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Investigation of ozone and coagulant material's (aluminum sulfate, ferric chloride, poly aluminum chloride and lime) efficiency in "Kerman Kork" industry wastewater treatment

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Abstract
Background: Entry of untreated wastewater of wool scouring factories has been associated with many environmental hazards because of high rate of pollution. Presently effective treatment methods and reducing the costs of operation and maintenance from treatment units have always been under the attention of this industry owners. The aim of this survey is to present a suitable method for the treatment of wool scouring.
Methods: In this study, chemical coagulant is used for wastewater treatment (aluminum sulfate, ferric chloride, poly aluminum chloride and lime). And then these materials with "ozone" as a strong oxidative in reduction of the amount of Chemical Oxygen Demand (COD) in wastewater effluent has been surveyed.
Results: The results of this work showed that only ferric chloride and aluminum sulfate among the above chemical coagulant had a high efficiency in the turbidity removal. Productivity of turbidity removal using ferric chloride with the pH of 4 to 5 was 99%, and turbidity was reduced from 166 Nephelometric Turbidity Units (NTU) to 1.5 NTU, and productivity of turbidity removal using aluminum sulfate within the pH 4.5 to 5 was 99.33% that reduced turbidity from 166 to 1.1 NTU. Results of ozonation with 5 g/hour and the oxygen flow of 2.5 litter per min with the pH assess of 15, 30, 45, 60, 90, 120, and 150 min to wastewater from chemical treatment with ferric chloride showed that the rate of COD was reduced from 24700 mg/L to 2940 mg/L. In ozonation to wastewater of chemical treatment with aluminum sulfate within the above rang of time, the rate of COD was reduced from 22500 mg/l to 4800 mg/l.
Conclusion: We can be hopeful that in near future, getting the cheap technology of ozone production in industrial scale, one can use this technology for the propose of removal of pollutants having removal preferable by help of advanced treatment approaches.
Keywords: Industrial wastewater, COD, Wool scouring, Chemical treatment, Ferric chloride, Aluminum sulfate, Ozonation, Turbidity
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Introduction
Wool scouring industry is a kind of industry wastewater which has high amount of pollution and always includes a lot of environmental dangers (1).
The main environmental impact comes from the very high oxygen demand, which is typically 45,000 mg/L Chemical Oxygen Demand (COD) (1). Seventy-five percent of the COD is due to emulsified wool wax, with the remaining portion due to water soluble compounds that are collectively known as suint (2).
The main difficulties in treating wool scour effluent are due to the combination of high oxygen demand, the slow biodegradability of the wool wax, the presence of high pollutant concentrations in both dissolved and insoluble form, and the difficulty in causing phase separation (3). This is continually becoming more difficult to treat due to improvements in scouring technology. The Biochemical Oxygen Demand (BOD) and COD of these effluents are also high. Owing to such high pollution load, treatment and disposal of these effluents are serious prob-

lems (1,4). Physico-chemical treatment allows to reduce dissolved, suspended, colloidal and non-settable matter as well as color from dyes. Depending on the wastewater characteristics, COD of a textile effluent can be reduced between by 50% and 70% after optimizing the operating conditions (pH, coagulant and flocculants concentrations) (4).

A modern wool scour consists of multiple hopper-shaped bowls arranged in series, which are operated as separate washing and rinsing sections (see Figure 1). The wash section commonly uses 1–5 litters of fresh water per kilogram of greasy wool processed, while the rinse section typically uses water volumes which can range from 6 liters per kilogram of wool washed to more than 15 litters per kilogram (5).

The wool is passed through squeeze presses after each bowl to prevent entrainment of waterborne contaminants from dirty bowls to cleaner ones, thus maintaining a counter current contacting of the wool and wash water. Squeeze pressing of the wool as it leaves the final rinse bowl and also minimizes the water loading in the wool was sent to the final dryer, thus significantly reduce the energy use of the overall process (Figure 2).

The key differences between a wash bowl and a rinse bowl are:

Wash bowl

✓ Contains detergent/surfactants

✓ Runs hot (50–60 °C, dictated by the type of detergent used)

✓ Is operated at a high suspended solids level (up to 8–12% solids in bowl 1)

✓ Relatively low water use.

