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Full Length Research Paper

Effects of different concentrations of bleaching agent on the micro hardness and shear bond strength of restorative materials – An *in vitro* study

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With the growing awareness of esthetics the use of bleaching agents are on the increase. Bleaching agents intended to be used at home affects the existing restorations. The aim of the present study was to evaluate the effect of bleaching with carbamide peroxide agents at concentrations 10 and 22% on the microhardness and shear bond strength of composites and compomers. For microhardness and shear bond strength evaluation, samples of both composite and compomer restorative materials were subdivided into control group (stored in artificial saliva), Experimental groups bleached with 10% CP and those bleached with 22% carbamide peroxide. Following 2 weeks of bleaching treatment, Vickers's hardness number was noted for each test specimen using a microhardness meter. For shear bond strength evaluation, the specimens were subjected to shear test under Instron universal testing machine. One way Analysis of Variance (ANOVA) was used for multiple group comparisons followed by Tukey's test for pair wise comparison. Bleaching with carbamide ceroxide at concentrations 10 and 22% caused reduction in hardness of composite restorative materials. However bond strength of both composite and a compomer restorative material was not affected following bleaching treatment. Hence it can be deduced that bleaching caused reduction in hardness of composite restorative materials.

Key words: Carbamide peroxide, bleaching, restoration, bonding, microhardness, composite resins, compomer.

INTRODUCTION

Tooth discoloration is a common problem affecting people of various ages and it can occur in both primary and permanent teeth (Unlu et al., 2004) . It is becoming a greater concern as more emphasis is being placed on esthetics. Esthetics, by definition, is the science of beauty: that particular detail of an animate or inanimate object that makes it appealing to the eye (Arens, 1989). In the modern civilized cosmetically conscious world well contoured and well aligned white teeth set the standard for beauty. Such teeth are not only considered attractive, but are also indicative of nutritional health, self esteem, hygienic pride and economic status (Arens, 1989). With the growing awareness of the esthetic options available, there is a greater demand for solutions to unsightly problems such as food staining, fluorosis and tetracycline staining (Sung et al., 1999).

Before the mid - 1980's various difficult, technique sensitive and potentially invasive procedures such as veneers and crowns were used. In recent years the demand for esthetic dentistry has grown, and simple, fast, in - office and at - home bleaching procedures have been popular and successful (Christensen, 1997). Tooth bleaching may involve internal bleaching of non vital teeth, external bleaching of vital teeth in the office, or external bleaching of vital teeth using prosthesis at home (Yap and Wattanapayungkul, 2002). Various bleaching

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Figure 1. Restorative materials.

Figure 2. Bleaching gel.

agents used include superoxol that contains 30% hydrogen peroxide and sodium perborate for non vital bleaching. However these treatments have been reported to cause an increased incidence of cervical root resorption. Bleaching agents available for vital teeth include hydrogen peroxide in concentrations from 30 - 50% and that for night guard bleaching are 5 - 22% sodium perborate gels. However, in office hydrogen peroxide tooth bleaching systems, such as dehydration and acid etching of teeth, and the need for extreme are to avoid harm to the soft tissue from the caustic levels that is 30 - 35% hydrogen peroxide (Brantley et al., 2001). The introduction of carbamide peroxide at-home bleaching has created resurgence in the area of bleaching primarily because of its relative ease of application, the safety of the material used, the lower cost, its general availability to all socio - economic classes of patients, and the high percentage of successful treatment (Attin et al., 2004). Carbamide peroxide is used in deciduous dentition as well and its use in children is primarily for fluorosis and genetically yellow permanent teeth (Campos, 2003).Nevertheless concerns about this relatively recent treatment remains and some of them are related to its effects on restorative materials either with regard to their physical properties or their adhesion to the dental structures. The bleaching agent may have a varying influence on the restorative materials and may even deteriorate them. Materials with different monomer systems, such as composites and compomers may show varying response to bleaching agents. Estimation of hardness may indicate deteriorating effects on the restorative material. Further, the adhesion of the resin restoration to the tooth should be able to prevent microleakage and withstand the forces exerted during mastication (Atash and Abbele, 2005). The aim of the present study was to evaluate and compare the *in-vitro* effects of bleaching with carbamide peroxide agents at concentrations 10 and 22% on the microhardness and shear bond strength of compomers and composite.

