



Removal of Microplastics from Synthetic Wastewater via Sono-Electrocoagulation Process: Modeling and Optimization by Central Composite Design

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ABSTRACT

Wastewater treatment plants are an important pathway for microplastics (MPs) to enter the environment. In recent decades, hybrid treatment technologies such as sono-electrocoagulation have been used to treat various types of wastewater. This study aimed to remove polypropylene microplastics from synthetic wastewater by sono-electrocoagulation process using central composite design. The central composite design was utilized to investigate the relationship among four independent variables including the number of MPs (0.003-0.03 MPs/L), sodium sulfate concentration (180-9000 mol/L), voltage (1-15 V) and reaction time (20-180 min) on the efficiency of polypropylene microplastic. Design Expert 13 software and central composite design method were used to design and analyze the experiments and results. The optimum number of concentration of MPs, sodium sulfate concentration, voltage, and reaction time were found to be 6343.36 MPs/L, 0.0181924 mol/L, 10.0356 V, and 62.21 min, respectively. In optimal conditions, polypropylene removal was found to be %90.34. Central composite design proposed a quadratic model for this process. Adequacy of the model using lack of fit statistical tests values, p-values, and F-values was checked, yielding the values of were 1.76, 0.0001 <, 19.51, respectively. The R², R² adjusted, R² predicted values which were 0.9367, 0.8776, 0.6959, respectively. Considering the proper removal efficiency, the sono-electrocoagulation process can be used to remove microplastics.

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INTRODUCTION

Nowadays, plastics are inevitably used in people's daily lifestyles, which causes excessive release of plastic materials in the environment. Plastic materials due to the low production cost are used in many industries such as packaging, construction, automotive, etc (Alvim et al.,

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2020). The production of plastic products in the world reached 359 million tons in 2018 (Shen et al., 2021a). Microplastics (MPs) are defined as plastic particles less than 5 mm in size, that are found in both primary and secondary forms in the environment (Takdastan et al., 2021).

MPs are synthetic polymers with high stability that pose long-term risks as anthropogenic pollutant in the environment (Shen et al., 2021a, Zhang et al., 2022). In natural environments, MPs undergo aging processes (including physical, chemical, and biological changes) that cause significant changes in their physicochemical properties in the environment. For example, aged MPs exhibit altered hydrophilicity, surface charge, and oxygen-containing groups that affect their interactions with co-pollutants (Zeb et al., 2023). MPs in aquatic environments are like food for many aquatic organisms, easily ingested by aquatic predators. Since MPs cannot be broken down by an enzymatic system, ingestion of MPs by organisms is harmful and potentially fatal in itself (Liu et al., 2023). There is a plethora of evidence that humans are exposed to MPs through eating and drinking (Cox et al., 2019).

MPs can be a source of chemicals. MPs due to their small particle size and large specific surface area, are highly amenable to absorb other environmental pollutants such as hydrocarbons, polycyclic aromatic compounds, antibiotics, and heavy metals (Zhang and Chen, 2020, Zhang et al., 2022).

Research shows that wastewater treatment plants are possible sources of MPs pollution in the environment, which can an important role in releasing MPs into the environment (Kazour et al., 2019). However, at present there is no specific regulation for the removal of MPs by wastewater treatment plants. Therefore, taking the potential risks of MPs into account, investigating the methods of removing MPs by different wastewater treatment technologies will be useful to identify the best removal technology (Yang et al., 2019). So far, various MPs removal technologies such as filtration, coagulation and flocculation, absorption process, foam flotation and advanced oxidation processes have been used for wastewater treatment, with each method having its own advantages and disadvantages (Adib et al., 2022, Yahyanezhad et al., 2021, Liu et al., 2023).

