

Effects of Time-Elapsed Bleaching on the Surface and Mechanical Properties of Dentin Substrate Using Hydrogen Peroxide-Free Nanohydroxyapatite Gel

Aftab Ahmed Khan¹, Abdulaziz Abdullah AlKhureif¹, Manal Almutairi², Abrar Nasser bin Nooh³, Saeed Awod bin Hassan⁴, Yasser M Alqahtani⁵

¹Dental Biomaterials Research Chair, College of Applied Medical Sciences, King Saud University, Riyadh, Saudi Arabia; ²Pediatric Dentistry and Orthodontics Department, College of Dentistry, King Saud University, Riyadh, Saudi Arabia; ³Restorative Dentistry Department, College of Dentistry, Princess Nourah Bint Abdulrahman University, Riyadh, Saudi Arabia; ⁴Restorative Dental Sciences Department, College of Dentistry, King Khalid University, Abha, Saudi Arabia; ⁵Restorative and Esthetic Dentistry Department, Ministry of Health, Abha, Saudi Arabia

Correspondence: Aftab Ahmed Khan, Dental Biomaterials Research Chair, College of Applied Medical Sciences, King Saud University, Room # 2307, Building # 24, Riyadh, 11451, Saudi Arabia, Email aftkhan@ksu.edu.sa

Introduction: There is a critical need to address concerns surrounding the potential impact of bleaching gels specifically on the tooth substrate. Therefore, this laboratory investigation aimed to assess the impact of a hydrogen peroxide (HP)-free bleaching (HiSmile™) in comparison to an HP-based bleaching (Opalescence Regular™) on the surface and mechanical characteristics of tooth substrate.

Methods: Sixty sound human premolar teeth were sectioned to produce dentin fragments and divided into two primary groups based on the bleaching agent used. Each group was subdivided into three subgroups (n = 10) per distinct bleaching regimens: (T₁) fragments underwent a 7-day immersion in distilled water at 37°C without any bleaching treatment, (T₂) fragments underwent a 7-day immersion in distilled water at 37°C, with the application of bleaching gel occurring on the seventh day for 10 minutes, and (T₃) fragments underwent a bleaching regimen for seven consecutive days, each session lasting for 10 minutes. The initial and final evaluations of surface roughness, nano-hardness, and elastic modulus were performed. Following the bleaching regimens of T₃, a composite stub was fabricated on the dentin fragments for the shear bond strength (SBS) test. Statistical testing was accomplished using the analysis of variance (P < 0.05).

Results: HP-based bleaching gel showed significant differences between measurement intervals in surface roughness, elastic modulus, and SBS parameters (P < 0.05). In contrast, HP-free bleaching gel showed insignificant differences within the group (P > 0.05). The SBS between dentin-composite was significantly affected with the use of HP-based bleaching gel, while HP-free bleaching gel showed insignificant difference between measurement intervals. The qualitative validation of the treatment's impact was further demonstrated using the scanning electron microscopy.

Conclusion: The findings suggest that the bonding stability of composite restorations to dentin may be compromised after bleaching with an HP-based gel, whereas immediate bonding procedures can be safely conducted following the application of an HP-free bleaching gel.

Keywords: dental bleaching gel, dentin, hydrogen peroxide, mechanical properties, phthalimidoperoxycaproic acid, nanohydroxyapatite

Introduction

In the last decade, there has been a considerable increase in the use of over-the-counter (OTC) tooth-bleaching products among the general population. The increased awareness of dental aesthetics could be the reason behind the increased impetus of OTC bleaching products. In 1989, the first tooth-bleaching product was introduced by Haywood and Heymaan.¹ Later, with advancements in technology and materials, bleaching products gained popularity.² At present, a wide variety of OTC bleaching products such as Opalescence Go™, iWhite™ Dark Stains Whitening Kitare and Opalescence PF™ are available in the market.^{3–5}

Cosmetic dentistry mostly involves tooth whitening and esthetic restorative procedures. Due to rising awareness about oral hygiene and the stigma associated with discolouration of teeth, a wide range of bleaching systems are available with varying constituents of bleaching agents.⁶ However, most of the formulations consist of potent oxidizing agents such as carbamide peroxide (CP) or hydrogen peroxide (HP).⁷

Despite tooth whitening being considered a viable and efficient procedure, it can have some negative consequences on the enamel and dentin, like variations in surface morphology, composition, micro-hardness, and surface abrasion.^{4,8} When applied to the tooth surface, HP, the main constituent of the bleaching product generates free radicals that interact with the organic pigmented compounds in the teeth (ie, chromogens), and thereby lightening the color of teeth. However, these free radicals also penetrate inside enamel and dentine,⁹ which hinders the polymerization of the resinous material.² The decomposition of HP interacts with the organic components of the teeth (proteins and lipids) resulting in organic loss and weakening/damaging of the tooth structure.¹⁰ This damage to the tooth structure also influences the optical properties of dental hard tissues.¹¹ These studies were conducted on extracted human teeth using a low concentration of HP or CP-based home bleaching gels. Since dental restorations often coincide with bleaching procedures, recommendations have been proposed to postpone the bonding process and allow for a waiting period of up to 10 days. This delay is advised to facilitate the restoration of lost surface minerals in the tooth substrate.^{12,13}

The impact of bleaching on the ability of composite resin restorations to adhere to tooth structure has been thoroughly studied on in-office bleaching. A study by Unlu et al, found that bonding on the tooth surface of enamel is reduced to 25–60% after bleaching.¹⁴ Alternative investigations where bonding was performed after 24 h of bleaching showed reduced bonding strength on the tooth surface with 40% HP compared to the CP bleaching product.^{15,16}

A new bleaching product has been introduced, containing organic compound, ie, phthalimidoperoxycaproic acid (PAP) with additives. Several ingredients such as potassium to protect dentine, nanohydroxyapatite to overcome the mineral loss and dentin softening, and potassium citrate as a buffer to maintain the pH level to normal were added to PAP to formulate PAP+. Using PAP+ prevents calcium chelation of enamel and dentin.^{17,18} In PAP+ bleaching, while the oxidation process decolorizes chromogens, the epoxidation of molecules with conjugated double bonds occurs without the formation of free radicals.¹⁷

