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Effect of Delayed Bonding and Antioxidizing Agent on Shear Bond Strength of Resin Composite to Enamel and Dentin after Tooth Bleaching

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Abstract. The present study aimed to investigate the effect of delayed bonding and antioxidizing agent on shear bond strength of bleached enamel and dentin. Forty extracted premolars were mounted in clear acrylic, ground to expose flat enamel and dentin surfaces, and bleached using 35% carbamide peroxide. Specimens were divided into two groups of 20, each was subdivided into two equal subgroups n = 10. In first and second subgroups, composite resin was applied to both enamel and dentin substrates immediately (T0) and after 7 days (T1) from bleaching respectively. In the third and forth subgroups, bleached substrates were pretreated with 10% sodium ascorbate before T0 and T1 composite application respectively. Shear bond strength was tested and data were statistically analyzed using ANOVA and "student's" *t*-test at $p \leq 0.05$. A significant increase in the shear bond strength was detected after sodium ascorbate treatment both in enamel and dentin substrates (from 8.57 ± 0.84 to 12 ± 1.97 MPa and from 11.51 ± 1.32 to 15.57 ± 1.55 MPa, respectively) with immediate application of composite resin ($p \leq 0.05$). Statistical significance ($p \leq 0.05$) was also found after delayed application of composite with and without sodium ascorbate in both enamel and dentin. It can be concluded that, Sodium ascorbate treatment for bleached enamel and dentin improved the bond strength, especially after delayed composite application.

Keywords: Antioxidant, Shear bond strength, Carbamide peroxide, Sodium ascorbate; In-office bleaching.

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Introduction

Most bleaching techniques use some form or derivative of hydrogen peroxide in different concentrations and application techniques. The mechanism action of bleaching with hydrogen peroxide is considered to be oxidation of organic pigments, although, the chemistry is not well understood[1].

With the overwhelming increase in esthetic demanding patients, the dentist should be aware of the limitations and shortcomings of the bleaching procedure; including its effect on structure and chemical composition of enamel and dentin, on restorative materials, as well as on bonding interface. With all the bleaching techniques, there is a transitory decrease in the potential bond strength of composite when it is applied to the bleached enamel and dentin. This reduction in bond strength might result from residual oxygen or peroxide residue in the tooth that inhibits the set of the bonding resin, precluding adequate resin tag formation in the etched enamel[1-4].

It has been reported that, the reduction in bond strength as a consequence of bleaching can be inverted by time, ranging from 24 hr[5], one week with water storage[6,7] or without water storage[8] to three wk[3].

On the other hand, Teixeira *et al.*[9], evaluated the effect of non-vital tooth bleaching on shear bond strength (SBS) of the composite resin/bovine enamel interface at different periods of time after bleaching. They found that, at 7, 14, and 21 days, no significant differences were observed from that found at 0 (base line time). Again, Basting *et al.*[10] found that 10% carbamide peroxide agents or placebo agent caused no differences in SBS of dentin after 15 days of storage in artificial saliva.

The use of antioxidants (reducing agents) is another method to preclude any oxygen or peroxide residue and allows a preferable immediate composite restoration. Sodium ascorbate (10%) was found to reverse the reduced bond strength to both dentin[11-13] and enamel[14].

Kum *et al.*[15] found that pretreatment of bleached tooth surface with catalase and ethanol reduce agents prior to bonding which significantly improves the composite-enamel shear bond strength. Recently, Bulut *et al.*[16] reported that, treating the bleached enamel surface with 10% sodium ascorbate or waiting for one week, enhances the reduced bond strength.

Based on this study, it was found the need to investigate the effect of delayed bonding and antioxidizing agent treatment on the shear bond strength of enamel and dentin tooth substrates. The null hypothesis was determined; time factor or sodium ascorbate antioxidizing agent will enhance the compromised tooth's shear bond strength after bleaching.

Materials and Methods

Materials

Forty human premolar teeth were used for the present study.

The category, detailed composition and manufacturer of the adhesive system, composite resin, carbamide peroxide and sodium ascorbate materials used in the study are presented in Table 1.

Table 1. Category, composition and manufacturer of the materials utilized in the study.

