

Adding Ferrous Sulphate to Hydrogen Peroxide Tooth Bleaching Gels Reduced the Required Bleaching Time

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Abstract

Objective To investigate the effect of ferrous sulphate (FeSO_4) as a chemical activator to hydrogen peroxide tooth bleaching gels on color change and treatment time. **Methods** One hundred and twenty extracted human upper premolars, hue A or B, were selected. One enamel-dentin specimen was prepared from each tooth. The specimens were divided into twelve groups ($n=10$), based on bleaching product, FeSO_4 and treatment time. Three in-office bleaching products were used in this study: Opalescence[®] Boost[™], PolaOffice[®] and ZOOM![®] Whitespeed[™]. Each product was used per its specific protocol: 1: untreated control (C), 2: FeSO_4 added, using the product's specified treatment time (Fe100 %), 3: FeSO_4 added, using approximately 75 % of the specified time (Fe75 %), 4: FeSO_4 added, using approximately 50 % of the specified time (Fe50 %). The color was measured before and immediately after bleaching, using a VITA Easyshade[®] V spectrophotometer based on the CIE-L*a*b* color system. Data between experimental groups of each product were analyzed using one-way ANOVA and Tukey's or Tamhane's test ($p=0.05$). **Results** Color parameter (ΔL , Δa , Δb and ΔE) evaluation indicated no differences between the C, Fe100 % and Fe75 % of each product. Compared with the C group, only Δb of the ZOOM![®] Whitespeed[™] Fe50 % group was significantly different ($p=0.001$). No differences were found between the Opalescence[®] Boost[™] and PolaOffice[®] C and Fe50 % groups with each material. **Conclusion** Adding FeSO_4 was effective in reducing the treatment time of Opalescence[®] Boost[™], PolaOffice[®] and ZOOM![®] Whitespeed[™] by at least 25 %.

Keywords: Bleaching, Chemical activation, Ferrous sulphate, Hydrogen peroxide, Tooth whitening

Received Date: Aug 14, 2017

Accepted Date: Nov 30, 2017

doi: 10.14456/jdat.2018.11

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Introduction

Whiter teeth are perceived as being associated with health and beauty. Tooth bleaching is a conservative treatment for discolored teeth compared with other restorative treatments, such as porcelain veneers, crowns or composite resin restorations.¹ In-office bleaching procedure is an alternative to home bleaching, especially in the case of mild to moderate discolorations, discoloration of single tooth, lack of patient compliance, or when rapid treatment is desired.^{2,3} In these bleaching procedures, hydrogen peroxide with concentrations varying from 17–50 % is commonly used.^{3,4} The operation time was usually around 30 minutes to 1 hour, depending on the commercial tooth bleaching product used.^{3,5} This long duration of treatment sometimes gave patients discomfort.^{6,7}

Many methods have been proposed to accelerate the dissociation of hydrogen peroxide and formation of free radicals to improve bleaching effectiveness; for example, physical activation method by using electric heating device or light sources such as blue light produced by quartz-tungsten-halogen lamps, plasma arc lamps, light emitting diodes, or LASER.^{3,5} However, it is still inconclusive concerning effectiveness and safety of these methods.^{8,9} Another method to promote dissociation of hydrogen peroxide molecule and free radical formation is using chemical activator, for instance, certain enzymes or transition metal salts, such as Fe, Cu, Cr, or Mn, to act as a catalyst. Many studies demonstrated that adding a metal salt to the bleaching gel improved final outcomes.¹⁰⁻¹⁷ This procedure caused increased free radical formation on tooth surface, resulting in a specific and rapid reaction with local chromophores, improving bleaching effectiveness and reducing quantity of unreacted hydrogen peroxide available for diffusion into tooth structure.¹⁸ The fast reaction may effect the overall time; therefore, the authors questioned that treatment time could be reduced.

Torres *et al.* found that addition of ferrous

sulphate (FeSO_4) or manganese gluconate in 35 % hydrogen peroxide bleaching gel produced a significant reduction of hydrogen peroxide penetration into the pulp chamber and a higher mean color change (ΔE) compared with the control group without a chemical activator.¹⁰ Travassos *et al.* also showed better ΔE results when adding FeSO_4 in 35 % hydrogen peroxide bleaching gel.¹⁷ In addition, Duque *et al.* found a significant increase of approximately 30 % in ΔE in the hydrogen peroxide mixed with FeSO_4 group compared with the control group without a chemical activator.¹⁸ However, most studies of chemical activators did not use or used limited brand of commercial tooth whitening product in their studies.¹⁰⁻¹⁶

The aim of this study was to investigate the effect of adding FeSO_4 to hydrogen peroxide containing commercial tooth bleaching products on color change and bleaching time required. The null hypotheses were: 1) There was no significant difference in tooth color between teeth bleached with hydrogen peroxide gel containing FeSO_4 and teeth bleached with hydrogen peroxide gel alone and 2) There was no significant difference in tooth color between teeth bleached with hydrogen peroxide gel containing FeSO_4 using reduced time and teeth bleached with hydrogen peroxide gel alone at the manufacturer's recommended time.

Materials and Methods

One hundred and twenty extracted intact non-carious and non-restored human upper premolars were collected with signed consent forms, under a protocol approved by the Ethics Committee of the Faculty of Dentistry, Chulalongkorn University (Study Code: HREC-DCU 2016-045), and conformed to the provisions of the Declaration of Helsinki. The selected teeth were debrided and stored in 0.1 % thymol solution (Merck KGaA, Darmstadt, Germany) at 4°C for up to 3

months after extraction. The teeth were examined under a stereomicroscope (ML 9300; Meiji, Saitama, Japan) at 20x magnification to exclude defects such as enamel cracks, fractures, caries and staining. The teeth in this study were of only hue A or B, using a VITA Easyshade® V spectrophotometer (Vita Zahnfabrik, Bad Säckingen, Germany). A NTI® sintered diamond disc (D352-220; Kerr, Orange, CA, USA) was used to obtain 5x5 mm enamel/dentin specimens from the labial surface of each tooth. The dentin side of the specimen was ground flat with 600, 800, 1,000, 1,200-grit silicon carbide sand paper (3M ESPE, St. Paul, MN, USA), under a constant water flow until the remaining dentin layer was 1 mm thick.

