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ELECTROCHEMICAL WASTEWATER TREATMENT WITH SIMULTANEOUS HYDROGEN PRODUCTION IN TEXTILE INDUSTRY

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The textile industry is a major contributor to water consumption and greenhouse gas emissions, ranking 4th and 5th respectively in terms of impact in the EU. The process of major concern are the finishing operations, such as brightening, dyeing, printing and mercerizing, which require water at high temperatures. These processes result in effluents with high levels of alkalinity and conductivity, making their wastewater ideal for electrochemical treatment. The industry has used various strategies to treat these wastewaters, but has not considered using electrochemical processes in conjunction with hydrogen co-generation, which could help decarbonize the textile finishing industry. The study evaluates the use of mercerizing and dyeing effluents as electrolytes for hydrogen production, coupled with color removal in the latter. The hydrogen flow rate depends solely on the effluent conductivity, with no detected effect from the present contaminants. Both effluents achieve hydrogen generation efficiencies of over 70%. The electrochemical treatment of dyeing effluent achieves 97-99% color removal and generates hydrogen equivalent to 19,5% of the electric energy consumed during treatment.

Keywords: textile wastewater; electrolysis; hydrogen production; wastewater treatment; Circular Economy

TRATAMIENTO ELECTROQUÍMICO DE AGUAS RESIDUALES CON PRODUCCIÓN SIMULTÁNEA DE HIDRÓGENO EN LA INDUSTRIA TEXTIL

La industria textil es un gran contribuyente en el consumo de agua y generación de emisiones de gases de efecto invernadero, siendo el 4º y 5º sector con mayor impacto en la UE. Los procesos que más preocupan son las operaciones de acabado, como el blanqueado, la tintura, el estampado y el mercerizado, que requieren agua a altas temperaturas. Estos procesos resultan en efluentes con altos niveles de alcalinidad y conductividad, convirtiéndolos en ideales para el tratamiento electroquímico. La industria ha utilizado diversas estrategias para tratar estos efluentes, pero aún no ha considerado la utilización de procesos electroquímicos en combinación con la cogeneración de hidrógeno, lo que podría ayudar a descarbonizar la industria de acabados textiles. El estudio evalúa el uso de efluentes de mercerizado y tintura como electrolitos para la producción de hidrógeno, emparejada con la eliminación de color en el segundo. El flujo de hidrógeno solo depende de la conductividad del efluente, sin efecto detectado por los contaminantes presentes. Ambos efluentes superan eficiencias de generación de hidrógeno del 70%. El tratamiento electroquímico del efluente de tintura consigue eliminar el 97-99% del color, con una generación de hidrógeno equivalente al 19,5% de la energía eléctrica consumida durante el tratamiento.

Palabras clave: agua residual textil; electrólisis; producción hidrógeno; tratamiento aguas residuales; Economía Circular

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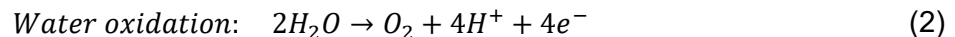
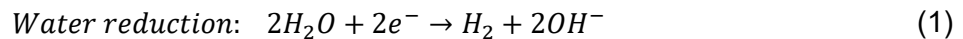
1. Introduction

Hydrogen is a well-known energy and chemical carrier that is gaining momentum in the last years as a partial solution to human activity's decarbonization. There are two main sources to obtain it: hydrocarbons and water (Nikolaidis & Poullikkas, 2017). The world focusses on the second source to get hydrogen because the electrolysis is the only path towards green hydrogen production when using renewable energy (European Commission, 2020; Gobierno de España - MITERD, 2020).

Producing hydrogen from water electrolysis presents two main issues: high electrical energy consumption and the demand of clean water, which makes these processes compete with hydric needs from ecosystems. Solving the first issue is mainly a matter of technical improvements and implementation of new technologies that will increase systems' performance and efficiency. Meanwhile, the second issue is addressed by studying water sources that are inadequate for human consumption, such as sea water (Asghari et al., 2022) and wastewater (Aydin et al., 2021).

As its name indicates, wastewater is considered a residue, while a residue is so called because its usability potential needs to be found and/or proven. When this usability potential is found, these residues are, then, considered by-products. Industrial processes generate diverse by-products that can be used across different industries, so knowing the by-products each one has available is a deal breaker for business profitability and for environmental responsibility.