In our previous study, we conducted a comparative analysis between peroxide-free and peroxide-containing bleaching gels to examine their respective influences on the mechanical properties of restorative composites.¹⁸ Our findings revealed that the utilization of peroxide-free gel did not yield noteworthy alterations in either the surface or bulk characteristics of the restorative composites. Considering the observed negligible impact on the mechanical characteristics of restorative composites in our prior investigation, the current investigation is predominantly oriented towards evaluating the influence of these two distinct categories of bleaching gels, each characterized by unique bleaching mechanisms, on the mechanical properties of the dentin structure. As OTC bleaching procedures have become popular, the present study targeted to ascertain the influences of two different home bleaching systems on dentin's surface and mechanical properties and the effect of these changes on the shear bond strength (SBS) of resin composite to dentin. It was hypothesized that there would be insignificant difference between the two bleaching systems across the measuring intervals. Additionally, it was postulated that the SBS of the resin composite to dentin would not differ significantly due to the use of the two bleaching systems at various measuring intervals.

Materials and Methods

Sample Preparation

Sixty human upper premolar teeth extracted for orthodontic reasons were collected from the Department of Orthodontics, King Khalid University Hospital, Riyadh; Saudi Arabia. Informed consent was obtained from patients aged 18–24, with a mean age of 21 years for utilizing their extracted sound and intact teeth for research. Teeth with caries, restorations and cracks were excluded from the research. This research adhered to the principles outlined in the Declaration of Helsinki and received approval from the Institutional Review Board at the College of Medicine, King Saud University, Saudi Arabia (Ref No: E-22-7366). The teeth were divided into two main groups according to the bleaching gel used. Each group was further divided into three subgroups (n=10) according to the bleaching regimens (Table 1). After amputation

Table 1 Bleaching Regimes for Study Subgroups

Bleaching Material	Composition	Study Subgroup	Bleaching Regime
None	–	T ₁	Dentin fragments specimens were stored in distilled water at 37°C for 7 days without any bleaching regime.
HP-based and HP-free	See composition footnotes [1],[2]	T ₂	Dentin fragment specimens were stored in distilled water at 37°C for 7 days. On the 7 th day, bleaching was applied as mentioned in subsection “bleaching regime”, and air-dried for 5 minutes.
HP-based and HP-free	See composition footnotes [1],[2]	T ₃	Dentin fragment specimens went through a bleaching as mentioned in subsection “bleaching regime” for seven consecutive days.

Notes: [1] HP-based: Glycerin, water, carbamide peroxide, xylitol, carbomer, PEG-300, sodium hydroxide, EDTA, potassium nitrate, sodium fluoride. [2] HP-free: Sorbitol, water, phthalimoperoxycaproic acid, propylene glycol, glycerin, potassium nitrate, PEG-8, hydroxyapatite, cellulose gum, hydroxyethylcellulose, xanthan gum.

of the roots, all teeth were stored in a 0.1% aqueous thymol solution for a week. Next, the teeth were cleaned and polished using a pumice powder with a rubber cup affixed to a low-speed handpiece. The crowns were affixed in wax and divided longitudinally along the mesiodistal axis using a dual-sided diamond disk attached to a low-speed handpiece. The enamel layer of the crowns was removed, and each half of the crown was further divided into incisal, mesial, distal, and cervical sections to produce square specimens measuring 4 mm in width and 2 mm in height. The standardized protocols for sectioning and finishing of the teeth were employed. All dentin fragments were sectioned using precision equipment to minimize surface damage and then subjected to a finishing process to ensure uniformity. This process yielded a total of 120 fragments, with 60 samples from the buccal face and 60 from the palatal face. Sixty teeth were allocated for the evaluation of pre- and post-bleaching effects on surface roughness, nano hardness, and elastic modulus, with the remaining sixty designated for assessing shear bond tests. Each dentin fragment was embedded in a self-cure acrylic resin using a polyvinyl chloride pipe having a 12 cm diameter.

Before the implementation of the bleaching regimen, baseline measurements were conducted to assess surface roughness, nano hardness, and elastic modulus of the dentin fragments as elaborated upon below. Since surface roughness, nano hardness and elastic modulus are non-destructive techniques, the same set of dentin fragments was utilized for both pre-and post-bleaching evaluations.

Bleaching Regimen

Two different home-based bleaching systems were assessed: Opalescence Regular™ (Ultradent Products, Inc., USA), a widely used bleaching product containing HP as the active agent; and HiSmile™ (Hismile Pty Ltd., Australia), which is marketed as a HP-free bleaching gel. To ensure consistency in the bleaching application, the bleaching gels were administered onto the dentin fragment surfaces using a syringe, thus standardizing the amount of gel for each application. In both study groups, the bleaching gel was evenly distributed on the samples and allowed to dwell for 10 min in each session. Subsequently, the samples were meticulously washed with a syringe having distilled water for 30 seconds. Following each bleaching session, the samples were placed in an incubation environment of distilled water at a temperature of 37°C until the subsequent bleaching session.

Surface Roughness Appraisal

Following the initial assessment of surface roughness (n = 10), dentin fragment specimens from each subgroup of both study groups were subjected to final measurements using a 3D optical non-contact surface profilometer (ContourGT, Bruker, Campbell, CA, USA). The roughness average (Ra), which represents the arithmetical mean of all absolute distances between the roughness profile and the centre line within the measuring length, was determined using non-contact scanning white light interferometry. The measurements were conducted using a standard objective camera with a 5× magnification. The precision of the measurements, control of surface roughness parameters, and creation of surface

roughness maps were facilitated by the Vision64 (v 5.30) application software (Bruker, Campbell, CA, USA). Both initial and final readings were obtained from the same predetermined region of interest.¹⁹

Nano Hardness and Elastic Modulus Evaluation

Following the initial measurements of nano hardness and elastic modulus (n=10), subsequent nano hardness and elastic modulus tests were conducted to obtain final readings using a nanomechanical device (UMT1, Bruker, CA, USA) equipped with a Berkovich diamond indenter nanotip. The tests were performed at a controlled room temperature of $21 \pm 1^\circ\text{C}$, with loading and unloading rates set at 0.5 mN/s and a dwell time of 10 seconds. The maximum applied load was fixed at 20.0 mN. Both the initial and final readings were obtained from the same predefined region of interest. The nano hardness and elastic modulus values were determined in gigapascals (GPa) using proprietary software associated with the testing device.

