

Article

Electrocoagulation with Fe-SS Electrodes as a Fourth Stage of Tequila Vinasses Treatment for COD and Color Removal

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Abstract: The tequila industry faces several environmental challenges due to its high yields of contaminants, especially tequila distillation stillage or tequila vinasses, with ten to twelve liters produced per liter of tequila. All treatments aim to shorten retention times to avoid the need for large equipment or new facilities and the saturation of residues within tequila distilleries. The complexity of tequila vinasses has led to treatments with several stages, whereby most of the organic matter content is reduced, but the treatment range results are insufficient. This study aimed to evaluate a fourth-stage tequila vinasse treatment using an electrocoagulation system that uses inexpensive electrodes (SS cathodes and iron anodes), has a low electrical consumption, and applies low voltages in order to meet safety, economic, and environmental criteria so as to comply with Mexican norm NOM-001-SEMARNAT-2021. Three sets of voltage–amperage controllable power source, a 4 mm cylindrical 304 stainless-steel cathode, and a 9 mm iron anode with 200 mL samples in 250 mL beakers were used; three replicas (R1, R2, and R3) underwent 2 h treatment at 1–6 volts to evaluate the voltage effect and 1–6 h of 5-volt treatment to assess the time effect. All samples were filtered with 8 µm and 0.25 µm meshes. Chemical oxygen demand, pH, electrical conductivity, turbidity, and color measurements (SAC for λ 436, 525, and 620 nm) were taken. The experiments determined the optimal voltage and time, considering a hydraulic retention time below 6 h. The results show that electrocoagulation of pretreated tequila vinasses effectively helps in the final removal of organic matter measured as COD, reaching values below 150 COD mg/L at 5–6 h with 5 V treatments and color reduction with 5 V, 1 h treatment. This leads to final polishing that complies with the Mexican wastewater discharge norm criteria.

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1. Introduction

1.1. Tequila

Tequila, Mexico's most representative spirit beverage, is produced primarily in Jalisco. Tequila production in Mexico is a significant industry, with the Agave tequilana Weber blue variety being a national icon and the only species cultivated for tequila production.

The plant faces challenges, such as phytoplasma infections, which affect its availability for tequila production [1].

The tequila market in Mexico has seen growth [2]; exports from Mexico have also been significant. In 2023, sales of tequila produced in Mexico reached a value of over MXN 70 billion (USD 3.5 billion), indicating the popularity and economic importance of the industry [3]. The COVID-19 pandemic has impacted the tequila market in the US, though recovery has been seen in the following years [4], with a yearly production peak in 2022 of 651.4 million liters of tequila [5]. Overall, tequila production in Mexico has a rich history and is a significant part of the country's culture and economy.

Tequila production involves several residues or by-products, including agave leaves, agave bagasse, bitter syrup, and primarily tequila vinasse (also known as tequila stillage). Tequila vinasse is a mixture of up to six distillation residual streams composed mainly of tequila distillation first-stage stillage, and it is the leading environmental challenge for the tequila industry [6]. Tequila vinasse yields reach ten to twelve liters per liter of tequila produced [7]. Tequila vinasse treatment includes several methods to reduce its environmental impact. Furthermore, a revised and stricter version of Mexican norm NOM-001-SEMARNAT-2021, "which establishes the permissible limits of contaminants in wastewater discharges into receiving bodies owned by the nation," [8] is a current legal obligation for all industries, resulting in the need for technological changes in tequila vinasse treatment.

1.2. Tequila Vinasse Characterization

Tequila vinasses are dark-colored wastewaters with low pH levels (3–5) and very high chemical oxygen demands (CODs) (60,000–100,000 mg/L) due to their organic matter content, with total solids contents in the range of 25,000–50,000 mg/L [7]. Regarding biodegradability, the BOD/COD ratio is 0.53–0.59 [9]. See Table 1 for an analysis of the main parameters.

Table 1. Tequila vinasse characterization.

Parameter	Value 1 [7]	Value 2 [9]
pH	3.4–4.5	3.6–4.6
Total COD (mg/L)	60,000–100,000	41,740–67,970
Total BOD (mg/L)	35,000–60,000	16,162–35,102
Total dissolved solids (mg/L)	23,000–42,000	4550–6750

These vinasses can be converted into valuable products, such as biodegradable films, or used as a nutrient source for xylitol production [10–12]. The characteristics of vinasses can vary depending on the tequila production process, with differences observed between processes that involve cooking agave cores and those that do not [13]. Anaerobic digestion of vinasses from the fermentation of Agave tequilana Weber has been studied, with thermophilic anaerobic digestion showing increased toxicity and sensitivity to environmental conditions [14,15]. Due to their specific characteristics, tequila vinasses can also be utilized as soil amendments for crops like agave, sugarcane, and sugar beet [16].

1.3. Environmental Normative Update and Tequila Industry Challenges

The changes to norm NOM-001-SEMARNAT-2021 have significant implications for various industries in Mexico. The new wastewater standard, which took effect on 11 March 2023, replaced the previous standard, NOM-001-SEMARNAT-1996 [17], and includes parameters for activities in rivers, natural bodies of water, and coastal waters [8]. This update is part of SEMARNAT's efforts to improve environmental regulations and

promote sustainable practices; the standard includes criteria for classifying wastes requiring special handling, emphasizing the importance of green supply chains, and reducing environmental impact [18]. In the context of wastewater treatment, compliance with the new standard is crucial. A study comparing treated wastewater characteristics with the NOM-001-SEMARNAT-2021 Mexican standards highlighted the importance of meeting the updated requirements. Furthermore, recent updates to the Climate Case Charts in February 2024 included references to NOM-163-SEMARNAT-ENER-SCFI-2013 [19], indicating the interconnectedness of environmental regulations in Mexico [20]. Changes to trade standards, such as eliminating exemptions to labeling requirements, also reflect the government's efforts to enhance compliance with environmental norms [21]. The changes to norm NOM-001-SEMARNAT-2021 signal a shift towards more stringent environmental regulations in Mexico. Businesses operating in the country need to adapt to these changes to ensure compliance and contribute to sustainable practices [22]. SEMARNAT's initiatives, including the General Law on Climate Change, highlight the government's commitment to addressing environmental challenges and promoting the use of ecological technologies [23].

The changes in NOM-001-SEMARNAT-2021 and the guidelines imply a shift in the rules for holders of a Discharge Permit, with technical standards being updated [8]. The main changes in NOM-001-SEMARNAT-2021 include modifications to Section 2, "Normative References", to update the list of Mexican standards [18]. Compliance with the requirements of NOM-001-SEMARNAT-2021 is mandatory; the standard also includes a Voluntary Compliance Program [22]. The standard aims to limit wastewater pollutants and protect the environment in Mexico [21]. The standard aims to update regulations and protect the environment from harmful pollutants [20]. Compliance with the standard is essential for holders of Discharge Permits to ensure the proper management of wastewater in Mexico [8].

This technical standard has implications for holders of Discharge Permits, signaling a shift in the rules governing wastewater discharge in the country [24]. One of the most notable changes introduced by NOM-001-SEMARNAT-2021 is the replacement of NOM-001-SEMARNAT-1996, signifying a departure from the previous standards and guidelines [25]. The new norm includes updated Tables of Permissible Limits, reflecting changes in permissible levels of pollutants in wastewater discharge [25]. The deadline for compliance with the requirements of NOM-001-SEMARNAT-2021 has been set for March 11, 2023, indicating the urgency and importance of adhering to the new regulations [26] and further emphasizing the shift towards updated standards and criteria for environmental protection [8]. NOM-001-SEMARNAT-2021 represents a significant step towards enhancing environmental protection and sustainability in Mexico. The publication of updated standards and guidelines reflects a commitment to addressing emerging pollutants and promoting green supply chains in the country [18,27]. By implementing these changes, Mexico aims to improve the management of wastewater discharge and ensure the protection of its natural resources for future generations. See Table 2 for the main changes in the discharge of waters to rivers and water bodies in Mexico.

With respect to international norms, there is still room for improvement. The current Mexican norm requires 150 mg/L COD (approximately related to 75–80 mg/L BOD₅), whereas the EPA (EE.UU., 40 CFR 133) and the UE (91/271/CEE) establish 30 and 25 mg/L, respectively. For SST, the same norms require up to 30 and 35 mg/L, respectively, and are therefore stricter than the Mexican norm [28,29].

Table 2. Changes in Mexican norm NOM-001-SEMARNAT-2021.

