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Post-Treatment of Real Textile Wastewater by Using a Hybrid System Comprising of Electrocoagulation and Fenton-like Oxidation in the Presence of Perovskite/Activated Carbon Composite Catalyst

Özlem BULCA , Burcu PALAS , Süheyda ATALAY , Gülin ERSÖZ*

[1]*Ege University, Faculty of Engineering, Department of Chemical Engineering, İzmir, Turkey*

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Anahtar Kelimeler

Gerçek Tekstil Atıksuları,
Elektrokoagülasyon,
Fenton Benzeri Oksidasyonu,
Ardışık Atıksu Arıtımı,
Perovskit,
Biyokütle Türevli Aktif Karbon

Graphical/Tabular Abstract (Grafik Özet)

Electrocoagulation and Fenton-like oxidation were applied sequentially to improve the quality and reusability of the treated textile wastewater. / Arıtılan tekstil atıksuyunun kalitesini ve tekrar kullanılabilirliğini artırmak için elektrokoagülasyon ve Fenton benzeri oksidasyonu ardışık olarak uygulanmıştır.

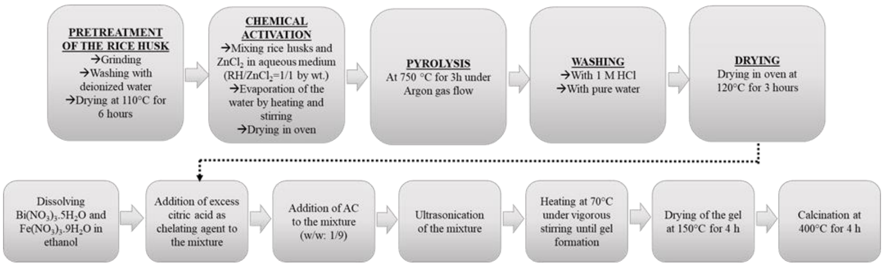

***Figure A**: Catalyst preparation steps /**Şekil A:**.Katalizör hazırlama basamakları*

Highlights (Önemli noktalar)

- *Real textile wastewater was subjected to sequential electrocoagulation and Fenton-like oxidation. / Gerçek tekstil atıksuyu ardışık elektrokoagülasyon ve Fenton benzeri oksidasyonuna tabi tutulmuştur.*
- *Perovskite/rice husk based activated carbon composite catalysts were used in the Fenton-like oxidation process. / Fenton benzeri oksidasyonu prosesinde perovskit/pirinç kabuğu bazlı aktif karbon kompozit katalizörler kullanılmıştır.*
- *The optimum Fenton-like oxdation conditions were determined as 1.5 g/L catalyst loading, pH 7, and 2 mM H_2O_2 concentration. / Optimum Fenton benzeri oksidasyonu koşulları 1,5 g/L katalizör yüklemesi, pH 7 ve 2 mM H_2O_2 konsantrasyonu olarak belirlenmiştir.*
- *In the presence of $BiNiO_3$/RHAC catalyst 32.7% TOC removal was achieved at the optimum reaction conditions. / $BiNiO_3$/RHAC katalizör varlığında optimum reaksiyon koşullarında %32,7 TOK giderimi sağlanmıştır.*

Aim (Amaç): *The aim of this study is the use of biomass-based environmentally friendly catalysts and the development of effective hybrid wastewater treatment systems to increase the quality and reusability of real textile wastewater. / Bu çalışmanın amacı gerçek tekstil atıksuların kalitesini ve tekrar kullanılabilirliğini arttırmak için biyokütle temelli çavre dostu katalizörlerin kullanılması ve ektili hibrit atıksu arıtım sitemlerinin geliştirilmesidir.*

Orginality (Özgünlük): *The performances of $BiFeO_3$/RHAC and $BiNiO_3$/RHAC catalysts were compared in Fenton-like oxidation applied subsequent to electrocoagulation and the most suitable reaction conditions were determined. / Elektrokoagülasyon sonrası uygulanan Fenton benzeri oksidasyonunda $BiFeO_3$/RHAC ve $BiNiO_3$/RHAC katalizörlerin performansları karşılaştırılmış ve en uygun reaksiyon koşulları belirlenmiştir.*

Results (Bulgular): *17% and 15.7% total organic removal efficiencies were achieved in electrocoagulation and Fenton-like oxidation processes, respectively, whereas the cumulative removal efficiency reached to 32.7%. / Elektrokoagülasyon ve Fenton benzeri oksidasyonu proseslerinde sırasıyla %17 ve %15,7 toplam organik giderim verimleri elde edilirken kümülatif giderim verimi %32,7'ye ulaşmıştır.*

Conclusion (Sonuç): *Perovskite/activated carbon composite materials have been effective in the treatment of industrial wastewater by using hybrid treatment technologies. / Perovskite/aktif karbon kompozit malzemeler, hibrit arıtma teknolojileri kullanılarak endüstriyel atıksuların arıtılmasında etkili olmuştur.*

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*Corresponding author, e-mail: gulin.ersoz@ege.edu.tr

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Post-Treatment of Real Textile Wastewater by Using a Hybrid System Comprising of Electrocoagulation and Fenton-like Oxidation in the Presence of Perovskite/Activated Carbon Composite Catalyst

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Abstract

A hybrid wastewater treatment method was used to improve the quality of the treated wastewater for possible reuses. Real textile wastewater was subjected to electrocoagulation and Fenton-like oxidation sequentially. In the Fenton-like oxidation process the performances of $BiFeO_3$/RHAC and $BiNiO_3$/RHAC (RHAC: Rice husk based activated carbon) catalysts were compared. $BiNiO_3$/RHAC was determined to be more effective in total organic carbon removal. A parametric study was carried out in the presence of the selected $BiNiO_3$/RHAC catalyst to investigate the effect of the catalyst loading, pH, and the oxidant dosage on total organic carbon removal. The most suitable reaction conditions were determined as 1.5 g/L of catalyst loading, pH 7, and 2 mM initial hydrogen peroxide concentration. 17% and 15.7% total organic removal efficiencies were achieved in electrocoagulation and Fenton-like oxidation processes, respectively, whereas the cumulative removal efficiency was 32.7%.

