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Treatment of Sugar Industry Wastewater by Using Subcritical Water as a Reaction Media

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The sugar industry is one of the most wastewater-producing industries and it contains high content of organic and inorganic substances. Treating and reusing wastewater has significant importance because sugar industry needs to use a high volume of water. In this study, sugar industry wastewater was treated under subcritical conditions and the impacts of reaction temperature and duration over TOC removal percentage were investigated. Additionally, the impact of NaOH concentration over TOC removal percentage was examined. The highest TOC removal was obtained almost 95% in the presence of 0.1 M of NaOH at 240 °C for 90 min of reaction duration. Treatment of sugar industry wastewater by subcritical water oxidation followed the second-order reaction kinetic model and the activation energy was found as 11.41 kJ/mol. Furthermore, the intermediate products were identified via GC-MS.

Introduction

Sugar industry placed within the most pollutant-generating industries in terms of volume and characteristics of produced wastewater. While it has been reported that $450x10^3$ m^3 of wastewater was generated by the sugar industry of India during six months running session, this value has been reported as 713.83×10^6 m^3 for the Mexican sugar industry for six months running session. It mainly contains the water used in cleaning processes and it could comprise lost cane juice and sugar solids, bagasse particles, detergents as well as oil and grease. In addition, it is characterized by high concentrations of nutrients and organic and inorganic contents.[1]

Turkey is one of the greatest sugar beet producer countries across the world. Turkey, the world's fifth- sugar beet producing country, is also the fourth largest country in Europe. In Turkey, 33 sugar factories with different capacities produce sugar from sugar beet and corn. These factories can produce sugar almost 3.5 million tons/year.[2] In these factories, at the end of the production step, large amounts of wastewater form. If this wastewater releases into nature without any treatment, it causes critical environmental concerns in our country as well as in the world. The sugar factories use high amounts of water for washing and flotation of sugar beets and the generated wastewater streams from sugar factories mainly compose of washing and flotation water. This wastewater contains organic substances with high carbohydrate content that mix with water from the surface of sugar beets. Additionally, Turkey is a water scarcity country so the treatment and reuse of wastewater generated by the sugar industry have gained great attention.

Alkaya and Demirer studied over the hydrolysis and acidification of the sugar industry wastes (sugar beet processing wastewater and beet pulp which were supplied from sugar factory in Amasya) for volatile fatty acid production by acidogenic anaerobic metabolism. The characterization study of the wastewater containing high amounts of hydrocarbons taken from the sugar plant (total Chemical Oxygen Demand (COD) of 6621 ± 113.2 mg/L, soluble COD of 6165 ± 517.1 mg/L) and pressed sugar beet pulp (1.22 ± 0.15) was performed.[3] The fact that sugar beet wastewater and pulp contain high amounts of hydrocarbons has enabled us to carry out studies to produce hydrogen, bioethanol, acetone, butanol, ethanol, and methane that can be obtained as valuable substances from these wastes. For example, Almohammed et al. studied over bioethanol production from sugar beet roots by pulsed electric field treatment.[4] Bellido et al., carried out a study to produce acetone, butanol, and ethanol with *Clostridium beijerinckii* from sugar beet pulp.[5] Demirel and Scherer performed a study over the production of methane from sugar beet silage.[6] Dhar et al. studied over the hydrogen production from sugar beet juice using an integrated biological hydrogen process consisting of dark fermentation and microbial electrolysis cells. The total hydrogen production obtained in this study was 25% of initial the COD amount. That is, this is equal to six moles of H_2/mol hexose. In addition, 57% of energy recovery was achieved from sugar beet juice using combined biological hydrogen production.[7] Kars and Alparslan studied over the production of biological hydrogen and 5-aminolevulinic acid from sugar beet molasses in the bio-refinery concept. In this study, *Rhodobacter sphaeroides O.U.001* was used to produce biological hydrogen and 5-aminolevulinic acid (5-ALA), and different culture media with various sugar content were tested. The results obtained indicate that molasses is a promising substrate for the production of biological hydrogen and 5-ALA. The highest amounts of biological hydrogen and 5-ALA were observed in media containing 28 g/L sugar (1.01 L H_2 L culture,

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23337 μM).[8] Consequently, the waste and wastewater of sugar industries could be used to produce value-added compounds.

In light of the literature survey, it could be concluded that various methods were used for the treatment of sugar industry wastewater. However, it has not been performed in the subcritical reaction media which is known as an effective wastewater treatment process. Subcritical water refers that the temperature of water changed between 100 and 300 °C when its pressure varied between 1 and 50 MPa, while supercritical water refers that temperature and pressure of water exceeding the critical pressure and temperature (374 °C and 22.1 MPa, respectively) of water. Additionally, hot compressed water (HCW) has different properties compared to ambient water. For instance; it has more ion value (10–11 at 250 °C) than water (10–14 at room temperature) and has a lower dynamic viscosity and a relatively low dielectric constant than its values under normal conditions.[9] It was used for the treatment of various wastewater streams such as Reactive Red 120, Acid Red 274, oil mill wastewater, and palm oil mill effluent.[10–13] However, up to our knowledge, sugar industry wastewater has not been treated via hydrothermal liquefaction. In this study, the treatment of sugar industry wastewater was achieved via hydrothermal liquefaction. In this context, the effect of reaction parameters that are reaction temperature (120–240 °C) and duration (1–2 h), sodium hydroxide (NaOH) concentration (0–0.1 M) over Total Organic Carbon (TOC) removal was investigated and intermediate products were identified via Gas Chromatography-Mass Spectrometry (GC-MS).