Rinse Bowl

✓ Contains minimal or no added detergents

✓ Either hot or cold water is used

✓ The water in the bowl is kept cleaner (seldom above 1.2% solids in bowl 4)

✓ A larger volume of water is generally used

Wool scouring effluent is a very stable emulsion not easy to treat by biological or physico-chemical processes. COD/BOD ratio is near to 3 (6).

The main components of the wastewater are wool wax, suint and dirt, stabilized by detergents (6).

In Iran, the most commonly used detergent is German Neodal.

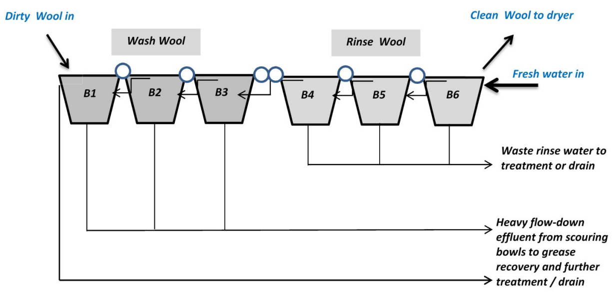

Figure 1. Conventional wool scour configuration

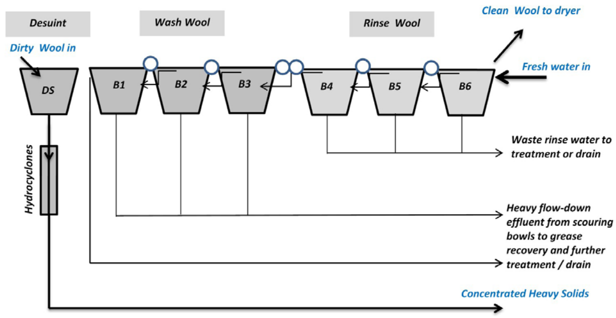

Figure 2. Use of an additional disunite bowl

The relative component contributed to COD is (7): suint 11%, total grease 71%, and dirt 18%.

Contaminant concentration in the wastewater varies with the type of wool and the scouring process employed. Average values are: BOD 5 to 40 g/L, COD 30 to 150 g/L, total grease 9 to 50 g/L, suspended solids 15 to 80 g/L, and pH 8 to 10 (8).

The main environmental impact comes from the very high oxygen demand, which is typically 45,000 mg/L chemical oxygen demand (1).

Effective scouring relies on: 1- Removing contaminants from the fiber into the scour liquor and 2- preventing the insoluble contaminants re-depositing back onto the fiber (1). Whilst scour effluent is difficult to degrade anaerobically (8).

Requiring typical retention times of 15–20 days (7,8), rapid anaerobic treatment of 4–10 days in a two-stage system achieved 60–86% COD removal by bio-flocculation of the wool wax (9).

Anaerobic treatment for 2–3 days followed by chemical flocculation was found to remove over 80% of wool wax, compared to the amount of only 30–50% by anaerobic treatment alone up to 8 days (10). Similarly for aerobic treatment, COD removal by chemical flocculation was enhanced by 15% after a rapid (3–5 h) aerobic treatment that removed less than 10% of pollutants itself (11).

Jar-tests allow the evaluation of a treatment to reduce dissolved, suspended, colloidal, and non settleable matter from wastewater by chemical coagulation-flocculation followed by gravity settling. Therefore, these tests are a valuable tool in wastewater treatment (4).

The major disadvantage of coagulation flocculation processes is the production of sludge (12).

Jar-tests were conducted in order to determine the optimum dosages of the used coagulants and flocculants. The results were evaluated using major ecological parameters, like COD and, NTU.

