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Sequential use of coagulation and adsorption methods for COD removal from soft drink industry wastewater

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Abstract: In this study, the effectiveness of sequentially applying coagulation and adsorption processes in treating soft drink industry wastewater was assessed based on COD removal. In the electrocoagulation method with iron electrodes, the highest COD removal occurred at 42%, achieved with a current of 9A and the natural pH of the wastewater at 5.51. In chemical coagulation, using $FeCl_3 \cdot 6H_2O$ as a coagulant, the highest removal rate of 23% was achieved at pH 5 with a coagulant dose of 2.5g/L. Activated carbon adsorption, in doses ranging from 10 to 40g/L, was applied to the effluents of both electrocoagulation and chemical coagulation at various contact times, up to 150 minutes, resulting in COD removal rates of 42% and 36%, respectively. According to the results, the COD removal efficiencies for the electrocoagulation-adsorption and chemical coagulation-adsorption systems were 66% and 51%, respectively. The findings of this study are important because they demonstrate the necessity of research on the use and development of physicochemical methods for the treatment of soft drink industry wastewater.

Introduction

The beverage industry faces a significant challenge due to excessive water consumption and the increasing demand for fresh water. Producing 1 liter of soft drink requires approximately 3-4 liters of clean water. Only 20-30% of this water remains in the product, while the rest turns into wastewater (Hsine et al. 2005, Salinas et al. 2019). Given the diversity of products in the sector beverage production involves numerous different substances (Hernandez et al. 2017). Process wastewater from this industry contains various sugars or artificial sweeteners, acid regulators, preservatives, flavoring and coloring substances, mineral salts, bicarbonates, and residues of fruit juice or vegetable extracts (Sheldon et al. 2016, Hernandez et al. 2017, Salinas et al. 2019). Additionally, it contains significant amounts of various chemicals used for cleaning purposes and oils used to lubricate mechanical parts such as production lines, equipment, and machines (Sheldon et al. 2016, Hernandez et al. 2017). Therefore, the wastewater from soft drink industry is considered to be highly resistant and poses an ecological threat (Sheldon et al. 2016, Remya and Swain 2019).

The most common wastewater treatment method used in the soft drink industry today is biological treatment (Hernandez et al. 2017, Zablocka et al. 2017, Remya and Swain 2019). However, in addition to the advantages of biological treatment systems, they are also known to be very sensitive to the composition of wastewater and can be time-consuming (Remya and Swain 2019, Kong et al. 2020). This sensitivity makes it challenging to treat the wastewater of the sector in question with biological treatment methods, considering the product groups, mixtures, and production programs. Additionally, in recent years, the sector's shift toward diet products with lower calorie and sugar contents–driven by public opinion and consumer pressure-has resulted in wastewater with insufficient organic matter content and unsuitable nitrogen-to-carbon ratios for effective biological treatment (Zablocka et al. 2017). While there is not much data on the treatment of these wastewaters in the literature, it is stated that oxidation processes, which are mostly effective in the removal of organic pollutants, can yield results (Hsine et al. 2005, Sheldon et al. 2016, Salinas et al. 2019). The literature also evaluates that processes adapted to the wastewater, compatible with each other as removal technologies, and applied in the correct order and manner can effectively treat these wastewaters (Hsine et al. 2005). At this juncture, electrocoagulation, which has been widely used in the treatment of industrial wastewaters containing resistant pollutants in recent years, is a treatment step whose potential for these wastewaters warrants investigation. Electrocoagulation can be described as an accelerated corrosion process, which is elucidated by the Pourbaix diagram in the literature (Casillas et al. 2007). The system operates based on initiation and conclusion of process involving species with potential coagulant properties from these corrosion products. During the electrolysis process, iron ions are released from the anode (Eqs. (1-2)) (Heidman and Calmano 2008).

$$Fe_{(s)} \rightarrow Fe_{(aq)}^{2+} + 2\acute{e} \qquad (1)$$

$$Fe_{(s)} \rightarrow Fe_{(aq)}^{3+} + 3\acute{e} \qquad (2)$$

In addition, electrolysis of water takes place simultaneously in the system, (Eqs. (3-4)), and O_2 is formed by the oxidation reaction and H_2 by the reduction reaction (Zongo et al. 2009).