Results And Discussion

The sugar industry wastewater was treated by using subcritical water as a reaction medium and the effects of reaction parameters such as reaction time, reaction temperature, and concentration of NaOH over the TOC removal were investigated. Firstly, the impacts of reaction temperature and duration were investigated and optimized. The results were given in Figure 1. Results showed that as the reaction temperature increased the TOC removal percentage enhanced and the highest TOC removal (45 %) was achieved at 240 °C for 2 hours. Additionally, an increase in reaction duration caused a slightly positive impact over the TOC removal, however, it did not cause a big difference in terms of TOC removal efficiency so the optimum reaction duration was selected as 1 hour. After that, the effect of reaction temperature over TOC removal was examined in the presence and absence of NaOH and the results were given in Figure 2. In this case, the addition of NaOH to the reaction media had a positive impact over TOC removal in each run and a synergetic effect of reaction temperature and NaOH concentration was observed. Almost the same TOC removal was observed at 180 °C and 210 °C in the presence of NaOH, however, it enhanced slightly at 240 °C in the presence of NaOH. In order to keep energy consumption at a minimum, the rest of the experiments were carried out at 180 °C since the TOC conversion was similar at 240 °C. Then, the effect of NaOH concentration (0, 0.01, 0.02, 0.04, and 1 M) over TOC removal efficiency was investigated and the results were given in Figure 3. Based on the results, the highest TOC removal (~ %70) was achieved using 0.1 M NaOH, and hence, subcritical water could be considered a promising wastewater treatment process for the removal of TOC from sugar industry wastewater.

Kinetic Study

The reaction kinetic mechanism of treatment of sugar industry wastewater under subcritical conditions was investigated at 180, 210, and 240 °C for 60, 90, and 120 min., and obtained data were linearized to figure out which reaction kinetic model was more appropriate. The R^2 values of the first-order reaction model were very low compared to the R^2 values of the second-order reaction model, and the linearized data for the second-order reaction kinetic model were given in Figure 4. Therefore, the subcritical treatment of sugar industry wastewater followed the second-order reaction kinetic model. Reaction rate kinetic constants of second order reaction kinetic model were found as 0.0001, 0.00008, and 0.00008 for 240, 210, and 180 °C, respectively. The Arrhenius plot of lnk versus 1/T was given in Figure 5 and the activation energy was calculated as 11.41 kJ/mol for this reaction.

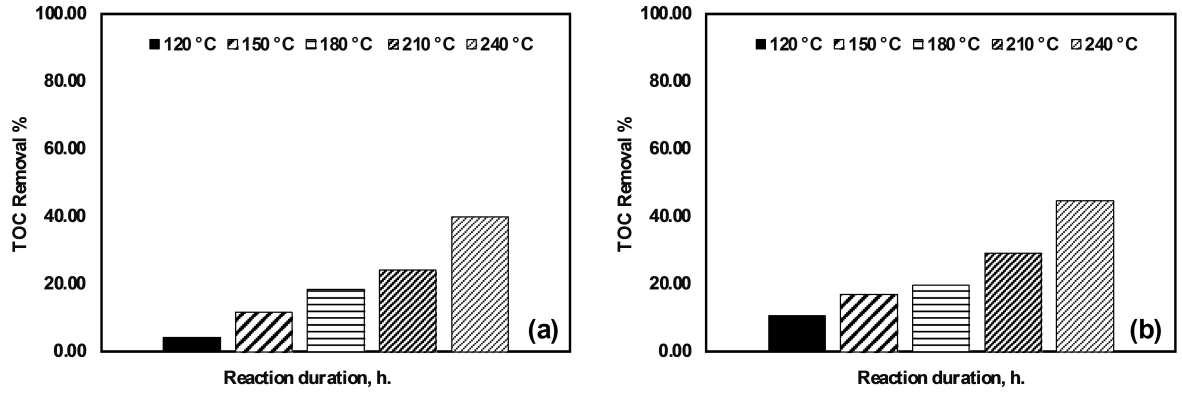

Figure 1. TOC removal % depending on reaction duration 1 h (a) and 2 h (b) (***reaction conditions:*** *100 ml of sugar industry wastewater, T = 120–240 °C, t = 1–2 h).*

