen that the best Fe$_3$O$_4$ magnetic powder dose is 120 mg/L, while for nickel and cobalt they are 80 mg/L and 100 mg/L respectively. The optimum performance depends on pH, treated water properties, coagulant type, and coagulant concentration [23]. It could be concluded that nickel magnetic powder exhibits an excellent performance, where its optimum dose is low in comparison with iron oxide and cobalt and it gives a removal efficiency reaches to 98.57%.

![Fig. 8. Effect of alum dose on turbidity removal, pH=7.](image)

![Fig. 9. Effect of pH on turbidity removal, Alum dose = 120 mg/L.](image)

![Fig. 10. Effect of magnetic powder dose on turbidity removal, alum dose = 120 mg/L & pH = 6.5.](image)

3.3. Results of the Third Set

After finding the best conditions of alum dose, pH, and magnetic powder doses, an evaluation for using different initial turbidities with the best findings are illustrated in Figure 11. It is clear that for alum alone, and alum with Fe$_3$O$_4$, the turbidity removal start to fall down at an initial turbidity of 61 and 90 NTU respectively, while Cobalt and Nickel still show good results at higher turbidities. This finding is in full agreement with that of [14] who stated that turbidity removal efficiency was decreased to a certain extent by increasing initial turbidity and application of higher coagulant dosage may improve turbidity removal from relatively high turbidity waters since high turbidity in addition to the dispersed Fe$_3$O$_4$ will need a lot of alum doses to neutralize their charges and overcome the mutual repulsion forces between suspended solids. They also found that there is an optimum magnetic dose for a specific initial turbidity range. However, when the raw water turbidity is altered, this optimal dosage will require an experimentally adjustment. They found that the optimum magnetic dose show a linear relationship with the initial turbidity. They stated that the coagulation process and turbidity removal was considerably effected by pH, coagulant dosage, as well as initial turbidity. Bahman (2014) reported that the increase in the turbidity removal with the increase of the initial turbidity might be attributed to other mechanisms such as sweeping flocculation rather than the neutralization of the surface charge of colloids.

For all experiment when using alum with magnetic powder, samples for the determination of removal efficiency are taken after 5 minutes while for alum only, samples are taken after 30 minutes settling. The removal efficiency when using alum (100 mg/L) and Fe$_3$O$_4$ (160 mg/L) at pH of 7.5 is 96.66% after 5 minutes settling while with employing alum only the turbidity removal efficiencies are 67.91% and 93.67% at settling periods of 5.0 minute and 30 minute respectively. This finding can give an increase of the treated volumes by 6 folds which is an essential matter for the field units.
Finally, from the present work results, it could be concluded that, there is no specific value for the independent variables that gives the best results. Therefore, these values should be determined for each case depending on the experimental laboratory results. It is also revealed that the nickel magnetic powder with alum give the best results by a comparison with Fe₃O₄ and cobalt magnetic powders.

4. CONCLUSIONS

The main conclusions of the present study could be summarized as follows:
1. An enhancing in the turbidity removal efficiency is achieved by utilizing magnetic flocculation technique.
2. The required period for settling is very short (five minutes) in comparison with conventional method.
3. There is a potential for applying the same operating conditions for various initial turbidities, but in reasonable limits.
4. It can be concluded that nickel magnetic powder has a superior performance in comparison with iron oxide and cobalt magnetic powders.
5. The maximum turbidity removal efficiency is 99.88% when applying magnetic flocculation technology, while with applying conventional flocculation the maximum turbidity removal efficiency is 92.89% at the same conditions.

Table 2
Experimental results of the first set for Iron Oxide.

| Turbidity Removal Efficiency (%) | pH  | 5.5 | 6.5 | 7.5 |
|----------------------------------|-----|-----|-----|-----|
| Iron Oxide Dose (mg/L)           |     |     |     |     |
| 0.0                              |     |     |     |     |
| 60                               |     |     |     |     |
| 80                               |     |     |     |     |
| 100                              |     |     |     |     |
| 120                              |     |     |     |     |
| 140                              |     |     |     |     |

Table 3
Experimental results of the first set for Nickel.

| Turbidity Removal Efficiency (%) | pH  | 5.5 | 6.5 | 7.5 |
|----------------------------------|-----|-----|-----|-----|
| Nickel Dose (mg/L)               |     |     |     |     |
| 0.0                              |     |     |     |     |
| 60                               |     |     |     |     |
| 80                               |     |     |     |     |
| 100                              |     |     |     |     |
| 120                              |     |     |     |     |
| 140                              |     |     |     |     |

Table 4
Experimental results of the first set for Cobalt.

| Turbidity Removal Efficiency (%) | pH  | 5.5 | 6.5 | 7.5 |
|----------------------------------|-----|-----|-----|-----|
| Cobalt Dose (mg/L)               |     |     |     |     |
| 0.0                              |     |     |     |     |
| 60                               |     |     |     |     |
| 80                               |     |     |     |     |
| 100                              |     |     |     |     |
| 120                              |     |     |     |     |
| 140                              |     |     |     |     |
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