$$H_2O \rightarrow 2H_{(aq)}^{+} + 1/2O_{2(g)} + 2\acute{e} \qquad E_0 = 1{,}229\ V/NHE \qquad (3)$$

$$2H_2O + 2\acute{e} \rightarrow H_{2(g)} + 2OH_{(aq)}^{-} \qquad E_0 = -0{,}828\ V/NHE \qquad (4)$$

Although there is no clear information about the dissolution of iron anodes, it is accepted that Fe^{2+} ions are mostly released during this process. In addition, Fe^{2+} can easily be oxidized to Fe^{3+} in the presence of oxygen (Eq. (5)), and the oxidation of Fe^{2+} at the anode should also be considered in this process (Eq. (6)). Furthermore, the dissolution of iron and the electrolysis of water are thought to compete, with the current and thus voltage being decisive factors in determining which reaction will proceed (Heidman and Calmano 2008, Zongo et al. 2009).

$$2Fe^{2+} + 1/2O_2 + H_2O \rightarrow 2Fe^{3+} + 2OH \qquad (5)$$

$$Fe^{2+} \rightarrow Fe^{3+} + \acute{e} \qquad (6)$$

The basic removal mechanism in electrocoagulation is built on Fe^{2+}/Fe^{3+} and hydroxyl ions released through oxidation and reduction reactions, respectively. Studies show that insoluble or low-solubility species are more effective at removing pollutants than soluble ionic iron hydroxides. Therefore, $Fe(OH)_{n(s)}$ (Eqs. (7–8)), which have very low solubility and remain as gelatinous suspensions in wastewater, are primarily responsible for pollutant removal (Ofir et al. 2007, Heidman and Calmano 2008, Zongo et al. 2009, Das et al. 2022). The efficiency of the process depends on the creation and maintenance of environmental conditions that lead to a certain saturation of wastewater with these complexes, in line the fundamental coagulation mechanism.

$$Fe_{(aq)}^{2+}\ 2OH^{-} \rightarrow +\ Fe(OH)_{2(s)} \qquad (7)$$

$$Fe_{(aq)}^{3+}\ 3OH^{-} \rightarrow +\ Fe(OH)_{3(s)} \qquad (8)$$

In this study, the effectiveness of primary physicochemical methods such as coagulation and adsorption in the treatment of wastewater from the soft drink industry was investigated based on the removal of Chemical Oxygen Demand (COD), which is one of the most important indicators of the organic pollutant load of wastewater. Due to its varying characteristics, complex structure, and fluctuating composition, this wastewater poses a significant environmental problem, especially given the market dynamics and production logic of the sector. Therefore, studies to identify alternative treatment stages and their potentials are gaining importance. Firstly, the effect of using electrocoagulation and chemical coagulation methods individually on COD removal from wastewater obtained from a soft drink facility was determined. Subsequently, the impact of sequential use of coagulation and adsorption methods on removal efficiency was examined and evaluated. The results obtained in the study are significant because they demonstrate the potential of the system l and addresses the lack of data in the literature regarding the sequential use of these processes for the mentioned wastewater.

Materials and methods

The wastewater used in the study was obtained from a facility producing soft drinks. Beverage production at the facility occurs in three stages: preparation of process water, preparation of soft drink syrup according to product characteristics, and dilution of the syrup with water before delivery to the filling line. The production process is completed by bottling the beverage on the filling line, capping the bottle, and labeling and packaging it. The characteristics of the process wastewater taken from the facility for use in the experiments, and preserved at 4°C in the laboratory, are given in Table 1.

Table 1. Characteristic properties of process wastewater.

Parameters	Units	Wastewater
COD	(mg/L)	5800
pH		5.51
Conductivity	(µS/cm)	1028
Turbidity	(NTU)	28.5
Temperature	(^{0}C)	20.3

In this study, COD analyses were carried out in accordance with the method titled 5220 C "Close Reflux, Titrimetric Method" given in the Standard Methods (Standard Methods 1995). COD is a crucial control criterion used for evaluating the effectiveness of treatment systems as well as the potential environmental effects of wastewater. It is also the main pollution parameter monitored in the wastewater of the relevant sector, according to the Water Pollution Control Regulation (Water Pollution Control Regulation 2004, Hu et al. 2022).

