Global NEST Journal, Vol 25, No 2, pp 7-12

Preliminary research on the efficiency of the coagulation and Fenton process for the removal of organic substances contained in wastewater from plastic lens production

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Received: 20/03/2022, Accepted: 29/05/2022, Available online: 06/07/2022
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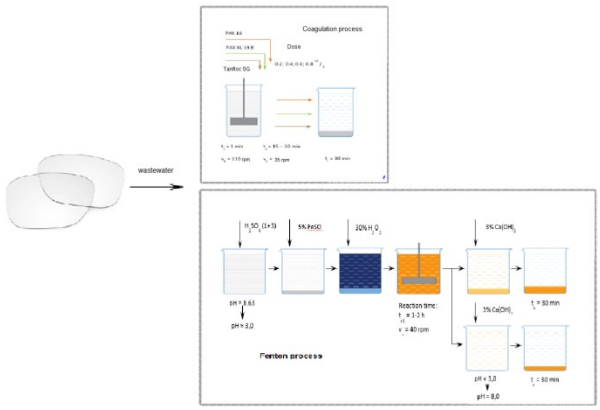
https://doi.org/10.30955/gnj.004308

Graphical abstract



Abstract

250 million people worldwide suffer from visual impairment. To correct this, 64% of them wear glasses of which, more than 80% have plastic lenses. Plastic lenses made of polycarbonate discs are created through machining, which requires the use of a coolant. The ensuing wastewater contains plastic chips, water, and coolant. In the facility studied, the chips are separated by filtration. Then, water and coolant are returned to the machine tool. Due to their low particle weight, some of the plastic dust floats and creates foam. If there is too much dust foam, a skimmer is used. To avoid machine damage and product quality deterioration, the coolant mixture in circulation is changed every 24 hours. For this study, wastewater was sampled from the plastic lens facility. It was characterized by a high COD value of 11200 - 20800 mg/L, a turbidity of 260-870 NTU, and a colour of about 200 mg/L. Wastewater with these parameters should not be discharged into the sewage system. For wastewater treatment, a coagulation process (PAX 18 and PAX XL 19 H, Tanfloc SG) and Fenton process were studied. The application of aluminium coagulants resulted in a maximum reduction of COD by 55%. Maximum COD reduction after the Fenton process was 50.7%.

Keywords: COD, industrial wastewater treatment, advanced oxidation process, Fenton reagent, coagulation

1. Introduction

The most popular forms of plastic processing take advantage of their flow at elevated temperatures (e. g. injection moulding, casting) (Wilczyński, 2000). However, there are products made of plastic, where the main production process is machining, which requires the use of coolant. An example of such products are plastic eyeglass lenses. The production process creates wastewater containing plastic chips, plastic dust, water, coolant. Sometimes a foam skimmer is used after which the wastewater is discharged into a sewer. If such wastewater contains fine suspended solids or colloids, it can clog the sewer it is discharged through. Synthetic coolants and oil-in-water emulsion coolants (mostly semi-synthetic, less commonly mineral) are used for machining plastics. Used emulsions and coolants should not be discharged into the sewerage system for multiple reasons, including the difficulties they may cause in municipal sewage treatment plants. This is because they are characterised by a high content of hydrocarbon chain compounds, due to which the COD value may be as high as several hundred thousand mg O_2/L, which may lead to a significant change in the load of organic pollutants in the biological reactor of the treatment plant. Further problems may arise in the sludge management. Oils contained in emulsions interfere with methane digestion of sludge. Furthermore, carcinogenic substances are present in some coolants and the oil phase emitted in the sewage pipes can lead to clogging. For these reasons, used coolants (especially emulsion coolants) should be treated before discharge into the sewage system, or they should be collected and recycled by specialised companies.

