

Surface Change of Dental Amalgam after Treatment with 10% Carbamide Peroxide

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The aim of this study was to explore the effect of bleaching agents on the release of metal ions and change in physical and chemical characteristics of amalgams. Amalgam surface changes resulting from bleaching on amalgam were assessed with inductively coupled plasma atomic emission spectrophotometer, an automatic mercury analyzer, a colorimeter, and atomic force microscopy. Analysis of the release of metal ions indicated that the bleaching agent caused more copper, tin, and mercury ions to be released into the immersion solution. Furthermore, metal ion release increased progressively as immersion time was increased from 42 to 84 hours. Morphological examination revealed no significant effects of the 10% carbamide peroxide bleaching agent on the surface morphology of any of the amalgams tested. These results demonstrated that bleaching using 10% carbamide peroxide did not adversely affect the two types of amalgam tested. In other words, a 10% carbamide peroxide bleaching agent can be safely used on a short-term basis in dentist-monitored bleaching of the dental amalgam.

Key words : Carbamide peroxide, Dental amalgam, Surface change

INTRODUCTION

Nowadays, various commercial bleaching products are commonly used at home and in the dental clinic. Most commercial products include carbamide peroxide-based gels, and are usually applied with a prefabricated tray to the external surfaces of teeth. Carbamide peroxide (CP) is available in concentrations varying between 3 and 50%^{1–3}. Popular commercial preparations contain about 10% CP and have an average pH of 5 to 6.5. The bleaching procedure takes place with CP decomposed into 6.4% urea, ammonia, carbon dioxide, and approximately 3.6% hydrogen peroxide^{4,5}. Against this background, controlled mouthguard bleaching procedures are considered relatively safe with regard to systemic effects.

However, of late, some controversy has arisen regarding their effect on restorative materials. To this controversy, several studies have allayed the related fears and concerns by reporting no significant changes^{6–9}. Hummert *et al.*⁶ suggested that when amalgam was exposed to 10% CP bleaching solution, mercury was released – but the amount was not significant according to World Health Organization (WHO) guidelines. Likewise, Rotstein *et al.*⁷ reported that increased mercury levels were found in solution after treatment with CP, but the increase did not exceed the WHO guidelines for mercury exposure. Ulukapi *et al.*⁸ demonstrated that bleaching with carbamide might alter the marginal leakage of resin composite restorations, but the amalgam resto-

rations were not affected adversely *in vitro*. Schemehorn *et al.*⁹ also showed that there were no significant effects of 6% hydrogen peroxide gel on the surface morphology of any of the admixed high-copper amalgams and hybrid resin composites. Nevertheless, a number of other researchers have indicated that the physical properties of certain permanent and temporary restorative materials were adversely affected^{10,11}. Haywood¹¹ reported green discoloration of the tooth-amalgam interface after using 10% CP bleaching for four months, and that this discoloration depended on amalgam brand and CP concentration. Additionally, some concern has been raised over bleaching agents causing adverse effects such as irritation to the gastric and respiratory mucosa when swallowed; alteration of the morphology and chemical composition of enamel, dentin, and cementum; and damage to composite restorations by softening and cracking the resin matrix^{12,13}.

Dental amalgams are generally intermetallic compounds of mercury, silver, tin, and copper^{14,15}. Sometimes, other selected metals – such as zinc or palladium¹⁶ – are added. Amalgam may be particularly susceptible to the strong oxidizing action of hydrogen peroxide¹⁷. Nevertheless, amalgam is still the most widely used restorative material for premolars and molars, and patients undergoing mouthguard bleaching are likely to have amalgam-restored teeth. Although bleaching gels are usually applied to the anterior dentition, excess bleaching material may inadvertently make contact with restored premolars

and molars and may increase the susceptibility of these amalgam restorations to corrosion and degradation. Thus, the interaction between amalgam and materials used for vital bleaching is of clinical significance. Despite the general, widespread use of tooth-whitening products, their efficacy and effects on the physical or chemical properties of dental amalgam still need to be verified.

The aim of the current study, therefore, was to explore the effect of a 10% CP bleaching agent on release of inorganic substances such as tin, copper, silver, zinc, and mercury ions and to examine changes in the amalgam surface.

