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The Effect of Ozone and Hydrogen Peroxide Bleaching on the Copper Number of Paper

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This preliminary study compared the oxidative damage to paper inflicted by ozone to that caused by another oxidative process commonly justified for use in conservation, bleaching. Paper samples were exposed to ozone at a concentration and duration comparable to that used by disaster remediation services for odour removal, and to conditions exceeding normal ozone treatment levels. Another set of samples was bleached with hydrogen peroxide. The degree of oxidation was characterized by determining the copper number of the paper before and after treatment. The results suggest that the degree of oxidation of cellulose produced by ozone at levels used for odour removal is comparable to that caused by mild oxidizing bleach.

Cette étude préliminaire a comparé les dommages causés au papier par l'oxydation due à l'exposition à l'ozone, à ceux engendrés par un autre traitement d'oxydation communément utilisé en restauration, le blanchiment. Des échantillons de papier ont subi deux types d'exposition à l'ozone : à une concentration et durée d'exposition comparables à celles utilisées pour le nettoyage désodorisant après sinistre, et à des conditions excédant les niveaux standards de traitement à l'ozone. Un second groupe d'échantillons a été blanchi au peroxyde d'hydrogène. Le degré d'oxydation a été défini en évaluant l'indice de cuivre du papier mesuré avant et après le traitement. Les résultats suggèrent que le degré d'oxydation de la cellulose produit par l'exposition à des niveaux d'ozone utilisés pour des traitements désodorisants est comparable à celui engendré par un blanchiment doux.

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Introduction

Conservation treatment of artifacts on paper is most often performed in response to deterioration of a visual or compositional nature; however, treatment may also be required to remove strong or unpleasant odour permeating the object. This is often the case after an object has been in a disaster situation such as a flood or fire, the odour being caused by fungal growth instigated by high relative humidity or dampness, or by deposits of particles of soot or ash carrying with them a strong smoke smell. Objects stored for long periods of time in damp conditions or in close proximity to cigarette smoke may also suffer from undesirable odours.

When a conservator is called upon to treat such artifacts, washing might be the first course of action considered; however, many artifacts may be unsuitable for these treatments because of media instability or structural complexity. Books, for example, are not easily subjected to washing, since the disbinding and reassembly are time consuming and highly interventive. When a flood or fire occurs in an archive, library or museum, strong residual odour may affect large numbers of objects, many of which may not be otherwise physically damaged. In such cases, odour removal through washing might not only be considered overly interventive but would also be extremely costly.

Exposure of an object to ozone (O₃) is a treatment that is known to eliminate odour effectively and with relative ease.¹ The treatment is currently in use by disaster remediation services to eliminate odour from objects that have been damaged by fire or water. The most common method, as described by remediation service companies,^{2,3} is to place the object to be treated in a closed chamber or room with an ozone generator, turn on the generator and leave it running for a period of hours or days. Restoration companies often use a mobile ozone generator unit either at the company's premises

or at the incident site. They typically begin by running the generator for one night, a period of approximately 16 hours. The next morning the generator is shut off, the chamber is aired out for several hours, and the articles are checked by an informal "smell test" to see if the odour has been eliminated. If odour still remains, the procedure is repeated for another night. The duration of the treatment depends on the size of the space and the number of objects.² On average, a treatment takes two overnight exposures but the duration may range from one night's exposure to seven nights.^{2,3}

The typical commercial generator produces ozone via a silent electrical discharge that splits double bonded oxygen (O₂) molecules from the ambient air, allowing the single oxygen molecules to bond with other O₂ molecules, creating O₃.⁴ The ozone molecules are unstable and prone to reacting with surrounding molecules; as they react with the volatile organic compounds (VOCs) that cause smoke odour, the compounds are oxidized and effectively deactivated.⁵ Ozone is also a potent germicide which is effective in killing some types of bacteria, making it an efficient method of removing musty odours caused by mould and fungal growth.⁶

The conservation field, however, is generally against the use of ozone for odour removal because oxidation is one of the key chemical reactions causing deterioration in most artifact materials.^{5,7} As a powerful oxidizing agent, ozone at various levels of exposure has been proven to cause fading of organic colourants in paint pigments and dyes,⁸ and deterioration in textile fibres⁹ and rubber.¹⁰ In cellulose, oxidation accelerates deterioration in several ways, including lowering the degree of polymerization by breaking cellulose chains, and also by increasing acidity, which in turn propagates deterioration by acid hydrolysis.^{11,12}

Much of the published research on the effects of ozone exposure on artifacts focuses on environmental ozone, at a concentration and duration which reflects pollution levels of the gas affecting museum collections in urban areas. The air pollution of regions like California, which is high in atmospheric ozone, can impact the interior air conditions of museums and galleries, and has been proven to have adverse effects on materials in the collections.¹³ However, these results cannot be reliably extrapolated to indicate the effects of higher concentrations or shorter durations.¹⁴ Research concerning the amount of deterioration caused by ozone to cellulose in particular is scarce. It is uncertain exactly how much damage may be caused by a strong but brief treatment comparable to that used by commercial restoration businesses.