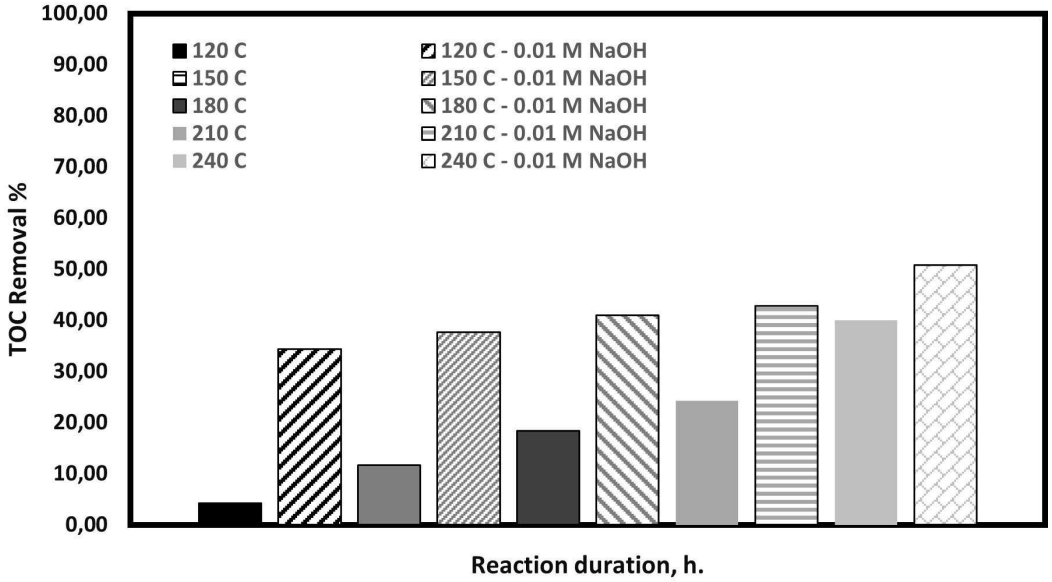

Figure 2. TOC removal % depending on reaction temperature (***reaction conditions:*** *100 ml of sugar industry wastewater, T = 120–240 °C, t = 1 h, $[NaOH]_0 = 0.01$ M).*

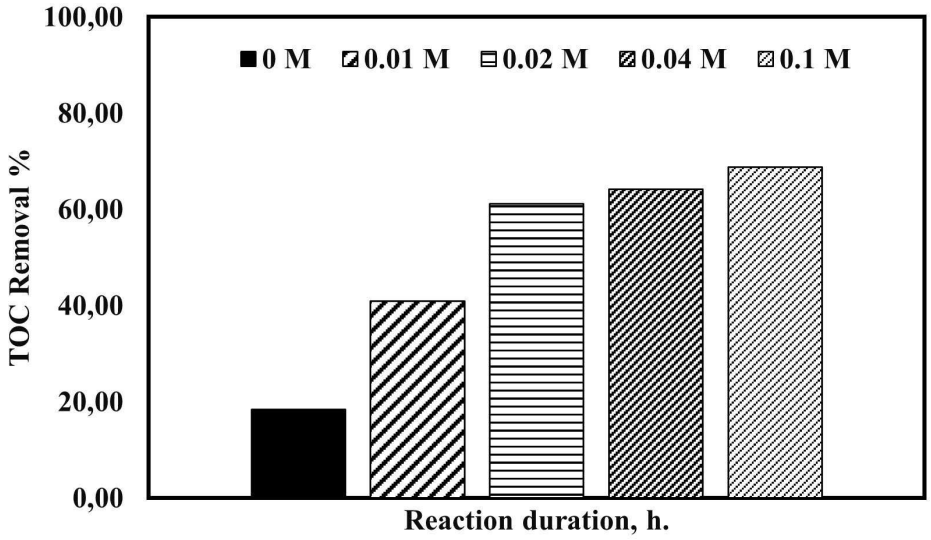

Figure 3. TOC removal % depending on NaOH concentration (***reaction conditions:*** *100 ml of sugar industry wastewater, T = 180 °C, t = 1 h, $[NaOH]_0 = 0–0.1$ M).*

Intermediate Products

GC-MS analysis was firstly performed to identify the compounds in untreated wastewater and the results showed that the compounds in untreated sugar industry wastewater were pentaethylene glycol (7.25 min.), (3-Methyl-oxiran-2-yl)-methanol (7.80 min.) and 1-butanol (8.78 min.). After the treatment of sugar industry wastewater under subcritical conditions, the intermediate products were identified via GC-MS analysis, and the list of formed intermediate products was given in Table 1 and some of these compounds are value-added.

The use of electrochemical processes for various wastewater streams is frequently encountered in the literature.

Table 1. Identified intermediate products by GC-MS.

Retention time (min)	Intermediate compounds
4.56	Ethanol
6.02	Acetonitrile
7.36	Carbonic acid, dimethyl ester
7.82	Ethyl oxamate
12.01	2-propanone, 1-hydroxy
14.48	Acetic acid
16.06	Dimethylamine
22.22	Acetamide
22.23	Ethanamine, N-methyl