METHODS

The present *in-vitro* study was carried out in the Department of Pedodontics and Preventive Dentistry, Bapuji Dental College and Hospital, Davangere, Karnataka, India in association with the Department of Textile, Bapuji Institute of Engineering and Technology, Davangere and the Department of Mechanical Engineering, Indian Institute of Science, Bangalore. Restorative materials used were Z100 composite and F2000 compomer (3M ESPE) (Figure 1). Carbamide peroxide bleaching gels at concentrations 10 and 22% (Polanight advanced tooth whitening system, SDI limited, Australia) were used (Figures 2 and 3).

Specimen preparation for microhardness evaluation

60 pellets were prepared with composite and compomer restorative materials of 30 each using brass molds of inner diameter 4 mm and a height of 2 mm. Brass molds were positioned on a Matrix strip placed on a glass plate and filled with each restorative material. The material was held under constant hand pressure using two glass slabs on either side prior to curing. Then the pellet was cured for 40 s with visible light curing unit (Figure 4) (Turker and Biskin, 2002). The pellets of each restorative material were further subdivided into 3 groups containing 10 samples each for the bleaching regime (Figure 5).

Sampling for shear bond strength evaluation

60 non carious premolars extracted for orthodontic reasons were selected for the study. Each tooth was embedded in acrylic resin exposing the buccal surface. Then the outer enamel surface was ground flat with the help of 320 grit sand paper to obtain a flat dentinal surface (Miguel, 2004). The dentin surface of each specimen was etched with 35% phosphoric acid for 15 s, washed and blotted dry. Two coats of single bond (Adper) was applied over each of the specimens and light cured for 20 s. A cylindrical Teflon mold measuring 2 mm in diameter and 4 mm in height was used to build the composite and compomer cylinders on the dentinal surface Figure 6. The restorative materials were filled in a two layer increment technique, each layer being light cured separately for 40 s using light cure unit (Miguel, 2004). 30 samples of each restorative material were subdivided into 3 groups of 10 each

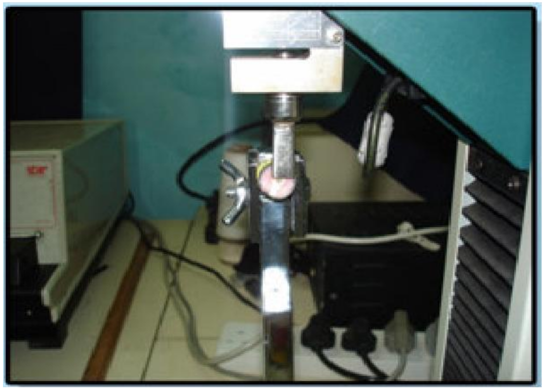

Figure 3. Specimen mounted on custom made jig, load applied at a cross head speed of 0.5 mm/min using a blade parallel to the dentin surface until the specimen shears.

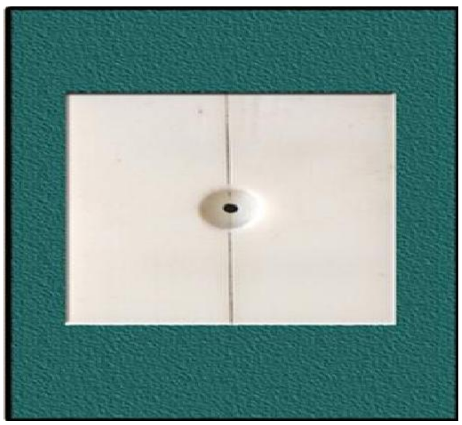

Figure 5. Teflon mold for preparation of shear bond strength specimens.

