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NON-THERMAL PLASMA MINI-REACTORS FOR WATER TREATMENT

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Abstract

The non-thermal plasma technologies (NTP) have been proved to be an alternative for air and water pollutants treatment. The goal of this paper is to emphasize the effect of the different non-thermal plasma mini-reactor configurations on the soluble organic compounds' removal from water. Therefore, a soluble organic dye (Reactive Blue 19) solution has been treated in an NTP experimental reactor using different electrodes configurations. The influence of the different geometrical configuration of the reactors, such as electrodes shape and distance between them, on Reactive Blue 19 dye decolourization have been studied. The dye solution decolourization has been measured using colorimetric methods and upon the measurements results the energy efficiency EEf (g/kWh) of the dye removal have been calculated.

The experimental results have shown that the electrochemical processes occurring in plasma and, consequently, the dye removal rate are significantly depending on the electrodes' shape. The best results have been obtained for using a reactor having one sharp active electrode and one horizontal plan section for the outlet electrode, while the distance between them was 4 mm.

Key words: dye, electrodes shape, non-thermal plasma.

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1. Introduction

Non-thermal plasma technologies could be considered as an alternative to other consecrated methods for water depollution such as sonolysis, photocatalysis, electrochemical, ozonolysis, adsorption, hydrolysis and biological methods (Siddique et al., 2011). The main advantages of the NTP technologies, in comparison to the other classical physical/chemical advance oxidative technologies, are a higher active power, high specific energy efficiency of the discharge and the environmental required operating conditions – atmospheric pressure and room temperature. The NTP technology does not require additional equipment for temperature and pressure control, which leads to simple technical solutions.

Many of the industrial applications such as textile dyeing, paper printing, leather shoes polish, plastics, food colouring etc., are using different

organic dyes that are later carried out in wastewater (Latif et al., 2010; McKay, 1979). Even though there have been serious concerns over the last few years to reduce the impact of pollutants on the environment and it is known that there are more than 100,000 types of commercial dyes available and over 7×10^5 tons of dye related compounds are produced annually (Jakumar and Ramamurthi, 2009). Very low concentrations of these dyes present in the effluent can be highly visible and undesirable, because of their chemical properties. They are resistant to light, water and oxidizing agents, and for these reasons, it is difficult to remove them from the aquatic systems (Kim et al, 2003; Kuo, 1992; Siddique et al., 2011; Zonoozi et al., 2018). Without adequate treatment, a soluble organic dye may be stable and may remain in the environment for years, in normal conditions (pH 7 and 25 °C) (Weber and Stickney, 1993). Due to the dyes chemical stability and low biodegradability, a

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wide range of methods has been developed for its removal from waters and wastewaters in order to decrease their impact on the environment (Siddique et al., 2011; Sizykh et al., 2018).

In various studies, RB-19 was selected as a model compound due to its low fixation efficiency (75-80%) on cellulose (does not fix on the cellulose fibre). The chemical structure of RB-19 is presented in Fig. 1 (Awad and Galwa, 2005; Weber et al., 1993).

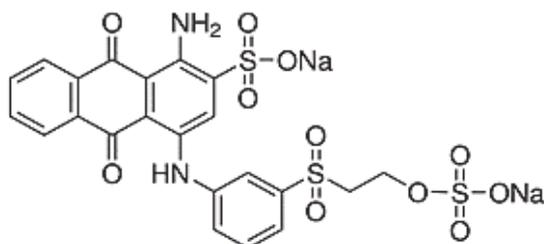


Fig. 1. Chemical structure of Reactive Blue 19 (C₂₂H₁₆N₂Na₂O₁₁S₃)

For these reasons, the soluble organic dye Reactive Blue 19 has been considered adequate to be used as a comparative study element, in order to emphasize the effects of NTP reactors electrodes shape on soluble organic compounds treatment. Ar was used as carrier gas instead of air, in order to avoid the nitrogen oxides influence on the treatment process Eqs. (3-7),

Radicals and highly energetic reactive molecular species are generated by non-thermal plasma electrical discharge in the gas phase, which interacts with water, and dye molecules, leading to dye degradation. These species depend on the nature of gas and the liquid treated and on the electrical discharge parameters (voltage and current waveforms, power).