In Poland, industrial plants sign a contract with the water and sewage company to discharge wastewater into the municipal sewerage system. In accordance with the law, the company decides the permissible COD value for the wastewater it discharges. The most common COD effluent discharge standard is 1000 mg/L. If the requirements are not met, an additional charge must be paid by the manufacturer. Therefore, for industrial plant wastewater, the most relevant indicator to be reduced is COD. The treatment of such wastewater can be carried out chemically (e. g. coagulation or advanced oxidation processes). Coagulation processes have been successfully used for the removal of suspended solids, colour and dissolved organic matter from different aqueous sources such as dyes wastewater, municipal sewage, micro-polluted water, oily wastewater and paper industry wastewater. Numerous coagulants and have been studied in order to improve the performance of the physicochemical process (Molina *et al.*, 2014). Fenton may be considered as a one of the most effective advanced treatment processes in the removal of hazardous organic pollutants from wastewater (Xu *et al.*, 2020).

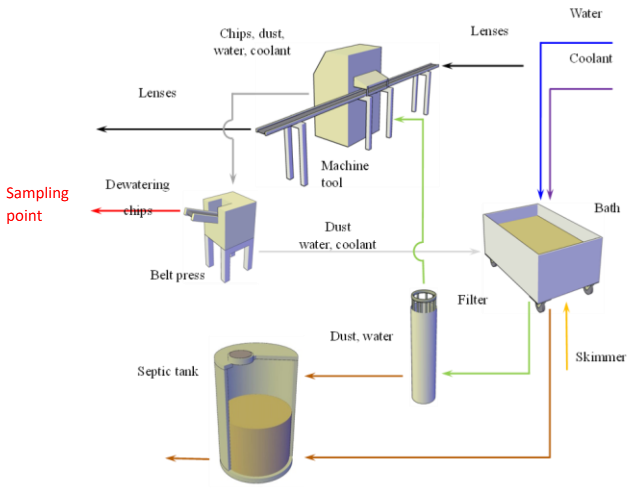


Figure 1. Plastic lens manufacturing process and the wastewater sampling point.

The Fenton process is based on the catalytic generation of free radicals, such as $OH^\bullet$ and $OOH^\bullet$, resulting from the chain reaction between ferrous ion and hydrogen peroxide at acidic pH. (Pliego *et al.*, 2012) Fe^{+3} ions, dissolved in water, can form different complexes; for instance, at pH close to 3, $[Fe(H_2O)_5(OH)]^{2+}$ becomes the predominant stable species (Gil Pavas*et al.*, 2017). Hydroxyl radicals with a high redox potential of 2.8 V, can effectively degrade most organic pollutants in a non-selective manner (Lin *et al.*, 2022). In order to optimise the Fenton process, reagent doses should be determined for each type of industrial wastewater. The Fenton process can be implemented simply, without requiring special equipment, and can be easily integrated into existing water treatment processes, such as coagulation, filtration, and biological oxidation (Ribeiro J.P., Nunes M.I, 2021). This paper reports on the feasibility of a solution for the treatment of plastic lens production wastewater using coagulation and Fenton processes. The effectiveness of different doses of aluminium coagulants (PAH 18, PAX XL 19 H) and Tanfloc SG coagulant, as well as different doses of Fenton reagent and oxidation times, were tested.

2. Materials and methods

2.1. Materials

Wastewater samples from plastic eyeglass lens production were collected from the septic tank of an industrial plastic lens manufacturer located in Poland. The plastic lens manufacturing process and the sampling point are shown in Figure 1.

2.2. Methods

The experiment was divided into two main stages that would examine two test processes:

1) coagulation process using coagulants: PAX18, PAX XL 19 H, Tanfloc SG, (series I-III);

2) advanced oxidation process using Fenton reaction, (series IV-VI).

Both stages were conducted at an ambient temperature of approximately 20 °C.

Chemical analyses in raw and treated wastewater were determined in accordance with the following standard methods:

pH - by electromotive method, PN-90-C-04540-01;

COD - by the titration method, PN-ISO 6060: 2006;

turbidity – by the nephelometric method, PN-EN ISO 7027-2:2019-04;

colour – by the comparison method, PN-EN ISO 7887:2012.