Ten holes were drilled in the middle area of an acrylic sheet, 6x12 cm and 2 mm thick (3A MEDES,

Goyang-si, Gyeonggi-do, Republic of Korea), using a 7 mm diameter trephine drill (Hu-friedy, Chicago, IL, USA) (Fig. 1a). Ten enamel/dentin specimens per sheet were then randomly positioned in the center of the holes, dentin side down, and embedded in clear epoxy resin (Super Silicone & Resin Art, Bangkok, Thailand) (Fig. 1b and c). The enamel side of each enamel/dentin block was ground flat using 600, 800, 1,000 and 1,200-grit sand paper (3M ESPE, St. Paul, MN, USA), under a constant water flow, until the enamel/dentin block was 2 mm thick. A 2 mm diameter carbide round bur (Kerr, Orange, CA, USA) was used to drill a hole 1 mm away from each specimen. The holes served as registration marks to assure that repeated color readings were performed at the same position (Fig. 1d).

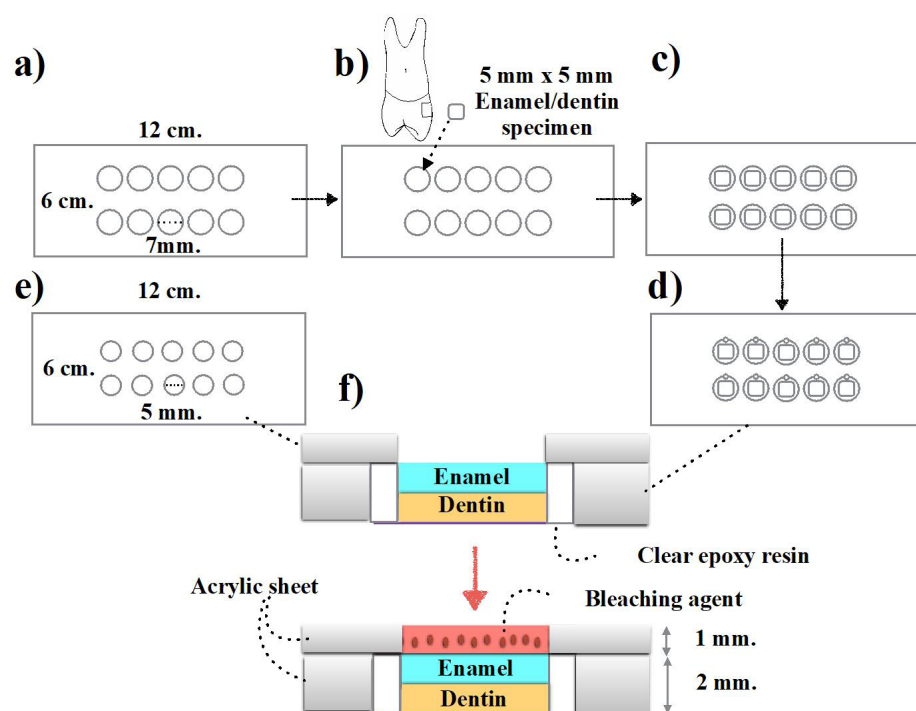


Figure 1 Schematic diagram of the study design. a) Ten 7 mm diameter, internal circular holes were drilled in the middle area of a 2 mm thick acrylic sheet. b) The enamel/dentin specimens were positioned in the center of circles. c) Specimens were embedded in clear epoxy resin. d) A 2 mm diameter hole was drilled 1 mm away from each specimen for repeatable positioning for color measurement. e) Ten 5 mm diameter internal circular holes were drilled in the middle area of a 1 mm thick acrylic sheet. f) The 1 mm thick acrylic sheet was used to control the amount of bleaching agent placed on the labial surface of each enamel/dentin specimen.

The twelve acrylic sheets were randomly allocated into twelve groups, according to three commercial bleaching products: Opalescence® Boost™ (OB) (Ultradent, South Jordan, UT, USA), PolaOffice® (PO) (SDI Limited, Victoria, Australia) and ZOOM!® Whitespeed™ (ZW) (Discus Dental/Philips Oral Healthcare, Stamford, CT, USA), the addition of FeSO₄ (Merck KGaA, Darmstadt, Germany) as a chemical activator to the gel, and the amount of time the gel would be applied (Table 1). A study determined that adding more than 0.004 % w/w FeSO₄ to a bleaching gel resulted in excessive bubble

formation that caused the gel to breakdown, thus, the concentration of 0.004 % w/w was used in the present study.¹⁰ Each product had 4 experimental groups: 1: Control, using the product's specified time without FeSO₄ (C), 2: FeSO₄ added, using the product's specified time (Fe 100 %), 3: FeSO₄ added, using approximately 75 % of the specified time (Fe 75 %), 4: FeSO₄ added, using approximately 50 % of the specified time (Fe 50 %). The manufacturer's recommended time was used as standard to compare.