Electrocoagulation is a cost-effective method used to treat all types of wastewater (Arka et al., 2022, David et al., 2015, Dai et al., 2022). This method which has been used to treat various wastewater pollutants is the most sustainable alternative for wastewater treatment due to its easy setup, the ability to treat a large amount of water without using chemicals, low sludge production, and compatibility with the environment (Tahreen et al., 2020, Moussa et al., 2017). The combination of electrocoagulation and ultrasonic processes can be more effective in removing various pollutants from wastewater (Moradi et al., 2021, Sadeghi et al., 2022, Afsharnia et al., 2018). The ultrasonic process increases the removal efficiency through the chemical and physical effects it creates. In physical effect, the cavitation bubbles generate intense shock waves that remove the insulating material from the electrode surface. The chemical effect of ultrasonic is related to the production of a large amount of oxidizing radicals (Moradi et al., 2021, Hassani et al., 2022). Therefore, the sono-electrocoagulation process causes the mass transfer rate to remain the same during the process, and consequently, energy consumption is reduced until the pollutants are completely destroyed. Sono-electrocoagulation increases the efficiency of removing pollutants, and the durability of the electrode, hence leading to a reduction of treatment costs (Prajapati, 2021, Mehralipour and Kermani, 2021). Shen et al. (2020) (Shen et al., 2022) reported polyethylene (93.2%), polymethylmethacrylate (91.7%), cellulose acetate (98.2%), and polypropylene (98.4%) removal at pH= 7.2 using electrocoagulation process. Considering the daily discharge of a large amount of MPs from wastewater treatment plants and lack of standards and legal restrictions regarding their discharge into the environment, it is indispensable to find an optimal removal method with high removal efficiency and low cost. Manikandan et al. (2023) (Manikandan and Saraswathi, 2023) removed reactive black-WNN dye from aqueous solutions by sono-electrocoagulation. Their results showed that it had

98.30% removal in optimized conditions (pH=6.6, current density=66.66 mA/cm², electrolysis time=25 min and ultrasonic power=100W). Moreover, sono-electrocoagulation had a more effective removal than electrocoagulation or sono alone.

To the best of authors' knowledge, there has not been a study about the sono-electrocoagulation process using for the treatment of MPs from the aquatic environment. Response surface methodology (RSM) has reduced the number of test runs, human error, and process optimization (Mehralipour et al., 2023, Mehralipour and Kermani, 2021, Khoramipour et al., 2021). As such, the present study will utilize the sono-electrocoagulation process for the removal of MPs from synthetic wastewater using the response surface method.

MATERIALS AND METHODOLOGY

Materials

In this study, polypropylene (PP) MPs were used. PP is the most widely used thermoplastic which is very popular due to having the lowest density among plastics and high resistance to temperature (Enfrin et al., 2019). PP granules were obtained from Maroon Petrochemical Company. Sulfuric acid (H₂SO₄), sodium sulfate salt (Na₂SO₄) and anionic surfactant sodium dodecyl benzene sulfonate (SDBS) were purchased from Merck company. In the current study, aluminum alloy 3105 was used as an anode and cathode.