SEM Evaluation

To evaluate the morphological changes due to bleaching protocols on dentin substrate, SEM (JSM-6360LV, JEOL, Japan) at 15 kV was employed. The pictograms of the study groups were obtained at 500x.

SBS Analysis

To evaluate the effect of bleaching protocols on SBS, the remaining sixty teeth were divided equally into two groups based on the type of bleaching gel used. Each bleaching group was further subdivided into three subgroups, with ten samples in each subgroup (n = 10), according to the specific treatment protocol (ie, T₁, T₂ & T₃) employed.

After completion of treatment protocols as mentioned in Table 1, the dentin fragment specimens were air-dried for 5 min. Next, a composite stub of 3 mm in diameter and 2 mm in height was fabricated on a dentin fragment using a silicon mold and micro-hybrid composite material of A2 shade, namely Filtek Z250 (3M ESPE, St. Paul MN, USA). A gentle pressure was applied with a hand instrument to ensure proper flow and adaptation of the composite material to the dentin fragment. The composite stub was light-cured for 40 seconds at an intensity of 650 mW/cm² using Elipar Freelight 2 (3M ESPE, Seefeld, Germany). Before composite stub fabrication, a single coat of dentin bonding agent, Harvard Bond SE Mono (Harvard Dental International, GmbH, Hoppegarten, Germany), was applied on the dentin fragment surface using a micro brush. The coating was air-dried for 20 seconds and light-cured for 20 seconds.

A universal testing machine (Model no. 3369 Instron, Canton, MI, USA) equipped with a 30 kN load cell and operating at a crosshead speed of 1.0 mm/min was employed for the testing procedure. The proprietary software, Bluehill ver. 2.4, automatically captured and recorded the maximum load at fracture and the corresponding SBS values in megapascals (MPa).

Fractographic Analysis

Following the completion of SBS testing, the failure mode was examined utilizing a stereomicroscope (Nikon SM2-10, Tokyo, Japan) with a magnification of 15 \times . The observed failure modes were subsequently classified as cohesive failure within the dentin, adhesive failure at the dentin–composite interface, or cohesive failure within the composite.

Statistical Analysis

Data analysis was conducted employing SPSS software (version 28.0 for Windows, SPSS, Chicago, IL, USA) with a significance level (α) set at 0.05. The assumption of normality was assessed using the Kolmogorov–Smirnov test, while the equality of variances was examined using Levene's test. To evaluate the impact of both the bleaching agent and treatment time, as well as their interactions, on the SBS, a two-way analysis of variance (ANOVA) was employed.

Results

Table 2 presents the surface roughness values of the study groups according to bleaching regimens. The influence of HP-based bleaching on surface roughness was observed insignificant at T₁ and T₂. However, a significant difference was observed between the initial and the final readings at T₃ ($p < 0.05$). In contrast, HP-free bleaching showed minimal damage to the dentin

Table 2 Means and Standard Deviations Surface Roughness (Ra, μm) of the Study Groups at Different Measurement Intervals

Bleaching gel	Measurement	Surface Roughness (μm)		
		T ₁	T ₂	T ₃
HP-based	Initial	0.98 \pm 0.15	1.02 \pm 0.17	0.96 \pm 0.25 ^A
	Final	1.00 \pm 0.12	1.11 \pm 0.22	1.51 \pm 0.27 ^A
HP-free	Initial	0.95 \pm 0.20	0.93 \pm 0.24	1.05 \pm 0.17
	Final	1.01 \pm 0.18	1.13 \pm 0.09	1.20 \pm 0.15

Notes: N.B. Uppercase letter "A" shows significant difference within the treatment groups of the same material. Groups without superscript letters did not show statistically significant differences when compared to each other. Statistical comparisons were conducted between all groups (T₁, T₂, T₃) within each bleaching gel.

surface. None of the bleaching regimens showed a significant difference in surface roughness between the initial and final readings ($p > 0.05$) using HP-free bleaching gel. Nonetheless, surface roughness gradually increased from T₁ to T₃, irrespective of the bleaching gel used. Figure 1 presents pictograms of the study groups following bleaching regimens.

Table 3 presents the nano hardness of the study groups according to the bleaching regimens followed. The influence of HP-based bleaching on nano hardness was observed to be insignificant in all bleaching regimens, ie, T₁, T₂ and T₃ ($p > 0.05$). Likewise, an insignificant difference in nano hardness was observed for HP-free bleaching between the initial and the final readings ($p > 0.05$). Nonetheless, nano hardness gradually decreased from T₁ to T₃, irrespective of the bleaching gel used.

Table 4 presents the elastic modulus of the study groups according to bleaching regimens. The influence of HP-based bleaching on elastic modulus was observed to be insignificant at T₁ and T₂ bleaching regimens. However, a significant difference was observed between the initial and the final readings at T₃ ($p < 0.05$). HP-free bleaching showed minimal damage to the elastic modulus of the dentin surface. None of the bleaching regimens showed a significant difference in elastic modulus between the initial and the final readings using HP-free bleaching gel ($p > 0.05$). Nonetheless, elastic modulus gradually decreased from T₁ to T₃, irrespective of the bleaching gel used.

Figure 2 depicts a graphical representation of the SBS of study groups following bleaching regimens. Among the HP-based bleaching group, the T₁ regimen (ie, without bleaching protocol) showed significant differences with T₂ and T₃ ($p < 0.05$). Additionally, the T₂ showed a significant difference in SBS with the T₃ bleaching regime ($p < 0.05$). In contrast, none of the regimens showed a significant difference using HP-free bleaching.