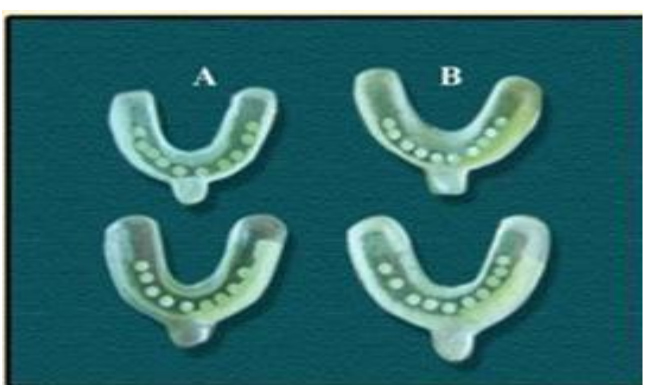

Figure 7. (A) Composite and (B) Compomer pellets subjected to bleaching treatment before microhardness evaluation.

(Figures 9 and 10).
The specimens for both microhardness and shear bond strength evaluation assigned for the bleaching regimes are as follows:

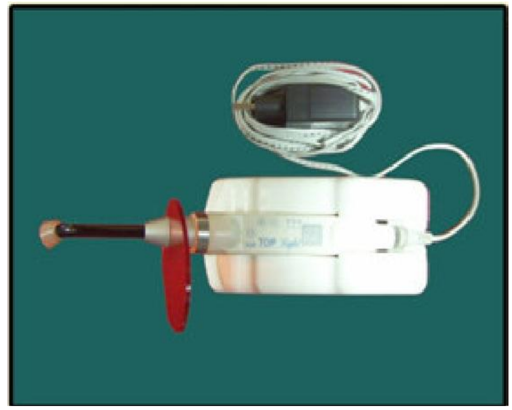

Figure 4. Light curing kit.

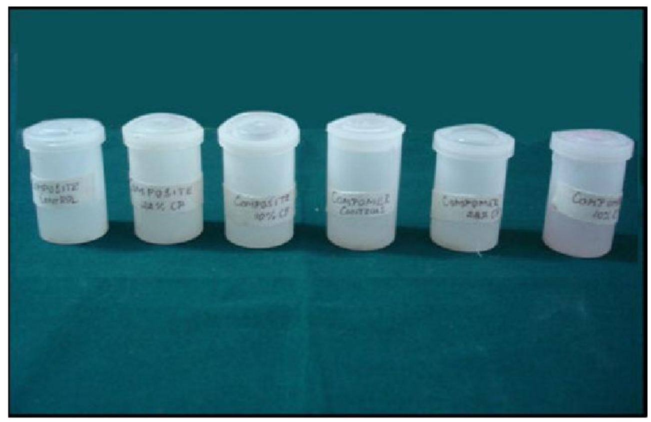

Figure 6. Composite and compomer pellets stored in plastic containers before microhardness evaluation.

A. ***Composite group:***
Group – I (Control) - stored in artificial saliva
Group – II (Experimental) - bleached with 10% carbamide peroxide
Group – III (Experimental) - bleached with 22% carbamide peroxide

B. ***Compomer group:***

Group – IV (Control) - stored in artificial saliva
Group – V (Experimental) - bleached with 10% carbamide peroxide
Group – VI (Experimental) - bleached with 22% carbamide peroxide

Bleaching regime

The test specimens of the experimental group were bleached with carbamide peroxide gels at 10 or 22%, 8 h daily for a period of 2 weeks. For microhardness samples, custom made trays were used wherein a drop of bleaching gel was applied in 10 areas within the tray and the pellets were placed within the applied area (Figure 7). For shear bond strength samples, the bleaching gel was applied at the bonded interfaces. After treatment with carbamide peroxide gel, the test specimens were abundantly washed under running water, dried with absorbent paper, and immersed in artificial saliva for remaining 16 h of the day (Turker and Biskin, 2002). The test specimens from the control group were kept in artificial saliva all the time, with the saliva being changed daily.