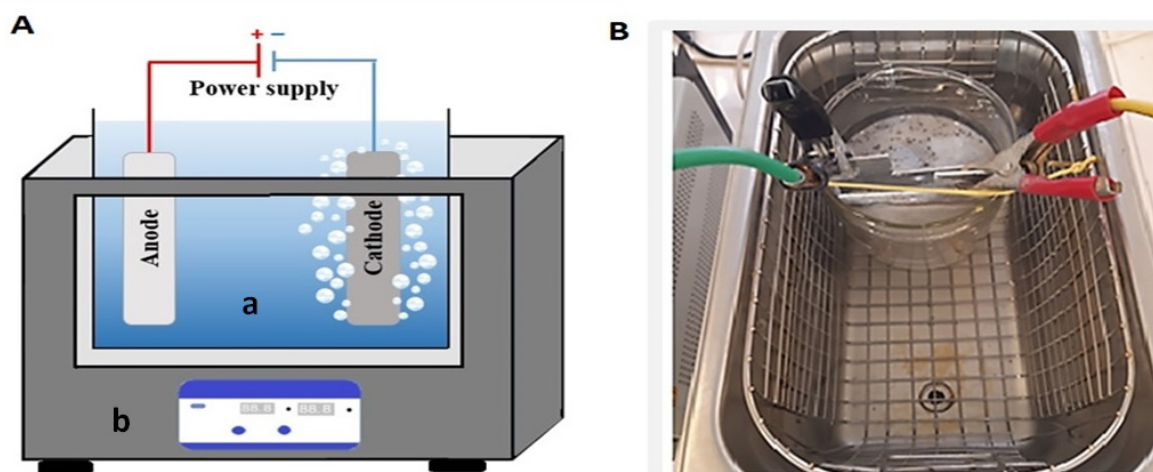
Experimental set-up

Synthetic wastewater was used in this study. To simulate real wastewater, PP materials were crushed by a ball mill (RETSCH-PM100) and passed through a standard sieve (TAK AZMA SD8-12) with a pore diameter of 300-425 μm. Number of MPs was obtained with an accurate balance (METTLER TOLEDO A8304-5). SDBS surfactant (20 mg/L) was added to the solution to disperse the MPs in water. Surfactant can help MPs form a uniform suspension and in addition, simulating the average concentration of surfactant in domestic wastewater (Shen et al., 2021b). For each experiment, 500 mL of uniform suspension of MPs was used. After preparation, the experiment was performed immediately. Aluminum electrode (5 cm × 7 cm × 1.25 mm) was used as an anode and cathode. It should be added that the distance between the electrodes was 1 cm. In the next stage, the cell electrocoagulation containing microplastic suspension was placed in ultrasonic cleaner (DSA-100W) and the electrodes were connected to the DC power source (Iran/Matrix mps-3005I) with a wire. During the test, the temperature of the tank of the ultrasonic bath was kept below 45°C. Based on the designed runs test, the time and voltage input to the pilot were adjusted. The schematic and experimental layout of the sono-electrocoagulation reactor is shown in Figure 1. After the experiment, the solution was stirred uniformly with a glass rod, and then the beaker was placed in a clean and closed place and settled for 16 h. All glassware was cleaned with ultrapure water before use. To remove the oxide layer formed during the experiment, the electrodes were placed in 1 M H₂SO₄ solution for 30 min after each run.

Determination of removal efficiency

After 16 h, MPs sank into the blanket of sludge formed at the bottom of the reactor and got trapped. After settling, the sludge was digested by sulfuric acid to separate the MPs trapped in the sludge from the aluminum flocs. Then, the digested solution from each sample was filtered by a 0.45 μm membrane and the filter was dried at 40°C for 24 h. The number of MPs on the dried filter was counted. Using Eq 1, MPs removal efficiency was calculated for each run as:

$$R = \frac{C_0 - C_t}{C_0} \times 100 \quad (1)$$



a: Cell electrocoagulation
b: Ultrasonic cleaner

Fig. 1. A) Schematic and B) experimental layout of the sono-electrocoagulation reactor

Table 1. Coded and actual values of numeric factors

Variables	unit	Symbol	Code variable level				
			-1	$-\alpha$	0	$+\alpha$	+1
Number of MPs	MPs/L	A	180	2385	4590	6795	9000
Na ₂ SO ₄ Concentration	mol/L	B	0.003	0.00975	0.0165	0.02325	0.03
Voltage	V	C	1	4.5	8	11.5	15
Reaction time	min	D	20	60	100	140	180

Where R is the removal efficiency of MPs; C_0 is the initial number of MPs in the solution (MPs/L); C_t is the number of MPs in the sludge (MPs/L).

Design of experiments

Recently, various experimental design methods have been used in the optimization of chemical and biochemical processes. RSM is a collection of mathematical and statistical techniques for designing experiments that are used to optimize and analyze the interaction effects of factors with a minimum number of experiments (Reji and Kumar, 2022). In this study response surface methodology and central composite design (CCD) were used for optimization. Four variables (number of MPs, electrolyte concentration, voltage density and reaction time) were selected at five levels ($-\alpha$, -1, 0, +1 and $+\alpha$). Table 1 shows the levels of independent variables in the design of the experiment. Based on the equation $N=2^k+2k+C_0$, where k and C_0 are the numbers of variables and the number of central points respectively, 30 experiments were designed for the sono-electrocoagulation process. Table 1 shows the design matrix and the range of parameters in the experiments.

