Cleaning soot-contaminated silk textiles using plasma generated atomic oxygen, a new technological approach.

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INTRO

The cleaning of soot from historic textiles is considered one of the most intricate challenges for conservators. Soot can be electrostatically bonded to the surface and penetrate into the substrate following fire damage. Conventional contact cleaning treatments, such as dry and solvent cleaning often result in compacting the particles deeper into the fibrous structure, giving rise to an incomplete and unsatisfactory outcome. Nevertheless, there are relatively few publications targeting soot removal from textiles.

Plasma technology has been utilized for surface cleaning in industries, due to its contactless, non-toxic and residue-free characteristics. In our current projects, a novel non-thermal plasma generated Atomic Oxygen (AO) is developed at UGent for removing carbon-based contaminations from a series of substrates as an innovative application for cultural heritage. As part of the European MOXY project, we are working on soot cleaning from silk textile using AO at UAntwerp.

Experimental

Preparation of mock-up

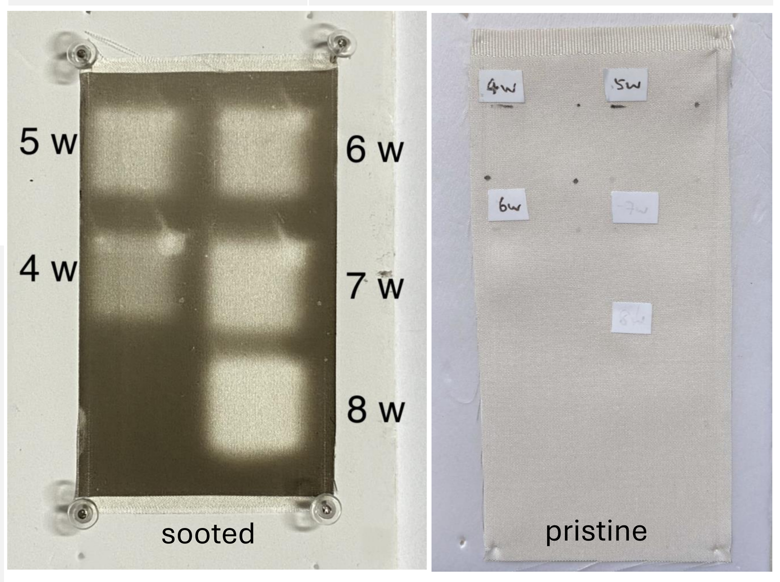
Fire soot deposition

- Soot was applied by combustion in a home-built drum device.
- Temperature and time controllable.
- Homogenous distribution of soot particulates.

Optimization of cleaning parameters

Cleaning test at selected conditions

Parameter	sooted	pristine
Exposure time (t/min)	7.5, 15, 30, 60 (at 6 W)	
Power (W)	4, 5, **6**, 7, 8 (at *t* = 7.5)	



Sample after AO treated at different power

Methods

- Input: RF 13.56 MHz, main gas - 4 slm helium and 30 mslm O_2-He admixture, shielding gas 4 slm argon.
- The output power is controlled below 8 w due to heating effect (80°C on sample at a fixed position). Distance set at 4 mm to ensure within the effective area - the lifetime of excited O atom is very short in atm condition being up to milliseconds.
- Each cleaned area is 2 x 2 cm square and homogenously cleaned on a programmed platform moving at constant speed in *x* and *y* axis.

