

## **SHEAR BOND STRENGTH OF ORTHODONTIC BRACKETS BONDED TO BLEACHED ENAMEL USING A NOVEL PAP-BASED BLEACHING AGENT- AN IN VITRO STUDY**

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### **ABSTRACT**

The aim of this study was to evaluate the effect of a novel phthalimidoperoxycaproic acid (PAP) and carbamide peroxide-based bleaching gels on shear bond strength between enamel and resin composite used to bond orthodontic brackets.

**Materials and Methods:** Fifty four human premolars were selected and divided into three equal groups (N=18); Group I (control): unbleached teeth (C), Group II: received carbamide peroxide-based bleaching material (CP) and Group III: received PAP-based bleaching material (PAP). Each group was further subdivided into two equal subgroups according to time of bonding of the orthodontic brackets either immediately after bleaching procedure (IB) or 14 days after bleaching procedure (DB) (n=9/subgroup). According to their assigned subgroups, labial enamel surfaces of teeth were bleached, embedded in acrylic resin blocks and orthodontic brackets were bonded using Transbond XT light cure resin composite (3 M Unitek, Monrovia, CA, USA). Teeth were mounted in a universal testing machine and shear bond strength were measured in MPa. Data were statistically analyzed by two-way ANOVA and Tukey's post hoc significance difference test were used to analyze the data.

**Results:** Two-way ANOVA showed that the two variables in this study had a statistically significant effect on enamel/resin composite shear bond strength, also there was an interaction between the two tested variables.

**Conclusions:** PAP-based bleaching agents may present a safe alternative to free radical-based bleaching agents. Delayed bonding may play a role in improving bonding to bleached enamel.

**KEYWORDS:** Shear bond strength, PAP-bleaching agent, Orthodontic brackets.

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## INTRODUCTION

Vital teeth bleaching (also known as teeth whitening) has been a common requirement for the public over the past few years. Patients who use bleaching agents at home refer to them as “at-home teeth bleaching” which either contain hydrogen peroxide (HP) or carbamide peroxide as an active ingredients<sup>(1)</sup>. When in contact with water, carbamide peroxide converts to hydrogen peroxide, which makes up 35% of its weight. Both bleaching agents are currently utilized at home or in a dental office (on a dental chair), and their bleaching effects get better with increased hydrogen peroxide concentrations and application times, following their manufacturers’ regulations<sup>(2)</sup>.

Both hydrogen peroxide and carbamide peroxide have the potential to oxidize chromogens (organic pigments), transforming them into simple and/or distinct structures with differing optical characteristics, which is what causes their bleaching effects<sup>(3)</sup>. Due to the presence of an unpaired electron that might react and become stable with conjugated systems of unsaturated organic molecules, these free radicals are unstable. This causes the oxidation reaction that breaks down the chromogen into smaller molecules. As a result of this oxidation process, the final products are smaller, less able to absorb light, and have less vibrant colors. According to the pH and activation process, hydrogen peroxide produces a variety of radicals<sup>(4,5)</sup>.

Although both hydrogen peroxide and carbamide peroxide produced satisfactory bleaching outcomes, they both have a number of drawbacks that limit their applicability. Use of these materials over an extended period of time, and/or repetitively, may cause hypersensitivity, irritation of the oral mucosa, and chemical alterations to the enamel, including erosion and a decrease in surface micro-hardness<sup>(6,7)</sup>. Despite the fact that professional application, which includes soft tissue isolation and the use of gingival barriers, is helpful to reduce or prevent soft tissue

irritation, it cannot prevent negative effects on the structure of the enamel<sup>(8)</sup>.

In addition to the variables already discussed, a decrease in bond strengths between resin-based materials and enamel just after bleaching has been documented in the literature. This was explained by the fact that resin polymerization is prevented from occurring by oxygen ions produced from bleaching chemicals into tooth structure<sup>(9)</sup>.

The removal of superficial enamel prior to bonding, the pre-treatment of enamel with alcohol, the use of adhesives containing organic solvents, and the use of antioxidants are just a few of the many trials that have been suggested to address the lower bond strength after bleaching<sup>(10-12)</sup>. The most well-known tactic is to postpone the restorative operation from 24 hours to two weeks in order to restore the bond’s usual strength<sup>(13,14)</sup>.