Table 1 Three Bleaching products and twelve experimental groups in this study

Bleaching Product	Group	Ferrous Sulphate	Treatment time (time x cycles)
Opalescence® Boost™ (Ultradent, South Jordan, UT, USA)	1 (C)	No	20x2= 40 min
40 % Hydrogen peroxide Lot.BC25B	2 (Fe 100 %)	Yes	20x2 = 40 min
	3 (Fe 75 %)	Yes	15x2 = 30 min
	4 (Fe 50 %)	Yes	10x2 = 20 min
PolaOffice® (SDI Limited, Victoria, Australia)	1 (C)	No	8x4 = 32 min
35 % Hydrogen peroxide Lot.1082345	2 (Fe 100 %)	Yes	8x4 = 32 min
	3 (Fe 75 %)	Yes	6x4 = 24 min
	4 (Fe 50 %)	Yes	4x4 = 16 min
ZOOM!® Whitespeed™ (Discus Dental/ Philips Oral Healthcare, Stamford, CT, USA)	1 (C)	No	15x3 = 45 min
25 % Hydrogen peroxide Lot.22-3764	2 (Fe 100 %)	Yes	15x3 = 45 min
	3 (Fe 75 %)	Yes	10x3 = 30 min
	4 (Fe 50 %)	Yes	5x3 = 15 min

Prior to treatment, baseline color of each specimen was assessed under standard conditions based on the CIE-L*a*b* system using a VITA Easyshade® V spectrophotometer (Vita Zahnfabrik, Bad Säckingen, Germany). An elastic ligature (Patented design 33898; Dent-mate, Bangkok, Thailand) was prepared as shown in Fig. 2a, b and c. This was then placed on the tip of the VITA Easyshade® V spectrophotometer (Fig. 2d, e and f). Each sample was dried with an absorbent gauze sponge (Union Science, Chiangmai, Thailand) for 3 seconds,

and the color was immediately measured. A neutral gray cloth was used as a background. During the measurements, the instrument tip was placed perpendicular to, and in complete contact with enamel surface of the enamel/dentin block. The 2 mm hole near each enamel/dentin specimen was used to register the sample position for color reading (Fig. 2g), by placing the 2 mm circle of the elastic ligature on top of the 2 mm hole of the acrylic sheet that was next to each enamel/dentin specimen (Fig. 2h and i). The color of each sample

was measured 5 times and averaged. The L*, a*, b* measurements served as the baseline color of each sample. All measurements were made by single investigator. Before any measurement, the device was

calibrated by placing a probe tip on the instrument's calibration port aperture, following the manufacturer's instruction.

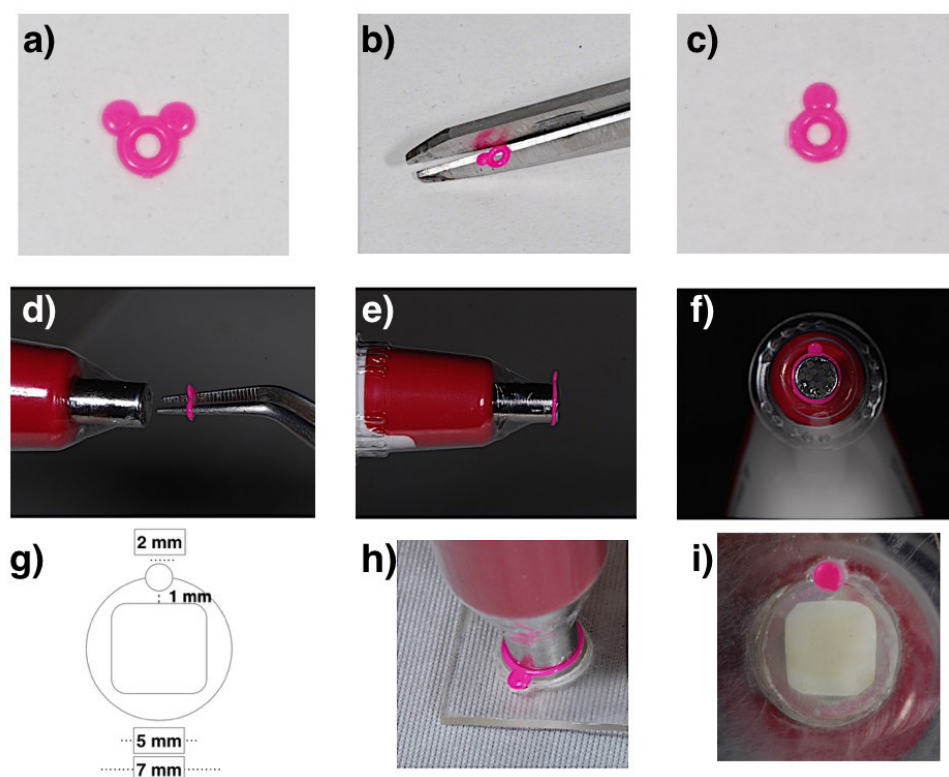


Figure 2 a-c) An elastic ligature was prepared. d-f) and placed on the tip of the VITA Easyshade® V spectrophotometer. g) A 2 mm diameter hole was drilled 1 mm away from each specimen. h) The 2 mm elastic ligature circle was placed on top of 2 mm hole in the acrylic sheet that was 1 mm away from each enamel/dentin specimen. This allowed for positioning each sample at the same spot for repeated color measurements. i) The probe tip of the VITA Easyshade® V spectrophotometer was completely covered by 5 mm x 5 mm specimen.

Before bleaching procedure, the enamel/dentine blocks were cleaned using a prophylaxis paste (fluoride-free pumice; 3M ESPE, St. Paul, MN, USA), with a polishing cup (3M ESPE, St. Paul, MN, USA) using a slow speed handpiece. The paste was removed using a water spray. Bleaching gels were then mixed according to manufacturer's recommendation, and transferred to a 5 ml glass beaker (Union Science, Chiangmai, Thailand). In the appropriate groups, 0.004 % (w/w) FeSO_4 (Merck KGaA, Darmstadt, Germany) was added to the gel and mixed on a magnetic stirrer (Bibby Scientific, Burlington, NJ, USA) using a

magnetic stirring bar, 3 mm in diameter and 5 mm in length (ProSciTech Pty Ltd, Queensland, Australia), for 1 minute. Only ZW used light activation (curing light unit obtained from the gel manufacturer) when the gel was placed on the samples.

