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Research Article

Cold Plasma Technology for Water Treatment: Design, Assembly and Technical Evaluation of A Pilot Plant

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ABSTRACT

The development of innovative oxidation process methods for wastewater treatment remains a major challenge worldwide. Cold discharge plasma is considered a promising remediation technology because it is a low-cost and easy-to-operate process. In this work, a system with a reactor for water treatment using plasma technology was designed, assembled and experimentally evaluated. The water to be treated was circulated by spraying under controlled conditions through a borosilicate glass cylindrical reactor at room temperature and a flow rate of 2.5 liters/min; to which a potential of up to 10,000 volts and a relatively high electrical frequency were applied. Under these conditions, the water is converted into a plasma state, in which ultraviolet light, hydrogen peroxide, H2O2, ozone, O3, and hydroxyl radicals, OH·, are generated. Organic materials, viruses and bacteria present in the water are oxidized. When synthesized samples were processed with treated water, ethyl alcohol and some coliforms, it was possible to quantify that their Chemical Oxygen Demand (COD) was reduced by 49% in best, and the microorganisms disappeared. It was found that the degradation of ethanol depended on the initial concentration of the synthesized samples, being more efficient for lower concentrations, so present system is proposed as a tertiary treatment.

Keywords: treatment, viruses, Organic, H2O2

1. INTRODUCTION

Due to rapid population growth and global climate change, the demand for drinking water has increased considerably, so other non-conventional water treatment alternatives have had to be developed through physical, chemical and biological processes. However, these techniques have the disadvantage of generating waste, and also to have high investment and operating costs. To avoid these problems, scientists have focused on developing advanced oxidation processes (AOP), such as photo-Fenton and non-thermal plasma (NTP) techniques.

Plasma water treatment has major advantages such as its simplicity, effectiveness in destroying toxic organic compounds in both raw water and waste water, lower investment than traditional technologies, lower operating costs, it does not generate waste, it does not consume reagents, it reduces the COD content in the treated water and eliminates pathogenic microorganisms, it tolerates highly conductive water and has reasonable energy consumption as it is a non-thermal process, since it is not necessary to heat the water as this occurs at room temperature.

Plasma oxidation has the advantages of high efficiency, no secondary pollution and green environmental protection [1]. In the process of plasma treatment, high-energy electrons and neutral radicals constantly collide to form active substances including electrons, ions, active radicals and ultraviolet light [1, 2] which cause various chemical reactions and physical changes.

1.1. Chemical Reactions

Plasma has been shown to be a highly selective and energy-efficient way to enhance chemical reactions [1]. Generally, there are three discharge modes to treat liquid solutions such as discharge above the liquid surface, direct discharge into the liquid and bubble/vapor discharge into liquid [3,4]. During these discharges, sufficient radicals and species will be produced.

The following summary of reactions occurring in the formation of plasma was taken from Yunqiu Cui et al. [5].

1. Reactions based on hydroxyl groups

Decomposition, ionization, rotational and vibrational excitation reactions will occur when high voltage electric energy is discharged into water. These processes can produce hydroxyl and hydrogen radicals as the reactions shown in equations (1)–(7) [1, 6, 7]:

$$H_2O + e \rightarrow OH\cdot + H\cdot + e \quad (1)$$

$$H_2O + e \rightarrow H_2O^+ + 2e \quad (2)$$

$$H_2O + H_2O^+ \rightarrow H_3O^+ + OH\cdot \quad (3)$$

$$H_2O + e \rightarrow H_2O^* + e \quad (4)$$

$$H_2O + H_2O^* \rightarrow H_2O + OH\cdot + H\cdot \quad (5)$$

$$H_2O + H_2O^* \rightarrow H_2O + O\cdot + H_2 \quad (6)$$

$$H_2O + H_2O^* \rightarrow H_2O + O\cdot + 2H\cdot. \quad (7)$$

Note H_2O^* is an activated water molecule

Hydroxyl plays a crucial role in water treatment. Shao P. R. et al. [8, 9] investigated the role of hydroxyl radical $OH\cdot$ in the degradation efficiency of sulfadiazine. A similar method was used in the study of wastewater treatment by Min Wang et al. [10], who also showed that $OH\cdot$ plays a major role in the degradation of antibiotic residues in equation (8). In addition, the degradation was also relative to other active particles such as $H\cdot$ and e_{aq}^- in aqueous, where electrons and radicals $H\cdot$ are strong reducing agents in plasma chemistry. $e_{aq}^-$is formed during the radiolysis of water and the radicals $H\cdot$ come from equations. (1) and (5) [10].