Figure 8. Microhardness testing unit and the view of an indentation.

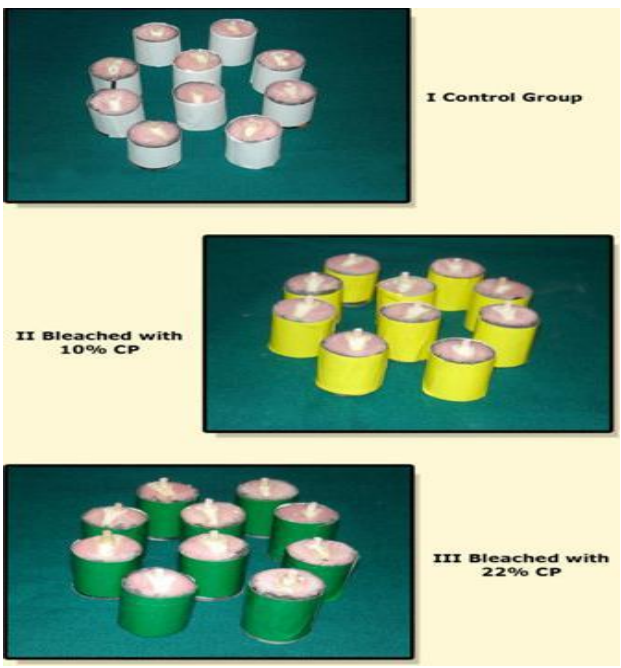

Figure 9. Color coded specimens of composite group for shear bond strength evaluation.

Figure 10. Universal testing machine.

Study of microhardness and shear bond strength

After 2 weeks of treatment, experimental and the control specimens were analyzed in a microhardness meter (Shimatzu HMV – 2000, Germany) Figure (8). The Vickers hardness measurement was taken of each test specimen in three different places previously established, applying a load of 25 g for 20 s. For each test specimen, the values were read referring to the size of the greater diagonal. The values were transformed into Vickers hardness number and the average of the values was calculated (Dahl, 2003). The shear bond test was performed using universal testing machine (Instron, USA) at a cross head speed of 0.5 mm min^{-1} in a compression mode using a blade parallel to the dentinal surfaces as the shearing element and the readings were noted. The values obtained were calculated in mega Pascal (Mpa) according to area of adhesion and subjected to statistical analysis (Goldstein, 1998).

Measurements were expressed as mean ± standard deviation and were compared between two groups by student's t test. One way ANOVA was used for multiple group comparisons followed by Tukey's test for pair wise comparison. P value less than 0.05 was considered for statistical significance.

RESULTS

The results and observations of microhardness and shear bond strength are summarized as follows:

Intergroup comparisons of microhardness evaluation

Table 1 represents comparison of microhardness of composite restorative material according to the treatment received; Group I (control) showed a statistically significant difference ($p < 0.01$) when compared with groups II and III, but the difference was not significant between groups II and III.

Table 2 represents comparison of microhardness of compomer restorative material according to the treatment received. There was no statistically significant difference between any of the groups compared.

Table 1. Comparison of microhardness between the composite groups.

Groups	Range (VHN)	Mean (VHN)	SD	Difference between groups		
				Groups compared	Mean difference	P - Value
Group I Stored in artificial saliva	53.4 – 59.1	55.8	1.9	I- II	8.7	<0.01,S
Group II Bleached with 10% CP	37.4 - 57.0	47.1	7.3	I - III	9.4	<0.01,S
Group III Bleached with 22% CP	35.8 – 53.3	46.4	7.2	II - III	0.7	NS

One way ANOVA; P < 0.01, Significant (S); P >0.05, not significant (NS).

