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APPLICATION OF ATMOSPHERIC PLASMA AND PLASMA-ACTIVATED WATER IN THE CONSERVATION OF HISTORICAL PAPER

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Abstract

This study presents research carried out on the combination of atmospheric plasma and plasma-activated water (PAW), applied to the conservation of graphic documents. The experiments were conducted on two samples of old paper, and the treatment results were assessed through visual and tactile analysis, transmitted light microscopy, and Energy Dispersive Spectroscopy (EDS). The results obtained demonstrate promising potential for treating pathologies associated with paper degradation, namely dirt, chemical alteration, acidification, and oxidation. The treated samples, compared to the reference samples, showed significant improvements in cleanliness and mechanical strength, as well as an apparent reduction in oxidation.

***Keywords**: Conservation; Paper; Plasma; PAW; Oxidation; Decontamination*

Introduction

Historical and archaeological objects of organic origin are subject to a range of degradation processes resulting from the passage of time and exposure to both natural and anthropogenic agents. The accumulation of particles and contaminating agents, originating from corrosion products, salts, and the build-up of dust and dirt on the surface or within the microstructures of materials, as well as microbial development by fungi and bacteria and exposure to harmful radiation such as UV, all affect the physical and chemical properties of these materials [1-4].

One such material is paper, the basis of most historical documentary heritage and commonly used as a medium for various types of records, including photographs, books, and paintings, among others. Due to its inherent fragility, paper conservation is a highly delicate and complex process [5-7]. The treatment methods traditionally employed rely on mechanical dry cleaning and/or chemical techniques, which present varying degrees of risk and difficulty for conservators. Physical contact between tools and the surface can cause vibrations and friction, potentially leading to mechanical damage in already weakened materials, while the use of chemical agents may react with and adversely alter the substrate's composition [8-10].

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The use of non-thermal or low-temperature plasmas has been tested in various ways in heritage conservation, particularly for metals, where it has been applied for chemical reduction and cleaning of oxidation layers [2], [4], [8]. Similarly, in recent years, research into the use of non-thermal plasmas has also been extended to organic materials, primarily for microbial deactivation as a means of disinfection, showing promising results [4], [9]-[12], as well as in the superficial cleaning of substrates [5].

In the specific case of paper conservation, research has focused on the use of cold plasmas for disinfection and cleaning [3], [13]-[16]. Noteworthy examples include the work of Vizàrová's team [3], who applied plasmas generated in Ar, oxygen, nitrogen, and carbon dioxide atmospheres to sterilize a range of microscopic fungi on paper, and the project led by Ioanid [16] employing cold plasma and corona discharge, which proved effective in decontaminating such materials. Another significant approach is the use of plasma combined with hexamethyldisiloxane for deacidifying old paper [17]. In all these cases, promising results were obtained, demonstrating high efficiency in microbial sterilization and pH stabilization.

However, with regard to the use of Plasma-Activated Water (PAW) applied to organic cultural heritage materials such as paper, no known studies have yet been conducted. Research in this specific area is very recent and has mainly been explored in fields such as medicine, as a surgical disinfectant [11], [18]-[20]; and, for instance, in agriculture, as a seed germination agent [21], [22].

Thus, the present study focuses on observing the effects of the combined use of plasma and plasma-activated water on aged paper, revealing its potential as a sustainable and safe alternative for the conservation of historical paper materials.

Theoretical Framework of the Experiment

Plasma, often referred to as the fourth state of matter, is defined as a quasi-neutral gas composed of charged and neutral particles that exhibit collective behavior [23]. According to Fridman, plasma is an ionized gas in which at least one electron is not bound to an atom or molecule [24]. While some partially ionized gases may not strictly fit Cheng's definition due to the absence of collective behavior, they are driven by electric fields and are considered stationary plasmas, owing to the ionization of neutral particles and the lack of thermal equilibrium between electrodes and ions [25].

There are two main types of plasma: thermal plasma and non-thermal plasma. In thermal plasma, all particles—electrons, ions, and neutral atoms—are in thermal equilibrium. In contrast, non-thermal plasma is characterized by a significant temperature disparity, where electrons reach high temperatures while ions and neutral particles remain at much lower temperatures, close to ambient conditions [26], [27].

Plasma can be generated by various methods, through the ionization of a specific gas or a mixture of gases. When atmospheric air is ionized by an electric field, the resulting state is referred to as atmospheric plasma. Plasma generation involves applying a strong electric field to a gas, causing changes and rearrangements in its chemical composition. This process produces reactive chemical species, such as Reactive Oxygen Species (ROS) and Reactive Nitrogen Species (RNS), which are oxygen and nitrogen radicals, respectively [23], [25], [28], [29].

