

# Tooth Bleaching Using Low Concentrations of Hydrogen Peroxide in the Presence of a Nonthermal Plasma Jet

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**Abstract**—To investigate the tooth bleaching effect of a low concentration hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) assisted by a nonthermal plasma, extracted human teeth were treated by a nonthermal plasma in the presence of varying concentrations of hydrogen peroxide gel. The results showed that combining plasma treatment with a low concentration hydrogen peroxide (6%) achieved a superior bleaching effect compared with a high concentration of hydrogen peroxide (35%) in the absence of a plasma. Furthermore, the bleaching efficacy did not significantly increase with increasing  $\text{H}_2\text{O}_2$  concentration above 6% in the presence of the plasma. Compared with the control group (hydrogen peroxide without plasma), the plasma generated two to three times as many  $\cdot\text{OH}$  radicals, which are believed to be one of the key reactive species that contribute to the bleaching effect.

**Index Terms**—Hydrogen peroxide, hydroxyl radical, nonthermal plasma, tooth bleaching.

## I. INTRODUCTION

**H**YDROGEN peroxide ( $\text{H}_2\text{O}_2$ ) has been widely used as the active ingredient in most tooth bleaching products. It serves as a source of free radicals, which break up the long-chain organic pigment molecules (that cause the coloration) into short-chain compounds, leading to the bleaching of the

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tooth surface [1]. The concentration of free radicals increases with the use of UV light or laser radiation [2]. Cosmetically acceptable bleaching effects are typically achieved by employing a high concentration (30%–35%) of  $\text{H}_2\text{O}_2$  in combination with UV light/laser activation [2], [3] in a dental office setting. However, safety issues related to the side effects stemming from the use of high concentrations of  $\text{H}_2\text{O}_2$  have been also reported, including oral soft tissue irritation [4], [5], severe eye and respiratory tract irritation or burns [5]–[8], and compromising cellular safety [9]. Furthermore,  $\text{H}_2\text{O}_2$  in such high concentrations is an unstable species that easily decomposes during transport and storage. As a consequence, effective bleaching treatments using low concentrations of  $\text{H}_2\text{O}_2$  are desirable. Many over-the-counter tooth bleaching products contain low concentrations of  $\text{H}_2\text{O}_2$ . However, they require much longer application times compared to the in-office high-concentration treatments.

Nonthermal plasmas combine a low gas temperature with high-energy electrons, which produce copious amounts of charged particles and reactive radicals in the plasma. Nonthermal plasmas have been reported to achieve a remarkable tooth bleaching effect, either as a stand-alone tool or in the presence of high concentrations of  $\text{H}_2\text{O}_2$  [10]–[13].

This paper is aimed at a systematic evaluation of the tooth bleaching by  $\text{H}_2\text{O}_2$  in different concentrations in the presence of a nonthermal plasma. We wanted to assess whether the combination of a nonthermal plasma and a low concentration of hydrogen peroxide (as low as 6%) can achieve effective tooth color alteration when compared with the conventional in-office tooth bleaching method, where a high concentration of hydrogen peroxide (35%) is used. Electron spin resonance (ESR) spin-trapping spectroscopy was used to analyze the generation of  $\cdot\text{OH}$  free radicals.

## II. MATERIALS AND METHODS

### A. Plasma Device

The plasma device used in this study (see Fig. 1) consists of two coaxial copper tubings as electrodes, which are separated by a dielectric tubing with a wall thickness of around 0.3 mm. The surfaces of the copper electrodes are separated by  $\sim 0.5$  mm. The inner electrode is powered by a dc high-voltage power supply (negatively biased), whereas the outer electrode is grounded for safety considerations. The nozzle opening of the

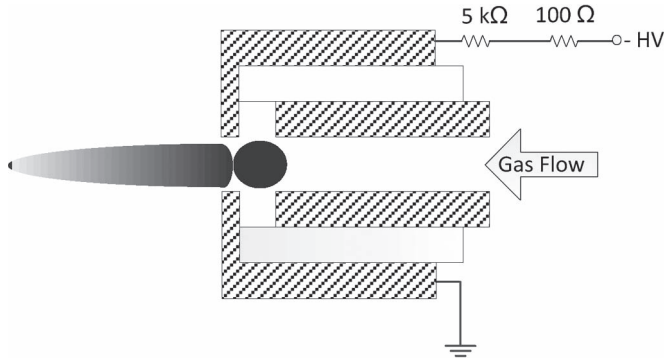


Fig. 1. Schematic of the plasma jet device.