RESULTS AND DISCUSSION

Development of model and validation

30 runs designed by CCD method were performed. Data analysis showed that the best model for interpreting the results of this study was the quadratic one. The results of the tests are presented in Table 2. As shown in the table, the highest and the lowest removal efficiency was

Table 2. The matrix of the experiments with CCD design and coded factor levels for PP removal

Run	Experimental design				Removal Efficiency (%)		
	Number of MPs (MPs/L)	Na ₂ SO ₄ Concentration (mol/L)	Voltage (V)	Reaction time (min)	Obtained	Predicted	Residual
1	2385	0.00975	4.5	140	81.57	79.65	1.92
2	4590	0.0165	8	100	89.95	85.75	4.20
3	4590	0.03	8	100	81.33	83.67	-2.34
4	4590	0.0165	8	100	85.4	85.75	-0.3500
5	6795	0.02325	4.5	140	87.68	88.81	-1.13
6	6795	0.02325	11.5	60	88.99	91.02	-2.03
7	4590	0.0165	15	100	70.7	68.77	1.93
8	4590	0.0165	8	100	84.8	85.75	-0.9500
9	4590	0.0165	1	100	68.94	71.02	-2.08
10	2385	0.02325	4.5	140	89.97	88.82	1.15
11	2385	0.02325	11.5	140	70.11	70.19	-0.0796
12	6795	0.00975	4.5	140	68.47	70.28	-1.81
13	2385	0.02325	4.5	60	80.64	78.23	2.41
14	4590	0.0165	8	100	86.14	85.75	0.3900
15	6795	0.00975	4.5	60	68.29	68.32	-0.0312
16	6795	0.02325	4.5	60	85.47	83.48	1.99
17	180	0.0165	8	100	72.43	75.64	-3.21
18	4590	0.003	8	100	70.36	68.17	2.19
19	4590	0.0165	8	100	83.93	85.75	-1.82
20	4590	0.0165	8	20	84.38	86.48	-2.10
21	2385	0.00975	11.5	60	76.13	75.11	1.02
22	2385	0.02325	11.5	60	74.16	72.09	2.07
23	6795	0.00975	11.5	140	71.66	74.18	-2.52
24	9000	0.0165	8	100	88.28	85.22	3.06
25	2385	0.00975	4.5	60	72.24	72.43	-0.1879
26	6795	0.02325	11.5	140	84.32	83.87	0.4471
27	2385	0.00975	11.5	140	68.11	69.84	-1.73
28	4590	0.0165	8	100	84.28	85.75	-1.47
29	6795	0.00975	11.5	60	83.81	84.70	-0.8896
30	4590	0.0165	8	180	88.5	86.55	1.95

found to be 89.97 and 68.11, respectively

Furthermore, the values of correlation coefficient (R^2), adjusted correlation coefficient (adj R^2) and predicted (Pred R^2) were found to be 0.9367, 0.8776 and 0.6959, respectively. The predicted R^2 was in reasonable agreement with the adjusted R^2 because the difference was less than 0.2. Adeq Precision measures the signal to noise ratio. A ratio greater than 4 was desirable. In this study, this ratio was found to be 11.9490, which indicates a sufficient signal. Additionally, the quadratic model with regression coefficients is presented in Eq 2.

$$\text{Removal Efficiency (\%)} = +28.90123 - 0.001054 * A + 2068.75717 * B + 6.75139 * C + 0.171776 * D + 0.157050 A * B + 0.000444 A * C - 0.000015 A * D - 93.43915 B * C + 3.12037 B * D - 0.022295 C * D - 2.73591 E^{-07} * A^2 - 53941.47234 * B^2 - 0.323588 * C^2 + 0.000119 * D^2 \quad (2)$$

Diagnostic plots based on residuals were used to ensure the adequacy of the proposed model. Figure 2-A shows the residual values versus the predicted response. According to this diagram, the distribution of the residuals was about zero, which confirms the assumption of