In non-thermal plasma treated water are generated highly oxidative radicals ($\bullet\text{OH}$ and others) and molecular species such as H_2O_2 , H_2 , O_2 . In addition, when the gas of non-thermal plasma is air, nitrates are formed in water (Locke and Shih, 2011; Bruggeman and Leyes, 2009; Brisset et al., 2008; Fridman, 2008; Locke et al., 2006; Malik et al., 2001; Sunka, 2001). Also, H_2O_2 may form in plasma through OH radical recombination by Eq. (1):



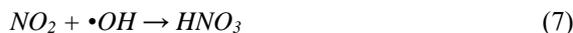
Reactions between hydrogen peroxide and OH radicals can also occur, leading to H_2O_2 destruction and water formation through reaction (Burlica et al., 2013) (Eq. 2):



If the carrier gas is air or nitrogen the following chemical reactions can occur (Eqs. 3-6):

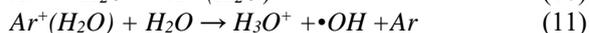


The nitrogen oxides affect the pH and the conductivity of the water through the formation of ions and acids described by reactions (Eq. 7) (Abdelmalek et al., 2006; Burlica et al., 2011):



There are studies based on using needle-to-plate non-thermal plasma (NTP) reactor which revealed a 59.9% decolourization rate of the reactive blue dye solution, treated in the Ar atmosphere, at room temperature and atmospheric pressure. (Sun et al., 2016).

For Ar as the carrier gas, along of hydroxyl radical ($\bullet\text{OH}$), active argons ions and electrons are generally the main reactive species responsible for the dye degradation (Bian et al., 2007; Clyne et al., 1969; Siddique et al., 2011), (Eqs. 8-11).



In addition, it is known that the removal efficiency increases if the solution to be treated is sprayed into the non-thermal plasma discharge zone formed between the electrodes. In the NTP reactors case, the water spray improves the efficiency of reactive species production and assure a better interaction between plasma and liquid phases (Burlica and Locke, 2008; Burlica et al., 2010; Locke and Thagard, 2009; Porter et al., 2009),

Non-thermal plasma (NTP) is generated by an electrical discharge between two electrodes, in a gas flow, in our case Ar, in order to maintain the low temperature of the plasma. The efficiency of the water treatment in NTP reactors was enhanced by spraying the solution into the plasma zone through a two-way nozzle.

The organic dye removal from the solution sprayed in the plasma has been measured using colorimetric methods, UV-VIS spectrometry. In order to optimize (from electrical and physical-chemical parameters point of view) the NTP treatment of the organic compounds dissolved in water, the energy efficiency (g/kWh) of the dye destruction has been calculated for different shapes of the electrodes.

2. Experimental design and set-up

In order to produce the non-thermal plasma discharge, a two electrodes configuration reactor has been used and the discharge was produced in a cylindrical Teflon chamber as presented in Fig. 2. The high AC voltage is applied between the electrodes E1 and E2 (a TRA-AC high voltage transformer (9 kV / 25 mA / 50 Hz with a fast dropping output electrical characteristic have been used), in series with the resistor, R (Fig. 2).