plasma device has a diameter of around 0.8 mm. Further details of the plasma device and the electrical circuitry can be found in [11]. Dry compressed air from a cylinder was used as the working gas and forced to flow through the inner electrode at a flow rate of 5 standard liters per minute (slm). The sustaining voltage was in the range of 400–600 V, with an operating current of 20–35 mA. The generated plasma plume typically extended to about 10 mm beyond the nozzle of the device.

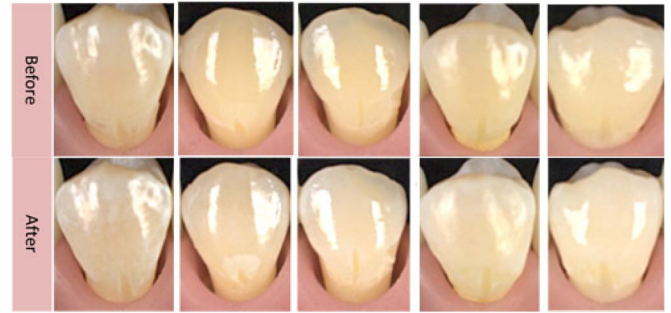
### B. Tooth Bleaching Experiments

Fifty human teeth extracted for orthodontic reasons were selected for our experiments. All teeth with intact crowns were free from caries, cracks, or other defects. The teeth were thoroughly cleaned and stored in a 0.1% thymol solution at 4 °C before use. They were randomly divided into five groups (A–E). Four groups of teeth (groups B–E) were treated by a nonthermal plasma in the presence of varying concentrations of H<sub>2</sub>O<sub>2</sub> gel (6%, 15%, 25%, and 35%, respectively). A control group (group A) contained teeth that only received 35% H<sub>2</sub>O<sub>2</sub> gel, but no plasma treatment. The plasma experiments were carried out at room temperature for 15 min. A layer of H<sub>2</sub>O<sub>2</sub> gel of ~1-mm thickness was applied on the surface of each tooth. In order to ensure an adequate amount of gel on the tooth throughout the treatment period, the gel was replenished at an interval of 30 s.

An Olympus spectrophotometer was used to evaluate the color of the teeth before and after plasma treatments. The color change of each tooth was based on the average of three measurements, as recommended in the Commission Internationale de L'Éclairage (CIE)  $L^*a^*b^*$ , or CIELAB color scale. This system expresses color as a combination of three values, i.e.,  $L^*$ ,  $a^*$ , and  $b^*$ . The  $L^*$  axis ranges from 0 (black) to 100 (white) and is a measure of the lightness (black/white contrast) of a sample. The  $a^*$  plane represents the green(–)/red(+) contrast, whereas the  $b^*$  plane denotes the blue(–)/yellow(+) contrast of the sample. The overall change of the colorimetric value, i.e.,  $\Delta E^*$ , is determined by the following formula [14]:

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}.$$

The amount of hydroxyl radicals ( $\cdot\text{OH}$ ) generated by different concentrations of H<sub>2</sub>O<sub>2</sub> before and after the plasma treatment was measured by using an ESR spin-trapping method. The spin trap reagent DMPO (5,5-dimethyl-1-pyrroline-1-oxide,



(a) 35% 6% H<sub>2</sub>O<sub>2</sub> 15% H<sub>2</sub>O<sub>2</sub> 25% H<sub>2</sub>O<sub>2</sub> 35% H<sub>2</sub>O<sub>2</sub>