When plasma comes into contact with water, it transfers these radicals to the liquid, temporarily imbuing it with disinfectant and cleaning properties [30], [31]. This plasma-treated liquid is known as Plasma-Activated Water (PAW).

PAW contains reactive species such as free radicals, ozone (O_3), hydrogen peroxide (H_2O_2), and other oxygen-based compounds generated during the activation process. These species give the water antimicrobial properties, making it effective in disinfecting surfaces, equipment, and even food. The free radicals and oxygen compounds can inhibit the growth of bacteria, fungi, and viruses [28].

The majority of studies [1], [3], [13] have demonstrated that low-temperature plasma is effective in inhibiting bacterial and fungal growth, with the efficacy largely depending on the presence of charged chemical species. In the case of paper, experiments have been conducted using plasmas from various gases, including oxygen and hydrogen. Results indicated that although oxygen plasma possesses strong biocidal activity, it also causes collateral damage to the substrate, particularly affecting the mechanical strength of paper due to its oxidative properties. Conversely, hydrogen plasma results in less degradation. Interestingly, when water was used as the plasma gas—containing both oxygen and hydrogen—only slight alterations were observed [1]. This observation suggests improved structural preservation when both gases are combined.

From a biological perspective, researchers have highlighted four main factors that influence microbial inactivation, which may act individually or in combination: heat, UV radiation, charged particles, and reactive neutral species [28]. These factors must be considered alongside parameters such as power, gas mixture, and flow rate, which determine the efficiency of bacterial deactivation [10].

Some studies in the medical field have shown that direct plasma exposure is significantly more effective and faster than treatment with post-glow plasma (indirect plasma). In non-thermal plasmas, results indicate that reactive chemical species play the most critical role in disinfection and bacterial inactivation, minimizing the impact of heat or UV radiation, which can contribute to substrate degradation [28].

Therefore, it is understood that non-thermal plasmas and PAW possess theoretically promising capabilities for their application in the conservation of organic historical artifacts, particularly graphic documents.

Methods

The process involved assembling equipment composed of a 35 kV DC power supply and a wooden structure with two horizontally aligned aluminum plates arranged in parallel to generate the electric field necessary for atmospheric plasma production (Fig. 1). Each aluminum plate was connected to one of the power supply's terminals: the lower plate to the negative pole and the upper plate to the positive pole.

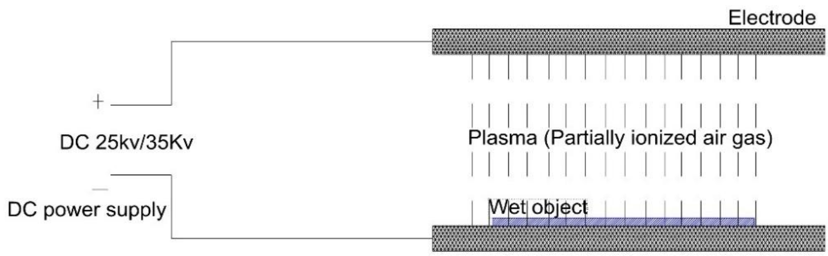

Fig. 1. Schematic figure of the system used in the experiment

In order to prevent the formation of electric arcs, the plate spacing was calculated based on the dielectric breakdown strength of air, estimated at 3 MV/m [32], or ±30 kV/cm. Given that the applied voltage was 35 kV, the distance between the plates had to exceed 1.2 cm. Additionally, it was necessary to account for environmental factors such as air humidity and temperature, particularly humidity, which significantly reduces the dielectric strength of air. Therefore, a plate distance of 4 cm was adopted to ensure a safety margin, since under humid conditions the dielectric strength can drop to 10 kV/cm [32].

Two paper samples were selected: one from an old newspaper (AP01) (Fig. 1) and another from kraft paper taken from the backing of a 19th-century picture frame (AP02) (Fig. 2). Two fragments were collected from each sample: one as a reference and the other for treatment (Table 1).

