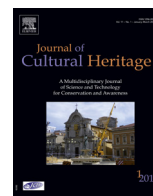




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Original article

## Fly-ash contribution to Nd:YAG laser yellowing and its mitigation using UV-B light

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### ABSTRACT

Nd:YAG Q-Switched laser cleaning of soiled stone at 1064 nm can sometimes result in yellower appearances than other conventional cleaning techniques. The yellowing effect is known to be at least partially due to the laser beam interaction with soiling matter, especially with infra-red absorbing components such as iron oxides, carbonaceous particles or fly-ash. In this study, pure plaster plates were artificially soiled with natural black crust and fly-ash powders. The morphology and crystalline structure of the soiling materials was characterized by scanning electron microscopy (SEM) and X-ray diffraction (XRD). Soiled plaster plates were then treated by Nd:YAG QS laser light at 1064 nm. The laser irradiation led to the appearance of a yellow tone in all cases, highlighting the fly-ash contribution to the yellowing effect. Afterwards, the irradiated plaster plates were exposed to UV-B light (313 nm) for a few dozen hours using an accelerated UV ageing chamber. This treatment resulted in a significant attenuation of the yellow colour for all the samples. Colour measurements revealed that this photo-bleaching effect was essentially due to brightness L\* and saturation C\* variations and not to hue h\* variation towards less yellow shades. As hue variation is often related to chemical transformations of chromophores, this result tends to indicate that the yellowing reduction was mainly due to scattering effects.

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### 1. Introduction

On stone and associated materials, Nd:YAG Q-Switched laser cleaning at 1064 nm can sometimes result in yellower appearances than other conventional cleaning techniques such as nebulization, chemical cleaning or microsanding [1]. The yellow colour can originate from different contributions amongst which an unveiling of pre-existing coloured layers hidden by black gypsum crusts or the presence of nano-sized residues at the stone surface after irradiation [2].

The nano-sized residues are generated by the laser beam interaction with the surface materials to be eliminated, especially infra-red absorbing components such as iron compounds [3–5] and carbonaceous particles [6,7] the interactions of which with the

laser being the most studied to date. The nature of the residues has been investigated through the irradiation of model crusts or artificially soiled samples containing hematite, lamp black or both components mixed with synthetic gypsum. Regarding the samples containing hematite, the residues analysed were found to be iron-containing nanostructures [5]. As for samples containing only carbon, the nature of the yellow residues has not yet been determined.

Other compounds widely present in black crust and much less studied are fly-ashes, mainly originating from coal or fuel combustion plants [8–10]. As fly-ashes contain iron oxides and carbonaceous particles in various proportions, they are also potentially infra-red absorbing compounds and thus may contribute to the yellowing effect. However, no studies have examined this possible contribution yet.

Parallel to the study of the origins of yellowing, attempts to get rid of this undesirable side effect have been performed, including poulticing tests [11] and use of UV radiation. In particular, the use of the Nd:YAG laser third harmonic (355 nm) in combination with the

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**Table 1**  
Compounds employed to prepare artificially soiled samples.

Soiling compounds	Provenance	Colour
Coal fly-ash	Gardanne, Surschiste company, France	Light grey
Fuel fly-ash	Porcheville, thermal power plant, France	Dark grey
Natural Black crust	Saint-Denis basilica, France	Dark grey

first harmonic (1064 nm) has shown very promising results [12,13]. Indeed black crusts can be removed efficiently without any noticeable discoloration by using the two wavelengths in variable fluence ratios [14]. A study on the effects of UV radiation has also been performed with a QUV weather chamber using fluorescent UV-B 313 nm lamps [7]. This work has demonstrated that the yellow tone observed on lamp black soiled samples cleaned by Nd:YAG (1064 nm) laser is greatly reduced after exposure of the samples to UV-B radiation. No experiments on natural black crust or other types of soiled samples using light emitted by UV lamps have been performed to date.

## 2. Research aims

The main purposes of this work are first, to study the possible contribution of both coal and fuel fly-ashes to the laser induced yellowing effect through the irradiation of fly-ash soiled technical samples and secondly, to study the effect of UV-B radiation on the colour of fly-ashes and black crust soiled technical samples cleaned by Nd:YAG laser.