Hence, the best approach to address high electrical consumption and clean water spending to produce green hydrogen is finding wastewater electrochemical treatment processes during which hydrogen is produced as a by-product. This can be achieved because water residues are eliminated by oxidation, and in order to complete the redox reaction water reduction is required, contrasting with regular water electrolysis, where balance is achieved by both oxidation and reduction of water, as shown in equations (1) and (2).



Textile industry is a suitable sector to find this kind of wastewater due to the diversity of its residual effluents. This sector features a high level of water consumption, having the 4th greatest hydric impact among European industrial sectors. In fact, textile finishing processes are the ones that account for the highest contribution to wastewater discharge in the sector, with a wide range of diversity in its residual effluents and consuming about 79,6m³ per tonne of produced textile (European Commission, 2023). Also, process water needs to be heated at plant boilers, implying CO₂ emissions.

The wastewater generated by the textile industry is mainly biodegradable, but there are non-biodegradable compounds that require treatment using tertiary processes such as physicochemical flocculation-coagulation and/or the addition of chemicals that counteract the pollutant. Therefore, this sector has a double impact on the environment: it generates residual effluents and greenhouse gas emissions (direct impact), and requires the addition of chemicals for the tertiary treatment of the effluents (indirect impact). As an alternative to classical treatments, the electro-oxidation process has shown promising results in the removal of non-biodegradable compounds from textile effluents (Rodríguez-Narváez et al., 2021). This innovative technology not only tackles the environmental impact caused by the direct discharge of non-biodegradable compounds but also reduces the need for the addition of chemicals for tertiary treatment, thus reducing the sector's indirect impact on the environment.

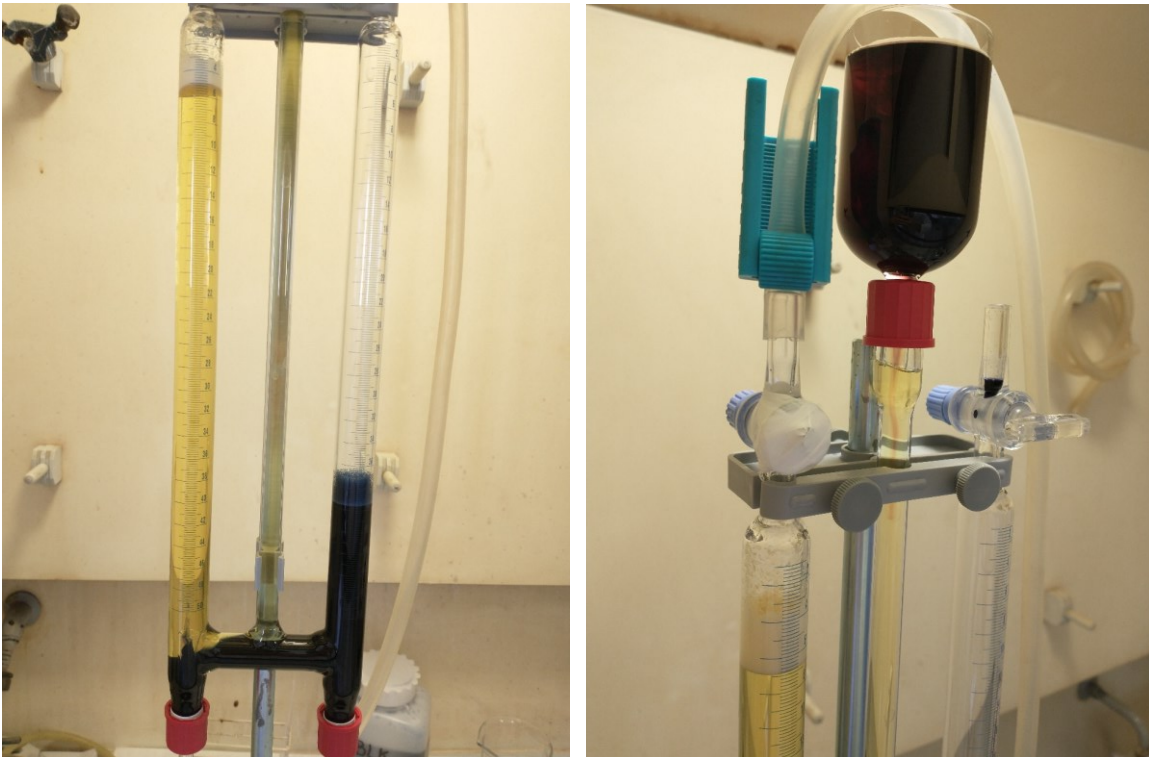
From the diverse textile finishing processes, reactive dyeing and mercerization are the ones with a higher potential to produce hydrogen when paired with electrochemical processes, as both show high conductivity and high pH due to its concentration in NaOH alkali, making possible the alkaline electrolysis of water with such effluents. In addition, reactive dyeing is the most common coloring method due to the high quality colors they give to the fibers, really intense and durable due to the reaction that attaches the dye to the textile. This type of dye have already been treated by electrochemical means achieving over 90% color removal rates or even reaching the complete color elimination depending on current density applied (López-Grimau et al., 2015).