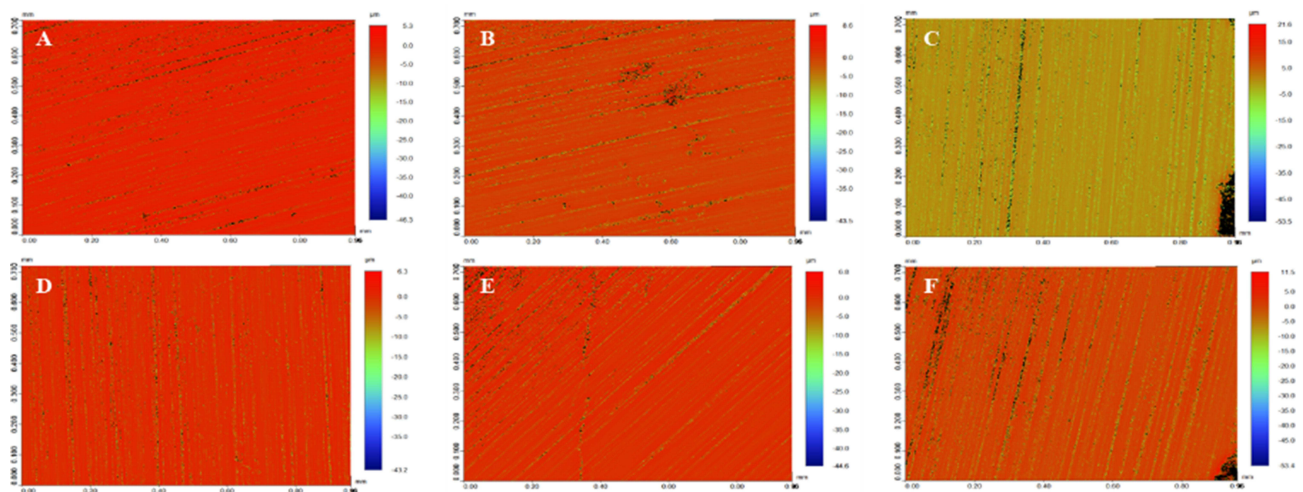


Figure 1 The post bleaching regimens 2D surface roughness profile images, (A–C): Dentin slabs using HP-based bleaching at T₁, T₂ & T₃, respectively and (D–F): Dentin slabs using HP-free bleaching at T₁, T₂ & T₃, respectively.

Table 3 Means and Standard Deviations Nano Hardness (GPa) of the Study Groups at Different Measurement Intervals

Bleaching gel	Measurement	Nano hardness (GPa)		
		T ₁	T ₂	T ₃
HP-based	Initial	3.49 ± 0.28	3.52 ± 0.41	3.51 ± 0.36
	Final	3.54 ± 0.36	3.43 ± 0.38	3.37 ± 0.41
HP-free	Initial	3.76 ± 0.53	3.71 ± 0.39	3.69 ± 0.44
	Final	3.71 ± 0.44	3.66 ± 0.43	3.61 ± 0.52

Notes: N.B. No statistical difference within the treatment groups at different measurement intervals.

Table 4 Means and Standard Deviations Elastic Modulus (GPa) of the Study Groups at Different Measurement Intervals

Bleaching Gel	Measurement	Elastic Modulus (GPa)		
		T ₁	T ₂	T ₃
HP-based	Initial	87.21 ± 6.74	88.62 ± 11.18	87.88 ± 6.75 ^A
	Final	86.55 ± 7.63	81.12 ± 8.14	73.78 ± 7.12 ^A
HP-free	Initial	85.11 ± 9.27	86.94 ± 10.41	85.58 ± 12.17
	Final	88.21 ± 8.57	83.87 ± 6.64	78.53 ± 8.35

Notes: N.B. Uppercase letter "A" shows significant difference within the treatment groups of the same material. Groups without superscript letters did not show statistically significant differences when compared to each other. Statistical comparisons were conducted between all groups (T₁, T₂, T₃) within each bleaching gel.

Figure 3 shows the scanning electron microscope (SEM) pictograms of the dentinal fragments. A smooth surface without pitting and irregularities was observed at the T₁ measurement interval using HP-based bleaching (Figure 3A). However, slight surface microstructural changes were noticed at T₂ measurement interval using HP-based bleaching (Figure 3B). A pitted surface with irregularities was observed at T₃ measurement interval using HP-based bleaching (Figure 3C). In contrast, HP-free bleaching exhibited unnoticeable surface microstructural changes at all-measurement intervals (Figure 3D–F).

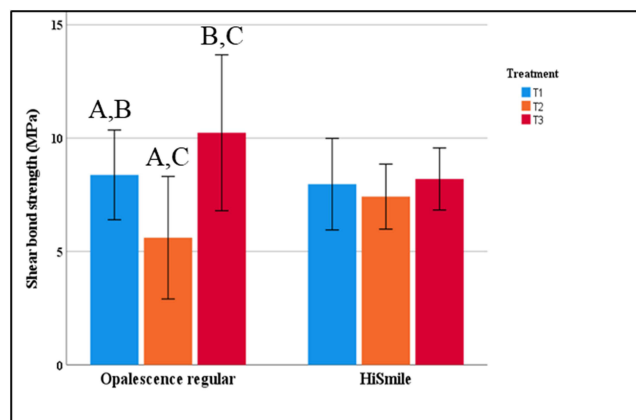


Figure 2 Comparison of mean SBS values among the bleaching gels used according to bleaching regimens. Same uppercase alphabets (ie, "A", "B", "C") represent a significant difference within the treatment groups for the same material depict significant difference within the treatment groups.

