Methylene Blue degradation using plasma: A comparative study between the microwave plasma jet over water surface and the nanosecond pulsed discharge in gas bubbles in water

Aloui Nadir and Belgacem Ibtissem

Abstract—This paper investigates the Methylene Blue degradation using two common reactors: the nanosecond pulsed discharge in oxygen bubbles and the Argon Microwave plasma jet above the treated solution. The aim of this study is to compare the evolution of the reactors performance when the electrical conductivity increases. In deionized water, the Methylene Blue degradation rate and the energy efficiency (0.19 min^{-1} and 99 mg/kWh, respectively) in the Nanosecond pulsed discharge configuration were significantly higher than the Argon microwave plasma jet (0.08 min^{-1} and 10 mg/kWh). However, when the solution electrical conductivity is increased from 5 to 200 µS/cm, the energy efficiency of the nanosecond pulsed discharge drops significantly by $\sim$82%, whereas the performance of the microwave plasma jet reactors decreases by $\sim$37% only for a greater variation of the electrical conductivity of the solution (from 5 to 10 000 µS/cm). Under these conditions the efficiency of both reactors becomes comparable, suggesting the necessity to consider various parameters to compare the reactors efficiency.

***Keywords*—Nanosecond pulsed discharge, Microwave plasma jet discharge, Methylene Blue, electrical conductivity, energy efficiency.**

NOMENCLATURE

NPD	Nanosecond Pulsed Discharge .
Ar MWPJ	Argon Microwave Plasma Jet .
MB	Methylene Blue.

I. INTRODUCTION

Water pollution is the result of introducing harmful substances into water bodies, causing degradation in their quality. This issue is becoming even more serious due to the increase in human activities such as urbanization [1] and industrialization [2], which introduce high levels of various pollutants into water resources, often referred to as "emergent pollutants" [3–6]. The emergent contaminants refer to a broad of compounds present in wastewater at low concentrations (ng/L-µg/L), that not have been previously monitored or regulated but are currently recognized as an important contributors to water pollution. These pollutants could classify into several groups such as: pharmaceuticals, personal care products, pesticides, micro plastics, industrial chemicals and hormone; also, they can be natural or synthetic substances [7, 8]. Despite the lack of knowledge about them, the emergent contaminants are identified as a potential threatens for both environment and human health [3, 9]. Therefore, the detection and the elimination of emergent contaminants is a crucial task nowadays. Recently, the plasma based technology has been proposed as an alternative to eliminate emergent contaminants from wastewater. In fact, when the plasma is coupled with water, large broad of reactive species such as Ozone (O_3) [10], hydrogen peroxide (H_2O_2) [10, 11] hydroxyl radical (OH) [12] oxygen atoms (O) [13], among others could be produced making it a promising technology for wastewater treatment. Effectively, the plasma showed a high potential to eliminate emergent pollutants such as: dyes [14, 15], pharmaceuticals products [16] and even pesticides [17]. However, plasma-based water treatment remains a relatively new technology that requires further development before it can be effectively used at larger scale. At this stage, one of the most critical tasks that should be considered to achieve this purpose is to evaluate the plasma technology for water treatment in order to establish a road map for further research. In this context, Malik, 2010 [18] made a comparative study between 27 types of plasma reactors to assess the most efficient configuration for water treatment. In the mentioned work, the energy yield at 50% was used as parameter to assess the plasma reactors performance. The results achieved by this study showed that the reactors efficiency depends on several parameters like the gas composition, the liquid distribution, i.e. deep layer, thin film or water sprayed and finally the signal of the power supply (pulse, DC or AC). Overall, the pulsed discharges in the gas phase and in contact with thin layers or fine droplets of solution are the most efficient reactors. However, despite the very important conclusions highlighted by Malik, 2010 [18], the comparison between plasma reactors should be extended to take into consideration other factors. In the present study, a different approach is adopted to compare the performance of two widely employed plasma-based water treatment reactors: the nanosec-

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ond pulsed discharge (NPD) reactor operating in gas bubbles within water and the argon microwave plasma jet (Ar MWPJ) in direct contact with the water surface. This comparison focuses on evaluating how the performance of these reactors evolves under varying operating conditions, particularly the electrical conductivity of the treated solution. Electrical conductivity was chosen as the primary variable because, in practical scenarios, the electrical conductivity of wastewater is significantly higher than that of deionized water. By systematically altering this parameter, the study aims to bridge the gap between controlled laboratory conditions and real-world applications, providing insights into the adaptability and efficiency of these reactors under conditions that are similar to actual wastewater compositions. This work highlights the critical role of solution conductivity in Plasma-based water purification, offering a deeper understanding of the challenges facing this technology.