Ozone is the strongest practical oxidant available for water treatment processes. The effectiveness of ozone is resulted with two phenomena:

- Solubility of ozone in water (transfer gas-liquid)
- Interactions between organic matter and oxidant

Chemical oxidation by ozone, or a combination of UV radiation and ozone and H_2O_2, have great interest, but their

costs are still very high (4).
Ozone is a very powerful oxidant for water and wastewater treatment. Once dissolved in water, ozone reacts with a high number of organic compounds in two different ways: by direct oxidation, as molecular ozone or by indirect reaction, through the formation of secondary oxidants, such as free radicals, particularly hydroxyl radical. By means of organic matter ozonation, it is expected to achieve color, COD elimination, and an increase of the biodegradable organic carbon for later biological stages (3).
The first ozone generator was built 150 years ago (1860s) at the beginning of the third millennium in Berlin by Siemens Company. The world's biggest industrial ozonation system with the generation power of 42 kgo3/hr per an industrial unit of paper paste whitener is working at Finland.
The usage of ozone in the treatment of wool scouring wastewater is not common yet.
The costs of ozone production have become more suitable during the last 50 years. In accordance with some researchers, the key of development in this technology is more ozone production at the level of electrode that is related to the technology of average frequency production (13).
-Producing more concentration of ozone by modern generator, 6 to 14 oxygen weight percent.
-Increasing the ozone production potential per a unit with the integral multiple of 2 or 3.
-40% reduction at the usage of special energy.
-Increasing the work safety by considering the work costs and investing, ozonation is not yet a cheap technology for the treatment of wastewater (14).

Materials and Methods

In the first step, to do jar-test, coagulant material – Ferric chloride and Aluminum sulfate – were prepared and jar unit was also installed, and became ready for the operation.
During sampling the following parameters which are shown in table 1 were examined: initial turbidity was 166 NTU, sample temperature 45 °C, and pH 8.33 (Table 1).
After moving the sample to laboratory, initial COD of the sample was measured that was 34800 mg/L in this sample.
COD experiment was carried out in accordance with the open reflux method (16).

Table 1. Initial parameters measured during sampling

Parameter	Initial sample
Temperature	45 °C
Turbidity	166 NTU
pH	8.33
COD (mg/l)	38400
Q (m^3/30 days)	2500

COD= Chemical Oxygen Demand; NTU= Nephelometric Turbidity Units

Experiments

Jar test

- Jar test with ferric chloride $FeCl_3\_6H_2O$
-Aluminum sulfate $(Al_2(SO_4)_3\_18H_2O$

Since the determination of the most suitable pH and optimum amount of coagulation material required in water and wastewater plants is not measurable theoretically and stoichiometric, these experiments must be determined empirically.
Jar and pH tests were carried out according to "Standard methods for the examination of water and wastewater". These tests are usually done by a unit called Jar. Turbidity was measured during all the steps of experiment by Lovibound turbidity meter unit.
We solved 2, 4, 6, 8, and 10 g/L of ferric chloride coagulant with chemical formula $FeCl_3\_6H_2O$ in water until it becomes completely homogenous. 25, 50, 75, 100, 150, and 200^{CC} of the obtained homogenous solution were added to jar cells in the following experiments and the unit did the rapid mix operation with 120 round per minute for 2 minute.
Slow mix operation done with 20 rounds per minute for 20 minutes time was recorded continuously. Final precipitation time was 30 minutes. The optimum dosage for coagulant material of ferric choleric was 1.1 g/L in this experiment.
pH was measured by CRISON model pH meter at 25 ºC. The range of optimum pH for using ferric chloride in these tests was 4–4.5.
Jar test for determination of optimum dosage and pH of coagulant material of aluminum sulfate with chemical formula $(Al_2 (SO_4)_3\_18H_2O$ was carried out precisely as above method.
The optimum amount of aluminum sulfate was 2g/L and the optimum range of pH was 5–5.5 .
After moving the wastewater samples to laboratory, the intended variances were measured in raw sample.
Then the variances were re-measured and examined for chemical treatment and determination of optimum amount and pH coagulation material.

Ozonation experiments

Required ozone in this test is provided by an ozone generator unit with the generation power of 5 g/hr and oxygen flow of 2.5 L/min (Figure 3).
After chemical treatment of sample with coagulant materials ferric chloride $FeCl_3\_6H_2O$ and aluminum sulfate $(Al_2(SO_4)_3\_18H_2O$, the required sample was provided for performing the ozonation tests.
For the purpose of performing ozonation tests, the provided sample was poured in a cylinder gauge. The outlet pipe of provided ozone from ozone generator unit attached to air stone at the most bottom of the cylinder was floated.

