CEST 2023

18th International Conference on Environmental Science and Technology

Athens, Greece, 30 August to 2 September 2023

Settling and filtration experiments for the removal of flocs produced by a hybrid sono-electrochemical process

Posavcıc H.[1], Halkıjevıc I.[2], Vouk D.[3,*], Nakıc D.[4]

[1] Water Research Department, Faculty of Civil Engineering, University of Zagreb, Kaciceva 26, Zagreb, Croatia

*corresponding author:
e-mail: hana.posavcic@grad.unizg.hr

Abstract Choosing the right method for floc separation after electrochemical treatment seemed to be a problem in a number of previous research papers. Therefore, ultrasonic and electrochemical (sono-EC) processes in circulating flow mode were combined with settling and filtration to treat the oily wastewater and to remove the produced flocs. Experimental results showed that the inclined settling, which represented lamellar settling, was not recommended for the low-density flocs, as it did not accelerate the settling process. Overall settling time was 45 min with the final mineral oil concentrations up to 25 mg/l. On the other hand, mineral oil removal efficiency was about 57 % using the combination of settling and filtration. Achieved mineral oil concentrations (8.76 mg/l and 28.63 mg/l) were in accordance with the European standards for discharge into the sewerage system or direct discharge into the water body. It was recommended that all three filter pore sizes are used together to achieve the complete removal of mineral oil, turbidity and safety, as the filter with the smallest pores would often clog due to the floc residue in the treated wastewater.

Keywords: electrochemistry; filtration; flocs; settling

1. Introduction

Uncontrolled discharge or inadequate treatment of oily wastewater can lead to environmental pollution because it can contain high concentrations of hydrocarbons, oil, phenol, benzene, chemical oxygen demand (COD), etc. Therefore, various biological, physical, and chemical technologies have been developed to separate oil components from water (Perez et al., 2015, Posavcic et al., 2022, Safari et al., 2016, Tir et al., 2008).

In this research hybrid ultrasonic electrochemistry (sono-EC) in circulating mode is combined with settling and filtration to remove the produced flocs (sludge). The optimal conditions for circulating flow sono-EC have already been determined in a previous study (Posavcic, 2022), and are as follows: 8 minutes of treatment time, a current density of 53.1 A/m^2, and a flow rate of 0.23 L/s. Although this hybrid sono-EC treatment was proved to be very effective (more than 90 % of the mineral oil was removed), separation of the produced flocs from the treated wastewater was a problem because of their low density and slow settling. The flocs had low density because they contained mineral oil, and because the sono-EC time was relatively short (8 minutes of treatment time).

Even though the problem of separating flocs from electrochemically treated wastewater appeared in many of previous research papers, only a few of them addressed this issue, for example Hakizimana et al., 2017, Sahu et al., 2014, Załeska-Chróst and Wardzyńska, 2016, etc. This number is even lower when only oily wastewaters are considered (Asselin et al., 2008, Hui et al., 2014., Safari et al., 2016). There are several reasons for this, such as the different origin of the wastewater, the different material of the electrodes, but mainly the lack of interest in floc separation due to the focus on the development of the electrochemical process itself (selection of the optimal operating parameters, such as current density, voltage, treatment time, pH, etc.) (Manilal et al., 2020, Safari et al., 2016, Tir et al., 2008).

Therefore, this paper is focused on settling and filtration experiments as the final step of sono-EC.

2. Materials and methods

The oily wastewater samples were collected at oil and grease separators, and their initial and final characteristics are listed in Table 1. Mineral oil concentrations were measured using the NEXIS GC-2030 (from Shimadzu, Japan), while pH, conductivity and temperature were measured continuously using the HI-98194 Multiparameter Waterproof Meter (from Hanna Instruments, Romania).

Table 1. Initial and final (after combined sono-EC, settling and filtration) characteristics of wastewater

	Sample 1		**Sample 2**	
Parameter	Initial	Final	Initial	Final
pH	7.47	7.74	7.20	7.64
Temperature (°C)	20.12	23.7	20.4	22.6
Conductivity (μS/cm)	631	422	724	501
Mineral oil concentrations (mg/l)	455.42	28.63	404.11	8.76

3. Experiments and experimental results

3.1. Settling

Two glass graduated cylinders (2000 ml) were used for gravity settling. One was placed vertically (90°), the other at an angle of 45°. Each cylinder was filled with 1000 ml of sono-EC treated wastewater containing flocs and allowed to settle by gravity for 45 minutes. The results showed that the settling rate was approximately the same regardless of the angle, Figure 1. However, the flocs were less dispersed in the inclined cylinder. After 45 minutes of settling, the final mineral oil concentrations were 25.1 mg/l for vertical settling and 23.2 mg/l for inclined settling.