Parameters	Unit	NOM-001-SEMARNAT-1996 MPL	NOM-001-SEMARNAT-2021 MPL
Temperature		40 °C	35 °C
Biological Oxygen Demand (BOD ₅)	mg/L	30–150	Deletion
Oils and Fats	mg/L	15	15
Settleable Solids	mL/L	1	Deletion
Total Suspended Solids	mg/L	40–150	20–60
Floating Matter		Absent	Deletion
Chemical Oxygen Demand (COD)	mg/L	Not required	100–150
Total Organic Carbon	mg/L	Not required	25–38
Total Nitrogen	mg/L	15–40	15–25
Total Phosphorous	mg/L	5–20	5–15
pH		5–10	6–9
True Color	SAC	Not required	436 nm/7.0 m ^{−1} max 525 nm/5.0 m ^{−1} max 620 nm/3.0 m ^{−1} max
Acute Toxicity	UT	Not required	2 (15 min exposure)
Arsenic	mg/L	0.1–0.2	0.1–0.2
Cadmium	mg/L	0.1–0.2	0.1–0.2
Cyanides	mg/L	1.0–2.0	1.0
Copper	mg/L	4.0	4.0
Chrome	mg/L	0.5–1.0	0.5–1.0
Mercury	mg/L	0.005–0.01	0.005–0.01
Nickel	mg/L	2.0	2.0
Lead	mg/L	0.2–0.5	0.2
Zinc	mg/L	10.0	10.0

1.4. Tequila Vinasses First-Stage Treatment

Tequila vinasses are challenging by-products to degrade due to their complex composition and high organic load concentrations [30], high turbidity, suspended solids contents [31], and color compounds [32]. The composition of the yeast community in tequila vinasses also impacts their treatment [33]. Furthermore, the generation and characterization of tequila vinasses have been explored with a focus on disposal and treatment problems in various regions [10]. Efforts have been made to reduce the organic load concentrations in tequila vinasses through various treatment methods, including flocculation studies [34].

López-López and Contreras Ramos [9] reported in 2015 that first-stage treatments aimed to remove suspended solids and regulate pH and temperature. The processes used were sedimentation ponds, sedimentation pools, dissolved air flotation, coagulation–flocculation, and neutralization. Cooling was achieved over time through the use of dedicated equipment or receptors. See Table 3 for the advantages and disadvantages of these technologies.

Table 3. Tequila vinasses first-stage treatment technologies.

Treatment	Advantages	Disadvantages
Neutralization	4–6 g/L CaCO ₃ Complies with requirement of pH levels of 6.5–8.0	Neutralization or pH adjustment must be performed after each treatment phase
Sedimentation Ponds	>80% sedimented solids removal	Organic matter concentration remains above 90%. Improper preparation leads to soil and subsoil pollution

Table 3. Cont.

Treatment	Advantages	Disadvantages
Sedimentation Pools	>80% sedimented solids removal	Organic matter concentration remains above 90%
Dissolved Air Flocculation (DAF)	>80% sedimented solids removal	Dissolved solids and biological oxygen demand have not decreased significantly
Coagulation–Flocculation	5 mg/L $\text{Al}_2(\text{SO})_4$ + 1.5 mg polymer 20–30% suspended solids and colloidal solids removal	Low chemical and biological oxygen demands reached

1.5. Tequila Vinasses Second-Stage Treatment

Over time, due to regulatory updates and increased production, pollution related to tequila distilleries has become more significant within society. Solid concentrations and chemical and biological oxygen demands were not adequately addressed with first-stage treatments, necessitating the incorporation of second-stage treatments. Several distilleries lacked wastewater treatment plants, indicating financial or technical constraints and, in some cases, insufficient space within the distillery facilities. Various treatment processes, including biological anaerobic and aerobic processes, have been implemented to treat tequila vinasses in the second stage, but their effectiveness varies, with discussions being conducted on the efficacy of different systems and technologies to meet environmental regulations in Mexico [7], and the need for proper treatment and disposal methods to prevent pollution of surface water bodies remains [10,35]. Further research and innovative approaches are still needed to manage and utilize this by-product of the tequila industry effectively [10,36,37].

Researchers are exploring various methods to regulate organic pollution levels in anaerobic digesters [38]. Dias et al. focused on the application of vinasses over time, highlighting the anaerobic treatment of tequila vinasses in a CSTR-type digester [39]. Mendiola-Rodríguez et al.'s study focused on anaerobic digestion for tequila vinasses treatment, aiming to reduce organic contents in wastewater while producing biogas [40]. Estrada-Arriaga et al. showed enhanced methane production and organic matter removal from tequila vinasses through specific treatment processes [41]. Silva-Martínez et al. pointed out that in Mexico, anaerobic treatment of tequila vinasses has been explored as a method for waste-to-energy conversion [42].

García-Depraet et al. indicated that the addition of lime can enhance the anaerobic digestion of tequila vinasses, although the difficulty in dissolving lime poses a challenge [43]. In another study by García-Depraet et al., they pointed out that despite the challenges posed by tequila vinasses, there is potential for their utilization in biohydrogen production and as a source of energy [44]. López-López et al. [9] provided a summary of anaerobic and aerobic technologies, which is summarized in Table 4. While anaerobic processes are capable of handling high COD values, aerobic treatment is recommended only for BOD levels lower than 3000 mg/L, noting that the BOD/COD ratio for tequila vinasses is close to 0.5.

Researchers have also optimized the treatment of alcohol vinasse using a combination of advanced oxidation processes with synthesized $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles [45]. A study on color compound removal from tequila vinasses introduced silica gel for treatment, along with advanced oxidation processes and coagulation methods [32]. Metabolic modeling tools have been utilized to produce aroma compounds from tequila vinasses, marking a novel approach in this area of research [46]. The sustainability of the tequila industry has also been a focus, with efforts having been made to address environmental concerns such as vinasse treatment, non-deforestation, and carbon capture by agave plants [47].

Table 4. Tequila vinasses second-stage treatment technologies.

Treatment	Type of Reactor	COD Removal
Anaerobic	UASB reactor	70–85%
	Anaerobic filter	65%
	UASB reactor + anaerobic filter	50–70%
	UASB without recirculation	36%
	UASB with recirculation	74%
	Fluidized bed	70%
Aerobic	CSTR (batch or continuous)	90–95%
	Activated sludge	5–10%
	Extended aeration	5–10%
	Aeration pools/ponds	5–10%
	SBR	5–10%

1.6. Tequila Vinasses Third-Stage Treatment

From second-stage treatment, effluents remove the main COD fraction, leaving residues (total suspended solids (TSS) and total dissolved solids (TDS)) that still need to be purged. For this purpose, coagulation–flocculation, sedimentation, and/or filtration reduce the COD from a value around 1000–3000 mg COD/L to 600–800 mg COD/L. Several works have reported this treatment, and it has also been noted that color is reduced. Coagulation–flocculation can reduce suspended solids, color, and COD in tequila vinasses by up to 99.4%, 86.0%, and 70.0%, respectively [48]. Biopolymers can serve as coagulants in tequila vinasses treatment. Ferral-Pérez et al. [49] reported chitosan as the most efficient biopolymer, as it removed up to 84.0% COD. In 2024, Zurita et al. [50] reported results with coagulation–flocculation using a natural coagulant, achieving an apparent color removal of 98%. Castillo-Monroy et al. reported a study on a coupled adsorption–electro-oxidation process as a tertiary treatment for tequila industry wastewater [51].

1.7. Tequila Vinasses Fourth-Stage Treatment

To remove the remaining color and COD in tequila vinasses and to comply with Mexican wastewater norms, several treatments have been proposed; these include advanced oxidation processes (AOPs), such as ozonation, electro-oxidation, photocatalytic oxidation, and electrocoagulation.

1.8. Electrocoagulation as Fourth-Stage Treatment

Electrocoagulation is a widely used method for wastewater treatment that involves the application of voltage to generate electrochemical reactions. Recent studies have shown that electrocoagulation effectively removes organic matter, usually measured as COD, BOD₅, total suspended solids (TSS), and color.

It is essential to note that contamination levels vary across every tequila distilleries, and the selected pretreatments used are not typically reported on an industrial scale, this information being kept as confidential as possible. It would be better if all cases, whether successes or failures, were reported to the environmental authority to converge on suitable technologies faster and more economically.

On the bright side, electrocoagulation is a process that involves low voltages and low electrical energy use. On the other hand, selecting anodes that produce low- or non-contaminating sludge is wise to avoid further pollution or complex treatment.

Considering the above criteria, cathodes can be made from several materials, typically using alloys that corrode less easily than those used for anodes. However, using anodes made from stainless steel, aluminum, and most alloys is not the right choice. The predominant soil in the Jalisco Highlands region is luvisol, at 34.6%; characterized by the

accumulation of clay, it is a reddish soil (high in iron) mainly used for agriculture, with moderate yields, and has a high susceptibility to erosion [52]. Thus, selecting iron anodes that produce iron oxides is a pertinent and environmentally safe choice.

A system with low electrical resistance, low electricity consumption, and cheaper and more suitable cathodes and anodes should be selected. These were the criteria for the current research, which aimed to evaluate the most appropriate electrocoagulation processes, economically and environmentally, for pretreated vinasses produced by the tequila industry.

The Environmental Protection Agency (EPA) does not mandate public water systems to act on iron as a contaminant, indicating that it is not considered a significant health risk [53]. Iron is also referenced in the context of cosmetic effects in water quality standards, suggesting that it may cause aesthetic issues rather than health concerns [54]. Furthermore, iron is not deemed hazardous in the quantities utilized for disinfection in drinking water [55]. This distinction between iron as a cosmetic parameter rather than a contaminant aligns with the FDA's regulation of cosmetics, according to which contaminants such as lead are considered more serious concerns [56]. The extant literature suggests that iron is not typically classified as a contaminant in drinking water due to its cosmetic rather than health-related effects.