MATERIALS AND METHODS

Two types of commercially available dental amalgam were selected for this study: SDI (Southern Dental Industries Ltd., Bayswater, Victoria, Australia) and Valiant Ph.D. (Dentsply Caulk Co., Milford, DE, USA). Each amalgam was prepared according to the manufacturer's instructions with a proportionate capsule and an amalgamator. Freshly prepared mix was condensed into an acrylic plastic mold 7 mm in diameter and 2 mm in depth. After 30 minutes, samples were removed from mold and immersed in distilled water for 48 hours. All specimens were highly polished with sandpaper (#800, #1,000, #2,000) and diamond paste (6 μm , 3 μm , 1 μm , and 0.1 μm) for surface standardization. Sixty specimens were then randomly divided into two groups: 30 specimens for control, and 30 for the experimental group where 10% CP was used as the bleaching material. While the control group was not bleached and only stored in distilled water for 14 days, the experimental group was exposed to CP for six hours per day for 14 days. The 10% CP was prepared by dissolving CP powder (Sigma, St Louis, MO, USA) in distilled water (pH 6.5).

Recommended exposure time was six hours per day in a mouthguard for seven days or 14 days. Hence, the numbers of ions released from the specimens were determined at 42 hours (6 hours \times 7 days) and 84 hours (6 hours \times 14 days) after 10% CP bleaching. All specimens were soaked in individual plates containing 5 ml of each solution. After bleaching, specimens were cleaned and dried for an hour at room temperature. Tin, copper, silver, and zinc ions in the soaking solutions were determined using an inductively coupled plasma atomic emission spectrophotometer (ICP-AES, JY38 Plus, Jobin-Yvon, France), and mercury was determined with an automatic mercury analyzer (FIMS-400, Bodenseewerk perkin-Elmer, Germany).

All specimens were assessed with a microhardness tester (MXT- α 7, Matsuzawa Co. Ltd., Japan) to detect changes in the surface hardness of the dental amalgams during the bleaching process at

0, 7, and 14 days.

A colorimeter (TC-8600A, Nippon Denshoku, Japan) was used to detect color changes before (0 day) and after bleaching (14 days). During baseline measurement, three measurements were performed for each specimen, and the mean of the readings was calculated. Next, the mean of each material was calculated using the CIE Lab uniform color scale¹⁸. The color characteristics of all specimens were compared to a white standard, which was a pressed powder tablet of barium sulfate. Magnitude of total color difference was represented by a single number, ΔE^* , which was calculated using the following equation:

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

where L^* represents lightness, a^* redness-greenness, and b^* yellowness-blueness.

Additionally, color change ΔE^* has been quantified by the National Bureau of Standards (NBS)¹⁹, according to NBS units of color difference. NBS units are expressed by the following formula:

$$\text{NBS unit} = \Delta E^* \times 0.92$$

The three-dimensional surface topography of amalgam surfaces was measured using atomic force microscopy (AFM, Nanoscope III Multimode, Digital Instruments Inc., USA) at 14 days. Roughness was measured on a 5 μm \times 5 μm flat area, 3.6 μm in height on the amalgam surface.

All collected data were analyzed with SAS 8.01. Differences between the two groups were then compared by Wilcoxon's rank sum test and repeated measures ANOVA at significance level of 0.05.

RESULTS

Tables 1 and 2 show the concentrations of metal ions in 10% CP. No ions were released in the control group. In the experimental group, copper, tin, and mercury ions were detected from the two types of amalgam in the 10% CP solution, while silver and zinc ions were not detected. Further, the concentrations of copper, tin, and mercury in the solution gradually increased as the immersion period of both amalgams increased from 42 to 84 hours. There was a significant increase in copper level up to 4.25 times for SDI (Table 1). There were also significant increases in tin and copper levels up to 1.22 and 1.4 times for Valiant Ph.D (Table 2).

Table 3 shows the microhardness values (VHN) of the control and 10% CP bleaching groups at 0, 7, and 14 days. There were no statistical differences in VHN between the control and 10% CP bleaching groups in all amalgams.