Although use of ozone on artifacts for odour removal is controversial among conservators, another process that causes oxidative deterioration of cellulose is often considered justifiable despite clear evidence that it causes degradation of cellulose.¹⁵ Bleaching, either by chemical or natural means (sunlight) is a treatment regularly used by conservators to reduce local stains or overall darkening on paper objects. Several chemical bleaching processes are oxidative in nature, including one of the most common, hydrogen peroxide. Hydrogen peroxide is recommended in the conservation literature as an appropriate treatment option for stain reduction.¹⁶ Its oxidative action can be controlled sufficiently to permit safe and gentle reduction of stubborn localized stains.¹⁷

If oxidative bleaching treatment can sometimes be justified in paper conservation practice, then perhaps so may other oxidative treatments, if the level of damage is similarly low. Comparing the oxidative damage caused by ozone exposure, at levels reflecting commercial ozone treatment for the removal of odour, to that caused by conservation bleaching with hydrogen peroxide should demonstrate whether ozone treatment results in greater, unacceptable levels of degradation. This preliminary study was designed to test this hypothesis.¹⁸

Methods

Ozone Treatments

Low Dose

Ozone treatment representative of that used for odour removal was carried out at a restoration company facility following procedures used by the industry. Maurizio Scatoza, on behalf of Munters Disaster Restoration (now Polygon Restoration) in Mississauga, Ontario, agreed to expose the samples with Munters' equipment. Three samples of Whatman filter paper no. 40, each measuring 23 x 28.4 cm and weighing 6.2 g were sent from Kingston, Ontario to Mississauga by courier, along with an Omega OM-EL-USB data logger for monitoring the environment during treatment. Grade 40 Whatman filter paper was selected because it is slightly more acidic and therefore better shows any deterioration. The samples and data logger were placed in a small interior room measuring approximately

1.5 m x 1.5 m x 2.5 m along with a Total Zone model TZ-2 ozone generator with an output of 3000 milligrams per hour (or approximately 50 ppm). Samples were exposed to ozone in a dedicated session, without any other materials present, for 63.5 hours. Temperature during treatment was 22–24°C while relative humidity rose from 17% to 30%, then fell back to 17% again. Following treatment, the samples and data logger were then returned to Kingston by courier. Samples were stored in polypropylene sleeves inside manila envelopes until further testing.

High Dose

A second set of samples was exposed to ozone at a significantly higher concentration – approximately 1% by weight or 10,000 ppm – using a Hankin Ozotec Ozone Generator. Three samples of Whatman filter paper no. 40, each measuring approximately 39 x 28.5 cm and weighing on average 10 g, were cut in half and placed together in a stainless steel pot in a fume hood. Black Norprene chemical tubing connected to the ozone generator was inserted into the pot, with the lid on ajar over it. The generator settings were: power control dial set to 75% (approximately 3 volts), 12 PSI, 1.72–1.91 standard litres per minute (SLPM). The generator was run for six hours on each of two consecutive days for a total exposure time of 12 hours. The environment in the fume hood during exposure, as recorded by an Omega OM-EL-USB data logger, was 23–25°C and 14–17% relative humidity. After ozone exposure, samples were stored in polypropylene sleeves inside manila envelopes until further testing.

Hydrogen Peroxide Bleaching

A third set of paper samples was bleached using stabilized hydrogen peroxide. Three samples of Whatman filter paper no. 40, each measuring approximately 39 x 28.5 cm and weighing approximately 10 g, were bleached with hydrogen peroxide in a manner simulating a conservation bleaching treatment.¹⁶ Each sample was individually deacidified for 20 minutes in a solution of distilled water brought to pH 9 using calcium hydroxide, followed by immersion in a fresh solution of 2% hydrogen peroxide in distilled water for 15 minutes. The sample was then washed for 20 minutes in tap water and then deacidified again in distilled water brought to pH 9 using calcium hydroxide. After this treatment, all samples were then left to dry on blotters and then stored in polypropylene sleeves.