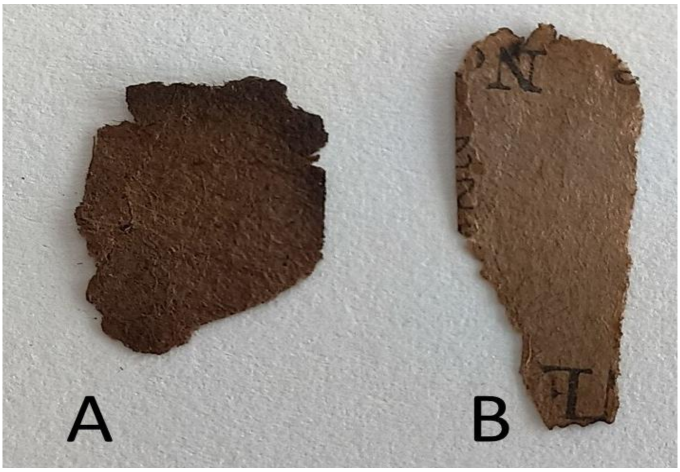

Fig. 2. Sample AP01 of newspaper paper: A – Reference sample without treatment; B – Test sample after treatment

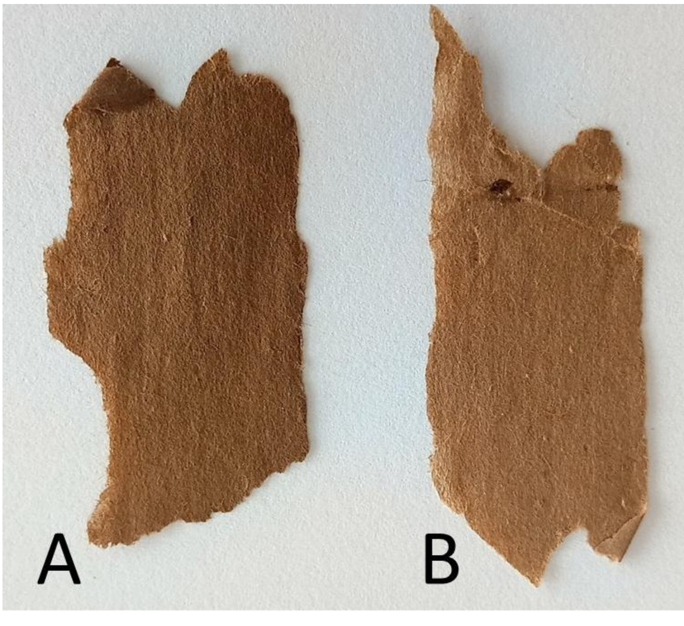

Fig. 3. Kraft paper sample AP02: A – Reference sample without treatment; B – Test sample after treatment

Table 1. Analyzed samples

Sample	Tipe	Sample ref. – (A)	Sample test – (B)
AP01	Papel de jornal	AP01 ref.	AP01 test
AP02	Papel Kraft	AP02 ref.	Ap01 test

The preparation of the test samples involved controlled humidification using tap water applied with a dropper. After preparation, the samples were individually exposed to the electric field, and the process was considered complete once the samples were completely dry.

Subsequently, the test samples were analyzed through visual inspection and compared to the reference samples to assess stiffness, mechanical strength, color, cleanliness, and texture, using a qualitative comparative criterion. Additionally, transmitted light microscopy analyses were performed, and the samples AP01 ref. and AP01 test were chemically analyzed using Energy Dispersive Spectroscopy (EDS).

Results and discussion

The AP01 test sample showed significant improvements in cleanliness and coloration. Under transmitted light microscopy at 100× magnification (Fig. 5-B) and 400× magnification (Fig. 6-B), the degree of cleaning can be clearly observed. Notably, the coloration of the fibers and the binder suggests a possible reversal of the oxidation process. No differences were observed in the surface texture.

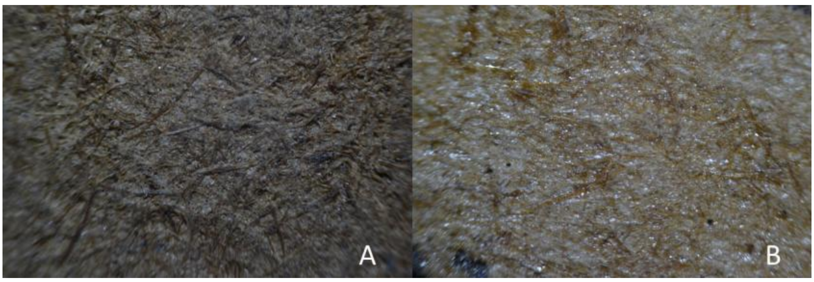

Fig. 4. Analysis by transmitted light microscopy, magnification 100×: A - Reference sample; B - Treated sample