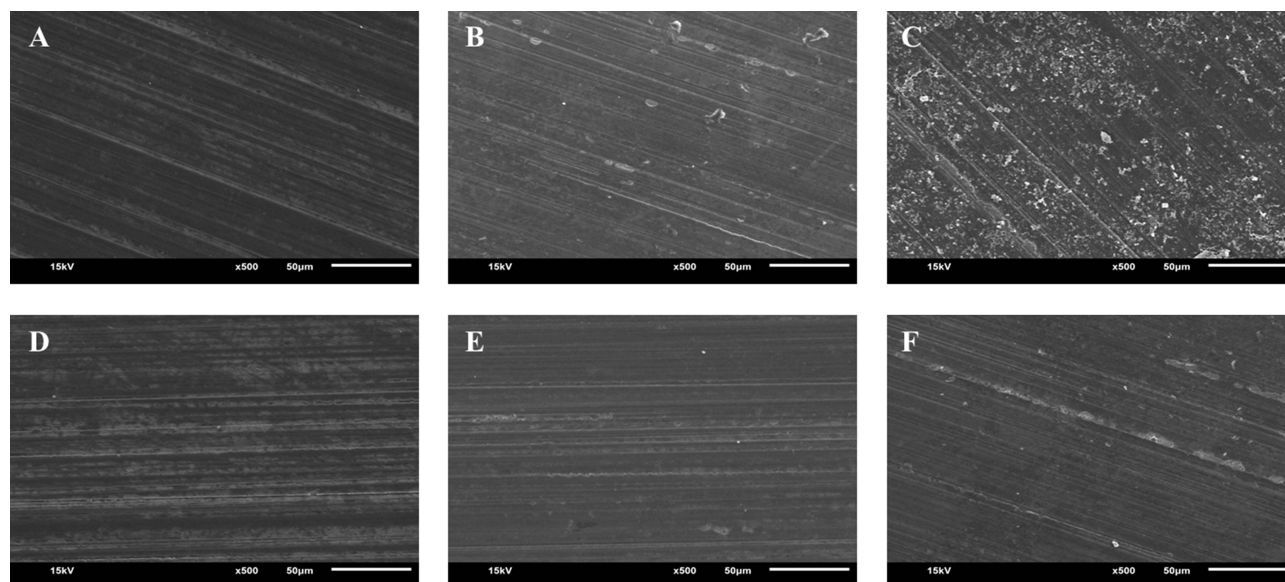


Figure 3 Post-bleaching SEM pictograms ($\times 500$) of the dentin fragment specimens: Figures (A–C) represent the surface morphology of T₁, T₂ & T₃ subgroups, respectively using HP-based bleaching. (D–F) represent the surface morphology of the T₁, T₂ & T₃ subgroups, respectively using HP-free bleaching.

Table 5 provides insights into the failure modes associated with the use of HP-based and HP-free bleaching products across different bleaching regimens. All the treatment regimens exhibited 100% adhesive failure at the dentin–composite interface except T₃ of the HP-based bleaching group where 90% of samples exhibited adhesive failure while 10% failures were cohesive within the dentin. The visual analysis can be observed in Figure 4A–F.

Discussion

This study investigated the influence of two different home-based bleaching systems on the surface and mechanical properties of dentin substrate. The results suggested insignificant variations in nano hardness while significant variations in surface roughness, elastic modulus, and SBS were observed. Therefore, the null hypothesis was accepted for nano hardness, while it was rejected for surface roughness, elastic modulus, and SBS.

The conventional cavity preparation method compromises the healthy tooth structure due to retention and resistance form.²⁰ However, in the era of minimally invasive dentistry, preservation of as much of the natural tooth structure as possible is principally aimed. This imperative underscores the necessity for bonded restorations to adhere well to the tooth surface to be successful clinically, and any interference with this adhesion might impact both its success and longevity.¹⁴ The study findings advocate that the active bleaching agent in HP-based gel caused roughening of the tooth surface and significantly affected the SBS.

Table 5 Failure Pattern Distribution in Dentin–Composite Interfaces Across Various Bleaching Regimens Using HP-Based and HP-Free Bleaching Gels

Bleaching Product	Treatment	Cohesive Failure within the Dentin (%)	Adhesive Failure at the Dentin-composite Interface (%)	Cohesive Failure within the Composite (%)
HP-based	T ₁	0	100	0
	T ₂	0	100	0
	T ₃	10	90	0
HP-free	T ₁	0	100	0
	T ₂	0	100	0
	T ₃	0	100	0

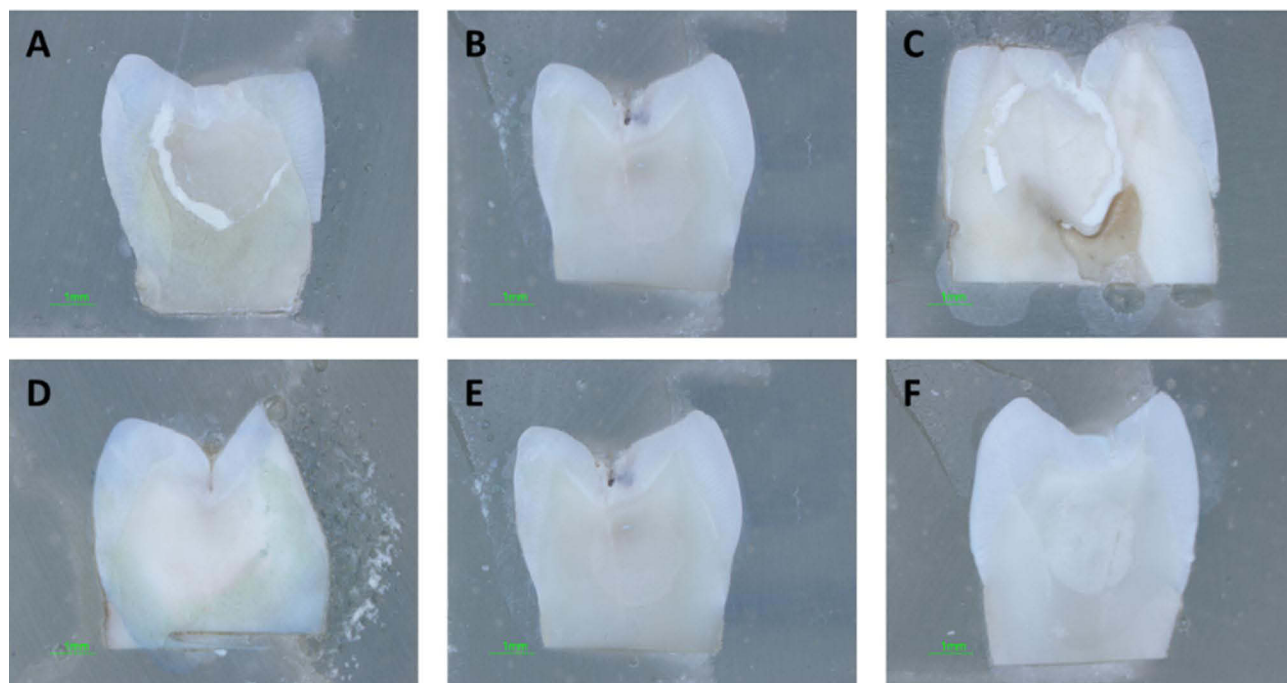


Figure 4 Stereomicroscopic images of debonding surfaces (scale bar showing 1 mm): From (A–C), adhesive failure at the dentin–composite interface except in image C where cohesive failure within the dentin can be seen using HP-based bleaching. While (D–F) shows adhesive failure at the dentin–composite interface using HP-free bleaching for T₁, T₂ and T₃, respectively.