$$OH\cdot + other\ species \rightarrow oxidized\ products \quad (8)$$

2. Ozone reactions

When plasma is generated in oxygen, oxygen can promote the formation of hydroxyl groups and also react with oxygen to form ozone equations (9)-(10). In addition, ozone can dissolve in water to generate hydroxyl radicals, whose oxidation ability is much stronger than that of ozone, as shown in equations (11) to (13) [10-14].

$$O\cdot + H_2O \rightarrow 2OH\cdot \quad (9)$$

$$O\cdot + O_2 \rightarrow O_3 \quad (10)$$

$$OH\cdot^- + O_3 \rightarrow HO_2\cdot + O_2^- \quad (11)$$

$$O_2^- + H^+ + O_3 \rightarrow HO_3\cdot + O_2 \quad (12)$$

$$HO_3\cdot \rightarrow OH\cdot + O_2 \quad (13)$$

3. Hydrogen peroxide reactions

Another active molecule is hydrogen peroxide, H_2O_2, which can be formed in the discharge as shown by equations (10)–(11). As is known, H_2O_2 is also widely used in industrial applications [15, 16]. In plasma, hydrogen peroxide will react with e_{aq}^- and $H\cdot$, at the same time as shown by equations (16)–(17) [17]; In addition, hydrogen peroxide will also react with ozone to form free radicals (equation (18)) [17, 18]:

$$OH\cdot + OH\cdot \rightarrow H_2O_2 \quad (14)$$

$$2H_2O \rightarrow H_2O_2 + H_2 \quad (15)$$

$$H_2O_2 + e_{aq}^- \rightarrow OH\cdot + OH\cdot^- \quad (16)$$

$$H_2O_2 + H\cdot \rightarrow H_2O + OH\cdot \quad (17)$$

$$H_2O_2 + O_3 \rightarrow HO_2\cdot + O_2 + OH\cdot \quad (18)$$

4. Hydroxyl groups formation

Ultraviolet (UV) irradiation is inevitable in plasma discharge. UV disinfection [23] and UV degradation [24] have become an alternative chemical treatment in water treatment. It can also decompose hydrogen peroxide into hydroxyl groups during plasma discharge, as shown in equation (19) [6, 21]:

$$H_2O_2 + hv \rightarrow OH\cdot + OH\cdot \quad (19)$$

The aforementioned reactions occur in the different reactors that have been designed for the production of plasma, in our specific case applied to water treatment.

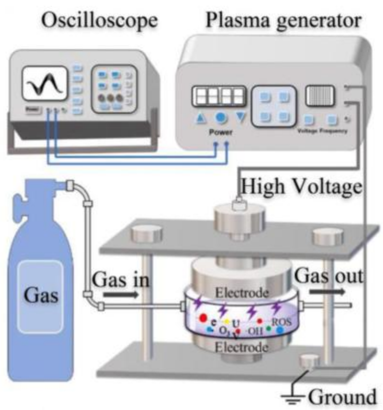
1.2. Plasma reactors

There are several types of plasma reactors that have been tested for wastewater treatment. The types of reactors depend on their configuration, contact form, electrode types, geometry, etc. In this work, we focus our designs and tests in two reactor types: Dielectric Barrier Discharge (DBD) Plasma Reactor, and Pulsed Corona Discharge (PCD) Plasma Reactor; following we show a generic description of them.

1. Dielectric Barrier Discharge (DBD) Plasma Reactor

The DBD is a non-thermal plasma that can be generated at atmospheric pressure, DBD plasma is generated by applying high voltage electrical energy to a gas between two electrodes separated by a dielectric material barrier as shown in figure (1) [24].