Material	Category	Composition	Manufacturer
Opalescence Quick	In-office bleaching system	35% carbamide peroxide, carbopol, glycerin and flavoring. - PH ~ 6.0	Ultradent Products Inc., South Jordan, UT USA
Sodium ascorbate 10%	Reducing agent	Prepared by dissolving 10 gm of sodium ascorbate powder ($C_6H_7NaO_6$) in 100 ml distilled water	Oxford Laboratory, Mumbai, India
Te-Econom Adhesive System	Total etch / two step bonding system	Total Etch: phosphoric acid (37 wt% in water), silicon dioxide and pigments. Adhesive: hydroxyethyl methacrylate (HEMA), di- and mono- methacrylates, inorganic fillers, initiators and stabilizers in an alcohol solution.	Ivoclar Vivadent AG, Schaan, Liechtenstein
Te-Econom Resin Composite	Light-curing microhybrid resin-based dental restorative material	The inorganic filler is silanated barium glass/silica. Its total content is 80 wt% (62 vol.%) with mean particle size of 0.04 - 7.0 µm. The resin matrix comprises Bisphenol A-Glycidyl Methacrylate (BIS-GMA), urethane dimethacrylate and triethylene glycol dimethacrylate (18.8 wt%). Additional contents: small amounts of catalysts, stabilizers and pigments (0.2 wt%).	Ivoclar Vivadent AG, Schaan, Liechtenstein

Teeth Selection

Forty caries-free premolar teeth which were extracted for orthodontic reason from an age group of 18-25 years were selected and used in this study. Selected teeth were cleaned, scaled, and checked under a magnifying length to be free from cracks.

Preparation of Teeth Specimens

Premolar teeth were mounted in clear acrylic resin blocks so that their buccal surfaces were facing upward (Fig. 1). Mounting was done using specially designed and constructed cubical shaped copper split mould to allow for a standardized tooth position and angle of mounting. The mould was utilized for embedment of teeth in acrylic resin blocks and for application of resin composite to the tooth substrate. Figure 2 shows the top view of the copper mould. Its external dimensions were 4.8×3.4×2 cm in length, width and height, respectively. The mould has a cubical cavity of 3×2×1.8 cm length, width and height, respectively. This was used to accommodate and embed the tooth in the acrylic block. The mould can be split into two halves along a horizontal split (Fig. 1a) and tightened by screws at the side of the mould (Fig. 1b). To standardize embedding of teeth in the acrylic block, the mould is marked vertically with a central groove (Fig. 1c) and horizontally with another groove (Fig. 1d) which divides the length of the internal window into an upper one third (1 cm) and lower two thirds (2 cm).

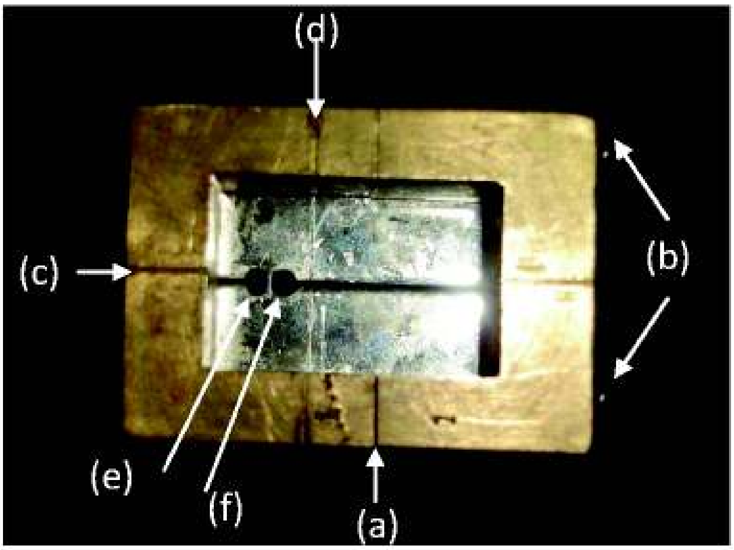

Fig. 1. Top view of the split copper mould. (a) horizontal split, (b) base screws, (c) central groove, (d) horizontal groove, (e) and (f) holes for application of resin composite to enamel and dentin, respectively.

Fig. 2. Acrylic resin block showing ground enamel and dentin substrates.

