

Cosmetic Wastewater Treatment Using Coagulation and Fenton Processes

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The cosmetic wastewater was treated by coagulation and Fenton process. COD of raw wastewater was 2888 mg/l. The effectiveness of these processes, defined as a decrease of COD was 66.4% for coagulation for a FeCl_3 dose 900 mg/l and 87.7% (to 295 mg/l COD) for Fenton process for $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ doses 3000/1000 mg/l. The contribution of coagulation in Fenton process was defined as 71.3% of the total treatment effect. The calculated H_2O_2 efficiency is high and for optimal doses reached 149.3%. The optimal pH of Fenton process is 3.0. Even a small change in its value leads to a rapid decrease in the efficiency of oxidation and hence in the effects of the process. The kinetics of the Fenton process can be described as $d[\text{COD}]/dt = -a \cdot t^m [\text{COD}]$, where t stands for time, while the a and m are constants, depending on the initial concentration of the reagents. It has been proved that the cosmetic wastewater is susceptible to purification by means of coagulation and Fenton process.

Keywords: cosmetic wastewater, wastewater treatment, coagulation, Fenton process

Introduction

For many years, cosmetic wastewater was deemed as harmless and as such was treated together with the domestic wastewater in the municipal wastewater treatment plant (WWTP). However, the presence of cosmetic ingredients in surface waters suggested that their removal in a biological WWTP was unsatisfactory and triggered researches in this area [1-5]. These ingredients were mainly synthetic musks (eg. galaxolide and tonalide) and UV filters (eg. benzophenone) [6]. These substances proved to be toxic for aquatic organisms [7, 8], human [9] and highly resistant to biochemical decomposition. Their removal is only by adsorption on activated sludge during biological treatment on WWTP [10].

Low effectiveness of biological treatment resulted in increased interest in physico-chemical methods and oxidation processes. These methods are not well examined for cosmetic wastewater and conclusions from different researches are not explicit. Results differ due to differences in composition of wastewater and pollutants concentrations. However, the larger plant and a wider range of produced personal care products (cosmetics), the more representative is wastewater for the cosmetics industry and the more general conclusion can be made.

Many authors have carried out studies on the application of coagulation [1, 11-14], electrocoagulation [11, 15], flotation [1], advanced oxidation processes [11, 12, 14-17], catalysts [18] and biological methods [19] for the wastewater treatment.

Many cosmetic factories used coagulation, flotation or their combination with biological methods for wastewater

treatment. An example may be Avon Operations Poland using flotation and biological treatment [20].

The aim of this study is to determine possibility of using the Fenton process for cosmetic wastewater treatment and estimation of the contribution of coagulation in the wastewater treatment total effect.

Materials and methods

Cosmetic wastewater was collected in May 2013 from the cosmetics factory located in Poland. Wastewater samples were refrigerated at 4°C. On the wastewater collection day, the factory produced creams, shampoos, powders and shower gels.

The following parameters were determined according to the EN or ISO Standards: COD, COD_{dis} (for sample filtered with 0.45 µm filter) (ISO 6060), total suspended solids (TSS) (EN 872), pH (EN ISO 10523), turbidity (EN ISO 7027), specific conductivity (EN 27888), and anionic surfactants (EN 903).

The mixed wastewater was left for 30 min for sedimentation in order to remove easily settleable solids. The wastewater after sedimentation was subjected to a Fenton process. All research on the wastewaters treatment processes was performed within 72 hours of collection.

The Fenton process was conducted in a 1L cylindrical reactor. The appropriate amount of acidic FeSO_4 solution was added to the reactor in Fe^{2+} concentration of 50 mg/mL (Stanlab, puriss p.a.). Furthermore, a 30% H_2O_2 solution was added (Stanlab, puriss p.a.). After 5, 15, 30, 60 and 120 min, the process was stopped by increasing the pH to 9.0. A pH of 9.0 was used in the final neutraliza-

tion/coagulation step to guarantee the complete decomposition of the remaining H_2O_2 during the night.

The efficiency of H_2O_2 was determined according to the Kang and Hwang equation as follows (Equation 1) [21]:

$$(1) \quad \eta (\%) = [\Delta COD / (0.4706 \cdot H_2O_2)] \cdot 100\%$$

where:

ΔCOD is the decrease of COD value (mg/l),

0.4706 is a constant factor for the amount of COD theoretically removed (mg) by 1 mg of H_2O_2 ,

$[H_2O_2]$ is the hydrogen peroxide dose (mg/l).