A laboratory study was conducted using equipment from various brands, including a VELP jar test unit (FC-6S), a Spectroquant thermoreactor (TR 620), a NÜVE shaker (SL 350), and a NÜVE centrifuge (NF 400). In the optimization studies, the pH was adjusted using 0.1 N H_2SO_4 and 0.1 N NaOH, while electrode cleaning was performed with an HCl solution.

Coagulation experiments

Wastewater treatment using the electrocoagulation method was carried out in a stainless steel electrocoagulator with a volume of approximately 3 L, at 6 different currents (2–11 amperes) and 9 different pH values (2–11). Iron plates with dimensions of 7.5 cm x 10 cm were used as electrode materials in the reactor. 2 mm thick plates were placed in the electrocoagulator with seven iron anodes and seven iron cathodes at 3 mm intervals.

The treatment of wastewater with chemical coagulation was carried out using a 6-piece conventional jar test setup with 600 mL volume beakers. In chemical coagulation, ferric chloride hexahydrate ($FeCl_3.6H_2O$), an iron-based coagulant effective over a wide pH range and widely used, was preferred was preferred to align with the electrocoagulation (Fe anode) study. $FeCl_3.6H_2O$ was applied to 300 mL wastewater samples in 10 different concentrations 0.4, 0.8, 1.2, 1.8, 2, 2.5, 2.8, 3, 3.2, and 3.5 g/L. In the jar test, based on the literature data optimizing coagulation conditions and preliminary studies, the samples were mixed rapidly at 150 rpm for 1 min. for

Table 2. Properties of the adsorbent.

Adsorbent	Average Particle Size (μm)	Surface Area (m²/g)	Average Pore Radius (Å)
Granular activated carbon	600	676.573	9.446

coagulation and slowly at 15 rpm for 30 min. for flocculation. Subsequently, the samples were kept at room temperature for 30 min to allow the flocs to settle (Ebeling et al. 2003, Ofir et al. 2007, Shak and Wu 20214, Ofir et al. 2007, Öztürk and Özcan 2021).

Adsorption experiments

The wastewater treatment by the adsorption method was carried out using effluents taken from the upper phase of the electrocoagulation and chemical coagulation processes after sedimentation. In the adsorption process, activated carbon is considered an effective adsorbent widely used for the removal of various organic and inorganic pollutants. Commercial granular activated carbon (Merck, Germany) was used as adsorbent in the study. The specific surface area and average pore size of granular activated carbon were measured by Quantachrome Quadrasorb SI BET surface area analyzer, based on the adsorption of nitrogen at 77 K using the BET method (Chang et al. 2010, El-Naas et al. 2010, GilPavas and Correa-Sanchez 2020). The surface area, average particle size, and pore radius of the granular activated carbon are given in Table 2.

Adsorption experiments were conducted with 50 mL effluent samples placed in 100 mL conical flasks. The experimental sets were designed based on adsorbent doses used in the studies conducted with real wastewater (El-Naas et al. 2010, GilPavas and Correa-Sanchez 2020). In each set of experiments where activated carbon was used at 4 different concentrations of 10, 20, 30, and 40 g/L (0.5-2g/50 mL), the samples were continuously mixed at 200 rpm using a mechanical shaker (Chang et al. 2010, Hu et al. 2022). Samples were taken at specific time intervals (0, 5, 20, 40, 60, 90, 120, and 150 min.) during the process between 0-150 min. and centrifuged at 4000 rpm for 10 min. to ensure phase separation.

Results and discussion

The treatability study of soft drink industry wastewater was carried out by monitoring COD removal through four experimental sets primarily based on coagulation and adsorption processes.

COD removal by electrocoagulation methods

In the electrocoagulation method, the process and removal efficiency depend on various factors. In this study, the electrocoagulation process was optimized based on current and pH. The variations in COD removal as a function of current and pH are shown in Figures 1 and 2, respectively.

Using a batch-operated experimental setup with Fe electrodes as the anode and cathode, the COD removal-current relationship was determined at current values of 2, 4, 6, 7, 9, and 11 amperes. The experiments, conducted at the original pH of the wastewater (5.51) and room temperature, showed that the efficiency varied between 26% and 42% for 6 different current values. The highest COD removal of 42% was achieved at a current of 9 ampere (Figure 1).

Following the current experiments, COD removal was monitored at the original pH of the wastewater and initial pH values of 2, 4, 5, 6, 7, 8, 9, and 11 to evaluate the effect of pH on the process. The 90-minute electrocoagulation results revealed that the efficiency varied between 21% and 42% across the 9 different pH values, with the highest COD removal occuring at the wastewater's original pH of 5.51 (Figure 2).