Previous works on tequila vinasses using advanced oxidation processes to comply with NOM-001-SEMARNAT-2021 include that by Carrillo Monroy et al. [51] in 2021. Prior to the establishment of this norm, a novel study that complied with it was presented by Jiménez López et al. [57] in 2016. Carrillo Monroy et al. [51] reached a low COD of 33–78 mg/L with a system using a coupled adsorption–electro-oxidation process with a very low cost (12.5–20.0 A/m²). Jiménez-López reported electrocoagulation using a 1.2 L stirred reactor with aluminum electrodes using 74.28–148.57 A/m², reaching COD values below 150 mg/L in 90 min.

The use of different electrode materials in electrocoagulation for stillage treatment has been investigated, with Al-Al, Al-Fe, and Al-SS combinations showing varying effectiveness. Al-Al electrodes demonstrate high efficiency in removing organic compounds and suspended solids but may introduce aluminum into the treated water. Al-Fe combinations offer a balance between removal efficiency and environmental impact, as iron is generally considered less harmful. Al-SS (stainless-steel) electrodes provide durability and resistance to corrosion, potentially extending the lifespan of the treatment system. The choice of electrode material impacts not only the treatment efficiency but also the composition of generated sludge and potential environmental implications. Factors such as pH, electrical conductivity, and specific contaminants in the stillage influence the performance of these electrode combinations, necessitating careful consideration in system design and operation [58,59].

Regarding previous works that used aluminum anodes, Jiménez-López [57] presented a work that aimed to polish pretreated tequila vinasses in compliance with the current Mexican norm, leading to a COD below 150 mg/L (94.41% removal), with a specific energy consumption of 18.75 kWh/m³ in 180 min. Montaña-Saavedra et al. [60] reported a 20% reduction in total dissolved solids contained in pretreated vinasses within 3 h. Dubey et al. [61] reported that in pretreated vinasses, the COD was reduced by 85.1% in half the time, 93 min. Prajapati et al. [62], in a study on distillery wastewater, reported that a 93% COD reduction was achieved in a highly efficient arrangement requiring 31.4 kWh/m³.

Khandegar et al. [63] reported several electrode combinations (Al-Al, Al-Fe, and Fe-Fe) using high-COD distillery spent wash with a higher surface electric current and obtained their best results with the Al-Al combination. Alizadeh et al. [64] also worked

with electrodes made of Al-Fe, using vinasse effluents, which achieved an 80.8% COD reduction in 45 min.

Regarding Fe anodes, Syaichurrozi et al., in 2020 [65], obtained a COD reduction of 51.67% in 6 h; in another work, in 2022 [66], they reached 67.62% COD removal with an 8 h treatment; and, finally, in a third paper, in 2023 [66], they analyzed the effects of agitation with poorer results. Finally, David et al. [67] reported a color removal of up to 83.75%, without noting a reduction in COD.

The work with the most similarities to the present research regarding objectives was the one reported by Jiménez-López [57]; he reported two experiments where Al concentration was measured: (a) Experiment 24 (223.85 A/m², 1.5 A, pH 5.0, 40 °C, 96.25% color removal): initial concentration: 0.05 mg/L, final concentration: 0.971 mg/L; (b) Experiment 26 (74.28 A/m², 0.5 A, pH 7.0, 40 °C, 86.00% color removal): initial concentration: 0.048 mg/L, final concentration: 0.484 mg/L. In general, he reached up to 2.1784 g Al anode consumption for 223.85 A/m² and an average of 0.6820 g Al anode consumption for 74.28 A/m² (similar to our research). Optimal conditions reported a DQO removal of 94.41% COD with a pH of 5.0, at 40 °C, 148.57 A/m², with a 0.9224 g Al anode consumption. All treatments lasted for 180 min.

References indicate that there is still considerable work to be done, considering COD values from untreated to significantly diluted vinasses. Energy consumption varies, and, in general, not all works report the same parameters. A higher COD removal above 90% was reported, and the pH increased with the process. Energy consumption is generally reported at values below 31.4 kWh/m³, except for one treatment, and the time range also varies from 45 to 480 min. See Table 5 for reported electrocoagulation systems.

Table 5. Previous works on electrocoagulation of vinasses.

Treatment	Electrode Material	Initial Condition	Highest Removal	Surface Electric Current A/m ²	Specific Energy Consumption kWh/m ³	Ref.
Pretreated tequila vinasses pH: 5 40 °C 180 min No stirring	Al	COD 304 mg/L	94.41%	148.57	18.75	[57]
Sugarcane vinasse 3 h 430 rpm	Al	TDS: 6810 ± 840 mg/L; TSS: 5200 ± 300 mg/L	TDS: 20% TSS: 96%	61	13.5	[60]
Distillery effluent 93 min pH: 3.5–9.5	Al	COD: 5.15 g/L	COD: 85.1%	135	NA	[61]
Distillery wastewater 0–30 V 1.5 L 2 h 0–5 A	Al	COD: 13.8 g/L	COD: 93%	89.3	31.4	[68]
Distillery spent wash 300 mL 2 h pH: 3 500 rpm	Al-Al Al-Fe Fe-Fe	COD: 120 g/L	COD: Al-Al: 73.3% Al-Fe: 60% Fe-Fe: 46.6%	1870	NA	[63]

Table 5. Cont.

Treatment	Electrode Material	Initial Condition	Highest Removal	Surface Electric Current A/m ²	Specific Energy Consumption kWh/m ³	Ref.
Vinasse effluents 1 A pH: 7 45 min	Al-Fe	COD: 46,550 mg/L	COD: 80.8%	167	NA	[64]
Vinasse waste from the bioethanol industry 0, 250, 500 rpm T 301.65 °K pH: 4.1 12.6, 12.3, 12.4 V Volume: 1 L	Fe	COD: 113.70 g/L	67.62%	NA	NA	[66]
Vinasse residues 12.5 V 200 rpm pH: 6 105–110 °C 6 h	Fe	COD: 100.16 g/L	51.67%	1021.41	264.75	[65]
Distillery wastewater pH: 6 2.98 A 10 V Time: 1 h 200 rpm	Fe	COD: 100.16 g/L	COD: 13.96%	1045.6	29.8	[69]
Distillery Effluent 100 rpm; Time: 2 h pH: 3–9 0.5, 1, 1.5, 1.9 A	Fe	COD: 140 g/L	Color: 83.75%	NA	NA	[67]

SS: stainless steel; TSS: total suspended solids; TDS: total dissolved solids; COD: chemical oxygen demand; NA: not available; Ref: reference.

1.9. Objective

This study aimed to evaluate the efficacy of using stainless-steel cathodes and steel anodes in electrocoagulation as a fourth-stage treatment method for tequila vinasses in compliance with the new regulatory standards in Mexico.

2. Materials and Methods

2.1. Equipment

Three kits, consisting of an Extech model 382213 power source (Teledyne FLIR, Thousand Oaks, CA, USA) with voltage–amperage control, a simple bipolar electrochemical cell configuration with a stainless-steel 304 (SS) rod with a 4 mm diameter as a cathode, and an ASTM A615/A615M iron rod with a 9 mm diameter as an anode connected with wires and attached to clamps on both sides, mounted on a laboratory stand with clamps, as shown in Figure 1, were used. Then, 200 mL samples were placed in 250 mL glass beakers. The anodes were sunk 6.7 cm with a surface area of 9.06 cm² for the SS cathode and 19.58 cm² for the iron anode. The electrode separation was measured at 2.5 cm from the respective outer diameters. The electrolyte temperature remained between 18 and 23 °C.



Figure 1. Electrocoagulation set used.

2.2. Pretreated Tequila Vinasses

Third-stage pretreated tequila vinasses were used (see Table 6 for their characterization), conserved at room temperature (18–23 °C). These tequila vinasses underwent pretreatment in the first stage, involving suspended solids removal through 40-mesh sieves; in the second stage, pH regulation was applied for both anaerobic and aerobic treatment, as well as clarification; in the third stage, treatment included coagulation–flocculation, sedimentation, and filtration.

Table 6. Third-stage pretreated tequila vinasses characterization.

Parameter	Units	Norm	Result
pH	[-]	6.5–8.0	7.81
COD	mg/L	<150	814
TDS	mg/L		3.577
Electrical conductivity	mS/cm		7.299
Color			
Abs 436 nm	m ⁻¹	7.0	34.7
Abs 525 nm	m ⁻¹	5.0	10.4
Abs 620 nm	m ⁻¹	3.0	3.7

Two independent variables were considered, time (in hours) and voltage (in volts), in the experiments. Time values were selected as 0, 1, 2, 3, 4, 5, and 6 h, given the expectation of relatively short process times for industrial applications; higher times would lead to the need for bigger equipment due to the high yield of tequila vinasses. Considering the data review, voltage values also ranged from 0 to 6 volts, covering 1, 2, and 5 volts for typical electricity commercial equipment; furthermore, higher voltage values could have increased the temperature of the samples, causing energy loss in the process.