The issues described before forced dentists to look for bleaching solutions other than hydrogen peroxide and carbamide peroxide. The use of organic peroxides as active bleaching agents, such as phthalimidoperoxycaproic acid (PAP), has recently attracted the attention of researchers. The most well-known industrial form of PAP is EURECOTM HC-L17TM (Solvay, Brussels, Belgium), which is a stable aqueous suspension of PAP crystals. PAP demonstrated a number of favorable safety traits, including being easily biodegradable, non-irritating, and non-toxic to humans and their skin<sup>(15)</sup>.

PAP’s efficacy as a teeth-whitening agent has recently been examined. A considerable whitening effect without any irritation to the oral mucosa or hypersensitivity was observed following a single treatment in a double-blind, placebo-controlled clinical trial<sup>(16)</sup>. Additionally, a lab study comparing a PAP-based gel to a traditional hydrogen peroxide-based gel was published in 2019. Both substances had a comparable bleaching impact on bovine teeth, however surface morphology and hardness studies revealed that the hydrogen peroxide-

based gel diminished the bleached teeth's surface microhardness while the PAP-based gel had no effect on the integrity of the enamel structure<sup>(17)</sup>.

Regarding adhesion to bleached enamel by PAP-based agent, literature lacked a formative research evaluating the effect of PAP on enamel bond strength. Based on this background, the present study was held to evaluate the effect of a novel phthalimidoperoxycaproic acid (PAP) and carbamide peroxide-based bleaching gels on shear bond strength between enamel and resin composite used to bond orthodontic brackets.

## MATERIALS AND METHODS

### Sample size calculation

This power analysis used shear bond strength as a primary outcome. The effect size  $f = (0.528114)$  was calculated based upon the results of Sedky and Abd Elhamid, 2021<sup>(18)</sup> using Alpha level of 5% and Beta level of 80%. The minimum estimated sample size was a total of 54 samples (9 sample per group). Sample size calculation was done using G\* Power version 3.1.9.2.

### Sample selection and preparation

In this in vitro study, 54 extracted, sound, human premolars were collected and stored in a 0.1% thymol solution. All of the teeth were observed to have intact buccal enamel; no pretreatment with any chemical agents; no cracks; no caries; and no restorations. Before any intervention, the enamel surfaces were polished with fluoride-free fine

pumice (Glove Club Ltd, Greenford, UK) and water coolant by using a slow-speed handpiece for 10 seconds<sup>(19)</sup>.

The samples were randomly divided into three equal groups (N=18), a control group and two experimental groups, according to the bleaching material applied as follow:

**Control group (C):** where teeth didn't receive any bleaching procedure

**Carbamide peroxide group (CP):** where teeth were bleached using carbamide peroxide-based at-home bleaching material (Opalescence, Ultradent, Utah, USA)

**Phthalimidoperoxycaproic group (PAP):** where teeth were bleached using PAP-based at-home bleaching material (hismile, HISMILE PTY LTD, Australia).

The bleaching materials used in the study, their composition and manufacturer are presented in **Table (1)**.

Each group was subdivided into two equal sub-groups (n=9/subgroup), according to time of bonding of the orthodontic brackets, as follow:

**Immediate Bonding Sub-group (IB):** where orthodontic brackets were bonded immediately after bleaching procedure.

**Delayed Bonding Sub-group (DB):** where orthodontic brackets were bonded 14 days after bleaching procedure.

TABLE (1) Bleaching materials, their composition and manufacturer

Material	Composition	Manufacturer
<b>Opalescence</b> (At-home bleaching material)	10% carbamide peroxide, Glycerin, water, xylitol, carbamide peroxide, carbomer, PEG-300, sodium hydroxide, potassium nitrate, EDTA, sodium fluoride.	Ultradent, Utah, USA
<b>Hismile</b> (At-home bleaching material)	4.1% phthalimidoperoxycaproic (PAP), potassium citrate and hydroxyapatite. (PAP+ formula).	HISMILE PTY LTD, Australia

### Teeth bleaching procedure

According to their manufacturers' instructions, bleaching materials were applied to the experimental groups as follow:

**CP group:** Bleaching material was applied directly into buccal surface of each tooth and left for eight hours. After eight hours, teeth were washed with water and surface-blotted lightly to remove excess moisture from the surface.