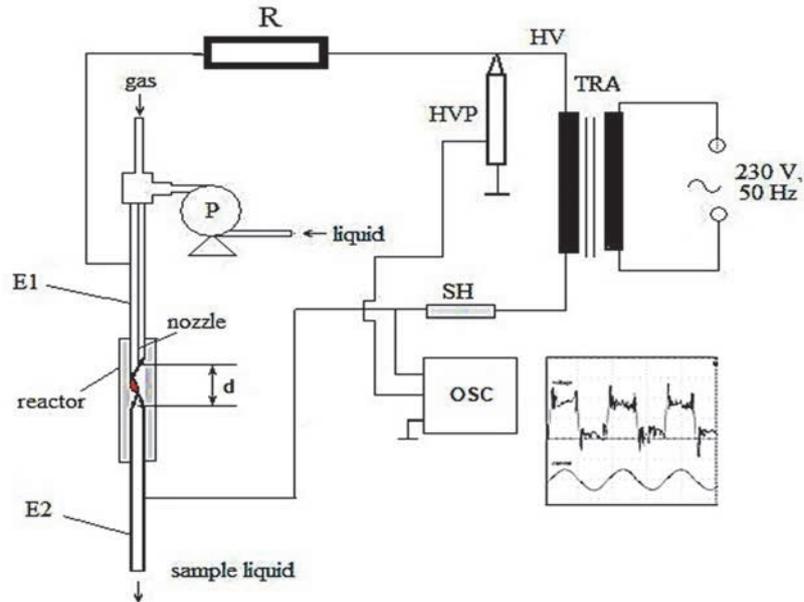


Fig. 2. Experimental set-up

In order to emphasize the influence of reactor electrodes, shape and position on dye removal Argon have been used as carrier gas. The reactive Blue 19 dye solution considered for experiments had an initial concentration of 50 mg/L and it was prepared using demineralized water. The solution to be treated was sprayed, through a two-port nozzle (Fig. 2), one for gas and another for solution, directly into the NTP reactor equipped with two cylindrical electrodes (E1, E2) placed in a narrow chamber, meant to confine the plasma and force the water droplets to get into contact with the reactive species formed in the gas phase. The liquid to be treated was injected into the reactor using a high-pressure pump, P, with a constant flow rate.

The NTP reactors were supplied by an AC high-voltage transformer (Tr, F.A.R.T 9 kV/25 mA). The electrical parameters as voltage and current have been measured by an oscilloscope OSC – LeCroy 454 where the SH is the shunt for current measurement and HVP is the high voltage probe (Tektronix) for voltage measurement. In order to avoid the influence, the electromagnetics perturbation induced by electrical discharge special measures of screening of the measurement equipment had been taken (Beniugă and Beniugă, 2017, Kováč et al., 2012).

The current measured was in the range of 4-5 mA while the power of the alternative discharge was about 4 W. The power was maintained quasi-constant for all the considered distances between the electrodes and reactors types being limited by electrical parameters of the high voltage power supply. The typical voltage and current waveforms are presented in Fig. 2. The low power of the electrical discharge used in the process to produce the non-thermal plasma does not thermally vaporize the water droplets in the reactor, and enhance the energy efficiency yields for organic dye removal (Burlica et al., 2010).

Experiments presented used Ar as the carrier

gas, in order to avoid the radicals quenching reactions by other molecular species such as nitrates and nitrites that are generated in air-plasma. The gas flow rate was $Q_g = 1.5$ L/min, constant, and the solution flow rates Q_s have been varied between 5-10 mL/min. In order to be sure that all the reactive species are generated due to the interaction between plasma and solution and there are no residual active species into the carrier gas, the gas has not been recirculated into the system, all the gas being released in atmosphere. If the scale-up of the system is required, for industrial application, the gas can be recirculated.

The initial concentration of the solution was 50 mg/L of Reactive Blue 19 and the absorbance measured (A) was $A = 0.415$ at 590 nm wavelength. The absorbance of the blue dye has been measured with Shimadzu UV-visible spectrophotometer Model UV-Mini, 1240 at the absorption peak, ($\lambda = 590$ nm), For 590 nm wavelength, the absorbance of the treated liquid has a linear variation with the reactive blue concentration in the solution.

The dye removal *energy efficiency* (EEf) was calculated in g/kWh for each experimental case, without following a specific degradation rate, considering the concentration variation of Blue 19 and the energy of the electrical discharge (Eq. 12):

$$EEf = \Delta[\text{Blue}19] \cdot Q_s \cdot t / W \text{ [g/kWh]} \quad (12)$$

where $\Delta[\text{Blue}19]$ is the concentration variation of Blue 19 dye (in mg/ L), calculated with the relation Eq. 13, Q_s is the solution flow rate in L/h, $W = P \cdot t$ is the discharge energy in kWh (P is the electrical power in kW) and t is the considered time (h).

The decreasing of the absorbance (intensity of the colour of the Reactive Blue 19 solution) after one pass through the NTP reactor has been measured. The results emphasized the significance of the NTP

experimental reactor configuration (electrodes shape and the distances between them) on dye degradation.

The variation of dye removal rate was evaluated with the relation (Eq. 13):

$$\Delta[\text{Blue19}] = C_i - C_e \text{ [mg/L]} \quad (13)$$

where: C_i is the initial concentration of dye solution sample to be treated (50 mg/L) and C_e is the solution dye concentration after NTP treatment. The evolution of the dye concentration was determinate based on the absorbance variation of the treated solution by NTP plasma.

Accordingly, the three repeated measurements of absorbance for each water flow rate have been performed, for the treated solution sample, a 10% error margin has been calculated.