Examining the results of this study, which determined the conditions for the highest COD removal based on current and pH, it was found that a 20% removal efficiency could be achieved with an electrolysis time of 30 minutes, except for the lowest applied current value. Under the optimal current of 9 amperes, the COD removal efficiency varied between 13% and 42%. Specifically, COD removal, which was 20% in the first 30 minutes, reached 40% in 60 minutes. The lack of a significant increase in COD removal during the final 30 minutes suggests that the system had reached equilibrium. In the pH experiments conducted over a wide range, the highest removal efficiency was achieved at the natural pH of 5.51, while at other pH values, the COD removal efficiency remained below 40% after the 90-minute electrocoagulation process. The results show that the electrocoagulation method achieved its highest removal efficiency of 42% at a current of 9 amperes and pH of

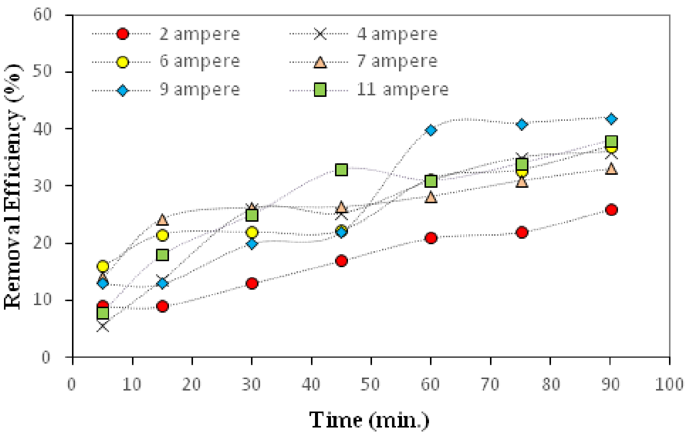

Figure 1. Effect of current (2-11 amperes) on COD removal efficiency.

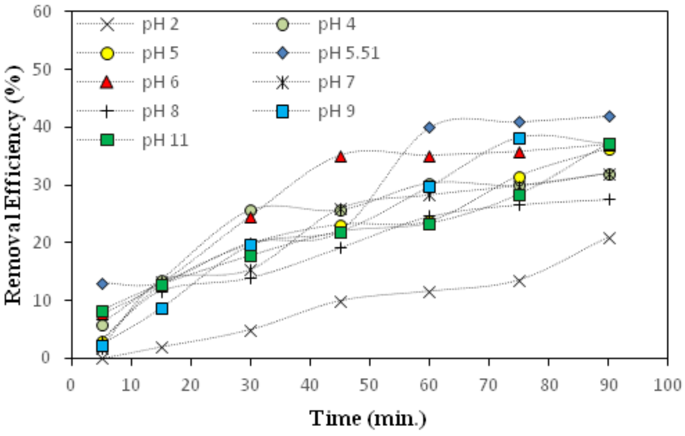

Figure 2. Effect of pH on COD removal efficiency.

5.51, reducing the effluent COD value to 3364 mg/L. Although the effect of this multicomponent structure of the wastewater in question on the treatment mechanism is not fully known, studies indicate that high concentrations of inorganic ions, such as sulphates, phosphates provide severe chemical stability to wastewater and limit the effectiveness of the applied treatment methods (Hernandez et al. 2017, Salinas et al. 2019).

The removal of COD from wastewater using the electrocoagulation method has shown varying efficiencies under different treatment conditions due to the complex nature of wastewater and the dynamic physicochemical changes occurring within the treatment cell (Ofir et al. 2007, Öztürk and Özcan 2021, Das et al. 2022). Hernandez et al. (2017) investigated the treatment of soft drink industry wastewater using a combined electrocoagulation-electrooxidation system, achieving 37.67% and 20% removal of COD and TOC, respectively, with electrocoagulation using Cu electrodes at pH 8. They further noted that the efficiency increased to 85% for COD and 75% for TOC when electrooxidation was applied subsequently (Hernandez et al. 2017). In another study by Tsioptsias et al. (2015), investigating the post-treatment of molasses wastewater by electrocoagulation COD removal rates between 10 and 54% were reported using copper and iron electrodes in various configurations (Tsioptsias et al. 2015). In a study conducted by Can (2014) on COD removal from wastewater from fruit juice production using electrochemical processes, efficiencies of 59.1% and 61.3% were achieved with aluminum electrodes at pH 5.5 and iron electrodes at pH 7.8, respectively (Can 2014). In the same study, COD removal rates of 64.7% and 84.4% were achieved through electrooxidation and electro-Fenton processes, respectively. Dia et al. (2018) investigated the pre-treatment of landfill leachate using the electrocoagulation process with aluminum anodes, achieving a COD removal efficiency of 37% (Dia et al. 2018). Inan et al. (2004) studied the treatment of olive oil wastewater via electrocoagulation, obtaining COD removal efficiencies of 42% and 52% with iron and aluminum electrodes at pH 6.2, respectively. The study also reported color removal efficiencies ranging between 90% and 97% (Inan 2004).