AO generation

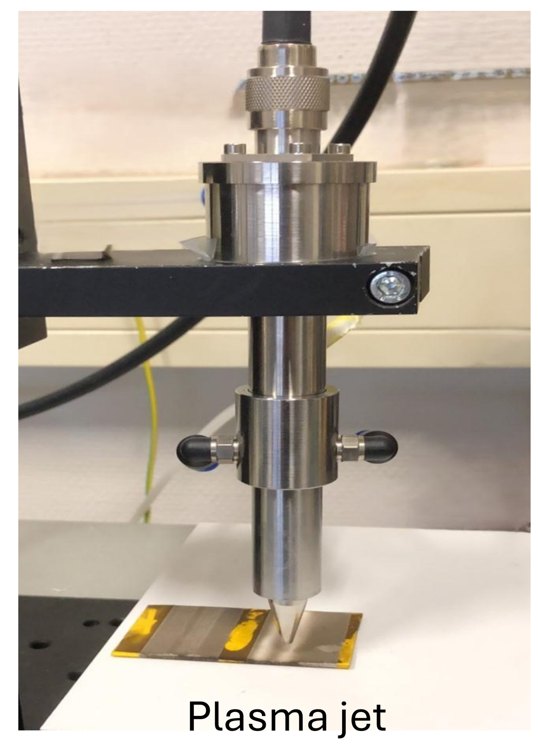
- Input - output power
- Gas composition and flow
- Jet design – nozzle dimension

Effective dose per area

- Exposure time
- Distance
- Moving speed

Key relationships :

- O_2-He flow rate ↑ Power ↓
- Power ↑ Temperature ↑
- Distance ↑ Temperature ↓



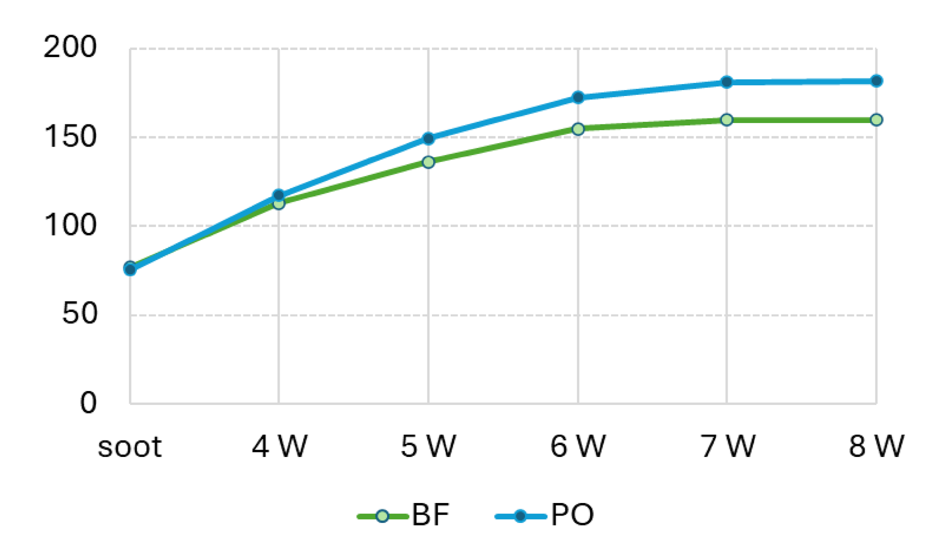
Plasma jet

Identification of emission species

A separate study on monitoring gaseous emission from the AO jet itself and from the cleaning of contaminants was conducted. O_3, CO_2 (and CO) were detected at very low level below 3 ppm. In preliminary study, gas phase FTIR is applied as a fast and useful technique to detect the emission product.