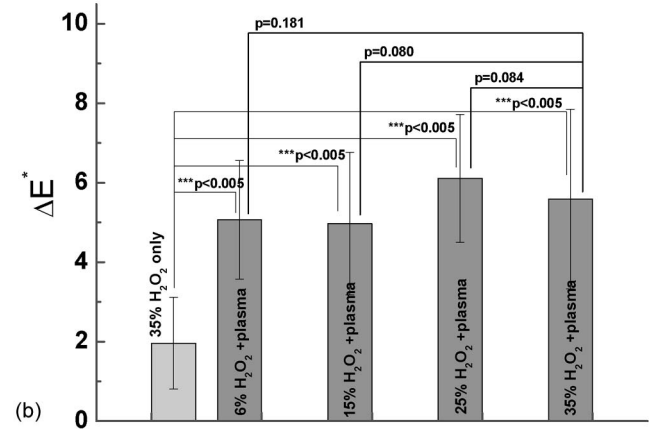


Fig. 2. (a) Photographs of one tooth from each group before and after the plasma treatment. (b) Results of the statistical analysis of the bleaching efficacy for all groups in terms of the overall color changes.

0.3 mol/L, 20  $\mu\text{l}$ ) was used to trap  $\cdot\text{OH}$ . In addition, 900  $\mu\text{l}$  of phosphate buffered saline solution was added into 100  $\mu\text{l}$  of hydrogen peroxide at each concentration. For each plasma treatment group, the plasma was applied at a fixed distance of 10 mm (from the exit nozzle of the device to the surface of the liquid) for 20 s. Samples of the solution (40  $\mu\text{l}$ ) before and after plasma treatment were imbibed by a glass capillary and detected in the resonator cavity of an ESR spectrometer (Bruker ER-200D-SRC) operated at room temperature.

The  $\Delta E^*$  values for various groups and the  $\cdot\text{OH}$  intensities with and without plasma treatment were statistically analyzed using an analysis of variance (ANOVA) test in the Statistical Package for the Social Sciences (IBM Corporation, V14). Differences were considered statistically significant when  $p < 0.05$ .

### III. RESULTS AND DISCUSSION

Fig. 2(a) shows photographs of one representative tooth from each of the five groups before and after the treatment. Visual inspection reveals a significant bleaching effect of the teeth in all plasma treatment groups compared with the control group. Fig. 2(b) plots the  $\Delta E^*$  values of the five groups. In the plasma treatment groups, the overall color change ( $\Delta E^*$ ) was 5–6 units, or about 2.5–3.0 times higher than that in the control group. Statistical analysis showed significant differences between the control group and all plasma treatment groups ( $p < 0.05$ ). On the other hand, no statistically significant

TABLE I  
MEAN VALUES AND STANDARD DEVIATION OF  $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$ , AND  $\Delta E^*$   
THAT DETERMINE THE COLOR FOR TEETH IN ALL FIVE GROUPS

Group	A	B	C	D	E
$\Delta L^*$	0.44±0.74	1.65±1.94	1.81±0.79*	2.42±1.27*	2.44±1.28*
$\Delta a^*$	0.14±0.46	0.02±0.29	0.01±0.34	-0.01±0.36	0.08±0.30
$\Delta b^*$	-1.08±1.82	-4.34±1.73*	-4.58±1.75*	-5.44±1.75*	-4.94±2.08*
$\Delta E^*$	1.96±1.15	5.07±1.50*	4.97±1.79*	6.11±1.61*	5.59±2.26*

differences were found in the bleaching effect among the teeth in the various plasma treatment groups.

