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Article

Treatment of Domestic Wastewater Through a Pilot Treatment Plant that Applies Selective Ion Flow Cell Technology

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Abstract: The capacity to treat synthetic and real wastewater of a small-scale pilot wastewater treatment plant (WWTP) using the Selective Ion Flow Cells (SIFC) technology (SIC patent title 37239) was evaluated, for this purpose, a one-factorial experimental design was carried out with samples of synthetic wastewater prepared in the laboratory. The relevant factor used was the flow of the sample and the response variables were hydrogen production (clean energy) and different physicochemical parameters: COD, fats and oils, color, pH, conductivity and total solids. The results obtained show that the best flow to treat wastewater was 50 mL/min with a hydraulic retention time (HRT) of 5.33 hours, reducing synthetic wastewater quality parameters such as COD by 90.54 wt %, fats and oils by 93.8 wt %, apparent color by 90.7 %, true color by 85.4 %, conductivity by 80.9 % and total solids by 83.7 wt % which comply with resolution No. 0631 of 2015 for discharges in Colombia.

Keywords: wastewater; green hydrogen; pilot WWTP; electrochemistry; selective ion flow cells

1. Introduction

The treatment of wastewater dates to the end of the 1800s [1–3], when hygiene mechanisms were intensified to prevent waterborne diseases. In 2002, Engineer Ricardo Rojas from the World Health Organization presented, in the Integral Course on Wastewater Treatment, a history of how wastewater treatment began, giving great relevance to the attempt of researchers to generate new technologies to reduce pollutants in water resources [4].

The production of wastewater is something inevitable and inherent to human beings, as long as human beings need to consume food, clothing, toiletries, technology, among others, the production of waste is permanent and continuous [5,6], much of this waste ends up in the water resources of the planet [7] so it is important to know the types of wastewaters that are generated and their composition in order to stipulate an appropriate treatment method [8].

Domestic wastewater (DWW) involves all wastes that are from living and service areas, generated mainly by human metabolism and domestic activities; domestic wastewater consists mainly of paper, soap, urine, feces, and detergents [9–11].

Industrial wastewater (IWW) presents varied wastes as they depend on the specific processes of the industries from which they originate [12–15]; in some cases, industrial wastes are released directly into the nearest water effluents which becomes a much greater polluting factor, due to the variability in the composition of industrial wastewater, the appropriate treatment is specific to each activity [16,17]. Therefore, in the present research work, it was decided to focus only on domestic wastewater.

Several authors mention that to treat domestic and industrial wastewater, it is very important to implement new technologies [18–23], procedures and treatment lines that involve “integrated unit processes” in order to eliminate the traditional idea that a Wastewater Treatment Plant is a simple

cement structure in which the water to be treated and different reagents are deposited, all of this in order to generate new alternatives and patents [24].

The Selective Ion Flow Cell System (SIFCS) is a patented system [25] that takes advantage of the oxidation of organic molecules in the excreta to generate high purity hydrogen as an alternative and clean energy source with low energy consumption [26–28]. This system separates the oxidation and reduction half-reactions in two different compartments by means of a membrane system and an electrical bridge system; SIFC combine two chemical phenomena, the phenomenon of electrolysis of organic matter (anode) and the law of chemical equilibrium (cathode); these cells allow the selective flow of ions between the two compartments.

The SIFC is a system that among its structure has a pair of membranes that serve as an exchange of substances between the anode and the cathode selective to certain species, it also has a system of electric bridges that help to direct ions by certain paths due to the electric fields (Figure 1). Because of the above, this electrolytic cell is called selective ionic flow cell [25].