COD removal by chemical coagulation methods

Chemical coagulation efficiency is known to be sensitive to various treatment conditions, with pH and coagulant dose considered key factors (Hu et al. 2022). In this study, the process effectiveness was determined by examining COD removal across pH values in the range of 2-11 and coagulant doses in the range of 0.4-3.5 g/L. The variations in efficiency based on pH and coagulant dose are shown in Figures 3 and 4, respectively.

In the chemical coagulation study conducted using $FeCl_3 \cdot 6H_2O$ in the jar test apparatus, the relationship between COD removal and pH was investigated for 10 different pH values of wastewater: 2, 2.69, 4, 5, 5.51, 6, 7, 8, 9, and 11. In experiments with a fixed coagulant dose of 2.5 g/L, the highest removal efficiency was obtained at pH 5 with 23%, and the COD removal varied between 4% and 23% for the given pH values (Figure 3).

This process begins with the rapid dissolution of $FeCl_3$, which is used as a ferric salt, in water (Eq. (16)) and continues with reactions involving various types of hydrolysis (Sincero and Sincere 2003).

$$FeCl_3 \cdot \rightarrow Fe^{3+} + 3Cl^- \qquad (16)$$

The amount of complex ions formed by these reactions, in which OH^- ions participate, and the binding of OH^- ions to metal complexes are controlled by pH or the OH^- concentration (Sincero and Sincere 2003). Under acidic conditions, Fe^{3+} ions tend to transform into multiple positively charged polynuclear cations by the reaction given in Eq. (17) (Ishak et al. 2018).

$$xFe^{3+} + yH_2O \cdot \rightarrow Fe_x(OH)_y^{(3x-y)} + yH^+ \qquad (17)$$

In the treatment process, the primary condition for successful coagulation, and ultimately for maximum sedimentation, is the creation of conditions in which a large portion of the ferric ions will be converted to solid $Fe(OH)_{3(s)}$. This mostly corresponds to the optimum pH (Sincero and Sincere 2003). There are many studies on the optimum conditions that provide the highest COD removal from wastewater. In these studies, the optimum pH conditions varied considerably, ranging from acidic to basic. It is accepted that this situation depends on the component matrix affecting the suspended solids load in wastewater (Ishak et al. 2018).

To determine the effect of the amount of coagulant on efficiency, COD removal at $FeCl_3 \cdot 6H_2O$ concentrations of 0.4, 0.8, 1.2, 1.8, 2, 2.5, 2.8, 3, 3.2, and 3.5 g/L was investigated. In the study, where the initial pH of the wastewater was adjusted to 5, it was determined that the COD removal efficiency in the

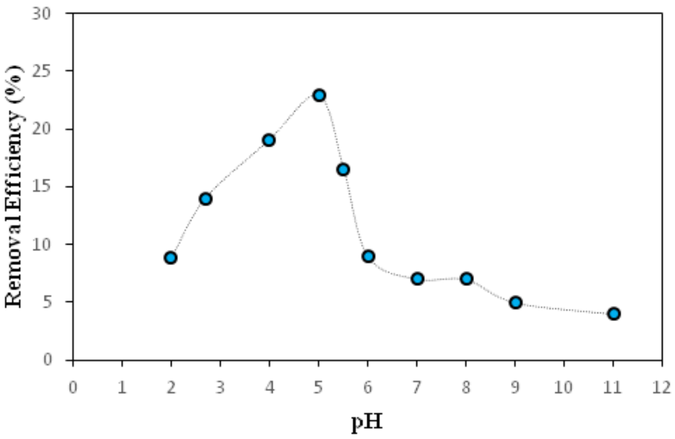

Figure 3. Effect of pH on COD removal efficiency.