The depth of mounting was also standardized by turning the mould face down, flushing it to a glass slab. The moulds were unscrewed after polymerization; the acrylic blocks with the mounted teeth specimens were removed. Grinding of the teeth specimen were then made using a grinding machine under copious water cooling to expose a flat, smooth enamel and dentin substrates in the same tooth specimen. Specimens were washed under running water and placed in distilled water at 37^{o}C, ready to receive different treatments.

Grouping of Teeth Specimens

Teeth specimens were randomly divided into two equal groups. Each group was further subdivided into two subgroups of 10 specimens, each (n = 10) for both enamel and dentin substrates. In the first and second subgroups, composite resin was applied to both enamel and dentin substrates immediately (T0) and after 7 days (T1) from bleaching respectively. In the third and forth subgroups, bleached enamel and dentin substrates were pretreated with 10% sodium ascorbate before the bonding and composite resin application (T_0S and T_1S), respectively.

Bleaching Procedure

The carbamide peroxide bleaching system was manipulated according to the manufacturer's directions. The bleaching gel was spread over the substrate tooth tissue, left for 30 min, then rinsed for 30 seconds with copious amount of water.

Delayed Bonding

Specimens assigned for delayed bonding were stored in distilled water for 7 days at 37^{o}C.

Reducing Agent Application

Freshly prepared 10% sodium ascorbate was dripped on the substrate tissue of specimens assigned for the application of reducing agent (S_1) and was agitated with a micro-brush (Ivoclar Vivadent AG, Schaan, Liechtenstein). After 10 min, the specimens were rinsed with copious amount of water for 30 seconds and were dried gently with oil-free compressed air.

Composite Application

Bonding procedures

Bonding procedures were performed according to manufacturer's instructions. A piece of single-sided adhesive tape with a punctured hole of 2.0 mm diameter was placed on each specimen surface to standardize the area of bonding. The area specified for adhesion either for enamel or for dentin was then marked using a fine marker. The adhesive tape was then placed on treated specimen such that the 2.0 mm hole would be limited to the substrate investigated; either enamel or dentin.

Etching

The phosphoric acid etching gel (Total etch, Ivoclar Vivadent AG, Schaan, Liechtenstein) was applied onto the substrate surface; following the manufacturer's directions. Enamel specimens were etched for 25 seconds and dentin specimens for 15 seconds. The acid was then rinsed with water for 15 seconds and dried gently with oil-free compressed air for 10 seconds, avoiding over drying of dentin.

Application of dentin adhesive

A generous amount of Te-Econom adhesive was applied on the substrate tissue using a fully saturated applicator tip (Ivoclar Vivadent AG, Schaan, Liechtenstein). The remaining excess solvent was evaporated with a slow stream of air for 10 seconds. The adhesive was then subjected to light-curing for 20 seconds using XL 3000 light curing unit (3M US: ESPE Dental Division, St. Paul, MN USA) afterwhich, the adhesive tape was removed from the substrate surface.

Application of composite resin

The specimens were re-placed in the copper mould with the tooth facing the bottom of the mould. The resin composite material Te-Econom (Ivoclar Vivadent AG, Schaan, Liechtenstein) was applied in one increment using a Teflon-tipped condenser (Dentsply Ash Instruments/Dentsply Ltd, UK). A matrix strip was applied to the surface to ensure removal of any excess of resin.

The composite was light cured for 40 seconds with the light curing tip in contact with the base of the copper mould. The light intensity was checked to be in the 420-500 nanometer range, using a built-in light meter. All specimens were then stored for 24 hours in distilled water at 37˚C before testing.

Shear Bond Strength Testing

Shear bond strength testing was done using Lloyd universal testing machine (Model LRX-plus; Lloyd-Instruments Ltd., Fareham, UK). Specimens in the acrylic blocks were fixed to the lower jag of the machine (Fig. 3) and loaded to failure at a crosshead speed of 0.5 mm/minute.

Fig. 3. The Lloyd Universal Testing Machine and the mounted specimen during shear bond strength testing.

The force required to break each specimen was recorded in Newtons and the obtained values were then converted into MegaPascal (MPA) units, according to the following equation:

$$\textit{Shear bond strength (SBS)} = F/A\ (N/mm^2)\ \text{or}\ (MPA)$$ [17]

Where F is the Breaking load in Newton and A is the Area of bonding interface = πr^2 (r = radius in mm).