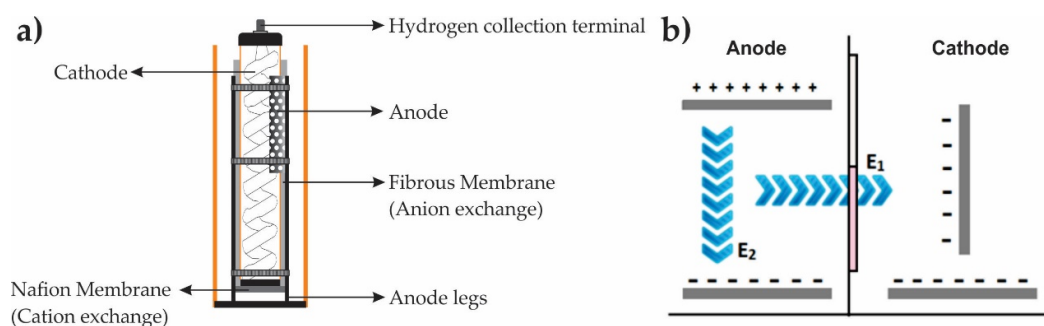


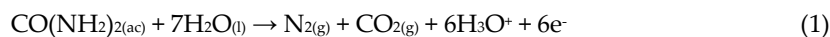
Figure 1. a) General scheme of the SIFC system; b) General representation of the directions of the electric fields in the cell.

Due to the configuration of the SIFC the tendency in the anode is that the H_3O^+ ions produced after oxidation in the positive plates migrate towards the negative plates of the same compartment because of the potential difference E_2 , however in this system, the potential difference E_1 is greater than E_2 , therefore the ions deviate from their first trajectory towards the cathode; they cross the cation exchange membrane (Nafion), lower membrane in Figure 1; it is also possible that a certain fraction of protons cross the stretch membrane (upper membrane in Figure 1) because the protons fit comfortably through the pores of this film which has a pore size around 30 Å, while the hydronium ions have a size close to 3 Å [25].

Figure 1 shows in a general way the SIFC system (a) and how the arrangement of the steel plates causes the directions of the electric fields represented by blue arrows (b). According to this figure, at the anode there are 2 types of plates, some positively charged (at the top) and others negatively charged (at the bottom). Between the anode plates a potential difference E_2 is generated with vertical direction, from the positive to the negative plate. At the cathode, the plates of importance that affect the direction of the electric field are negative; the potential difference generated between the anode and cathode plates is called E_1 . The SIFC has a system of electrodes and electric bridges that generate two electric fields perpendicular to each other. The negative plates at the lower end of the anode and cathode in Figure 1 represent the electric bridge [25].

As already mentioned, the oxidation reaction at the anode occurs in a basic medium, but the research of Lozada-Castro, 2020, has shown that considering the chemical equilibrium of water, the reaction also occurs in a neutral medium (using a salt as electrolyte). In Equations (1) to (3) the above is evident taking urea as example: at the anode urea is oxidized only in the presence of water generating nitrogen, carbon dioxide, hydronium ions and electrons; at the cathode hydronium ions are reduced by electrons to generate hydrogen and water.

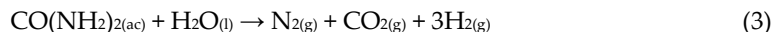
Anode



Cathode



Total equation



Equations (1) and (2) give a clearer explanation of hydrogen production from the oxidation of urea in an electrolytic cell with cathode and anode separated into two compartments. The explanation is because the hydrons generated at the anode migrate through the membranes to the cathode where they participate in the cathodic half-reaction.

The explanation in this process of the chemical equilibrium effect of water is based on the shift of equation 4 to the right (towards the products). As evidenced in this equation, there is production of hydronium ions which at equilibrium react with hydroxyls to generate water, however, if this reaction is subjected to external disturbances, it can shift to one of the two sides of the equation (either towards the products or towards the reactants). In SIFC, the need for hydroxyl ions in the urea oxidation half-reaction and the need for hydrons to produce hydrogen at the cathode at slightly elevated potentials incentivizes the shift of the water chemical equilibrium reaction to the right.



In an experiment in which aqueous urea and sodium bicarbonate were added to the anode as electrolyte, and water and phosphoric acid to the cathode, initially, in the absence of electric field, the transport of protons would be from the cathode to the anode, this contrasts with what has been explained so far. The reason for this counterproductive shift is because the acid constant of H_3PO_4 is much higher than the constant of water, which leads to the concentration of hydronium ions being much higher at the cathode, and because of the diffusion phenomenon, the clear tendency is for the protons to travel towards the anode. However, when the cell is subjected to the electric field, the phenomenon of diffusion in the transport of hydronium ions is no longer relevant since in this case the phenomenon of migration predominates, strongly influenced by the differences in applied potential in the cell.

