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**Preliminary evaluation of decontamination with a 10-volt-supplied plasma jet: made possible by piezoelectric generator**

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# Preliminary evaluation of decontamination with a 10-volt-supplied plasma jet: made possible by piezoelectric generator

Jordan Stekke, Claire Tendero, Pascal Tierce, Christian Courtois, Gaetan Rauwel, Juslan Lo, Philippe Guillot, Francois Pigache

**Abstract**— This paper deals with preliminary decontamination performances obtained from a plasma jet generated by a piezoelectric transformer. This low voltage supply solution (<10V) is investigated as a compact and safe solution for the decontamination of medical thermo-sensitive devices. Promising reduction rates are obtained on *Pseudomonas aeruginosa* and *Staphylococcus aureus* bacteria with an argon plasma jet at atmospheric pressure about 2.5W input power.

**Index Terms:** piezoelectric transformer; atmospheric plasma jet; decontamination

## I. INTRODUCTION

Although the initial studies started focusing on the disinfection abilities of low temperature plasma in the early 90's, this scope is still active at the present time [1-4]. Numerous plasma generators have been developed to satisfy the disinfection requirements of thermos-sensitive or chemo-sensitive tools. Some examples of plasma jet (or needles) solutions for different applications are briefly described in Table 1. Many difficulties such as a high disinfection level, a minimal duration of treatment, a shape ratio or complex geometry of the tools still have to be overcome [5].

TABLE 1: EXAMPLES OF PLASMA JET SOURCES

Application field	Operating conditions	Target / issue
Oncology	Pulsed RF source 1.2 kV RMS / 3 W Gaz flow: 3 slm , 98% Ne / 2% Ar	Melanoma cells (in vitro & in vivo tests) [6]
Dentistry	2.6 kHz 1.1 W Air	<i>E. Faecalis</i> [7]
Desinfection	27.12MHz RF source 20 W Gaz flow: 20 slm Ar	<i>B. Atrophaeus</i> <i>E. coli</i> [8]
	4-6kV pulsed source 100ns pulse width 2kHz repetition Gaz flow: 5 slm, He / 1% O <sub>2</sub>	<i>B. Atrophaeus</i> [9]
Wound healing	Array of $\mu$ plasma jets 20 kHz	2nd degree burns [10]

	1 kV RMS Gaz flow: 1.5 slm, He	
	kINPen (neoplastools) 1MHz 1 kV / 3.5 W Gaz flow: 3 slm, Ar	Chronic wounds on domestic animals, clinical observations on leg ulcers [11]
Risk assessment	kINPen	Genotoxicity and mutagenic potential of argon plasma [12]
	25 kHz 3 kV / 2mA Gaz flow: A few slm, He	Skin damage on mouse [13]

Although these different sources operate at various frequencies ranging from kHz up to MHz with different DBD designs (most of the time a metal needle inserted in a quartz tube), they all require a high voltage source as input in order to initiate and sustain the discharge (depending on the gas, flow, dielectric configurations...). In some applications and depending on the required output power, such high voltage generators can be cumbersome. The proposed technology in this paper may bring about an alternative.

## II. DESCRIPTION OF THE PLASMA SOURCE

### A. Principle of a piezoelectric plasma generator

This section briefly introduces the piezoelectric properties and then focuses on the use of a piezoelectric transformer device for plasma discharge. The piezoelectric property is the ability of some (mono or poly crystalline) materials have the capacity to develop an electric potential when mechanical stress is applied [14-15]. Moreover this property is reversible.

The research on this topic really started in 2000's leading to a significant understanding of this technology [16-17]. Since then, the piezoelectric transformer as plasma generator has been explored in various applications such as an ozonizer [18], an excimer lamp VUV light source [19], an ignitor for pulsed plasma thrusters [20], a micro-thruster for satellite positioning control [21], an ionic wind source [22], a source for deuterium

or hydrogen neutron production [23], or a patented source for ambient mass spectrometry at atmospheric pressure [24].