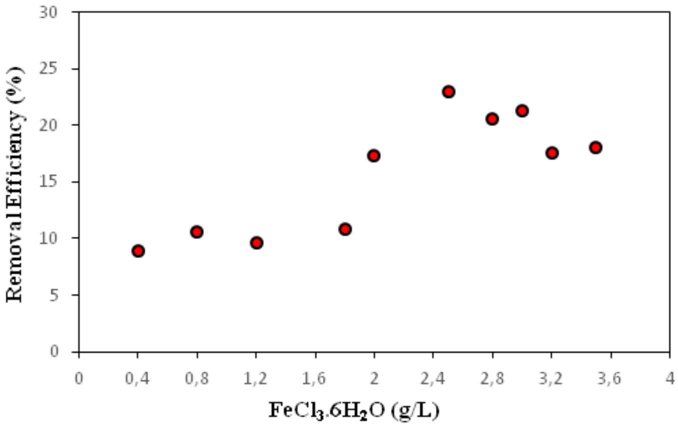

Figure 4. Effect of coagulant amount on COD removal efficiency.

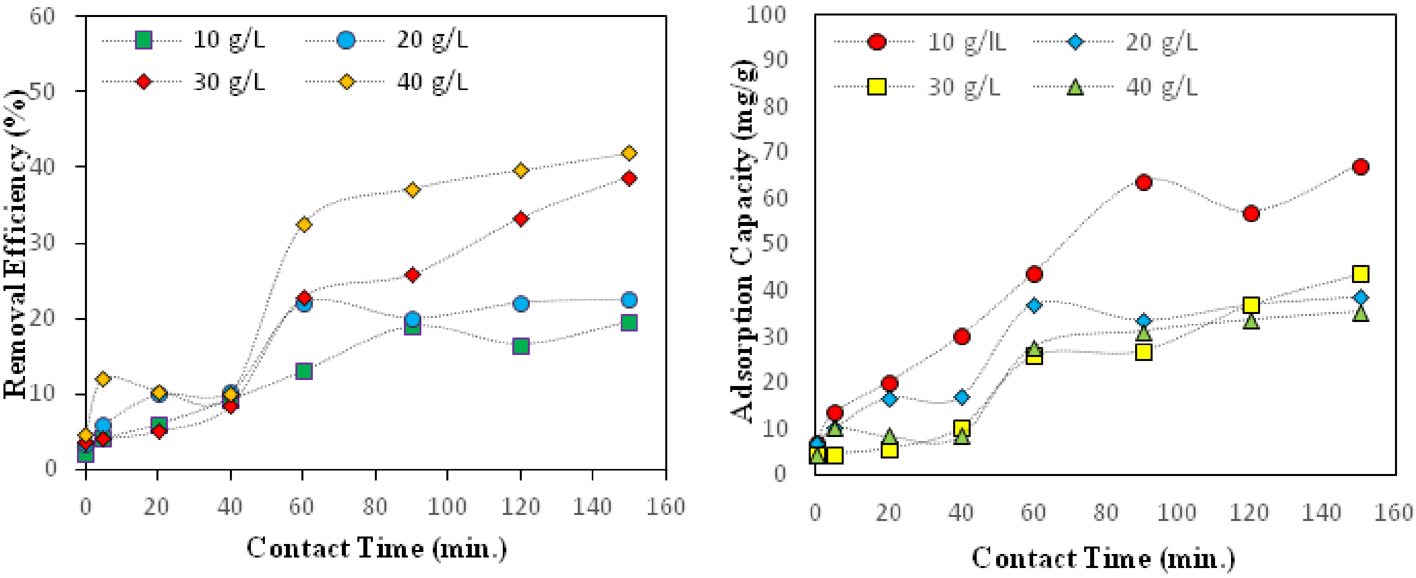

Figure 5 a-b. Effect of adsorbent dose on COD removal efficiency **(a).** Adsorption capacity of activated carbon **(b).**

system varied in the 9–23% range for the 10 different coagulant amounts applied, with the most appropriate coagulant dose used being 2.5 g/L (Figure 4).