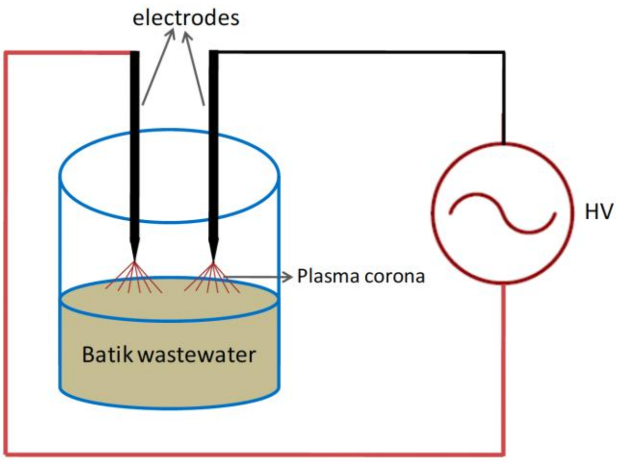
Figure (1) Dielectric barrier discharge (DBD) plasma reactor



2. Pulsed Corona Discharge (PCD) Plasma Reactor

A corona discharge occurs when a high voltage, with direct or alternating current is applied between two electrodes and separated by a neutral fluid such as air or water vapor, by ionization of this fluid, a plasma is then created and the electrical charges propagate from the ions to the molecules of the neutral gases Figure (2) [23].

Figure (2) Pulsed Corona Discharge (PCD) Reactor



2. METHODOLOGY

This work was carried out in five main phases (Figure 3):

1.- Research on different water treatment systems using plasma technology

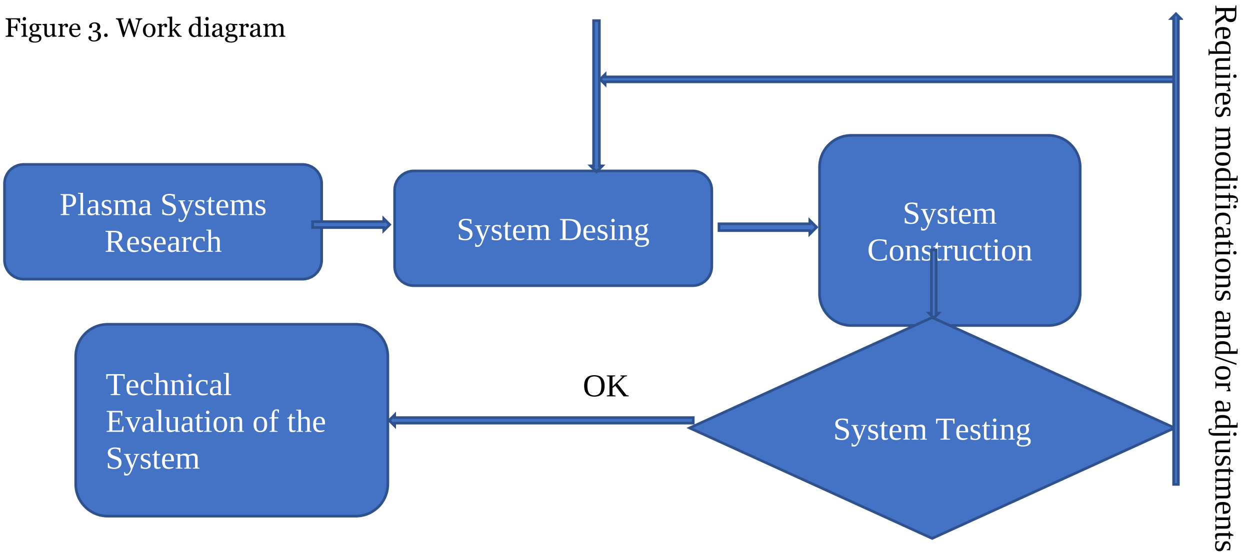
2.- Design of the water treatment system using plasma technology.