A non-contact surface profilometer enables precise measurement of surface roughness and texture without altering the sample, ensuring accurate and non-destructive analysis.²¹ This technique was therefore employed in the current study. The T₁ and T₂ treatment conditions showed insignificant effects on the surface roughness parameter of the dentin fragments. While T₃ treatment condition showed a significant difference between the initial and final values using an HP-based bleaching gel. The reason could be the acidic nature of CP gels, which can demineralize the tooth structure.²² Prolonged exposure to the acidic gel may cause the erosion of enamel and dentin by removing mineral content, leading to the development of surface roughness.²³ The lack of a notable surface roughness effect at T₁ and T₂, coupled with a significant effect at the T₃ measurement interval, suggests that the duration of exposure could be a determining factor. In contrast, peroxide-free bleaching gel exhibited negligible differences in all treatment conditions, suggesting that the absence of peroxide from the formulation reduces the potential damage to the enamel or dentin.¹⁸ The current findings are in agreement with the previous study that advocated an adverse effect on the roughness of the enamel surface.²⁴ However, our study contradicts the findings of Ozdemir and Surmelioglu, who found no correlation between bleaching application on surface roughness.²⁵ The varied outcomes could be ascribed to diverse treatment regimens, uncontrolled variables, complex biological or chemical interactions and instrument calibration. Conversely, the inclusion of nano-hydroxyapatite and potassium citrate in the HP-free bleaching formulation may serve to sustain a nearly neutral pH during the bleaching application. This near-neutral pH may have a non-detrimental impact on the tooth surface.

In this study, nano hardness and elastic modulus were assessed to explore the effect of bleaching at nanoscale. In the T₁ regimen, a slight alteration in nano hardness between the initial and final values for both bleaching gels might imply that water immersion of dentin fragments does not induce any change in their nano hardness. However, over subsequent measurement intervals (T₂ and T₃), both products caused a decrease in nano hardness. Though the difference became less evident, HP-free is typically retained to have slightly greater nano hardness than HP-based bleaching. This suggests that the dentin's nano hardness declined due to the bleaching procedure, which is potentially linked to the chemical procedures used to whiten teeth that may damage the tooth structure.²⁶ While no significant variations were found between the initial and final readings when using any bleaching material at each measurement interval, a marginally

higher decrease in nano hardness with HP-based bleaching during the T_2 and T_3 measurement intervals may indicate the impact of the composition's pH and active ingredients. The dentin surface may be eroded by a lower pH.²⁷

A complicated interaction between the bleaching chemicals and the tooth structure could be another contributing factor. The active ingredients aim to break down and remove stains on the teeth, they can also potentially affect the mineral content of enamel and dentin,²⁸ leading to reduced hardness. The specific chemical reactions and interactions with the tooth's mineral content may differ between these two products. Despite earlier studies suggesting a decline in hardness following the bleaching process,^{4,24,25} it is crucial to recognize that direct comparisons with our study are not viable. Specifically, our study employed a nano hardness tester to gauge hardness at the nano level, whereas previous studies relied on microhardness testing.

It is well established that HP exhibits potent oxidizing properties.⁷ Additionally, its acidity is also a noteworthy characteristic.^{4,8} Both of these attributes are likely to play a role in influencing the observed outcomes, albeit to varying degrees. A prior study proposed that HP's robust oxidative influence on the organic matrix of intertubular dentin predominantly influences the alterations in intertubular dentin. As the collagen scaffold deteriorates, the demineralization of intertubular dentin is exacerbated by the acidic pH of HP, resulting in a reduction in hardness and elastic modulus.²⁹

In dental research, assessing the adhesive quality between materials and tooth structures, forecasting restoration durability, and contrasting various bonding agents depend on SBS. SBS is widely used method because of its relative simplicity.³⁰ The obtained results may suggest that the question of adhesive bonding with dentin either immediately after the bleaching or delayed is material-dependent. It was observed that SBS was significantly reduced from T_1 to T_2 . This may be because of the hydroxyapatite crystals that make up the mineral component of dentin that may change the structure of dentin. The formation of residual free radicals due to the breakdown of HP penetrates in dentin structure causing compositional changes.³¹ Previous studies have also reported a 20–60% reduction in bond strength between composite resin and enamel.^{31,32} Our study also observed a reduction of 39.59%. However, the results need to be cautiously interpreted as we used dentin substrate for bonding, while the previous studies used enamel as bonding substrate. Unlike the reduction of SBS at the T_2 stage using HP-based bleaching, a significantly higher SBS at the T_3 stage may suggest the demineralization process over time increased the surface roughness of the dentin, permitting resin tag formation in the etched dentin. Additionally, HP can break down and remove organic contents from within the dentin and create spaces for resin tags.¹⁰

No significant change in the SBS values at any measurement interval suggests that this new bleaching system with an active ingredient, ie, PAP, though it is an organic peracid containing a high potential of oxidation, however, due to the presence of hydroxyapatite nano powder and a citrate buffer in the formulation, the pH of the formulation remains neutral during application. Thus, preventing the dentin surface from erosion.¹⁷ The presence of nano-hydroxyapatite in the formulation helps in re-mineralization of tooth substrate besides potassium nitrate that aids in reducing the sensitivity, making the whitening process more comfortable for the user.¹⁸ It is also believed that the PAP+-based bleaching system works without the formation of free radicals.¹⁷ The bonding process between the resin composite and dentin substrate might not be affected due to the absence of free radicals.