The aim of this study is to test reactive dyeing and mercerization wastewaters, both generated by the textile finishing industry, as sources for hydrogen gas production via electrochemical processes. In the case of dyeing wastewater, the electrochemical process is already employed to treat the non-biodegradable dye, making hydrogen a by-product of the treatment process. In contrast, mercerization effluents lack residues to be oxidized, being composed solely by water and NaOH. However, this composition is also the one for the electrolyte used in water alkaline electrolysis, making it very interesting for this purpose.

2. Methodology

In this study, a Hofmann Voltammeter is utilized. This device allows both electrodes to be placed at a fixed distance with no separator in between, and enables the measurement of gas formation in the anode and the cathode columns individually, with a maximum capacity to measure 50mL of gas per column. Figure 1 displays the voltammeter treating a dyeing wastewater, which clearly shows the discoloration at the anode and the volume of produced hydrogen at the cathode column.

Figure 1. Hofmann Voltammeter: (L) dyeing effluent treatment with platinum electrodes (anode-yellow water; cathode-black water) (R) overflow funnel of the center column's top opening



The electrochemical process is carried out at constant voltage conditions due to the low current found during experimental design testing, which prevents to use the full range of up to 10A from the DC source, which voltage could go up to 20V. The test runs until the amount of hydrogen reaches the maximum the system allows. Either caused by achieving more than 45mL of H₂ production or because the water displaced by both H₂ and O₂ formation makes the effluent get close to overflowing voltammeter's center column. The three types of effluents are the ones described in Table 1, being their composition the most common found in industrial environments. In the case of the dyeing wastewater, black dye is chosen because it is the one needing a greater effort to be eliminated.

Table 1. Simulated effluents composition

Effluent	Nomenclature	NaOH	Other compounds
<i>Alkaline Electrolysis</i>	AlkSim25	25%	-
<i>Mercerization</i>	MerSim02	2%	-
<i>Dyeing</i>	BlkSimAA	add till pH11	NaCl: 20 g/L Remazol Black B-133: 1 g/L

Two types of electrodes are used, graphite (C) and platinum (Pt). The graphite electrodes are cylindrical with a length of 87mm and a diameter of 8mm, providing a contact surface area with the effluent (S_C) of 22,37cm². On the other hand, the platinum electrodes are sheets of 5x10mm, with a contact surface area (S_{Pt}) of 1cm².

In Table 2 tests are identified depending on 3 factors: effluent type (AlkSim25, MerSim02 or BlkSimAA), electrode material (C or Pt) and working voltage (3V to 20V). The lower voltage limit is set to 3V even water electrolysis thermo-neutral voltage is 1,48V and its gas evolution voltage is 1,65-1,7V (Rashid et al., 2015). This is because the system working at voltages lower than 3V produce too little hydrogen, and the current measured by the DC source is 0mA due to its minimal significance decimal, making impossible calculating the electrical energy consumption.