The present piezo plasma generator uses a classical so-called Rosen-type transformer and its design is illustrated in Fig. 1. The first patent of piezoelectric transformer appears in 1931, explaining the concept [25]. More than 20 years later, in 1954, C.A. Rosen describes some designs and still used at the present time [26].

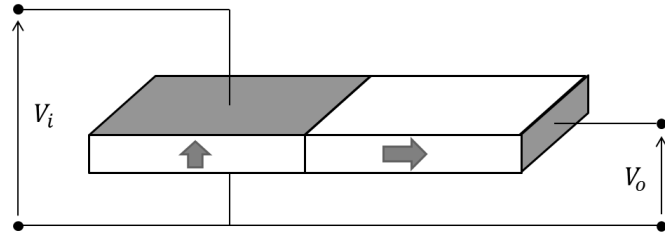


Fig. 1 Design of the step-up Rosen type transformer [27].

The principle of so called Rosen type piezoelectric transformer relies on the association of two (or more) piezoelectric elements that are mechanically coupled. The first is excited by an external electric supply (less than 10Vpk sine voltage) applied by electrodes, leading to mechanical vibrations (the reverse piezoelectric effect). It is qualified as the primary side (I), later referred to as “primary”. The second element also vibrates, inducing an electric field (direct piezoelectric effect) and it is qualified as the secondary side. Thus, the operation relies on two successive electromechanical conversions, enabling a voltage/current adaptation, an impedance adaptation, a high insulation level. Since this conversion results from the conversion of mechanical energy (propagating vibrations), the performances of such a component are strongly dependent on the design, the operating frequency, the electrode distribution and the piezoelectric material properties. Significant description and modelling of piezoelectric transformer designs and particularities are further discussed in research publications [28-29].

In the frame of plasma generation, the most interesting architectures are the high step-up voltage transformers: they can reach a voltage gain higher than 100 giving the opportunity to design a compact generator supplied with a very low input voltage.

For optimal efficiency and a maximum output voltage, piezo-transformers should be supplied at an operating frequency close to a resonant frequency of the structure. When the primary side is supplied close to one of the longitudinal resonant modes, a standing wave vibration occurs in the material and an electric potential distribution is obtained on secondary.

For plasma generation, the piezoelectric transformers can be used in two main different configurations:

- As a simple two-port step-up voltage component. The electric charges are collected on the secondary side by a metal needle [30], or between two electrodes with a narrow gap and separated by a dielectric layer [31].
- Another configuration directly involves the secondary surface as a dielectric layer which supports the discharge

[32-33]. In such configuration, there is no need for the secondary electrode. This particular configuration avoids sparking (DBD-like configuration) and it benefits from the high dielectric constant of the material. Let us remember that the relative permittivity of PZT ceramic is about several hundred units ( $\epsilon_r \approx 1000$  for hard PZT ceramic). This latter configuration has been selected for this study.

Contrary to a passive dielectric layer in conventional DBD, the electric potential on the piezo surface is not uniform and depends on the excited resonant vibratory mode. Consequently, for the first longitudinal vibratory modes, the electric potential distribution is illustrated in Fig. 2(a). This distribution drives the plasma discharge location surrounding the transformer surface (Fig. 2(b)). Moreover, the electric potential amplitude is strongly a function of the input voltage supply, the mechanical damping coefficient and the operating angular pulsation. With the appropriate operating conditions this maximum potential value can reach several kV.