The presence of the electric field allows current flow, therefore for the current to be maintained it is necessary that the ions are transported in the established directions which are: hydrons from the anode to the cathode and hydroxyls from the cathode to the anode (Figure 2). It is necessary to clarify that not only through the membranes there are flows of positive ions, but there is also transport of negative ions (through the stretch membrane).

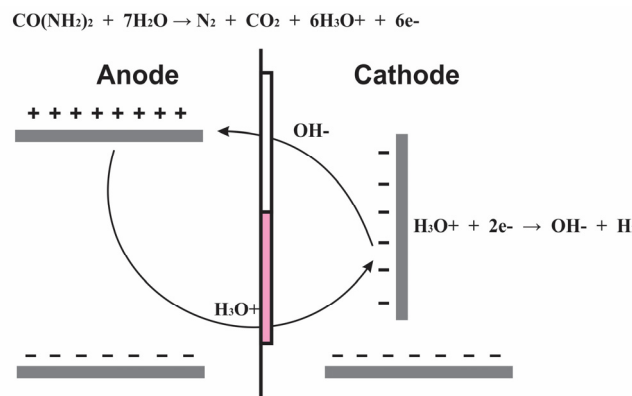


Figure 2. Graphical representation of the movement of ions.

Figure 2 shows in a general way the processes mentioned so far, mainly representing how the movement of ions through the membranes is carried out. The hydroniums produced at the anode cross the lower membrane (nafion), while the hydroxyls generated at the cathode cross the upper membrane (stretch) to the anode.

In this order, it was considered to include selective ionic flow cells in a process involving different mechanisms in order to evaluate the capacity to treat synthetic and real wastewater to evaluate the reduction of pollutants and the production of hydrogen gas in order to propose an alternative to conventional wastewater treatment and stipulate whether it is possible to produce energy while decontaminating the wastewater.

In this work, the capacity to treat domestic wastewater of a pilot WWTP that applies the technology of selective ionic flow cells (SIFC) was evaluated with wastewater samples prepared in the laboratory and its application to wastewater samples from the city of Pasto Colombia by determining the reduction capacity of different physicochemical parameters that are regulated for discharges and also by calculating the amount of hydrogen produced.

2. Materials and Methods

This WWTP applies several new technologies, among which are the Selective Ion Flow Cells (SIFC), in Figure 3 we can see in a general way the parts of the WWTP.

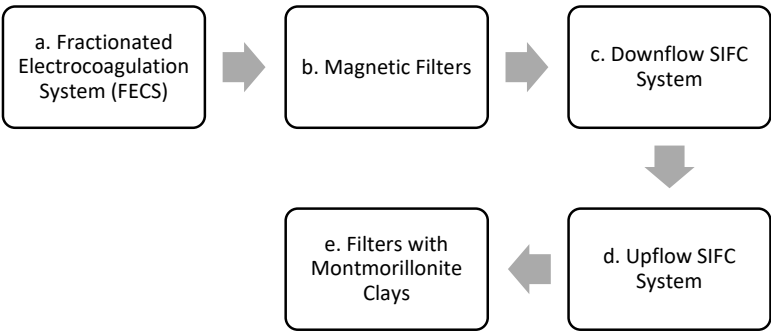


Figure 3. **a.** Technology pending intellectual protection, **b.** Technology pending intellectual protection, **c,d.** SIFC technology, **e.** Conventional system.

The portable pilot WWTP built has a capacity of 1 L/min, is fully functional (see Figure 4), which is used for research and previous studies to scale up to a real WWTP (in-situ simulation, modeling, scaling and designs of the projects to be developed), according to previous studies carried out.

