The impact of etching time and material on bond strength of self-adhesive resin cement to eroded enamel

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INTRODUCTION

Loss of tooth structure as a result of dental erosion is an issue of growing solicitude¹,²), and the process loss of mineral content of tooth structure is usually progressive, slow, and pain-free³). Erosive tooth wear is often unnoticed by patients and even dentists and habitually recognized at a progressive phase when teeth damage has occurred⁴,⁵).

The causes of dental erosion may be intrinsic, as a result of gastroesophageal reflux, chronic alcoholism, and bulimia nervosa or extrinsic, usually by the action of dietary acids⁶-¹⁰). Gastroesophageal reflux disease, which is the main reason of intrinsic erosion, is a condition where gastric acids are regurgitated back to the oral cavity, thus give rise to persistent oral exposure to an acidic medium.

Initially the process of acid erosion begins with softening of the exterior surface of enamel and the effect can be varied based on the acids pH and the time of exposure¹¹-¹⁵). In patients with a record of intensive vomiting and gastroesophageal reflux, serious lingual-occlusal erosion is almost existing and in more sophisticated conditions, inclusive mineral sacrifice can produce tooth shortening, which can contribute to aesthetic and structural challenges¹³,¹⁶-¹⁸).

As the management for erosive teeth wear should be primarily focused on the etiology, prevention and to overcome the dilemma of excessive loss of tooth structure¹⁹,²⁰), the restorative approach necessitates a precise planning, based on the extent of loss²¹). With the improvements in field of adhesive technologies, it has become possible to restore eroded teeth in a minimal invasive mode. Ultra-thin bonded ceramic veneers have been discussed as an alternative treatment option of severe erosive lesions compared to the conventional onlays and complete coverage crowns¹¹,¹²). Bonded composite resins and ceramics address the previously described biomimetic principles of ultimate conservation of teeth tissue and esthetics²³-²⁵).

For adhesive cementation, luting resin cements are the recommended option⁴,⁶). While surface modification of the dental hard tissues are required before cementation, therefore, resin cements can be categorized as total etch resin cements needs phosphoric acid etching and bonding adhesive application before cementation; Self-adhesive resin cements have the capability to bind to tooth structures in absence of extra adhesives; and Self-etch resin cements requires an acidic monomer that is integrated in its composition, to alter the surfaces of dental tissue before curing²⁶).

Long-term stability of bonding to tooth structure remains questionable. The results of enamel bond strengths to indirect restorations utilizing self-adhesive cements have many conflicting reports and to promote the adhesion process, the application of phosphoric acid before cementation has been suggested¹¹,¹²). Although, it is likely that phosphoric acid etching of the enamel surface created rough irregular surface that may be a causative factor of enamel bonding failure²⁶), especially in patients with a history of enamel surface dissolution as a result of acid erosion²⁷,²⁸). Therefore, to minimize the extent of enamel surface damage, alternative conditioners, such as maleic acid, nitric acid, polyacrylic acid and ethylenediaminetetra acetic acid, have been used to obtain clinically durable bond strengths by reducing the depth of enamel dissolution²⁹,³₀).

Therefore, the current study directed to estimate
the effects of use lactic acid, phosphoric acid for etching the previously eroded enamel. The effectiveness of eroded enamel surface conditioning before material implementation was also tested. The hypothesis of this study was that, the use of 20% of lactic acid for surface conditioning of eroded enamel increases the bond strength to self-adhesive cement.

MATERIALS AND METHODS

Enamel sample preparation

Thirty extracted human caries-free anonymized molars were recruited in this research. The selected molars were cleaned, disinfected using 0.5% chloramine and stored in distilled water until use. Approval to use human teeth was obtained from the Research Ethics Committee (No.0613118) at the Faculty of Dentistry, Mansoura University, Egypt.

Following mesio-distal sectioning (Isomet 1000TM, Buehler, Lake Bluff, IL, USA) of the coronal part of the teeth, the enamel bonding sites (5×3×2 mm) were prepared from the mid-coronal section of the buccal and lingual surfaces.

So as to remove superficial enamel layer and to create a polished flat surface, the teeth were ground with a with 4000-grit polishing paper under copious air-water coolant.