Gerçek Tekstil Atıksularının Perovskite/Aktif Karbon Kompozit Katalizör Varlığında Elektrokoagülasyon ve Fenton Benzeri Oksidasyondan Oluşan Hibrit Sistem Kullanılarak Son Arıtımı

Makale Bilgisi

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Biyokütle Türevli Aktif Karbon

Öz

Arıtılan atıksuyun kalitesini olası bir yeniden kullanım için iyileştirmek amacıyla bir hibrit atıksu arıtım yöntemi kullanılmıştır. Gerçek tekstil atıksuyu sırasıyla elektrokoagülasyon ve Fenton benzeri oksidasyonuna tabi tutulmuştur. Fenton benzeri oksidasyonu prosesinde $BiFeO_3$/RHAC ve $BiNiO_3$/RHAC (RHAC: Pirinç kabuğundan elde edilen aktif karbon) katalizörlerin performansları karşılaştırılmıştır. $BiNiO_3$/RHAC'nin toplam organik karbon gideriminde daha etkili olduğu belirlenmiştir. Katalizör yüklemesin, pH ve oksidan dozajının toplam organik karbon giderimi üzerine etkisini araştırmak için seçilen $BiNiO_3$/RHAC katalizör varlığında parametrik çalışma gerçekleştirilmiştir. En uygun reaksiyon koşulları 1,5 g/L katalizör yüklemesi, pH 7 ve 2 mM başlangıç hidrojen peroksit konsantrasyonu olarak belirlenmiştir. Elektrokoagülasyon ve Fenton benzeri oksidasyonu proseslerinde sırasıyla %17 ve %15,7 toplam organik giderim verimleri elde edilirken kümülatif giderim verimliliği %32,7'dir.

1. INTRODUCTION (GİRİŞ)

Water pollution is one of the major global challenges and environmental problems of the world. In recent years, minimization and reuse of water have gained importance and popularity due to decline in the clean water sources and increasing water pollution. Textile industry wastewater is one of the most significant contributor of water pollution and approximately 17–20% of industrial water contamination arises from textile processing [1,2]. Textile industry effluents contain many complex chemicals affecting the aquatic organisms living in receiving water sources due to the large amount of chemical consumption in scouring, bleaching, mercerizing, warping, spinning, and dyeing processes.

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*Corresponding author, e-mail: gulin.ersoz@ege.edu.tr

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The textile mill effluents are deeply colored and decrease photosynthetic activity by blocking sunlight penetration, which significantly reduces the amount of dissolved oxygen [3,4]. The effective treatment of textile industry wastewaters has a great significance to minimize harmful effects of contaminants.

Conventional physical, chemical and biological wastewater treatment processes often cannot achieve complete removal of contaminants from wastewaters. Classical chemical methods have some drawbacks including generation of larger quantities of sludge and higher salinity in the effluents. Biological methods are generally not adequate for the treatment of highly toxic wastewaters and the microorganisms are sensitive to environmental conditions [5].

Advanced oxidation processes (AOP) have the capability of degradation of persistent compounds present in the industrial wastewaters due to generation of highly oxidant radicals which converts the complex chemicals into simple harmless compounds [6]. These processes have the significant advantages over conventional methods due to the formation of strong reactive oxygen species such as hydroxyl radicals. Fenton oxidation is one of the most popular advanced oxidation methods due to its low cost, easy operation, high degradation rate, and ability to reach complete mineralization. The process is based on the formation of hydroxyl radicals, which have high oxidation capacity for the degradation of organic pollutants. Hydroxyl radicals are formed in the reactions between metal ions and hydrogen peroxide [7–9].

Electrocoagulation is an effective wastewater treatment technology due to combination of electrical and chemical methods. It has been applied for the removal a wide variety of pollutants including the organics, inorganics, metals and colloidal particles present in textile, pulp and paper, mining, and food industries [10–12]. Electrocoagulation is based on the formation of the coagulant material by electrical oxidation mechanisms through suitable electrodes in aqueous medium. The metal ions are transferred from anode surface to the aqueous medium by the current. At the same time, the water is reduced to hydrogen gas and hydroxyl ions. Consequently, metal hydroxides are produced as a coagulant material that have highly pollutant adsorption capacity [13]. Electrocoagulation is easy to implement, and require compact equipment and small amount of chemicals. Therefore, this method has economic and environmental advantages compared to other more conventional techniques which need extensive use of chemicals. The combination of electrocoagulation and the Fenton process is extremely effective due to the formation of powerful oxidizing agents [14].

Electrocoagulation followed by Fenton oxidation is considered as excellent for the treatment of high-strength industrial wastewaters. Fenton oxidation can be ineffective for the treatment of intensively contaminated wastewaters. Initial electrocoagulation treatment reduces the pollutant load significantly [15]. In most of the sequential electrocoagulation and advanced oxidation studies, electrocoagulation is adopted as the pre-treatment followed by the Fenton oxidation. This combination is beneficial since the suspended solid and the colloid content of the wastewater is reduced by electrocoagulation. Therefore, the efficiency of the Fenton process can be improved remarkably. The sequential application of the electrocoagulation and Fenton processes is reported as effective for the treatment of dye containing wastewaters [16,17]. In addition, electrocoagulation can provide an efficient pre-treatment before Fenton oxidation. It may have a synergic effect since the residual metal remaining in the solution after electrocoagulation may serve as the catalyst for the Fenton oxidation reactions [18].