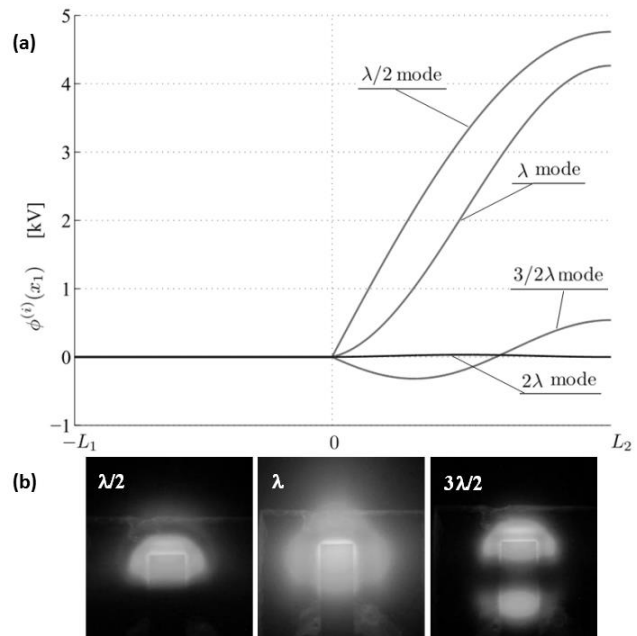


Fig. 2 (a) The theoretical electric potential on the transformer surface for the first four longitudinal vibratory modes. (b) Plasma discharges for first three modes obtained at 10mbar air pressure.

The various dimensions of the Rosen type transformer used in this study are summarized in Table 2.

TABLE 2: PIEZO TRANSFORMERS CHARACTERISTICS

	Hard PZT
Ceramic	Hard PZT
Total length (mm)	30.4
Width (mm)	7.6
Thickness (mm)	2.3
Operational vibratory mode	$\lambda/2$
Operating frequency (kHz)	$\sim 53$ kHz

As the Piezoelectric Transformer (PT) is a resonant device, it can be accurately simulated by an equivalent electrical circuit. This equivalent circuit is valid in the vicinity of a selected resonant mode and element values are dependent on the geometry and the material properties.

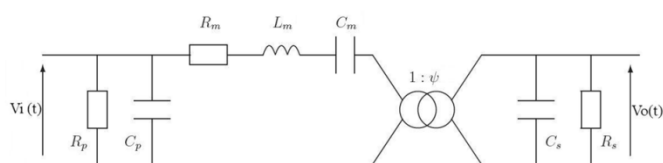


Fig. 3 The electric equivalent circuit of piezo transformer

As emphasized in Fig. 3, the PT is essentially capacitive due to the primary side. Its electrical behavior changes when supplied at its mechanical resonant frequency simulated by the two parameters  $L_m$  and  $C_m$ . The voltage gain is depicted by factor  $\psi$ . The expressions of the equivalent circuit parameters are not given in detail in this paper. However there is a considerable body of research devoted to the modelling [34].

As a consequence of the non-linear behavior of the plasma discharge and the load dependency of the voltage gain, the output voltage on the transformer termination is rather complicated to evaluate. It should be noted that an important difference exists here compared to conventional plasma generators where, most of the time, the electric quantities applied to the termination can be directly measured by voltage or Pearson current probes. In the present case, and considering that the plasma is directly generated on the secondary side surface, only electrical measurements from the primary side are possible.

The voltage gain of the transformer highly depends on the concordance of the device's supply and the resonant frequencies of the device. Since the resonant frequency tends to slightly shift with the steady state electric load (plasma load) and the transformer temperature, systematically rises during such operations. Thus a control feedback is required to maintain a stable plasma jet. This optimal operating point is maintained by the admittance phase tracking [35].

For the preliminary bactericidal tests carried out with this piezoelectric technology, an elementary plasma stylus design has been made as illustrated in Fig. 4.

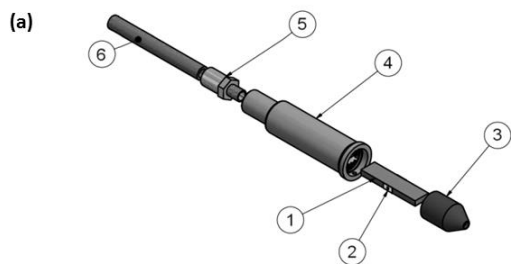


Fig. 4 Design of the piezo-plasma jet stylus (a) : (1) PT (2) electric connectors (3) stylus nozzle (4) stylus body (5) gas connector (6) gas tube. The piezo-plasma jet in operation (b) with a 1slm Ar gas flow