Table 2. Experimental nomenclature depending on effluent, electrode and voltage

Effluent	Electrode	
	Graphite	Platinum
<i>Alkaline Electrolysis</i>	AlkSim25_C_xxV *	AlkSim25_Pt_xxV
<i>Mercerization</i>	MerSim02_C_xxV	MerSim02_Pt_xxV
<i>Dyeing</i>	**	BlkSimAA_Pt_xxV

* "xxV" defines the working voltage, for example *MerSim02_C_16V* for the 16V test of the mercerization effluent with graphite electrodes, and *MerSim02_C* in the case of referencing to the whole voltage range results

** Graphite not used with dyeing effluent since Cl⁻ presence would degrade the material excessively

The focus during testing are: evaluating H₂ generation speed and electricity to H₂ efficiency during electrochemical processes for different effluents, and how these effluents react to this process. In the dyeing effluent case, color before and after treatment is measured at the wavelength of maximum absorbance in the pre-treated effluent (585nm), which corresponds to the specific wavelength, within the visible spectrum, that defines the color of the effluent.

Table 3 depicts all the experimental measurements and calculated values this work involves, while Table 4 lists the devices used for measurements.

Table 3. Experimental measurements and calculated values

Experimental measurements		Calculated values		
<i>Initial and final values</i>		<i>From</i>	<i>To</i>	
[pH]	Effluent pH			
[Cond]	Effluent conductivity (mS/cm)			
[T]	Effluent temperature (°C)	[t], [vH ₂]	[ΔvH ₂ /t]	H ₂ generation speed (mL/min)
[Col]	Color absorbance at 585nm	[T], [vH ₂]	[EH ₂]	HHV energy of H ₂ produced (kJ)
<i>During test measurements</i>		[I], [V], [t]	[E]	Electrical energy spent (Wh)
[t]	Experiment time (min)	[EH ₂], [E]	[%EH ₂]	H ₂ production efficiency (%)
[vH ₂]	H ₂ gas volume (mL)	[Col]	[%disCol]	Color elimination (%)
[vO ₂]	O ₂ gas volume (mL)	[E], [V], [t]	[I _{AVG}]	Average current (mA)
[I]	Current (mA)	[I _{AVG}], [S]	[I/S]	Current density (mA/cm ²)
[V]	Voltage (V)			

Table 4. Devices used for variables measurements

Variable	Device
[pH]	Hach sensION+ PH1
[Cond]	Crison Conductimeter GLP 31
[Col]	Shimadzu UV-2041PC Spectrophotometer
[vH ₂] \ [vO ₂]	Hofmann Voltammeter (graduated columns from 0mL to 50mL + 0,2mL)
[I] \ [V]	Blausonic FA-210 (DC source)

For each test at a specific voltage, a graphical representation of [vH₂] as a function of time [t] gives a lineal regression, slope from which the hydrogen production rate [ΔvH₂/t] is extracted.

Because of the center column top funnel opening, gas pressures inside the voltammeter are at atmospheric conditions and is possible applying ideal gas hypothesis to convert H₂ volume produced [vH₂] to moles, see equation (3). The temperature considered is the one measured in the end of the test for the effluent, as it is the one more in contact with the gas. From moles of hydrogen to energy [EH₂], equation (4) is used, where HHV refers to the combustion high heat value: 285,84 kJ/mol (Harrison et al., 2010). The main reason to take the combustion HHV is the textile finishing processes requiring much more hot water than electricity, meaning that energy recovered from hydrogen is more likely to be obtained via H₂ combustion to heat process water.

$$p \cdot v = n \cdot R \cdot T \quad \rightarrow \quad n_{H_2} = \frac{p_{atm} \cdot v_{H_2}}{T \cdot R} \quad \{R = 0,082057 L \cdot atm/K\} \quad (3)$$

$$EH_2 = n_{H_2} \cdot HHV \quad (4)$$

On the other side, the electrical energy spending in the test [E] is determined calculating the electrical power at each measurement done: from the given voltage [V] and current [I]; and then integrating between each pair of measures taking into account the time [t] at which each one was taken, just as seen in equation (5).

$$E = \sum_0^i \frac{(V_{i+1} \cdot I_{i+1}) + (V_i \cdot I_i)}{2} \cdot (t_{i+1} - t_i) \quad (5)$$

The efficiency of hydrogen production [%EH₂] is obtained comparing the equivalent energy of the hydrogen generated [EH₂] and the electrical energy spent [E], such as shown at equation (6).

$$\%EH_2 = \frac{EH_2}{E} \cdot 100 \quad (6)$$

Discoloration [%disCol] is calculated using equation (7), giving the amount of color that disappeared from the effluent between the initial sample and the post-treated one.

$$\%disCol = \frac{Col_f - Col_i}{Col_i} \cdot 100 \quad (7)$$

In cases where different types of electrodes have to be compared, the best way is representing the results as a function of current density [I/S] instead of the voltage [V]. To do so, the average current [I_{AVG}] is calculated from equation (8), taking the total electrical energy spent [E], the experimental voltage [V_{exp}] (encoded as “xxV” at test nomenclature) and the total time of the test [t_i]. From average current [I_{AVG}] to current density equation (9) is used.