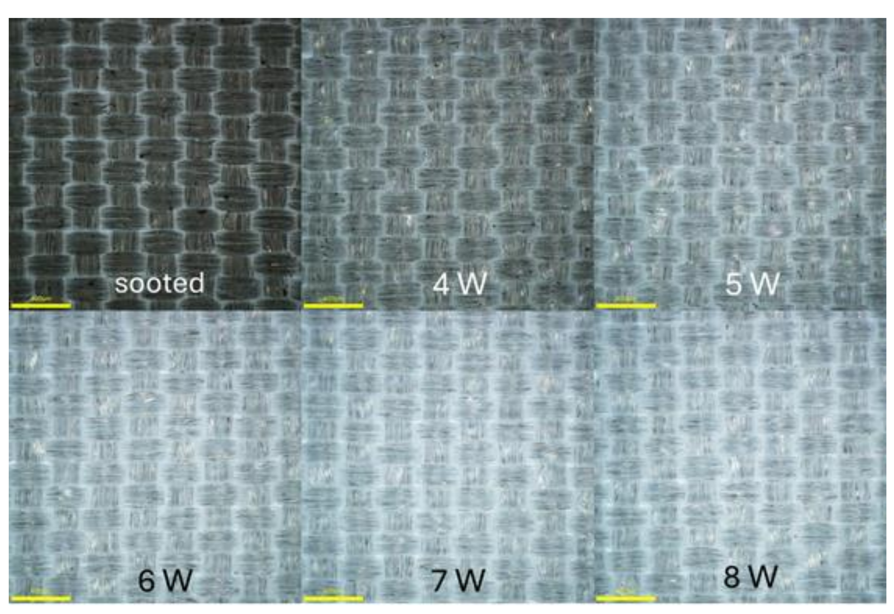
Visual assessment

Brightness value – RGB image from OM



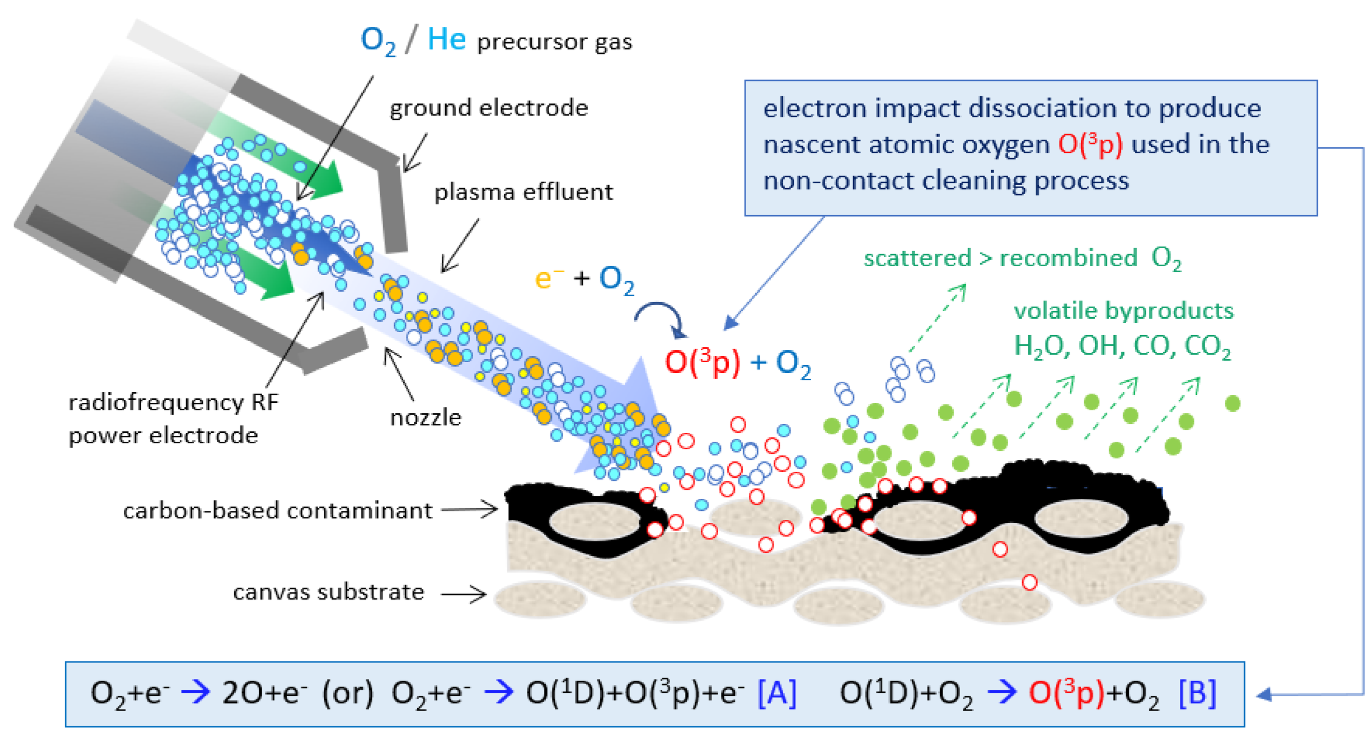
Since soot particles are black in nature, the amount of soot can be quantified roughly by the brightness of RGB images from OM. RGB pixels are calculated to brightness values using the formula V=(R+G+B)/3 by imageJ. Both average values (blue bars) of bright field (BF) and polarized light (PO) increase at higher power.