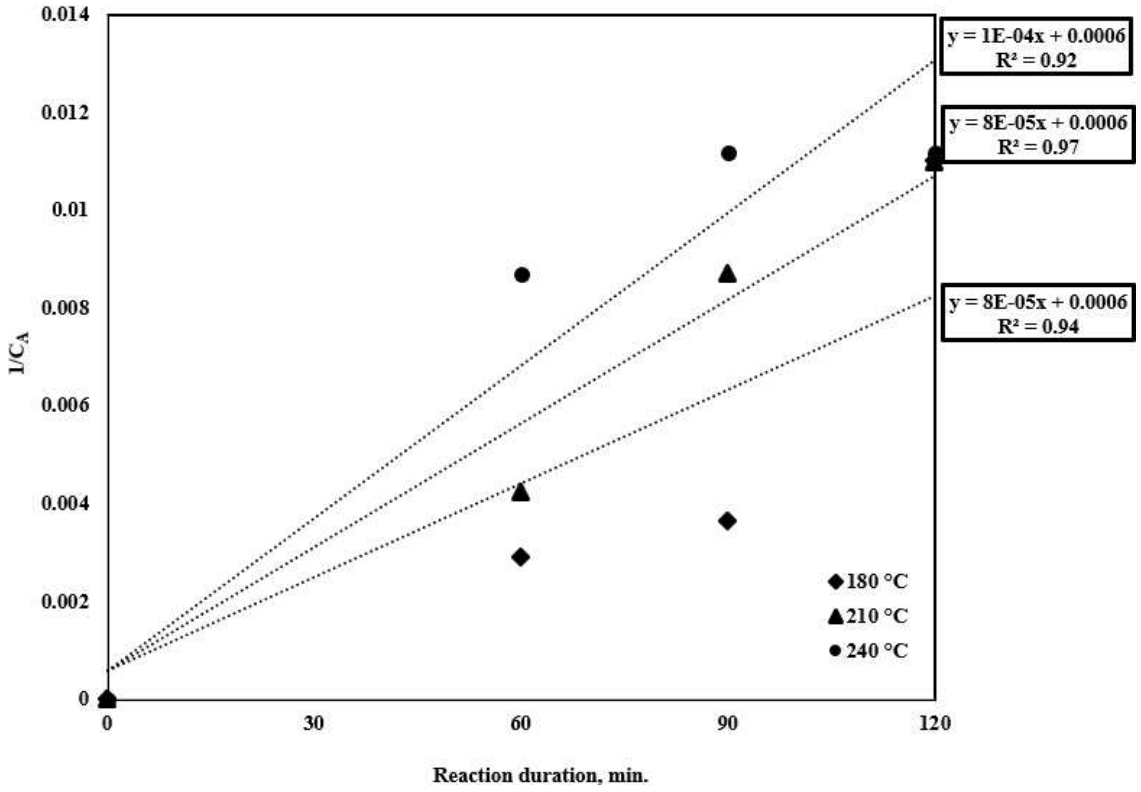

Figure 4. Linearized second-order kinetic plot (***reaction conditions:*** *100 ml of sugar industry wastewater, T = 180, 210, and 240 °C, t = 60, 90, and 120 min., $[NaOH]_0 = 0.1$ M*).

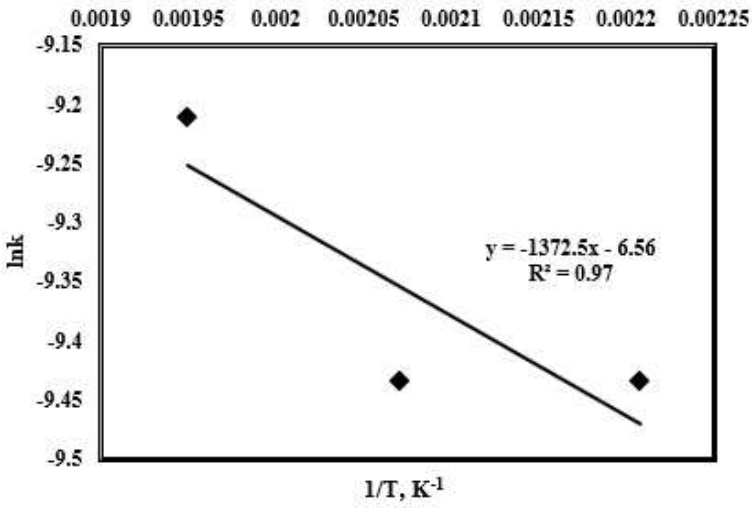

Figure 5. lnk vs 1/T.

Especially promising results have been found in studies on wastewater from the sugar industry. Electrochemical treatment was applied on sugar industry wastewater by Sahu et al with the aluminum electrode at pH = 7, electrode gap (EG) = 20 mm, current density (CD) = 178 A/m^2, and COD and color reduction were 79%, and 78% respectively.[14] Also, Soho et al. reported maximum yield as 84% COD and 86% color reduction, respectively, at the following reaction conditions: pH = 6, CD = 178 A/m^2, electrode gap 20 mm using iron as an electrode in 120 min of treatment.[15] The combined experiment of ferrous sulphate catalyst and electrodes was performed and 97.8% COD reduction and 99.7% color removal were achieved in 15 min treatment time at pH = 6, CD = 156 A/m^2 with electrode distance 20 mm.[16] When using aluminum electrodes at 1.5 cm