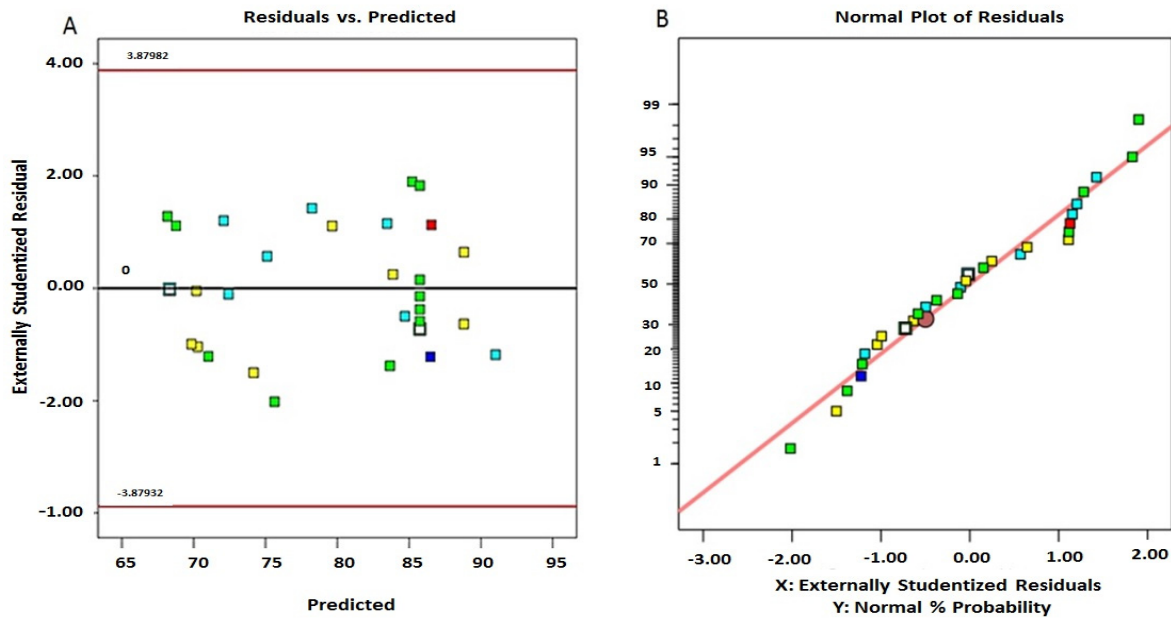


Fig. 2. ANOVA results of the PP removal modeling A) residuals vs. predicted, B) normal probability.

Table 3. ANOVA results for PP removal by sono-electrocoagulation process using response surface (for coded factor).

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	1624.32	14	116.02	15.86	< 0.0001	significant
A- number of MPs	137.57	1	137.57	18.80	0.0006	
B- Na ₂ SO ₄ Concentration	360.38	1	360.38	49.25	< 0.0001	
C-Voltage	7.62	1	7.62	1.04	0.3238	
D-Time	0.0067	1	0.0067	0.0009	0.9763	
AB	87.42	1	87.42	11.95	0.0035	
AC	187.55	1	187.55	25.63	0.0001	
AD	27.62	1	27.62	3.77	0.0711	
BC	77.97	1	77.97	10.66	0.0052	
BD	11.36	1	11.36	1.55	0.2319	
CD	155.88	1	155.88	21.30	0.0003	
A ²	48.53	1	48.53	6.63	0.0211	
B ²	165.68	1	165.68	22.64	0.0003	
C ²	430.98	1	430.98	58.90	< 0.0001	
D ²	1.00	1	1.00	0.1368	0.7166	
Residual	109.76	15	7.32			
Lack of Fit	85.47	10	8.55	1.76	0.2768	not significant
Pure Error	24.29	5	4.86			
Cor Total	1734.08	29				

$R^2=0.9367$, Adjusted $R^2=0.8776$, Predicted $R^2=0.6959$, Adeq Precision=11.9490

constant variance. Therefore, the proposed model can predict the removal of PP by the sono-electrocoagulation process. The normal probability plot showed the normal distribution of residuals (Figure 2-B).

Table 3. ANOVA analysis shows the results pertaining to the removal of MPs. As shown in Table 3, the F-value of the model indicates that the model was significant and there is only a

