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Can Sodium Ascorbate Increase the In Vitro Bond Strength of the Interface between a Composite and Bleached Enamel?

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Abstract: Recently, the use of antioxidants before the bonding of bleached enamel was considered effective for reversing the reduction in the bond strength. This article aimed to assess the influence of different sodium ascorbate (SA) presentations (liquid, gel, and semi-gel) on the composite resin–enamel bond strength after a bleaching protocol. Sound human anterior teeth were collected, cleaned, prepared for a bond strength test, and randomly allocated into groups according to the bonding procedure. Group 1 acted as a control, without bleaching treatment, and without applying an antioxidant agent. In groups 2–6, specimens were bleached using 10% carbamide peroxide. In groups 3, 4 and 5, 10% wt% SA was applied for 10 min as an antioxidant in the form of a liquid, gel, and semi-gel, respectively. In group 6, samples were bleached and immersed in fresh human saliva for 14 days. After the bleaching process, the materials were restored by means of an adhesive system and a resin composite material. The analysis revealed that the differences between the shear bond strength (SBS) between the different groups were statistically significant ($p = 0.0469$). The highest SBS was achieved for the group where the 10 wt% SA liquid was applied before the bonding procedures. The application of liquid 10 wt% SA might reverse the negative impact that bleaching has on the bond strength of a resin composite and enamel.

Keywords: antioxidants; bleaching; bond strength; enamel; in vitro

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1. Introduction

In the interest of looking good, humankind has continually struggled to advance people's facial characteristics. Since the alignment and the appearance of teeth can impact one's personality, they have received significant attention [1,2]. With a rising understanding of aesthetic dentistry, bleaching has become a common procedure amongst the numerous treatment approaches available to address this demand, since it is a well-accepted, conservative, safe, efficient, and non-invasive treatment [3].

Patients are generally displeased with the color of their teeth, and the best solution for this problem is bleaching. This can be achieved either by the dental practitioner using bleaching products with a high concentration of a bleaching agent, or by the patient dispensing a low concentration of bleaching agents into a custom-fit tray [4].

One should bear in mind that the active component existing in most bleaching agents is hydrogen peroxide (HP) [5]. This durable oxidizing component breaks down the large, pigmented molecules that cause tooth discoloration into smaller and less pigmented ones [6]. However, the negative impact of bleaching on the dental pulp, periodontal tissues, and enamel topography has been described [7]. Following the bleaching procedure, the enamel surface presents residual oxygen free radicals which hinder the photopolymerization of resin dental materials, subsequently resulting in a decline in the bond strength of enamel [8]. This adverse impact can be reversed in a stage lasting from 24 h to 3 weeks [9], and a minimum postponement of 14 days has been recommended before the execution of any adhesive treatment [10]. Nevertheless, in a clinical situation, such a delay may have problematic results, because patients often need restorative treatments immediately after the process of bleaching [11], in addition to bracket cementation [12]. A delay in the restorative procedure can lead to unexpected consequences, such as tooth fracture [13].

Numerous treatment modalities for immediately reversing this negative effect have been proposed, including the treatment of the bleached enamel with alcohol prior to restoration, the elimination of the superficial coat of enamel before restoration, or the use of an adhesive comprising organic solvents [13,14]. If the management of bleached enamel before bonding is helpful for reversing the lessened bond strength of composite resin, it could be a sustainable alternative to the delayed bonding approach after an enamel bleaching procedure [15]. Furthermore, another study assessed the use of sodium ascorbate (SA) (an antioxidant agent) formulated as a solution or a hydrogel and established no statistical variances between the preparations, indicating that bond strengths were meaningfully improved following the use of SA [16]. Specifically, SA, when applied for 10 min as a 10% solution, permits the free radicals of the adhesive monomers to continue their photopolymerization without early interruption by reestablishing the altered redox potential of the oxidized adherent, accordingly, improving the enamel's bonding to the resin composite [17]. In this manner, different viscosities of SA might be helpful for clinicians to improve the enamel's bond strength to the resin composite after HP application.

Therefore, this article sought to evaluate the effect of diverse SA presentations on the composite resin–enamel bond strength after a bleaching protocol. According to the null hypothesis, the different SA presentations do not have any effect on the composite resin–enamel bond strength after a bleaching protocol.