### B. Plasma Diagnostics

The characterization of piezo-plasma jet was made by optical emission spectroscopy and by bidimensional emission imaging. An optical spectrometer (HR2000+, Ocean Optics, slit 10  $\mu\text{m}$ , 200 to 1100 nm) coupled to an optical fiber (core diameter 600  $\mu\text{m}$ ) was used to identify the plasma emission lines [36]. Firstly, the measurement was realized facing the plasma source exit in order to identify the typical optical emissions of the carrier gas and of the reactive species due to the ambient air. Secondly, the optical fiber was placed perpendicular to the piezo-plasma jet on a micrometric translation stage. Moving the fiber along the piezo-plasma jet, the linear translation system allowed to characterize the axial distribution of the species excited or ionized by the plasma. In parallel, a fast ICCD camera (Princeton PIMAX-2K-RB, 1024x1024) was placed beside the jet, perpendicularly to the source, in order to assess the 2D distribution of plasma emissions within the plasma jet [36]. The jet length was determined from the ICCD camera data. The experimental setup has been described previously in [36-37].

#### Optical emission spectrometry

In the standard experimental conditions of the piezo-plasma jet stylus, the emission spectra of the discharge were measured. Fig. 5 highlights the main emission lines of the plasma, observed by many authors [38-41] on a reduced wavelength range (300-800 nm).

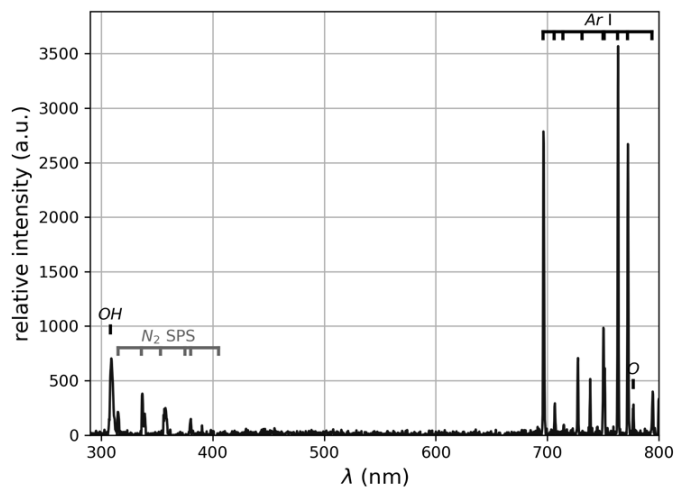


Fig. 5 Main emission lines of the plasma on a selected range of wavelengths

The UV and visible emissions identified in Fig. 5 correspond to the radiative de-excitations of argon, hydroxyl (OH radical), molecular nitrogen and oxygen. The presence of emission lines relative to ambient air species demonstrates the interaction of the plasma jet with its environment. In these experimental conditions, facing the plasma, the emissions are dominated by the argon emission lines [37-40]. The presence of hydroxyl in the UV-B region (308 nm) is clearly visible [39] and related to processes involving water molecules. The nitrogen second positive system can also be observed [39-40], these emissions are situated in the 300 to 450 nm range. The production of the excited nitrogen molecule can come from electron collisions

from the fundamental as well as from Ar metastable excitation transfer processes to the nitrogen molecules. Oxygen (777 nm) is the last sign of plasma and ambient air interaction [40].

When the optical fiber was placed perpendicular to the piezo-plasma jet stylus, the micrometric translation stage allowed the characterization of the emission's spatial variations. Two spectra were recorded in this work. The first one corresponds to the plasma emissions produced inside the source, close to the piezo transformer edge (Fig. 6 (a)). The second one was obtained at the top of the plasma jet, 15 mm far from the stylus outlet (Fig. 6 (b)).