Table 2. Comparison of micro hardness between the compomer groups.

Groups	Range (VHN)	Mean (VHN)	SD	Difference between groups		
				Groups compared	Mean difference	P-Value
Group IV Stored in artificial saliva	40.4 – 54.4	46.2	4.5	IV- V	1.2	NS
Group V Bleached with 10% CP	37.6 - 51.3	45.0	5.3	IV - VI	2.6	NS
Group VI Bleached with 22% CP	37.7 – 49.0	43.6	4.4	V - VI	2.4	NS

One way ANOVA; P < 0.01, Significant (S); P >0.05, Not significant (NS).

Table 3. Comparison of shear bond strength between the composite groups.

Groups	Range (Mpa)	Mean (Mpa)	SD	Difference between groups		
				Groups compared	Mean difference	P-Value
Group I Stored in artificial saliva	5.68 – 11.93	8.54	1.80	I- II	0.28	NS
Group II Bleached with 10% CP	7.37 - 10.5	8.26	0.86	I - III	0.5	NS
Group III Bleached with 22% CP	6.15 - 10.93	8.04	1.57	II - III	0.02	NS

One way ANOVA; P >0.05, not significant (NS); P < 0.001, highly significant.

Intergroup comparisons of shear bond strength evaluation

Table 3 shows comparison of shear bond strength values of composite restorative materials. There was no statistically significant difference in the shear bond strength values between the groups of which was stored in artificial saliva and those treated with carbamide peroxide at 10 and 22%.

Table 4 represents comparison of shear bond strength of compomer restorative material according to the treatment received. There was no statistically significant

Table 4. Comparison of shear bond strength between the compomer groups.

Groups	Range (Mpa)	Mean (Mpa)	SD	Difference between groups		
				Groups compared	Mean difference	P- Value
Group IV Stored in artificial saliva	3.56 - 6.28	4.95	0.9	VI - V	0.12	NS
Group V Bleached with 10% CP	3.34 - 6.21	4.83	0.88	IV - VI	0.24	NS
Group VI Bleached with 22% CP	3.34 - 6.46	4.71	0.96	V - VI	0.12	NS

One way ANOVA; P >0.05, not significant (NS); P < 0.001, highly significant (HS).

difference between any of the groups compared.

DISCUSSION

Tooth discoloration varies in etiology, appearance, localization, severity, and adherence to tooth structure. It may be classified as intrinsic, extrinsic, and a combination of both. Intrinsic discoloration is caused by incorporation of chromatogenic material into dentin and enamel during odontogenesis or after eruption. Exposure to high levels of fluoride, tetracycline administration, inherited developmental disorders, and trauma to the developing tooth may result in pre - eruptive discoloration. After eruption of the tooth, aging, pulp necrosis, and iatrogenesis are the main causes of intrinsic discoloration. coffee, tea, red wine, carrots, oranges, and tobacco give rise to extrinsic stain (Khin, 2000). In the past, the demand for esthetic dentistry has dramatically grown and so has the rapid development of new non restorative treatment for discolored teeth. Bleaching is a conservative treatment alternative for discolored teeth.

Since enamel and dentin are porous tissues, the scientific basis for bleaching vital teeth is sound (Basting et al., 2003). Clinicians all over the world have incorporated the vital bleaching into their profession and oxygenating agents like carbamide peroxide or hydrogen peroxide are used for effective bleaching (Garcia-Godoy et al., 2002).