In order to use as catalyst in heterogeneous Fenton-like oxidation processes perovskites in the form of ABO_3 (A is a rare earth metal with large ionic radius or a post transition metal such as Bismuth; B is a transition metal with a small ionic radius) are known to be a good alternatives over other metal catalysts due to their resistance to high temperatures, high activity and easy separation from wastewater [19,20].

Development of low cost and effective catalysts is one of the main concern of advanced oxidation studies. In the present study $BiFeO_3$/RHAC and $BiNiO_3$/RHAC (RHAC: Rusk husk based activated carbon) were used as catalyst in Fenton-like oxidation process. In order to prepare eco-friendly catalysts by using low cost and nontoxic materials, rice husk was used as raw material in activated carbon preparation process. Several post-treatment methods have been applied sequentially to satisfy the wastewater discharge limits and to increase the quality of the treated wastewater for its possible reuse. Hybrid wastewater treatment systems comprising of electrocoagulation and advanced oxidation methods have been used effectively for the treatment of industrial wastewaters [18,21–25]. According to the literature survey on industrial wastewater treatment, the application of Fenton-like oxidation in the presence of perovskite/activated carbon catalysts subsequent to electrocoagulation has not been tested so far. In this context, the main original approach of this study is the comparison of the performances of $BiFeO_3$/RHAC and $BiNiO_3$/RHAC catalysts and determination of the most suitable reaction conditions in Fenton-like oxidation subsequent to electrocoagulation.

2. MATERIALS AND METHODS (MATERYAL VE METOD)

2.1. Chemicals (Kimyasallar)

In-situ pre-treated real textile wastewater was provided from a textile factory in İzmir, Turkey. In the pre-treatment step, physical, biological and chemical treatment methods were applied at the factory. Discharge properties of in situ pre-treated wastewater are shown in Table 1. Zinc chloride and hydrochloric acid (37%) were purchased from Merck to synthesize activated carbon from rice husk. Sulphuric acid (Merck, 95-97%) and sodium hydroxide (Merck, pure) were used for pH adjustment in Fenton-like oxidation process. Bismuth nitrate pentahydrate (98%), nickel nitrate hexahydrate (99–102%), iron nitrate nonahydrate (99–102%), and citric acid mono hydrate (99.5-100.5%) were supplied from Merck and used to synthesize $BiFeO_3$/RHAC and $BiNiO_3$/RHAC composite catalysts.

Table 1. Processing parameters (İşleme parametreleri)

Wastewater Characteristics		Value
TOC (mg/L)		30-32
COD (mg/L)		310
Turbidity (NTU)		4.9
Conductivity (mS/cm)		10
Salinity (ppt)		5.5
pH		8-8.5
Dissolved O_2 (mg/L)		3.20
Color, m^{-1}	436 nm	9.41
	525 nm	5.08
	620 nm	3.16

2.2. Catalyst preparation (Katalizör hazırlanışı)

The activated carbon used as catalyst support was prepared from rice husk by using zinc chloride as the activating agent. The weight ratio of the rice husk to zinc chloride was 1:1. The pyrolysis was performed at 750°C for 3 hours. $BiFeO_3$/RHAC and $BiNiO_3$/RHAC catalysts were synthesized by sol-gel method Bismuth nitrate pentahydrate, transition metal nitrate (nickel nitrate hexahydrate or iron nitrate nonahydrate), and citric acid monohydrate were dissolved in ethanol solution. The molar ratio of Bi:M:Acid (M: Fe or Ni) was equal to 1:1:3. The required amount of RHAC was inserted into the mixture to obtain 10% perovskite and 90% activated carbon (by wt.) in the catalyst structure. The mixture was heated and stirred until the gel was formed. After drying, calcination was carried out at 400°C for 4 h [24]. The details of the catalyst preparation steps are shown in Figure 1.

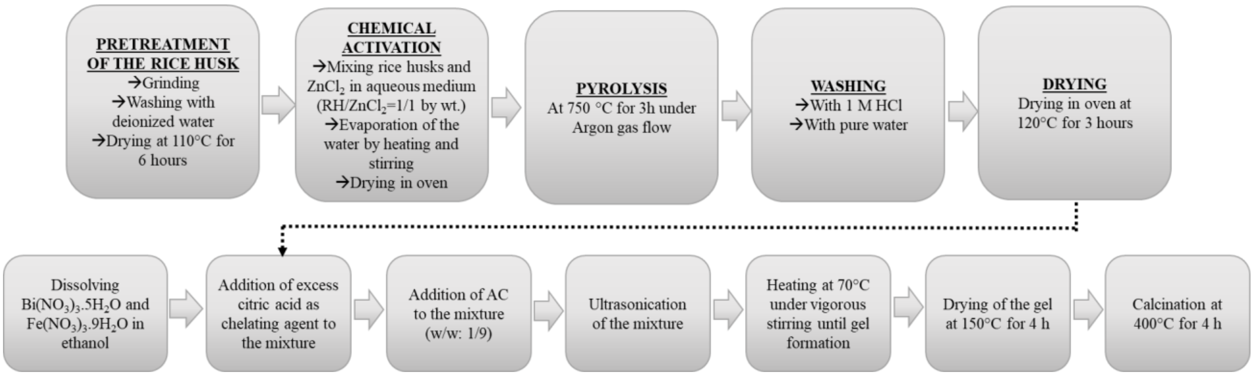

Figure 1. Catalyst preparation steps (Katalizör hazırlanış basamakları)

2.3. Experimental set-up and procedure (Deney düzeneği ve prosedürü)

In-situ pretreated textile wastewater was subjected to electrocoagulation and Fenton-like oxidation sequentially. In the electrocoagulation process Aluminum electrodes were used at a current density of 25 mA/cm^2 and pH=5 when the mixing rate was 220 rpm. The most operational conditions of electrocoagulation were determined in the previous study [26]. The initial TOC content of the wastewater supplied from the textile mill was 30-32 mg/L and the TOC was reduced to 24-26 mg/L after the application of electrocoagulation.