Table 4 shows the color changes of the two amalgam types after 10% CP bleaching treatment for 14 days. Mean of color change was calculated using

Table 1 Concentrations of metal ions in 10% CP solution from SDI at 42 and 84 hours ($\mu\text{g/ml}$)

	SDI	
	42 hours	84 hours
Sn	3.78±0.82	4.24±0.96
Cu*	0.04±0.05	0.17±0.07
Hg	0.59±0.19	0.83±0.08

Values are indicated as mean ± SD.

*p<0.05

Table 2 Concentrations of metal ions in 10% CP solution from Valiant Ph.D. at 42 and 84 hours ($\mu\text{g/ml}$)

	Valiant Ph.D.	
	42 hours	84 hours
Sn*	5.76±0.71	7.00±1.26
Cu*	0.88±0.12	1.23±0.44
Hg	0.20±0.03	0.23±0.03

Values are indicated as mean ± SD.

*p<0.05

Table 3 Change of microhardness (VHN) of SDI and Valiant Ph.D. with time

	SDI		Valiant Ph.D.	
	DW	10% CP	DW	10% CP
0 Day	88.37±2.68	88.88±3.19	122.13±3.26	121.11±3.21
7 Days	88.57±2.40	88.17±4.33	121.67±2.64	121.26±3.56
14 Days	88.57±3.48	87.17±3.37	121.27±1.42	121.42±3.37

Values are indicated as mean ± SD.

No statistically significant differences exist according to repeated measures ANOVA (p>0.05).

Table 4 Color changes and NBS units of each amalgam

	SDI	Valiant Ph.D.
DW	0.79±0.28 (0.73)	0.56±0.32 (0.52)
10% CP	1.80±1.00 (1.66)	2.20±0.36 (2.02)

Values are indicated as mean ± SD.

() : Critical remarks of color difference by NBS system.

No statistically significant differences exist according to Wilcoxon's rank sum test (p>0.05).

the CIE lab uniform color scale. Following which, the mean ΔE^* values of all amalgams were also converted to NBS units to reveal whether color changes of amalgams were clinically detectable. ΔE^* level and NBS units less than 3.3 is considered clinically insignificant. Hence, although there were slight differences in color change and NBS units between the two amalgam types, these differences were not statistically significant.

Fig. 1 shows the AFM images of the 3D surface topography of amalgams in the control and 10% CP bleaching groups after 14 days. No differences were observed between the control and 10% CP bleaching groups on any of the amalgams tested. For SDI, mean surface roughness (Ra) values corresponding to the control and 10% CP bleaching groups were 7.875 nm and 7.473 nm respectively. For Valiant Ph.D., similar results were observed where Ra values corresponding to the control and 10% CP bleaching groups were 9.011 nm and 10.531 nm respectively.

DISCUSSION

Carbamide peroxide is synonymous with urea peroxide, hydrogen peroxide carbamide, and perhydrol urea. The usual tray bleaching method uses 10 to 15% carbamide peroxide decomposing into 3 to 5% hydrogen peroxide and 7 to 10% urea once the solution comes into contact with moisture^{4,5}). Hydrogen peroxide is the active ingredient contained in the bleaching agent. For example, 10% carbamide peroxide is equivalent to about 3.5% hydrogen peroxide. This content of hydrogen peroxide is equivalent to one-tenth of 35% hydrogen peroxide. A concentration of 30-35% hydrogen peroxide is widely used for in-office teeth bleaching treatments²⁰), whereby one hour per week for two to four appointments is a common routine. On the other hand, 10% carbamide peroxide generally requires two weeks of at-home bleaching because of the lower hydrogen peroxide concentration. Nonetheless, nightguard vital bleaching became popular due to its simplicity. This method was first described and published by Haywood and Heymann¹), where 10 to 15% carbamide peroxide was used in a custom-made mouthguard for several weeks. Nowadays, these systems use 10% carbamide peroxide with carboxypolymethylene polymer as a thickening agent to improve tissue adherence and for sustained release of the whitening agent.