The zeta potential can be adjusted to an appropriate range by adding different amounts of coagulant for the basic mechanism, which is based on the separation of colloidal pollutants from wastewater flow as solid sediment by converting them into large flocs (Hu et al. 2022). Colloidal particles in wastewater are generally negatively charged at pH 5–9, and by adding an appropriate amount of $FeCl_3$, the optimum concentration of Fe^{3+}cations needed to neutralize the system can be provided. However, the excessive addition of $FeCl_3$ triggers restabilization by reversing the particle surface charge (Ishak et al. 2018, Hu et al. 2022).

At this stage of the study, it was observed that a maximum of 23% COD removal could be achieved with chemical coagulation in the wastewater in question, reducing the effluent COD value to 4466 mg/L. The results obtained with both coagulation methods are important because they show the inadequacy of coagulation methods alone for the treatment of these wastewaters. This highlights the necessity of using two or more processes together in the treatment of high-resistance food industry wastewaters, such as those from the soft drink industry, in parallel with the literature (Tsioptsias et al. 2015, Hernandez et al. 2017, Dia et al. 2018).

COD removal by the sequential use of coagulation and adsorption

In this study, the effectiveness of the adsorption process, which has attracted great attention in recent years, particularly in the removal of resistant pollutants, was investigated in the treatment of electrocoagulation and chemical coagulation process effluents. The adsorption study, using 4 different activated carbon doses varying between 10 g/L and 40 g/L, was carried out at room temperature and at a stirring speed of 200 rpm. In the treatment of electrocoagulation and chemical coagulation effluents by the adsorption method, the change in COD removal efficiency and the adsorption capacity depending on the amount of activated carbon are shown in Figures 5 (a)-(b) and 6 (a)-(b), respectively.

Adsorption studies were conducted by applying 4 different doses of activated carbon -10, 20, 30, and 40 g/L - to the effluents after coagulation, with 8 samples taken at certain intervals during the contact time of 0-150 minutes for each experimental set. When the results of the adsorption experiments conducted under the same conditions were examined, it was determined that the COD removal rate varied between 20% and 42% in the electrocoagulation effluent and between 19% and 36% in the chemical coagulation effluent. The adsorption capacities of the activated carbons were between 67 mg/g and 35 mg/g for electrocoagulation and between 85 mg/g and 40 mg/g for chemical coagulation. In this study, it was calculated that for the 40 g/L adsorbent dose, where the highest removal efficiency was obtained by adsorption, the COD value decreased to approximately 1951 mg/L with a 42% removal efficiency in the electrocoagulation effluents and to approximately 2858 mg/L with a 36% removal efficiency in the chemical coagulation effluents.

In activated carbon adsorption, factors that affect the interaction of pollutants with activated carbon, and thus their transfer to the solid surface, determine the process efficiency. Particularly in adsorption, which is a surface process, the amount of activated carbon is a parameter that directly affects the removal efficiency. Increasing the amount of activated carbon in the process generally increases the surface area available for COD adsorption, thereby enhancing the removal efficiency (Hu et al. 2022, Kuśmierek et al. 2023).

Within the scope of the study, the total COD removal efficiencies of the electrocoagulation-adsorption (5800 mg/L-1951 mg/L) and chemical coagulation-adsorption processes (5800 mg/L-2858 mg/L) were determined to be approximately 66% and 51%, respectively, based on the COD values at the inlet and outlet of the sequential systems. In the literature, various studies have investigated and reported different results regarding the combined use of coagulation and adsorption processes. In a study by Hu et al. (2022) investigating the COD removal of wastewater from hydrothermal carbonization of food waste by combined coagulation and activated carbon adsorption, it was reported that the optimum doses for polyaluminum ferricsulfate (PAFS), polyacrylamide (PAM),