The coagulation process was conducted in a 0.5L cylindrical reactor. The appropriate amount of $Fe_2(SO_4)_3$ and $FeCl_3$ was added to the reactor in Fe^{3+} concentration of 100 mg/mL (Stanlab, puriss p.a.). Experiments were performed at pH 9.0. The samples were stirred 5 min for fast, followed by 10 min for the slow stirring. The wastewater was left to sedimentation in the measuring cylinder, followed by determination of the treated wastewater parameters.

Results and Discussion

Raw wastewater

Raw wastewater parameters are shown in table 1. COD of raw wastewater was 2888 mg/l. Raw wastewater was characterized by a small amount of the easily suspended solids (ESS). The volume of ESS was 3 ml/l, a further extension of observation period up to 24h resulted in increase to 3.5 ml/l. Removal of easily suspended solids through sedimentation resulted in a noticeable decreasing of the COD of 485mg/l (16.8%). COD_{dis} accounted for 44.2% COD_{sed} . These values are comparable with other cosmetic wastewater samples [13, 14].

Coagulation

The results of coagulation process are shown in Fig 1. The highest efficiency of the process, 66.4% decrease of COD was observed at a $FeCl_3$ dose of 900 mg/l. The effect of the process was similar and amounted to 59.2-66.4% COD removal. Regardless of a dose, $FeCl_3$ resulted in a better effect than $Fe_2(SO_4)_3$. $FeCl_3$ optimal dose of 900 mg/l was determined on the basis of a COD removal. COD values

Table 1. Raw wastewater parameters

Parameter	Unit	Value
COD_{raw}	mg/l	2888
COD_{sed}	mg/l	2403
COD_{dis}	mg/l	1062
TSS	mg/l	964
ESS	ml/l	3.0
Anionic surfactants	mg/l	150
pH	-	6.3
Conductivity	mS/cm	1.524
Turbidity	NTU	1200
Colour	-	grey

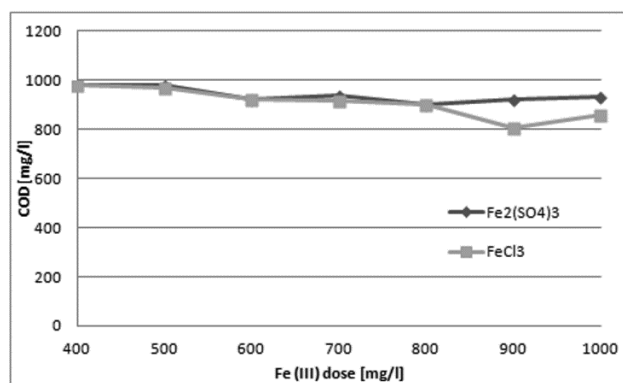


Figure 1. The effectiveness of the coagulation process for different doses of coagulants

of treated wastewater in a coagulant dosage range of 600 - 1000 mg/l differs by less than 5%. The COD values of treated wastewater were smaller than those presented by other authors [1, 11, 13, 14].

The effectiveness of the coagulation process was evident not only in decreasing the COD value. An additional effect of treatment was almost 100% colour and turbidity removal. In case of colour removal, it was precipitation or adsorption rather than coagulation [22].

After coagulation, the COD decreased to a value below COD_{dis} , 1062 mg/l, which means that as a result of coagulation, in addition to suspensions and colloids, 24.1% of the dissolved compounds has been removed.

The high efficiency of coagulation is expected – as wastewater is characterized by a large amount of suspensions. Another reason for the high efficiency of the coagulation could be the presence of large amounts of polycyclic musks (aromatic compounds). Polycyclic musks are characterized by a high lipophilicity, in particular those used in the largest quantities: galaxolide (HHCB) and tonalide (ATHN) [1, 3]. Much less lipophilic are musk xylene (MX) and musk ketone (MK). The presence of these musks has been established based on known production profile. Studies on coagulation of the hospital wastewater [24] have shown that musks are removed in a 70-84%. These compounds are removed primarily by adsorption. The obtained results show that sorption processes play a crucial role in removing dissolved organic compounds. At pH 9.0, the predominant form of iron in wastewater is $Fe(OH)_3$ [1]. The efficiency of wastewater treatment by Aloui et al [11] was less and the obtained COD values of treated wastewater were significantly higher, despite the use of higher doses of coagulant and the use of lime.