**PAP group:** The teeth were embedded in putty addition silicon (elite HD+, Zhermack dental, Italy) and arranged in a U-shaped arch to fit inside the ready-made plastic tray attached with the kit. The bleaching material was applied on buccal surface of each tooth and in the plastic tray. The loaded tray was applied on the mounted teeth then the LED device, attached with the kit, was switched on [Figure (1)]. After 10 minutes, LED device was switched off automatically indicating the end of the bleaching procedure. Teeth were washed with water and surface-blotted lightly to remove excess moisture from the surface.

For both groups, six applications were performed daily for six consecutive days (one application/day). Teeth were stored overnight in distilled water at room temperature between applications to minimize the effects of dehydration<sup>(20)</sup>. After finishing the six applications, teeth were removed from silicon mold.

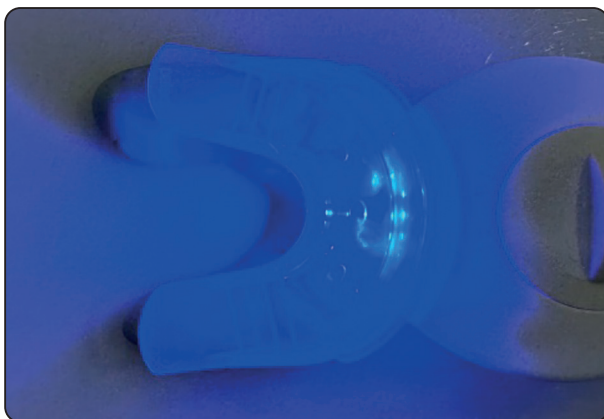


Fig. (1) Bleaching procedure for PAP group

### Teeth mounting in acrylic molds

Selected teeth were embedded in self-cured acrylic resin (Acrostone Dental & Medical Supplies, Cairo, Egypt) cylinders with their occlusal surfaces facing upwards, and parallel to the horizontal plane. The cemento-enamel junction (CEJ) was located slightly above the acrylic resin surface by 2 mm.

### Orthodontic bracket bonding

According to their subgroups, premolar metallic orthodontic brackets [0.022-inch (Roth), discovery smart, Dentaauram, Germany] were bonded to teeth either immediately after bleaching procedure or after 14 days of bleaching procedure, i.e. In (DB) sub-groups, teeth were stored in distilled water at room temperature till time of orthodontic brackets bonding.

Each tooth's enamel surface was polished for 10 seconds<sup>(21)</sup> with pumice and a rubber cup, followed by a water spray and air drying. The 37% orthophosphoric acid solution (Ormco Corporations, USA) was applied to the enamel surface for 30 seconds, followed by a thorough 15-second rinsing and another 15 seconds of air-drying. The enamel surface was coated with Transbond XT adhesive (3M Unitek, Monrovia, CA, USA) and cured by LED dental curing light (Bluephase N, Ivoclar Vivadent, Germany) for 20 seconds. Premolar brackets were attached to the treated enamel surfaces in accordance with the manufacturer's instructions using Transbond XT light cure composite from 3 M Unitek, Monrovia, California, USA. The buccal enamel surface was used to place and seat the brackets, and any extra resin composite was removed before curing using a dental probe<sup>(22)</sup>. Finally, the resin composite was then cured for 40 seconds.

### Shear bond strength (SBS) test

The lower fixed head of the universal testing device (Instron model 3345 England) was attached with the acrylic blocks that had the teeth embedded in them. The upper movable head of the testing apparatus was equipped with a uni-beveled chisel

with a 0.5 mm wide blade. Compression mode force was applied using the chisel blade at a crosshead speed of 1.0 mm/min up until the bracket was completely debonded [Figure (2)]. The shear bond strength in MPa was calculated using the attached machine software (BlueHill 3 Instron England) by dividing the force necessary for failure in (N) by the surface area of the orthodontic bracket ( $\text{mm}^2$ ).



Fig. (2) A tooth embedded in an acrylic block attached to universal testing machine for shear bond strength testing.

### Statistical analysis

Numerical data from the experiment was collected, tabulated and checked for normality using test of normality (Shapiro–Wilk test). The data was found to be normally distributed and a parametric test, two way ANOVA, was used to compare between

different groups, and post-hoc test was used to detect significance if present. The significance level was set a  $p \leq 0.05$ . IBM SPSS statistics for windows, was used for statistical analysis.