Optical Microscope (OM)



- Significant colour change after cleaning at all testing condition.
- No visible alternation of the shape and morphology of silk after treatment even at the highest dose.

AO cleaning mechanism



Spectrocolorimetry

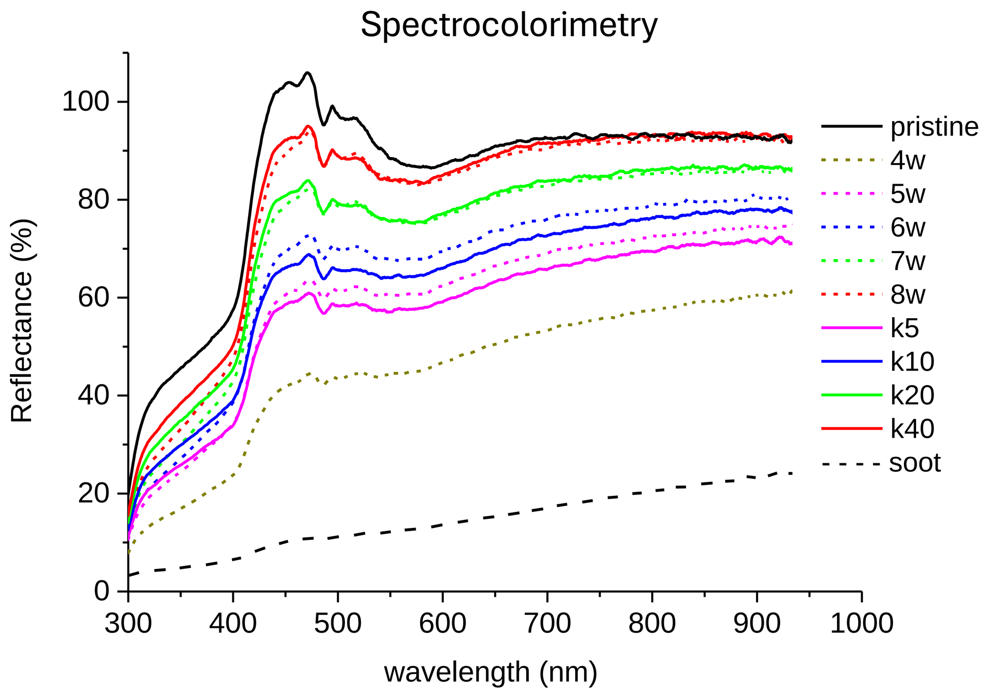


Table 1. Cleaning efficiency based on ΔE_{00}

Test sample	Dose	ΔL	efficiency%	ΔE	efficiency%
1.exposure time	sooted	41.67	/	31.76	/
	7.5 min	13.10	68.56	8.98	71.73
	15 min	10.97	73.68	7.54	76.26
	30 min	3.78	90.94	2.48	92.19
	60 min	-0.02	100.04	0.96	96.98
2.power	sooted	51.78	/	41.68	/
	4 W	21.37	58.72	15.56	62.67
	5 W	11.21	78.35	8.13	80.49
	6 W	7.97	84.60	6.05	85.48
	7 W	3.94	92.40	3.54	91.51
	8 W	0.05	99.90	2.71	93.50

*Reference colour = pristine silk

- Averages and colour differences ΔE_{00} (CIE00 formula) were calculated for sooted sample and after AO cleaning.
- *ΔE* decreases with the increase of exposure time and power, indicated more soot is removed at higher dose. The minimum *ΔE* is 0.96 at the longest exposure time (k=40) at 6w. Such small difference is not perceivable.
- To quantify the cleaning efficiency, a way is to calculate the ratio of colour change after and before cleaning using the formula: **$(1-\Delta E/\Delta E_{soot}) \times 100$ %**, when using pristine as reference colour (see in Table1).
- Cleaning efficiency increases from 63% up to 97% at k5 at 4 w and k40 at 6 w, respectively. At 6 w, the efficiency increased by 25% from k5 to k40 (7 to 60 mins), while it increased by 30% from 4 to 8 w with same duration.