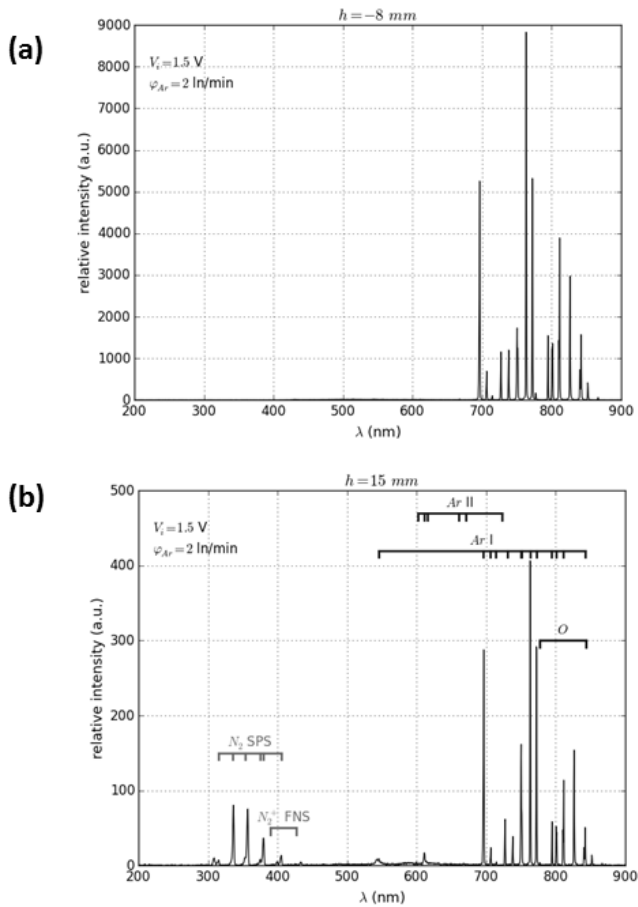


Fig. 6 Main emission lines of the plasma, (a) inside the source, 8 mm before the stylus outlet, and (b) in the plasma jet, 15 mm away from the source outlet.

Inside the stylus (Fig. 6 (a)), the plasma emission is mainly composed by Ar emission lines. Even if gas impurities (99.995% argon purity) or device leaks may cause weak emissions at the corresponding wavelengths (nitrogen, oxygen), this was not recorded in our work. Outside the plasma source structure (Fig. 6 (b)), the intensity of argon emission lines are lower and decrease from the source exit to the tip of the plasma jet. Concerning the ambient air species, an increase of the corresponding emission lines intensity can be seen between the plasma created inside the source and the plasma jet located outside the source. Indeed, when the argon plasma jet, generated inside the stylus, flows through the ambient air, its energy is high enough to excite the corresponding external species.

### 2D imaging of plasma emissions

The fast ICCD camera gives information on the spatial distribution of the plasma. Measurements were made in the standard working conditions of the piezo-plasma jet source defined previously. The region of interest analyzed with the ICCD camera and an example of plasma distribution are presented on Fig. 7. The spectral range of the camera is 195-920 nm.

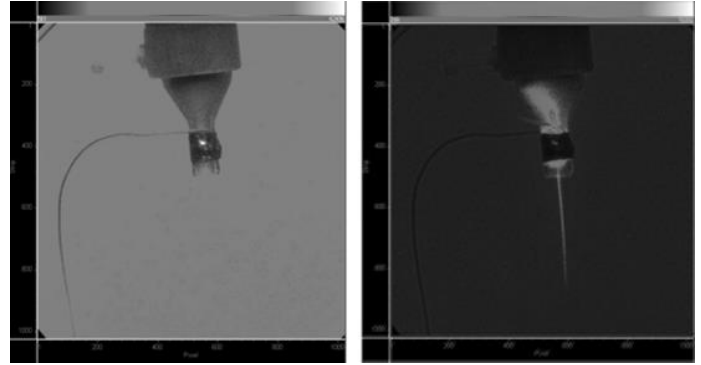


Fig. 7 2D imaging of the source, without (left) and with (right) plasma (all emission range, no filter)

The discharge intensity is highest inside the stylus, in the plasma creation zone. From the outlet of the nozzle the plasma jet has lower overall intensity, which decreases gradually from the exit to the extinction point (over 20 mm long).