II. METHODS AND MATERIALS

In this study, we investigate the Methylene Blue (MB) degradation at initial concentration of 5 and 10 mg/L using NPD in O_2 bubbles and the Ar MWPJ above water surface, respectively.

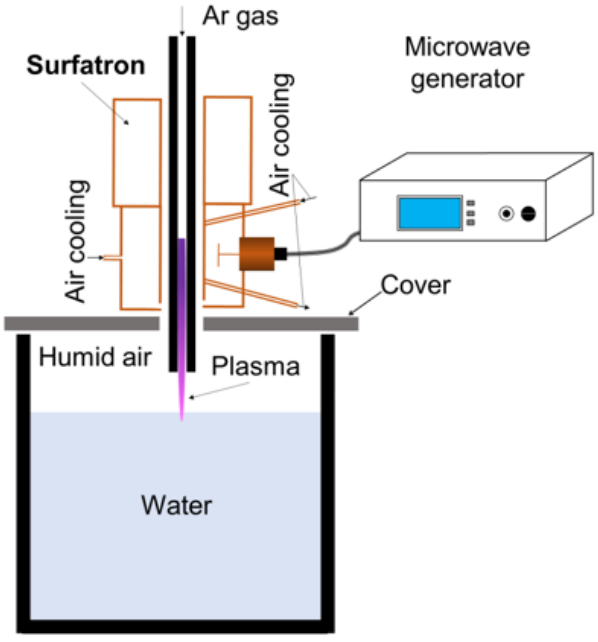

Fig. 1: Schematic of the the Ar MPWJ setup used to degrade MB solution.

The Fig.1 shows the experimental setup of the Ar MWPJ. The energy provided by the microwave generator (SAIREM GMS200W) operating at 2.45 GHz is coupled to the Argon gas (purity >99.995%) using a Surfatron device (SAIREM S-WAVE 6 A). The Argon gas is supplied at a flow rate of 0.5 L/min through a quartz tube with inner and outer diameters of 2 mm and 6 mm, respectively. The experiments are conducted using a closed cell covered with an aluminum circular plate. The quartz tube is inserted into the closed cell through a 7 mm diameter hole in the aluminum plate to generate the MWPJ in contact with 100 mL of MB solution. During the experiments, the compressed air is used to cool the Surfatron device and the quartz tube. The microwave power is maintained at 100 W throughout the process.

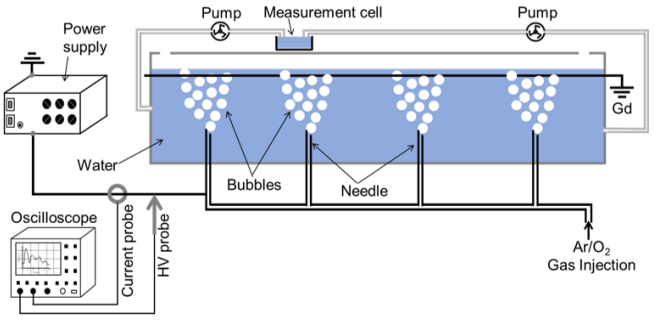

Fig. 2: Schematic of the the NPD in O_2 bubbles setup used to degrade MB solution.

Regarding the NPD in O_2 bubbles we used the setup shown in (Fig.2). The discharge is produced using a nanosecond negative polarity pulsed power supply (NSP 120-20-N-500-TG-H, Eagle Harbor Technologies) at an amplitude of -20 kV, a pulse width of 500 ns and a frequency of 1kHz. The high voltage was connected to four hollow-needles-like (inner and outer diameter of 0.3 and 0.5 mm, respectively), whereas the grounded electrode was a stainless-steelwire (1 mm diameter) placed at 8 mm above the needles. A mass flow controller was used to inject O_2 (purity >99.995%) into MB solution through the hollow needles at flow rate of 4 L/min (1 L/min per needle). The volume of the MB solution was 300 mL. 200 mL was filled in the cylindrical cell where the electrodes are mounted, whereas the remaining 100 mL is filled in cell to perform the absorbance measurements. Two pumps (Ismatec™ MS-2/6 Reglo Analog Pump) were utilized at flow rate of 50 mL/min to ensure the MB solution recirculation.