Initial assessment of enamel hardness

Initial assessment of enamel hardness was carried out utilizing a Vickers indenter linked to a microhardness tester (FutureTech, Tokyo, Japan). Six indentations per test were made at the middle section of each specimen at 300 μm interval. The load for indentation was 100 g with 15 s dwell time. The specimens with mean hardness values 10% above or below the general mean were ignored (mean surface hardness of 334.56±30.71 VHN)\(^{30}\).

One hundred-twenty specimens were recruited, one hundred specimens were submitted to erosive challenge and twenty specimens were used as a control.

Assessment of enamel hardness following simulated erosive lesion

The erosive lesion was attained by subjecting the enamel specimens to short-term acidic exposure to HCl solution (0.01 M, pH 2.3) for 30 s, with agitation at speed of 50 rpm at room temperature (25°C). This protocol provoked erosion of the composite resin bonding surfaces were performed utilizing 50-μm Al\(_2\)O\(_3\) for 10 s at a distance of 10 mm, washed under distilled water, and air dried. Subsequently, a silane primer (Ceramic Primer, 3M ESPE) was dispensed to the composite bonding surface, allowed to dry for one minute, and air dried for 30 s.

Composite resin cylinders were bonded to the treated enamel surfaces with either Panavia SA or Multilink speed self-adhesive resin cement (n=10 for each subgroup) based on the recommendation of the manufacture (Table 1).

All specimens were stored in distilled water prior onset and in the middle of all steps of testing.

A total of 120 composite resin cylinder (Filtek Z250, 3M ESPE, Seefeld, Germany) were condensed into 2.38 mm diameter , 3 mm height transparent plastic molds and photo-polymerized for 3 min followed by further polymerization for 10 min inside a polymerization unit (Triad 2000 System, Dentsply Trubyte, York, PA, USA) at 1,000 mW/cm\(^2\). Airborne-particle abrasion of the composite resin bonding surfaces were performed utilizing 50-μm Al\(_2\)O\(_3\) for 10 s at a distance of 10 mm, washed under distilled water, and air dried. Subsequently, a silane primer (Ceramic Primer, 3M ESPE) was dispensed to the composite bonding surface, allowed to dry for one minute, and air dried for 30 s.

Composite resin cylinders were bonded to the treated enamel surfaces with either Panavia SA or Multilink speed self-adhesive resin cement (n=10 for each subgroup) based on the recommendation of the manufacture (Table 1).

All specimens were stored in distilled water at 37°C for one day. Then, the specimens were subjected to thermocycling for 6,000 cycles between 5±2°C and 55±2°C, with a dwell time of 20 s and a transfer time of 5 s.

Each specimen was locked within a special designing device mounted on the universal testing machine (Instron 4204, Instron, Canton, MA, USA). A load was exerted to the bonded zone between the enamel and composite resin cylinder from a notched-edge fixture and a crosshead speed of 1 mm/min (Fig. 5). The recorded maximum loads were divided by the surface area of the enamel-composite interface to calculate the shear bond strength (SBS) in megapascals (MPa).

After SBS test, the tooth surface and composite resin were viewed under a light optical stereomicroscope (Carl-Zeiss, Oberkochen, Germany) at 40× magnification to detect the mode of failure. The portion of adhesive
Fig. 1 SEM micrographs (1,000×) showing eroded enamel surface.

Fig. 2 SEM micrographs (1,000×) eroded enamel surface treated with 35% phosphoric acid etching for 15 s.

Fig. 3 SEM micrographs (1,000×) eroded enamel surface treated with 35% phosphoric acid etching for 30 s.

Fig. 4 SEM micrographs (1,000×) eroded enamel surface treated with 20% Lactic for 30 s.