3.- Construction of the system.

4.- Operation tests, modifications and adjustments to the design.

5.-Technical evaluation of the system.

Figure 3. Work diagram



2.1. Plasma Systems Research

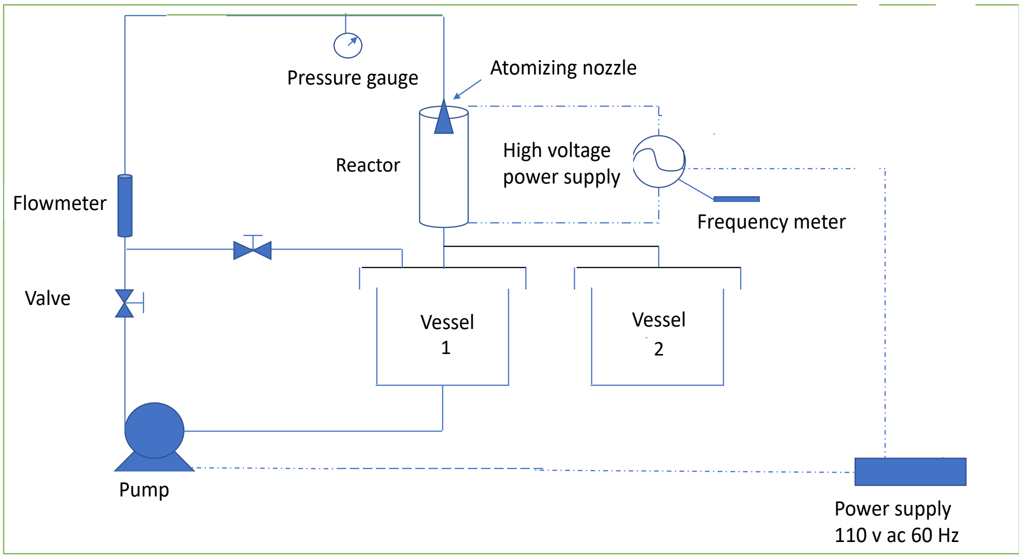
To begin this work, articles on water treatment systems using plasma technology were reviewed in order to consider previous experiences and different system proposals; which would serve as a basis for the design of the system that we propose.

2.2. System Design

In a first step, a coaxial DBD type reactor was designed because it produces a non-thermal plasma that does not significantly raise the temperature; therefore, it does not require a cooling system, making the system cheaper to build. After several initial tests, due to difficulties in forming plasma, it was necessary to modify the design to apply the high voltage current directly to the sprayed water stream using cylindrical rod electrodes.

The designed system is shown in Figure (4): The water to be treated is circulated by spraying it under controlled conditions through a reactor in which it is exposed to a high voltage and high frequency electric energy current, which causes the water to pass into a plasma state.

Figure 4. Diagram of the pilot water treatment system using Plasma technology



The System is basically made up of:

1 20 l fiberglass or plastic container for the water to be treated.

1 1 HP centrifugal pump

1 Fluxometer

1 Pressure gauge

1 Atomizing nozzle

1 Reactor (2 inch diameter borosilicate glass tube)

1 High voltage generator (maximum 20 kv)

1 Frequency meter

Accessories, pipes and connections.

2.3. System Construction

The system was built following the work diagram in figure (1); on a wooden frame, in order to avoid high voltage current jumps In the pipes and accessories polyvinyl chloride and polyethylene tubings were used, it was tried to avoid as much as possible metallic parts in particular in the different sprays that were tested. The exception were the electrodes that initially were of copper and finally of stainless steel in order to avoid carbonization and damage to the electrodes. The reactor was built with borosilicate glass tube of different diameters until finding the most adequate in our case of 2 inches. The High voltage generator has a maximum capacity of 20 Kv of alternating current.