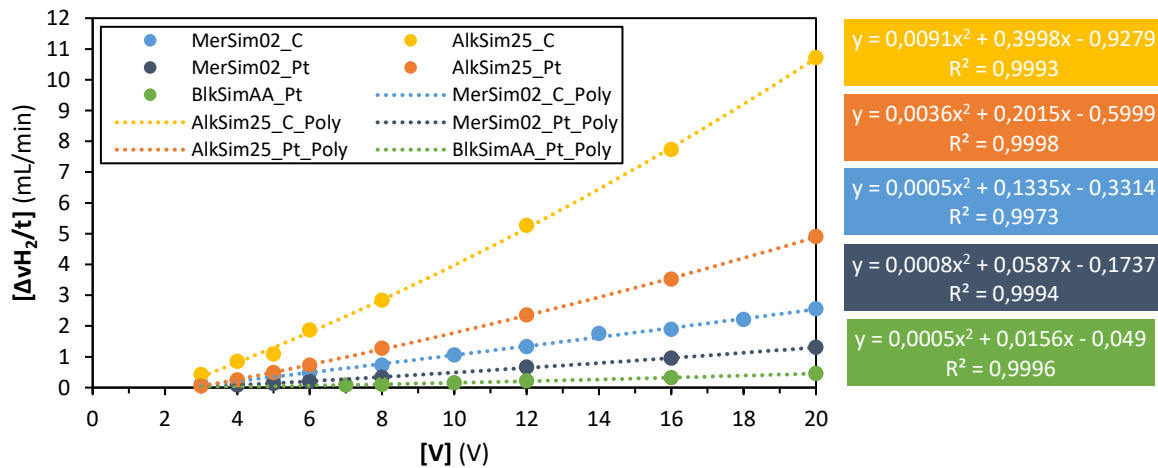
$$I_{AVG} = \frac{E}{V_{exp} \cdot t_f} \quad (8)$$

$$I/S = \frac{I_{AVG}}{S_{electrode}} \quad (9)$$

3. Results

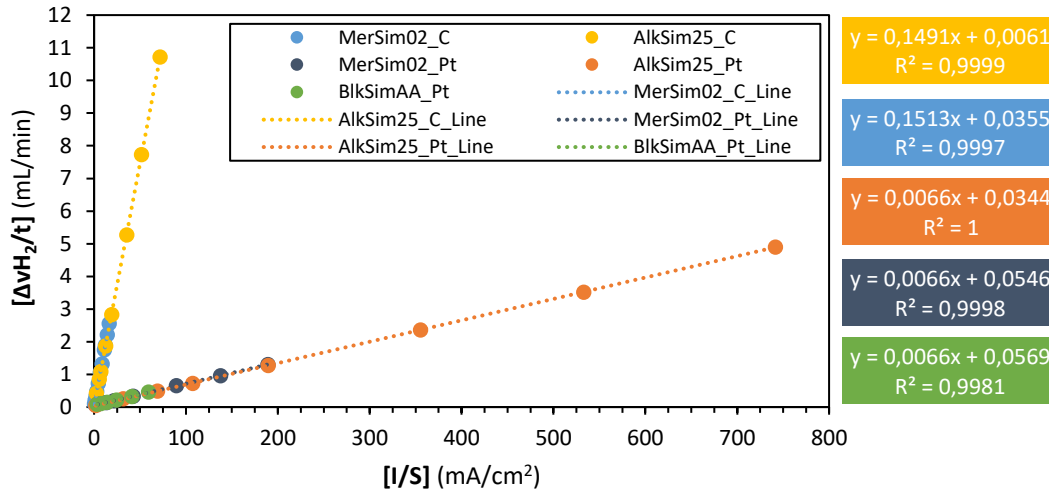
The first results are represented at Figure 2, where H₂ production flow with respect to the applied voltage is shown. As stated in the methodology, at too low voltages hydrogen production is too low to make the test feasible in this. On the other side, great hydrogen flows are found at the highest voltage (20V), with a ratio of effluents processed with graphite electrodes (C) getting double the flow compared to those using platinum electrodes (Pt), behavior seen between the result pairs: yellow-orange (AlkSim25) and light-dark blue (MerSim02).