Table 1 Name, composition, manufacture and method of application of self-adhesive cement used in this study

<table>
<thead>
<tr>
<th>Material</th>
<th>Composition</th>
<th>Manufacturer</th>
<th>Application technique</th>
</tr>
</thead>
</table>
| Panavia SA plus| A paste: Bis-GMA, TEGDMA, dimethacrylate, 10-MDP, silanized Ba glass filler, silanized colloidal silica, photo-initiator, chemical-initiator B paste: Bis-GMA, dimethacrylate, silanized Ba glass filler, silanized colloidal silica, silanized NaF, chemical accelerator, pigment. | Kuraray, Osaka, Japan | 1. Dispense the cement paste in 1:1 ratio from double-push syringe and mix 2 pastes on mixing pad before restoration is loaded.  
2. Load the intaglio surface of restoration with mixed cement.  
3. Seat the restoration in place and hold. Immediately, remove excesses cement with disposable brush and wait for 3 min. |
| Multilink Speed CEM | Base paste: UDMA, TEGDMA, polyethylene glycol dimethacrylate  
Catalyst paste: polyethylene glycol dimethacrylate, TEGDMA, methacrylated phosphoric acid ester, UDMA | Ivoclar Vivadent, Schaan, Liechtenstein | 1. Dispense the cement paste in 1:1 ratio from double-push syringe and mix on mixing pad directly before restoration is placed.  
2. Load the intaglio surface of restoration with mixed cement.  
3. Seat restoration in place and hold. Immediately, remove excesses with disposable brush and wait for 3 min. |
resin surface with adherent enamel and visible remains was calculated roughly to consider the style of failure (adhesive failure, cohesive failure in composite cylinder, cohesive failure in enamel or mixed failure).

### Statistical analysis

Statistical analysis was carried out using SPSS 17.0 software for Windows (SPSS, Chicago, IL, USA). Levene’s normality and Kolmogorov-Smirnov tests showed that SBS data (MPa) were homogeneously distributed. Therefore, parametric (ANOVA) analysis test was performed to test the difference of the SBS values between groups under study. Tukey’s HSD post-hoc test for multiple comparisons was made. $p$ Values less than 0.05 were regarded statistically significant.

### RESULTS

Table 2 lists SBS values and standard deviation for the tested groups. Tukey’s test revealed significant difference ($p<0.05$) in SBS of L30 and Ph15 treated

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**Table 2** Mean and SD of SBS values of tested groups

<table>
<thead>
<tr>
<th>Etching</th>
<th>cement</th>
<th>Mean</th>
<th>Std. Deviation</th>
<th>$n$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>Panavia SA</td>
<td>14.8a</td>
<td>3.49427</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td>Multilink SP</td>
<td>13.4a</td>
<td>1.64655</td>
<td>10</td>
</tr>
<tr>
<td>without etching</td>
<td>Panavia SA</td>
<td>19.2b</td>
<td>2.42</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td>Multilink SP</td>
<td>16.0a</td>
<td>3.0</td>
<td>10</td>
</tr>
<tr>
<td>Ph etching 15 s</td>
<td>Panavia SA</td>
<td>25.3c</td>
<td>3.6</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td>Multilink SP</td>
<td>19.9b</td>
<td>2.7</td>
<td>10</td>
</tr>
<tr>
<td>Ph etching 30 s</td>
<td>Panavia SA</td>
<td>15.0a</td>
<td>3.5</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td>Multilink SP</td>
<td>13.4a</td>
<td>4.2</td>
<td>10</td>
</tr>
<tr>
<td>Lact etching 30 s</td>
<td>Panavia SA</td>
<td>26.3c</td>
<td>2.8</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td>Multilink SP</td>
<td>21.8b</td>
<td>4.6</td>
<td>10</td>
</tr>
<tr>
<td>Lact etching 60 s</td>
<td>Panavia SA</td>
<td>21.2b</td>
<td>3.0</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td>Multilink SP</td>
<td>19.8b</td>
<td>2.8</td>
<td>10</td>
</tr>
</tbody>
</table>

Groups with same letters do not have significant differences in columns ($p>0.05$)

**Table 3** Summary of 2-way ANOVA for representation of interactions between etchant type/resin cement variables of shear bond strength to eroded enamel surface