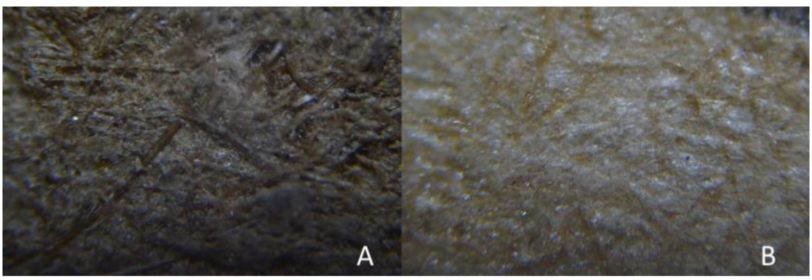

Fig. 5. Analysis by transmitted light microscopy, magnification 100×: A - Reference sample; B - Treated sample

A significant improvement was recorded in both malleability and mechanical strength, to the extent that the reference sample AP01 ref. showed signs of fragility and fragmentation—a condition not observed in the treated AP01 test sample. The ink of the letters does not appear to have been affected (Fig. 2).

In the case of sample AP02, significant improvements were noted in coloration, which became very close to that of new Kraft paper. Under transmitted light microscopy at 100× magnification (Fig. 7) and 400× magnification (Fig. 8), the degree of cleaning and the brightening of the vegetal fibers in the AP02 test sample are also visible. Increased flexibility and effective cleaning were also observed. No perceptible changes were identified in the surface texture.

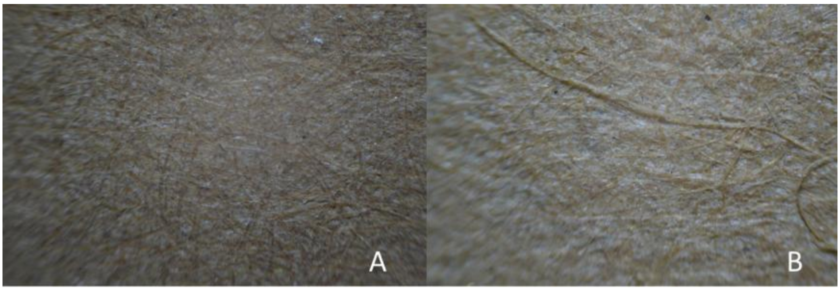

Fig. 6. Analysis by transmitted light microscopy, magnification 100×: A - Reference sample; B - Treated sample

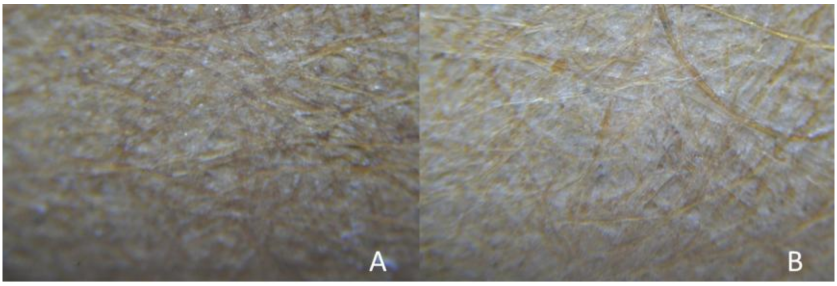

Fig. 7. Analysis by transmitted light microscopy, magnification 400×: A - Reference sample; B - Treated sample

Samples AP01 ref. and AP01 test were also analyzed using EDS. Tables 2 and 3 present the results of the EDS analyses and the corresponding chemical spectra (Figs. 9 and 10).

Table 2. Chemical composition of AP01 ref.

Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	Abs. error [%] (1 sigma)	Rel. error [%] (1 sigma)
O	8	18595	49,99	49,99	45,25	6,49	12,98
C	6	18307	43,48	43,48	52,43	5,66	13,01
Fe	26	1350	3,57	3,57	0,93	0,18	4,99
Si	14	1937	1,03	1,03	0,53	0,08	7,59
Ca	20	736	0,69	0,69	0,25	0,06	8,66
S	16	1159	0,64	0,64	0,29	0,06	8,97
Al	13	1030	0,6	0,6	0,32	0,06	10,51
Sum			100	100	100		

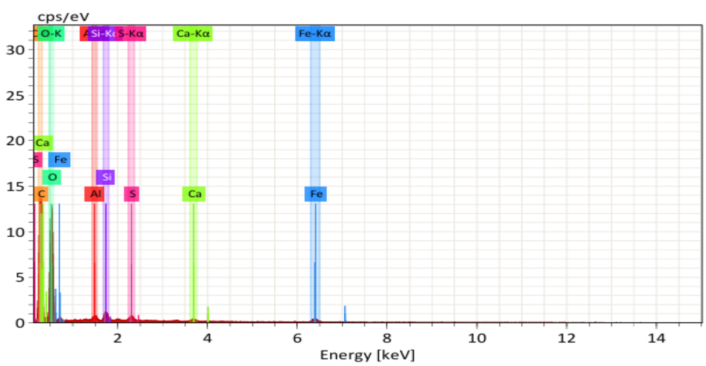

Fig. 8. Chemical Spectrum of the AP01 Reference Sample (EDS)