Fenton process

The results of Fenton process are shown in Figures 2a-d, 3 and 4. The changes in the COD values (Fig. 2a-d) were relatively sharp, with rapid changes in COD removal within the first 5 min. This may suggest that the final neutralization/coagulation is the main mechanism of COD re-

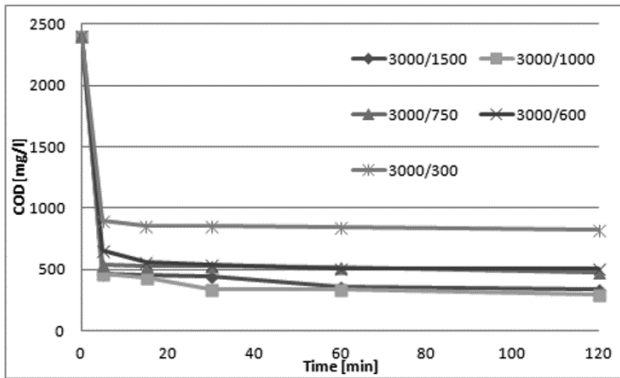


Figure 2a. The effectiveness of the process of Fenton H_2O_2/Fe^{2+} doses

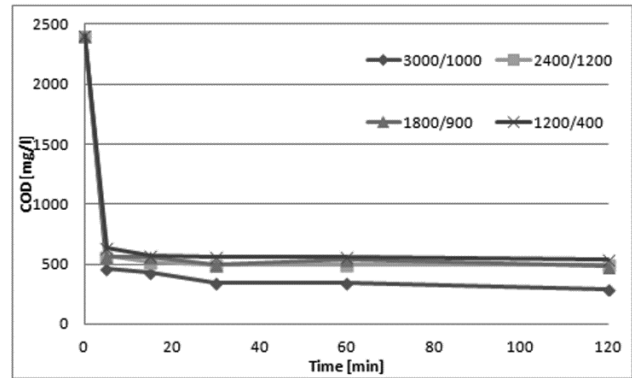


Figure 3. The effectiveness of the Fenton process for different reagent H_2O_2/Fe^{2+} doses

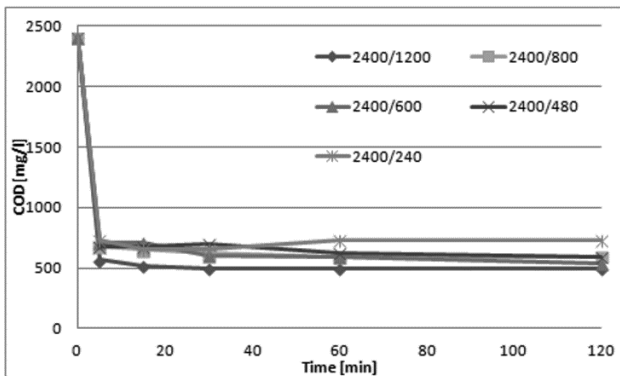


Figure 2a. The effectiveness of the process of Fenton H_2O_2/Fe^{2+} doses

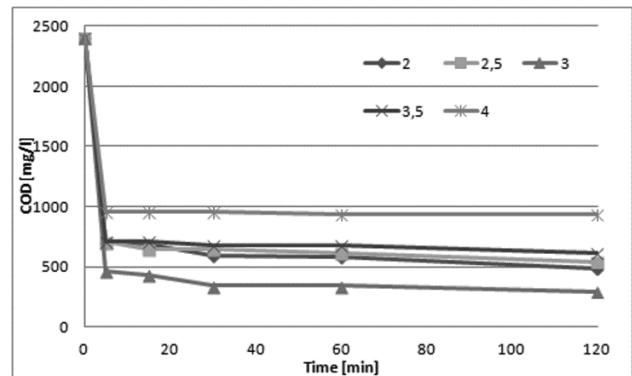


Figure 4. The effectiveness of the Fenton process for reagent H_2O_2/Fe^{2+} doses 3000/1000mg/l for different pH