The photo Fenton-like oxidation experiments were performed in a beaker to treat 100 mL of wastewater taken from electrocoagulation effluent. A 100 Watt halogen lamp (Philips Plusline ES, 78 mm) was placed above the reaction medium and a water bath was used to keep the temperature at 25°C. Homogeneous mixing is maintained by using a magnetic stirrer. In a typical run, the pH of the wastewater is adjusted by using dilute sulphuric acid or sodium hydroxide solutions. Required amounts of catalyst and hydrogen peroxide are added into the wastewater simultaneously. At the end of 2 hours, samples are taken from the wastewater to analyze.

In a previous study, the pretreated textile wastewater was subjected to directly photo Fenton-like oxidation, after pH adjustment, 16.12% and 15.92% TOC removal was obtained in the presence and in the absence of visible light irradiation, respectively, by using $BiFeO_3$/activated carbon [27]. Additionally, in the preliminary studies, photo Fenton-like oxidation was applied after electrocoagulation and adsorption. In the photo Fenton-like oxidation step, the TOC removal efficiencies changed between 1.54% and 5.61% in the presence of $BiFeO_3$/RHAC and $BiNiO_3$/RHAC catalysts. Since the application of advanced oxidation after electrocoagulation and adsorption was not effective, and the influence of light irradiation was negligible in the previous studies, Fenton-like oxidation (without light irradiation) was applied subsequent to electrocoagulation for the post treatment of real textile wastewater in the present study. The electrocoagulation and advanced oxidation experiments were repeated three times.

2.4. Analysis (Analiz)

In order to determine the wastewater characteristics total organic carbon (TOC), chemical oxygen demand (COD), pH, concentration of dissolved oxygen, conductivity, color, and turbidity were measured.

Total organic carbon was determined by a Shimadzu TOC Analyzer. TOC removal efficiencies were calculated by using Equation 1:

$$\text{TOC } \% = \frac{(\text{TOC}_0 - \text{TOC}_F)}{\text{TOC}_0} \times 100 \qquad \text{(Eq.1)}$$

Where TOC_0 and TOC_F are the initial and final total organic carbon contents in mg/L.

COD analysis was performed by using Spectroquant Merck COD cell tests, a NOVA 400 spectrophotometer, and a WTW thermoreactor.

Dissolved oxygen, conductivity, and pH were measured via a multimeter (WTW Multi 340i) whereas the turbidity was determined via a WTW Turbo 550IR.

The absorbance values at 436, 525, and 620 nm, which correspond to yellow, red, and blue colors, respectively, were measured in a 10S UV-VIS Spectrophotometer to determine the color.

The spectral absorption coefficient (SAC) is calculated according to the Equations 2-4.

$$\text{SAC}_{436} = \frac{A_{@436\text{ nm}}}{d} \qquad \text{(Eq.2)}$$

$$\text{SAC}_{525} = \frac{A_{@525\text{ nm}}}{d} \qquad \text{(Eq.3)}$$

$$\text{SAC}_{620} = \frac{A_{@620\text{ nm}}}{d} \qquad \text{(Eq.4)}$$

A: Absorbance

d: UV cuvette thickness, [m]

3. RESULTS AND DISCUSSION (BULGULAR VE TARTIŞMA)

3.1. Catalyst screening (Katalizör seçimi)

In the catalyst screening study, Fenton-like oxidation performances of $BiFeO_3$-RHAC and $BiNiO_3$-RHAC catalysts were tested at two different catalyst loadings and pH values to select the most efficient catalyst. The effluent of the electrocoagulation process was used as the wastewater in the catalyst screening study.

During the Fenton-like oxidation the organic pollutants, ferric ions in the $BiFeO_3$/RHAC catalyst structure react with H_2O_2 to produce ferrous ions and hydroperoxyl radicals (Eq. 5). The ferric ions react with H_2O_2 to form more reactive hydroxyl radicals (Eq. 6).

$$Fe^{2+}+H_2O_2\rightarrow Fe^{3+}+HO^{\bullet}+OH^{-} \qquad \text{(Eq.5)}$$

$$Fe^{3+}+H_2O_2\rightarrow Fe^{2+}+HO_2^{\bullet}+H^{+} \qquad \text{(Eq.6)}$$

Bismuth metal ions with a high valence state react with Ni^{2+} and transfer to the previous valence state (Eq. 4), which have the ability to activate the oxidant. Then, the Ni^{3+} and H_2O_2 react to generate Ni^{2+} and hydroperoxyl radical (Eq 7). The hydroperoxyl radicals are converted into superoxide radicals and hydrogen ions (Eq. 8) during the catalytic oxidation. The produced hydroxyl and superoxide radicals react with the organic pollutants and the oxidation products are formed (Eqs. 9 and 10) [28,29].