Results

Figure 3. Ozone generator unit with generation power of 5 g/hr

In this study, the efficiency of coagulant materials ferric chloride $FeCl_3\_6H_2O$, aluminum sulfate $(Al_2(SO_4)_3\_18H_2O$, and poly aluminum chloride in removal turbidity and suspended COD of effluent of wool scoring industries was examined.

Ferric chloride and aluminum sulfate have a very high efficiency for high turbidity removal, but poly aluminum chloride is suitable for low turbidity removal. Turbidity removal efficiency of ferric chloride is >98% and of aluminum sulfate is >99%.

Initial COD of the sample was 38400 mg/L. After chemical treatment with ferric chloride, COD was reduced from 38400 mg/L to 24700 mg/L. COD removal efficiency of ferric chloride was 35.6% mg/L. Ferric chloride is in the form of a brownish liquid, and it is odorless. The chemical formula of ferric chloride is $FeCl_3\_6H_2O$.

Physical properties: Special gravity is about 1.43 g/cm^3, and its color is brown.

Chemical analysis: The rate of COD was reduced 22500 mg/L after chemical treatment with aluminum sulfate.

COD removal efficiency of Aluminum sulfate was obtained about 41.4%.

The rate of COD was reduced to 35400 mg/L after chemical treatment with poly-aluminum chloride. COD removal efficiency of with poly-aluminum chloride was about 7.8%, which is in comparison with the two previous materials, the efficiency of turbidity removal is very lower. Chemical formula of this material in deride form (without water) is $(Al_2(OH)_{6-x}(Cl_xYH_2O)_z$. Amount of Z is varied in the range of 12-18.

Poly-aluminum chloride is a mineral polymer. Its monomers are duel-core complexes of aluminum.

Three inorganic coagulants $FeCl_3\_6H_2O)$, $(Al_2(SO_4)_3\_18H_2O$, $FeSO_4\_7H_2O$, and commercial cationic flocculants, either alone or as a combination, were tested to purify the wool scouring effluents.

The results of determined COD after ozonation of the sample from chemical treatment with ferric chloride are shown in Figure 4. The results of determined COD after ozonation of the sample from chemical treatment with aluminum sulfate are shown in Figure 5.

Discussion

The findings showed that in this study the COD removal efficiency of the coagulants aluminum sulfate is higher than that of ferric chloride and poly-aluminum chloride, but both aluminum sulfate and ferric chloride with small differences are very suitable for turbidity, removal and COD.

In accordance with the statistical tests analysis and variation (ANOVA) for analysis of data related to jar tests for determining the best coagulant, it can be concluded that in the analysis table of four coagulants, aluminum sulfate is the most suitable material for performing the process of coagulation. This is because of its lower standard deviation and higher efficiency average than the other coagulants but there is not a significant statistical difference between ferric chloride and fluminum sulfate. According to the optimal rate of these two materials, which is 1 g/L for ferric chloride and 2 g/L for aluminum sulfate, ferric chloride was economically suggested for doing chemical treatment. In this statistical test, alfa was 0.05.

The optimal pH range to use ferric chloride was 4–4.5 and for aluminum sulfate was 5–5.5.

Working with ferric chloride it should be noticed that the slug from ferric chloride is very corrosive, and it should be used with special strategies. In ozonation of wool scouring wastewater, the removal efficiency was suitable at 120 min. At the present time, ozonation is not considered a cheap technology.

Looking at Figure 4 to 6, we can observe the process of COD reduction resulted from ozonation to wastewater of chemical treatment with coagulant like ferric chloride and aluminum sulfate. It can be seen from Figure 4 that the COD rate has reached from 24700 mg/L to 2940 mg/L

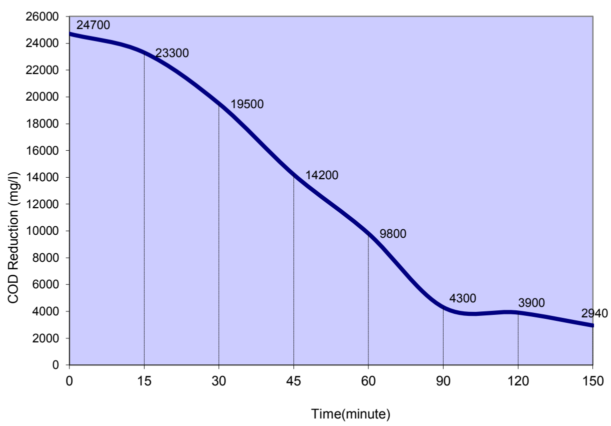

Figure 4. COD reduction after ozonation samples treated with ferric chloride.