The reactor is composed of two cylindrical electrodes placed in a teflon cylinder having also the role to isolate the plasma produced from the outer environment in order to enhance the solution treatment. The inlet electrode has an inner diameter of 0.10 mm, the outlet electrode has an inner diameter of 1 mm, both placed in a 2 mm teflon tube diameter. The distance between the electrodes allows to modify the length of the plasma channel and consequently, to change the electrical parameters of the discharge and as well the chemical processes between the solution and plasma. The evaluation of dye removal efficiency was determined for three distances between the electrodes, 2, 4 and 6 mm. The distance (d) has been measured on the reactor vertical simetryc axes (as shown in Fig. 2), between the center of the plane sections of the electrodes. In Table 1 a summary of the four different types of reactors that have been studied is presented.

The experimental NTP reactors configurations used are the following: reactor type “R1” has both electrodes identically, with a horizontal plan sections and are placed face to face; reactor type “R2” has the inlet electrode with a sharp shape cut at 45 degrees and the outlet electrode with horizontal plan section; reactor

“R3” has both identically electrodes with 45 degrees sharp shape cut. The “R3” model has two types of configurations: R3_1 has a parallel position of the transversal electrodes section, respectively perpendicullar position in R3_2 configuration (Table 1). For evaluation of each type of reactor performances, the dye removal rate was determined for all the three considered distances between electrodes mentioned above, for different solution flows, between 6.5 and 9.5 mL/min, while the gas flow rate, $Q_g = 1.5$ L/min, was kept constant.

The 50 mg/L Reactive Blue 19 solution has been treated in the NTP reactor and the decreasing of colour intensity has been measured. For the same sample treated solution, three repeated measurements of absorbance have been made for each water flow rate and the average of the values measured was reported.

3. Results and discussion

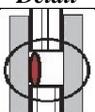
In order to emphasize the influence of distance between electrodes, the energy efficiency (g/kWh) of the dye removal from the solution, for different electrical (voltage, current, electrical power, P) and physical parameters (solution flow rate, Q_s , electrodes shape, distances between electrodes), has been calculated.

3.1. Influence of distance between electrodes on dye removal

For all configurations of NTP reactors, the decolourization of the treated dye solution was determined. Measurements of the variation of dye concentration of the treated solution for 2, 4 and 6 mm distance between electrodes for each reactor configuration have been made.

The R1 configuration of NTP reactor used parallel horizontal sections of electrodes, Table 1. In Fig. 3, the variation of dye removal with solution flow rate injected into the reactor, for different distances between electrodes is presented.

Table 1. Electrodes shapes

Reactors	Detail	Description
R1		The electrodes are identical, with horizontal plan sections, disposed of in a parallel position of transversal sections;
R2		The inlet electrode has a 45 degrees plan section and the outlet electrode has a horizontal plan section;
R3_1		Both electrodes are identical, with a 45 degrees plan section, and there are disposed of in a parallel position of the transversal sections;
R3_2		Both electrodes are identical, with a 45 degrees plan section, and there are disposed of in an orthogonal position of the transversal sections.

For the R1 configuration (parallel horizontal sections of electrodes) of NTP reactor, the most significant dye removing has been obtained for 6.5 mL/min solution flow rate for all considered distances between electrodes. For liquid flow rates near to 10 mL/min, the variation of dye concentrations converges to the same value for all distances between electrodes.

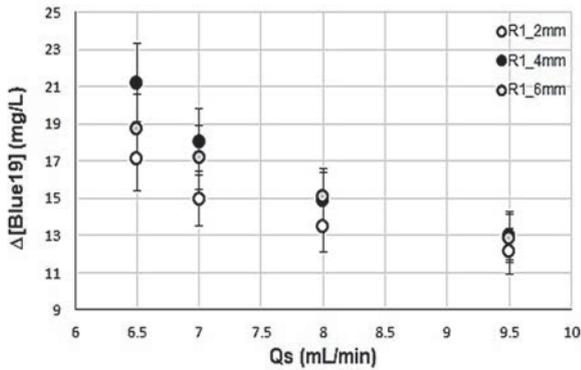


Fig. 3. Variation of dye removal with flow rate for different distances between electrodes of R1 NTP reactor

The results obtained for R1 NTP configuration, for 4 mm distance between electrodes and 6.5 mL/min solution flow rate, show more than 21 mg/L dye degradation, meaning 42% dye reduction. At the same water flow rate (6.5 mL/min) for 2 mm distance between electrodes, the removed dye concentration was about 17 mg/L (34 % dye reduction).