### RESULTS

Two way ANOVA was used to test the effect of the two tested variables in this study on shear bond strength [Table (2)]. The first variable which was the type of bleaching material applied (M), and it had three levels [either control (C), carbamide peroxide (CP), or phthalimidoperoxycaproic (PAP)]. While the second variable was the time of bonding of the orthodontic brackets (T), it had two levels either immediate bonding (ID) or 14 days after bleaching (DB). Statistical analysis was performed with IBM SPSS Statistics Version 20 for Windows.

Two way ANOVA showed that different type of bleaching materials had a statistically significant effect on the bond strength at an F value of 83.212 and a  $p$ -value = 0.0001, also statistical significant effect was noticed for the time of bonding of the orthodontic brackets on shear bond strength (F value = 70.555 and  $p$ -value = 0.0001). Results also showed that there was an interaction between the two tested variables at F value = 63.4053 and  $p$ -value = 0.0001.

Post hoc test revealed that the control groups whether IB or DB showed the highest mean shear bond strength with no statistically significant difference in-between. While the lowest statistically significant mean value was recorded for the CP/IB. The time of bonding of the orthodontic brackets significantly affect the CP group, such effect was not evident in case of C or PAP groups [Table (3) & Figure (3)].

TABLE (2) Two way ANOVA statistical evaluation for the effect of the two tested variables on shear bond strength.

Variable	df	Sum of Square	Mean Square	F Statistic	P-value
T	1	130.27	130.27	70.555	0.00001
M	2	307.28	153.64	83.212	0.00001
T*M	2	234.14	117.07	63.4053	0.00001



TABLE (3) Means, standard deviation (MPa) and significance of shear bond strength for all tested groups.

	C			CP			PAP		
	Mean	SD	sig	Mean	SD	sig	Mean	SD	sig
IB	12.377	2.28723	a	1.63055	0.58229	d	8.35197	0.64529	c
DB	11.38443	0.96725	ab	10.44898	0.50191	b	9.84535	1.97581	bc

*Different letters show statistical significance  $p \leq 0.05$*

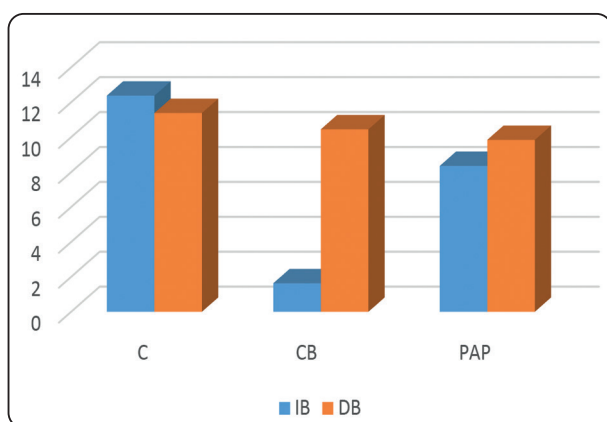


Fig. (3) A bar chart showing the effect of the two variables on shear bond strength for all tested groups.

## DISCUSSION

There have been numerous teeth-whitening products, both for professional and non-professional usage, in the last ten years, many of which are carbamide peroxide or hydrogen peroxide-based. An orthodontic or restorative appointment may be scheduled for the patient soon after bleaching<sup>(23)</sup>. Unfortunately, bleaching agents that contain hydrogen peroxide or carbamide peroxide lead to the production of a destabilized adhesive interface between resin and tooth tissues, which has a negative impact on the bond strength of the resin to tooth tissues<sup>(24)</sup>. On teeth that had recently undergone restoration following bleaching, scanning electron microscopy studies revealed reduced resin tag production and an erratic hybrid layer structure<sup>(25,26)</sup>.

Due to its clinical relevance, this early decline in enamel bond strength following bleaching is seen as a serious issue that needs to be handled and fixed. Although numerous researchers have looked into a variety of strategies to strengthen the link between adhesive restorations and resin-bonded brackets and bleached enamel, no clear-cut and widely accepted technique has yet to be introduced<sup>(14)</sup>.

One of these methods was the addition of a radical-free bleaching substance, such as phthalimidoperoxycaproic (PAP), which performed better when compared to a 6% hydrogen peroxide gel in a previous laboratory study of its efficiency on polyphenol stains<sup>(15)</sup>. Furthermore, according to Qina et al., PAP gels exhibit a same whitening effect to HP gels<sup>(17)</sup>. Our study's objective was to assess the impact of new carbamide peroxide- and phthalimidoperoxycaproic acid-based bleaching gels (Opalescence, Ultradent, Utah, USA) on the shear bond strength between enamel and resin composite used to bond orthodontic brackets.