2. Materials and Methods

2.1. Study Design

In this study, the bond strength of a composite resin to bleached enamel was assessed according to the following factor: (1) the presentation of a 10%wt SA solution in three states—liquid, gel, and semi-gel. The gel state has been described phenomenologically as a solid or solid-like material containing two or more constituents, one of which is a liquid, present in a considerable quantity. However, the semi-gel state has been defined as a semi-solid, or semi-solid-like material. A cohesive powder was used as an explosive in this type. A group where the enamel was not bleached was used as a negative control in this experiment. Additionally, a group bonded immediately after bleaching the enamel was used as a positive control. In addition to this, a group consisting of bleached specimens bonded after 14 days of storage in saliva was used for comparison purposes. The focus of this article was the bond strength (n = 6), which served as the primary response variable. To determine the sample size, a prior study [4] that examined the bond strength of a resin-based substance on bleached enamel in a comparative study design with six separate groups was used as a reference. The estimation considered a mean minimum detectable difference of 7.92, with a standard deviation of 2.03, a power of 0.8, and a significance level of 0.05. The estimation of the sample size was performed using G*Power 3.1.9.6 software for Mac OS X. The products tested in this study were prepared and provided by Optident (Ilkley, UK). The proportion of the components and details for the preparation are not provided due to limitations based of the protection intellectual property.

2.2. Specimen Fabrication

After the approval of the study protocol by the Institutional Review Board of Saint-Joseph University (ref.# USJ-2023-47) of Beirut, Lebanon, sound human anterior teeth (n = 36; age range: 18–30 years) free from caries and restorations extracted for orthodontic or periodontal reasons were used in this study.

The roots of each tooth were sectioned around 2 mm from the cemento-enamel junction (CEJ) apically using (Exakt Technologies Inc., Norderstedt, Germany). The individual crown of each tooth was later cut in the mesio-distal direction, and the buccal side of crowns was employed in this article. The buccal side of crowns was fixed in an acrylic resin (Supacryl Ortho, Faprodent, Marrakech, Morocco) block, keeping only the buccal part exposed, and were exposed to 600 grit silicon carbide paper (Buehler Ltd., Lake Bluff, IL, USA) to attain a flat and rough enamel surface. Next, all specimens were observed under a light microscope to validate enamel exposure rather than dentin.

Afterwards, all specimens were allocated randomly into 6 groups comprising 6 samples each. Group 1 (which served as a control) did not undergo bleaching treatment or antioxidant application. Samples were acid etched with 37% phosphoric DENTOETCH acid (Itena Clinical, Paris, France) for 30 s, rinsed for 30 s and air-dried for 10 s. A thin layer of adhesive agent (Iperbond max universal adhesive, Itena Company, Paris, France) was applied for 20 s with a rubbing motion on the demineralized enamel and slightly spread with an air stream until it was not possible to see any freely moving liquid film. Next, light curing was conducted at room temperature for 20 s by using a light curing unit equipped with a light emitting diode (LED) multiwave curing pen (Eighteeth, Changzhou, China) using an irradiance of 1000 mW/cm^2. Following the adhesive protocol, a cylindrical polyethylene mold with an internal diameter of 2.38 mm and a height of 2.15 mm (Bonding Jig, Ultradent Products, Inc., South Jordan, UT, USA) was then employed on the surface of the samples. Afterward, the molds were covered with a flowable resin composite (Reflectys flow, Itena Company, Paris, France) cylinder for each specimen in at a 2 mm level or less, and then light-cured with the same light-curing material for 40 s. Thus, the approximate bonding area was circular in form.

In groups 2–6, specimens were bleached using 10% carbamide peroxide (CP) (White Dental Beauty, Optident, Ilkley, UK) according to the manufacturer's directions. The vestibular surface of the teeth was covered with a bleaching gel for 8 h daily, for 14 days. Following daily application, teeth were completely rinsed with distilled water and air-dried for 30 s. For the remainder of the day, they were conserved in distilled water at room temperature. So, in group 2, only bleaching was performed without any additional treatment. Furthermore, in groups 3, 4 and 5, 10%wt SA (Sodium L-ascorbate, Sigma Aldrich, Serva, Heidelberg, Germany) was applied as an antioxidant in the form of a liquid, gel, and semi-gel, respectively. The experimental hydrogels which have not yet been commercialized, the SA gel, and semi-gel were applied on the enamel surface using a syringe (1 mL) for 1 min, and then the teeth were left until achieving an application time of 10 min. Same for the 10%wt SA solution. After that, the enamel was then rinsed using distilled water for 30 s. Then, the teeth were restored using flowable resin composite (Reflectys flow, Itena Company, Paris, France) as in Group 1. In Group 6, specimens were bleached and immersed in human saliva for 2 weeks. After the storage time, they were restored in a similar way to as in Group 1. To reach 10% SA, 10 g of SA crystals was dissolved in 100 mL of distilled water.