Table 3. Chemical composition of AP01 test

Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	Abs. error [%] (1 sigma)	Rel. error [%] (1 sigma)
O	8	81184	52,35	52,35	47,13	6	11,45
C	6	69923	41,83	41,83	50,16	4,84	11,57
Si	14	14615	1,86	1,86	0,95	0,11	5,66
Al	13	12961	1,76	1,76	0,94	0,11	6,23
Ca	20	4973	1,16	1,16	0,42	0,06	5,58
Fe	26	549	0,35	0,35	0,09	0,04	12,68
S	16	1889	0,28	0,28	0,13	0,06	13,42
K	19	1002	0,12	0,12	0,05	0,03	17,75
Mg	12	1145	0,18	0,18	0,11	0,04	20,54
Ti	22	98	0,03	0,03	0,01	0	11,16
P	15	1	0	0	0	0	122,78
Sum			100	100	100		

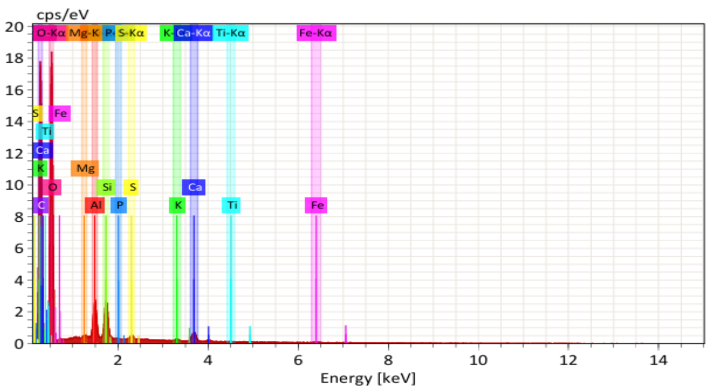

Fig. 9. Chemical Spectrum of the AP01 Test Sample (EDS)

The significant increase in chemical elements typically present in paper observed in the treated sample, compared to the reference sample, combined with the sharp decrease in iron content (from 3.57% to 0.35%) on the surface, suggests that in sample AP01 (ref), the remaining chemical elements were likely obscured by a layer of iron oxides covering the surface. This oxidation layer contributed to the darkening of the paper, which is also evident in the previously discussed microscopic analysis.

The apparent increase in oxygen concentration and the decrease in carbon content in the AP01 test may indicate a slight degree of oxidation of the paper substrate. However, when these findings are cross-referenced with the transmitted light microscopy analysis (Figs. 5 and 6), which shows the opposite trend, it can be assumed that this variation in oxygen and carbon concentration may be misleading and potentially due to rearrangement and alteration of the chemical spectrum between the two samples. Although these particular data are not entirely conclusive, the combination of the different analyses demonstrates significant improvements in the paper and highlights the effectiveness of this conservation treatment.

Conclusions

The preliminary results from the experiments conducted with the combination of atmospheric plasma and plasma-activated water suggest the viability of this technology for the conservation of graphic documents on paper substrates. The ability for cleaning, as well as the improvement in physical properties and aesthetic appearance, was clearly evident. Although samples with biological agents such as fungi were not tested, the cleaning achieved in the treatment hints at a potential efficacy in addressing such issues, commonly encountered in historical books or documents.

The potential reduction in paper oxidation is another significant advantage of this technology, ensuring a process of deacidification, restoration of plant fibers, and microstructural improvements that are reflected in the physicochemical properties of the paper.

Building on the accumulated experience, a specific equipment setup is being developed, incorporating systems for controlling and monitoring the electric field. This will allow for a more structured understanding of the processes involved, as well as testing different properties and parameters, particularly in observing the effects of varying electrical voltages on treatments. In the same vein, further experiments will be conducted on a broader range of pathologies associated with such materials, particularly those of biological origin. These adjustments will enable the calibration of the process and its adaptation to the specific needs of each condition, aligning with conservation and restoration criteria. Additionally, more chemical characterization analyses will be carried out on new samples, which may complement and confirm the results presented in this article.

Beyond the already demonstrated benefits for the quality of conservation and restoration treatments for graphic documents, the use of this technology eliminates the need for chemicals, some of which are harmful, thereby protecting the environment and user health. This approach aligns with green technology methodologies.

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