distance at pH = 7, and current as 1 A in 60 min treatment time, COD removal and color reduction were found as 69.2%, and 88.6%, respectively.[17] In addition, Sahu et al. reported that 90% COD and 94% color removal were achieved at optimum conditions pH = 7, CD = 178 A/m^2, electrode distance 20 mm, and using 0.5 M NaCl solution.[18] Also, the treatment of sugar industry wastewater was achieved using the same molarity of NaCl solution via electrocoagulation at the following conditions: pH = 6.5, EG = 20 mm, CD = 156 A/m^2, and 85% of COD and 89% of color removal were obtained.[19] Gupta et al. studied on the treatment of wastewater from the sugar industry using electrocoagulation, and the optimum reaction conditions were found as pH = 6.5, CD = 133.9 A/m^2, and (copper) EG = 1.5 cm. Totally 1274 mg/dm^3 of COD was removed and 95.67% color reduction was successfully achieved.[20] Güven et al. studied with iron electrodes in the presence of NaCl electrolyte. Optimum conditions were determined at 100% waste concentration, 33.05 g/L supporting electrolyte concentration, and 12 V of applied voltage. COD removal and COD initial removal rate were found as 79.66%, and 33.69 mg/L, respectively.[21] Sharma et al. reported the optimum conditions for electrooxidation on sugar beet industry process wastewater as pH = 5, and CD = 49.1 mA/cm^2, in 294 min operation time, so 75% of COD and TOC were achieved.[22] Asaithambi et al. studied on the percentage of COD removal in a batch electrochemical reactor, and they investigated different parameters such as current density, the concentration of electrolyte, and flowrate. Maximum 80.74% of COD removal was achieved where optimum conditions were determined as 5 A/dm^2 and 5 g/L.[23]

Contrary to electrochemical processes, studies on wastewater treatment of sub/supercritical processes are limited. There are several applications of sub/supercritical water oxidation for various wastewater streams except sugar industry in literature. Crystal violent was degraded at 120–200 °C and 2.5–6 MPa in the presence of a catalyst (Ni/MgAlO) and 98% dye removal was obtained.[24] Daskalaki et al. studied over the degradation of Reactive Red 120 using H_2O_2 (hydrogen peroxide) as an oxidant under subcritical conditions and they observed that TOC removal % was within 20–64%.[10] Hosseini et al. studied over the decomposition and decolorization of 4-(2-Hydroxynaphthylazo) benzenesulfonic acid sodium salt (AO7, acid dye) as a model for textile wastes by using a flow-type subcritical water system. The operation pressure was not effective over decomposition reactions up to 25 MPa. AO7 totally decomposed at higher operating temperatures or longer residence time and the reaction followed the first-order reaction. Additionally, activation energy was found as 102.2 kJ/mol for the overall reaction.[25] Zhang et al. focused on the decomposition and decolorization of methyl orange solution (MO) which represents the textile wastewater via hydrothermal treatment in a flow-type reactor packed with MnO_2 catalyst. Nearly complete decomposition of this solution (TOC_0:300 mg/L) was achieved in hot steam at 300 °C, 1 MPa, 10 s, and 1.5 oxygen supply ratio and this reaction followed a second-order kinetic model. The highest TOC removal (97.6%) was achieved at the following conditions: 300 °C, 10 MPa, 1921.8 s, for subcritical water oxidation of MO. A two-step first-order kinetic model was used to describe the kinetics of subcritical water oxidation.[26] Enteshari and Martínez-Monteagudo studied about the treatment of ice cream wastewater under subcritical conditions (T: 130–230 °C and P: 20–60 bar). The degree of hydrolysis significantly enhanced depending on the increase of reaction duration and temperature and its maximum value was 40.99 ± 0.81after 200–240 min at 230 °C. Additionally, the activation energy of apparent reaction was calculated as 37.53 ± 5.21 kJ/mol.[27] Yabalak worked about the mineralization of ampicillin in subcritical water medium using H_2O_2 and TOC removal (81.59%) was achieved at 403 K of reaction temperature, 80 mM of H_2O_2, and 147.3 min of reaction duration.[28] In addition, he studied over the degradation of ticarcillin in subcritical water medium using H_2O_2, and 81.99% of TOC, 79.65% of COD, and 94.35% of ticarcillin removal were observed.[29] Also, Yabalak et al, studied on the treatment of agricultural chemicals production plant wastewater with the subcritical water oxidation method. They achieved 59.45% TOC and 97.92% color removal rates at 433 K, 1.2 M of H_2O_2, in 100 min.[30] Meng et al. conduct a study to treat methyl vanillin wastewater in a subcritical Fenton oxidation (HCFO) system. In this system, Fe^{2+} was used as a catalyst, and TOC removal was 94.3% at the following conditions: temperature of 340 °C, pressure of 24 MPa, oxidant multiple of 1.5, residence time of 197 s (flow rate of 2.0 mL min^{-1}). Additionally, the reaction followed the first-order reaction model, and the activation energy was found as 32.6 kJ/mol.[31] Yüksel et al. worked over the degradation of Orange G between 180–250 °C and at 7 MPa and achieved greater than 99% TOC removal in 30–90 of treatment time.[32] Zhang et al. showed that supercritical water could be successful in the decomposition of CI Reactive Orange 7 dye. According to the results COD and TOC removal were reported as 98%, and 88%, respectively.[33] 99.8% of COD removal was obtained by Li et al. from dyeing sludge, at 600 °C, and 25 MPa with 1.2 oxidation coefficient within 600 s of treatment time in the batch reactor.[34] Söğüt and Akgün reported 99.99% of TOC removal between 450–600 °C and 24–28 MPa during rapid supercritical water oxidation within 9–19 s in a continuous flow reactor.[35] Wang et al. studied about the treatment of petrochemical sludge (PS) in subcritical and supercritical conditions (350-450 °C) for 30 min and the oil phase was degraded. During the hydrothermal treatment of PS, 44.98%–59.64% of the oil (organic matter) in the sludge was decomposed into aqueous and gas products. The TOC and COD concentrations in liquid phase were 14,960–19,050 mg/L and 9029–10,870 mg/L, respectively. The main constituents of the gas products were H_2 and CO_2. In addition, this study shows that PS transforms from being hazardous waste to general solid waste so that its treatment/disposal cost is reduced significantly. Thus, the hydrothermal treatment of PS could have potential applications to manage the disposal of petrochemical industry.[36]