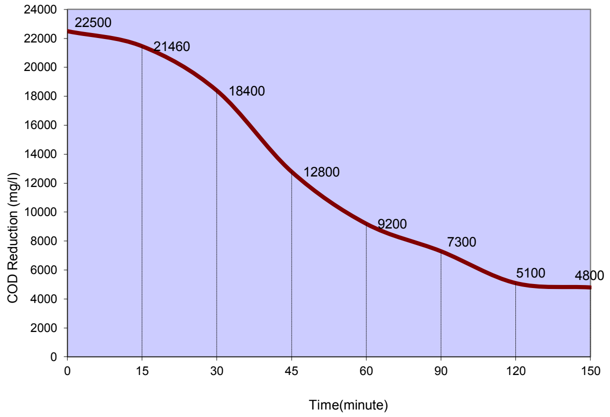

Figure 5. COD reduction after ozonation samples treated with aluminum sulfate

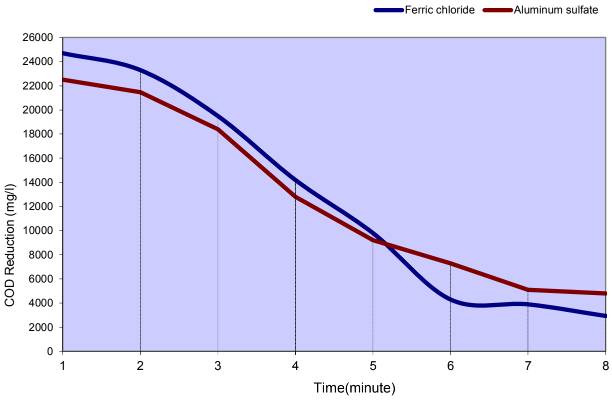

Figure 6. The merged diagram of COD reduction after ozonation.

which shows the 88% efficiency.

The most amount of removal takes place at the time 90 minutes. It can be observed in Figure 5 that the COD rate reached from 22500 mg/L to 4800 mg/L, and this shows the 78% efficiency. The most amount of removal takes place at the time 120 minutes.

In Figure 6 a mixing of Figures 4 and 5 can be seen reduction of suspended COD of wool scouring wastewater and turbidity removal resulted from TSS, one should preferably use ferric chloride with the chemical formula of $FeCl_3\_6H_2$ by 1 g/L because of cheap production in the country. It is more economical for this industry owner. But, on the other hand, the required approaches should be considered because of strong corrosion of the produced sludge.

In ozonation tests of wastewater from chemical treatment with ferric chloride, the rate of COD was reduced from 24700 mg/L to 2940 mg/L that showed 88% removal efficiency. In ozonation of wastewater from chemical treatment with Aluminum sulfate, the COD rate is also reduced from 22500 mg/L to 4800 mg/L that here the efficiency of COD reduction is equal to 78% which shows a very desirable process in removal of solute wool scouring wastewater COD.

One of the researchers has reported ozone doses in level of 2 mg/L to result in virtually complete removal of color and hard pollutants, such as detergents in textile wastewater effluent (19).

Conclusion

At the end, we can be hopeful that in near future, getting the cheap technology of ozone production in industrial scale, can use this technology for the propose of removal of pollutants having removal preferable by help of advanced treatment approaches.

Ethical issues

We certify that all data collected during the study is presented in this manuscript and no data from the study has been or will be published separately.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

AHJ, Mohammad Malakootian, Mohsen Mehdipour conceived and designed the study. Mohsen Mehdipour performed the literature search and wrote the manuscript. All authors participated in the data acquisition, analysis and interpretation. All authors critically reviewed, refined and approved the manuscript.

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