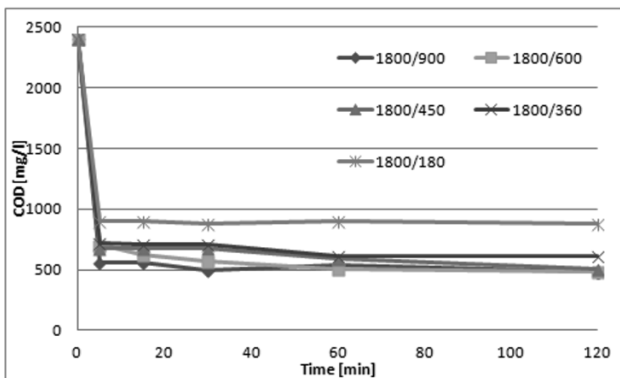


Figure 2c. The effectiveness of the process of Fenton H_2O_2/Fe^{2+} doses

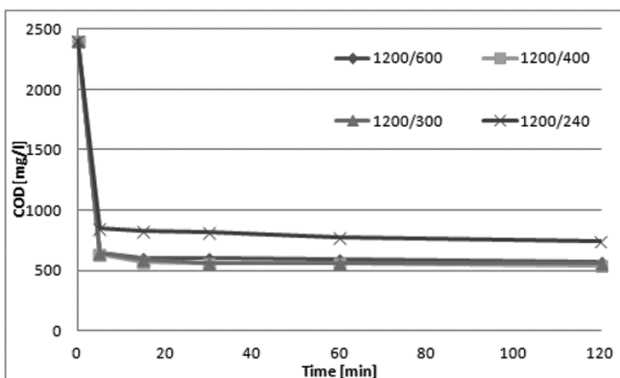


Figure 2d. The effectiveness of the process of Fenton H_2O_2/Fe^{2+} doses

removal and Fenton reaction played a less significant role. Further extension of the process time, causes only slight COD decreasing. The greatest efficiency of the process, 87.7% COD decreasing, to the 295mg/l, were obtained for H_2O_2/Fe^{2+} 3000/1000mg/l doses. Regardless of doses, the effect was at least 63.5%, corresponding to a final COD value 876mg/l. Usually [25] the effect of the Fenton process increases with increasing dosage of the reagents, what was also observed in the presented work.

On the other hand excess of one of the Fenton reagents resulted in HO^{\bullet} and HO_2^{\bullet} decay according to the following reactions:

- (2) $H_2O_2 + HO^{\bullet} \rightarrow HO_2^{\bullet} + H_2O$
 $k_2 = 1.7-4.5 \cdot 10^7 \text{ (l mol}^{-1} \text{ s}^{-1}\text{)}$
- (3) $HO_2^{\bullet} + HO^{\bullet} \rightarrow O_2 + H_2O$
 $k_3 = 1.4 \cdot 10^{10} \text{ (l mol}^{-1} \text{ s}^{-1}\text{)}$
- (4) $HO^{\bullet} + HO^{\bullet} \rightarrow H_2O_2$
 $k_4 = 5-8 \cdot 10^9 \text{ (l mol}^{-1} \text{ s}^{-1}\text{)}$
- (5) $Fe^{2+} + HO^{\bullet} \rightarrow Fe^{3+} + OH^-$
 $k_5 = 2.5-5 \cdot 10^8 \text{ (l mol}^{-1} \text{ s}^{-1}\text{)}$
- (6) $HO_2^{\bullet} + HO_2^{\bullet} \rightarrow O_2 + H_2O_2$
 $k_6 = 0.8-2.2 \cdot 10^6 \text{ (l mol}^{-1} \text{ s}^{-1}\text{)}$

Increasing the H_2O_2 dose up to 3000 mg/l enhanced the COD removal. Optimal $\text{H}_2\text{O}_2/\text{COD}$ ratio was 1,25/1. The effects of this process were dependent on the $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ ratio. For certain H_2O_2 doses, the best COD removal effect was obtained when this ratio was 2 – 3. The H_2O_2 concentration played a crucial role in the efficiency of the degradation process. Degradation of pollutants increased as the H_2O_2 dose increased. The deterioration regarding the effects of this process for a $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ ratio higher than 3 potentially resulted from the rate of HO^\bullet scavenging (Equations 2-6).

For each $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ doses, the worst treatment effect was observed for a mass ratio of 10/1, which in addition to HO^\bullet scavenging may be associated with insufficient iron dose at the final coagulation. Furthermore, due to the large amount of H_2O_2 decomposing during the final coagulation, final sedimentation was very difficult.

Increasing Fe^{2+} dose caused an increase in the COD removal, up to 1000 mg/l. Further increasing the Fe^{2+} dose to 1500mg/l caused deterioration in the efficiency of the process. This phenomenon is observed for $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ doses 3000/1500mg/l and can be associated with overdose of coagulant. COD value for $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ doses 3000/1500mg/l is 16% higher than for 3000/1000mg/l (341 and 295mg/l respectively).