With these elements and the diagram in figure (2) the system was built. It is important to mention that adjustments were made after several tests, such as going from an initial design of the reactor of dielectric barrier discharge type to one of corona discharge pulse type, such as changing the material and diameter of the electrodes in order to find the separation between electrodes that allowed a better plasma formation. Various types of nozzles were tested, initially discarding those with metal parts because they formed electric arcs with the electrodes and then searching among different designs of plastic nozzles, to find those that gave the best result. In the reactor covers, some made of nylamid, rubber and Teflon were tested, also different designs of the covers were tested, in order to avoid water leaks and obtain a better electrodes connection. Additionally, two types of pumps were tested, centrifugal and of diaphragm.

As adjustments were made that allowed for better plasma formation, the constructed system varied, as shown in Photographs (1 and 2) until it finally appeared as shown in Photograph (3).

Photograph 1. Water Treatment System using plasma technology, the reactor was of the pulsed corona type, the borosilicate glass tube is 4" in diameter and copper electrodes, the frame was originally metallic.

Photograph 2. Water Treatment System using plasma technology in 2nd stage, the reactor is pulsed corona type, the borosilicate glass tube is 2" in diameter and stainless-steel electrodes, the frame is made of wood.

Photograph 3. Water Treatment System using plasma technology in 3rd stage, similar to 2nd stage but with improvements in nozzle feed tubing, reactor caps design, and adjustments in water pressure and flow.

2.4. System Testing

When finally, the system was built with all changes, the Operating Manual was prepared, which is important because the system handles high voltage and high frequency electric current, so precautions must be taken to avoid electric arcs and possible burns. For the personnel safety, the next basic instructions described in the manual must be followed.

Steps to follow for the system operation:

1.- Fill the container to 70% with the water to be treated, which has been previously analyzed to subsequently evaluate the performance of the System. This operation must be carried out with all electrical equipment disconnected, making sure there are no leaks.

2.-Allow water to fill the suction pipe and pump by opening the discharge valve.

3.- Energize the pump until there is a stable flow of water sprayed into the reactor (Flow between 2.5 and 4 l/min and pressure between 35 and 50 psi). In the event of any failure in the operation of the pump, the instructions in the pump manufacturer's operating manual must be followed.

4..-Make sure that there are no metal elements near the high voltage generator and its cables.

5.- Energize the high voltage generator according to its User Manual.

6.-Once the system operation has stabilized, samples of the treated water can be taken for analysis at different times to assess the system's performance.

After that, several tests were carried out following the steps mentioned above, until the operating conditions were found that allowed the best plasma formation.

2.5. Technical Evaluation of the System

For the evaluation of the efficiency of the System, batches of water to be treated with ethyl alcohol were prepared at concentrations such that the Chemical Oxygen Demand (COD) of the batches were between 300 and 700 mg/l. Subsequently, these samples of a volume of 15 l were circulated through the system where the formation of plasma was induced (see photographs (4-7). With the water circulating through the system and returning to the feed container, samples were taken at intervals of 15 minutes. The samples were analyzed to determine their COD level and the removal percentage.

Photo 4. Plasma formation (a) Photo 5. Plasma formation (b)

Photo 6. Plasma formation (c)

Photo 7. Plasma formation (d)

3. RESULTS AND DISCUSSION

The results obtained are shown in Table (1) and in Graphs (1-4).

COD removal and percentage of removal efficiency were calculated as:

$$\text{COD removed} = COD_o - COD_f \quad (20)$$

$$\%\ \text{Removal Efficiency} = (COD_o - COD_f)*100/COD_o \quad (21)$$

Table (1) Analysis results of: Initial, Middle and Final COD; COD Removed and Removal Efficiency (%) of the different tests.