2.3. Methods

2.3.1. Analytical Methods

The methods used for assessment of the above parameters were as follows: (a) pH: Standard Method for the Examination of Water and Wastewater 4500-H+B: pH in Water by Potentiometry [70]; (b) COD: Method 410.4, Revision 2.0: The Determination of Chemical Oxygen Demand by SemiAutomated Colorimetry [71]; (c) Total Dissolved Solids and Electrical conductivity: Standard Method for the Examination of Water and Wastewater 2510 Conductivity (2017) [72]; (d) Color: ISO 7887:2011—Examination and determination of colour [73]; (e) Turbidity: Standard Method for the Examination of Water and Wastewater 2130 Turbidity (2017) [74].

2.3.2. Calculations

Electric Power:

$$P = UI \quad (1)$$

P : Electric power (W).

I : Current intensity (A).

U : Applied voltage (V).

Surface electric current:

$$SC = \frac{I}{A} \quad (2)$$

SC : Surface electric current (A/m^2).

I : Current intensity (A).

A : Electrode effective area.

For industrial applications, energy consumption is a critical parameter for determining costs. Specific energy consumption (SEC) refers to the energy needed for organic matter, such as COD, ammonia nitrogen, colorant, and others [59].

Specific energy consumption per volume:

$$SEC1 = \frac{UIt}{1000V} \quad (3)$$

$SEC1$: Specific energy consumption per volume unit (kWh/m^3).

U : Applied voltage (V).

I : Current intensity (A).

t : Electrolysis time (h).

V : Volume of treated wastewater (m^3).

This specific energy consumption can be assessed in the following manner:

$$SEC2 = \frac{UIt}{1000(COD_i - COD_e)V} \quad (4)$$

$SEC2$: Specific energy consumption per removal unit ($kWh/kg\ COD$).

U : Applied voltage (V).

I : Current intensity (A).

t : Electrolysis time (h).

V : Volume of treated wastewater (m^3).

COD_i : Chemical oxygen demand before treatment (kg/m^3).

COD_e : Chemical oxygen demand after treatment (kg/m^3).

Faraday's law is used to calculate the material decay of an electrode [59].

$$C = \frac{ItM}{1000zFV} \quad (5)$$

C: Electrode consumption (kg/m³)

I: Current intensity (A).

t: Electrolysis time (s).

M: Molar mass of electrode consumed (g/mol).

z: Electron transfer number.

F: Faraday constant (96,487 C/mol).

V: Volume of treated wastewater (m³).

Faraday efficiency:

$$FE = \frac{QnF}{It} \quad (6)$$

FE: Faraday efficiency.

Q: COD moles.

n: Molar electron transfer number.

F: Faraday constant (96,487 C/mol).

I: Current intensity (A).

t: Electrolysis time (s).

The operating cost will be the sum for energy consumption and electrode consumption, in USD/m³.

$$OC = EC + ED \quad (7)$$

OC: Operating cost (USD/m³).

EC: Energy cost (USD/m³).

ED: Electrode consumption (decay) cost (USD/m³).

2.4. Experimental Design

Data were collected by conducting experiments with three replicates (R1, R2, and R3) for each time (0, 1, 2, 3, 4, 5, and 6 h) and voltage setting (0, 1, 2, 3, 4, 5, and 6 volts), specifically by measuring the changes in chemical oxygen demand (COD) content, pH, electrical conductivity (EC), total dissolved solids (TDS), turbidity, and color (over voltages with 2 h treatment and over time with 5-volt treatment). Also, the mean electrical current consumption was measured.

3. Results

3.1. Effect of Voltage

Table 7 shows the electrical current density values for the reported voltages, considering initial and final electrical currents.

Table 7. Electrical current density for reported data.

Voltage V	Initial Electrical Current, Amp I ₁	Final Electrical Current, Amp I ₂	Electrical Current Density, Amp/m ² ECD
1	0.02	0.02	10.2
2	0.06	0.06	30.6
3	0.08	0.08	40.9
4	0.14	0.14	71.5
5	0.16	0.15	79.2
6	0.18	0.17	89.4

The first parameter considered is the chemical oxygen demand (COD), which showed good progress in reducing organic matter content, reducing it from 814 to 227 mg/L;

however, the latter value is still above the MPL of 150 mg/L requested by the Mexican norm. See Table 8 and Figures 2 and 3.

Table 8. COD removal results with 2 h treatment and variable voltage.

Voltage, V	COD, mg/L		
	R1	R2	R3
0	814	814	814
1	594	553	614
2	489	415	365
3	367	410	318
4	331	268	355
5	247	303	277
6	227	242	235

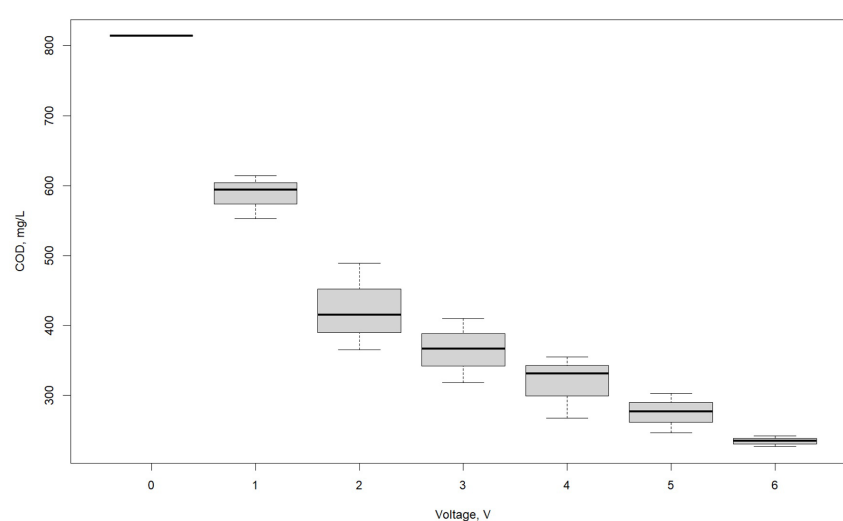


Figure 2. COD vs. voltage with 2 h treatment.



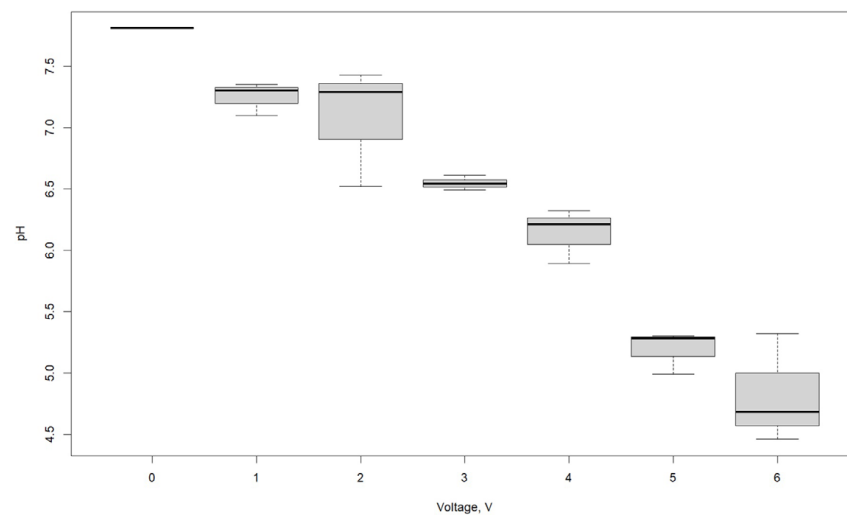
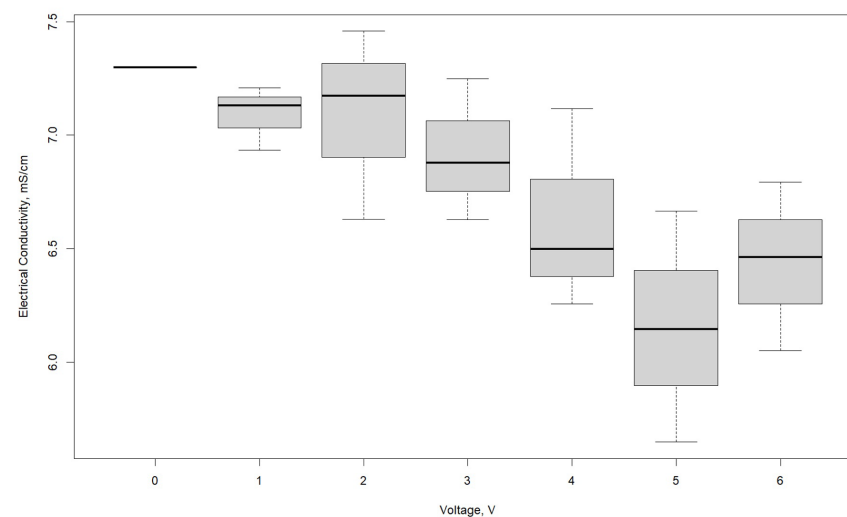
Figure 3. Voltage effect for 2 h treatments. From left to right: pretreated vinasses, 1 V, 2 V, 3 V, 4 V, 5 V, 6 V, and pure water.

Regarding pH, from the third-stage treatment, its value decayed from 7.81 to 4.46 with increasing voltage, though the latter is also outside the range permitted by the Mexican norm. See Table 9 and Figure 4.