The ICCD camera was also coupled to bandpass interference filters (Thorlabs, 10 nm as bandwidth) centered on specific central wavelength values. Based on the lines of interest, determined from the emission spectra, two filters that allowed the characterization of species production at the outlet of the nozzle in contact with ambient air have been used. The first one, centered on 750 nm, allowed the assessment of argon distribution. The second one, centered on 380 nm, showed the nitrogen distribution. These results are presented on Fig. 8.

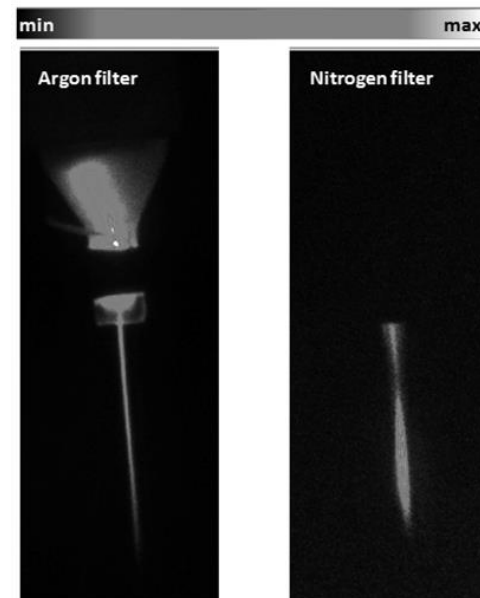


Fig. 8 2D normalized imaging of the plasma inside and outside the source, 750 nm argon filter (left) and 380 nm nitrogen filter (right).

The highest argon emission intensity corresponds to the interior of the source. From the exit of the nozzle, argon emission intensity decreases until plasma extinction at several tens of millimeters from the exit. Nitrogen emissions start at the exit of the source. The emission intensity increases, reaches a maximum and then decreases. This maximum emission is located in the intermediate region of the plasma jet. Before this maximum, argon-air ratio is too high and plasma is dominated by argon emissions [42]. After this maximum value is reached, argon-air proportion is too small and the plasma jet is not able anymore to excite ambient air species [41].

### III. EXPERIMENTAL SECTION – BACTERICIDAL TEST CONDITIONS

The evaluation of the piezoelectric plasma jet efficiency used for the first time as a disinfection device is carried out with two different bacteria strains (Gram-positive and Gram-negative):

- *Pseudomonas.aeruginosa* - Institut Pasteur - CIP 103 467
- *Staphylococcus.aureus* - Institut Pasteur - CIP 4 83

The methodology that was carried out relies on the adaptation of the EN 13697 protocol (bactericidal effect on non-porous surface). This standard protocol implies operating at ambient temperature ( $\sim 20^{\circ}\text{C}$ ) and a significant effect is considered when the reduction rate is superior to 4log compared to the control sample. Each test has been duplicated at least twice to four times to ensure robustness of the experimental results.

The samples are prepared as follow: a 50 $\mu\text{L}$  droplet of the initial suspension (that contains between 1.5e8 CFU / mL and 5.0e8 CFU/ mL of bacteria in tryptone salt diluent) is deposited on a 2cm-diameter stainless steel substrate. Then samples are dried at  $37^{\circ}\text{C}$  in an incubator during 20 minutes before being treated with the plasma jet. It consists in directly and vertically applying the plasma jet on the surface carrying the bacteria inoculum, as illustrated in Fig. 9.