Figure 1. Vertical (left) and inclined (right) settling

3.2. Filtration

Three filters were used in filtration experiments. In each experiment, treated wastewater was filtered through three filters, from larger to smaller pores (50 µm, 20 µm and 5 µm). The 50 µm pore size filter was made of washable nylon mesh, the 20 µm pore size filter was a combination of polypropylene wool and activated carbon, and the 5 µm pore size filter had a sintered activated carbon cartridge (Figure 2). Two cases were observed, with and without prior settling. In the first case, filtration followed immediately after sono-EC treatment, with the treated wastewater that contained unsettled flocs. In the second case, the flocs were allowed to settle in a tank for 30 minutes, then the decanted wastewater (treated wastewater separated from the precipitate (flocs)) was filtered. In both cases, mineral oil concentrations were analyzed before and after sono-EC and after filtration. As expected, a lower removal efficiency was obtained when there was no settling before filtration. In this case, the mineral oil removal efficiency after sono-EC was 85.69 % (65.19 mg/l) and after filtration was 93.71 % (28.63 mg/l), Figure 3. Without settling, it was not possible to bind the remaining free oil to the flocs formed. Further, since the wastewater passing through the filters was not completely clear, some flocs remained on the filter. In the second case, when decanted wastewater was filtered, few residual flocs remained on the filters. This is also noticed in Figure 4. where that water sample was completely clear. The mineral oil concentration after sono-EC and settling was 21.27 mg/l (94.74 %), and in the effluent (after filtration) was 8.76 mg/l (97.83 %), Figure 3 and Table 1.