A 10% carbamide peroxide bleaching agent is the most commonly used at-home bleaching product. A range of concentrations of carbamide peroxide available to the dental professional include 15, 20, 22 and 30% solutions (Khin, 2000) . It has been reported that whitening of teeth can be achieved faster with higher concentrations compared with the results for lower concentrations of carbamide peroxide (White et al., 2003). The mechanism of action of bleaching agents on tooth structures apparently is due to oxidation of enamel and dentin molecules causing changes in color. There is a need for prolonged contact of the agent with the dental structure to allow the oxidation process to take place. This contact also occurs between the agent and preexisting restorations, with the latter being exposed to the same condition (Hayacibara, 2004). Similarly any chemical softening resulting from bleaching has implications on the clinical durability of restorations (Atash and Abbele, 2005). So the present *in vitro* study was conducted with the objective to evaluate and compare the effects of different concentrations of bleaching agents on the microhardness and Shear bond strength of composites and compomers. Polanight advanced tooth whitening system used in this study is a neutral pH, high viscosity, crystal clear sustained release gel. It is available in both 10 and 22% concentrations of carbamide peroxide.

The bleaching protocol in the present study was designed to simulate treatment of teeth under cycling conditions of bleach and saliva exposure which is encountered under in situ conditions (Kudalkar and Damle., 1997). The control test specimens of restorative materials were stored in artificial saliva throughout and the experimental test specimens while they were not being “bleached” in order to simulate oral conditions as closely as possible. Artificial saliva comprised of Sodium chloride (NaCl) 0.4 g, Potassium chloride (KCl) 0.4 g, calcium chloride ($CaCl_2.H_2O$) 0.795 g, sodium-di-hydrogen phosphate ($NaH_2PO_4.H_2O$) 0.69 g, sodium sulfide ($Na_2S.9H_2O$) 0.005 g and distilled water 1000 ml. The pH was adjusted to 7 (Attin, 1996). it has been reported that the substances present in the composition of saliva may act as accelerators in degrading carbamide peroxide and may minimize its adverse effects by means of the salivary remineralizing potential. Furthermore, the salivary biofilm appears to control the adhesion of oral bacteria to the restorative materials after the lightening treatment (Turker and Biskin, 2002). The time frame for use

use of the agents in this study was based on the manufacturer's instructions which ranged from 6 - 8 h for a two week period.

Microhardness

Hardness is defined as the resistance of a material to indentation or penetration. As hardness is related to a material's strength, proportional limit and its ability to abrade or to be abraded by opposing dental structures/materials, any chemical softening resulting from bleaching has implications on the clinical durability of restorations (Atash and Abbele, 2005). Surface deterioration of composite and compomer restorative materials bleached with carbamide peroxide were evaluated. The restorative materials were placed in a brass mold which ensured standardization of the shape and size of each pellet. The setting material was covered with matrix strips on either side to avoid early moisture contamination and was held under constant hand pressure using glass slabs on either side in order to obtain polished surface. This polished flattened surface was essential to prevent distorted indentation for hardness measurement on any material (Anusavice, 2003). The VHN for each pellet was evaluated using surface microhardness tester (Shimatzu HMV- 2000, Germany). Vickers hardness measurements fulfill the requirements of the standard test method of materials as defined by American society for testing and materials (Garcia - Godoy, 1993).

The microhardness tester was standardized prior to indentation on each pellet. The testing parameter of 25 g for 20 s initiated no cracks on the surface of the material, thereby providing a size of indentation that allow measurement of surface hardness of these materials (Turker and Biskin, 2002). The results of this study showed that the composite restorative materials when submitted to bleaching with carbamide peroxide gel at 10 and 22% showed significant reduction in hardness when compared to the control group, which was stored in artificial saliva. On the contrary, the reduction in the hardness showed by the compomer group was not statistically significant. This shows that the effect of carbamide peroxide gels may be material dependent. However, the impact of low concentrated carbamide peroxide gels on surface microhardness of composite restorative materials has showed a varied response in the previous studies. It has been reported to increase (Cooley and Burger, 1991) or decrease (Bailey and Swift, 1992) or it may remain unchanged (Nathoo et al., 1994; Attin et al., 2004). Such wide variations in data suggest that some tooth colored restorative materials may be more susceptible to alterations and few bleaching agents are more likely to cause those alterations. The latter may be attributed to the differences in pH between bleaching agents (Attin et al., 2004). Further, the use of remineralization solutions could inhibit the decrease in Microhardness caused by the bleaching agents. Remineralization exists in saliva substitutes that contain calcium and phosphate ions, (Hayacibara, 2004) such as artificial saliva used in this study.