From our results, it appeared that the two amalgam brands tested released ions after 42 and 84 hours of exposure to CP. Although the 84-hour continuous exposure is longer than the usual

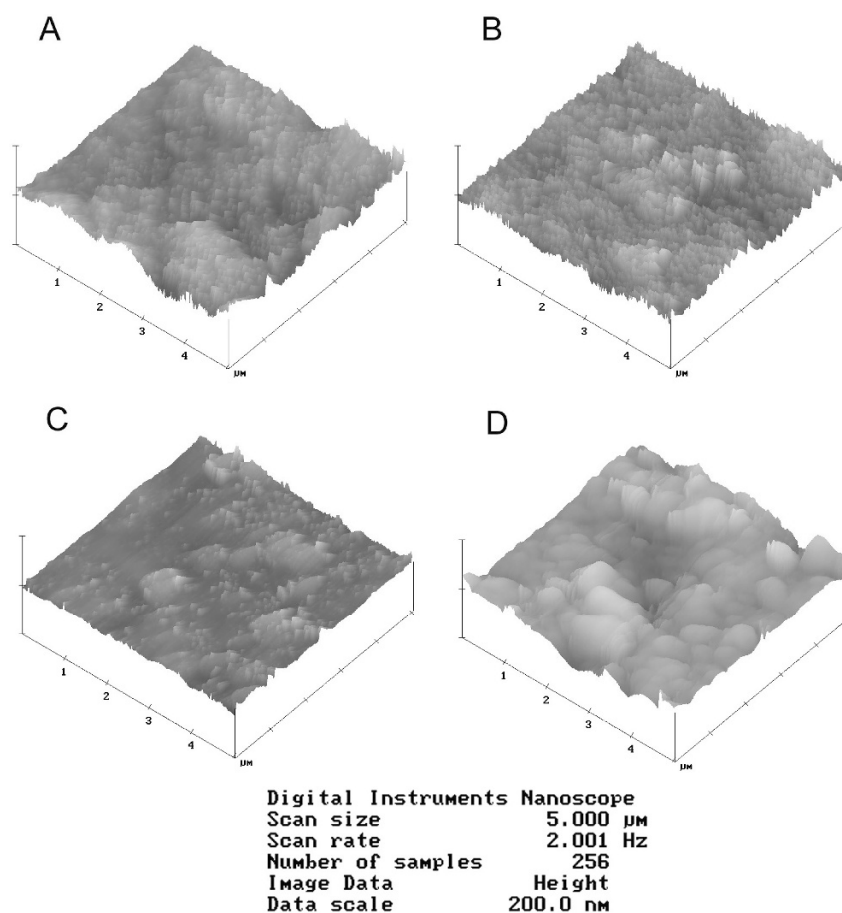


Fig. 1 AFM images of unbleached (A and C) and bleached amalgams with 10% CP (B and D). Amalgams were treated 6 hours per day for 14 days (A, B: SDI; C, D: Valiant Ph.D.).

intermittent exposure in patients, the results reported here are of clinical significance. The current results demonstrated that metal ion release from dental amalgams was dependent on exposure duration, and that the amount of metal ions released varied among different amalgam brands; however, there was no significant increase in mercury. Moreover, the increase in mercury amount was not alarming when viewed in terms of the WHO guidelines (0.3 mg/week/person)²¹⁾ for mercury exposure. Our results supported the reports of Hummert *et al.*⁶⁾ and Lee *et al.*²²⁾ which demonstrated higher amount of mercury in solutions of CP-treated amalgam as compared with DW. These researchers also showed variations in ion release among different amalgam brands. Additionally, in the current study, copper release levels were not related to mercury – an observation similar to the results obtained by Ferracane *et al.*²³⁾. Nonetheless, although the amount of mercury was lower than the WHO guideline, clinicians and patients must exercise caution and take measures to minimize undesirable mercury release.

In our study, the amount of tin ions released from the two types of amalgam (SDI: 4.24 $\mu\text{g}/\text{ml}$; Valiant Ph.D.: 7.00 $\mu\text{g}/\text{ml}$) was much lower than the figure recommended in WHO guideline²⁴⁾. According to the WHO guideline, the most common adverse effect of tin on humans is acute gastric irritation. However, to date, no evidence of adverse effects associated with chronic exposure to tin has been reported. Meanwhile, copper is an essential nutrient for humans, and the recommended minimum amount is 0.9 mg/day for both adult men and women. In our current study, the amounts of copper ions released from the two types of amalgam (SDI: 0.17 $\mu\text{g}/\text{ml}$; Valiant Ph.D.: 1.23 $\mu\text{g}/\text{ml}$) were well within the range recommended by the guideline of the National Academy of Sciences' Food and Nutrition Board (NASFNB)²⁵⁾, *i.e.*, no more than 10 mg/day.