Characterization of Visual and Tactile Changes

Colour, brightness, surface texture, flexibility and strength of paper samples were noted before and after treatments. Colour change of the paper samples exposed to ozone was also determined using a Minolta Chromo Meter CR-300 before and after treatment. The measurements were recorded in L*a*b* colourspace using a D65 light source. Measurements were performed in four different locations on each sample and averaged to give values for unexposed paper and for each ozone treatment. Total colour change, delta E ('76), was calculated against the values for unexposed paper.

Table I: Results of Colorimeter Testing on Whatman Filter Paper no. 40, Before and After Ozone Exposure (mean and standard deviation).

Treatment	L*	a*	b*	$\Delta E(^{\circ}76)$
Control (no treatment)	94.88 \pm 0.06	-0.28 \pm 0.02	+0.75 \pm 0.04	
Low Dose Ozone (~50 ppm; 63.5 hours)	97.26 \pm 0.07	-0.20 \pm 0.03	+1.94 \pm 0.11	2.66
High Dose Ozone (~10, 000 ppm; 12 hours)	94.49 \pm 0.11	-0.38 \pm 0.02	+2.01 \pm 0.11	1.32

Copper Number Testing

The relative degree of oxidation caused by ozone and hydrogen peroxide bleaching treatments of Whatman filter paper no. 40 was assessed using TAPPI standard test method T430 om-94 for the Copper Number of Pulp, Paper and Paperboard.^{18,19} This test, which determines the grams of metallic copper (as Cu₂O) resulting from the reduction of CuSO₄ by 100 g of paper fibres, was chosen for the analysis because it is sensitive enough to detect minute changes in the molecular structure of cellulose. Copper number has also been shown to exhibit a linear relationship to the wet zero-span tensile index, indicative of fibre strength.²⁰ Three tests per sample were completed on samples from each treatment, and the results were used to calculate copper number as follows:

Copper number = $[6.36 (V-B) N]/W$, where

V = amount of potassium permanganate needed to titrate the filtrate from each sample (ml)

B = amount of potassium permanganate needed to titrate a blank filtrate: 0.30 ml

N = normality of potassium permanganate solution: 0.05

W = moisture-free weight of test specimens measured following TAPPI standard test method T550 om-08²¹:

1.446 g, (moisture content of 3.6%)

Results and Discussion

Visual and Tactile Observations

Changes in colour and brightness were observed and measured (Table I) in the samples exposed to both levels of ozone. Exposure to the low ozone dose resulted in just noticeable yellowing (increase in b*) and brightening (increase in L*) but only when viewed next to the control, which is consistent with a delta E ($^{\circ}76$) of 2.66, slightly over the just visible threshold of 2. The paper exposed to high ozone exhibited clear yellowing along the edges (approximately 1 mm border). Colorimeter measurements indicated yellowing similar to the low dose samples (increase in b*) and a slight darkening in comparison to the control (decrease in L*). The delta E ($^{\circ}76$) value of 1.32 suggests that visible change was limited to the borders of the samples.

Changes in surface texture, flexibility and strength were noted while handling the Whatman

filter paper samples before and after ozone and bleaching treatment. Before treatment, the samples had a rough texture, almost chalky surface, but very good flexibility and strength. After low-dose ozone exposure no changes in texture or flexibility were observed. The hydrogen peroxide treatment similarly resulted in no change. After the high dose of exposure to ozone, however, marked differences were apparent. The paper had become extremely brittle and its surface felt very chalky. A 45° fold resulted in the paper breaking, as did the pressure of just a thumb on the surface. While cutting with scissors to obtain pieces small enough for the copper number testing, the high-dose paper strips frequently broke.

Copper Number

Copper number test results are presented in Figure 1 and Table II. The difference between the results for the control, hydrogen peroxide bleached and low dose ozone samples was small: the copper number of low-dose ozone samples was only slightly higher than that of the bleached samples which was slightly higher than that of the controls. All values are lower than copper number values interpreted by Annis and Reagan²² as indicative of no significant change in chemical structure of cellulose in bleached cotton fibres. High-dose ozone samples, however, exhibited a copper number over 5 times higher, suggesting significant change in molecular structure which is consistent with the observed increase in brittleness.