Concerning electrical conductivity (EC), the third-stage treatment value started at 7.299 mS/cm and dropped to 5.649 with increasing voltage. The Mexican norm does not cover this parameter; however, it is related to ions and suspended particles; thus, a decrement is expected. See Table 10 and Figure 5.

Table 9. pH results with 2 h treatment and variable voltage.

Voltage, V	R1	pH R2	R3
0	7.81	7.81	7.81
1	7.35	7.30	7.10
2	7.43	6.52	7.29
3	6.54	6.61	6.49
4	6.32	6.21	5.89
5	4.99	5.28	5.3
6	4.46	4.68	5.32

**Figure 4.** pH vs. voltage with 2 h treatment.**Figure 5.** EC vs. voltage with 2 h treatment.

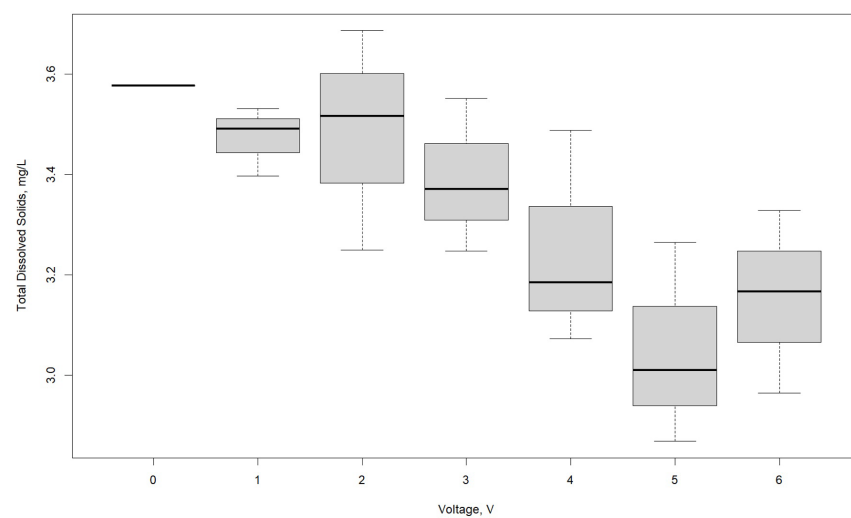
Total dissolved solids (TDS) was measured by the electrode method; the third-stage pretreated vinasses value started at 3.577 mg/L, and the value changed with increasing voltage to 2.869 mg/L. Mexican norms do not have a specific limit for this parameter, but its decrement was expected too. See Table 11 and Figure 6.

Table 10. EC results with 2 h treatment and variable voltage.

Voltage, V	EC, mS/cm		
	R1	R2	R3
0	7.299	7.299	7.299
1	7.130	6.933	7.208
2	7.459	7.174	6.631
3	6.628	7.248	6.879
4	7.117	6.258	6.498
5	5.649	6.666	6.145
6	6.463	6.793	6.051

Table 11. TDS results with 2 h treatment and variable voltage.

Voltage, V	TDS, mg/L		
	R1	R2	R3
0	3.577	3.577	3.577
1	3.491	3.397	3.532
2	3.687	3.516	3.25
3	3.248	3.552	3.371
4	3.488	3.073	3.185
5	2.869	3.265	3.011
6	3.167	3.329	2.965

**Figure 6.** TDS vs. voltage with 2 h treatment.

Turbidity also decreased, from an initial value of 1.50 NTU to 0.62 NTU. See [Table 12](#) and [Figure 7](#).

Table 12. Turbidity results with 2 h treatment and variable voltage.

Voltage, V	Turbidity, NTU		
	R1	R2	R3
0	1.50	1.50	1.50
1	1.41	1.17	1.42
2	1.03	1.05	1.04
3	0.94	0.96	0.94
4	0.80	0.72	0.82
5	0.79	0.67	0.78
6	0.75	0.62	0.62

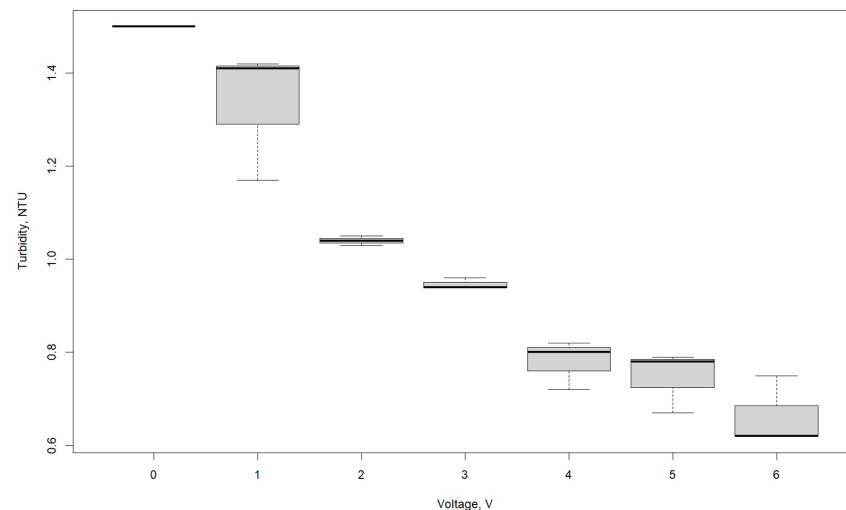


Figure 7. Turbidity vs. voltage with 2 h treatment.

The Mexican norm considers three parameters for color measurement, the first one with a 436 nm wavelength. The maximum permitted limit (MPL) for the spectral absorption coefficient (SAC) is 7 m^{-1} . SAC values obtained with 4, 5, and 6 volts in 2 h treatment comply with the norm. See Table 13 and Figure 8.

Table 13. SAC λ 436 nm with 2 h treatment and variable voltage.

Voltage, V	SAC λ 436 nm, m^{-1}		
	R1	R2	R3
0	34.7	34.7	34.7
1	24.4	25.6	21.2
2	11.2	9.6	6.3
3	6.9	7.5	5.1
4	4.6	4.7	3.9
5	4.4	3.2	4.5
6	4.4	3.1	3.8

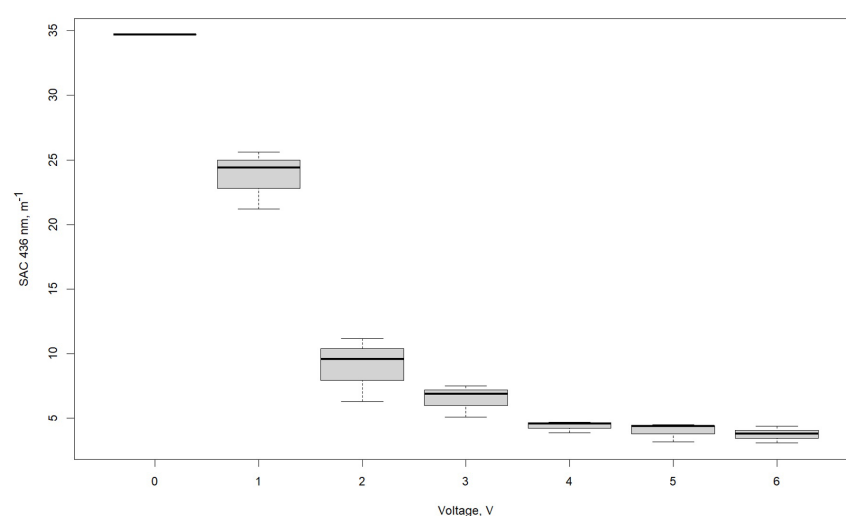
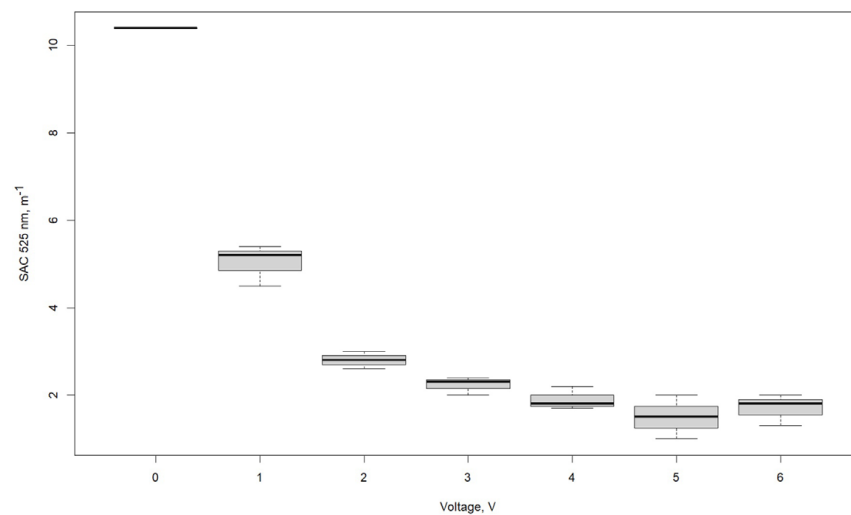


Figure 8. SAC λ 436 nm vs. voltage with 2 h treatment.

The second color parameter is the 525 nm wavelength. The MPL for this SAC is 5 m^{-1} . SAC values obtained with 2, 3, 4, 5, and 6 volts with 2 h treatment comply with the norm. See Table 14 and Figure 9.