After the restorative procedures, the samples were kept in distilled water at 37 °C for 24 h. Next, they were exposed to a shear bond strength (SBS) test. A stainless-steel wire (0.2 mm diameter) was looped around the restoration and associated with the bonded interface for the shear test. The universal testing machine YL-01 (YLE GmbH, Bad König, Germany) was employed to measure the SBS at a crosshead speed of 0.5 mm/min and with a load cell of 1000 N. The bond strength value was calculated by dividing the measured debonding force by the size of the area bonded. This was performed in accordance with the following formula: R = F/A (R: the bond strength in MPa, F: the failure force in Newtons, and A: the bonding area in mm^2).

2.3. Statistical Analysis

The statistical tests were performed by means of a Sigma Plot v12.0 software. The data were analyzed to authenticate the normal distribution and variance homogeneity. A one-way analysis of variance (ANOVA) test was implemented to compare the SBS among the different groups. Multiple comparison procedures were executed by means of a Tukey test. The level of significance was fixed to $p < 0.05$ for all tests.

3. Results

The differences between the SBS among the different groups were statistically significant (Table 1, $p = 0.0469$). The results of SBS for every single group are presented in Table 2. The highest SBS was achieved for the group where the 10 wt% SA liquid was applied before the bonding procedure, and this group demonstrated statistically significant differences only when compared with the group which corresponds to the bleached enamel being bonded immediately (G2).

Table 1. Analysis of variance (ANOVA).

Source of Variation	Sum of Squares	d.f.	Variance	F	*p*
Between groups	232.56	5	46.512	2.5779	0.0469
Within groups	541.278	30	18.042		
Total	773.8380	35			

Table 2. Shear bond strength (SBS) of the evaluated groups.

Group	Mean	RSD *
G1: Non-bleached enamel	15.9 (2.9) ^{ab}	18.2%
G2: Bleached enamel bonded immediately	11.4 (3.6) ^{b}	31.6%
G3: Bleached enamel bonded after application of 10% sodium ascorbate liquid.	19.5 (2.8) ^{a}	14.4%
G4: Bleached enamel bonded after application of 10% sodium ascorbate gel.	13.0 (3.9) ^{ab}	30%
G5: Bleached enamel bonded after application of 10% sodium ascorbate semi-gel.	14.2 (5.16) ^{ab}	36.3%
G6: Bleached enamel bonded after 14 days of storage in saliva	15.4 (6.1) ^{ab}	39.6%

Different superscript letters indicate the presence of statistically significant differences ($p < 0.05$). * Relative standard deviation.

4. Discussion

In this article, the bond strength of a flowable resin composite to bleached enamel was tested according to the protocol of SA application in three forms (liquid, gel, and semi-gel). A group without bleaching and without SA application was used as a control. Additionally, a group where bleaching was performed but the restoration procedures were performed after 14 days of storing the specimen in fresh human saliva was used as reference. The results of this analysis reveal that the differences between the SBS among the different groups were statistically significant ($p = 0.0469$). Accordingly, the null hypothesis stating that the different SA viscosities do not have any effect on the composite resin–enamel bond strength after a bleaching protocol can be rejected.

One should note that home bleaching by means of 10% to 22% CP was considered an approach applied by the patient [18]. Previous reports confirmed that the enamel bond strength lessened after bleaching with CP in numerous concentrations [18,19]. The bleaching agents employ free radicals such as hydroxyl or peri-hydroxyl ions and nascent oxygen when they are used on the dental surface. It is important to mention that a free radical is a molecule that has one unpaired electron, providing a high reactivity. So, these molecules can react with electron-rich areas of the pigmented dental structure, henceforth breaking down the large, pigmented molecules into smaller ones with less pigmentation [20]. This feature of bleaching products is detrimental to the bonding of resin materials to the dental substrate. The impact of bleaching agents on the bonding demonstrates that peroxides, along with their byproducts present inside the dental structure, interfere with the process of photopolymerization of the adhesive material [21–23].

According to the results of this study, a reduction in SBS was seen after bleaching the enamel when compared to the group with unbleached enamel. However, the variance was not statistically noteworthy. In addition, the bond strength significantly increased in the group where 10 wt% SA was used as an antioxidant in the form of a liquid after bleaching the enamel and was significantly higher compared to the other groups ($p = 0.0469$).

Furthermore, the RSD in 10% liquid group is the smallest compared to other groups, meaning that the bond strength is the most reliable (in terms of less data scattering), even better than non-bleached enamel. On the other hand, these outcomes demonstrate that the enamel–resin SBS is meaningfully reduced when the restoration is completed directly after bleaching with 10% CP gels. This agreed with a previous manuscript displaying that a lower concentration of CP lessened enamel bond strength [24]. The reasons for the reduction in bond strength directly after enamel bleaching are as follows: the residual oxygen present on the enamel surface precludes the penetration of the resin monomers inside demineralized enamel or partly restricts their photopolymerization; the highly reactive oxygen reacts with the free radicals of the adhesive, hindering their photopolymerization and creating polymers with inferior mechanical properties [6,25]; and the drop in the surface energy of collagen fibers after bleaching leads to a structural deformation and reduced bond strength [26].