$$Bi^{5+}+2Ni^{2+}\rightarrow Bi^{3+}+2Ni^{3+} \qquad \text{(Eq.7)}$$

$$Ni^{3+}+H_2O_2\rightarrow Ni^{2+}+HO_2^{\bullet}+2H^{+} \qquad \text{(Eq.8)}$$

$$HO_2^{\bullet}\rightarrow H^{+}+{}^{\bullet}O_2^{-} \qquad \text{(Eq.9)}$$

$$HO^{\bullet}\left(\text{or } {}^{\bullet}O_2^{-}\right)+\frac{\text{Organic}}{\text{Pollutant}}\rightarrow CO_2+H_2O \qquad \text{(Eq.10)}$$

The formation of hydroxyl radicals is enhanced in the presence of the colored substances presented in the textile wastewaters. In the presence of the dyestuffs visible light irradiation results in reduction of Fe^{3+} into Fe^{2+} by intermolecular electron transfer from an excited state of dye molecule (Equations 11 and 12) [30].

$$\text{Dye}+\text{Visble light}\rightarrow \text{Dye}^{*} \qquad \text{(Eq. 11)}$$

$$\text{Dye}^{*}+Fe^{3+}\rightarrow Fe^{2+}+\text{Dye}^{*+} \qquad \text{(Eq. 12)}$$

The TOC removal performances of the perovskite/activated carbon catalysts at 0.25, 0.5 and 1.0 g/L catalyst loadings are presented in Figure 2. Though the catalytic performances of the nickel and iron based catalysts were close to each other, slightly higher TOC removal efficiencies were achieved in the presence of $BiNiO_3$/RHAC catalyst at each of the catalyst loadings.

The maximum allowable discharge limits for iron and nickel were 0.3 and 0.2 mg/L, respectively, according to the WHO guideline [31–33]. Since the difference between the allowable limits of these metals are not too high, either of the iron or nickel containing heterogeneous catalysts can be used in wastewater treatment processes. Based on the high purity chemical use and laboratory scale production, the cost of the $BiNiO_3$/RHAC catalyst required to treat 1 liter wastewater is 0.023$ higher than the cost of $BiFeO_3$/RHAC. The catalyst cost can be decreased by using bulk chemicals at high scale production. Despite its higher cost, nickel shows high catalytic activity in pollutant removal processes and nickel-containing catalysts have the potential to be used more effectively than iron containing catalysts in the presence of various oxidants such as peroxymonosulfate [34] and air [35] in advanced oxidation processes.

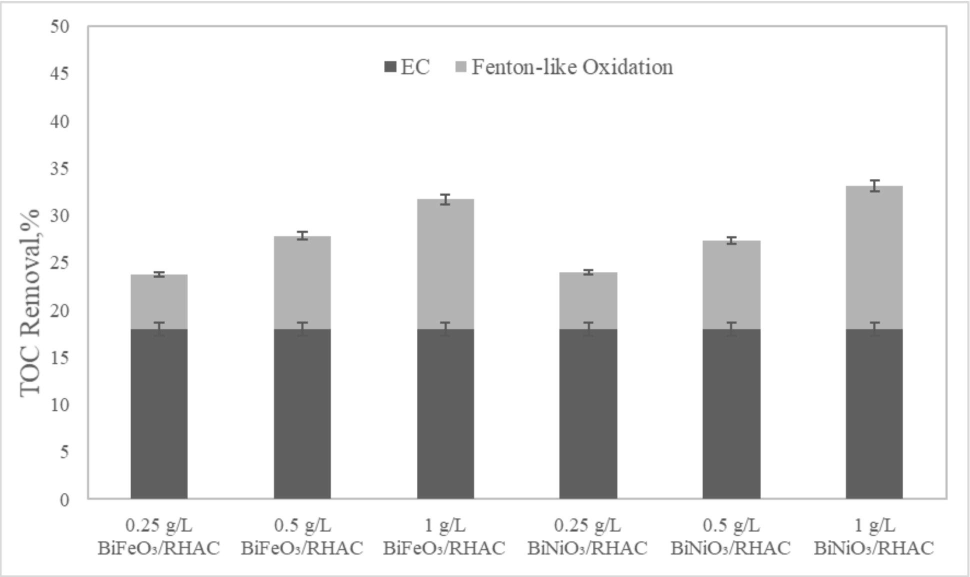

Figure 2. Comparison of catalytic performances of perovskite/activated carbon catalysts at various catalyst loadings, $[TOC]_o$= 30-32 mg/L, room temperature, pH=8-8.5. (Çeşitli katalizör yüklemelerinde perovskite/aktif karbon katalizörlerin katalitik performanslarının karşılaştırılması, $[TOC]_o$= 30-32 mg/L, oda sıcaklığı, pH=8-8,5)

The total organic carbon removal efficiencies were improved by the increase in catalyst dosage. The enhancement in organic pollutant removal is attributed to the increase in the number of reaction sites and the increasing probability of collision between the organic molecules and catalyst particles [36].

The reactivity of metal containing composite catalysts strongly depends on the pH of the wastewater. The pH range tolerance of the perovskite/activated carbon catalysts was studied at various pH values and the results are presented in Figure 3.