Statistical Analysis

The results were recorded, tabulated, and statistically analyzed using one-way ANOVA test and "student's" *t* test. The level of significance was established at $p \leq 0.05$ for all statistical tests.

Results

The mean shear bond strength values and standard deviations in MPA are shown in Tables 2 and 3. Generally, sodium ascorbate antioxidant application to the bleached enamel and dentin substrates increased the shear bond strength of the adhesive system (Te-Econom). One-way ANOVA (Table 2) revealed that, the mean values for the increased SBS was statistically significant (p = 0.0001). However, the difference in shear bond strength between enamel and dentin substrates was not statistically significant (p = 0.7554). The delayed application of sodium ascorbate raised the shear SBS for enamel substrate from 12 ± 1.97 to 16.79 ± 1.59 (Fig. 4). This increase, however, was found statistically insignificant (p = 0.1603) for both enamel and dentin substrates collectively (Table 2).

Table 2. Mean shear bond strength values (MPA) of Te-Econom to enamel and dentin and for the various variables of the study using one-way ANOVA.

Factor	Variable	Mean	± SD	± SE	F-value	P-value	Significance
Substrate	Enamel	11.83	3.05	0.47	0.10	0.7554	ns
	Dentin	11.70	3.37	0.53			
Antioxidant	None	9.91	2.20	0.33	52.46	0.0001	*
	Ascorbate	13.75	2.90	0.46			
Time	Immediate	12.12	3.26	0.50	2.01	0.1603	ns
	Delayed	11.39	3.11	0.49			

One-way ANOVA

F-value	26.13	
p-Value	≤ 0.05	****

SD = standard deviation *SE = standard error*
**= Significant at p ≤ 0.05* *ns = not significant*

Table 3. The shear bond strength of resin composite (MPA) to enamel and dentin substrates with and without sodium ascorbate at the tested time intervals "student's" *t*-test.

Substrate	Time Lapse (T)	No Ascorbate (S_0)		Ascorbate (S)		"Student's" t-value	p Value
		Mean	± SD	Mean	± SD		
Enamel (E)	Immediate (T_0)	8.57	0.84	12	1.97	3.62	0.0151*
	Delayed (T_1)	13.76	1.47	16.79	1.59	3.29	0.0094*
Dentin (D)	Immediate (T_0)	11.51	1.32	15.57	1.55	4.70	0.0011*
	Delayed (T_1)	9.64	1.32	12.38	1.96	2.59	0.0319*

**= Significant at $p \leq 0.05$*

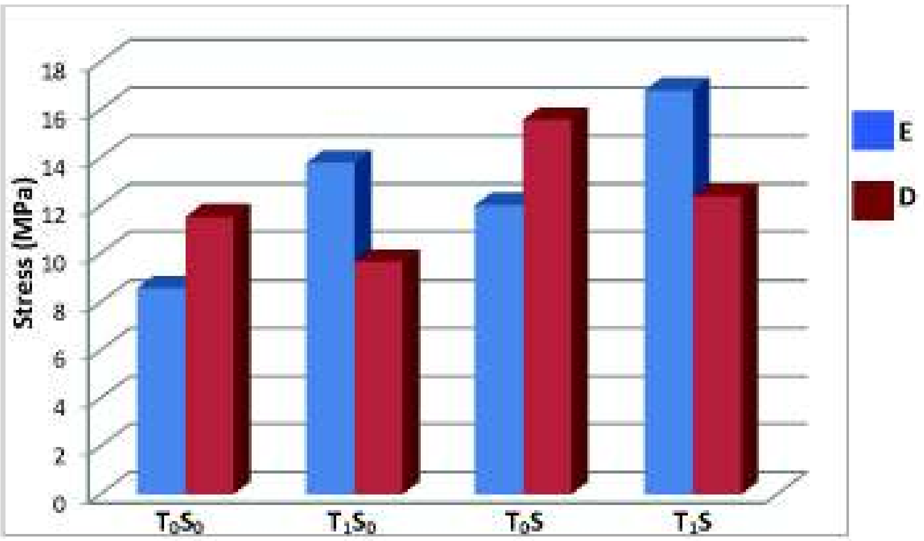

Fig. 4. Comparison of Shear bond strength of resin composite in (MPA) of enamel (E) and dentin (D) substrates with and without sodium ascorbate at the tested time intervals.