Figure 2. H₂ production speed vs working voltage



Just as defined during methodology, in order to compare the results effectively, these require being represented in function of the current density. This is the case at Figure 3, where H₂ generation speed is coupled to current density, obtaining a completely lineal relation between these variables instead of the 2nd polynomial degree relation from Figure 2.

Figure 3. H₂ production speed vs current density



The 5 lineal regressions obtained can be grouped into 2 cases depending on the type of electrode used for the electrochemical process: 0,15 slope in the case of carbon electrodes and 0,0066 slope for the platinum ones. These results show that H₂ generation speed and current density relationship is unaffected by the effluent pollutants, so it remains a characteristic linked to the electrode type. Therefore, when looking for hydrogen production for a specific wastewater, the only parameter that will set the performance of the process will be conductivity, and this means conductivity increment can make any residual effluent suitable for hydrogen production.

The previously stated relation shown between the effluents links higher conductivities ones to higher current densities at same voltages, consequently being able to reach higher H₂ generation speed. This relation is about the same proportion at Table 5, where maximum current densities, conductivities and H₂ production flow between different effluents are compared, so a proportional relation between these three variables can be assumed.

Table 5. Relation between effluents' average conductivity, maximum current density and H₂ production flow

Test 1 / Test 2	Avg. [Cond] relation	Max. [I/S] relation	Max. [ΔvH ₂ /t] rel.
<i>AlkSim25_C / MerSim02_C</i>	(331,1)* / 77,1 4,29	71,7 / 16,5 4,35	10,71 / 2,55 4,20
<i>AlkSim25_Pt / MerSim02_Pt</i>	(330,6) / 94,7 3,49	741,6 / 189,1 3,92	4,90 / 1,30 3,76
<i>MerSim02_Pt / BlkSimAA_Pt</i>	94,7 / 31,4 3,02	189,1 / 59,22 3,19	1,30 / 0,46 2,85

* [Cond] out of conductimeter's measure range (max 200mS/cm), (*values*) are theoretical based on equations described at Brauns and Turek (2020) work, as a function of the 25% NaOH concentration and the temperature [T]

Efficiency regression curves are shown at Figure 4, where both residual effluents (MerSim02 and BlkSimAA) have been able to reach high efficiencies independently from the electrode material: MerSim02_C_03V (81,7%), MerSim02_Pt_04V (100%) and BlkSimAA_Pt_07V (70,2%).

In contrast with the H₂ generation speed growth from Figure 3, Figure 4 shows a decrement in energy efficiency to produce this hydrogen while current density increases. From this observation, a balance between H₂ flow and process efficiency seems required, and this balance depends on the priorities at every given application and situation.

Another issue is how lower conductivity effluents give lower efficiencies for the same current density. Finding such behavior permits determining that a higher conductivity will increase H₂ production efficiency apart from its generation speed. Also, platinum electrodes tests show higher efficiencies over graphite ones, with AlkSim25_C and BlkSimAA_Pt giving similar efficiencies, although the first one having a ten times greater conductivity compared to BlkSimAA_Pt.