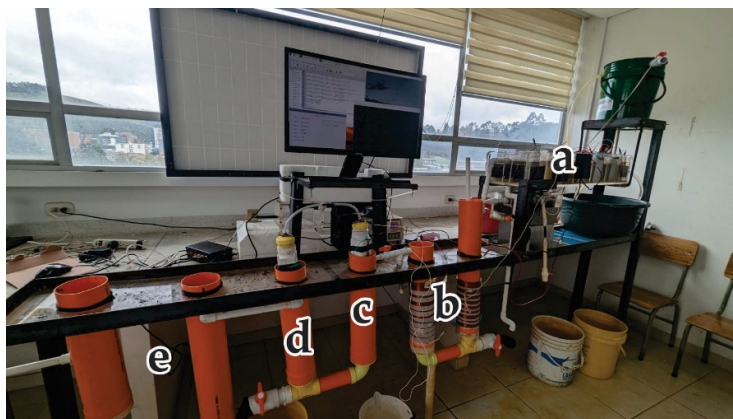


Figure 4. Fully functional portable WWTP applying SIFC technology, with a capacity of 1L/min, useful for simulation studies, modeling and scaling of full-scale WWTPs. a) Fractionated Electrocoagulation System (FECS), b) Magnetic Filters, c—d) SIFC, e) Filters with Montmorillonite Clays.

The implementation of the WWTP required the development of other complementary technologies: FECS, magnetic filters, which will be subject to intellectual property protection.

2.1. Fractionated Electrocoagulation System (FECS)—a

The photograph shows that it has been built in an acrylic box 1 cm thick (80 cm x 22 cm x 12 cm), the box has 4 sets of stainless steel electrodes connected in Series with an approximate potential in each set of 3V, this system combines polarities of the electrodes allowing the ions to remain longer in the box, which resembles an ion trap in liquid phase; this allows the nucleation processes to occur in this box with the cations present in the wastewater, coagulation, flocculation and sedimentation in continuous flow, it should be noted that the flow is ascending; this innovation has solved the problem of foam generation and other undesirable reactions such as electrolysis.

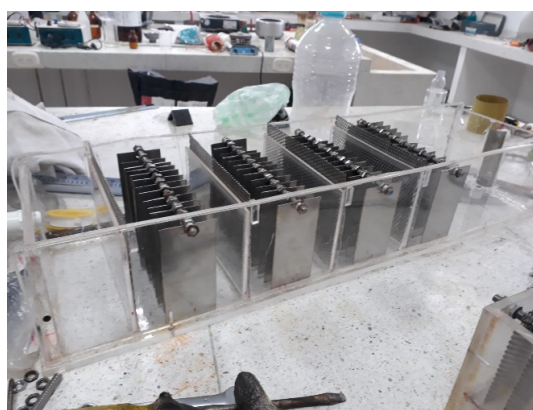


Figure 5. Fractionated electrocoagulation system built.

2.2. Magnetic Filters—b

These filters use an induced magnetic field, which is obtained by winding a coil around the 4 inches tube, internally a filler material is used which has a high magnetic permeability and becomes magnetically charged and when ions pass through this material they are attracted to the surface of the particles and are retained; the coil operates at 6V each and 0.5A.



Figure 6. Magnetic filters built.

2.3. SIFC—c, d

This system separates the oxidation and reduction half-reactions into two distinct compartments by means of a membrane system that allows the selective flow of current through the ions formed in the reactions by the electrolytic system, so the oxidation of organic molecules in the wastewater takes place in an anode compartment, generating electrons that migrate to the cathode, where the reduction and production of hydrogen occurs, all of this according to [25].

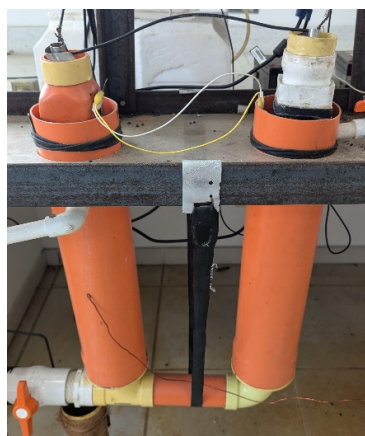


Figure 7. Selective ion flow cells.

2.4. Clay Filters—e

These are filters with clays formed sedimentarily in volcanic areas such as montmorillonites which use the adsorption principle to remove ions and other particles in the final stage of wastewater treatment. Industrial applications of montmorillonites include their use as oil decolorizers, as adsorbents for pesticides or heavy metals in wastewater and as heterogeneous catalysts to promote chemical reactions [29]. The clays were pre-washed with 0.1 M HCl and hot water; about 2 kg of clays were used in each filter.



Figure 8. Two clay filters with montmorillonites.

2.5. Synthetic Wastewater Sample Preparation

After reviewing the general composition of domestic wastewater and the available materials, the decision was made to prepare the sample as follows:

Table 1. Materials used in the preparation of the synthetic wastewater sample.