Colorimetry is a branch of color science and is based on the digital expression of the color perceived from an object^{3,26,27)}. The American Dental Association recommends the use of CIE Lab color differential system¹⁸⁾ in which all colors in nature are obtained through the blending of three basic colors –

red, blue, and green – in various proportions. Canay and Cehreli²⁸⁾ found that after bleaching with CP, color changes were noted for the tested composites. However, differences among the restorative materials were not statistically significant and that the color changes varied depending on the material. Similarly, in the present experiment, there were slight differences in color change and NBS units between the two types of amalgam. However, the ΔE^* level and NBS units were less than 3.3, which is clinically and statistically insignificant.

Campos *et al.*²⁹⁾ observed that treatment with 10 to 15% CP caused a reduction in the microhardness of various tested restorative materials: compomer, hybrid glass ionomer, and silver amalgam. However, the treatment did not alter the microhardness of the two composites tested (Charima and Durafill). In contrast, the current study showed that there were no statistical differences in VHN between the control and 10% CP bleaching groups in all amalgams. In other words, treatment with 10% CP for 14 days did not seem to cause any changes to amalgam surface hardness. In terms of the surface levels of amalgam metallic components, Rotstein *et al.*⁷⁾ demonstrated that 10% CP could cause significant changes. These changes appeared to be caused by enhanced oxidation, corrosion, and dissolution of the amalgam surface by CP and its byproducts. In stark contrast to the results of Rotstein *et al.*⁷⁾, Lee *et al.*²²⁾ suggested that there were no significant differences in surface roughness and morphology when they examined the effect of 35% CP. Concerning the results of Lee *et al.*²²⁾, AFM used in the current study did not detect any significant differences between the control and 10% CP bleaching groups on the two types of amalgam tested.

Based on the results of the present study, it could be concluded that a 10% CP bleaching agent appeared to have no significant effects on dental amalgam surface. This was because by means of ICP-AES, automatic mercury analyzer, microhardness tester, colorimeter, and AFM, we failed to observe any critical changes on the dental amalgam surface by 10% CP bleaching agent – either physically or chemically.

In terms of metal ion release, the bleaching agent caused more copper, tin, and mercury ions to be released into the solution as compared to distilled water. However, the concentrations of the metal ions released were lower than the guidelines recommended by WHO and NASFNB. Therefore, further studies that entail *in vivo* experiments and clinical trials are needed for a more precise evaluation of the present *in vitro* study's results. If verified to be safe for human use, the present 10% carbamide peroxide concentration could then be applied in bleaching agents for clinical use.