Figure 4. H₂ production efficiency vs current density

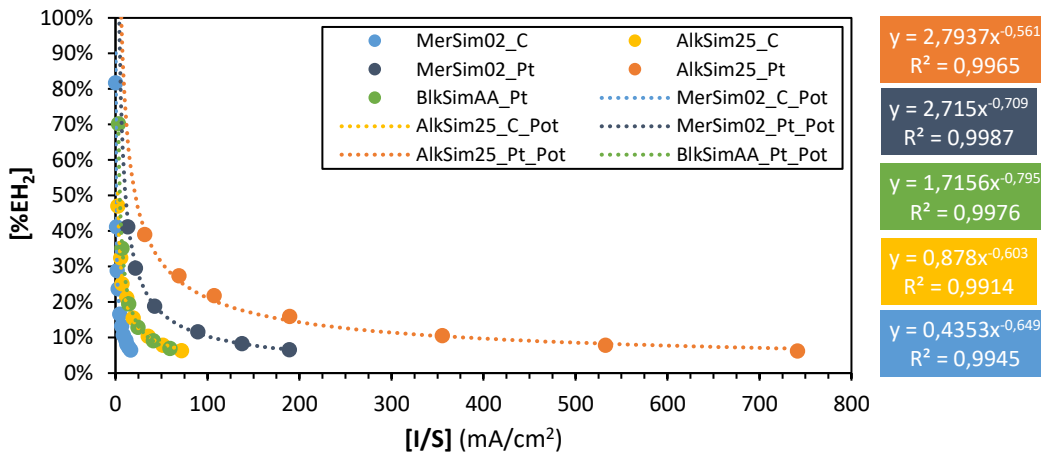
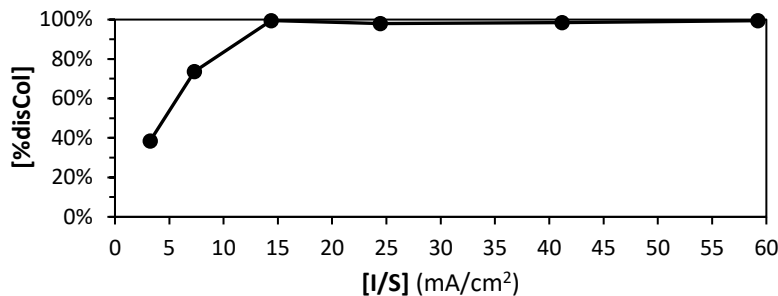


Figure 5 depicts performance during dyeing effluent treatment, evaluated considering discoloration of the effluent (%disCol).

Figure 5. Color reduction vs current density



There is a breaking point at current density after which the discoloration reaches 97-99%. In this experimental case, the breaking point corresponds to 14,38mA/cm², with the hydrogen production characteristics being: a generation speed of 0,1517mL/min and a 19,5% efficiency. This second value has to be seen as the maximum amount of energy that could be recovered from the hydrogen produced during color elimination.

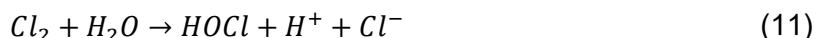
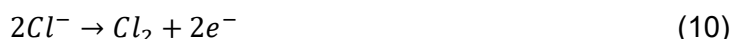
From the potential regressions at Figure 4, H₂ production at 14,38mA/cm² are 62,5% for the ideal effluent (AlkSim25_Pt) and 41,0% for the not so ideal effluent (MerSim02_Pt). Taking into account the efficiency dependency on conductivity, it would be possible to increase the energy recovery

potential substantially. In case of similar behavior between BlkSimAA_Pt and MerSim02_Pt, incrementing dyeing effluent conductivity from 30mS/cm to 90mS/cm would double the energy reduction for the electrochemical treatment (from 19,5% to 41,0%).

This extra conductivity could be found in dyeing effluents with higher NaCl concentration since some industrial effluents achieve up to 80g/L of salt for specific purposes. For these effluents would be expected greater production, but could represent a much higher corrosion environment for electrodes, lowering their life expectancy.

The color elimination mechanism is identified as an indirect oxidation process based on the formation of aqueous sodium hypochlorite (NaOCl), commonly known as bleach, from which hypochlorite anion (OCl⁻) is an oxidant agent. This hypothesis is coherent to the statements:

1. There is almost no gas formation at the anode, meaning that the water oxidation producing O₂ gas is substituted by the production of aqueous specimens, such as NaOCl.
2. There is a much higher pH reduction when higher discoloration. These findings can be explained by indirect oxidation reaction mechanisms, which are described at equations (10) to (12) (Martínez-Huitle & Panizza, 2018) and show generation of protons (H⁺) that acidify the effluent lowering its pH.



3. These mechanisms also explain how NaCl salt in a high alkalinity solution makes possible the formation of OCl⁻ by chlorine oxidation, which interacts with Na⁺ ion from the salt to represent the disassociated NaOCl (bleach). Although, equation (12) only occurs at highly alkaline effluents, such as the pH11 from this dyeing effluent (Fukuzaki, 2006).

To end with, average conditions before and after tests are included at Table 6.

