



## Treatment of natural rubber industry wastewater through a combination of physicochemical and ozonation processes

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### Original Article

#### Abstract

In any type of rubber product manufacturing (including tires), the primary concerns are environmental. The aim of the present study was to survey a treatment combination of ozonation and physicochemical processes in the rubber industry. Wastewater samples were collected from the discharge unit of the rubber processing sewage system in Kerman Barez Tire Factory, Kerman, Iran. The wastewater samples used for chemical oxygen demand (COD), biochemical oxygen demand (BOD), total suspended solids (TSS), and oil and grease determinations were collected directly into bottles. After collection, samples were transferred to the laboratory for examination. The 2 methods of physicochemical process and ozonation process were used to treat wastewater. The study results suggest that the use of a chemical coagulation process with ferric chloride ( $\text{FeCl}_3 \cdot 7\text{H}_2\text{O}$ ) in the first stage of this study reduced COD by 37% of the original amount (0.56 g/l). The optimum dosage and pH range were 0.775 g/l and 6.5, respectively. When using  $\text{Al}_2(\text{SO}_4)_3$ , the COD reduction rate was 42%, and the optimum dosage and pH range were, respectively, 0.45 g/l and 6.5-7. After the ozonation process, COD was reduced by 70.75% and 90.6%. In accordance with these results and with respect to the high contamination load of this industry's wastewater and its many environmental hazards, the complete treatment of this industry's wastewater is crucial. One scientific and practical approach to wastewater treatment is the use of a combination of processes.

**KEYWORDS:** Aluminum sulfate, Coagulation, Ferric Chloride, Ozonation, Physicochemical, Wastewater, Treatment

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#### Introduction

Industries are major sources of pollution in all environments.<sup>1</sup> Depending on the industry, different levels of pollutants are discharged into the environment directly or indirectly through the sewer outlet. In recent years, a rapid growth has been observed in industries due to the development of technology. Therefore, the volume of waste

produced by different industries has also increased. The rubber industry produces environmental pollutants, which are highly objectionable, from natural rubber processing. The high concentrations of nitrogen and organic and inorganic loading in rubber wastewater pose serious threats to the environment.<sup>2</sup> Industrial wastewater includes employees' sanitary waste, production process discharge, wash waters, and contaminated water from heating and cooling and other operations.<sup>3</sup> To produce 20

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tons of rubber, a rubber factory produces, on average, 410,000 liters of effluent per day.<sup>4</sup> Environmental pollution caused by the daily discharge of about 80 million liters of untreated rubber effluent into near streams and rivers has been reported in Malaysia.<sup>2,5,6</sup> Without appropriate treatment, the discharge of rubber industry wastewater into the environment may have serious, dangerous, and prolonged consequences. Therefore, suitable technologies must be used to treat this wastewater.<sup>7</sup> Various methods for treating this type of waste exist in the world, the most important of which are biological,<sup>8</sup> aerobic,<sup>9</sup> anaerobic,<sup>10</sup> and physicochemical methods, and facultative ponds. Advanced methods include natural process,<sup>11</sup> electrochemical methods,<sup>12</sup> ozonation process followed by batch activated sludge, and methods combining physicochemical and biological methods (e.g., the gas injection technique and sludge process).<sup>13</sup> None of these studies, however, used the combination of physicochemical and ozonation processes.<sup>8-13</sup> The current study investigated the treatment efficiency of the combination of physicochemical treatment and ozonation process for chemical oxygen demand (COD) removal from rubber industry effluents.

## Materials and Methods

In this study, a testing unit was conducted at a laboratory-scale in the Kerman Barez Tire Factory, Iran, to investigate the treatment efficiency of physicochemical and ozonation processes in a combined treatment method on rubber industry wastewater effluent.

The experimental stage was divided into 2 stages. In the first stage, experimental studies were conducted with a physicochemical treatment process in which effluent was treated through coagulation followed by flocculation using aluminum sulfate [alum,  $\text{Al}_2(\text{SO}_4)_3$ ], and ferric chloride ( $\text{FeCl}_3$ ). Different variables, including contact time, coagulant material dosage, and pH, were tested. In the second stage, ozonation was performed in the batch reactor as post

treatment, and different variables, including contact time, ozone dosage, pH, and COD removal efficiency, were tested.