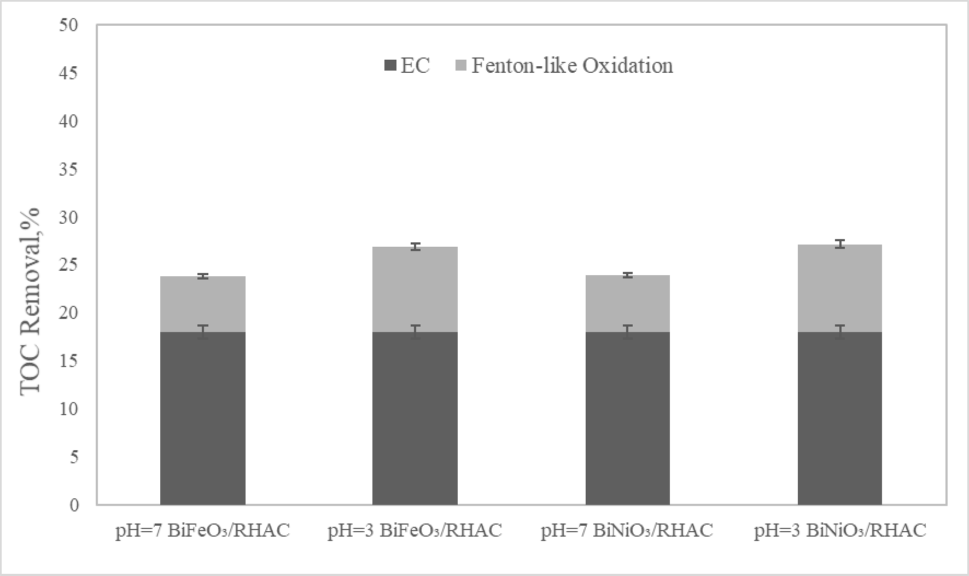

Figure 3. Comparison of catalytic performances of perovskite/activated carbon catalysts at various pH values, $[TOC]_o$= 30-32 mg/L, room temperature, 0.25 g/L of catalyst loading.(Perovskite/aktif karbon katalizörlerin çeşitli pH değerlerinde katalitik performanslarının karşılaştırılması,$[TOC]_o$= 30-32 mg/L, oda sıcaklığı, 0,25 g/L katalizör yüklemesi)

Both of the $BiFeO_3$/RHAC and $BiNiO_3$/RHAC catalysts showed higher performances at pH 3. The generation of hydroxyl radicals was promoted at acidic medium since at lower pH values the perovskite catalyst surface is positively charged which enhanced the adsorption of the negatively charged molecules such as the anionic dyes present in the textile wastewater [37,38]. When the pH was 7, only a slight decrease in TOC efficiencies was observed. This trend indicated that the present system works well under acidic or neutral conditions.

According to the assessment of the catalyst screening study, $BiNiO_3$/RHAC and $BiFeO_3$/RHAC catalysts exhibited very similar catalytic performances and they were approximately equally effective in TOC removal under all of the reaction conditions tested.

Since the removal efficiencies evaluated in the presence of $BiNiO_3$/RHAC catalyst were slight higher, this catalyst is recommended to be used in Fenton oxidation process subsequent to the electrocoagulation for the post-treatment of real textile wastewaters.

3.2. Investigation of the influences of the reaction parameters (Reaksiyon parametrelerinin etkilerinin incelenmesi)

In the parametric study, the effects of the catalyst loading, pH, and the initial hydrogen peroxide concentration on TOC removal were examined in the presence of $BiNiO_3$/RHAC catalyst which was determined as slightly more effective in pollutant removal. Various catalyst loadings varying between 0.25 and 2.0 g/L were tested to observe the impact of the catalyst amount on TOC removal. The wastewater obtained from electrocoagulation process was at neutral pH and no further pH adjustment was performed before the Fenton-like oxidation. Figure 4 depicts that the TOC removal efficiencies were increased with the increasing catalyst dosage. A similar trend was reported in the literature [39–41].

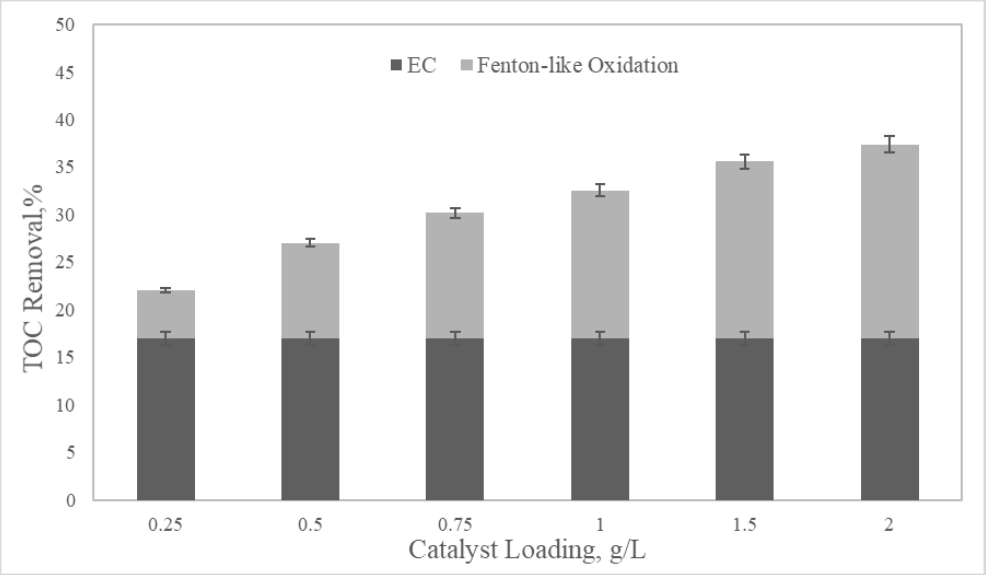

Figure 4. The effect of catalyst loading on TOC removal efficiencies, $[TOC]_o$= 30-32 mg/L, room temperature, neutral pH) (Katalizör yüklemesinin TOK giderim verimi üzerine etkisi $[TOC]_o$= 30-32 mg/L, oda sıcaklığı, nötr pH)