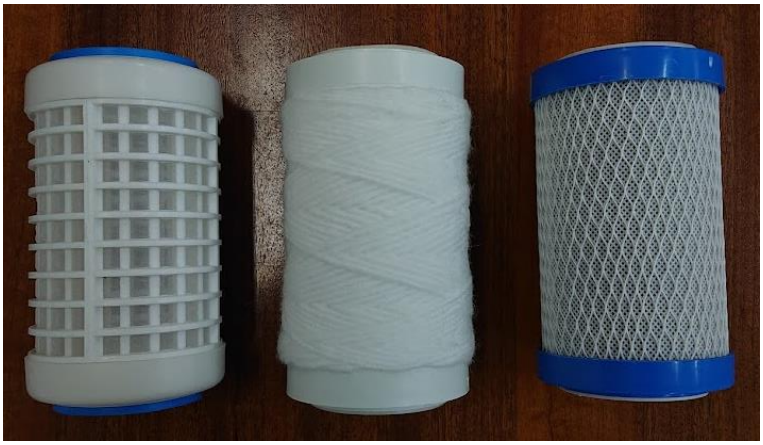

Figure 2. Filter pore sizes from left to right: 50 µm, 20 µm and 5 µm

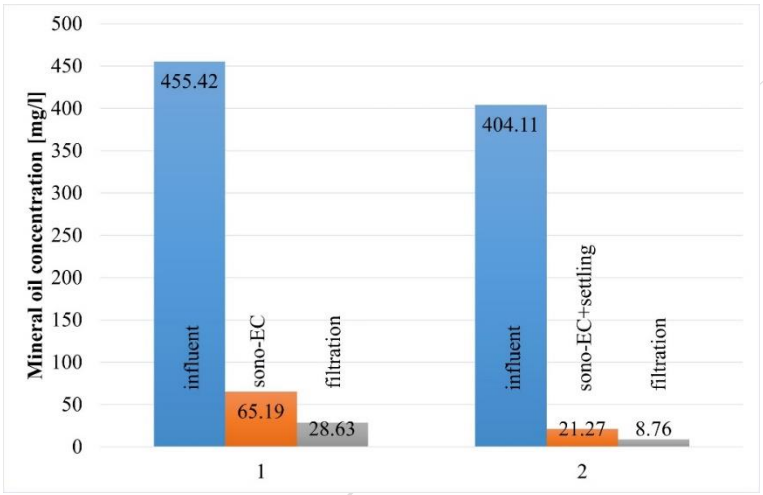

Figure 3. Mineral oil concentrations of samples 1 and 2

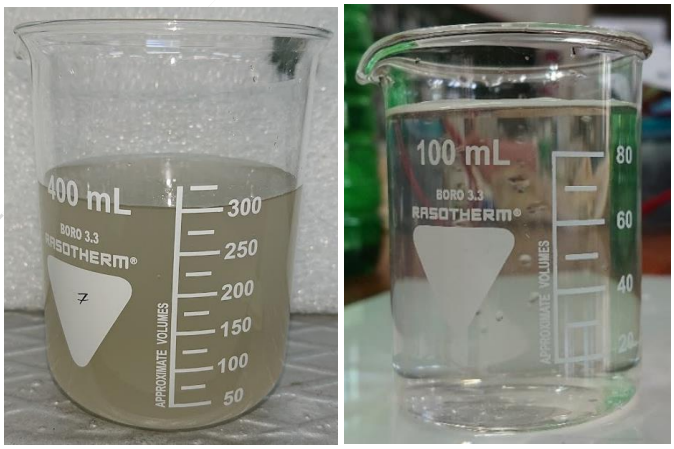

Figure 4. Water samples after filtration: without (left) and with (right) previous settling

4. Conclusion

Since previous research focused on developing the electrochemical process itself and lacked interest in separating the flocs from the water after the process, a space for additional research emerged.

Settling experiments showed that the inclined settling, which represented lamellar settling, is not recommended for the low-density flocs, as it does not accelerate the settling process. It is emphasized that there were several attempts to construct lamella clarifier with continuous flow, however laminar flow could not be ensured, flocs started to break, and swam out through the space between lamellas (4mm).

However, excellent results were obtained during filtration where the removal efficiency of the remaining mineral oil was about 57 % overall. According to the results (8.76 mg/l and 28.63 mg/l), the achieved mineral oil concentrations were in accordance with the European standards (30 mg/l of mineral oil for discharge into the sewerage system, and 10 mg/l for direct discharge into the water body). Additionally, each filter was tested separately (15.06 mg/l for 50 µm, 14.47 mg/l for 20 µm and 14.95 mg/l for 5 µm), however the highest mineral oil removal performance was still achieved when all three filters were used together. It is also recommended that all three filter pore sizes are used together to achieve complete removal of mineral oil, turbidity and safety, as the filter with the smallest pores can often clog due to the floc residue in the treated wastewater.

In conclusion, the combination of settling and filtration is recommended as a final step in EC and sono-EC processes.

References

Asselin, M., Drogui, P., Brar, S.K., Benmousa, H. ¸ & Blais, J.F. (2008) Organics removal in oily bilgewater by electrocoagulation process, *Journal of Hazardous Materials*, **151**, 446-455.

Hakizimana, J, Gourich, B., Chafi, M., Stiriba, Y., Vial, C., Drogui, P. & Naja, J. (2017) Electrocoagulation process in water treatment: A review of electrocoagulation modeling approaches, *Desalination*, **404**, 1-21.

Hui, L., Yan, W., Juan, W. & Zhongming, L. (2014) A Review: Recent Advances in Oily Wastewater Treatment, *Recent Innovations in Chemical Engineering*, **7**, 17-24.

Manilal, A., Soloman, P. & Basha, C.A. (2020) Removal of Oil and Grease from Produced Water Using Electrocoagulation, *Journal of Hazardous, Toxic, and Radioactive Waste*, **24**, 1-13.

Perez L.S., Rodriguez O.M., Reyna, S., Sanchez-Salas, J.L., Lozada, J.D, Quiroz, M.A. & Bandala, E.R. (2016) Oil refinery wastewater treatment using coupled electrocoagulation and fixed film biological processes, *Physics and Chemistry of the Earth*, **91**, 53-60.

Posavcic, H., Halkijevic, I., Vouk, D. & Cvetnic, M. (2022) Circulating flow hybrid ultrasonic and electrochemical process for the treatment of mineral oil wastewaters. *Journal of Water Process Engineering*, **49**, doi:10.1016/j.jwpe.2022.103024

Safari S., Azadi Aghdam M. & Kariminia H.R. (2016) Electrocoagulation for COD and diesel removal from oily wastewater. *Int J Environ Sci Technol*, **13**(1), 231–242.

Sahu, O., Mazumdar, B. & Chaudhari, P. (2014) Treatment of wastewater by electrocoagulation: A review, *Environmental Science and Pollution Research*, **21**, 2397-2413.

Tir M. & Moulai-Mostefa N. (2008) Optimization of oil removal from oily wastewater by electrocoagulation using response surface method, *J Hazard Mater*, **158**(1), 107–15.

Załeska-Chróst, B. & Wardzyńska, R. (2016) Analysis of sludge aggregates produced during electrocoagulation of model wastewater, *Water Science and Technology*, **73**, 161-166.