Rubber wastewater samples were collected from 9 production halls of a rubber wastewater processing factory in Kerman Province, Iran. Wastewater sampling was conducted at the output point, before discharge into the storage pond. After collection, the samples were fixed, transported to the laboratory, and immediately examined. First, each production unit was tested separately (9 units), and then, samples were mixed to make up 1 sample for the physicochemical treatment process.

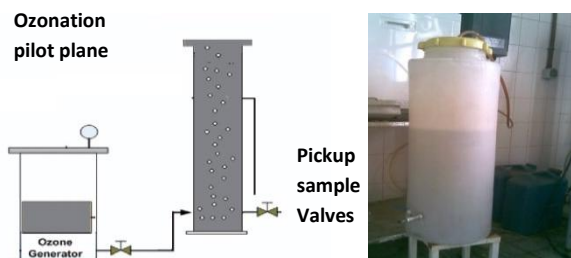
The successful application of various coagulant materials in water and wastewater treatments has been reported in many studies.<sup>14</sup> Various types of natural and synthetic organic polymers have been used for the coagulation-flocculation process in wastewater treatment.<sup>15</sup> The main disadvantage of a physicochemical treatment process is the high volume of sludge it produces.<sup>16</sup>

## Jar Tests

One well-known apparatus for selecting coagulant material for physicochemical wastewater treatment is the jar testing device, the results of which show treatment efficiency in terms of suspended matter and organic/inorganic matter removal.<sup>17,18</sup> Chemicals and coagulant materials are selected and optimum operating conditions (pH and exact amount of coagulant materials) are determined by means of jar testing.

Physicochemical experiments were carried out in a six-stirrer jar-test device (Phipps & Bird, USA). For the tests, 1000 ml of the sample was introduced into the jars. Then, the coagulant material was added by a beaker and the mixture was mixed rapidly (100 rpm) for 2 minutes. Subsequently, paddle velocity was decreased to 20 rpm for 20 minutes, and the flocculants were added into the tests in which ferric chloride ( $\text{FeCl}_3$ ) and aluminum sulfate [alum,  $\text{Al}_2(\text{SO}_4)_3$ ] were used. Finally, the paddles were withdrawn so that the particles could settle for a 30-minute period.<sup>19</sup>

Rubber wastewater treated through physicochemical process is discharged into the ozonation reactor (50 liters) for treatment by ozone (Figure 1).



**Figure 1. Schematic and photo of an ozonation pilot-scale**

In all tests, COD, turbidity, pH, oil and grease content of the clarified water, and sludge volume after 30-minute sedimentation ( $V_{30}$ ) were measured. COD and oil and grease contents were assayed using standard protocols given in section 5220 A.<sup>20</sup> In addition, pH was measured using a cation apparatus (Analytical Technology Inc., USA).

The ozone generator used in this study was a Compact Ozone Generator (OZONEUF, Model No. 6-5-11015, France Co., Ltd) (Figure 2). It was applied at various voltages and different ozone gas flow rates of 2-5 gr/hour. The ozone generation was determined by spectrophotometry using the standard potassium iodide (KI) absorption procedure.<sup>21</sup>

In this section of the treatment, ozonation was carried out in a 50-liter reactor fitted with a sand diffuser. A total of 6 samples were withdrawn periodically from the reactor by pickup sample valves. COD samples were collected at 7 contact times (15, 30, 45, 60, 90, 120, and 150 minutes). The initial value of COD was measured before the

ozonation process was started.

The schematic and a photo of the ozonation pilot-scale are provided in figure 1.



**Figure 2. Compact Ozone Generator**

## Results and Discussion

The tire and rubber industry has a great variety of uses for water and depends on it to cool its various types of equipment. Each use may have its own quality requirements.

Production Process in Kerman Barez Tire Factory:

The rubber tire manufacturing process includes the 11 steps of mixing, milling, extruding, calendaring, bead making, cementing and marking inks processes, cooling and culture, tire-building, lubricating, curing, and tire finishing.<sup>22</sup>

All the steps mentioned above are associated with the consumption of large amounts of water which produces a volume of 2500 cubic meters of sewage per month in the studied factory.

Wastewater characterization:

The parameters analyzed were conductivity, pH, COD, oil and grease, and turbidity. The features of raw effluent are summarized in table 1.