Table 14. SAC λ 525 nm with 2 h treatment and variable voltage.

Voltage, V	SAC λ 525 nm, m^{-1}		
	R1	R2	R3
0	10.4	10.4	10.4
1	5.4	4.5	5.2
2	2.8	2.6	3.0
3	2.3	2.4	2.0
4	1.8	1.7	2.2
5	1.0	1.5	2.0
6	2.0	1.8	1.3

**Figure 9.** SAC λ 525 nm vs. voltage with 2 h treatment.

The third color parameter is the 620 nm wavelength. The MPL for this SAC is 3 m^{-1} . SAC values obtained with all voltages applied in 2 h treatment comply with the norm. See Table 15 and Figure 10.

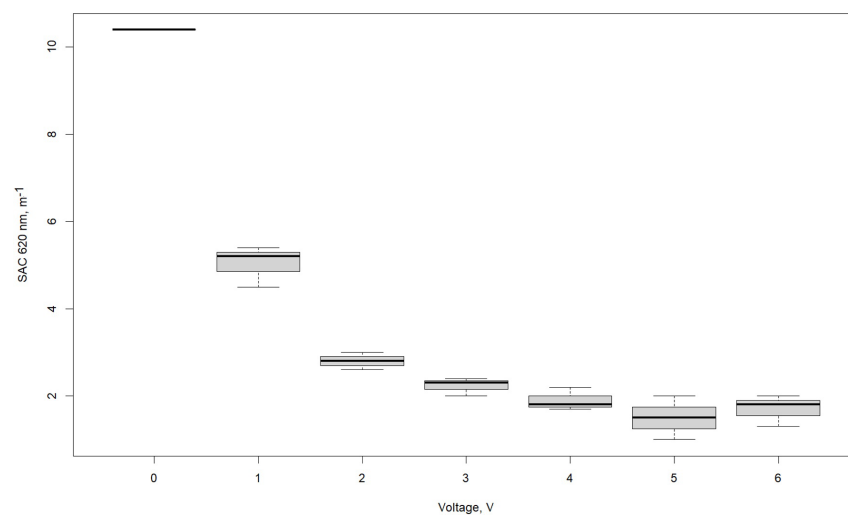
**Figure 10.** SAC λ 620 nm vs. voltage with 2 h treatment.

Table 15. SAC λ 620 nm with 2 h treatment and variable voltage.

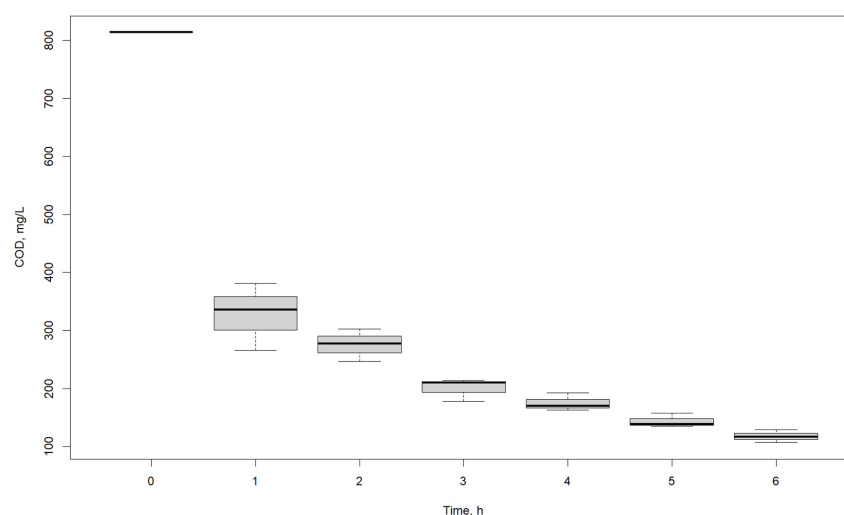
Voltage, V	SAC λ 620 nm, m^{-1}		
	R1	R2	R3
0	10.4	10.4	10.4
1	5.4	4.5	5.2
2	2.8	2.6	3
3	2.3	2.4	2
4	1.8	1.7	2.2
5	1	1.5	2
6	2	1.8	1.3

3.2. Effect of Time

The same parameters were analyzed for treatments maintaining 5 volts for different times. Chemical oxygen demand (COD) levels complied with the maximum of 150 mg/L in 5–6 h established in the Mexican norm. See Table 16 and Figures 11 and 12.

Table 16. COD removal results with 5-volt treatment and variable time.

Time, h	COD, mg/L		
	R1	R2	R3
0	814	814	814
1	381	266	336
2	247	303	277
3	214	178	210
4	170	193	163
5	135	158	139
6	117	129	107

**Figure 11.** COD vs. time with 5-volt treatment.

Concerning pH, the value of pretreated tequila vinasses decayed from 7.81 to 3.33 with treatment time, which is outside the values permitted by the Mexican norm. See Table 17 and Figure 13.

Electrical conductivity (EC) values ranged from 7.299 to 5.378 mS/cm, dropping over time. See Table 18 and Figure 14.



Figure 12. Time effect for 5 V treatments. From left to right: pretreated vinasses, 1 h, 2 h, 3 h, 4 h, 5 h, 6 h, and pure water.

Table 17. pH results with 5-volt treatment and variable time.

Time, h	R1	pH R2	R3
0	7.81	7.81	7.81
1	6.52	6.18	6.28
2	6.32	6.21	5.89
3	5.69	5.76	5.30
4	5.10	5.66	5.49
5	4.54	4.17	4.80
6	3.76	3.52	3.33

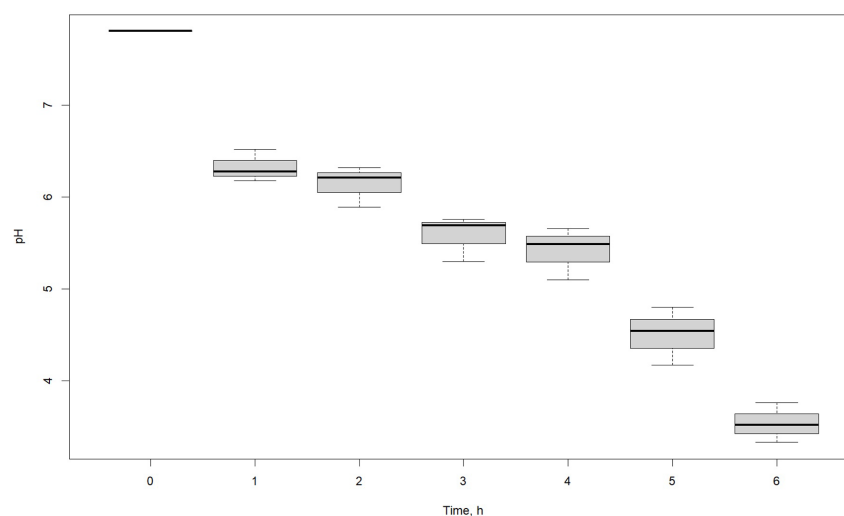


Figure 13. pH vs. time with 5-volt treatment.

Table 18. EC results with 5-volt treatment and variable time.

Time, h	R1	EC, mS/cm R2	R3
0	7.299	7.299	7.299
1	6.436	6.457	6.623
2	5.649	6.666	6.145
3	5.378	6.128	6.014
4	6.489	6.290	6.302
5	5.678	6.025	5.509
6	5.783	5.850	5.698

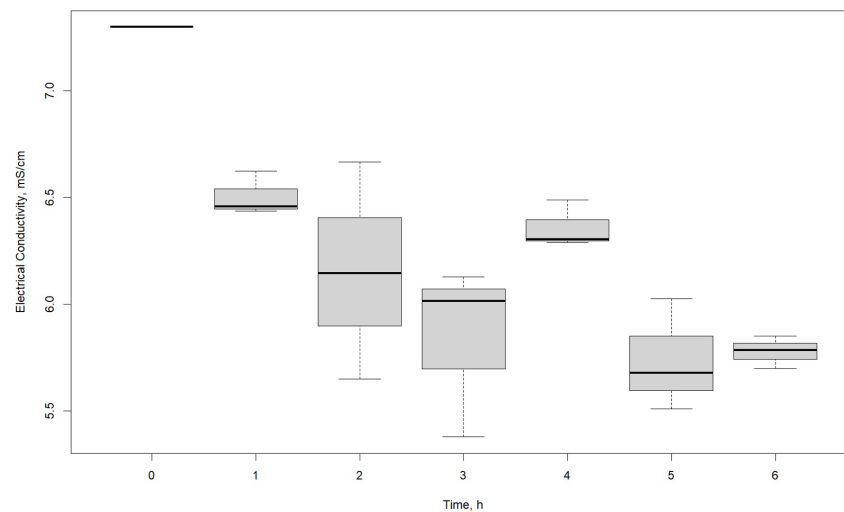


Figure 14. EC vs. time with 5-volt treatment.

Total dissolved solids (TDS) for pretreated vinasses value ranged from 3.577 to 2.658 mg/L over time. See Table 19 and Figure 15.

Table 19. TDS results with 5-volt treatment and variable time.