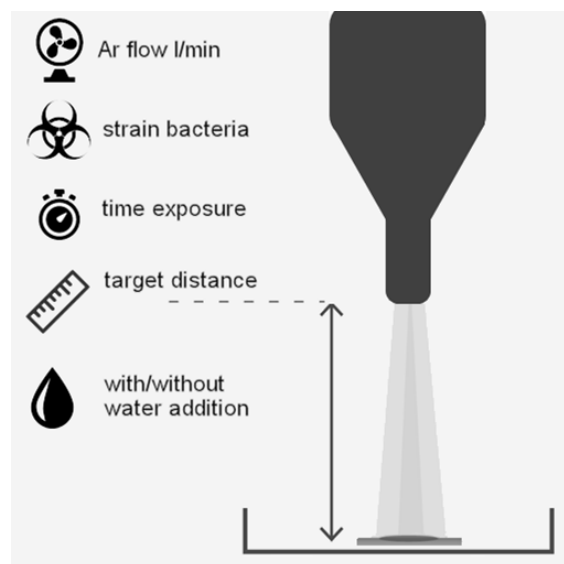


Fig. 9 The plasma jet treatment protocol

After the plasma treatment, the stainless steel samples are transferred into a 10 mL container of the tryptone-salt diluent

where the microorganisms are removed from the disk by shaking with glass beads. This mixture is 10 to 100 diluted and 1mL is grown in TSA (15ml to 20ml) in an incubator at  $36^{\circ}\text{C}$  during 2\*24h. Only Petri dishes ranging from 15 up to 300 CFU + -10% are used for counting.

The operating plasma discharge regime is comparable to a glow discharge, obtained by simply placing the plasma jet in contact with the inoculum without any particular layout.

The experimental details of the procedure are presented in Table 3. The log reduction is finally evaluated for different exposure times for both bacteria species. The authors are aware that several bactericidal actions operate during the exposure period, leading to the slope variation of the log reduction [43]. In this preliminary study, the purpose is to observe if the piezoelectric generator can provide significant bactericidal effect (4 log reduction and beyond) during a sufficient exposure period.

TABLE 3: EXPERIMENTAL CONDITIONS

Bacteria strain	Operating conditions
<i>S. Aureus</i>	<ul style="list-style-type: none"> <li>▪ ON / OFF device</li> <li>▪ without / with 100 <math>\mu\text{L}</math> droplet water</li> <li>▪ 1.5 cm target distance</li> </ul>
<i>P. Aeruginosa</i>	<ul style="list-style-type: none"> <li>▪ Gaz flow rate : <math>\sim 1\text{slm}</math> argon</li> <li>▪ Exposure duration: 2, 5, 10, 15, 20 mn</li> </ul>

Due to the fact that the presence of water in any plasma activated water (PAW) significantly favors the biocidal effect [44-45], tests are carried out in two configurations: dry conditions and with the addition of 100 $\mu\text{L}$ -distilled-water droplet on the inoculum surface. Log reduction on both bacteria strain is presented in Fig. 10.

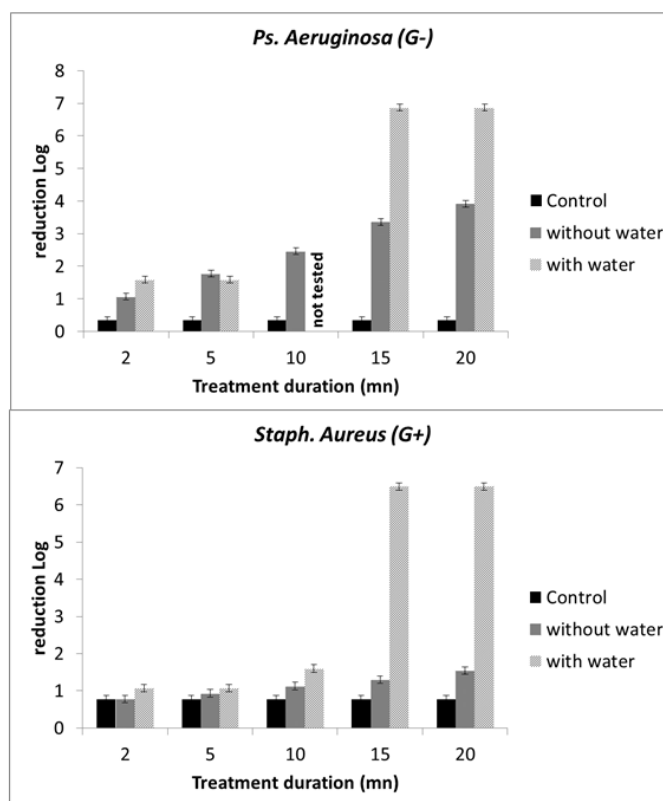


Fig. 10 The log reduction according to the exposure time and operational conditions