A 6 cm x 12 cm, 1 mm thick acrylic sheet (3A MEDES, Goyang-si, Gyeonggi-do, Republic of Korea) was drilled with 5 mm diameter trephine drill (Hu-friedy, Chicago, IL, USA) to create ten 5 mm internal diameter holes in the middle area of the acrylic sheet (Fig. 1e). This acrylic sheet was placed on top of the enamel/

dentin specimen acrylic sheet, serving as a template, to accommodate a 1 mm layer of bleaching gel on the enamel surface of each specimen (Fig. 1f). An aspiration cannula was used to remove the gel between each application, simulating a clinical bleaching treatment. The specimens were then washed with a water spray. After bleaching, the same method as for the initial color measurement was used. Color of each sample was measured 5 times and averaged. The averaged L^* , a^* , b^* measurements served as the after bleaching color of each sample. In order to observe the immediate effect of FeSO_4 as a chemical activator, bleaching was performed on one experimental group at a time, and color of the samples in each group was measured within 10 minutes after bleaching.

The results of color measurement were quantified in three coordinate values (L^* , a^* , b^*), established by the Commission Internationale de l'Eclairage (CIE), which located the color of an object in three-dimensional color space. The L^* axis represented the degree of lightness within a sample, ranging from 0 (black) to 100 (white). The a^* plane represented the degree of green/red, and the b^* plane represented the degree of blue/yellow in the sample. Comparing tooth color before and after treatment, color changes (ΔE) and differences (ΔL , Δa , Δb) were calculated with the following color definition of respective positive (+) and negative (-) values: $\Delta L = (+)$ white, $(-)$ black; $\Delta a = (+)$ red, $(-)$ green; $\Delta b = (+)$ yellow, $(-)$ blue.

Determination of the color change of each specimen after the bleaching procedure was made by calculating the variation of L^* (ΔL), a^* (Δa), and b^* (Δb). The total color change (ΔE) were calculated according to the following formula:

$$\Delta E = [(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]^{1/2}$$

where $\Delta L = L_{\text{Final}} - L_{\text{Initial}}$, $\Delta a = a_{\text{Final}} - a_{\text{Initial}}$ and $\Delta b = b_{\text{Final}} - b_{\text{Initial}}$.

The data were statistically analyzed using SPSS 17.0 for Windows (Chicago, IL, USA). After using the Shapiro-Wilk test to determine a normal distribution, color measurement data (ΔE , ΔL , Δa and Δb) were analyzed using one-way ANOVA to compare the differences between experimental groups of each commercial product, followed by the Tukey's homogeneous test. If Levene's test showed that homogeneity of variance was violated, the Tamhane's test was used instead of Tukey's homogeneous test. The level of significance was determined as $p=0.05$.

Results

Means and standard deviations of ΔL , Δa , Δb and ΔE of the groups were determined as shown in table 2. One-way ANOVA indicated that there were no significant differences in ΔL , Δa , Δb and ΔE between experimental groups of each product ($p>0.05$), except ΔL between the PO groups ($p=0.016$) and Δb between the ZW groups ($p=0.001$), as shown in Table 3. Table 3 also presents the results of Tukey's homogeneous test for ΔL of the PO groups. The data analysis revealed that only ΔL of the PO Fe 100 % and Fe50 % were significantly different from each other. The Levene statistics showed that the homogeneity of variance of Δb between the ZW groups was violated ($p=0.021$), therefore, the Tamhane's test was used instead of Tukey's homogeneous test to analyze these results. Table 3 presents the results of Tamhane's test for Δb of the ZW groups. Δb of the ZW C and Fe 100 % groups were significantly different from that of the Fe 50 % group.

Table 2 Results of one-way ANOVA

Product	Parameter	df	SQ	QM	F	p-level*
Opalescence®	ΔL	3	16.277	5.426	1.014	0.398
Boost™	Δa	3	0.027	0.009	0.045	0.987
	Δb	3	18.842	6.281	1.385	0.263
	ΔE	3	0.478	0.159	0.105	0.956
PolaOffice®	ΔL	3	30.861	10.287	3.951	0.016*
	Δa	3	0.654	0.218	1.177	0.332
	Δb	3	3.119	1.040	0.545	0.654
	ΔE	3	4.903	1.634	2.077	0.120
ZOOM!®	ΔL	3	54.255	18.085	0.858	0.472
Whitespeed™	Δa	3	4.933	1.644	2.816	0.053
	Δb	3	258.903	86.301	6.386	0.001*
	ΔE	3	43.093	14.364	2.228	0.102

Abbreviation – df: Degree of Freedom; SQ: Sum of Squares; QM: Mean Square

* Significant differences ($p < 0.05$).

Where; ΔL = LFinal – LInitial; Δa = aFinal – aInitial; Δb = bFinal – bInitial and ΔE = EFinal – EInitial.