When the catalyst loading was increased from 0.25 g/L to 2 g/L, TOC removal efficiency was increased from 5% to 20.4%. The improvement of the catalytic activity could be associated to the increase in number of available active sites for pollutant adsorption and hydroxyl radical production. Though the generation of hydroxyl radicals was enhanced up to an optimum catalyst loading, the excessive amount of catalyst may result in decrease in removal efficiencies due to the self-scavenging effect of hydroxyl radicals by excess of metal ions (M^{n+}) (Eq.13) [40]:

$$HO^{\bullet}+M^{n+}\rightarrow OH^{-}+M^{n+1} \qquad \text{(Eq.13)}$$

The recommended $BiNiO_3$/RHAC catalyst loading is 1.5 g/L for the Fenton-like oxidation of the textile wastewater since the difference in TOC removal efficiencies evaluated at 1.5 and 2 g/L of catalyst loading was below 2% and the improvement in TOC removal above 1.5 g/L dosage become insignificant gradually. Since the real industrial wastewaters exist various pH values, it is very significant to determine the effect of pH on the pollutant removal efficiency. The influence of pH was investigated in the range of 3-10 and results obtained at acidic, neutral and basic medium are shown in Figure 5.

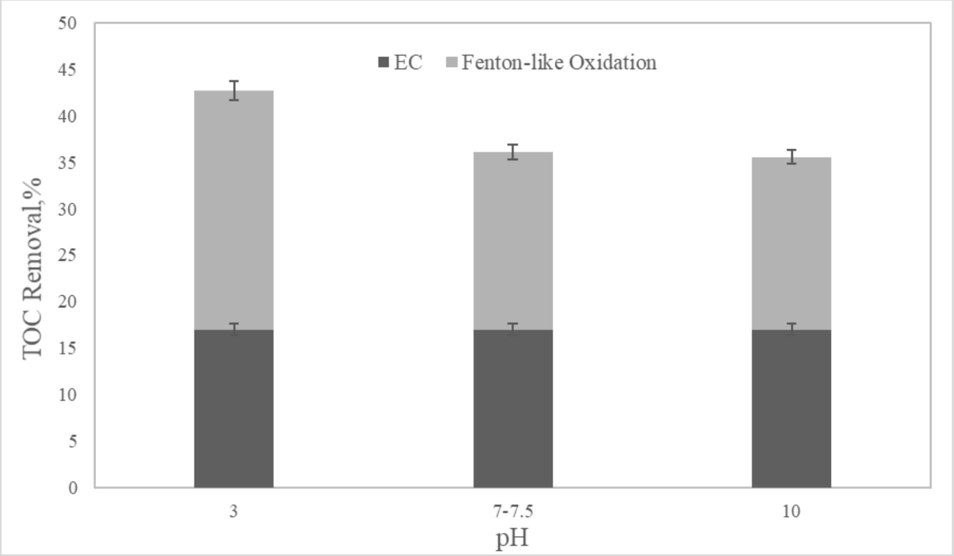

Figure 5. The effect of pH on TOC removal efficiencies, $[TOC]_o$= 30-32 mg/L, room temperature, 1.5 g/L of catalyst loading. (pH'nın TOK giderim verimi üzerine etkisi, $[TOC]_o$= 30-32 mg/L, oda sıcaklığı, 1,5 g/L katalizör yüklemesi)

It is well known that the Fenton reactions are favored at acidic medium. Therefore, the highest TOC removal efficiency was obtained at pH 3 whereas the post-treatment system was slightly inhibited in the alkaline solution. Under the alkaline conditions the oxidant is consumed due to the deprotonation reaction of H_2O_2 (Eq. 14) [42].

$$H_2O_2 \rightarrow HO_2^- + H^+ \qquad \text{(Eq. 14)}$$

In addition, at higher pH values excessive OH^- ions may react with the metal ions to form metal hydroxide precipitates deposited on the catalyst surface leading to the reduction of hydroxyl radical generation.

When the wastewater pH was decreased from 10 to 3 the TOC removal efficiencies increased by 7.1%. The results obtained in this study are consistent with the literature data reported for the Fenton oxidation of various industrial pollutants [43,44].

Even though using acidic medium promoted the TOC removal, pH 7 was determined as the most suitable operating pH since it is the original pH of the electrocoagulation effluent and there is no need a further pH adjustment step to operate at neutral pH. By considering the economic and environmental aspects, neutral pH is determined to be more suitable when compared to the corrosive acidic solutions.

The oxidant dosage is another important parameter in Fenton processes. Hydroxyl radicals are the main free radicals eliminating the pollutants in the wastewaters and H_2O_2 was used as the oxidant and the hydroxyl radical source in the present study. In order to investigate the effect of the initial H_2O_2 concentration, 2, 4 and 8 mM oxidant dosages were tested at pH 7 and 1.5 g/L catalyst loading. At 2 mM H_2O_2 concentration, 15.7% TOC removal was accomplished and approximately the same TOC removal was recorded at 4 mM H_2O_2 concertation. A further increase in oxidant dosage up to 8 mM resulted in enhancement of the TOC removal slightly. 17.7% TOC removal efficiency was evaluated when the initial H_2O_2 concentration was 8 mM. Even though using the H_2O_2 promotes the pollutant efficiency above the optimal oxidant dosages the produced hydroxyl radicals can be consumed by the scavenging effect of the excess H_2O_2 (Eq. 15) [45]:

$$H_2O_2 + HO^{\bullet} \rightarrow HO_2^{\bullet} + H_2O \qquad \text{(Eq. 15)}$$

Considering the insignificant improvement in TOC removal efficiencies and the possible scavenging effect of the high amounts of oxidant, 2 mM was selected as the most suitable H_2O_2 concentration for the Fenton-like oxidation operated in the presence of $BiNiO_3$/RHAC catalyst.