Component	Brand	Formula	Amount (mg/L)
Powdered milk	El Rodeo	----	250
Unflavored gelatin	Gel'hada	----	250
Potassium sulfate	----	K ₂ SO ₄	125
Ammonium chloride	----	NH ₄ Cl	125
Sugar (sucrose)	----	C ₁₂ H ₂₂ O ₁₁	250
Human urine	----	----	8,3 mL/L

The urine used for the preparation of the samples was collected for several days in a dark container, prior to mixing with each sample the pH was adjusted to 11 with 30% ammonium hydroxide; the powdered milk, gelatin and sugar were mixed together in a beaker with hot water until complete dissolution, the salts were dissolved in cold water directly in the 20 L flask, once everything was in the flask the urine was added with the pH adjusted and the water was added until the 20 L were completed, after stirring constantly for 10 minutes the sample was divided into 2 flasks and 5 L of water were added to each one to complete a total of 30 L of sample. Now after addition of urine, the sample changes from a whitish color to a dark color and takes on a characteristic odor, very similar to sewage water. Note that 30 L of sample were prepared for each experiment. Finally, the sample was ready to be fed to the pilot WWTP.

2.6. Hydrogen Collection and Quantification

The hydrogen produced inside the SIFC was collected by means of a vacuum pump that pulls it through the flow, hydrogen concentration and current sensors. The H₂ was measured by the Mhydros® system [30] and collected at low pressure in a conventional iron cylinder.

These sensors were connected using a Raspberry electronical network that through a software, shows the hydrogen concentration, temperature, pressure, among other data; Figure 9 shows an example of these observations.

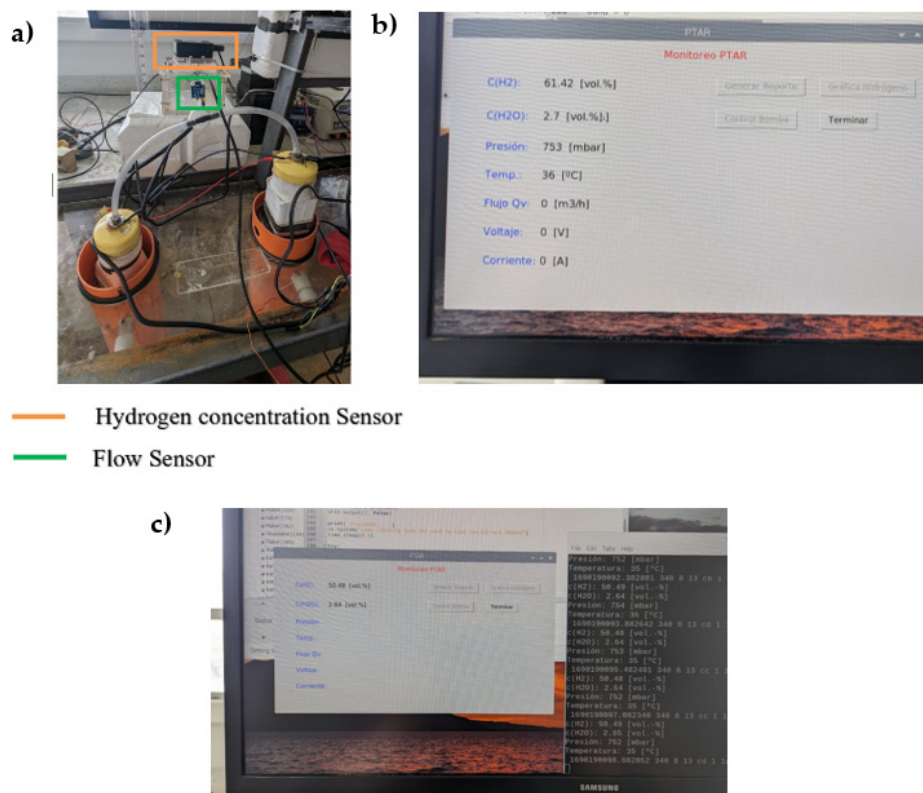


Figure 9. Hydrogen quantification method Mhydros®; a) Sensors organization, b—c) Observation examples.