The aforementioned related studies from the literature were given as a literature comparison table in Table 2. As a result, in the literature, the COD removal % was reported between 69.2 and 97.8 during the treatment of sugar industry

Table 2. Literature comparison.

Process	Wastewater Type	Optimum Reaction Conditions	TOC removal (%)	COD removal (%)	Decolorization (%)	Reference
Electrochemical	Sugar Industry	Aluminum electrode pH = 7, EG = 20 mm, CD = 178 A/m^2	–	79	78	Sahu [14]
		Iron electrode pH = 6, EG = 20 mm, CD = 178 A/m^2, time = 120 min	–	84	86	Sahu [15]
		Ferrous sulphate catalyst pH = 6, EG = 20 mm, CD = 156 A/m^2, time = 15 min	–	97.8	99.7	Sahu [16]
		Aluminum electrode pH = 7, EG = 1.5 cm, current as 1 A, time = 60 min	–	69.2	88.6	Sahu [17]
		Aluminum electrode pH = 7, EG = 20 mm, CD = 178 A/m^2, 0.5 M NaCl solution	–	90	94	Sahu [18]
		Iron electrode pH = 6.5, EG = 20 mm, CD = 156 A/m^2, 0.5 M NaCl solution	–	85	89	Sahu [19]
		Copper electrode pH = 6.5, EG = 15 mm, CD = 133.9 A/m^2	–	–	95.7	Gupta [20]
		Iron electrodes NaCl solution, 12 V	–	79.7	–	Güven [21]
		pH = 5, CD = 49.1 mA/cm^2, time = 294 min	–	75	75	Sharma [22]
		CD = 5 A/dm^2, Flowrate = 5 g/L	–	80.7	–	Asaithambi [23]
Sub/Supercritical	Reactive Red 120	200 °C, 1% w/v H_2O_2, time < 70 min	20–64	–	–	Daskalaki [10]
	AO7, acid dye	220–300 °C and 10–25 MPa	> 99	–	> 99	Hosseini [25]
	MO	300 °C, 10 MPa, 1921.8 s	97.6	–	–	Zhang [26]
	Ampicillin	403 K, 80 mM of H_2O_2, 147.3 min	81.6	–	–	Yabalak [28]
	Ticarcillin	453.5 K, 80 M H_2O_2, 80 min	82	79.7	–	Yabalak [29]
	Agricultural chemicals production plant	433 K, 1.2 M of H_2O_2, 100 min	59.4	97.9	–	Yabalak [30]
	Methyl vanillin	340 °C, 24 MPa, Fe^{2+}, 197 s	94.3	–	–	Meng [31]
	Orange G	180–250 °C, 7 MPa, 30–90 min	> 99	–	–	Yüksel [32]
	CI Reactive Orange 7 dye	550 °C, pH = 8, 10.5 min	88	98	–	Zhang [33]
	Dyeing sludge	600 °C, 25 MPa, 600s	–	99.8	–	Li [34]
	CI Basic Blue 41	450–600 °C, 24–28 MPa, 9–19 s	> 99	–	–	Söğüt [35]
	Sugar Industry	240 °C, 90 min., 0.1 M NaOH	94.9	93.1	–	This study

wastewater via the electrochemical process.[14–23] Additionally, TOC removal % was reported within 20 and almost complete.[10,25,26,28–35] In the present study, 94.9% of TOC removal and 93.1% of COD removal % were achieved, and comparing the literature, higher TOC and COD removal % were obtained at milder conditions.

Consequently, the literature survey showed that hydrothermal treatment of various wastewater streams was studied, and it is a promising wastewater treatment process. Up to our knowledge, sugar industry wastewater has not been treated via this process and in this study, it was treated by using subcritical water as a reaction media. Compared to the literature, treatment of sugar industry wastewater by using subcritical water as a reaction media could be achieved at milder reaction conditions and higher TOC removal percentages could be achieved with lower activation energy.

Conclusion

The sugar industry needs a high volume of water to remove impurities from the surface of sugar beets and sugar canes. Additionally, a high volume of water is required during the sugar process. The reusability of water in the sugar industry has importance and thus the wastewater produced from the sugar industry should be treated. In this study, sugar industry wastewater was treated by using subcritical water as a reaction media. The optimum reaction time and temperature were found as 1 h and 180 °C, respectively. Then, the effect of NaOH concentration was investigated and almost 95% of TOC removal was achieved using 0.1 M of NaOH at 240 °C for 90 min of reaction duration. Intermediate products of subcritical treatment of sugar industry wastewater were identified via GC-MS analysis and based on the results, value-added compounds (i.e., ethanol, acetic acid) formed during the wastewater treatment. This reaction followed the second-order kinetic model and the activation energy was determined as 11.41 kJ/mol.