REFERENCES

- 1) Haywood VB, Heymann HO. Nightguard vital bleaching. *Quintessence Int* 1989; 20(3): 173-176.
- 2) Fasanaro T. Bleaching teeth: History, chemicals, and methods used for common tooth discolorations. *J Esthe Dent* 1992; 4(3): 71-78.
- 3) Goo DH, Kwon TY, Nam SH, Kim HJ, Kim KH, Kim YJ. The efficiency of 10% carbamide peroxide gel on dental enamel. *Dent Mater J* 2004; 23(4): 522-527.
- 4) Rotstein I, Walton RE. Bleaching discolored teeth: Internal and external. In: Principles and practice of endodontics, Walton RE, Torabinejad M (eds), WB Saunders, Philadelphia, 2002, pp.405-423.
- 5) Steinberg D, Blank O, Rotstein I. Influence of dental biofilm on release of mercury from amalgam exposed to carbamide peroxide. *J Biomed Mater Res B Appl Biomater* 2003; 67(1): 627-631.
- 6) Hummert TW, Osborne JW, Norling BK, Cardenas HL. Mercury in solution following exposure of various amalgams to carbamide peroxides. *Am J Dent* 1993; 6(6): 305-309.
- 7) Rotstein I, Mor C, Arwaz JR. Changes in surface levels of mercury, silver, tin, and copper of dental amalgam treated with carbamide peroxide and hydrogen peroxide *in vitro*. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 1997; 83(4): 506-509.
- 8) Ulukapi H, Benderli Y, Ulukapi I. Effect of pre- and postoperative bleaching on marginal leakage of amalgam and composite restorations. *Quintessence Int* 2003; 34(7): 505-508.
- 9) Schemehorn B, Gonzalez-Cabezas C, Joiner A. A SEM evaluation of a 6% hydrogen peroxide tooth whitening gel on dental materials *in vitro*. *J Dent* 2004; 32(1): 35-39.
- 10) Bailey SJ, Swift EJ Jr. Effects of home bleaching products on composite resins. *Quintessence Int* 1992; 23(7): 489-494.
- 11) Haywood VB. Greening of the tooth-amalgam interface during extended 10% carbamide peroxide bleaching of tetracycline-stained teeth: A case report. *J Esthet Restor Dent* 2002; 14(1): 12-17.
- 12) Rotstein I, Dogan H, Avron Y, Shemesh H, Steinberg D. Mercury release from dental amalgam after treatment with 10% carbamide peroxide *in vitro*. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 2000; 89(2): 216-219.
- 13) Lopes GC, Bonissoni L, Baratieri LN, Vieira LC, Monteiro S Jr. Effect of bleaching agents on the hardness and morphology of enamel. *J Esthet Restor Dent* 2002; 14(1): 24-30.
- 14) Horasawa N, Takahashi S, Marek M. Evaluation of corrosion degradation of amalgams by immersion and fracture test. *Dent Mater J* 2003; 22(4): 452-459.
- 15) Lim SD, Takada Y, Kim KH, Okuno O. Ions released from dental amalgams in contact with titanium. *Dent Mater J* 2003; 22(1): 96-110.
- 16) Koike M, Ferracane JL, Fujii H, Okabe T. Evaluation of the amalgamation reaction of experimental Ag-Sn-Cu alloys containing Pd using a mercury plating

- technique. *Dent Mater J* 2003; 22(3): 280-291.
- 17) Combe EC. Notes on dental materials, 6th ed, Churchill Livingstone, Edinburgh, 1992, pp.100-107.
 - 18) Council on Dental Materials and Devices. Revised American Dental Association specification no. 12 for denture base polymers. *J Am Dent Assoc* 1975; 90(2): 451-458.
 - 19) Shotwell JL, Razzoog ME, Koran A. Color stability of long-term soft denture liners. *J Prosthet Dent* 1992; 68(5): 836-838.
 - 20) Park HJ, Kwon TY, Nam SH, Kim HJ, Kim KH, Kim YJ. Changes in bovine enamel after treatment with a 30% hydrogen peroxide bleaching agent. *Dent Mater J* 2004; 23(4): 517-521.
 - 21) World Health Organization. Inorganic mercury, environmental health criteria 118, WHO/ICPS, Geneva, 1991, pp.108-110.
 - 22) Lee YE, Jung CW, Choi YE, Song KB. Effect of 35% carbamide peroxide on surface of dental amalgam: Release of metal ions and morphological change. *J Korean Acad Dent Health* 2004; 28(4): 524-535.
 - 23) Ferracane JL, Mafiana P, Cooper C, Okabe T. Time-dependent dissolution of amalgams into saline solution. *J Dent Res* 1987; 66(8): 1331-1335.
 - 24) World Health Organization. Guidelines for drinking-water quality, 2nd ed, Vol 1, WHO, Geneva, 1993, pp.388-389.
 - 25) National Academy of Sciences' Food and Nutrition Board. Food and Nutrition Board Recommended Dietary Allowances, 10th ed, National Academy Press, Washington DC, 1989, pp.1-9.
 - 26) Kanie T, Arikawa H, Fujii K, Ban S. Adhesive strength of paint-on resins to crown and bridge composites. *Dent Mater J* 2004; 23(4): 441-446.
 - 27) Fujita M, Kawakami S, Komatsu H, Sano H. Translucency and characteristics of newly developed polymer-based dental tooth coating material. *Dent Mater J* 2005; 24(1): 111-116.
 - 28) Canay S, Cehreli MC. The effect of current bleaching agents on the color of light-polymerized composites *in vitro*. *J Prosthet Dent* 2003; 89(5): 474-478.
 - 29) Campos I, Briso AL, Pimenta LA, Ambrosano G. Effects of bleaching with carbamide peroxide gels on microhardness of restoration materials. *J Esthet Restor Dent* 2003; 15(3): 175-183.