Shear bond strength results after instant bonding (IB) of orthodontic brackets to bleached enamel labial surfaces of teeth in this study revealed that PAP group had a statistically significant higher mean value than CP group. This may be attributed to PAP which can oxidize and decolorize chromogens by epoxidizing molecules with conjugated double bonds [Figure (4)]. Free radicals are thought to be the primary cause of tooth sensitivity, gingival irritability, and a decrease in the bond strength of the enamel during teeth whitening using hydrogen

peroxide and carbamide peroxide<sup>(27)</sup>. This technique does not result in the creation of free radicals. Moreover, in order to reduce enamel surface loss through dental erosion and a decrease in surface microhardness, hydroxyapatite and a citrate were added to the hismile bleaching substance used in this study (PAP+ formula). Previous research demonstrated that bleaching gels lacking in bio-available calcium caused enamel erosion and mineral loss to be more worse<sup>(28,29)</sup>. The degradation of the inorganic and organic components of the tooth surface, mostly brought on by the actions of free radicals, has also been linked to changes in microhardness, according to a number of in vitro studies<sup>(30-32)</sup>.

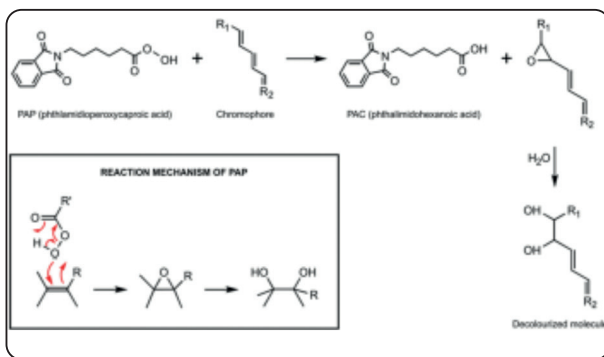


Fig. (4) The way that PAP affects chromogens <sup>(15)</sup>

In all tested (IB) subgroups, orthodontic brackets were bonded to the teeth with Transbond XT adhesive (3 M Unitek, Monrovia, CA, USA) which showed the highest mean shear bond strength in previous studies<sup>(33,34)</sup>. However, in CP (IB) subgroup, the mean shear bond strength was 1.63055 MPa while the mean shear bond strength of PAP (IB) subgroup was 8.35197 MPa. It should be noted that Reynolds investigation recommended that shear bond strength ranged from 5.9 to 7.8 MPa are acceptable and sufficient to make orthodontic brackets tolerate intraoral forces during the orthodontic treatment period<sup>(35)</sup>.

On the other side, shear bond strength results after delayed bonding (DB) of orthodontic brackets to bleached labial enamel surface of teeth showed

no statistical significant difference between groups. However, time of bonding of the orthodontic brackets had a statistically significant effect on CP group while it showed non-significant effect on the other two groups. In CP group, DB subgroup showed a statistically significant higher mean value than IB subgroup. Remaining oxygen from the bleaching chemical, which prevents resin polymerization, may be the cause of the drop in shear bond strength in the IB subgroup. The effect of residual oxygen appears to be eliminated by the 14-day delay in bonding. When bleached teeth were immediately bonded, Spyrides et al. found that bond strengths were reduced; however, bond strengths significantly increased when bonding was postponed for a week<sup>(36)</sup>. To avoid the negative effects of the bleaching agent's residual oxygen, which inhibited resin polymerization and hampered proper resin infiltration, other researchers advised delaying bonding for two to four weeks after bleaching<sup>(14,37)</sup>.

According to Gungor et al., bleaching done at home had a larger negative impact on bond strength. They explained that the extended application times involved with the at-home bleaching procedure led to changes in the enamel's surface structure and that the calcium loss may also become more pronounced with time<sup>(38)</sup>. According to another study, the restored normal bond strength values in at-home bleaching groups were likely due to the decreased peroxide contents in these bleaching products. The teeth were also kept in distilled water after each daily whitening. This might have removed the leftover peroxide that the enamel had absorbed<sup>(20)</sup>.

## CONCLUSIONS

According to the conditions of our study, the following could be concluded:

1. PAP-based bleaching agents may present a safe alternative to free radical-based bleaching agents.
2. Delayed bonding may play a role in improving bonding to bleached enamel.

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