Time, h	TDS, mg/L		
	R1	R2	R3
0	3.577	3.577	3.577
1	3.174	3.206	3.246
2	2.869	3.265	3.011
3	2.658	3.003	2.971
4	3.180	3.083	3.089
5	2.787	2.953	2.734
6	2.878	2.867	2.793

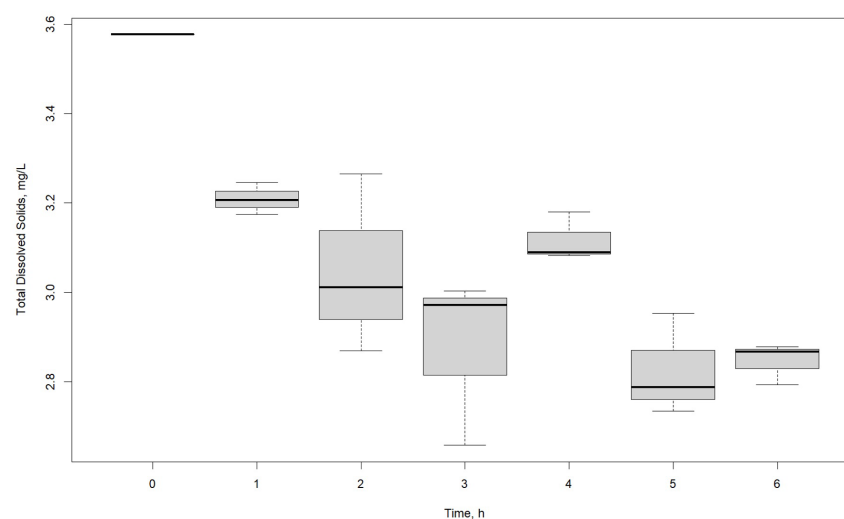
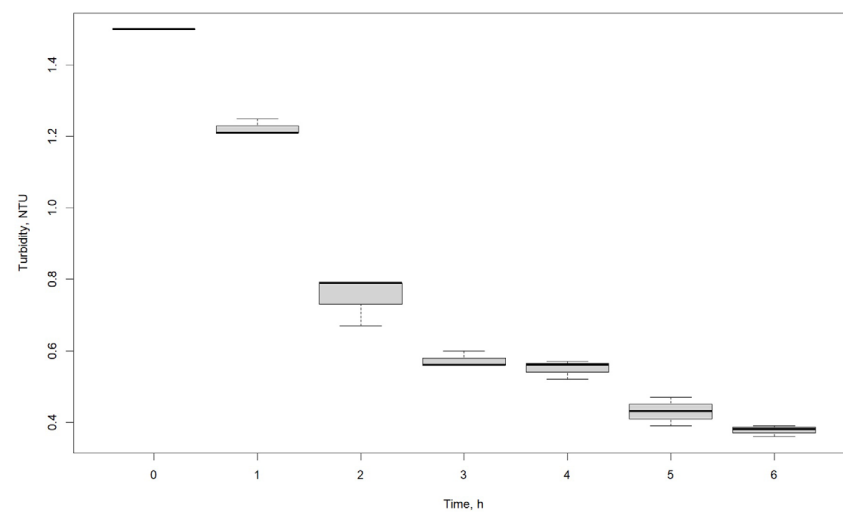


Figure 15. TDS vs. time with 5-volt treatment.

Turbidity values decreased from 1.50 NTU to 0.36 NTU over 6 h with 5-volt treatment. See Table 20 and Figure 16.

Table 20. Turbidity results with 5-volt treatment and variable time.

Time, h	Turbidity, NTU		
	R1	R2	R3
0	1.50	1.50	1.50
1	1.21	1.21	1.25
2	0.79	0.67	0.79
3	0.60	0.56	0.56
4	0.56	0.57	0.52
5	0.47	0.39	0.43
6	0.36	0.39	0.38

**Figure 16.** Turbidity vs. time with 5-volt treatment.

SAC values for the λ 436 nm wavelength obtained after 1 h with the 5-volt treatment complied with the Mexican norm. See Table 21 and Figure 17.

SAC values for the λ 525 nm wavelength obtained with the 5-volt treatment for all times complied with the Mexican norm. See Table 22 and Figure 18.

SAC values for λ 620 nm for all times applied in the 5-volt treatment complied with the Mexican norm. See Table 23 and Figure 19.

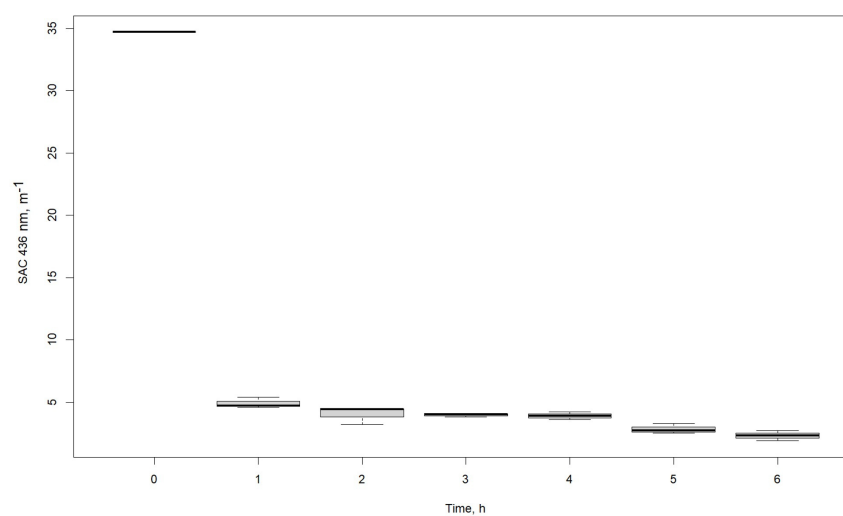
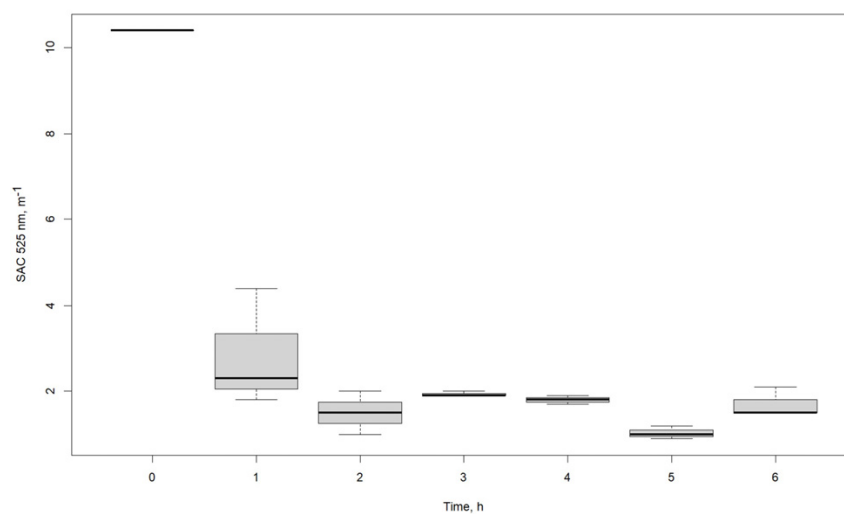
**Figure 17.** SAC λ 436 nm color vs. time with 5-volt treatment.

Table 21. SAC λ 436 nm with 5-volt treatment and variable time.

Time, h	SAC λ 436 nm, m^{-1}		
	R1	R2	R3
0	34.7	34.7	34.7
1	4.7	4.6	5.4
2	4.4	3.2	4.5
3	3.8	4.0	4.1
4	3.6	4.2	3.9
5	2.7	2.5	3.3
6	2.3	1.9	2.7

Table 22. SAC λ 525 nm with 5-volt treatment and variable time.

Time, h	SAC λ 525 nm, m^{-1}		
	R1	R2	R3
0	10.4	10.4	10.4
1	2.3	1.8	4.4
2	1.0	1.5	2.0
3	1.9	2.0	1.9
4	1.7	1.9	1.8
5	1.2	0.9	1.0
6	1.5	1.5	2.1

**Figure 18.** SAC λ 525 nm vs. time with 5-volt treatment.**Table 23.** SAC λ 620 nm with 5-volt treatment and variable time.

Time, h	SAC λ 620 nm, m^{-1}		
	R1	R2	R3
0	3.7	3.7	3.7
1	1.2	1.1	1.6
2	1.1	0.9	1.0
3	1.2	1.1	1.1
4	0.9	1.3	1.0
5	1.2	1.4	1.4
6	1.3	0.8	1.2

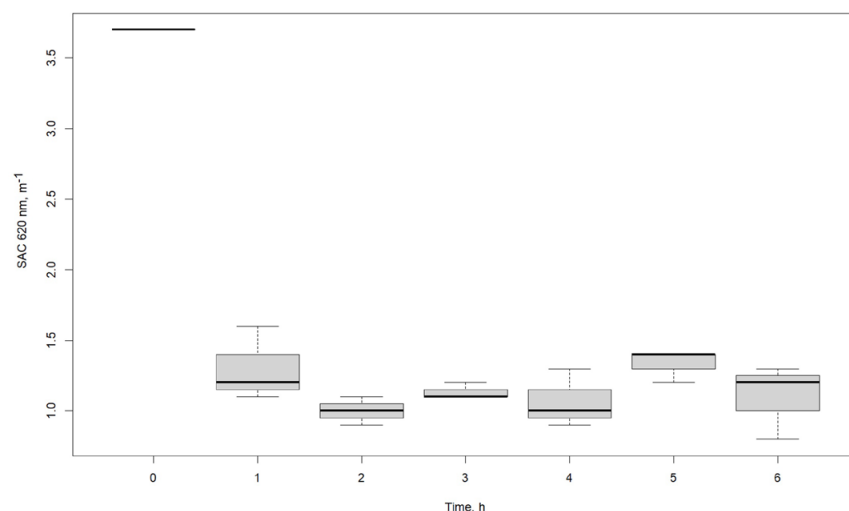


Figure 19. SAC λ 620 nm vs. time with 5-volt treatment.