Table 3 Mean and Standard deviation values of ΔL, Δa, Δb and ΔE

Product	Group	Mean of ΔL (sd)	Tukey's homogeneous groups *	Mean of Δa (sd)	Mean of Δb (sd)	Tamhane's test*,**	Mean of ΔE (sd)
Opalescence®	C	2.40 (1.55)		-0.59 (0.52)	-2.21 (1.21)		3.66 (1.21)
Boost™	Fe100 %	0.69 (2.65)		-0.51 (0.36)	-1.31 (2.81)		3.89 (0.98)
	Fe75 %	1.93 (2.50)		-0.54 (0.47)	-2.24 (0.97)		3.78 (1.23)
	Fe50 %	1.30 (2.40)		-0.54 (0.41)	-0.59 (2.80)		3.61 (1.45)
PolaOffice®	C	1.88 (1.34)	AB	-0.09 (0.26)	-0.11 (1.36)		2.51 (0.82)
	Fe100 %	1.98 (1.43)	A	0.02 (0.44)	0.18 (1.47)		2.66 (0.98)
	Fe75 %	0.41 (2.07)	AB	-0.13 (0.44)	-0.60 (1.62)		2.41 (1.13)
	Fe50 %	-0.01 (1.51)	B	0.20 (0.54)	-0.14 (0.98)		1.74 (0.49)
ZOOM!®	C	0.43 (2.88)		-0.30 (0.70)	-6.68 (2.78)	C	7.41 (2.33)
Whitespeed™	Fe100 %	-1.25 (6.16)		0.24 (0.92)	-5.07 (1.70)	C	7.89 (1.94)
	Fe75 %	-0.46 (4.32)		-0.39 (0.87)	-2.34 (5.60)	CD	6.81 (2.40)
	Fe50 %	-2.74 (4.40)		0.44 (0.50)	-0.04 (3.47)	D	5.14 (3.29)

Where; ΔL = LFinal – LInitial; Δa = aFinal – aInitial; Δb = bFinal – bInitial and ΔE = EFinal – EInitial.

* The groups accompanied by the same letters do not present significant differences.

** Equal variances not assumed.

Discussion

The first null hypothesis was accepted due to no significant difference in tooth color between the Fe 100 % and C groups. The second null hypothesis was partially accepted, as the results show no significant difference in tooth color between the Fe 75 % and C groups, however, Δb of the ZW Fe 50 % group was significantly different from that of the C group.

If the ΔE resulting from increased ΔL and decreased Δb is 2 or greater, the bleaching efficacy of the product is deemed to be acceptable, per ISO 28399.¹⁹ Our results demonstrated a ΔE of more than 2 for every commercial product used in this study, indicating that every product is acceptable to use for tooth bleaching.

ΔE results of the Fe 100 %, Fe 75 % and Fe 50 % groups were not significantly different compared with the C groups of all products (Fig 3). The data showed

that for the OB, PO and ZW materials, ΔE of the Fe 100 % group was little higher compared with that of the C group at 6.3 %, 5.9 % and 6.5 % respectively, similar to that found by Travassos *et al.*¹⁷ In contrast to the results of the present study, other studies found significantly improved ΔE results when adding FeSO_4 as a chemical activator.^{10,18} It should be noted that most studies related to chemical activators did not use or use limited brand of commercial whitening product in their investigations.¹⁰⁻¹⁶ Every commercial bleaching product has different composition. Even products, labeled as having the same concentration of hydrogen peroxide, may provide different bleaching effects.^{20,21} Furthermore, some studies used a coloring solution to darken the tooth samples before bleaching, which may have facilitated the observation of significant differences.^{15,17,18}

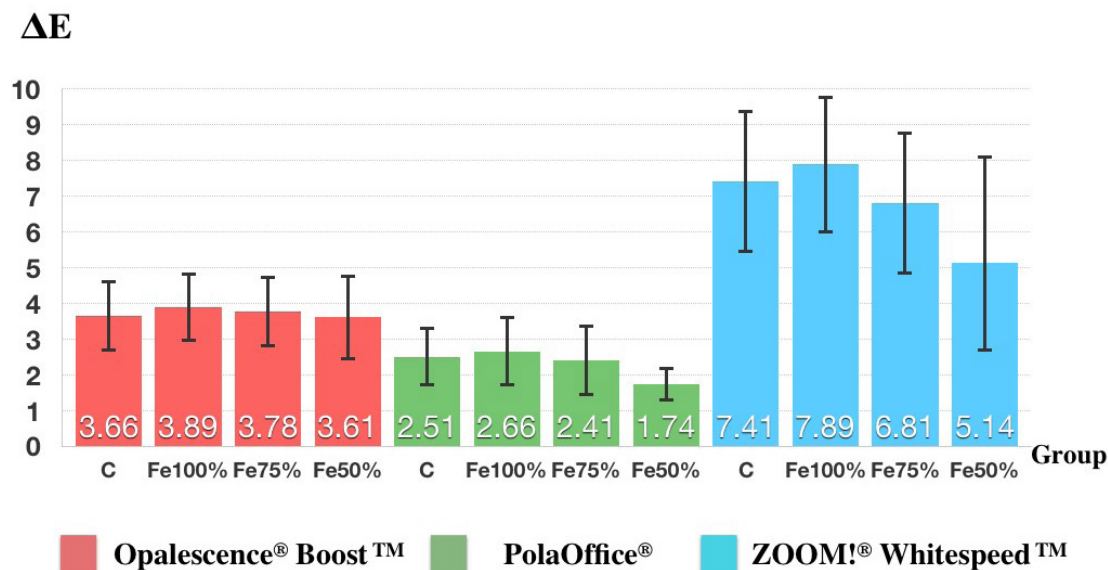


Figure 3 Mean ΔE of experimental groups

Our results showed that ΔE of the Fe 75 % and Fe 50 % groups in each product were not statistically different from that of the C groups. The use of an iron derivative catalyzer resulted in a reaction between the