In both configurations, the MB degradation is monitored using an UV-Vis absorption spectrophotometer (Cary 5000 UV–Vis-NIR, Agilent). The decoloration efficiency is assessed based on the absorbance intensity at 664 nm, and the MB concentration was deduced based on a calibration curve. The KCl was used to alter the solution electrical conductivity and the H_2O_2 production is measured is distilled water (without MB) using drop count titration method based on Sodium Thiosulfate titrant.

III. RESULTS AND DISCUSSION

A. MB degradation

The (Fig.3a) shows the evolution of the normalized absorbance (at 664 nm) as function of plasma processing time for both configurations the Ar MWPJ and the NPD in O_2 bubbles. The results show that the MB degradation efficiency is considerably higher in the NPD than the Ar MWPJ; the $t_{50\%}$ which represents the time required to eliminate 50% of MB molecules is 15 min in the Ar MWPJ configuration. Whereas in the NPD reactor the $t_{50\%}$ is only 5min.

To estimate the MB degradation rate, a kinetic study is carried out. Since the MB concentration decreases exponentially during processing (Fig.3a), we assume that its degradation is described by first order kinetics (1).

$$-ln(C_t/C_O) = kt \quad (1)$$

With C_t (mg/L) is MB concentration at time t, C_0 (mg/L) is the MB initial concentration and k (min^{-1}) is the first order rate

constant.

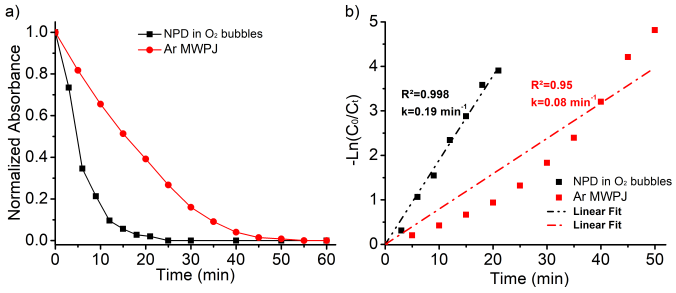

Fig. 3: (a) Temporal evolution of the normalized absorbance (at 664 nm) of MB solution in the Ar MWPJ and NPD in O_2 bubbles reactors. (b) First order kinetics of MB degradation solution in the Ar MWPJ and NPD in O_2 bubbles reactors.

The results of the kinetic study (Fig.3b) show that the correlation coefficient R^2 is 0.95 and 0.998 in the Ar MWPJ and the nanosecond pulsed discharge configurations, respectively. This confirms our assumption indicating that MB degradation is represented by first-order kinetics.

The constant degradation rate in Ar MWPJ is 0.08 min^{-1}, whereas, in the NPD in O_2 bubbles, it is 0.19 min^{-1}. These results demonstrate that the MB degradation in this latter configuration is ∼2.5 time faster than the Ar MWPJ. To understand the reason behind the superiority of the NPD in MB degradation, the H_2O_2 production in water (without MB) is measured during plasma processing. The presence of H_2O_2 in water indicates the production of hydroxyl (OH) radicals which are known to be responsible for the degradation of MB [19, 20].

The (Fig.4) shows the H_2O_2 production as function of plasma processing in both configurations. In the NPD in O_2 bubbles, the H_2O_2 production increases linearly after 30 min of processing to reach ∼54 mg/L with rate of ∼1.9 mg/Lmin. On the other hand, in the Ar MWPJ, the H_2O_2 production reaches 20 mg/L after 40 min of processing with rate of ∼0.5 mg/Lmin. After this period no further increase in H_2O_2 production is registered.

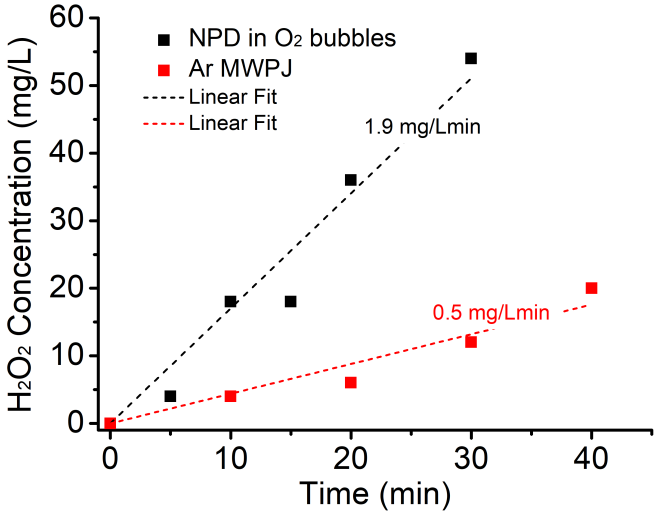

Fig. 4: H_2O_2 production using the NPD in O_2 bubbles and the Ar MWPJ.