"Student's" *t* test demonstrated that there was a statistically significant difference in the mean shear bond strength between T_0S_0 and T_0S (P = 0.0151 and p = 0.0011) for both enamel and dentin substrates, respectively (Table 3 and Fig. 4). Again, a statistically significant difference was found in the mean shear bond strength between T_1S_0 and

T_1S (p = 0.0094 and p = 0.0319) for enamel and dentin substrates, respectively (Table 3 and Fig. 4).

Discussion

In the present *in vitro* study, freshly extracted sound premolar teeth from the same young age group were used. Teeth were assigned for extraction as a part of orthodontic treatment plan. The specially designed and constructed split mould was allowed for a standardized tooth position and angle of mounting of teeth specimens. It was utilized for embedment of teeth in standardized acrylic resin blocks. The bottom of the copper mould had two holes of 2 mm diameter and 2 mm high each for a standardized application of Te-Econom resin composite to enamel and dentin substrates, respectively.

Enamel and dentin substrates were subjected to a simulated in-office bleaching using 35% carbamide peroxide which was applied for a period of 30 min.

The time of application of Sodium ascorbate was selected to be 10 min. Lai *et al.*[14] immersed the bleached specimens in 10% sodium ascorbate solution for 3 h. While in most of the other studies, the duration of anti-oxidant treatment was 10 min[12,16,18,19]. This application time for sodium ascorbate was used in the current study as it was reasonable to the clinical situations.

The other half of the specimens bonding was applied either immediately after bleaching or delayed after seven days. Submersion of bleached tooth specimens in distilled water in a trial to restore the compromised SBS was repeatedly recommended[8,3,20-23].

The use of alcohol or acetone-based adhesive systems has been proposed to result in less compromised composite bond strength, when restorative procedures are to be completed immediately after bleaching [14,24-26]. In the present study, an alcohol based adhesive (Te-Econom) was utilized to bond resin composite to the bleached tooth substrate. Spyrides *et al.*[8] demonstrated that adhesives containing ethanol and water, reversed the decreased bond strength in 10% CP treated dentin. Ethanol is known to decrease the surface water and increase the enamel bond strength. Sung *et al.*[28] used an alcohol-based bonding agents, that showed no significant difference between bleached and unbleached enamel bond strength, and thus it was possible to apply composite restoration immediately after enamel bleaching. This water-chasing behavior of high pressure solvents (acetone and alcohol) is accentuated in dentin, owing to its intrinsic wetness.

The results of the present study revealed that sodium ascorbate pretreatment increased the shear bond strength values recorded for enamel and dentin substrates compared to the untreated ones. The difference was statistically significant. This marked increase was found both in immediately treated samples as well as those treated after a time delay of seven days. These results concur with[11,13,14,25,27,28] who have shown that reduced bond strength of composite resin to bleached enamel and dentin was effectively upturned by anti-oxidant treatment (sodium ascorbate).

Lai *et al.*[11] suggested that sodium ascorbate allows free-radical polymerization of the adhesive resin to proceed without premature termination by restoring the altered redox potential of the oxidized bonding substrate, and hence, reversed the compromised bonding. It was also reported, that this anti-oxidant is capable of quenching reactive free radicals that can help neutralize and reverse the oxidizing effect of hydrogen peroxide in the biological system[14]. A contradicted result, however was found by Uysal *et al.*[29]. They found no significant difference in shear bond strength between teeth specimens which were bleached, treated with antioxidant agent, then bonded and the control unbleached group.

The increase in the shear bond strength was found to be highly statistically significant in enamel -in the delayed application- and in dentin -in the immediate application ($p \leq 0.01$). Kaya *et al.*[12] reported that under scanning electron microscopy, interfaces between resin and bleached enamel were substantially different from those formed between resin and unbleached enamel. A granular and porous aspect with a bubbled appearance was observed, which was suggested to be due to gaseous bubbling, which could be the result of oxidizing reactions due to the entrapment of peroxide in the subsurface layer of the enamel. Hydrogen peroxide in various formulations was reported to cause loss of calcium, decrease in microhardness and alterations in the organic substance resulting in a decrease in enamel bond strength[30,31].