Test	COD (mg/l) Initial	COD (mg/l) Middle	COD (mg/l) Final	COD (mg/l) Removed	Removal Efficiency (%)
1a	741	714	667	74	9.98650472
1b	734	708	678	56	7.62942779
1c	756	720	672	84	11.1111111
1d	761	718	650	111	14.586071
1e	731	703	659	72	9.8495212
2a	464	444	307	157	33.8362069
2b	480	453	335	145	30.2083333
2c	412	375	297	115	27.9126214
2d	448	440	300	148	33.0357143
2e	471	461	315	156	33.1210191
3a	408	318	245	163	39.9509804
3b	433	337	261	172	39.7228637
3c	389	319	269	120	30.848329
3d	415	310	278	137	33.0120482
3e	422	325	255	167	39.5734597
4a	336	230	183	153	45.5357143
4b	325	214	181	144	44.3076923
4c	341	241	198	143	41.9354839
4d	315	210	167	148	46.984127
4e	351	249	178	173	49.2877493

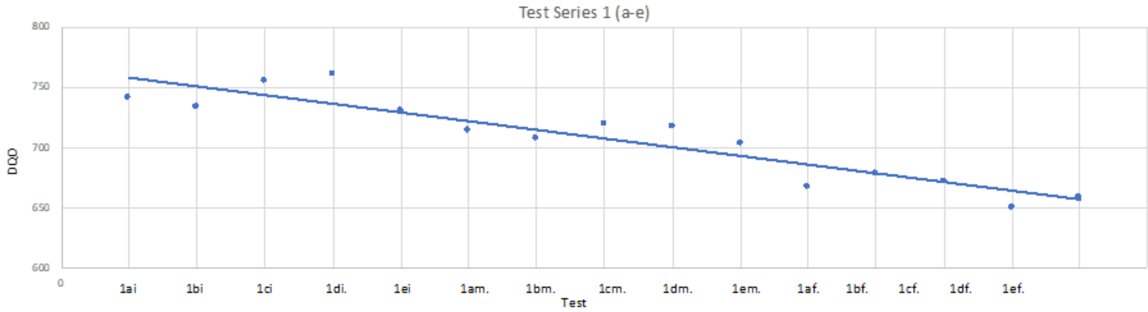


Chart (1). Test Series 1 (a-e). High initial COD. i = initial, m = mean, f = final.

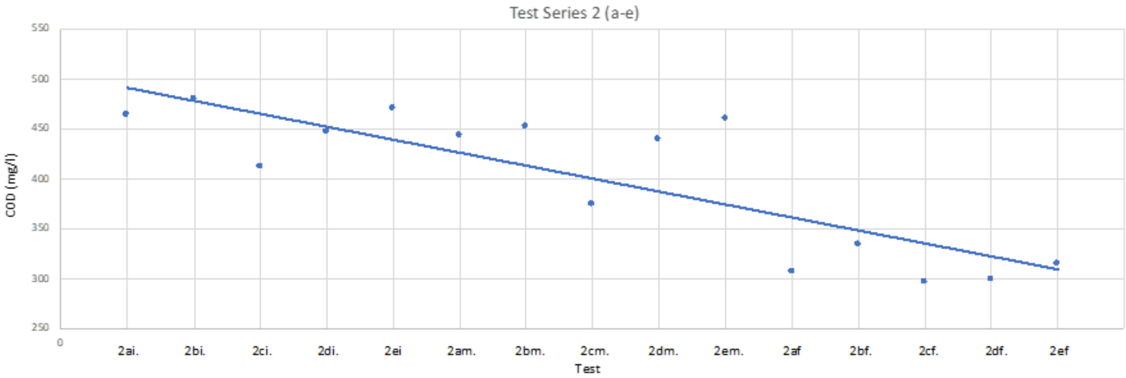


Chart (2). Test Series 2 (a-e). Medium high initial COD. i = initial, m = mean, f = fina