**Table 1. Characteristics of raw wastewater of Kerman Barez Tire Factory (separate production units)**

No.	Unit production/Variable	pH	COD (mg/l)	TSS (mg/l)	Oil and grease (mg/l)
1	Old Banbury Mixer (hall 1)	9.9	26145	34900	26249
2	New Banbury Mixer (hall 2)	8.9	6391	1031	2925
3	Effluent of water boilers	10.3	3403	-	255
4	Effluent of cementing process	6.6	5146	77	7903
5	Effluent of makeup tube	8.5	3237	1328	853
6	Effluent of makeup tire	7.0	2830	1080	730
7	Effluent of reverse osmosis unit	8.0	270	300	0
8	Effluent of boiler blowdown	2.1	3901	31	3901
9	Effluent of curing process	6.6	3071	220	481

COD: Chemical oxygen demand; TSS: Total suspended solids

**Table 2. Characteristics of mixed raw wastewater of Kerman Barez Tire Factory (final mixed samples)**

No.	Parameters	Deal	Industrial effluent standard <sup>23</sup>
1	COD (mg/l)	5613	< 120 (not to exceed 400)
2	TSS (mg/l)	560	< 50 (not to exceed 150)
3	Oil and grease (mg/l)	5651	-
4	pH	8.3	5.5-9.0

COD: Chemical oxygen demand; TSS: Total suspended solids

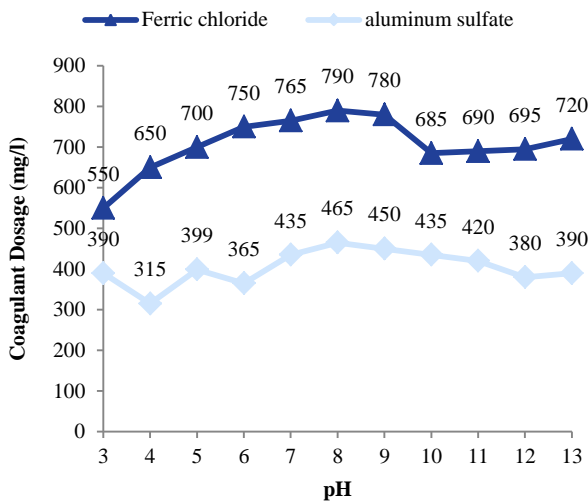
The characteristics of mixed raw wastewater of Kerman Barez Tire Factory are presented in table 2.

The concentrations of COD, total suspended solids (TSS), and oil and grease were much higher than discharge effluent standards.

Wastewater characteristics after treatment through a 2-stage process:

**First stage: Physicochemical Process**

The relationship between pH and coagulant material dosage (ferric chloride and alum) is shown in figure 3.

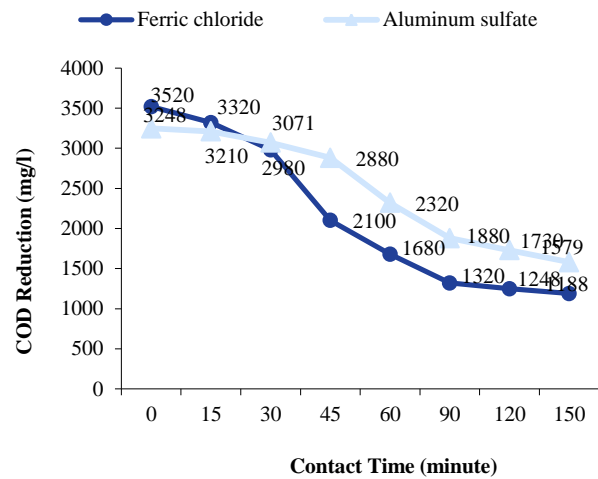


**Figure 3. Relationship between pH and coagulant material dosage (ferric chloride and alum)**

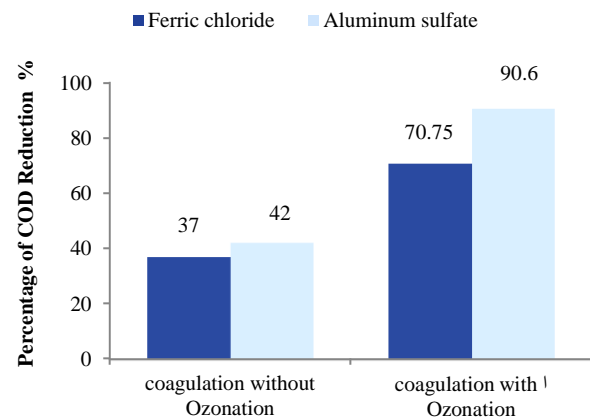
COD variation and removal efficiency in ozone pilot plane are shown in figure 4.