The coagulants used in this study can be classified into two types, i. e. chemical and natural coagulants (Sukmana *et al.*, 2021). Chemical coagulants used were PAX 18 and PAX XL 19 H. PAX 18 is an aqueous solution of polyvinyl chloride with the following physicochemical properties:

colour: light yellow;

Al_2O_3 content: 17.0 ±0.6%;

Al^{+3} ion content: 9.0 ±0.3%;

Cl- ion content: 21,0 ±2,0%;

alkalinity: 41,0 ±3,0%;

density at 20°C: 1360 ±10 kg/m^3;

viscosity at 20°C: approx. 20 mPa·s;

pH: 1.0 ±0.2.

PAX XL 19 H is a slightly turbid aqueous solution of polyglycine chloride with higher Al_2O_3 content than PAX 18. It has the following physicochemical properties:

colour: slightly grey;

Al_2O_3 content: 23.6 ±0.6%;

Al^{+3} ion content: 12.5 ±0.3%;

Cl- ion content: 8,5 ±1,0%;

alkalinity: 85.0 ±5.0%;

density at 20°C: 1340 ±30 kg/m3;

viscosity at 20°C: approximately 20 mPa·s;

pH: 3.5 ±0.4.

Tanfloc SG is a natural polymer of plant origin, produced from the wood of black acacia (*Acacia mearnsii*) grown in Brazil. The technological process for producing Tanfloc SG is protected by the patent 6.478.986 B1, and the commercial product itself contains up to 33% tannin, (Beltrán-Heredia *et al.*, 2010).

Tannin is composed of many gallic acid molecules and D-glucose molecules, and it combines with most heavy metal salts which can precipitate many alkaloids from solution (Biliński at al., 2012). It is used in water and wastewater technology as a coagulant and flocculant (either alone or as a supporting agent).

The tannin coagulant used in this study was a dark brown, opaque concentrate with the following physicochemical properties:

colloidal particle content: 30-34%;

viscosity measured with a Ford cup size 4, at 25°C: maximum 50 s (1.75 cm2/s);

pH: 1.3-2.3.

According to the producer's claims, the coagulant is effective in the pH range from 4.5 to 8.0 and does not affect the pH, as it does not affect the alkalinity of treated wastewater.

Table 1. Wastewater quality after coagulation processes - series I

Parameter	Unit	Raw wastewater	PAX 18				PAX XL 19-H				Tanfloc SG			
Dose	mL/L	-	0.2	0.4	0.6	0.8	0.2	0.4	0.6	0.8	0.2	0.4	0.6	0.8
pH	-	8.41	8.04	7.90	7.86	7.57	8.33	8.26	8.11	7.97	8.19	8.20	8.20	8.20
COD	mg O_2/L	13600	4320	4720	4640	4280	4240	4800	4640	4320	4800	4720	4880	4720
COD reduction	%	-	68.2	65.3	65.9	68.5	68.8	64.7	65.9	68.2	64.7	65.3	64.1	65.3
Turbidity	NTU	870	8	14	23	15	13	15	11	10	17.2	19.5	21.2	20
Colour	mg Pt/L	200	25	25	22	22	25	22	20	20	25	27	27	30
Sludge	mL/L	-	70	90	113	123	91	127	129	187	97	99	111	96

Table 2. Wastewater quality after coagulation processes - series II

Parameter	Unit	Raw wastewater	PAX 18				PAX XL 19-H				Tanfloc SG			
Dose	mL/L	-	0.2	0.4	0.6	0.8	0.2	0.4	0.6	0.8	0.2	0.4	0.6	0.8
pH	-	8.84	8.38	8.33	8.37	8.34	8.55	8.47	8.47	8.41	8.60	8.59	8.56	8.56
COD	mg O_2/L	13200	9040	8880	9120	8800	9040	8800	8800	8960	8640	8880	9200	9360
COD reduction	%	-	31.5	32.7	30.9	33.3	31.5	33.3	33.3	32.1	34.5	32.7	30.3	29.1
Turbidity	NTU	490	7.2	14.7	13.8	7.1	6.2	5.7	5.1	6.4	9.4	12.9	15.2	33.2
Colour	mg Pt/L	200	27	27	25	25	25	25	23	27	30	35	40	40
Sludge	mL/L	-	44	62	90	104	35	51	66	82	14	7	25	10