Table 6. Average initial and final parameters values from effluents

Test	pH _i	pH _f	Cond _i (mS/cm)	Cond _f (mS/cm)	T _i (°C)	T _f (°C)
AlkSim25_C	14,00	14,00*	(314,8)**	(347,4)	17,7	20,9
MerSim02_C	13,36	13,29	77,8	76,5	18,3	20,3
AlkSim25_Pt	14,00	14,00*	(310,7)	(350,5)	17,3	21,2
MerSim02_Pt	13,38	13,28	95,5	93,9	20,2	21,6
BlkSimAA_Pt	11,05	9,98	31,1	31,6	21,2	22,6

* AlkSim25 [pH] remained the maximum as 25% NaOH concentration is totally alkaline and it is already 14 from a concentration of 4% NaOH

** Theoretical (*values*) due to out of range conductimeter measures (max 200mS/cm)

First of all, a reduction of pH is seen, especially in the case of dyeing effluent treatment (BlkSimAA_Pt). This is a positive outcome because any effluent out of legal deposition ranges require adding acids or bases. So electrochemical pH neutralization seems an option to reduce the addition of neutralization agents to wastewater, lowering economic and environmental costs of using such acids and bases. The regional law, Decret 130/2003 (Catalonia, 2003), defines a maximum pH10 to permit sending effluents to be treated at public waste water treatment plants (WWTP), so dyeing wastewater would be already in legal ranges after electrochemical treatment.

Second, an increment in effluent's temperature between 1,4°C and up to 3,9°C, behavior that can be interpreted as an energy loss in the system, because such an increment has no benefit in the treatment of the wastewater or hydrogen production.

Third and lastly, conductivity decreases in the case of MerSim02, while it slightly increases in the case of dyeing effluent treatment (BlkSimAA_Pt). Both increment and decrease are not significant (less than 2% in both cases), so further research with a more efficient and compact system should be performed to see whether such tendency goes further or remains at low variation values.

4. Conclusions

The production of hydrogen via alkaline electrolysis of 2 different textile wastewaters, mercerization (MerSim02) and reactive dyeing (BlkSimAA), perform similarly to the one using an ideal electrolyte for alkaline electrolysis (AlkSim25) while using platinum electrodes. The main factors determining the H₂ generation speed as a function of current density are the electrodes' materials and the effluent's conductivity, independently of its actual composition.

The effluent's conductivity fixes the current density for a specific working voltage and, more importantly, the hydrogen production rates, all showing a directly proportional relationship. For example: MerSim02_Pt has a conductivity of 3,02 times BlkSimAA_Pt and their current densities and hydrogen generation speeds relations are 3,19 and 2,85 respectively. Additionally, conductivity affects to the H₂ generation efficiency, resulting into higher efficiencies in effluents of higher conductivities. In fact, conductivity is identified as the main determining factor for H₂ production performance, factor that can be improved artificially to increase the performance.

In the case of the dyeing effluent, a dual process occurs: hydrogen production at the cathode and color elimination at the anode (Figure 1). Hydrogen is generated following the same behavior as the other 2 effluents tested with platinum electrodes (AlkSim25_Pt and MerSim02_Pt) and confirms its independency from the content on other compounds and contaminants such as the ones in dyeing wastewater: dissolved salt (NaCl) and black textile dye (Remazol Black B-133).

The maximum color removal found is 99%, proving the electrochemical treatment of color based on reactive dyes in textile wastewaters is possible while hydrogen is produced. Consequently, energetic and economical performances of the treatment system can be improved. In the case of using hydrogen as a substitute of natural gas at the water boilers, its combustion would be able to recover up to 19,5% of the electrical energy consumed during the electrochemical treatment.

Some future works identified include: changing the Hofmann Voltammeter used by a more compact electrochemical cell, and identifying economic and environmental benefits over current treatments used. In the first case, placing electrodes closer should increase system's performance and allow more industrially scalable testing conditions. The second case is required to push electrochemical treatment processes of wastewater into industry. Some of the benefits expected to be found are: electrification of wastewater treatment, independence from chemicals supply chain, carbon neutrality of the process via renewable energy self-consumption, and CO₂ emissions reduction via H₂ by-product substitution of natural gas for process water heating.

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Communication aligned with the Sustainable Development Objectives