Thermal analysis

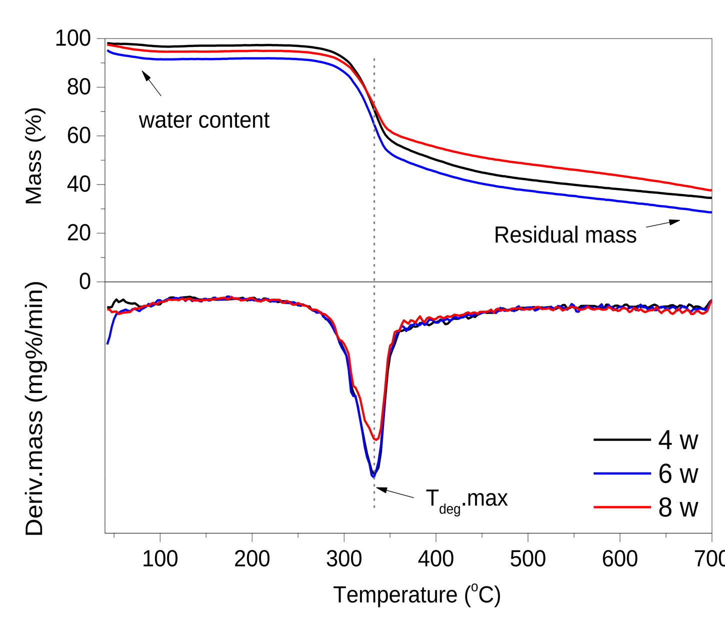


Table 2. TGA results of AO cleaned samples

Sample/mass	T_{deg}	T_{onset}	Water %	Residual %
pristine	336.6	306.9	4.0	24.1
4w	332.4	305.2	1.4	34.5
6w	330.3	304.9	4.5	28.6
8w	338.2	304.0	3.0	37.7
k10	333.8	305.8	1.0	34.5
k20	338.3	307.1	1.0	34.6
k40	335.5	306.0	1.1	34.9
Average	335.0	305.7	2.29	32.69

- TGA was applied to investigate if there is a change of thermal degradation property after AO treatment on pristine silk(without soot).
- The average thermal degradation temperature is found at around 335.0°C with onset point at 305.7 °C. There is no clear relationship with the exposure time or power.
- A decrease in water content after cleaning except for 6w sample. [*It has been reported that the water content is highly related to the RH condition before the analysis. And it is involved in the glass transition process – amorphous phase to helical or beta-sheet crystal.*]
- The residual mass increases after any cleaning treatment from 24% up to 38%.
- Other techniques e.g. DMA and DSC will be performed to investigate the physio-chemical properties with more information.

** All the cleaning was accomplished on a programmed platform moving at a selected speed and path. The cleaning time may vary if use different script.*

Conclusion and project perspective

The results indicate that soot can be safely removed from undyed silk fabric with AO under controlled parameters. The relationship of cleaning effect and exposure time (and power) were investigated, and the optimized working conditions did not cause visual or morphological alternation to the silk substrate. Thermal analysis shows there is no clear shift in thermal degradation temperature. Other results including FTIR, SEM, and mechanical test will be discussed in InArt postprint. Benchmarking other cleaning method to be presented separately as well. This research is a part of the EU funded MOXY project as well as Plasmart.

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MOXY.project

Green Atmospheric Plasma-Generated Monoatomic Oxygen Technology for Restoration of Works of Art

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