Table 3 Wastewater quality after coagulation process - series III

Parameter	Unit	Raw wastewater	PAX 18	PAX XL 19-H	Tanfloc SG
Dose	mL/L	-	0.8	0.8	0.8
pH	-	8.43	8.13	8.25	8.37
COD	mg O_2/L	20800	9280	9200	9600
COD reduction	%	-	55.4	55.8	53.8
Turbidity	NTU	490	5.5	10.1	15.4
Colour	mg Pt/L	200	27	27	40
Sludge	mL/L	-	120	101	51

The main factors that determine the removal of contaminants from wastewater during the coagulation process are: coagulant dosage, settling time, and pH. Coagulant doses of 0.2; 0.4; 0.6 and 0.8 ml/l were used in this study. The destabilization time was 1 minute, flocculation time was 15 - 30 minutes and sedimentation time was 30 minutes.

Due to the limited effectiveness of coagulation in reducing organic compounds expressed by COD, it was decided to test wastewater treatment by advanced oxidation processes using Fenton's reagent.

2.3. Advanced oxidation process using Fenton reagent

The overall homogeneous Fenton chemistry is highly complex, and includes both oxidation and coagulation reactions (Ribeiro, Nunes 2021). In all tests, before the Fenton process was initiated, the pH was lowered to 3.0 with sulphuric acid (Gulkaja *et al.*, 2006; Xu *et al.*, 2020). In test series IV, reagent doses were used such that the weight ratio of Fe^{+2}/H_2O_2 was 0.125. According to Riberio (Ribeiro, Nunes 2021), chemical coagulation is dominant at lower H_2O_2/Fe^{2+} ratios, while chemical oxidation is dominant at higher ratios. Thus, test series IV was dominated by chemical coagulation. In test series IV the reaction time was only 15 min. In test series V and VI, the reagent doses were changed such that the weight ratio of Fe^{+2}/H_2O_2 was in the range 0.14-0,20 and the reaction time was extended to 1 or 2 h (Cetinkaya *et al.*, 2018; Kuo *et al.*, 2012). In these series, there were two types of alkalinization (alkalinization of the sample and decanted liquid only).

3. Results and discussion

3.1. Coagulation process

The results obtained in test series I-III for the coagulation process are shown in Tables 1-3.

In all tests, raw sewage was characterised by high COD concertation in the range 11200 mg/L – 20800 mg/L. In series I, for each of the applied coagulants and successive doses, after the process was complete, similar values of COD in treated wastewater were obtained in the range of 4240-4880 mg O_2/L, and percentage reductions in the range of 64.1-68.5%. During this series, there was no significant influence of the dose level or the type of coagulant on the efficiency of organic compound removal from wastewater. The benefit of conducting this process was to obtain treated wastewater with very low turbidity in the range of 8-20 NTU, which will not have the ability to precipitate suspended solids in municipal sewage.

The coagulation process generates sludge. When the aluminium-based coagulants were used, the amount of sludge formed after sedimentation increased with increasing coagulant dose and ranged from 7.0-18.7%. In the case of the Tanfloc SG coagulant, the amount of sludge formed after coagulation was independent of the dose and ranged from 9.6-11.1%.