#### IV. RESULTS AND DISCUSSION

##### C. Bactericidal performances

Several observations can be deduced from results presented in Fig. 10. Firstly, the exposure of the dried inoculum to an argon gas flow without the plasma discharge does not result in significant bactericidal effect: whatever the duration of the exposure period is, the log reduction does not change in these conditions. On the contrary, after a few minutes exposure period to plasma discharge, log reduction is observable for both bacteria strains. Therefore, the following conclusions can be drawn:

- The longer the exposure time, the higher the reduction of bacteria, which is consistent with any other plasma treatments presented in research.
- As already demonstrated in research the *Staphylococcus aureus* (gram-positive) is more resistant than the *Pseudomonas aeruginosa* (gram-negative) [46].
- The presence of water in the decontamination process radically improves the efficiency, including those observed with the *S. aureus*. After a 15- minutes-exposure, both strains have reached the maximum measurable reduction factor (over 6.73 log). Please note that the droplet water has completely evaporated after 10 minutes of exposure.

In spite of the low input power applied to the piezoelectric device (about 2.5W @53kHz), the disinfection capacity is clearly visible. Obviously, the exposure time treatment has to be reduced to generate more interest in this technology. The piezoelectric component can be highly improved to be adapted to this new use. However these results are quite promising with regard to the maximum measurable value obtained.

##### D. The technical limits and improvement perspectives

Even if the piezoelectric technology can bring some advantages in terms of product safety, compactness, low voltage supply, it is also subjected to some technical limits which require attention. Firstly, the electric potential significantly depends on the amplitude vibration produced in the piezo material. The vibration implies mechanical stress in a precise location. All necessary precautions should be taken to prevent brittleness, for example by a specific transformer design or by limiting the mechanical stress during the operation [47]. Moreover, depending on the plasma location and its regime, excessive discharge power density is likely to occur at an unsuitable location of the transformer surface. This can damage the material by erosion and local heating. This phenomenon is observed at the edge of the primary electrode [46]. Such a sparking event can be prevented by depositing a thin coat of a dielectric material on the piezo surface. However this dielectric layer should also be able to withstand the vibration of its substrate. Finally, the piezoelectric property can vanish making the transformer inoperative if its temperature exceeds the Curie temperature: indeed at this temperature a reversible change in the crystalline occurs and results in the depolarization of

ferroelectric material. The temperature rise is essentially due to the self-heating induced by the internal friction force. The typical Curie temperature limit is below 250°C for standard PZT hard ceramic.

All these technical precautions actually limit the power of such a piezo plasma generator to a few watts. However, specific design and precautions may enhance the power availability and thus the resulting bactericidal properties.

#### V. CONCLUSION

Preliminary decontamination tests have been carried out with an atmospheric pressure plasma jet generated by a piezoelectric transformer. An elementary plasma stylus has been designed with a standard off the shelf transformer, supplied by a 10 volt voltage source. To prevent the frequency discrepancy due to the thermal effect, a simple phase locked loop is added to maintain plasma discharge over time. This plasma jet relies on 1 slm argon gas flow and is finally applied to two different bacteria strains following the recommendations of EN 13697 protocol. The resulting reduction rate obtained with this argon plasma jet has reached significant bacteria reduction and the maximal measurable value in the most convenient configurations. This low voltage solution gives rise to the opportunity to develop safety low voltage plasma generators. Nevertheless at this development stage, a specific design of the piezoelectric element is required. This should be associated to an appropriate feedback control to significantly improve these promising preliminary results.

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