Therefore, the use of antioxidant agents to reverse this side effect has been recommended to allow the use of the immediate adhesive method [27]. SA is a synthetic antioxidant that has been widely reviewed for this purpose [28]. It was discovered that a 10 wt% SA solution applied to the enamel surface for a period of 10 min improved the bond strength of the surface on which it was applied [4]. This was in accordance with the outcome of this research article. However, the antioxidant was converted into gel and semi-gel form and used in these different viscosities in the current manuscript, showing reduced bond strength, contradicting the results obtained by a preceding article which showed no variance in enamel bond strength with respect to the use of two forms, a solution and hydrogel of SA [16].

It is essential to note that the gel form significantly increased the enamel bond strength when applied for 120 min or more [18]. This was not in agreement with the methodology of this research, since different viscosities were applied for 10 min, and only the solution form was effective. When chemical substances are transformed into the gel form, their drug relief rates are slower than when they are in a solution form [29,30]. So, their effectiveness might be better over a longer timeframe. These observations confirm that an antioxidant gel or semi-gel becomes efficient on the enamel surface after a long period. A preceding report confirmed that a gel form applied for 3 h increased enamel bond strength, and as the application period of the antioxidant on the enamel substrate increased, the bond strength of the composite to the enamel surface also improved [16].

In addition, while the liquid form reversed the compromised bond strength, and the salt of ascorbic acid prevented the double etching effect of ascorbic acid on enamel, permitting the complete photopolymerization of the adhesives without premature termination. These effects agree with the results of previous manuscripts [31–33], where the antioxidant restores the distorted redox potential, enables free radical polymerization, and reverses the compromised enamel bonding. Therefore, it can be stated that 10 wt% SA in solution form is recommended for a shorter application time, disregarding the necessity for delaying composite restorations after bleaching.

It should be noted that a 14-day storage period during which the peroxide ions decompose, and the subsequent replaced hydroxyl radicals reinstate the apatite level, leads to the removal of structural modifications produced by the incorporation of peroxide ions [34,35]. Thereafter, postponing the bonding for 1 to 3 weeks following dental bleaching treatment was proposed [36]. This explained the non-difference between the unbleached enamel and the bleached enamel undergoing 14 days of storage in saliva obtained in this study.

Moreover, it can be suggested that the enamel substrate interacts with air and may contain oxygen. In addition, it is covered with water and organic molecules and contain oxygen. So, the oxygen component on the unbleached enamel may likewise hinder photopolymerization at the interface between enamel and resin. Subsequently, the superior bond strength obtained with antioxidants in the form of a liquid when compared to unbleached specimens could be due to the neutralization aptitude of oxygen, which is provided not only by the bleaching product but also by the enamel or the air [37].

It was essential to prepare the antioxidant liquid, gel, or semi-gel on same day since the efficacy of 10 wt% SA solution is short-lived. In the present article, if the container cover was not opened, the efficiency of these solutions can be maintained for a long time. Consequently, the use of this antioxidant is easier for dentists. The limitations of this research should also be considered. First, an in vitro design has some limitations, and the results cannot be extrapolated to a clinical situation. Meanwhile, the SBS was only tested immediately, and future studies should take in account the evaluation of the bond strength after aging with a failure mode. Additionally, only one brand of adhesive system, resin composite and bleaching agent was tested, respectively. Further research should be encouraged to conduct well-designed clinical trials to explore these features in a clinical scenario. Furthermore, alternative formulations that have the potential to counteract the decrease in bond strength following bleaching can be investigated. This exploration may involve the utilization of natural hydrogels and natural antioxidants [38,39]. Moreover, the molecular constituents of dental hard tissues such as enamel have been the subject of research using a diversity of methods, such as electronic microprobes, infrared spectroscopy, and Raman spectroscopy. X-ray microanalysis by means of energy-dispersive X-ray analysis (EDAX) was used previously in the study of matrix elements such as phosphate and calcium [40]. Thus, this approach should be further evaluated. Finally, studies may be required to implement similar bleaching procedures and highlight the effect of different viscosities of SA on the color change of the enamel structure in both in vitro and clinical scenarios.

5. Conclusions

Taking into account the limitations of the present research, it could be determined that the application of liquid 10 wt% SA might reverse the negative impact that bleaching has on the bond strength of a resin composite and enamel. Additionally, it can be hypothesized that the influence of an antioxidant depends on its viscosity, form, and duration of application.

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