The results obtained for 6 mm distance between electrodes case, for R1 NTP reactor, are in between the results obtained for the 4 mm respectively 2 mm electrode distances for lower flow rates (6.5 and 7 ml/min). As observed, the influence of the distance between the electrodes becomes less important with the increasing of the solution flow rate ($Q_s > 7$ mL/min), around 13 mg/L (26%).

Similar, for the R2 configuration of NTP reactor (the inlet electrode with a sharp shape at 45 degrees and the outlet electrode with horizontal plan section), the variation of dye removal with solution flow rates variation for different distances between electrodes has also the same behaviour, as shown in Fig. 4. In the case of R2 NTP configuration, for 4 mm distance between electrodes and 6.5 mL/min solution flow rate, the dye degradation was about 25 mg/L RB-19 dye (50% dye reduction), meantime for 2 mm and 6 mm distance between electrodes dye degradation is about 20 mg/L (40%).

Starting with 7 mL/min solution flow rate, for the above-mentioned shape of electrodes, the distance between electrodes has an insignificant influence on dye removal. Similar to the previous case of the R1 reactor, for 9.5 mL/min solution flow rate, the 19 Blue dye removals has the same value, about 13 mg/L (26%), for all considered distances between electrodes. In Fig. 5 and Fig. 6 is shown the variation of Reactive Blue 19 dye concentration, with solution flow rates for different distance between electrodes,

for the R3_1 respectively R3_2 electrode configurations of NTP reactors.

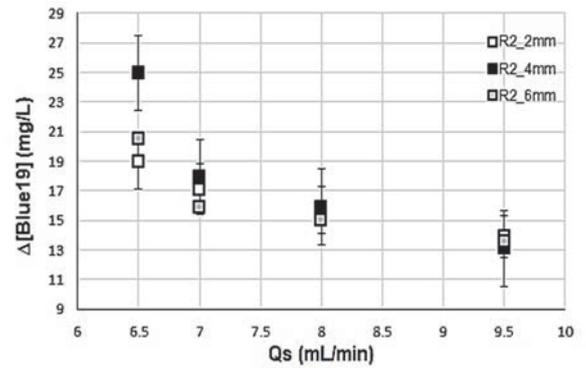


Fig. 4. Variation of dye removal with flow rate for different distances between electrodes of R2 NTP reactor

R3 electrode configuration has 45 degrees sharp shape for both electrodes. For R3_1 NTP experimental reactor with 6 mm distance between electrodes, the Reactive Blue 19 treated solution reach a rate of dye removal between 17 mg/L (34%) at 6.5 mL/min and 11.5 mg/L (23%) at 9.5 mL/min flow rate (Fig. 5). For R3_1 NTP reactor with 2 and 4 mm distance between electrodes the dye concentration variation is between 16 mg/L (32%) and 11 mg/L (22%) for solution flow rates from 6.5 mL/min to 9.5 mL/min.

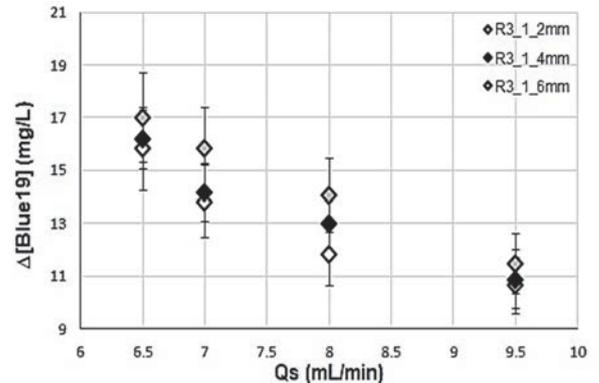


Fig. 5. Variation of dye removal with flow rate for different distances between electrodes of the R3_1 reactor

For 50 mg/L initial concentration of Reactive Blue 19 treated solution in the R3_1 reactor, small differences in dye removing have been found, for all distance between electrodes. The results for the sample dye solution ($[RB-19] = 50$ mg/L) treated in the R3_2 NTP reactor, are presented in Fig. 6.

For R3_2 NTP with 6 mm distance between electrodes (perpendicular position electrode sharp sections), the variation of dye removal values was found to be between 17 mg/L (34%) at 6.5 mL/min and 13 mg/L (26%) at 9.5 mL/min flow rate. For 2 or 4 mm distance between electrodes, the measured variation rate of dye removal is between 15 mg/L (30%) and 10 mg/L (20%) for the flow rate variation between 6.5 mL/min to 9.5 mL/min.