Fe^{2+} ion from FeSO_4 with hydrogen peroxide, known as the Fenton Reaction (Fig. 4).^{22,23} Addition of an iron ion caused an increase in the oxidative force of hydrogen peroxide, resulting in an increase in its degradation

speed, causing free radicals, a hydroxyl radical and a peroxide radical, to form more rapidly.^{22,24} The hydroxyl radicals are strongly oxidizing, leading to the breakdown of chromogens.^{10,17,24} From the three products evaluated in our study the results suggested that FeSO₄ increased the speed of hydrogen peroxide decomposition. FeSO₄ increased the formation of free radicals on the tooth

surface more rapidly.^{10,17,18,22} Therefore, these results implied that addition of FeSO₄ could reduce treatment time. We had not any groups without FeSO₄ using reduced time because the manufacturer's recommended time should represent the best outcome of each product. Reducing treatment time produced significantly lower ΔE value than the traditional in-office protocol.^{25,26}



Figure 4 Fenton's reaction: Fe²⁺ is oxidized by hydrogen peroxide to Fe³⁺, forming a hydroxyl radical and a hydroxyl anion. Fe³⁺ is then reduced back to Fe²⁺ by another molecule of hydrogen peroxide, forming a peroxide radical and a proton

The ΔE results of the PO Fe 75 % and Fe 50 % groups were not significantly different compared with the C group. However, the Fe 50 % group, which treatment time was reduced by half, showed a ΔE of 1.74±0.49, which is not acceptable according to ISO 28399. We also found that L* in this group did not increase after bleaching. Although ΔL of the Fe 50 % group was significantly different from that of the Fe 100 % group, it was not significantly different compared with the C group. The PO 75 % group demonstrated ΔL, Δa, Δb and ΔE results that were not significantly different from that of the C group. Our data suggested that if FeSO₄ was added to PO as a chemical activator, the treatment time may be reduced by 25 %, from 32 minutes to 24 minutes. The ΔE of the ZW Fe 50 % group was not significantly different from that of the C group. However, Δb of the Fe 50 % group was significantly different from that of the C group. This result indicated that the bleaching agent in this group did not decrease b*. We also found that L* in the Fe 50 % group decreased. Thus, the bleaching procedure in this group was inefficient. However, the ZW Fe 75 % group ΔL, Δa, Δb and ΔE were not significantly different from those of the C group. These

data suggested that when adding FeSO₄ as a chemical activator to ZW, the treatment time may be reduced by 33 % from 45 minutes to 30 minutes. It was also found that the OB Fe 75 % and 50 % groups demonstrated the values of ΔL, Δa, Δb and ΔE that were not significantly different compared with the C group. These data suggested that addition of FeSO₄ as a chemical activator in OB, facilitated treatment time reduction from 40 minutes to 20 minutes or 50 % of treatment time reduction.

Our study indicated that the use of FeSO₄ as a chemical activator may be an alternative method for increasing the speed of hydrogen peroxide decomposition. Because only 0.004 % of FeSO₄ was required, this method could, therefore, be considered inexpensive. This metal salt is not toxic when used in the right concentration, since it is used as nutritional supplement in human diet.¹⁰ Duque *et al.* evaluated the effect of FeSO₄ on the transenamel and transdental cytotoxicity of the bleaching gel to two culture lineages obtained from dental pulp in an *in vitro* study. The viability of odontoblast-like cells (MDPC-23) and Human dental pulp cells (HDPCs) from tooth bleached with 35 % hydrogen peroxide gel mixed with FeSO₄ was not

significant different from those bleached with 35 % hydrogen peroxide gel alone.¹⁸ However, when using this method a large amount of hydroxyl free radicals and hence a large amount of oxygen are released in a very short time. One concern is that the active generation of oxygen bubbles may result in gel contacting gingiva, leading to irritation.¹¹

There are many benefits of reducing bleaching time. Patients undergoing bleaching procedures frequently complained of painful or uncomfortable sensations arising in the treated teeth. This long duration of pain had affected patient's satisfaction with their bleaching experience and might be so severe that patient's discontinue treatment.^{6,7} Shorten treatment time reduced tooth sensitivity.^{6,25,27} It also reduced the amount of hydrogen peroxide penetrating into pulp chamber, associated with decreased adverse effects to pulp cells.^{25,26}

In the present study, ZW groups surprisingly showed the highest ΔE results, despite the lowest concentration of hydrogen peroxide among the three products. The gels containing higher hydrogen peroxide concentrations did not appear to have a greater effect on whitening,²¹ in contrast to other studies.^{3,28,29} The reasons why ZW generated a higher ΔE may be due to its longer contact time, high pH and the effect of light activation. Studies have shown a trend toward improved whitening with increased contact time.^{21,25,29,30} However, this effect would be limited by the activity of the peroxide. In a high pH (>7) environment, hydrogen peroxide became destabilized and released hydroxyl and peroxide free radicals and oxygen gas.¹¹ The pH of OB is 7, while those of PO and ZW are 5.5 and 8.9, respectively. Thus, only the pH of ZW is greater than 7. A number of studies demonstrated that light activation increased bleaching gel efficacy.³¹⁻³⁵ However, some studies showed that the increased bleaching gel efficacy from light activation was limited or were not significant.^{8,9,36,37} Studies have speculated that light may cause temporary dehydration, resulting in lighter appearance of teeth that relapsed

over time.^{21,36} More research is required to resolve this issue. The manufacturer of OB informed that teeth would continually be whitened 12 to 24 hours after whitening treatment is completed. However, our study focused only on immediate color change after whitening treatment.

Because the ΔL and Δb raw data in each group was quite varied, the SD was higher than the mean. These results may have occurred because of tooth color variation. Evaluation of the effect of LASER tooth whitening revealed that teeth with hue A showed greater shade improvement than those of teeth with hue C or D.³⁸ However, in our study we used human upper premolar teeth of hue A or B to mitigate this issue. We also found that even samples of the same hue, samples with high chroma showed greater ΔE results than those with low chroma. Somehow further studies should consider using teeth of the same initial tooth color, both hue and chroma.