Table 4. Wastewater quality after advanced oxidation processes using Fenton reagents - **series IV**

Parameter	Unit	Raw wastewater	Fenton D_1	Fenton D_2	Fenton D_3	Fenton D_4	Fenton D_5	Fenton D_6
Dose	mg H_2O_2/L	-	288	576	846	1100	1176	1768
	mg $FeSO_4$/L		100	200	300	300	400	600
pH	-	8.62	8.02	8.09	8.20	8.16	8.20	8.20
COD	mg O_2/L	13600	9280	9440	9120	8480	9120	8800
COD reduction	%	-	31.8	30.6	32.9	37.6	32.9	35.3
Turbidity	NTU	260	1.08	1.08	1.05	0.95	0.92	1.67
Colour	mg Pt/L	170	22	25	30	30	35	40

Table 5. Wastewater quality after advanced oxidation processes using Fenton reagents (t_{r1} = 1h) series V

Parameter	Unit	Raw waste water	Fenton D_1		Fenton D_2		Fenton D_3	
			-	Decanted liquid	-	Decanted liquid	-	Decanted liquid
Dose	mg H_2O_2/L	-	3500		3000		2500	
	mg $FeSO_4$/L		1357		1357		1357	
pH	-	8.63	7.85	7.92	7.84	7.93	7.94	7.91
COD	mg O_2/L	11200	5920	6400	6240	7200	5760	6240
COD reduction	%	-	47.1	42.9	44.3	35.7	48.6	44.3
Turbidity	NTU	571	0.48	0.6	0.53	0.38	0.33	0.28
Colour	mg Pt/L	200	40	30	40	30	50	35
Sludge	mL/L	-	54	34	60	62	58	72

In the second test series, with the same doses of coagulant but with an extended flocculation time to 30 minutes, a lower reduction of organic compounds was obtained with a range of 29.1-34.5%. In series II, no effect of dose on the reduction of COD values was observed for the aluminium coagulants, while for Tanfloc, an increase in dose caused a decrease in the reduction of organic compounds. Achieving only a low reduction in COD values may have been influenced by changing the coolant or surfactant used in the production process. The amount of sludge formed in series II, with the same doses of coagulants, was much smaller than in series I, but the correlations were the same as in series I (for aluminium coagulants, an increase in dose caused an increase in the amount of sludge, for Tanfloc SG coagulant, no such correlation). The amount of sludge formed after Tanfloc SG coagulation is significantly lower than the amount formed after the application of the aluminium coagulants. In series III, the efficiency of the coagulation process (at 30 minutes flocculation) was checked at the highest dose of coagulant: 0.8 mL/L. Again, no differences in process efficiency were observed depending on the coagulant used. In the case of aluminium coagulants, the COD removal efficiency was about 55%, while for Tanfloc SG, 53%. During treatment of textile wastewater by coagulation process using aluminium coagulants, a reduction COD values from 2073 mg/L to 322 mg/L was achieved (84%) by Favero (Favero *et al.*, 2020), but

GilPavas (GilPavas *et al.*, 2017) achieved a COD reduction of only 48% (from 845 mg/L to 450 mg/L) with a dose of 700 mg $Al_2(SO_4)_3$/L. Wolf (Wolf *et al.*, 2015) achieved a COD reduction of 77.28% and a sludge volume of 27.3 mL/L during coagulation of dairy wastewater with Tanfloc SG at a dose of 20 mg/L.

The study of the Tanfloc SG coagulants used in the treatment of galvanic industry wastewater described by Vaz *et al.* (2010) obtained results of high colour (96.77%) and turbidity (99.38%) removal using Tanfloc SG. Bortolatto *et al.* (2017) obtained about 96% for turbidity removal and apparent colour removal of 78% treating the wastewater of a swine-slaughterhouse using Tanfloc SG as the coagulant in the coagulation process.(Land *et al.*, 2020). In test series I – III, a near equally high reduction in colour and turbidity was achieved with each coagulant.