In addition to the TOC content, chemical oxygen demand (COD), turbidity, conductivity, pH, dissolved oxygen, and color of the textile wastewater treated under the most suitable Fenton-like oxidation conditions were determined. The wastewater characteristics determined after the subsequential application of electrocoagulation and Fenton-like oxidation were shown in Table 2.

Table 2. Treated wastewater characteristics after the application of electrocoagulation and Fenton-like oxidation (Elektrokoagülasyon ve Fenton benzeri oksidasyon uygulandıktan sonra arıtılmış atık su özellikleri)

Characteristic		**Final Value**	**Maximum Removal (%)**
TOC (mg/L)		20-25	32.7
COD (mg/L)		230-240	25.8
Turbidity (NTU)		0.30-0.40	94
Conductivity (mS/cm)		10.15	-
Salinity (ppt)		5.6	-
pH		7-7.8	-
Dissolved O_2 (mg/L)		2.25	-
Color, m^{-1}	436 nm	4.81-4.95	48
	525 nm	2.0-2.16	60
	620 nm	0.8-0.91	71

Under the most suitable electrocoagulation and Fenton-like oxidation conditions the TOC and COD removal efficiencies reached up to 32.7% and 25.8%, respectively, whereas the conductivity (or salinity) could not be reduced by the application of the hybrid post-treatment method. The post-treatment system was very effective in turbidity and color removal.

Color limits determined in terms of European Norm EN ISO 7887 are 7 m^{-1} for 436 nm (yellow), 5 m^{-1} for 525 nm (red), and 3 m^{-1} for 620 nm (blue) [46,47]. The color of the post-treated wastewater is acceptable considering the European Norm. According to the national irrigation water quality classification, pH range of 6.5–8.5 and the total suspended solids up to 30 mg/L were accepted as satisfactory for reuse as irrigation water. Therefore, the irrigation water criteria for pH and turbidity were met by the application of post-treatment. The conductivity, TOC, and COD limits were 3 mS/cm, 8 mg/L and 50 mg/L, respectively, for irrigation applications [27,48]. The TOC and COD contents and salinity (in terms of conductivity) of the post-treated wastewater are above the allowable limits set for the irrigation water. Therefore, an additional treatment method such as nanofiltration and reverse osmosis can be applied as the last step of the hybrid treatment to improve the quality of the treated wastewater, which increase the potential reuse of the wastewater. The beneficial use of water include drinking water, recreational use, fisheries and shellfish production, irrigation, and industrial process water [49]. It may be possible to reuse the treated wastewater in various fields by increasing the reuse potential of the wastewater via hybrid treatment methods.

4.CONCLUSIONS (SONUÇLAR)

Electrocoagulation and heterogeneous Fenton-like oxidation methods were applied sequentially for the post-treatment of real textile wastewater. The electrocoagulation was operated at 25 mA/cm^2 current density, pH 5 and 220 rpm agitation speed. The effluent of the electrocoagulation process was subjected to Fenton-like oxidation. $BiFeO_3$/RHAC and $BiNiO_3$/RHAC catalysts showed similar catalytic performances in the Fenton-like oxidation process. In the parametric study the influences of catalyst loading, pH, and the initial H_2O_2 concentration on total organic carbon removal were investigated. According to the results, increasing the catalyst and oxidant dosage improved the catalytic pollutant removal while decreasing the pH enhanced the removal efficiencies. Considering the environmental and economic aspects, the most suitable reaction conditions were determined as 1.5 g/L of $BiNiO_3$/RHAC, neutral pH, and 2 mM of H_2O_2 concentration. Under these conditions, cumulative TOC, COD, and turbidity removal efficiencies were 32.7, 25.8, and 94%, respectively. The color removal efficiencies varied between 48% and 71% for yellow, red and blue colors. The results indicated that the novel perovskite/activated carbon composite materials are promising alternatives for the post-treatment of real industrial wastewaters in green hybrid treatment technologies.

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DECLARATION OF ETHICAL STANDARDS (ETİK STANDARTLARIN BEYANI)

The author of this article declares that the materials and methods they use in their work do not require ethical committee approval and/or legal-specific permission.

Bu makalenin yazarı çalışmalarında kullandıkları materyal ve yöntemlerin etik kurul izni ve/veya yasal-özel bir izin gerektirmediğini beyan ederler.

AUTHORS’ CONTRIBUTIONS (YAZARLARIN KATKILARI)

Özlem BULCA**:** She conducted the experiments, analyzed the results and contributed to the writing process.

Deneyleri yapmış, sonuçlarını analiz etmiş ve makalenin yazımına katkıda bulunmuştur.

Burcu PALAS**:** She analyzed the results and contributed to the writing process.

Deney sonuçlarını analiz etmiş ve makalenin yazımına katkıda bulunmuştur.

Süheyda ATALAY**:** She analyzed the results and contributed to the writing process.

Deney sonuçlarını analiz etmiş ve makalenin yazımına katkıda bulunmuştur.

Gülin ERSÖZ**:** She analyzed the results and contributed to the writing process.

Deney sonuçlarını analiz etmiş ve makalenin yazımına katkıda bulunmuştur.

CONFLICT OF INTEREST (ÇIKAR ÇATIŞMASI)

There is no conflict of interest in this study.

Bu çalışmada herhangi bir çıkar çatışması yoktur.

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