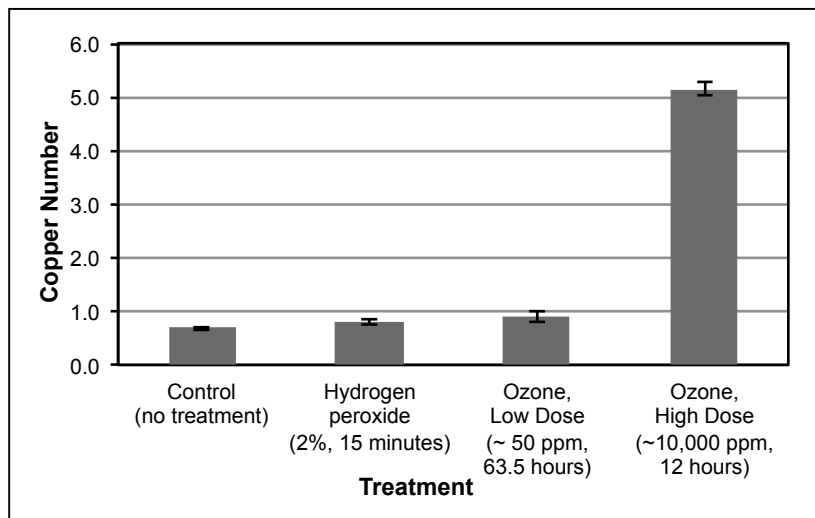


Figure 1. Copper Number (mean and standard deviation) of Whatman filter paper no. 40 treated with hydrogen peroxide and ozone.

Table II: Copper Number of Whatman Filter Paper no. 40 (average of 3 samples and standard deviation).

Treatment	Copper Number (g copper / g paper)
Control (no treatment)	0.7 ± 0.1
Hydrogen peroxide (2%, 15 minutes)	0.8 ± 0.1
Low Dose Ozone (~50 ppm; 63.5 hours)	0.9 ± 0.1
High Dose Ozone (~10,000 ppm; 12 hours)	5.1 ± 0.1

Conclusions

This preliminary study has demonstrated that the exposure of one type of paper to ozone at low concentration for a duration comparable to that used by commercial restoration companies to eliminate odour causes damage comparable to that of the oxidation effects of a mild bleach, hydrogen peroxide, while affecting the paper's colour and handling qualities very little. A much higher concentration of ozone, by contrast, resulted in greater chemical damage, as indicated by a large increase in copper number and noticeable loss of strength. The high dose effects illustrate the potential for serious damage by ozone if exposure is poorly controlled. More research would be needed to determine acceptable yet effective treatment dose if ozone were to be considered an appropriate conservation treatment option for removal of undesirable odours from paper artifacts.

If properly controlled, therefore, exposure to ozone for odour removal may not cause more immediate damage to paper than hydrogen peroxide bleaching treatment. More rigorous testing, equivalent to that used to study bleaching treatments for paper,^{15,23} might identify treatment protocols that adequately manage the risk of oxidative damage to paper due to ozone while eliminating odour. In addition, since ozone is toxic to humans in the concentrations required for these treatments,^{24,25} an alternative might be to consider hydroxyl technology, a treatment recently introduced by the restoration industry for odour removal that is safer for humans but that requires longer exposure to be effective.²⁶ Since, unlike bleaching treatments, local application is not possible with the current technology, research would also need to take into consideration the possible damage to components other than paper and to long-term effects on paper aging as has been studied for various bleaching treatments^{15,23,27} and for exposure to light and near ultraviolet.²⁸ Such research might ultimately provide conservators with new options for odour removal for some artifacts, whether done in collaboration with the restoration industry or adapted for the conservation laboratory.

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Materials

Copper number test (hydrogen peroxide, H₂O₂; copper sulfate, CuSO₄; sodium carbonate, Na₂CO₃; sodium bicarbonate, NaHCO₃; sodium molybdate, Na₂MoO₄; phosphoric acid, H₃PO₄; sulfuric acid, H₂SO₄; potassium permanganate, 0.05N KMnO₄; Whatman Filter Paper no. 44): Fisher Scientific Company, 112 Colonnade Road, Ottawa, Ontario K2E 7L6, Canada; Tel.: 1-800-234-7437; <www.fishersci.ca>

Hankin Ozotec Ozone Generator, Type S, Model 3, 50Hz: Hankin Ozone Systems Limited, 690 Progress Avenue, Unit 12, Scarborough, Ontario M1H 3A6, Canada; Tel.: 416-439-7860; <www.hankinozone.com/contact.html>

Norprene™ chemical tubing: Fisher Scientific

Ozone exposure chamber (stainless steel cooking pot, approximately 1 cubic foot): hardware stores

Total Zone model TZ-2 Ozone Generator: International Ozone Technologies Group Inc., 1100 "J" S. W. 10th. Street, Delray Beach, Florida 33444, USA; Tel.: 1-877-406-9663; <www.internationalozone.com/OZONE%20GENERATORS.htm>

Whatman Filter Paper no. 40: Fisher Scientific

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