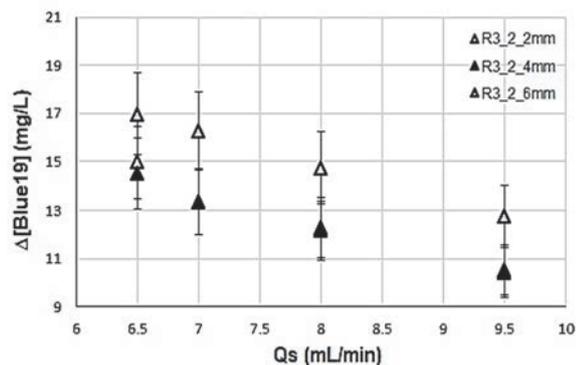


Fig. 6. Variation of dye removal with flow rate for different distances between electrodes of R3_2 NTP reactor

3.2. Influence of electrodes shapes on dye removal

Fig. 7 shows the variation of dye removal, comparative diagrams, for the NTP experimental reactors for d=2 mm (in Fig. 7a), d=4 mm (Fig. 7b) and d=6 mm (Fig. 7c) distance between electrodes.

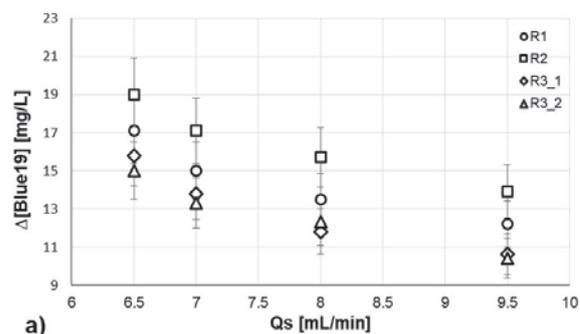
The R2 reactor configuration, sharp inlet electrode (45 degrees) and the horizontal section for the outlet electrode provided the highest dye removal rate of the treated solution, for d= 2 mm distance between electrodes. Contrary, for the R3_2 experimental configurations, sharp shape for both electrodes (45 degrees), the dye removing was the lowest (4 mg/L lower). Dye concentration variation had the same pattern when the distance between the electrodes was 4 mm, as is shown in Fig. 7b. In this case, the highest value for dye removal in R2 NTP experimental reactor was 25 mg/L at 6.5 mL/min solution flow rate. R3_2 configurations, with 4 mm distance between electrodes, the maximum dye removal was 15 mg/L, for 2 mm distance between electrodes.

Even when the electrodes are fixed at d=6 mm distance, the experimental results for dye removing had the same evolutions like in the other two reactor configurations (R1 and R3_1), as it can be observed in Fig. 7c. In these cases, the dye concentration removed vary from 21 mg/L to 11 mg/L, for solution flow rates between 6.5 mL/min to 9.5 mL/L. For 6 mm distance between electrodes in the R2 NTP experimental reactor, for a solution flow rate of 6.5 mL/min, highest dye removal was about 21 mg/L. Meantime the R3 configuration (sharp electrodes parallel or orthogonal sections position) dye removal was the lowest, about 17 mg/L after treatment. For solution flow rates highest than 7 mL/min, the final dye solution concentrations, after treatment, converge to the same value.

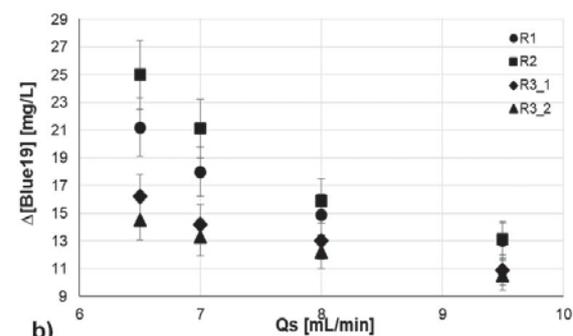
3.3. Energy efficiency on dye removal for different electrode shapes

The energy efficiency of dye removal had been calculated with relation (Eq. 12) for each reactor configurations. For each NTP experimental reactor configuration, the shape of electrodes and distance