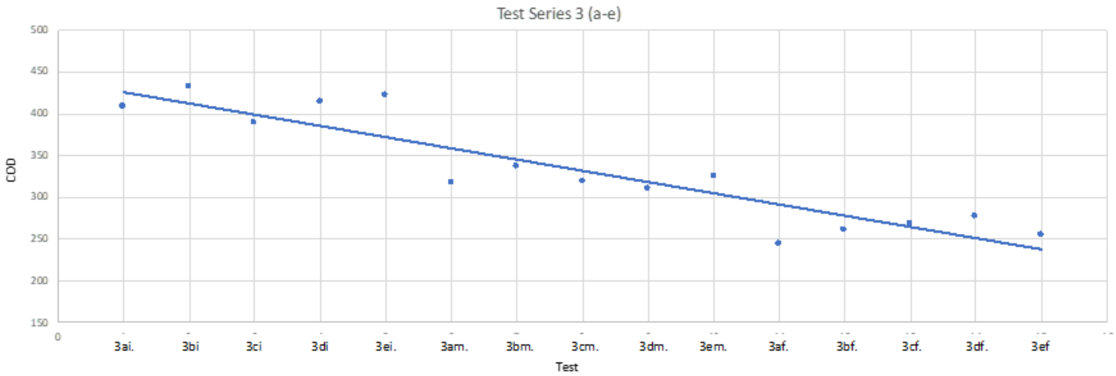


Chart (3) . Test Series 3 (a-e). Medium low initial COD. i = initial, m = mean, f = final.

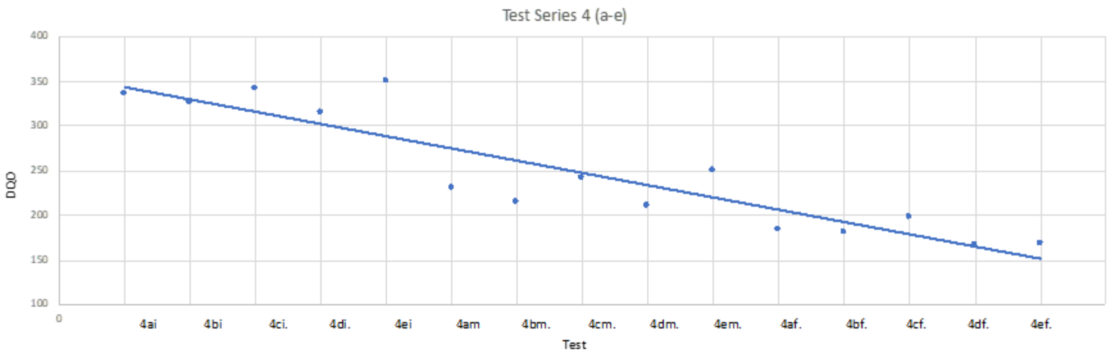


Chart (4). Test Series 4 (a-e). Low initial COD. i = initial, m = mean, f = final.

In the above information, we can observe that when initial COD was about 700 mg/l, as an average the COD removed was 79.4 mg/lt and removal efficiency 10.63%, when initial COD was between 400 and 500 mg/l, they had as an average 148 mg/l of COD removed and a removal efficiency of 34.12%, and when initial COD was between 300 and 400 mg/l, as an average the COD removed was 152.2 mg/lt and removal efficiency 45.61%.

On the basis of above information, it is evident that the higher the initial COD value, the lower COD removed and the removal efficiency, so it is recommended to use the developed system as a tertiary treatment, coinciding with what was proposed by Naicker, KI. et al [22].

On the other hand, in the best case, the COD level of the prepared water batches was reduced by a maximum of 49.28%.

Additionally, during the tests it was observed that the water to be circulated through the System must be free of suspended solids and/or colloidal suspensions, therefore, if wastewater with the aforementioned characteristics is to be used, the water must be passed through a filter. It was also observed that the higher the temperature of the water to be treated, the removal efficiency, and even the formation of plasma, decreases, so a temperature range of 15°C to 30°C in the water to be treated turned out to be the best for the operation of the System. The latter agrees with that reported by Petri Ajo et al. [12], in their supplementary discussion.

4. CONCLUSIONS

The equipment designed and built allows the formation of plasma with the water vapor that is formed when spraying the water to be treated and reducing its organic load present in the water through the formation of $OH\cdot$ radicals, Ozone, H_2O_2 and UV light.

It is important to note that the system is now assembled and ready to operate, so that, in subsequent work, tests can continue to be carried out in order to find the operating conditions that allow optimizing the removal of organic load, and with the same purpose, test the use of catalysts such as ferrous sulphate or titanium dioxide, or even the combination of plasma technology with another type of treatment, for example, filtration with activated carbon.

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