Decreasing the Fe^{2+} dose deteriorated the final coagulation effect. However, increasing Fe^{2+} dose inhibited the Fenton reaction, probably due to the greater efficiency of HO^\bullet scavenging (Equation 5). From these two opposite effects there is a greater impact on the whole process of coagulation and because of that the optimal doses of $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ were 3000/1000 mg/l.

The cause of a much larger efficiency of Fenton process compared with coagulation (21.3%) is a chemical oxidation followed by a final coagulation. Dissolved compounds are subjected to chemical oxidation. They are almost indelible in coagulation in which their removal mechanism is likely to be limited to adsorption. The compounds responsible for COD_{dis} were removed 72.2%. Oxidation reactions occurring in the Fenton process cause the conversion of the compounds originally contained in the wastewater into another compounds. The intensity of oxidation reaction decreases during the process due to consumption of the reagent i.e. H_2O_2 . This explains the lower intensity of COD decrease for longer process times. For optimal $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ 3000/1000mg/l doses, compared to the maximum effect of COD decreasing caused by coagulation, 1503mg/l, the oxidation reaction results in an additional decreasing of 605mg/l, which is 28.7% of the total effect. On the other hand, the results indicate dominating contribution of the final neutralization/coagulation in the total treatment effect. At the final coagulation there are compounds which were present in the wastewater from the beginning and new substances which were produced during the chemical oxidation. It is possible, that some com-

pounds created during oxidation reaction have higher susceptibility to coagulation, than compounds from which they arise. Oxidation of functional groups (for example carboxylic) on the surface of suspensions facilitates aggregation into larger flocs in the final coagulation. The reason for this phenomenon is higher susceptibility to coagulation of oxidized compounds containing polar functional groups [26]. Similarly to the coagulation, wastewater pretreated in Fenton process, are characterized by a complete removal of colour and turbidity. The reason is the final coagulation/neutralization occurring after chemical oxidation stage.

Confirmation of the dominant contribution of coagulation in Fenton process thesis was achieved studying the effect of pH on COD removal effect. At the same doses of H_2O_2 , Fe^{2+} and the same pH value of the final coagulation, differences in treatment effect must be related only to the intensity of the oxidation reaction, which is closely related to the pH value at which occurs. Clearly the best results were obtained at pH 3.0. Decreasing or increasing of pH, resulted in a significant deterioration of wastewater treatment effect. The results obtained coincide with studies published by other authors [27-30].

At higher pH non-reactive iron forms appear in wastewater: oxohydroxides or hydroxides precipitates and acts as radical scavengers. In this situation, the amount of iron available for radical generation is significantly lower than for the same dose, but in a lower pH. In addition, the redox potential of HO^\bullet decreases with increasing pH [25]. The effect of the process carried out at pH 4.0 is comparable to the effect of coagulation alone. On the other hand, lowering the pH below 3.0, results in the appearance of numerous iron aquacomplexes and hydration H_2O_2 to $[\text{H}_3\text{O}_2]^+$, which is more stable and therefore less reactive with Fe^{2+} .

H_2O_2 effectiveness

Efficiency of H_2O_2 is shown in Table 2a-d. Due to the high efficiency of Fenton process, calculated values are high and range from 112% to 329%. The highest efficiency was calculated for a $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ doses 1200/400 mg/l. COD value for this dose was 542 mg/l and was almost twice higher that COD for optimal $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ doses 3000/1000 mg/l.

In this case, despite a decrease in COD value to 295 mg/l, due to much higher dose of H_2O_2 its performance was of 149% and was one of the lowest, which further confirms the thesis of predominant contribution of the final coagulation/neutralization in the overall result of the Fenton process. This allows to conclude that the higher dose of Fe^{2+} and the lower the dose of H_2O_2 , the greater the Fenton process efficiency.

Because of the complex nature of the treatment process (oxidation and coagulation), this parameter do not describe the efficiency of H_2O_2 but total purification effect in relation to the H_2O_2 dose.