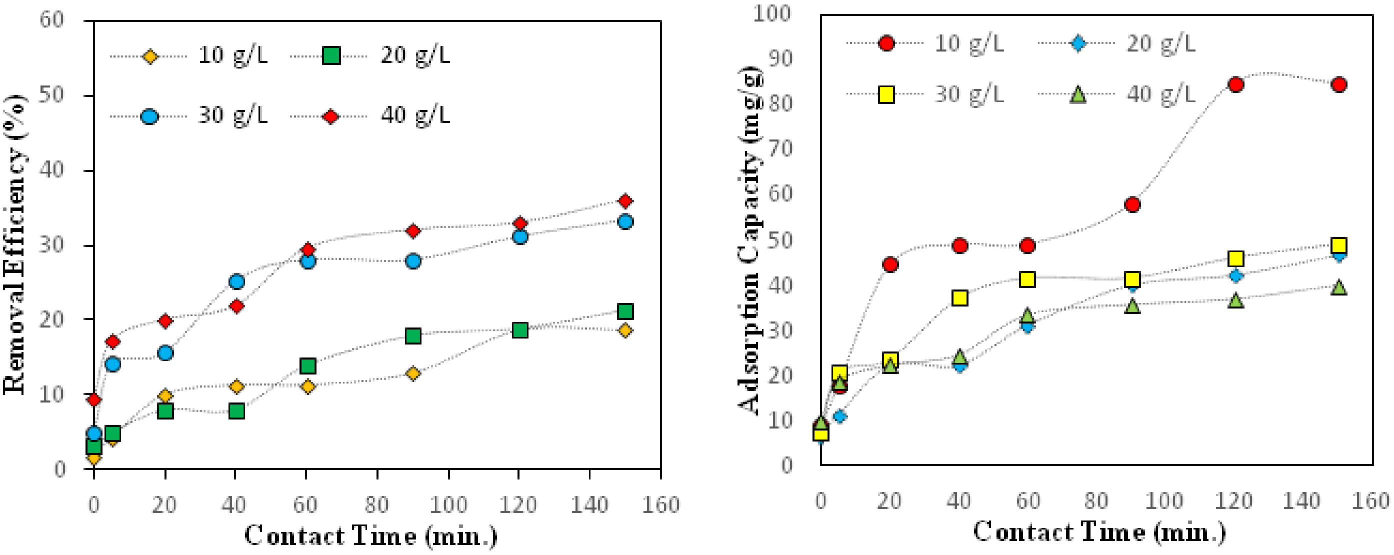

Figure 6 a-b. Effect of adsorbent dose on COD removal efficiency **(a)**. Adsorption capacity of activated carbon **(b)**.

and activated carbon (AC) were 6 g/L, 10 mg/L, and 30 g/L, respectively, achieving 68.41% COD removal (Hu et al. 2022). Chang et al. (2010) studied the removal of Reactive Black 5 from synthetic wastewater using combined electrocoagulation-granular activated carbon adsorption and reported that 39% COD removal with Fe electrodes during electrocoagulation. In a subsequent phase of the study, they found that the percentage of residual COD (COD/COD_0) decreased from 61% to 7% with an optimum granular activated carbon dose of 20 g/L and 4 hours of adsorption time (Chang et al. 2010). Literature data and the results obtained in this study show that the sequential use of these processes can also be developed as an option for soft drink wastewater.

Conclusion

The soft drink industry poses serious environmental risks worldwide due to its water use and wastewater management practices. A critical issue in addressing these risks is the industry's variable, multi-component, and resistant wastewater. This situation becomes even more important in today's conditions, depending on the nature of the process and consumer trends. In particular, variations in wastewater composition and fluctuations pose challenges to biological treatment applications, highlighting the importance and necessity of researching physicochemical methods for the treatment of these wastewaters. In the study, the effectiveness of sequential physico-chemical systems employing coagulation and adsorption methods was investigated for treating soft drink industry wastewater. Initially, experiments optimizing electrocoagulation and chemical coagulation conditions revealed maximum COD removal efficiencies of 42% and 23%, respectively. The results obtained clearly demonstrated that coagulation methods alone are insufficient for complete wastewater treatment but can serve as effective pre-treatment steps. For this reason, in this study, adsorption method was employed, which is effective in the removal of dissolved organic and inorganic pollutants. In continuation of the study, 42% COD removal in electrocoagulation effluent and 36% COD removal in chemical coagulation effluent were achieved via sequential activated carbon adsorption. When the obtained results were evaluated, it was determined that the COD removal efficiencies from soft drink industry wastewater with sequential systems were 66% for electrocoagulation-adsorption and 51% for chemical coagulation-adsorption. Accordingly, the effluent COD values decreased to 1951 mg/L and 2858 mg/L, respectively. Based on these findings, further optimization of the adsorption process could potentially enhance removal efficiencies. Additionally, it is believed that placing an additional treatment step between coagulation and adsorption processes, through detailed studies, may reveal recycling opportunities for these challenging wastewaters.

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