## 3. Materials and methods

### 3.1. Sample preparation and characterization

Artificially soiled gypsum plates were prepared following the same procedure as that described by De Oliveira et al. [7] but using fly-ash and a natural black crust instead of lamp black mixtures. A very pure calcium sulphate hemihydrate (ALDRICH, 97%) was chosen to avoid the presence of any impurities, especially iron, which could interact with the laser beam. Three different soiling materials were prepared, using fly-ash originating either from coal or fuel combustion plants and natural black crust collected on the walls and vaults of a limestone arch, located on a terrace at the top of the west front of the Saint-Denis basilica. The black crust was ground in an agate mortar (see Table 1). The different dry compounds were gently rubbed with a gloved finger onto the surface of the gypsum plates, thus forming a very thin soiling layer.

Fuel fly-ash and black crust chemical compositions were already determined in previous studies [15,16]. The chemical composition of coal fly-ash is provided in the product technical data sheet [17] and the results are summarized in Table 2.

Soiling compounds morphology were characterised using a digital microscope (OM) 3D VHX-5000 from KEYENCE Corporation and a JEOL JSM-5600 scanning electron microscope (SEM) operating at 20 kV coupled with a EDX spectrometer (OXFORD, Pentafet Link

**Table 2**  
Soiling materials chemical composition [15–17].

Elements (wt%)	Si	Al	Fe	Ca	S	Ti	K	P	Na	
Coal fly-ash	23–26	11–13	6	1.8	0.1–1	0.6	1–3	0.1	0.4	
Fuel fly-ash	3.7	1.4	3.5	1.26	6.42	0.05	0.06	0.43	1.46	
Black crust	N.D.	0.81	0.73	22.1	16.8		0.25	0.05	0.16	
Elements (wt%)	C	V	Ni	N	Cd	Zn	Pb	Co	Cu	Cl
Coal fly-ash	2–7									0.04
Fuel fly-ash	61.13	4.66	1.28	0.85	0.17	0.08	0.05	0.03	0.02	
Black crust	2.96			<0.01						

6587) for chemical composition analysis. Fly-ash powders were put on carbon tabs before being metallised with carbon for SEM observation. As for natural black crust, a small fragment was metallised with gold for a cross-section observation.

X-ray diffraction (XRD) was performed on fly-ash powders and natural black crust crushed into powder using a Panalytical X'Pert MPD – Bruker D8 diffractometer fitted with a copper anode. Experimental conditions: 5–80° 2θ, total scan: 40 min.

### 3.2. Laser cleaning

All the samples were irradiated with a Nd:YAG Q-switched laser from Thomson BM Industries. The irradiation conditions were similar to those used for on-site cleaning applications. The laser, operating at a wavelength of 1064 nm, produced discrete pulses of laser energy up to 0.4 J with a pulse length of 15 ns. The pulse was delivered using an articulated mirrored arm and a hand-piece equipped with a 70 cm focal length convergent lens. The fluence used slowly increased from 0.2 to 0.6 J/cm<sup>2</sup> during the cleaning process. The pulse frequency was 10 Hz and the sample was irradiated at a rate of one minute per cm<sup>2</sup>. The surface of the sample was sprayed with water prior to irradiation.

### 3.3. UV-B (313 nm) exposure

After laser cleaning, the plates were irradiated by UV-B using an accelerated UV ageing chamber (QUV LAB Product). The temperature was maintained constant at 55 °C and one unsoiled pure gypsum plate was exposed as reference. The exposure was performed for 69.4 hours at an irradiance of 1.34 W/m<sup>2</sup> and attained a cumulative fluence of 33.3 J/cm<sup>2</sup>. The extremity of each sample was covered with aluminium foil prior to the UV-B exposure in order to keep a reference area safe from UV-B exposure.

### 3.4. Colour measurement

Colour measurements were carried out with a Hunterlab MiniScan XE Plus System spectro-colorimeter using a standard D65 illuminant and a 8 mm diameter beam of diffuse light with a 10° viewing angle geometry. The colour-systems used were CIE L\*a\*b\* and CIE L\*C\*h\* where L\* represents brightness and a\* (red-green) and b\* (yellow-blue) are the chromatic coordinates. The attributes of chroma C\* (saturation or colour purity) and hue h\* (colour) were obtained from the equations:

$$C^* = \sqrt{a^{*2} + b^{*2}} \quad (1)$$

$$h^* = \left( \tan^{-1} \left[ \frac{b^*}{a^*} \right] \right) \times \frac{180}{\pi} \quad (2)$$

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