The qualitative analysis of the dentin fragments at $\times 500$ indicated that the HP-based bleaching gel deteriorated the dentin surface over time. A marked difference in terms of surface texture and roughness at T_1 (Figure 3A), T_2 (Figure 3B) and T_3 (Figure 3C) measurement intervals suggest that the use of HP-based bleaching causes surface irregularities that help in enhancing resin composite to dentin bonding. The HP-free bleaching induces minimal to no surface alterations. Therefore, unaltered SBS values were observed at all measurement intervals using HP-free bleaching gel.

The consistent 100% adhesive failure at all measurement intervals at the dentin-composite interface suggests that the bond strength is not affected using HP-free bleaching (Figure 4D–F). In contrast, a 10% cohesive failure within dentin at the T_3 measurement interval for the HP-based bleaching suggests the occurrence of resin tag formation resulting from surface irregularities (Figure 4C). Hence, a strong SBS between a dentin-composite interface. The application of HP-containing bleaching gel on the dentin surface may increase SBS between resin composite and dentin due to surface irregularities induced by HP on dentin. However, over time, the dentin may undergo a remineralization process as dentin is a dynamic tissue capable of undergoing remineralization processes over time. As the dentin remineralizes, the surface

irregularities may be partially or filled in with mineral deposits, altering the topography of the dentin surface and leading to stress concentration at the resin composite and dentin interface, which could potentially contribute to bonding failure.

While the benefits of HP-free bleaching in terms of adhesion promotion and dentin damage reduction are highlighted in our findings, the product's efficacy in whitening teeth was not evaluated. However, a double blinded placebo-controlled short-term study advocates significant whitening effects immediately and 24 h after a single-use treatment.³³ Another study proved the efficacy of PAP-based bleaching equivalent to that of HP- or CP-based bleaching.³⁴

Like many laboratory investigations, this study too has its set of limitations. The investigation was limited to the immediate effects after the bleaching regimens. Long-term effects on the mechanical properties of dentin substrate and tooth-composite bonding strength were not addressed. Additionally, the potential impact on the pulp tissue was ignored. In the future, in vivo and clinical studies would be appropriate to observe the effects of bleaching gels in a more realistic oral environment, considering factors such as saliva, temperature variations, and dynamic tissue capability of dentin.

Conclusion

This laboratory assessment of the two different bleaching gels revealed that the HP-based bleaching gel demonstrated notable changes in surface roughness, elastic modulus, and SBS parameters over time, indicating its impact on the dentin substrate. In contrast, the HP-free bleaching showed insignificant differences within the group, suggesting a comparatively milder effect and safe for immediate bonding procedures. The demand for efficient teeth whitening ought to be balanced with the necessity to preserve tooth structure when selecting a bleaching material. Consequently, precaution is advised when performing immediate restorative procedures after HP-based bleaching application.

Funding

The authors extend their appreciation to the Deputyship for Research & Innovation, Ministry of Education in Saudi Arabia for funding this research (IFKSURC-1-0403).

Disclosure

All authors declare no conflicts of interest in this work.

References

1. Da Silva Machado J, Cândido MSM, Sundfeld RH, De Alexandre RS, Cardoso JD, Sundfeld M. The influence of time interval between bleaching and enamel bonding. *J Esthet Restor Dent.* 2007;19(2):111–118. doi:10.1111/j.1708-8240.2007.00077.x
2. Topcu FT, Erdemir U, Ozel E, Tiriyaki M, Oktay EA, Yildiz E. Influence of bleaching regimen and time elapsed on microtensile bond strength of resin composite to enamel. *Contemp Clin Dent.* 2017;8(3):451. doi:10.4103/ccd.ccd_234_17
3. Takamizawa T, Aoki R, Saegusa M, et al. Whitening efficacy and tooth sensitivity in a combined in-office and at-home whitening protocol: a randomized controlled clinical trial. *J Esthet Restor Dent.* 2023;35(6):821–833. doi:10.1111/jerd.13033
4. AlShehri A, AlRefeai MH, AlZamil F, AlOtaibi N, AlKinani Y. Effect of over-the-counter tooth-whitening products on enamel surface roughness and microhardness. *Appl Sci.* 2022;12(14):6930. doi:10.3390/app12146930
5. Martins KV, Zanatta RF, Palhari FTL, et al. Deleterious effects over enamel surface bleached with low and high concentration gels containing calcium—in vitro evaluation. *Int J Odontostomat.* 2022;16(1):120–124. doi:10.4067/S0718-381X2022000100120
6. Kwon SR, Wertz PW. Review of the mechanism of tooth whitening. *J Esthet Restor Dent.* 2015;27(5):240–257. doi:10.1111/jerd.12152
7. Seto TH, Grymak A, Mudliar V, Choi JJE. Effect of enamel bleaching on the bond strength of ceramic—a systematic review. *Oral.* 2022;2(2):182–197. doi:10.3390/oral2020018
8. Cheng Y-L, Musonda J, Cheng H, Attin T, Zheng M, Yu H. Effect of surface removal following bleaching on the bond strength of enamel. *BMC Oral Health.* 2019;19(1):50. doi:10.1186/s12903-019-0742-4
9. de Oliveira Duque C, Soares D, Briso A, et al. Influence of tooth pigmentation on H2O2 diffusion and its cytotoxicity after in-office tooth bleaching. *Oper Dent.* 2020;45(6):632–642. doi:10.2341/19-013-L
10. Sastrodihardjo S. *The Effect of Bleaching on the Morphology of Enamel.* Atlantis Press; 2018:152–154.
11. Cavalli V, Ries A, Giannini M, Ambrosano G. The effect of elapsed time following bleaching on enamel bond strength of resin composite. *Oper Dent.* 2001;26(6):597–602.
12. Oz FD, Kutuk ZB. Effect of various bleaching treatments on shear bond strength of different universal adhesives and application modes. *Restor Dent Endod.* 2018;43(2):e20. doi:10.5395/rde.2018.43.e20
13. Gurgan S, Alpaslan T, Kiremitci A, Cakir FY, Yazıcı E, Gorucu J. Effect of different adhesive systems and laser treatment on the shear bond strength of bleached enamel. *J Dent.* 2009;37(7):527–534. doi:10.1016/j.jdent.2009.03.012