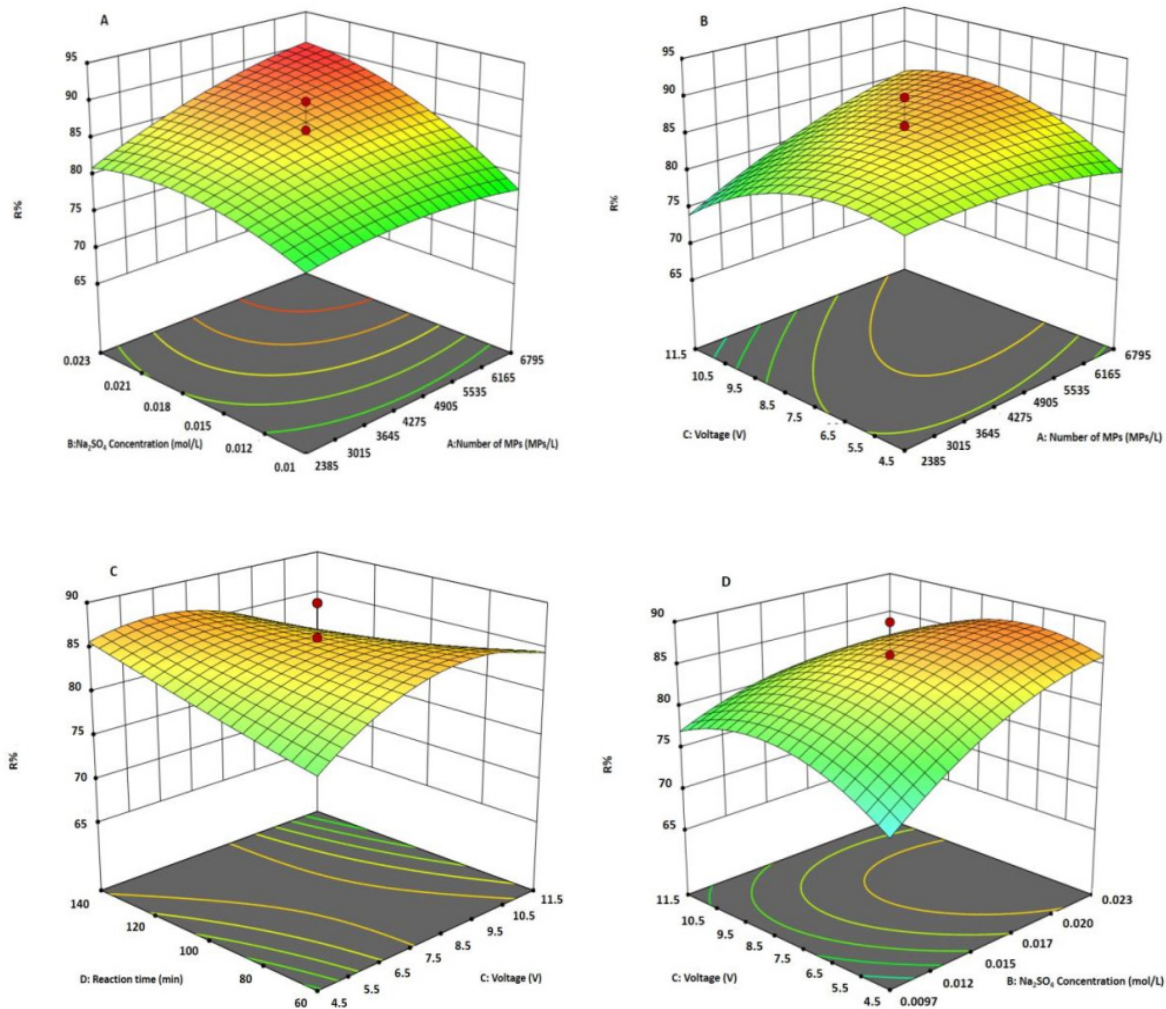


Fig. 3. Response surfaces plots for PP removal, A) number of MPs -electrolyte concentration, (B) number of MPs -voltage, C) voltage -reaction time, D) sodium sulfate salt and voltage.

0.01% chance that an F value of this magnitude was due to error. A p-value lower than 0.05 confirms the significance of the model.

The influence of variables and their interactions

Three-dimensional diagrams (Figure 3) were used to investigate the interactive effects between different parameters such as number of MPs, sodium sulfate salt concentration, voltage and reaction time on the removal of MPs by sono-electrocoagulation and determining their optimal values.

The Figure 3-A shows the interaction between number of MPs and sodium sulfate salt concentration on removal efficiency. At a constant voltage of 8 and reaction time of 100 min, with an increase in the number of MPs from 2385 to 6795 MPs/L and the concentration of sodium sulfate salt from 0.00975 to 0.026 mol/L, the removal efficiency increases from 78.38% to 92.34% and after that, it starts to decrease.

Figure 3-B showed the impact of number of MPs and voltage on PP removal efficiency. At a fixed value of sodium sulfate salt concentration of 0.0165 mol/L and a reaction time of 100 min, as the number of MPs increases from 1080 to 8625 MPs/L, the removal efficiency increases in turn and the voltage goes up from 4.5 to 10.50 V. After that, the removal efficiency decreases with the increase of voltage and the number of MPs.