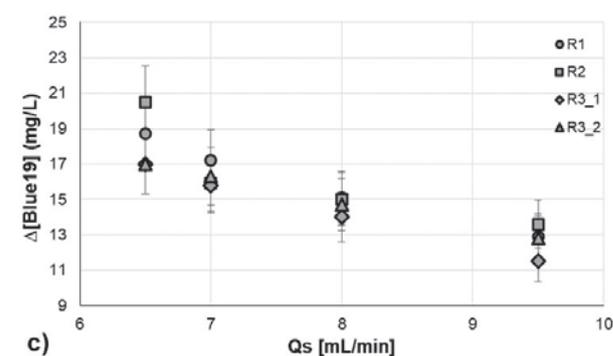
between electrodes the maximum value of energy efficiency has been determined.



a)



b)



c)

Fig. 7. Variation of blue dye removal with flow rate for different electrode shapes of reactors at the same distance between electrodes: a) 2 mm, b) 4 mm, c) 6 mm

The influence of electrodes shape configuration on the energy efficiency was represented in the graphs below for each electrodes shape and distances d between them: Fig. 8a for d= 2 mm, Fig. 8b for d=4 mm and Fig. 8c for d=6 mm. The highest Eef values, obtained in different conditions for each reactors and solution flow rates are presented. For a 2 mm distance between the electrodes, the highest Eef was obtained for a 9.5 mL/min solution flowrate for all the reactors. In the case of 4 and 6 mm distance between the electrodes, the efficiency is increased when a lower solution flowrate is considered (6.5 mL/min).

The reactor R3_3 presents an exception: for all the three distances considered for experiments, the highest Eef, although generally lower than the one corresponding to R1 and R2, was obtained for the highest solution flowrate (9.5 mL/min).