In the present study, higher shear bond strength was recorded in enamel after delayed application of the bonding agent with or without sodium ascorbate treatment. This might be explained on the basis that water immersion can effectively remove most of the hydrogen peroxide from the treated enamel. Titley *et al.*[18] reported that one week storage in distilled water seems to be appropriate for reversal of the reduced enamel bond strength. However, for dentin, this storage protocol might not be appropriate. Again, this could be a result of the interaction of the storage medium with dentin, as suggested in a study by Spyrides *et al.*[8] who found a decrease in bond strength values after one week delayed bonding even to non-bleached dentin. He also reported that, when the bleached dentin is exposed to artificial saliva for one week, the reduced bond increases for the in-office (35% carbamide peroxide) regimens.

When bonding agent was applied immediately with or without sodium ascorbate treatment; shear bond strength was recorded to be lower in enamel than in dentin. The reason for the relatively lower effect of bleaching on the bonding strength of the immediately bonded dentin might be that the time of application of the 35% carbamide peroxide (30 min) is not enough to cause accumulation of oxygen into dentin. Same explanation was reported also by Cavalli *et al.*[3]. The shown differences between enamel and dentin substrate in their reaction to the test variables are logic because of the difference in physical and chemical characteristics of these two dissimilar dental tissues.

Conclusion

Within the limitations of this study, the null hypothesis determined; time factor or sodium ascorbate antioxidizing agent will reverse the tooth's shear bond strength after bleaching was accepted. Accordingly, it can be concluded that:

1) Delayed bonding is a viable treatment modality after an in-office bleaching of enamel.
2) The use of 10% sodium ascorbate antioxidant enhances the compromised bond strength of bleached enamel and dentin, especially when immediate restoration is planned.
3) Further research with different age groups might be needed to accurately investigate the effect of shear bond strength of resin composite to enamel and dentin after tooth bleaching.

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تأثير تأخير الربط والمادة المضادة للأكسدة على قوة الربط القصي للراتنج المركب إلى المينا والعاج بعد تبييض السن

عبدالغني إبراهيم ميرة

قسم العلاج التحفظي، كلية طب الأسنان

جامعة الملك عبد العزيز

جدة – المملكة العربية السعودية

المستخلص. كان الغرض من البحث هو اختبار تأثير عامل الوقت أو استعمال أسكوربات الصوديوم المضادة للأكسدة على قوة الربط القصى للأسنان بعد التبييض، واستخدم في هذا البحث ٤٠ ضاحك غرست في الأكريليك الشفاف وتم بردها لإظهار سطح أملس من المينا والعاج ثم تبييضها بكارباميد بيروكسيد ٣٥٪. قسمت العينات إلى مجموعتين متساويتين من ٢٠ عينة وكل مجموعة إلى مجموعتين فرعيتين ن=١٠. في المجموعات الفرعية الأولى والثانية تم تطبيق الراتنج على ركائز المينا والعاج مباشرة بعد التبييض، و٧ أيام بعد التبييض على التوالى. فى المجموعات الثالثة والرابعة تم علاج الركائز المبيضة ب ١٠٪ أسكوربات الصوديوم قبل تطبيق الراتنج (مباشرة أو بعد ٧ أيام من التبييض على التوالى). تم اختبار قوة الربط القصي وحللت النتائج إحصائيا. تم الحصول على القيم العليا ذات دلالة إحصائية لقوى القص الربطى بعد علاج الركائز المبيضة ب١٠٪ أسكوربات الصوديوم لكل من ركائز المينا والعاج (من ٨,٥٧ ±٠,٨٤ إلى ١,٩٧ ± ١٢ ومن ١١,٥١±١,٣٢ إلى ١٥,٥٧ ±١,٥٥على التوالى) بعد تطبيق الراتنج مباشرة بعد

التبييض. أيضا تم الحصول على القيم العليا ذات دلالة إحصائية لقوى القص الربطى بعد التطبيق المتأخر للراتنج (٧ أيام) بعد التبييض في الركائز المستخدمة. إن علاج الركائزبأسكوربات الصوديوم بعد التبييض يعزز قوة الربط خاصة بعد التطبيق المتأخر للراتنج.