Visual color matching is subjective and influenced by variety of factors. Observer-related variables such as sex, color blindness, experience and eye fatigue could influence the final results.^{39,40} More accurate results were reported with the use of dental spectrophotometer compared with visual methods.⁴¹⁻⁴⁵ Furthermore, studies have demonstrated that the performance of the VITA Easyshade® spectrophotometer was more accurate than that of a naked eye.^{41,46,47} One problem with shade matching, especially for bleaching studies, is positioning the tip of the measuring device. A flat surface is required for accurate placement of measuring tip. Therefore, the present study used 2 mm thick enamel/dentin specimens, which were grounded flat on both sides, to eliminate problem of tooth convexity. In addition, 5 mm x 5mm specimens were used in order to accommodate the 5 mm diameter probe tip of the measuring device.

The purpose of this study was only to improve material efficacy. However, the pulpal reaction is still the most important aspect that should be concerned. This is to prevent the misuse of the report. Further

studies should include groups using reduced time with and without FeSO_4 in order to confirm role of FeSO_4 . Additional studies should also focus on the safety and efficiency when using a bleaching agent mixed with FeSO_4 *in vivo*. Studies concerning color relapse and the exact amount of time that can be reduced when adding FeSO_4 to other commercial bleaching products should also be conducted. This information will allow for greater understanding of the effects of FeSO_4 as a chemical activator. Furthermore, there are other potential chemical activators that may improve bleaching results and simultaneously reduce treatment time such as manganese gluconate, manganese chloride, etc.

Conclusion

Within the limitations of this study, it can be concluded that adding 0.004 % w/w FeSO_4 reduced the treatment time of these commercial bleaching products, while obtaining the same bleaching results. For PolaOffice®, the treatment time could be reduced by 25 %, from 32 minutes to 24 minutes, ZOOM!® Whitespeed™ 33 %, from 45 minutes to 30 minutes, and by 50 % for Opalescence® Boost™, from 40 minutes to 20 minutes.

Acknowledgements

Our sincere thanks go to Assoc. Prof. Chanchai Hosawaun for statistic consultation and Dr. Kevin Tompkins for academic English proofreading.

Disclosure statement

The authors do not have any financial interest in the companies whose materials are included in this article.

References

1. McEvoy SA. Chemical agents for removing intrinsic stains from vital teeth. I. Technique development. *Quintessence Int*

1989;20:323-8.

2. Barghi N. Making a clinical decision for vital tooth bleaching: at-home or in-office? *Compend Contin Educ Dent* 1998;19: 831-8;quiz 40.

3. Sulieman M. An overview of bleaching techniques: 3. In-surgery or power bleaching. *Dent Update* 2005;32:101-4,7-8.

4. Kihn PW. Vital tooth whitening. *Dent Clin North Am* 2007;51:319-31,viii.

5. Alqahtani MQ. Tooth-bleaching procedures and their controversial effects: A literature review. *Saudi Dent J* 2014;26:33-46.

6. Hewlett ER. Etiology and management of whitening-induced tooth hypersensitivity. *J Calif Dent Assoc* 2007;35: 499-506.

7. Leonard RH Jr, Haywood VB, Phillips C. Risk factors for developing tooth sensitivity and gingival irritation associated with nightguard vital bleaching. *Quintessence Int* 1997;28:537-34.

8. Al Shethri S, Matis BA, Cochran MA, Zekonis R, Stropes M. A clinical evaluation of two in-office bleaching products. *Oper Dent* 2003;28:488-95.

9. Mena-Serrano AP, Garcia E, Luque-Martinez I, Grande R, Loguericio AD, Reis A. A Single-Blind Randomized Trial About the Effect of Hydrogen Peroxide Concentration on Light-Activated Bleaching. *Oper Dent* 2016;41:455-64.

10. Torres CR, Wiegand A, Sener B, Attin T. Influence of chemical activation of a 35 % hydrogen peroxide bleaching gel on its penetration and efficacy—in vitro study. *J Dent* 2010;38:838-46.

11. Banerjee A, Friedman J, inventors; AdDent Inc, assignee. Dental bleaching gel composition, activator system and method for activating a dental bleaching gel. United States patent US 6,485,709 B2. 2002 Nov 26.

12. Chen JH, Xu JW, Shing CX. Decomposition rate of hydrogen peroxide bleaching agents under various chemical and physical conditions. *J Prosthet Dent* 1993;69:46-8.

13. Feng J, inventor; Go smile Inc, assignee. Tooth whitening accelerator formulation and method of using the same. United States patent US 8,906,350 B2. 2014 Dec 09.

14. Freedman B, inventor. Booster and activator for tooth-whitening agents. United States patent US 2004/0191188 A1. 2004 Sep 30.

15. Gaffar A, inventor. Oral compositions having accelerated tooth whitening effect. United States patent US 5,648,064. 1997 Jul 15.

16. Zhao J, inventor. Whitening Compound. United States patent US 2005/0042185 A1. 2005 Feb 24.

17. Travassos AC, Rocha Gomes Torres C, Borges AB, Barcellos DC. In vitro assessment of chemical activation efficiency during in-office dental bleaching. *Oper Dent* 2010;35:287-94.