3.3. Kinetic Model

The kinetic model fits a second-order type, where an average value of k is $0.04194 \text{ [mM]}^{-1}\text{h}^{-1}$ with a mean R^2 value of 0.9768. These values were considered, and the data are shown in Table 15, modifying COD from mg/L to mM. See Figure 20.

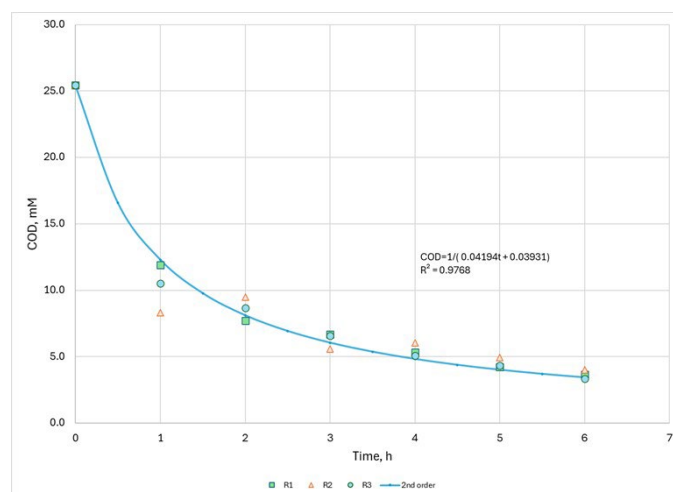


Figure 20. Second-order kinetic model analysis.

3.4. Economic Analysis

The treatment incurs two main costs: those for electricity and those for the anode material. In the laboratory, we calculated that the cost for electricity was 0.21 USD/kWh and that the cost for steel was 1.05 USD/kg (values for Mexico in May 2025), resulting in costs of 0.79 USD/m³ for electricity and 0.57 USD/m³ for the steel anode (material consumption)—a total of USD 1.36 per cubic meter of treated vinasse. See Table 24. Anode material consumption was measured at -4.7419 g with 32 h for kit 1, -3.8149 with 30 h for kit 2, and -1.7408 with 29 h for kit 3 (0.1118 g/h at 5 V and 0.155 A).

Table 24. Economic analysis of proposed treatment.

Parameter	Data
Optimal laboratory conditions	
Sample volume	0.2 L
Average electric current	0.155 A
Voltage	5 V
Treatment time	6 h
Calculated power	0.775 W
Electrode surface	19.58 cm ²
Initial COD	814 mg/L
Average final COD	118 mg/L
Anode consumption	0.1118 g/h 0.1442 kg/kWh
Data for industrial applications	
Surface electric current	79.16 A/m ²
Unitary energy consumption	3.775 kWh/m ³
Specific energy consumption	33.31 kWh/kg COD
Theoretical electrode consumption	4.84 kg/m ³
Faraday efficiency	0.51
Energy cost	0.21 USD/KWh 0.79 USD/m ³
Anode consumption	0.1442 kg/KWh 0.544 kg/m ³
Anode cost	1.05 USD/kg 0.57 USD/m ³
Treatment cost	1.36 USD/m ³

3.5. Sludge Generation

An indirect way of measuring sludge generation is to measure the filtered volume vs. the initial volume (200 mL). The sludge generation ranged from 5.32% at a voltage of 1 volt to 36.35% at 6 volts. See Table 25 and Figure 21.

Table 25. Sludge volume yield with 2 h treatment and variable voltage.

Voltage	Replicate	Filtered Volume, mL	Sludge Percentage
1	R1	183.72	8.86%
	R2	188.68	6.00%
	R3	189.90	5.32%
2	R1	178.83	11.84%
	R2	176.23	13.49%
	R3	174.56	14.57%
3	R1	162.64	22.97%
	R2	169.12	18.26%
	R3	170.08	17.59%
4	R1	157.19	27.23%
	R2	155.31	28.77%
	R3	155.69	28.46%
5	R1	153.17	30.57%
	R2	151.11	32.35%
	R3	155.01	29.02%
6	R1	146.68	36.35%
	R2	149.29	33.97%
	R3	150.16	33.19%



Figure 21. Sludge volume yield after two hours of treatment with variable voltage. From left to right: 6 V, 5 V, 4 V, 3 V, 2 V, and 1 V.

It is important to note that a further sludge analysis will be needed to check the dry solids percentage.

4. Discussion

Regarding treatment time, the proposed period is one of the longest at 360 min. Seven of the nine reported works in Table 5 reported lower times; however, the period is very suitable for tequila distilleries, reaching up to four treatments in a day, reducing equipment volume.

This treatment differs from those in all the other works regarding pH, since they reported an increment in pH values vs. a decrement reported in this research. The treatment presented in this work will need further alkalization, as in most of the reported works.

With respect to COD levels, six of the nine works reported presented direct distillery wastewater (COD above 100 g/L). Alizadeh et al. [64] also used this origin but with half the COD. Only three works used pretreated vinasses [57,60,61], and only Jiménez-López reached COD values below 150 mg/L, as established by the Mexican norm, though they did not comply with EPA or European norms. Color reduction, in general, achieves high values (>80% reduction) in very short times of around 60 min. Turbidity, in general, is associated with TSS and is usually lowered by filtration after electrocoagulation.

Regarding surface electrical current, values vary from treatment to treatment. Three works reported values above 1000 A/m², while the rest reported ones below 200 A/m². This work presented a lower value: 79.16 A/m².

For specific energy consumption, this work yielded a lower value of 3.775 kWh/m³, which is consistent with COD levels as compared with all the reported works. Only with respect to this parameter is the process cost lower than those of the treatments in all the other presented works.

Out of all the reported research, Jiménez López [57] presented the most similarities, reaching COD values below 150 mg/L, with 3 h versus 6 h in this work, as well as double the surface electric current, five times the energy consumption, and a similar proportion in terms of anode consumption (0.6 g/h vs. 0.14 g/h). It should be noted that using aluminum electrodes will impact the generated sludge in terms of contaminants and ions in treated wastewater, while iron electrodes will not have a significant environmental impact if used as a precursor of compost or biofertilizers.

5. Conclusions

This study demonstrates that electrocoagulation using stainless-steel cathodes and iron anodes is an effective fourth-stage treatment for tequila vinasses to meet the updated regulatory standards in Mexico. The process successfully reduced COD levels to below 150 mg/L after 5–6 h of treatment at 5 V, complying with NOM-001-SEMARNAT-2021 requirements. Color removal was achieved within 1 h at voltages of 4–6 V. The treatment

showed promising results for industrial scale-up, with a relatively low energy consumption of 72.4 A/m² and economical costs of USD 1.36 per cubic meter treated. While pH adjustment is still needed post-treatment, the use of iron anodes produces environmentally benign sludge that could potentially be used as a soil amendment. Overall, this electrocoagulation process provides an efficient and sustainable solution for tequila distilleries to meet stringent wastewater discharge regulations and improve the environmental performance of the tequila industry. This proposed process can be limited by the electricity availability within distilleries owing to several factors, such as the power available in the region from the national electric grid; therefore, solar-powered electricity might be needed, and the changing of anodes is also a concern, since they must be replaced frequently. In future work, the sludge generated can be further studied as a fertilizer or for other potential uses.

Author Contributions: Conceptualization, R.G.P., A.L.F.M. and E.M.O.; methodology, R.G.P., A.L.F.M. and E.M.O.; software, E.M.O., N.S.O. and J.N.A.; validation, N.S.O., J.N.A. and C.G.G.; formal analysis, R.G.P., A.L.F.M., E.M.O., N.S.O., J.N.A. and C.G.G.; investigation, R.G.P., E.M.O. and N.S.O.; resources, R.G.P., A.L.F.M. and E.M.O.; data curation, J.N.A. and C.G.G.; writing—original draft preparation, R.G.P.; writing—review and editing, A.L.F.M. and E.M.O.; visualization, N.S.O. and J.N.A.; supervision, A.L.F.M.; project administration, R.G.P. and E.M.O.; funding acquisition, R.G.P. and E.M.O. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflicts of interest.

Abbreviations

The following abbreviations are used in this manuscript:

COD	Chemical Oxygen Demand
EC	Electrical Conductivity
SAC	Spectral Absorption Coefficient
TDS	Total Dissolved Solids
TSS	Total Suspended Solids

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