Table 3. Elemental analysis of sugar industry wastewater.

Elements	Sugar industry wastewater [mg/L]
TKN (N)	51.8
Phosphorus (P)	–
Potassium (K)	157.8
Cobalt (Co)	0.9607
Iron (Fe)	5.203
Molybdenum (Mo)	0.1064
Nickel (Ni)	1.654
Selenium (Se)	1.785
Calcium (Ca)	579.4
Magnesium (Mg)	99.51

Materials And Methods

Materials

Sodium hydroxide, NaOH, (Merck, analytical grade) was used as an electrolyte, instead of using chlorine-containing salts, it was selected to conserve the reactor from corrosion. Sugar industry wastewater was supplied from a sugar factory in Eskişehir, Turkey and its characterization was performed via ICP-MS (Agilent 7500 ce) and results were given in Table 3. TOC and COD of this wastewater were 1690 mg/L and approximately 6200 mg/L, respectively.

Experimental Set-up and Procedure

The hydrothermal liquefaction of sugar industry wastewater was performed in a batch reactor (Parr 5500 High-Pressure Compact Reactor, V: 300 mL) made from Type 316 Stainless Steel. The components of the reactor are illustrated in Figure 6.

The hydrothermal liquefaction of sugar industry wastewater was carried out in the Parr batch reactor using 100 mL of sugar industry wastewater. After the introduction of sugar industry wastewater, firstly, it was started to stir and then, purged with nitrogen to remove air from the reaction media. After that, it was heated up to desired reaction temperatures (120, 150, 180,

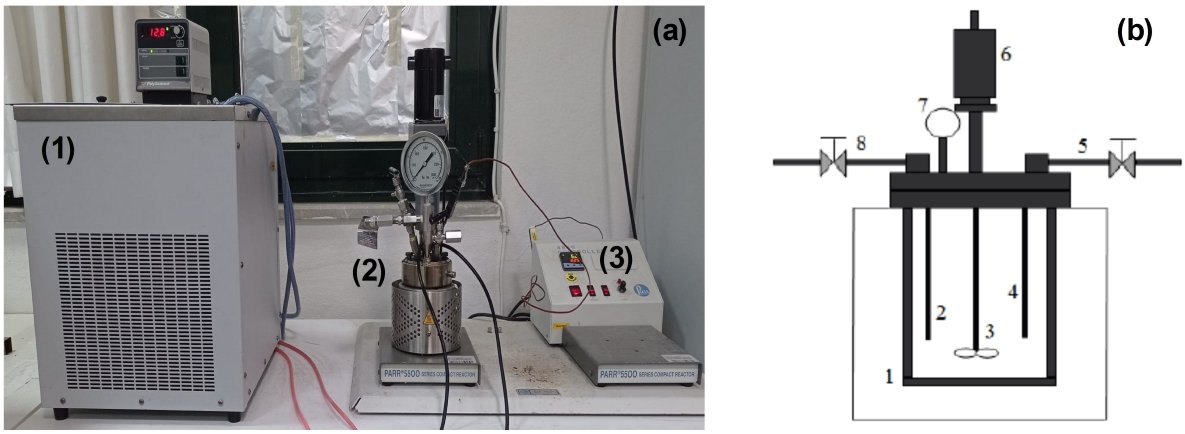

Figure 6. (**a**) Experimental Setup for subcritical wastewater treatment: (**a-1**) Chiller, (**a-2**) Batch reactor, (**a-3**) Heater; (**b**) Batch reactor for hydrothermal liquefaction of sugar industry wastewater: (**b-1**) stainless steel beaker, (**b-2**) thermocouple, (**b-3**) stirring impeller, (**b-4**) gas inlet, (**b-5**) input nitrogen gas, (**b-6**) magnetically driven stirrer, (**b-7**) pressure gauge, (**b-8**) gas sample collecting valve.

210, and 240 °C) and the reactions lasted for 1 and 2 hours. Additionally, the effect of NaOH over the removal of TOC was also examined using various concentrations of NaOH (0.01, 0.02, 0.04, and 0.1 M) at the optimum reaction temperature and duration. A chiller was used to keep the reaction temperature constant and to cool it to get samples at the end of reactions. The batch reactor was opened at almost 50 °C and the solid and liquid fractions were separated by filtration. The liquid samples were analyzed via TOC (Shimadzu TOC-Vcph TNM-1/SSM-5000 A) while FT-IR (Shimadzu FT-IR 8400S) was used to analyze the solid samples. Additionally, to identify the intermediate products of the hydrothermal liquefaction of sugar industry wastewater, GC-MS (Agilent 6890N/5973N Network, USA) was performed and thus, a reaction pathway could be developed for this process. In this analysis, helium (20 ml/min of flow rate) was used as the mobile phase and the detector was operated at 250 °C. In addition, the oven temperature program is 50 °C for 3 min, 100 °C (5 min with an increase of 50 °C/min), 200 °C (5 min with an increase of 50 °C/min), and 250 °C (7 min with an increase of 50 °C/min).