As shown in Figure 3-C, with the increase of voltage from 4.5 to 7 V and the reaction time

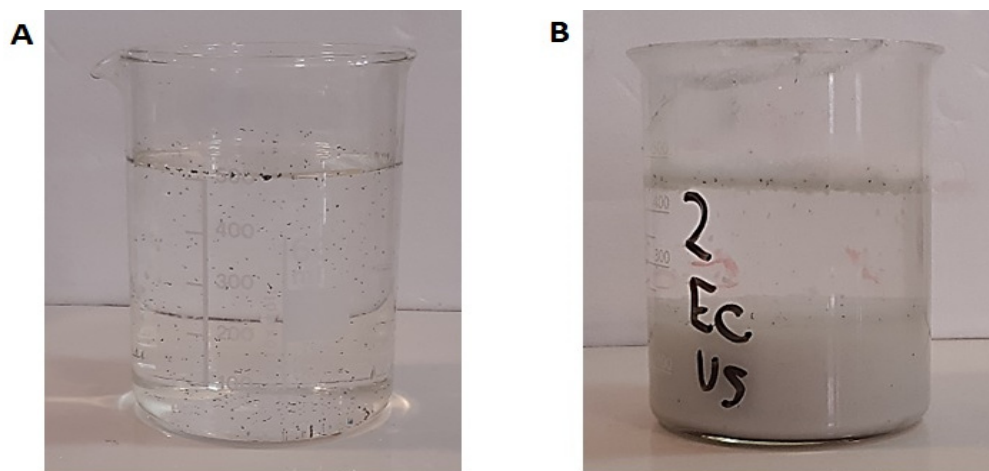


Fig. 4. A) Before and B) after the sono-electrocoagulation process.

from 60 to 120 min, the removal efficiency increases, and then it decreases with the increase of voltage from 8 to 11.5 V.

As shown in Figure 3-D, with an increase in voltage from 4.5 to 6.98 V and an increase in the Na_2SO_4 concentration from 0.00975 to 0.0227 mol/L, the efficiency has reached from 74% to 87.65%, then it has decreased with the increase in voltage.

An important factor in the removal of pollutants from wastewater with the sono-electrocoagulation process is their initial concentration (Moradi et al., 2021). Based on the results of other studies, due to differences in lifestyle and local conditions, the presence of MPs in wastewater has varied from tens to tens of thousands (Shen et al., 2022). Increasing the initial concentration of pollutants increases oxidation reactions. The reason for this decrease may be the lack of aluminum hydroxides in sufficient quantity for pollutant precipitation because by applying a certain voltage, almost a certain amount of ions is released in the solution. Hence, when the contaminant concentration increases, more coagulant is required to complete the sedimentation of the contaminant and trap it in the sludge (Sadeghi et al., 2022, Moradi et al., 2021). Asaithambi et al. showed that increasing the concentration of NaCl increases color removal and COD removal while further increase in NaCl concentration had no significant effect on removal efficiency (Asaithambi et al., 2017).

By the increase in voltage, the current density increases, and subsequently the amount of formed hydroxide complexes also increases; more flocs are formed, ultimately leading to an increase in the removal of MPs (Liu et al., 2023). However, when the voltage density exceeded its optimal value, the increase in voltage density led to a decrease in the removal efficiency of MPs, which might have been due to the rupture of clots due to gas bubbles, and the other one is the creation of additional coagulation, which leads to more sludge (Liu et al., 2023). Therefore, operating the process at the appropriate voltage intensity improves MPs removal efficiency and saves energy. The time to start producing excess sludge in the process depends on the reactor conditions, especially the flow density.

In the study by Shen et al, the efficiency of PP removal by the electrocoagulation process increases with increasing voltage. However, increasing the voltage from 10 to 15 volts during the reaction time of 4 hours does not lead to an increase in the removal efficiency (Shen et al., 2022). In addition, in the study by Afsharnia et al., COD and TSS removal efficiency was investigated by sono-electrocoagulation process. The results showed the efficiency increased with the increase in voltage, which is due to the increase in the rate of released coagulants (Afsharnia et al., 2018).