14. Unlu N, Cobankara FK, Ozer F. Effect of elapsed time following bleaching on the shear bond strength of composite resin to enamel. *J Biomed Mater Appl Biomater*. 2008;84(2):363–368. doi:10.1002/jbm.b.30879
15. Elmourad AM, Alqahtani MQ. Effects of pre-and post-simulated home bleaching with 10% carbamide peroxide on the shear bond strengths of different adhesives to enamel. *Saudi J Dent Res*. 2014;5(2):81–92. doi:10.1016/j.sjdr.2014.01.002
16. Katib AH. *The Effect of 40% Sodium Ascorbate on Shear Bond Strength of Composite Resin Immediately After Bleaching with 40% Hydrogen Peroxide*. Nova Southeastern University; 2021.
17. Pascolutti M, de Oliveira D. A radical-free approach to teeth whitening. *Dent J*. 2021;9(12):148. doi:10.3390/dj9120148
18. Khan AA, Abdullah Alkhureif A, Bautista LS, Alsunbul H, Vellappally S. Peroxide-free bleaching gel: effect on the surface and mechanical properties of nano-and micro-hybrid restorative composite materials. *Appl Sci*. 2023;13(10):5935. doi:10.3390/app13105935
19. Khan AA, De Vera MAT, Mohamed BA, Javed R, Al-Kheraif AA. Enhancing the physical properties of acrylic resilient denture liner using graphene oxide nanosheets. *J Vinyl Addit Technol*. 2022;28(3):487–493. doi:10.1002/vnl.21895
20. Mount GJ, Hume WR, Ngo HC, Wolff MS. *Preservation and Restoration of Tooth Structure*. John Wiley & Sons; 2016.
21. Ružbarský J. The difficulty of measuring the roughness of glossy surfaces using the triangulation principle. *Appl Sci*. 2023;13(8):5155. doi:10.3390/app13085155
22. Benahmed AG, Gasmi A, Menzel A, et al. A review on natural teeth whitening. *J Oral Biosci*. 2022;64(1):49–58. doi:10.1016/j.job.2021.12.002
23. Ribeiro MES, Lopes RM, Aranha ACC, Medeiros IS, Lima RR, Loretto SC. Is prolonged bleaching more harmful to dental enamel than daily dietary and hygienic oral habits? *Braz Oral Res*. 2021;35:e113. doi:10.1590/1807-3107bor-2021.vol35.0113
24. Goyal K, Saha SG, Bhardwaj A, Saha MK, Bhapkar K, Paradkar S. A comparative evaluation of the effect of three different concentrations of in-office bleaching agents on microhardness and surface roughness of enamel—an in vitro study. *Dent Res J*. 2021;18(1):49. doi:10.4103/1735-3327.318944
25. Ozdemir ZM, Surmelioglu D. Effects of different bleaching application durations on enamel in terms of tooth color, microhardness, and surface roughness. *Color Res App*. 2022;47(1):204–212. doi:10.1002/COL.22710
26. Alqahtani MQ. Tooth-bleaching procedures and their controversial effects: a literature review. *Saudi Dent J*. 2014;26(2):33–46. doi:10.1016/j.sdentj.2014.02.002
27. Joiner A. Review of the effects of peroxide on enamel and dentine properties. *J Dent*. 2007;35(12):889–896. doi:10.1016/j.jdent.2007.09.008
28. Lertsukprasert N, Locharoenrat K. Efficiency of tooth bleaching agent on staining and discoloration characteristics of nicotine stained dental enamel model. *BMC Oral Health*. 2020;20(1):221. doi:10.1186/s12903-020-01207-2
29. Chng H, Ramli H, Yap A, Lim C. Effect of hydrogen peroxide on intertubular dentine. *J Dent*. 2005;33(5):363–369. doi:10.1016/j.jdent.2004.10.012
30. Placido E, Meira JB, Lima RG, Muench A, de Souza RM, Ballester RY. Shear versus micro-shear bond strength test: a finite element stress analysis. *Dent Mater*. 2007;23(9):1086–1092. doi:10.1016/j.dental.2006.10.002
31. Vidhya S, Srinivasulu S, Sujatha M, Mahalaxmi S. Effect of grape seed extract on the bond strength of bleached enamel. *Oper Dent*. 2011;36(4):433–438. doi:10.2341/10-228-L
32. Ergün Kunt G, Yılmaz N, Şen S, Dede D. Effect of antioxidant treatment on the shear bond strength of composite resin to bleached enamel. *Acta Odontol Scand*. 2011;69(5):287–291. doi:10.3109/00016357.2011.568958
33. Bizhang M, Domin J, Danesh G, Zimmer S. Effectiveness of a new non-hydrogen peroxide bleaching agent after single use—a double-blind placebo-controlled short-term study. *J Appl Oral Sci*. 2017;25(5):575–584. doi:10.1590/1678-7757-2016-0463
34. Qin J, Zeng L, Min W, Tan L, Lv R, Chen Y. A bio-safety tooth-whitening composite gels with novel phthalimide peroxy caproic acid. *Compos Commun*. 2019;13:107–111. doi:10.1016/j.coco.2019.04.002

International Journal of Nanomedicine

Dovepress

Publish your work in this journal

The International Journal of Nanomedicine is an international, peer-reviewed journal focusing on the application of nanotechnology in diagnostics, therapeutics, and drug delivery systems throughout the biomedical field. This journal is indexed on PubMed Central, MedLine, CAS, SciSearch®, Current Contents®/Clinical Medicine, Journal Citation Reports/Science Edition, EMBase, Scopus and the Elsevier Bibliographic databases. The manuscript management system is completely online and includes a very quick and fair peer-review system, which is all easy to use. Visit <http://www.dovepress.com/testimonials.php> to read real quotes from published authors.

Submit your manuscript here: <https://www.dovepress.com/international-journal-of-nanomedicine-journal>