Table 6. Wastewater quality after advanced oxidation processes using Fenton reagents (t_{r2} = 2h) - series VI

Parameter	Unit	Raw waste water	Fenton D_1		Fenton D_2		Fenton D_3	
			-	Decanted liquid	-	Decanted liquid	-	Decanted liquid
Dose	mg H_2O_2/L	-	3500		3000		3000	
	mg $FeSO_4$/L		500		500		600	
pH	-	8.63	8.07	7.93	8.00	7.88	7.93	7.90
COD	mg O_2/L	11200	6560	5600	5760	6000	6080	5520
COD reduction	%	-	41.4	50.0	48.6	46.4	45.7	50.7
Turbidity	NTU	571	0.57	0.43	0.48	0.47	0.36	0.33
Colour	mg Pt/L	200	40	40	40	40	40	35
Sludge	mL/L	-	110	76	70	74	96	55

The use of the coagulation process did not reduce the COD value to the limit required by the discharged wastewater contract with the municipal sewage system according to the Polish law. Consequently, the industrial plant would still have to pay an additional fee, however, it will be lower, due to the observed reduction of the COD.

3.2. Advanced oxidation process using the Fenton reaction

The results obtained in test series IV-VI are shown in Tables 4-6.

Series IV was conducted as an exploratory series. During the experiment, the colour of the sample changed (milky white colour after lowering the pH to 3.0, dark blue after adding $FeSO_4$, and red after adding H_2O_2). During a 15-minute reaction, a reduction in COD values in the range of 30.6-37.6% was obtained. In the next test series, the reagent doses and Fe^{+2}/H_2O_2 ratios were increased. The oxidation time was extended to 1 hour (series V) and 2 hours (series VI). Another factor whose influence was studied, was the method of alkalinisation. After oxidation, the samples sedimented for 30 minutes. The pH was then corrected in two ways: either with samples containing sludge or samples containing just decanted liquid.

Significantly increasing the doses of reagents as well as the reaction time resulted in the reduction of the COD values in the wastewater. In series V, the COD was reduced from 11200 mg O_2/L to values in the range 5520-7200 mg O_2/L (35.7-48.6%). However, the actual percentage reduction is much lower than that obtained by Sindhi (Sindhi *et al.*, 2014) for wastewater from the textile and pharmaceutical industries. The results of series V showed the lack of influence of reagent dose and oxidation time on treatment efficiency. The influence of the reaction time on COD reduction is evident in the samples which were alkalinised without sludge. In this case, the COD value is lower in all samples where the pollutants were oxidised for 2h (series VI). Increasing the oxidation time to 2 hours (series VI) and changing the amount of reagents resulted in a higher reduction of COD, 46.4-50.7% for samples in which decanted liquid was alkalinised. Rao (Rao, Shrivastava, 2020) demonstrated that in the treatment of complex recalcitrant wastewater, increasing the oxidation time during the Fenton process from 30 min to 150 min resulted in an increase in COD removal efficiency from 46.9% to 72.8%, but the greatest increase in reduction was seen between 30 and 60 minutes (from 46.9 to 66.7%). Cetinkaya (Cetinkaya *et al.*, 2018) investigated the effect of oxidation time from 10 to 180 min on COD removal efficiency from textile wastewater. There was a significant increase for the classic Fenton process up to 60 min. This result implies that the effect of time after 60 min did not change the COD removal significantly.

The Fenton process has some inherent disadvantages, which limit its application and adoption. One of them is the accumulation of ferric sludge that affects the oxidation efficiency (Xu *et al.*, 2020). Alkalinisation of the decanted liquid after the Fenton process, at certain doses, reduced the amount of sludge formed.

4. Conclusions

Coagulation was effective at removing suspended solids and colloids from plastic lens manufacturing wastewater, which are responsible for the clogging of the sewerage networkovergrowth in. This is the main problem faced by the manufacturing plant. As seen from the simultaneous removal of the largest amount of suspended solids and the greatest reduction in COD values, the coagulant dose of 0.8 mL/L proved to be the most effective. The COD reduction of the effluent after the Fenton process was slightly lower. However, increasing the reaction time and the reagent dose resulted in better removal of organic compounds. Further studies are needed to select the appropriate process parameters for the deepened wastewater treatment.

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