18. Duque CC, Soares DG, Basso FG, Hebling J, de Souza Costa CA. Bleaching effectiveness, hydrogen peroxide diffusion, and cytotoxicity of a chemically activated bleaching gel. *Clin Oral Investig* 2014;18:1631-7.
19. ISO23899. Dentistry-Products for external tooth bleaching. 2011.
20. Thitinanthapan W, Satamanont P, Vongsavan N. *In vitro* penetration of the pulp chamber by three brands of carbamide peroxide. *J Esthet Dent* 1999;11:259-64.
21. Matis BA, Cochran MA, Franco M, Al-Ammar W, Eckert GJ, Stropes M. Eight in-office tooth whitening systems evaluated *in vivo*: a pilot study. *Oper Dent* 2007;32:322-7.
22. Dunford HB. Free radicals in iron-containing systems. *Free Radic Biol Med* 1987;3:405-21.
23. Liochev SI, Fridovich I. The Haber-Weiss cycle -- 70 years later: an alternative view. *Redox Rep* 2002;7:55-7;author reply 9-60.
24. Young N, Fairley P, Mohan V, Jumeaux C. A study of hydrogen peroxide chemistry and photochemistry in tea stain solution with relevance to clinical tooth whitening. *J Dent* 2012;40Suppl2:e11-6.
25. Soares DG, Basso FG, Pontes EC, Garcia Lda F, Hebling J, de Souza Costa CA. Effective tooth-bleaching protocols capable of reducing H₂O₂ diffusion through enamel and dentine. *J Dent* 2014;42:351-8.
26. Soares DG, Ribeiro AP, da Silveira Vargas F, de Souza Costa CA. Efficacy and cytotoxicity of a bleaching gel after short application times on dental enamel. *Clin Oral Investig* 2013;17:1901-9.
27. Kose C, Calixto AL, Bauer JR, Reis A, Loguercio AD. Comparison of the Effects of In-office Bleaching Times on Whitening and Tooth Sensitivity: A Single Blind, Randomized Clinical Trial. *Oper Dent* 2016;41:138-45.
28. Kashima-Tanaka M, Tsujimoto Y, Kawamoto K, Senda N, Ito K, Yamazaki M. Generation of free radicals and/or active oxygen by light or laser irradiation of hydrogen peroxide or sodium hypochlorite. *J Endod* 2003;29:141-3.
29. Heymann HO. Tooth whitening: facts and fallacies. *Br Dent J* 2005;198:514.
30. Reis A, Tay LY, Herrera DR, Kossatz S, Loguercio AD. Clinical effects of prolonged application time of an in-office bleaching gel. *Oper Dent* 2011;36:590-6.
31. Allred P, inventor. Two-part dental bleaching systems having improved gel stability and methods for bleaching teeth using such systems; 2003 Jan 07.
32. Luk K, Tam L, Hubert M. Effect of light energy on peroxide tooth bleaching. *J Am Dent Assoc* 2004;135:194-201; quiz 28-9.
33. Bhutani N, Venigalla BS, Patil JP, Singh TV, Jyotsna SV, Jain A. Evaluation of bleaching efficacy of 37.5 % hydrogen peroxide on human teeth using different modes of activations: An *in vitro* study. *J Conserv Dent* 2016;19:259-63.
34. Park S, Kwon SR, Qian F, Wertz PW. The Effect of Delivery System and Light Activation on Tooth Whitening Efficacy and Hydrogen Peroxide Penetration. *J Esthet Restor Dent* 2016;28:313-20.
35. Klaric E, Rakic M, Marcus M, Ristic M, Sever I, Tarle Z. Optical effects of experimental light-activated bleaching procedures. *Photomed Laser Surg* 2014;32:160-7.
36. Jones AH, Diaz-Arnold AM, Vargas MA, Cobb DS. Colorimetric assessment of laser and home bleaching techniques. *J Esthet Dent* 1999;11:87-94.
37. Buchalla W, Attin T. External bleaching therapy with activation by heat, light or laser--a systematic review. *Dent Mater* 2007;23:586-96.
38. Lin CH, Chou TM, Chen JH, Chen JH, Chuang FH, Lee HE, et al. Evaluation of the effect of laser tooth whitening. *Int J Prosthodont* 2008;21:415-8.
39. Haddad HJ, Jakstat HA, Arnetzl G, Borbely J, Vichi A, Dumfahrt H, et al. Does gender and experience influence shade matching quality? *J Dent* 2009;37 Suppl 1:e40-4.
40. Capa N, Malkondu O, Kazazoglu E, Calikkocaoglu S. Evaluating factors that affect the shade-matching ability of dentists, dental staff members and laypeople. *J Am Dent Assoc* 2010;141:71-6.
41. Bahannan SA. Shade matching quality among dental students using visual and instrumental methods. *J Dent* 2014;42:48-52.
42. Fani G, Vichi A, Davidson CL. Spectrophotometric and visual shade measurements of human teeth using three shade guides. *Am J Dent* 2007;20:142-6.
43. Paul S, Peter A, Pietrobon N, Hammerle CH. Visual and spectrophotometric shade analysis of human teeth. *J Dent Res* 2002;81:578-82.
44. Gehrke P, Riekeberg U, Fackler O, Dhom G. Comparison of *in vivo* visual, spectrophotometric and colorimetric shade determination of teeth and implant-supported crowns. *Int J Comput Dent* 2009;12:247-63.
45. Paul SJ, Peter A, Rodoni L, Pietrobon N. Conventional visual vs spectrophotometric shade taking for porcelain-fused-to-metal crowns: a clinical comparison. *Int J Periodontics Restorative Dent* 2004;24:222-31.
46. Browning WD, Chan DC, Blalock JS, Brackett MG. A comparison of human raters and an intra-oral spectrophotometer. *Oper Dent* 2009;34:337-43.
47. Alsaleh S, Labban M, AlHariri M, Tashkandi E. Evaluation of self shade matching ability of dental students using visual and instrumental means. *J Dent* 2012;40 Suppl 1:e82-7.