**Second stage: Ozonation Process**

In figure 5, the histogram of physicochemical treatment by ferric chloride and alum with and without ozonation is presented.



**Figure 4. Chemical oxygen demand (COD) variation and removal efficiency in ozone pilot plane**



**Figure 5. Physicochemical treatment by ferric chloride and alum with and without ozonation**

The results of this study showed that, in the first stage, wastewater was treated through chemical coagulation with ferric chloride (FeCl<sub>3</sub>.7H<sub>2</sub>O) for a contact time of 30 minutes. In this stage, COD reduction was 37% of the original amount (5600 mg/l). The optimum dosage of chloride (FeCl<sub>3</sub>.7H<sub>2</sub>O) and the pH range were 0.775 g/l and 6.5,



respectively. In the next stage of the physicochemical treatment with  $\text{Al}_2(\text{SO}_4)_3$ , the COD reduction rate was 42% of the original amount (5600 mg/l), and the optimum dosage and pH were 0.45 g/l and 6.5-7, respectively. In the second stage, after physicochemical treatment by  $\text{FeCl}_3 \cdot 7\text{H}_2\text{O}$  and  $\text{Al}_2(\text{SO}_4)_3$ , the wastewater was further treated by an ozonation process with 7 contact times (15, 30, 45, 60, 90, 120, and 150 minutes).

Ozone dosage was 5 gr/hour. The COD removal efficiency rates for the final effluent after the combined process are shown in figure 4 and are compared to the rates with and without ozonation. The removal efficiency of the COD parameter with the physicochemical process as a pretreatment was significantly higher than that with ozonation. This indicates that the combination process is better in terms of pollutant removal from the effluent of this industry. In the physicochemical stage of COD reduction, no significant difference in removal efficiency was observed with  $\text{Al}_2(\text{SO}_4)_3$  (42%) and  $\text{FeCl}_3 \cdot 7\text{H}_2\text{O}$  (37%). In 2010, wastewater from the cheese industry was treated through coagulation-flocculation using  $\text{FeSO}_4$ ,  $\text{Al}_2(\text{SO}_4)_3$ , and  $\text{FeCl}_3$ .<sup>24</sup> A ferric salt concentration of 250 mg/l resulted in a 40% to 60% reduction in COD. The optimum condition was found to be 3000 mg/l of  $\text{FeCl}_3$  at pH of 5.6, resulting in a 76% removal of COD. Coagulation-flocculation using  $\text{Al}_2(\text{SO}_4)_3$ ,  $\text{FeCl}_3$ , and aluminum polychlorosulfate showed low efficiency for the removal of avian influenza virus subtype  $\text{H}_5\text{N}_1$  and the swine-origin influenza A virus.<sup>25,26</sup>

A medical waste sterilization plant was pretreated with coagulation-flocculation using  $\text{FeCl}_3$ , ferrous sulfate, and  $\text{Al}_2(\text{SO}_4)_3$  by Ozkan et al.<sup>27</sup> Their results showed that a 60% COD removal and a high removal of suspended solids, nitrogen, and phosphorous were achieved through the application of 300 mg/l of  $\text{FeCl}_3$  at pH of 10.<sup>27</sup> Paint manufacturing wastewater containing