Shear bond strength

Shear stress is considered to be more representative of the clinical situation. Bond strength is the force per unit area that is required to break a bonded assembly with failure occurring in or near the adhesive/ adherend interface (Farik et al., 2002). For shear bond strength evaluation, a total of 60 non carious premolars extracted due to orthodontic reasons were selected and stored in distilled water before and during the study period, since the distilled water does not affect the dentin permeability and bond strength compared to saline. The buccal surfaces of all the 60 teeth were considered in this study, because this surface allowed the shearing force to be exactly perpendicular to the bonded specimen (Shinohara, 2004).

The dentin surface was etched with 35% phosphoric acid for 15 s.The scotch bond™ multi-purpose etchant is a versatile system which is recommended for bonding all classes of restorations. "Single Bond", a fifth generation (type 2) adhesive, was used having both primer and adhesive in one bottle. The same bonding agent was used for all the groups according to the manufacturer's instructions. The main reason to use an adhesive was to facilitate the penetration of composite into the etched dentin surface to provide a better bond to tooth structure.

Single bond adhesive was found to provide up to 97% retention rate. Few studies have reported that carbamide peroxide does not interfere in the adhesion of composite resin with dentin. The reaction of carbamide peroxide is immediate, and it is probable that the residues of hydrogen peroxide leach rapidly. Furthermore, dentin is a porous substrate, and the peroxide residues of carbamide peroxide may release the oxygen more easily to the dentin tubules than to enamel.

In this study, although there was reduction in bond strength of the groups treated with Carbamide peroxide at 10 and 22% for both composite and compomer restorative materials when compared with the control groups that were stored in artificial saliva, it was not statistically significant. This is in agreement with the previous studies (Kudalkar and Damle, 1997). It is conceivable that storage in saliva might have modified or attenuated the hydrogen peroxide impact by the formation of a surface protection salivary layer on the restorative material (Atash and Abbele, 2005).

Reduction in resin – to - dentin bond strengths after bleaching treatment also has been reported. Toko and Hisamitsu reported that the reduction of adhesion can be related to the alteration of the dentin organic content.

However, these *in-vitro* studies do not consider many of the clinical variables present when bleaching teeth *in vivo*. Factors such as dentin thickness, sclerosis of dentinal tubules, and most importantly natural saliva are the clinical variables that can significantly influence the effect of bleaching treatment on dentin (Goldstein, 1998). Most of the studies have reported that major changes are not present in tooth structure or in common restorative materials when immersed in the currently available tooth bleaching solutions, and that the bleaching materials are effective clinically (Dahl, 2003; Garcia-Godoy et al., 2002; White et al., 2003). Although composites showed a significant reduction in hardness after bleaching, compomers revealed no change in hardness following bleaching. However, no significant difference in shear bond strength was observed between the control and bleached groups for both the restorative materials. Therefore, the use of at - home bleaching employing carbamide peroxide agents does not affect the adhesion of bonded restorations to tooth structures.

Conclusion

Following conclusions were drawn from the study:

- Carbamide peroxide bleaching gel at concentrations 10 and 22% caused a reduction in hardness of composite restorative material whereas that of compomer restorative material remained unchanged.
- Bleaching had no effect on the bond strength of composite as well as compomer restorations to the tooth structure. We advice further *in-vivo* studies to evaluate the other associated effects of bleaching on restorations such as the surface roughness, microleakage and color changes in tooth colored restorative materials which can significantly enhance the findings of the present study.

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