2.7. Experimental Design

A single-factor experimental design ¹⁴ was developed taking the hydraulic retention time (HRT) as the only factor, which refers to the time in which the synthetic DWA sample is in contact with the plates of the electrocoagulation system and the SIFC; to this it was necessary to control the flow at which the sample moves through the hydraulic system of the pilot WWTP, for this purpose two keys were used at the outlet of the sample storage vessel; to measure the flow a stopwatch and a measuring cylinder were used, the amount of sample leaving the electrocoagulation system in one minute was taken; each flow was measured three times.

The flow rates used were:

F1: 50 mL/min—F2: 100 mL/min—F3: 200 mL/min—F4: 300 mL/min.

All the raw samples obtained at the different flow rates were obtained in triplicate, labeled and stored refrigerated at 5 °C for subsequent analysis of the parameters mentioned above:

- COD: Standard Methods Edition No. 23 5220- D—Technique: Colorimetric. [31]
- Fats and oils: Standard Methods Issue No. 23 5520—D—Technique: Gravimetric. [31]
- Apparent and true colors: Standard Methods Edition No. 23 2120—C—Technique: Colorimetric. [31]

3. Results and Discussion

3.1. Hydraulic Retention Time (HRT)

As shown in Figure 4, the pilot WWTP consists of several sections, to determine the HRT for each flow it was necessary to measure the volume of each of the sections and thus, with triplicate tests, calculate the time that the sample is under treatment for each flow, using Equation (5).

$$HRT = \frac{V(m^3)}{Q(m^3/h)} \quad (5)$$

where:
HRT: Hydraulic retention time
V: Volume in cubic meters
Q: Flow in cubic meters per hour
Equation (5) was applied for each flow in triplicate, the data obtained are shown in Table 2.

Table 2. HRT for each flow.

Sign	Flow (mL/min)	Total HRT (h)
F1	50	5,33
F2	100	2,67
F3	200	1,34
F4	300	0,88

By means of these data, it was possible to obtain an estimate of the hydraulic retention time that a large-scale WWTP should have, in its totality and in each section, to achieve results approximating those presented in this work with synthetic domestic wastewater.

3.2. . Experimental Design Results

After treating the samples in triplicate, applying the different flows, the physicochemical parameters mentioned above were measured, obtaining the data shown in Table 3.

Table 3. Parameters of samples treated in the pilot WWTP at different flows in triplicate.

Sign	COD (mg O ₂ /L)	Fats and oils (mg/L)	Apparent Color (UPC)	True Color (UPC)	pH	Conductivity (μS/cm)	Total Solids (mg/L)
Raw Sample	955,3	162,4	287,5	54,5	8,5	1676	824
Raw Sample	962,1	159,2	288,1	56,0	8,5	1712	833
Raw Sample	958,6	163,4	287,7	54,6	8,5	1684	831
F1	92,0	<10	28,4	<8	7,3	321	121
F1	89,2	<10	25,2	<8	7,4	311	147
F1	90,8	<10	26,9	<8	7,3	335	138
F2	292,0	<10	28,6	<8	7,1	445	343
F2	315,5	<10	30,8	<8	7,4	421	334
F2	303,9	<10	31,3	<8	7,1	457	345
F3	338,8	<10	58,1	<8	7,7	387	354
F3	317,4	<10	55,0	<8	7,5	391	374
F3	343,8	<10	62,8	<8	7,5	373	360
F4	453,5	93,1	83,4	<8	8,2	865	550
F4	447,5	66,8	88,1	<8	8,0	844	513
F4	423,1	56,7	75,5	<8	8,0	868	541

From the data obtained after treating the synthetic domestic wastewater sample with the Selective Ionic Flow Cells technology and the other systems such as magnetic filters and the Fractionated Electrocoagulation System, through the pilot WWTP, it was determined that the F1 sample complies with the discharge requirements stipulated in Resolution No. 0631 of March 17, 2015 of MINAMBIENTE—Colombia [32].

Table 4. Comparison of the average parameters of samples treated in the pilot WWTP with the maximum normative for discharges.