Moreover, the time of electrolysis is one of the effective factors in pollutant removal. As the

electrolysis time increases, it is followed by an increase in the pollution removal efficiency. However, once the optimum electrolysis time is reached, the pollutant removal efficiency remains constant and does not improve with increasing electrolysis time. Dissolution of the anode produces metal hydroxides. As the duration of electrolysis and constant current density increase, the number of metal hydroxides produced increases in turn. Increasing the formation of clots with longer duration electrolysis leads to an increase in the efficiency of pollutant removal. However, when the electrolysis time exceeds the optimal electrolysis time, the pollutant removal efficiency is not improved because there is a sufficient number of clots available to remove the pollutant (Boinpally et al., 2023). Dia et al. found that the removal of tetracycline using electrocoagulation increased with the increase in reaction time (Dai et al., 2022).

The reason for the increase in efficiency is the increase in ionic strength, followed by the trapping of MPs in the flock. Because increasing the concentration of metal cations or ionic strength decreases the stability of particles (Sharma et al., 2021). Asgharian et al. showed that by increasing the concentration of electrolyte up to 0.02 mM, the efficiency of pollutant removal increased while the removal efficiency decreased at higher concentrations (0.04 mM) due to the production of more oxygen and hydrogen bubbles (Asgharian et al., 2017).

FEATURES OF SLUDGE CAUSED BY SONO-ELECTROCOAGULATION PROCESS

Ultrasonic technique has attracted a lot of attention due to the advantages of not causing secondary pollution, high decomposition speed, and simple equipment in urban sludge treatment (Xu et al., 2019). The sludge produced in the electrocoagulation process contains various types of organic materials, metals, and non-metals. In processes where aluminum electrodes are used, there are aluminum complex compounds such as $Al(OH)]^{2+}$, $[Al(OH)_2]^+$, $[Al_2(OH)_2]^+$, $Al(OH)_3$ in the sludge (Aker et al., 2022). Based on Figure 4, after the sono-electrocoagulation process, sludge with a high percentage of solids is formed. Ultrasonic waves cause the particles and the environment to vibrate at the same time, as a result of which the collision and adhesion of the particles increases and the sludge has less water and less volume (Xu et al., 2019). Recent studies showed that the application of ultrasonic technology for sludge treatment increased the total solids content of the sludge by 16.2%, reduced the volume by 60.9%, while the viscosity of the sludge also decreased, which resulted in improved sludge filtration performance. As a result, it can be said that the ultrasonic process increases sludge dewatering (Ruiz-Hernando et al., 2013).

CONCLUSION

The purpose of this study was to investigate the efficiency of the sono-electrocoagulation process in removing PP MPs using RSM statistical analysis. The advantages of using the response surface methodology and the central composite design are achieving the best results with minimum tests, investigating the simultaneous interaction of two parameters on the response, drawing three-dimensional diagrams, and determining the presence or absence of a relationship between two variables. PP microplastics with a density of 0.88 float in water. Via using the sono-electrocoagulation process, MPs particles are removed by aluminum hydroxides and different mechanisms and subsequently settle in the sludge. The optimal amount of MPs is 6343.36 MPs/L, the concentration of sodium sulfate salt is 0.0181924 mol/L, the voltage is 10.0356 V and the time is 62.21 min; in this condition, the removal efficiency will be 90.3465%. The results showed that the sono-electrocoagulation process can be useful in removing PP microplastics.

According to the obtained results and the limitations of the current study, it is suggested that in future studies investigate the effect of different variables such as dissolved oxygen and

content of dissolved organic matter, power and frequency of the ultrasonic device, temperature, etc. in the removal of microplastics.

In order to realize the widespread application of sono-electrocoagulation process for the removal of microplastics, research and development for the use of sustainable energy can be taken as the future research direction. However, in a practical large-scale operation, the problems of high power consumption and plate passivation may limit the application of electrocoagulation. To overcome these shortcomings, the type of power supply can be improved, namely by the development of renewable energy sources such as solar or wind energy.

GRANT SUPPORT DETAILS

This research received financial support from Qom University of Medical Sciences (code of ethics IR.MUQ.REC.1401.165).

CONFLICT OF INTEREST

The authors declare that there is not any conflict of interests regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/ or falsification, double publication and/or submission, and redundancy has been completely observed by the authors.

LIFE SCIENCE REPORTING

No life science threat was practiced in this research.

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