The high production rate of H_2O_2 in the NPD reactor indicate that this configuration produces more OH radicals, explaining thus, the high MB degradation rate observed in this reactor. In fact, when the NPD in O_2 bubbles is used, the oxygen atoms could be produced (2) to generate supplementary OH radicals (3) and ozone (O_3) (4) [21]. This latter (O_3) play an important role in MB degradation [22] by a direct oxidization or by generating more OH radicals when reacting with H_2O_2 (5) [23]. Overall, the production of O atoms and O_3 in the NPD configuration can enhance the OH production which is beneficial for MB degradation. Besides the ozone production, the generation of the discharge in gas bubbles enhances the mass transfer of reactive species to the liquid bulk, making this configuration more efficient.

$$e + O_2 \rightarrow 2O + e \tag{2}$$

$$O + H_2O \rightarrow 2OH \tag{3}$$

$$O + O_2 \rightarrow O_3 \tag{4}$$

$$O_3 + H_2O_2 \rightarrow OH + HO_2 + O_2 \tag{5}$$

B. Energy efficiency

The energy yield at 50% ($Y_{50\%}$) is used in this study to assess the energy efficiency of the studied configurations (NPD in O_2 bubbles and the Ar MWPJ). This parameter represents the amount of eliminated contaminants per unit of delivered energy at 50% degradation of MB, and it is expressed by equation (6) [18]:

$$Y_{50\%} = \frac{V \times C_0 \times \frac{C_0 - C}{C_0}}{P \times t_{50\%}} \tag{6}$$

With V(L) is the volume of MB solution, C_0(mg/L) is the initial concentration, C(mg/L) is the MB concentration at 50% of conversion, P(kW) is the delivered power, and $t_{50\%}$(h) is the time to reach 50% of MB degradation.

The delivered power by the microwave generator was set at 100 W, whereas in the NPD in $O2$ bubbles, we measured the dissipated power using a monitor that was added to the plug wall where the pulse generator is connected. The consumed power in that case was 181 W.

The table I shows once again the superiority of the NPD in O_2 bubbles compared to Ar MWPJ; the $Y_{50\%}$ in NPD in O_2 bubbles (99 mg/kWh) is 10 times higher than the Ar MWPJ (10 mg/kWh). This significant difference between the two reactors is not attributed only the fast MB degradation in the NPD configuration, but also due to the physic of both plasmas. In the Ar MWPJ an important portion of power is dissipated to heat the surrounding air, resulting in a local temperature increase. This is evident from the previous experimental results who indicated that the plasma gas temperature can reach very high values (few thousands of Kelvin) [15, 24, 25]. On the other hand, in the NPD, the short duration of the voltage pulse allows only electrons the gain a high energy to initiate the ionization process, whereas the heavier ions remains close to room temperatures [18]. This feature indicates that the NPD allows plasma ignition with less energy dissipation, explaining the high energy efficiency of this configuration.

Table. I
THE ENERGY YIELD ($Y_{50\%}$) OF THE NPD IN O_2 BUBBLES AND THE AR MPWJ

Configuration	$t_{50\%}$ (min)	P (W)	$Y_{50\%}$ (mg/kWh)
NPD in O_2 bubbles	5	181	99
Ar MWPJ	15	100	10

C. *The effect of electrical conductivity on the reactors performance*

Most of the previous studies investigated the plasma based water treatment using deionized water, but in reality the wastewaters exhibit very high electrical conductivities. Therefore, it is crucial to assess the reactors efficiency under conditions of high values of solution electrical conductivity. Herein, we investigate the impact of this parameter on the performance of the NPD in O_2 bubbles and the Ar MWPJ. It is worth to mention that during our experiments we observed that increasing the electrical conductivity beyond 200 µS/cm in the NPD configuration prevents plasma ignition, probably due to the fast current dissipation in the liquid [26]. On the other hand, the electrodeless design of the Ar MWPJ allowed increasing the solution conductivity to very high values (> 50 000 µS/cm) without altering the plasma jet ignition. At this stage, we believe that the Ar MWPJ is more suitable for highly conductive solutions.