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

Keywords: Hydrothermal Treatment · Subcritical Hydrolysis · Sugar industry · Wastewater

[1] J. P. Kushwaha, *A Desalin. Water Treat.* **2013**, *53(2)*, 309–318.
[2] www.sekerkurumu.gov.tr.
[3] E. Alkaya, G. N. Demirer, *Biomass Bioenergy* **2011**, *35*, 32–39.
[4] F. Almohammed, H. Mhemdi, E. Vorobiev, *Appl. Energy.* **2016**, *162*, 49–57.
[5] C. Bellido, C. Infante, M. Coca, G. González-Benito, M. T. García-Cubero, *Bioresour. Technol.* **2015**, *190*, 332–338.
[6] B. Demirel, P. Scherer, *Biomass Bioenergy* **2008**, *32*, 203–209.
[7] B. R. Dhar, E. Elbeshbishy, H. Hafez, H.-S. Lee, *Bioresour. Technol.* **2015**, *198*, 223–30.
[8] G. Kars, Ü. Alparslan, *Int. J. Hydrogen Energy.* **2013**, *38*, 14488–14494.
[9] I. J. Ochuma, O. O. Osibo, R. P. Fishwick, S. Pollington, A. Wagland, J. Wood, J. M. Winterbottom, *Catal. Today* **2007**, *128*, 100–107.
[10] V. M. Daskalaki, E. S. Timotheatou, A. Katsaounis, D. Kalderis, *Desalination.* **2011**, *274(1-3)*, 200–205.
[11] B. Kayan, B. Gözmen, *J. Hazard. Mater.* **2012**, *201–202*, 100–106.
[12] L. Hadhoum, M. Balistrou, G. Burnens, K. Loubar, M. Tazerout, *Bioresour. Technol.* **2016**, *218*, 9–17.
[13] Z. S. Lee, S. Y. Chin, C. K. Cheng, *Heliyon.* **2019**, *5*, 01792.
[14] O. P. Sahu, V. Gupta, P. K. Chaudhari, V. C. Srivastava, *Int. J. Environ. Sci. Technol.* **2015**, *12(11)*, 3519–3530.
[15] O. P. Sahu, P. K. Chaudhari, *J. Electroanal. Chem.* **2015**, *739*, 122–129.
[16] O. Sahu, D. G. Rao, A. Thangavel, S. Ponnappan, *Int. J. Sustain. Eng.* **2018**, *11(1)*, 16–25.
[17] O. Sahu, B. Mazumdar, P. K. Chaudhari, *Int. J. Environ. Sci. Technol.* **2019**, *16(3)*, 1527–1540.
[18] O. Sahu, *Int. J. Ind. Chem.* **2019**, *10(4)*, 335–347.
[19] O. Sahu, *S. Afr. J. Chem. Eng.* **2019**, *28*, 26–38.
[20] V. Gupta, B. Mazumdar, N. Acharya, *Int. J. Energy Technol. Policy* **2017**, *13*, 177–187.
[21] G. Güven, A. Perendeci, A. Tanyolac, *Chem. Eng. J.* **2009**, *151(1-3)*, 149–159.
[22] S. Sharma, H. Simsek, *Chemosphere* **2020**, *238*,124669–124680.
[23] P. Asaithambi, M. Matheswaran, *Arab. J. of Chem.* **2016**, *9*, S981-S987.
[24] G. Ovejero, A. Rodríguez, A. Vallet, J. García, *Ind. Eng. Chem. Res.* **2012**, *51*, 11367–11372.
[25] S. D. Hosseini, F. S. Asghari, H. Yoshida, *Water Res.* **2010**, *44*, 1900–1908.
[26] G. Zhang, I. Okajima, T. Sako, *J. Supercrit. Fluids.* **2016**, *112*, 136–142.
[27] M. Enteshari, S. I. Martínez-Monteagudo, *Food Bioprod. Process.* **2018**, *111*, 104–113.
[28] E. Yabalak, *J. Environ. Chem. Eng.* **2018**, *6*, 7132–7137.
[29] E. Yabalak, *J. Environ. Sci. Health A* **2018**, *53:11*, 975–985.
[30] E. Yabalak, A. M. Gizir, *J. Environ. Sci. Health A* **2020**, *55*(12), 1424–1435.
[31] S.-A. Meng, J.-R. Bu, C.-M. Lin, *J. Indian Chem. Soc.* **2021**, *98(9)*, 100115–100121.
[32] A. Yuksel, M. Sasaki, M. Goto, *J. Hazard. Mater.* **2011**, *190*, 1058–1062.
[33] J. Zhang, S. Wang, Y. Guo, D. Xu, Y. Gong, X. Tang, *Color. Technol.* **2012**, *128*(4), 323–330.
[34] J. Li, S. Wang, Z. Jiang, C. Yang, H. Xu, In *IOP Conf. Ser.: Earth Environ. Sci.* **2019**, 233(5),052004-052010..
[35] O. Ö. Söğüt, M. Akgün, *J. Ind. Eng. Chem.* **2009**, *15*(6), 803–808.
[36] C. Wang, C. Wu, H. Zhang, J. Lai, X. Luo, Y. Liang, J. Tian, *Chemosphere.* **2021**, *278*, 130392–130401.

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