Sign	COD (mg O ₂ /L)	Fats and oils (mg/L)	Apparent Color (UPC)	True Color (UPC)	pH	Conductivity (μS/cm)	Total Solids (mg/L)
Maximum Normative	200,00	20,00	NR	NR	6–9	NR	100,00
Average raw sample	958,67	161,67	287,8	55	8,5	1690,7	829,33
Average F1	90,66	<10	26,8	<8	7,3	322,3	135,33
Average F2	303,85	<10	30,2	<8	7,2	441	340,67
Average F3	333,30	<10	58,6	<8	7,6	383,7	362,67
Average F4	441,39	72,2	82,3	<8	8,1	859	534,67

The statistical analysis showed that there are significant differences between the flows used to treat the synthetic wastewater samples, which influence all the response variables, except for fats and oils and true color. Multiple range tests were performed to determine the correlation between flows within the same treatment. Some examples are shown in Figure 10.

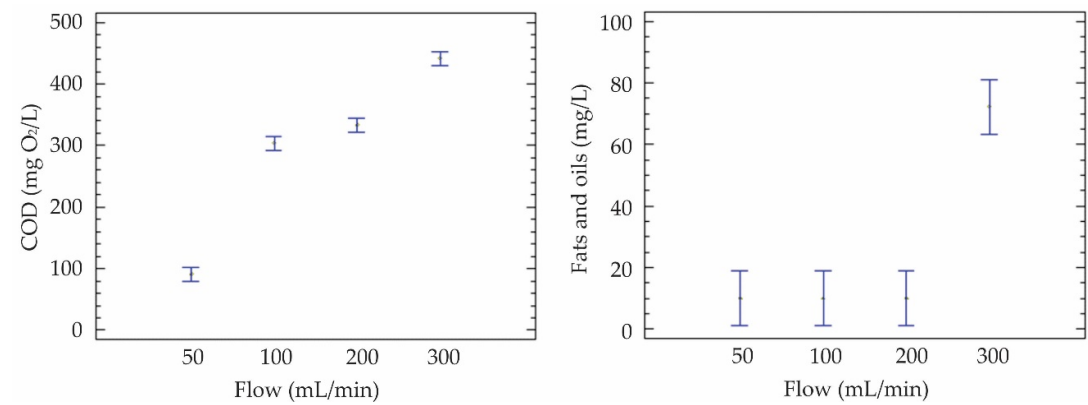


Figure 10. Average graphs for COD and fats and oils.

Figure 11 shows photographs of the treated samples, the raw sample had a cloudy yellow color and as they were treated, it became clearer until it became translucent, like potable water.

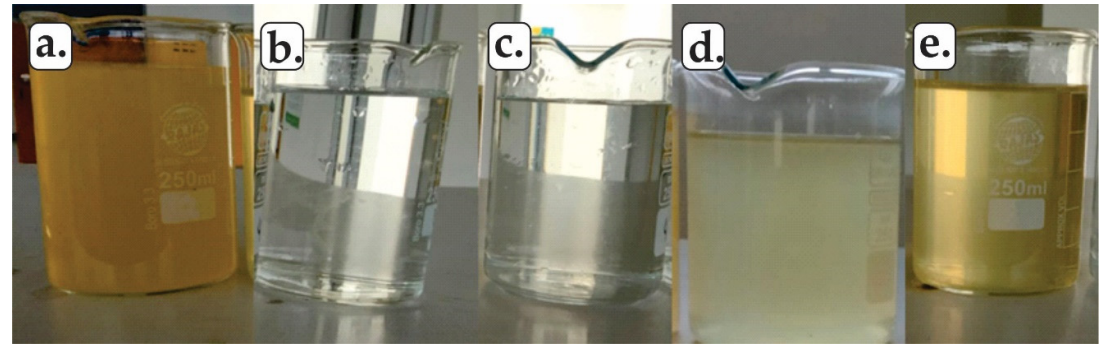


Figure 11. Photographs of samples: a) Raw sample, b) Treated sample F1, c) Treated sample F2, d) Treated sample F3, e) Treated sample F4.

In addition to the previously mentioned, a search for COD reduction data with conventional techniques was carried out, in [33] a comparison of 7 reactors was made determining the different

percentages of COD reduction, the results obtained in this research show that they were able to reduce COD levels by 84%, 80%, 16%, 93%; 67%, 58% and 68%; which shows that the SIFC technology is more efficient in the reduction of organic matter; however, it is important to carry out a study of energy efficiency in terms of the use of the hydrogen produced and the energy consumed, which can be obtained from solar panels.