Table 2a. Oxidant efficiency and kinetics for Fenton process

H ₂ O ₂ /Fe ²⁺ doses [mg/l]	3000/1500	3000/1000	3000/750	3000/600	3000/300
η [%]	146	149	136	134	112
a	0,085	0,114	0,026	0,065	0,026
m	-0,941	-0,92	-0,982	-0,947	-0,976
n	0,059	0,0797	0,018	0,053	0,025
k	1,449	1,43	1,445	1,229	1,049
R ²	0,832	0,922	0,679	0,883	0,896

Table 2b. Oxidant efficiency and kinetics for Fenton process

H ₂ O ₂ /Fe ²⁺ doses [mg/l]	2400/1200	2400/800	2400/600	2400/480	2400/240
η [%]	169	160	165	160	148
a	0,039	0,041	0,075	0,037	-0,009
m	-0,972	-0,965	-0,929	-0,969	-1,007
n	0,0276	0,0346	0,0706	0,0314	-0,007
k	1,4064	1,1945	1,0568	1,1703	1,2636
R ²	0,803	0,950	0,873	0,610	0,033

Table 2c. Oxidant efficiency and kinetics for Fenton process

H ₂ O ₂ /Fe ²⁺ doses [mg/l]	1800/900	1800/600	1800/450	1800/360	1800/180
η [%]	227	226	224	211	180
a	0,0375	0,0930	0,0687	0,0503	0,0064
m	-0,973	-0,913	-0,937	-0,954	-0,993
n	0,0271	0,0874	0,0628	0,0462	0,0066
k	1,3870	1,0647	1,0949	1,0903	0,9700
R ²	0,533	0,992	0,683	0,733	0,430

Table 2d. Oxidant efficiency and kinetics for Fenton process

H ₂ O ₂ /Fe ²⁺ doses [mg/l]	1200/600	1200/400	1200/300	1200/240
η [%]	324	329	327	294
a	0,031	0,042	0,044	0,037
m	-0,976	-0,967	-0,968	-0,961
n	0,0245	0,0329	0,0353	0,0389
k	1,2748	1,2847	1,2601	0,9614
R ²	0,941	0,851	0,802	0,918

Kinetics

Kinetics calculation results are shown in Table 2a-d. Many authors undertook an attempt to describe the kinetics. Pseudo – first and pseudo – second order equations were used [16, 31, 32]. More complex approach was also proposed. Wu et al. (2011) [33] suggest a two-stage model consisting phase of rapid COD decreasing and the phase of stagnation. The application of these models does not give satisfactory results. The Fenton process is very complex and simple approach does not take into account aspects such as the final coagulation, changes in the concentration of individual substrates or the complexity of the oxidation reactions that occur over time.

To describe the process, the following kinetic models were used:

– pseudo-first order equation in relation to COD value (7)

$$(7) \quad d[\text{COD}]/dt = -k_1[\text{COD}]$$

– pseudo-second order equation in relation to COD value (8)

$$(8) \quad d[\text{COD}]/dt = -k_2[\text{COD}]^2$$

– empirical equation taking into account other factors that occur during the process: Balcerzak Equation (9)

$$(9) \quad d[\text{COD}]/dt = -a t^m [\text{COD}]$$

where: t - time, a and m - constants, depending on the initial concentration of the reagents.

After integration and use of double logarithm the following equation (10) was obtained:

$$(10) \quad \ln \ln[\text{COD}]_0 / [\text{COD}] = (m+1) \ln t + \ln a - \ln (m+1)$$

where: [COD]₀ – initial COD

Calculations using the pseudo – first (7) and the pseudo-second (8) order kinetics model did not give satisfactory results and are not presented. Only the kinetics calculated by the Balcerzak equation (9) shows significant correlation coefficients R². This finding was inconsistent with the results of the studies presented by Bautista et al. (2007) [16], who stated that treating cosmetics wastewater using the Fenton process might be described by the second-order reaction equation.

The highest compatibility was achieved for the largest and the smallest doses of H₂O₂. In other cases, H₂O₂/Fe²⁺ mass ratio 10/1 can not be described by this model (low R² value). The highest compatibility was achieved for H₂O₂/Fe²⁺ doses 1800/600, 2400/800 and 1200/600mg/l.

Conclusions

Cosmetic wastewater is susceptible to purification by coagulation and Fenton process. The effectiveness of these processes, defined as a decrease of COD, amounts up to 66.4% for a FeCl₃ dose of 900 mg/l and 87.7% for H₂O₂/Fe²⁺ doses of 3000/1000 mg/l. Contribution of coagulation in Fenton process was defined as 71.3% of the total treatment effect. Despite this, calculated H₂O₂ efficiency is high and for optimal parameters reached 149.3%. The optimal pH for Fenton process is 3.0. Even a small increase or decrease of its value resulted in significant decrease in efficiency of oxidation and thus in the effects of the whole process. The kinetics can be described by following equation $d[\text{COD}]/dt = -a t^m [\text{COD}]$, where t - time, while the a and m - constants, depending on the initial concentration of the reagents.

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