In terms of the overall change of the colorimetric values, Tavares *et al.* [1] showed that bleaching occurs mainly by an increase in lightness (higher  $L^*$ ) and a decrease in yellowness (lower  $b^*$ ), and to a lesser extent by a decrease in redness (lower  $a^*$ ). The  $\Delta E^*$  values along with the individual  $\Delta L^*$ ,  $\Delta a^*$ , and  $\Delta b^*$  values are listed in Table I. In the plasma treatment groups, the yellow shades ( $\Delta b^*$ ) decreased by 4–5 units, and the lightness ( $\Delta L^*$ ) increased by 1.5–2.5 units. A more detailed analysis of the results revealed that there were noteworthy differences in the whiteness ( $\Delta L^*$ ) ( $p < 0.05$ ) between the control group and all plasma treatment groups. Significant differences were also found in the yellow shades ( $\Delta b^*$ ) between the control group and all plasma treatment groups. Therefore, the improved values of  $\Delta E^*$  in our study were mainly the result of reduction in the yellow shades ( $\Delta b^*$ ) and an increase in the whiteness ( $\Delta L^*$ ) of the teeth. The red/green contrast ( $\Delta a^*$ ) was not significantly affected by the treatment. This result is consistent with what was reported by Joiner *et al.* [15]: bleaching works most efficiently for teeth with a yellow hue. Joiner also suggested that the alteration in tooth color may be influenced by many factors, such as peroxide diffusion, reaction with organic colored materials, the generation of per-hydroxyl anions ( $\text{HO}_2^-$ ), and an increase in the concentration of hydroxyl radicals. However, the actual mechanism of tooth bleaching by oxidizing materials (such as hydrogen peroxide and carbamide peroxide) is not yet fully understood [16].

$\cdot\text{OH}$  radicals were generated during the plasma treatment and were evaluated via ESR spin-trapping spectroscopy, as described earlier. The direct spin-trapping reaction between DMPO and  $\cdot\text{OH}$  produces the spin adduct DMPO-OH that is characterized by a quartet ESR spectrum with a peak intensity ratio of 1 : 2 : 2 : 1 [11]. Half heights of the second peak of the quartet spectra were chosen to represent DMPO-OH intensities and are plotted in Fig. 3. The DMPO-OH signal intensity due to the plasma treatment increases by a factor of 2–3 compared with the untreated cases. We also found an increase in the hydroxyl radical generation with increasing concentration of  $\text{H}_2\text{O}_2$ .

Fig. 3 aims to show that the presence of a plasma correlates with an increase in the  $\cdot\text{OH}$  concentration, and there is a significant difference in DMPO-OH signal intensities between the case in which only  $\text{H}_2\text{O}_2$  was applied and the case in which both  $\text{H}_2\text{O}_2$  and plasma were applied. The hydroxyl radical concentration significantly increased with the addition of plasma for each concentration of  $\text{H}_2\text{O}_2$ . Meanwhile, a significant difference in DMPO-OH signal intensities was found between the

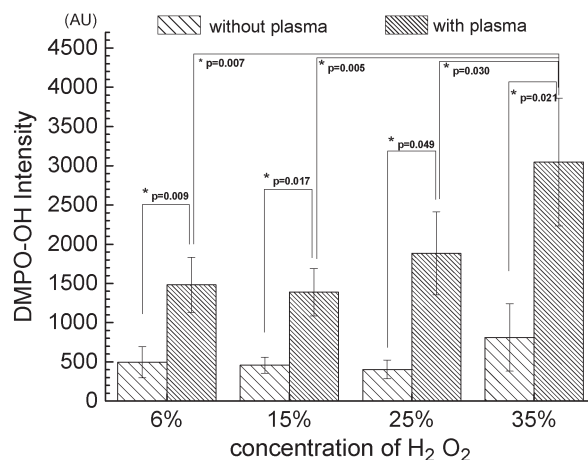


Fig. 3. DMPO-OH signals at different  $\text{H}_2\text{O}_2$  concentrations before and after employing plasma.

6%, 15%, and 25%  $\text{H}_2\text{O}_2$  plasma treated groups and the 35%  $\text{H}_2\text{O}_2$  plasma treated group. The fact that the increase in  $\cdot\text{OH}$  concentration in the 35% group did not, however, result in a better whitening efficacy leads us to believe that  $\cdot\text{OH}$  is not the only radical that contributes to the whitening process.