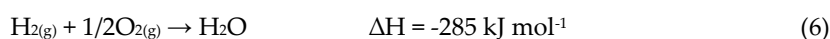
3.3. Hydrogen Production

The hydrogen produced by the Selective Ion Flow Cells (SIFC) was quantified by the Mhydros® system [30], an example shows in **Figure 9**, the results are shown in Table 5.

Table 5. Hydrogen produced values.

Sample	% H ₂ (w/v) Average	H ₂ Flow (L/h)	Moles of H ₂ /h
F1	96	120	3,53
F2	92	120	3,38
F3	90	120	3,32
F4	88	120	3,28

Taking into account the data obtained from the hydrogen produced and the enthalpy of combustion, the energy potential was determined as follows:



$$(3.53 \text{ molH}_2) \left(\frac{285 \text{ kJ}}{1 \text{ molH}_2} \right) = 1006.05 \text{ kJ} \quad (7)$$

As can be seen, treating wastewater with this technology produces an estimated 3.53 moles of hydrogen gas per hour, which is equivalent to approximately 1006.05 kJ per hour of energy that could be obtained from a fuel cell.

3.4. Wastewater Sample from Pasto City

The same physicochemical parameters that were analyzed with the synthetic samples were evaluated with this sample; this sample was treated with the F1 flow (50 mL/min), since this value was the one that yielded the best treatment results. The data obtained are shown below.

Table 6. Parameters of real DWW sample treated in the pilot WWTP with F1 flow.

Sign	COD (mg O ₂ /L)	Fats and oils (mg/L)	Apparent Color (UPC)	True Color (UPC)	pH	Conductivity (μS/cm)	Total Solids (mg/L)
Raw Sample	712,0	96,9	462,9	81	7,5	1232	663
F1	98,7	20,0	8,6	<8	7,3	244	112
Maximum Regulation	200,0	20,0	NR	NR	6–9	NR	100,0

It was possible to determine that the real DWW sample meets most of the requirements of Resolution No. 0631 of 2015 for discharges in natural tributaries; as with the synthetic DWW samples the only parameter that does not meet the maximum standard is the Total Solids, it is recommended to conduct further studies in this regard.

5. Conclusions

Through this research it was possible to treat samples of synthetic domestic wastewater by means of a pilot WWTP using SIFC technology. It was determined that the best flow to treat the DWW samples in the pilot WWTP is 50 mL/min, with a hydraulic retention time of 5.33 hours.

It was possible to reduce COD by 90.54 wt %, Fats and oils by 93.8 wt %, Apparent Color by 90.7%, True Color by 85.4%, Conductivity by 80.9% and Total Solids by 83.7 wt % in synthetic wastewaters complying with the regulations for discharges related to the measured parameters, said regulations set forth in Resolution No. 0631 of 2015, it was possible to reduce the pollutant load of organic matter by 86.14 wt % through an approximate treatment time of 5.33 hours for a sample of DWW from the City of Pasto through the WWTP—SIFC.

Through the WWTP/SIFC a maximum hydrogen production of 3.53 moles of hydrogen per hour was obtained at an DWW flow of 50 mL/min with 14V and 1.3A.

Author Contributions: Conceptualization, Lozada-Castro, Juan José.; methodology, Bastidas-Obando, Jonnathan David; formal analysis, Bastidas-Obando, Jonnathan David and Lozada-Castro, Juan José; investigation, Bastidas-Obando, Jonnathan David and Guerrero-Fajardo, Carlos Alberto.; resources, Guerrero-Fajardo, Carlos Alberto.; data curation, Bastidas-Obando, Jonnathan David; writing—original draft preparation, Bastidas-Obando, Jonnathan David; writing—review and editing, Bastidas-Obando, Jonnathan David and Guerrero-Fajardo, Carlos Alberto; visualization, Bastidas-Obando, Jonnathan David and Lozada Castro, Juan José; supervision, Guerrero-Fajardo, Carlos Alberto; project administration, Lozada-Castro, Juan José and Guerrero-Fajardo, Carlos Alberto; funding acquisition, Lozada-Castro, Juan José and Guerrero-Fajardo, Carlos Alberto. 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

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