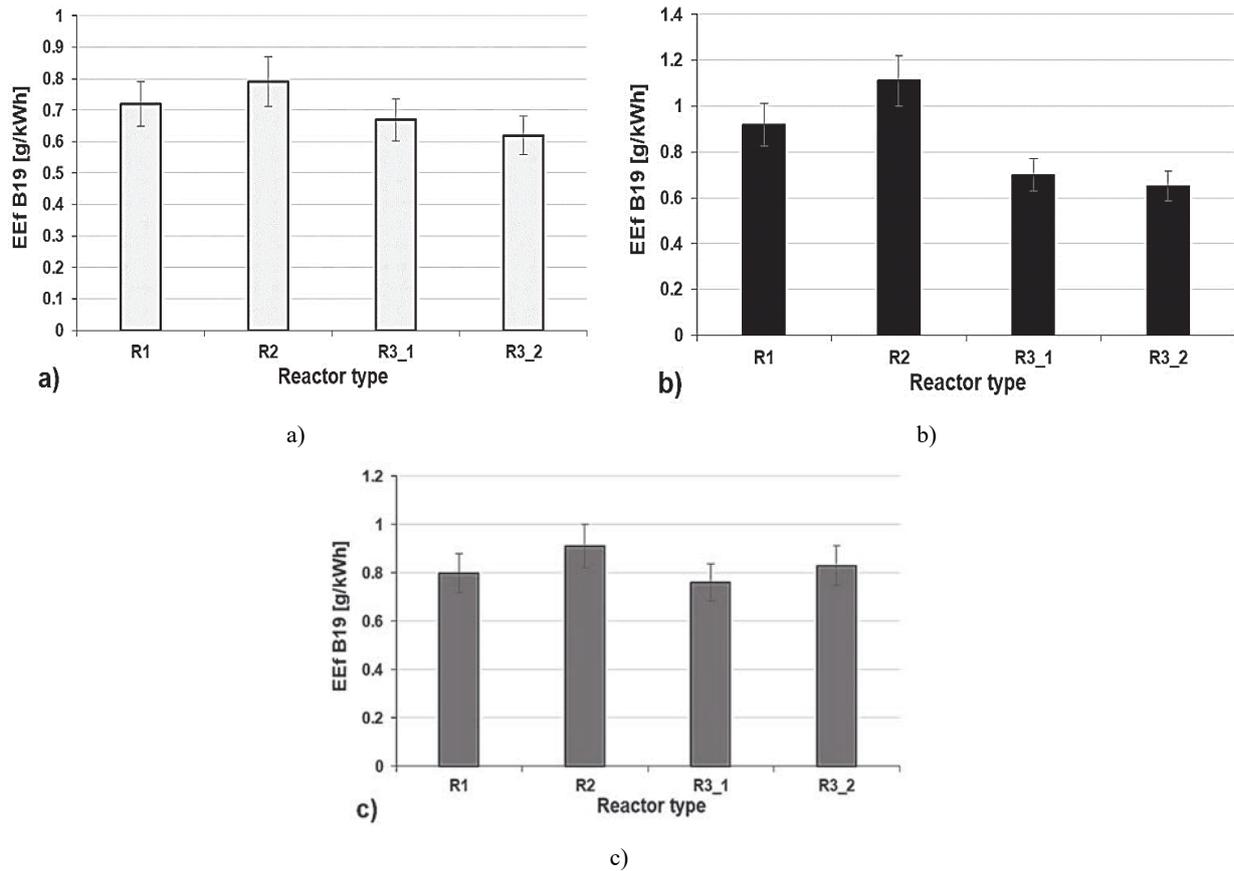


Fig. 8. Dye removal energy efficiency for different electrode shapes NTP reactor at the same distance between electrodes: a) 2 mm, b) 4 mm, and c) 6 mm

Fig. 8a shows a 0.8 g/kWh efficiency for the R2 NTP experimental reactor set up (inlet electrode with a sharp shape, 45 degrees, and a horizontal section for the outlet electrode) for $d=2$ mm distance between electrodes. The lowest energy efficiency, around 0.6 g/kWh, was calculated for the R3_2 NTP experimental reactor set up (sharp shape both electrodes in an orthogonal section position). For $d=4$ mm electrode distance (Fig. 8b) of NTP discharge, the calculated energy efficiency was significantly higher for R2 NTP experimental reactor, about 1.1 g/kWh, in comparison with 0.65 g/kWh for the R3_2 reactor configuration.

For the NTP experimental reactors configurations with $d=6$ mm distance between electrodes the range of energy efficiency values calculated is narrower (from 0.75 g/kWh to 0.9/kWh). In this case, the maximum *energy efficiency* value was obtained for R2 reactor configuration while the lowest *EEf* value was obtained for R3_2 reactor configuration as it is shown in Fig. 8c.

The electrode shapes, for $d=2$ mm distance between them, significantly affect the energy efficiency in dye removing especially for lower dye solution flow rates. The highest $EEf = 1,1$ g/kWh, has been determined in the case of R2 NTP reactor for a distance $d=4$ mm between the electrodes, while the lowest *EEf* values, 0.75 g/kWh, corresponds to R3_2

NTP reactor configuration for $d=2$ mm distance between electrodes.

Based on the followed output parameter the reactor configuration could be adapted. If a higher removal rate or higher energy efficiency are considered as output parameters, then the distance between electrodes should be 4 mm while the solution flowrate 6.5 mL/min. If higher solution volume is required to be treated with respect to energy efficiency, then 2 mm distance between electrodes and 9.5 mL/min solution flowrate should be considered.

Along with electrical parameters such as power and the current/voltage of the electrical discharge the physical parameters as solution flow rate, the shape of electrodes and the distance between them play an important role in NTP solutions treatment efficiency.

Changing the configurations of NTP reactors geometry using different forms for electrodes and adjusting the distances from them allows adapting different solutions treatments to various practical applications.

4. Conclusions

The Blue 19 dye removal in NTP mini-reactors significantly depends on NTP mini-reactor configuration, the shape of electrodes and the distance between them.

The dye removing decrease with the treated solution flow rates for all NTP experimental reactors configurations. The dye removal rate is higher for low solution flowrates due mainly to the higher residence time in the plasma region for all the considered reactors but still this does emphasize an optimum for the energy efficiency of dye degradation, in all cases of reactors configurations and distances between the electrodes.

For 2 mm distance between electrodes, a higher solution flowrate of 9.5 mL/min assures better results for the energy efficiency, while for distances of 4 and 6 mm a lower flowrate of 6.5 mL/min offered better results.

The highest variation of dye degradation was obtained for all the distances between the electrodes and for the considered flowrates for the R2 type NTP reactor having a 45 degrees plan section inlet electrode and a horizontal plan section outlet electrode. It is followed by the R1 type reactor that has to identical electrodes, with horizontal plan sections, disposed of in a parallel position of transversal sections.

The highest energy efficiency was also obtained when the R2 type reactor was used for all the considered distances between the electrodes. The highest value was obtained for a distance of 4 mm between the electrodes and a 6.5 mL/min flowrate.

The lowest performances were founded for the both configuration of the R3 type NTP reactor, which was identical electrodes, with a 45 degrees plan section.

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