Lee *et al.* [10] reported that  $\cdot\text{OH}$  was the key reactive species in tooth bleaching. Our observation of an increase in the concentration of hydroxyl radicals with increasing  $\text{H}_2\text{O}_2$  concentration while the tooth bleaching efficacy remained essentially independent of the  $\text{H}_2\text{O}_2$  concentration from 6% to 35% suggests that, while hydroxyl radicals are important, they may not be the only radicals that cause tooth bleaching. We speculate that other species such as atomic oxygen (O), the superoxide anion ( $\text{O}_2^-$ ), and singlet molecular oxygen ( $^1\text{O}_2$ ) must also play a role in the tooth bleaching process. In a different study with the same plasma device, we successfully detected O,  $\text{O}_2^-$ , and  $^1\text{O}_2$  in a plasma–water system [17].  $\text{O}_2^-$  may serve as the precursor of  $\cdot\text{OH}$  and participate in the bleaching process indirectly [17]. O and  $^1\text{O}_2$  may be generated directly in the plasma. The lifetime of O in air is of the order of 10–20  $\mu\text{s}$  [18]. The lifetime of  $^1\text{O}_2$  (in particular  $\text{O}_2(^1\Delta_g)$ ) was calculated to be 72 min [19]. This number is considerably reduced at higher concentrations of oxygen and in the presence of other molecular species to the range of milliseconds in the gaseous phase. Given the flow rate of 5 slm and the geometric dimension of the device, these species are estimated to be delivered at a speed of  $\sim 100$  m/s to the tooth surface. Thus, O can only travel roughly 1–2 mm in an ideal case. Although O may not contribute to the oxidation process on the tooth surface directly, the O atoms may serve as precursors of longer-lived



reactive species that may contribute to the bleaching process. However,  $^1\text{O}_2$  can travel for 10 cm in principle. We suspect that, even in the collisional environment within the air flow channel, it is very probable that a certain amount of  $^1\text{O}_2$  reaches the tooth surface and participates in the bleaching process directly. The interplay caused by the presence of the various radicals, i.e.,  $\cdot\text{OH}$ ,  $\text{O}$ ,  $\text{O}_2^-$ , and  $\text{O}_2(^1\Delta_g)$ , during the bleaching process will be the subject of future investigations.

It is well recognized that enamel dehydration can produce significant shade reduction or a whiter appearance of tooth [14]. Our experiments were designed with this in mind: the amount of bleaching gel on the tooth surface was kept adequate throughout the experiments via replenishment at an interval of 30 s. In an earlier study [12], saline solution was used instead of  $\text{H}_2\text{O}_2$  gel and replenished every 30 s. At an air flow (no plasma) of 5 slm, no significant color change of tooth was observed, therefore excluding dehydration as one of the bleaching factors. In this paper, the viscosity coefficient of the  $\text{H}_2\text{O}_2$  gel is 3.7–4.5 Pa · s, a value that is much higher than that of the saline solution ( $0.72 \times 10^{-3}$  Pa · s). Therefore,  $\text{H}_2\text{O}_2$  gel maintains moist on the tooth surface more effectively, and tooth bleaching due to dehydration is not considered important in this process.

Acidity change on the tooth surface may also influence the tooth color appearance after treatment due to demineralization. When the plasma jet used in this study was applied to 20-ml water without any buffer for 4 min, the pH value dropped from 7.5 to 3.4. This is attributed to the reaction of reactive nitrogen species with water [20]. However, we are not able to evaluate the pH change on the tooth surface at the current stage, making it difficult to assess whether acid pickling significantly contributed to the tooth bleaching.

#### IV. CONCLUSION

In conclusion, we have found that the addition of a non-thermal plasma significantly improves the bleaching efficacy of  $\text{H}_2\text{O}_2$  gel at various concentrations. The bleaching effect is independent of the concentration of  $\text{H}_2\text{O}_2$  between 6% and 35%. This observation implies that 1) there is no advantage of using high concentrations of hydrogen peroxide in conjunction with a plasma in terms of tooth bleaching efficacy and 2) even a low concentration of hydrogen peroxide (6%) in conjunction with a plasma achieves cosmetically satisfactory results. It is noteworthy that one can assume the existence of a “saturation point” in terms of hydrogen peroxide concentration when used in conjunction with a plasma and that this saturation point must occur for very low hydrogen peroxide concentrations, i.e., 6% or even lower. Since the concentration of  $\cdot\text{OH}$  radicals was found to increase with increasing  $\text{H}_2\text{O}_2$  concentration, we conclude that reactive species other than  $\cdot\text{OH}$  radicals must play a role in the tooth bleaching chemistry.

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