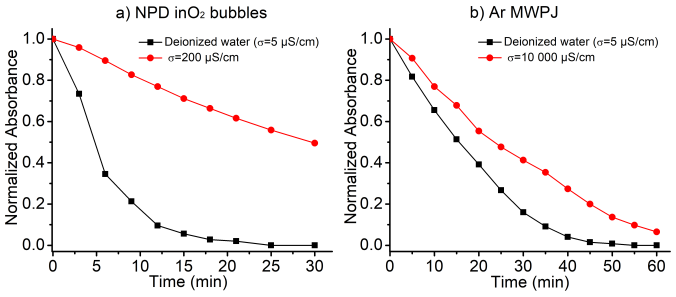

Fig. 5: Effect of the solution electrical conductivity on the MB degradation rate in: a) the NPD in O_2 bubbles, and b) the Ar MWPJ

The Fig.5a and Fig.5b illustrate the effect of the electrical conductivity on MB degradation in the NPD in O_2 bubbles and the Ar MPWJ, respectively. In the former configuration, increasing the electrical conductivity to 200µS/cm decreases the MB degradation rate considerably where the $t_{50\%}$ increases from 5 min at deionized water (5µS/cm) to 30 min at 200 µS/cm. this reduction could be explained by the fact that at high electrical conductivity the contact plasma-water is reduced (due to short plasma channels) which reduces the reactive species production. Regarding the Ar MWPJ configuration, the initial solution conductivity is adjusted at 10 000 µS/cm (for $\sigma <$ 10- 000 µS/cm no significant change in the MB degradation rate is observed). At this electrical conductivity, the MB degradation rate decreases; the $t_{50\%}$ increases from 15 min at deionized water (5 µS/cm) to 24 min at 10 000 µS/cm. In fact, this reduction is considered minor compared to the significant increase in solution electrical conductivity. Moreover, the observed reduction in the MB degradation rate is not caused by the presence of ionic products in general, but mainly by Cl^- ions (Provided from KCl dissociation) that have an inhibitory effect on OH radicals [27–29].

The table II shows the variation of the energy yield ($Y_{50\%}$) as function of the electrical conductivity. The $Y_{50\%}$ in the NPD configuration drops significantly by 82% (from 99 to 17 mg/kWh) when the electrical conductivity is increased from 5 to 200 µS/cm, whereas in the Ar MWPJ, the $Y_{50\%}$ drops only by 37% to reach 6.3 mg/kWh when the electrical conductivity is increased to 10 000 µS/cm. Overall, the results discussed in this section suggest the possibility to use the Ar MWPJ configuration for solutions with high electrical conductivities. On the other hand, the NPD in O_2 bubbles loses its efficiency rapidly for small electrical conductivity variations. These observations highlight the necessity to consider different operating conditions to assess the reactors performance.

Table. II
THE EFFECT OF ELECTRICAL CONDUCTIVITY ON THE ENERGY YIELD ($Y_{50\%}$) IN THE NPD IN O_2 BUBBLES AND THE AR MWPJ CONFIGURATIONS.

Configuration	NPD in O_2 bubbles		Ar MPWJ	
conductivity (µS/cm)	5	200	5	10 000
$Y_{50\%}$(mg/kWh)	99	17	10	6.3

IV. CONCLUSION

In the present study, we investigated the MB degradation using two configurations: the NPD in O_2 bubbles and the Ar MWPJ above the water surface. In the deionized water, the NPD in O_2 bubbles demonstrated superior MB degradation because of its ability to generate higher concentration of reactive species such as O_3, O, OH and H_2O_2. Furthermore, the energy yield ($Y_{50\%}$) at 50% demonstrated that the energy efficiency in the NPD in O_2 is higher compared to the Ar MWPJ (99 vs 10 mg/kWh, respectively). Unlike the Ar MWPJ, where a significant portion of power is dissipated to heat to the surrounding environment, the NPD ensures plasma ignition with minimal power dissipation, making it highly effective in terms of energy efficiency.

the other hand, at high solution conductivities, the NPD configuration prevents plasma ignition which causes a significant drop of energy efficiency that decreases from 99 mg/kWh in deionized water to achieve 17 mg/kWh at 200 µS/cm, whereas the performance of the Ar MWPJ drops by 37% only (from 10 to 6.3 mg/kWh) when the electrical conductivity is increased to 10 000 µS/cm . These findings suggest the possibility to use Ar MWPJ configuration for high conductive solutions.

The results of this study reveal the complexity of the comparison task between plasma reactors, suggesting thus the necessity to take into consideration other parameters to properly estimate the reactors performance.

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