polyvinyl alcohol (PVA) was treated with alum.<sup>28</sup> The results showed that 80% COD removal was obtained with 2000 mg/l of both coagulants, whereas more than 90% removal was achieved for latex-based wastewaters with  $\text{FeCl}_3$ .<sup>28</sup> Chiavola et al. used alum, lime, and iron chloride as coagulants to treat olive mill wastewater.<sup>29</sup> Lime was selected as the best coagulant with 51% COD removal compared with alum and iron chloride. The results showed that the effluent was suitable for the subsequent biological treatment.<sup>29</sup> Kiril et al. found that the process of flocculation and coagulation and fission of acid improve oil biodegradation of olive oil mill wastewater.<sup>30</sup> Their results showed COD and phenol removal percentages of more than 67% and 72%, respectively.<sup>30</sup> Iron trichloride and aluminium polychloride were used as coagulation-flocculant material by Castrillon et al. to treat old landfill leachate.<sup>31</sup> The results of their study showed that 62% to 73% of non-biodegradable organic matter and more than 97% of turbidity and color were removed.<sup>31</sup> Samadi et al. studied the effects of different metal salt coagulants of polyaluminum chloride (PACl), alum, and ferrous on landfill leachate  $\text{Fe}_2(\text{SO}_4)_3$  (1500 mg/l) which resulted in a higher than 71% COD removal at pH of 12.<sup>32</sup> Zazouli et al. used lime to treat fresh leachate.<sup>33</sup> The optimum concentration of lime as a coagulant was found to be 2.4 g/l and optimum pH was 9.5. Heavy metals and COD removal efficiencies of 79%-88% and 25%, respectively, were obtained.<sup>33</sup> Maranon et al. used  $\text{FeCl}_3$ , alum, PACl, and polyacrylamide polyelectrolytes for the coagulation-flocculation treatment of landfill leachate.<sup>34</sup> They found that ferric chloride concentration of 0.6 g/l at pH of 5-5.5 resulted in 73% COD, 98% color, and 100% turbidity removal.<sup>34</sup>

In another study, alum,  $\text{FeCl}_3$ , and ferrous sulfate showed the same performance for COD removal in the coagulation by precipitation (C/P) process, while for coagulation/dissolved air flotation (C/DAF),

the order of removal efficiency was alum > FeCl<sub>3</sub> > ferrous sulfate. Pre-ozonation was used before coagulation to investigate its effect on the formation, breakup, and regrowth of the flocs. Increased O<sub>3</sub> concentration showed an adverse effect on floc formation with limited regrowth of broken flocs.<sup>35</sup> The effects of the application of O<sub>3</sub> on the COD and color removal efficiencies of a textile industry were investigated by Avsar and Batibay.<sup>36</sup> O<sub>3</sub> was effective in removing COD and color and improved COD and color removal in addition to the current chemical treatments.<sup>36</sup> In 2010, Demin used ozone/biological activated carbon (BAC)/TiO<sub>2</sub> to treat phenolic wastewater.<sup>37</sup> The results showed that when phenol concentration was 0.1 g/l, the O<sub>3</sub>-containing air flow rate was 0.05 m<sup>3</sup>/hour, O<sub>3</sub> concentration was 3.58 mg/l, pH value was 7.5, and treatment time was 30 minutes. Moreover, the phenol removal rate was 99% and COD removal rate was 55%.<sup>37</sup>

Orta de Velasquez et al. investigated the effect of O<sub>3</sub> on dissolved organic matter during wastewater coagulation using alum.<sup>38</sup> Adding O<sub>3</sub> to the coagulant treatment enhanced the quality of the final effluent compared with conventional coagulation treatments.<sup>38</sup> Lafi et al. investigated the use of a combination of coagulation and advanced oxidation processes (AOPs) for the removal of organic pollutants from olive oil mill wastewater. The percentage of COD removal of coagulation with O<sub>3</sub> was a little lower than that with O<sub>3</sub>/UV and H<sub>2</sub>O<sub>2</sub>/UV.<sup>39</sup> Hernandez-Ortega et al. reported combined electrocoagulation-ozonation to be a suitable pre-treatment for traditional biological processes and as a complete treatment for the discharge of industrial effluents into municipal sewers.<sup>40</sup> The combination of a biological treatment with the electrocoagulation-ozonation process led to a high-quality effluent.<sup>40,41</sup>

## Conclusion

The combination of a physicochemical

process with Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> as a pretreatment to the ozonation process can improve the efficiency of COD removal by up to 90.6%. This combined process was found to be very effective in removing the pollutants present in rubber industry wastewater. Moreover, it produced a final effluent which was low in suspended solids, clear, and odorless. However, the individual processes and the combined process (physicochemical and ozonation) were not sufficient to completely treat the highly polluted rubber wastewater. Thus, it is necessary to complete the treatment process with a method such as activated sludge. To reach industrial effluent standards, complementary processes such as activated sludge can be used.

## Conflict of Interests

Authors have no conflict of interests.

## Acknowledgements

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