<table>
<thead>
<tr>
<th>Source</th>
<th>Type III sum of squares</th>
<th>df</th>
<th>Mean square</th>
<th>F</th>
<th>Sig.</th>
<th>Partial Eta squared</th>
<th>Noncent. parameter</th>
<th>Observed power$^b$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Corrected model</td>
<td>2,117.949$^a$</td>
<td>11</td>
<td>192.541</td>
<td>18.028</td>
<td>0.000</td>
<td>0.647</td>
<td>198.304</td>
<td>1.000</td>
</tr>
<tr>
<td>Intercept</td>
<td>42,755.650</td>
<td>1</td>
<td>42,755.650</td>
<td>4,003.230</td>
<td>0.000</td>
<td>0.974</td>
<td>4,003.230</td>
<td>1.000</td>
</tr>
<tr>
<td>Etching</td>
<td>1,780.937</td>
<td>5</td>
<td>356.187</td>
<td>33.350</td>
<td>0.000</td>
<td>0.607</td>
<td>166.750</td>
<td>1.000</td>
</tr>
<tr>
<td>Cement</td>
<td>261.370</td>
<td>1</td>
<td>261.370</td>
<td>24.472</td>
<td>0.000</td>
<td>0.185</td>
<td>24.472</td>
<td>0.998</td>
</tr>
<tr>
<td>Etching$^a$ cement</td>
<td>75.641</td>
<td>5</td>
<td>15.128</td>
<td>1.416</td>
<td>0.224</td>
<td>0.062</td>
<td>7.082</td>
<td>0.483</td>
</tr>
<tr>
<td>Error</td>
<td>1,153.471</td>
<td>108</td>
<td>10.680</td>
<td>—</td>
<td>—</td>
<td>—</td>
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</tr>
<tr>
<td>Total</td>
<td>46,027.070</td>
<td>120</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Corrected total</td>
<td>3,271.420</td>
<td>119</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
</tbody>
</table>

a. R Squared=0.647 (Adjusted R Squared=0.611)
b. Computed using alpha=0.05
groups compared to L60, Ph30, N and C experimental treatments. No significant differences were found among SBS values of Group C and PH30. The highest overall bond strength (MPa) was observed in 30 s lactic acid treated group; 26.3±2.8 for Panavia SA and 21.8±4.6 for Multilink SP, followed by both Ph15; 25.3±3.6 for Panavia SA and 19.9±2.7 for Multilink SP, followed by L60 group; 21.2±3 for Panavia SA and 19.8±2.8 for Multilink SP self-adhesive cement. The control group showed the lowest SBS; followed by Ph30 group; 5±3.5 for Panavia SA and 13.4±4.2 for Multilink SP. Eroded group without any surface treatment showed SBS comparable to Ph30 group; 19.2±2.4 for Panavia SA and 16±3 for Multilink SP. There was a significant difference (p<0.05) in SBS of L30, Ph15 and L60 groups to control group. Two-way ANOVA (Table 3) revealed that etching material and cement type had a significant influence (p<0.05) on SBS values while there was no significant (p>0.05) interaction between the two factors.

In all study groups, adhesive failures were recorded (C>N>Ph30>Ph15>L60>L30).

However, an increase in cohesive failure within the cement film was noticed in relation with eroded enamel with Panavia SA resin cement. In contrast, an increase in adhesive failure happened when enamel was etched for the self-etch resin cement, Multilink SP resin cement.

**DISCUSSION**

Although bonding to enamel is much easier than bonding to dentin surface, there are three potential obstacles to establish an optimal bonding to enamel surface. First, 1–2 μm-thick hydrophobic smear layer that cover the surface of ground enamel, which reduces the surface wettability. Second, the mineral component of enamel is approximately 96% by weight with only 4% organic components and water which results in hydrophobic enamel surface compared to dentin. Third, the variation in resistance against acid dissolution of prismatic and a-prismatic enamel leads to different etching pattern. Enamel pre-etching with phosphoric acid removes the smear layer, changing the surface properties and increases the wettability of enamel surface, by exposing the hydroxyl groups of the enamel, therefore producing a consistent enamel etching pattern.

When erosive demineralization occurs, it leads to the production of a superficial mineral dissolution with an irregular structure similar to the pattern of etched enamel surface known from well-established adhesive bonding. Till now, not too much is recognized about how deep the partly demineralized area extends; values spread between a not many microns up to around 100 μm have been reported. When the acid exposure continues, mass enamel loss occurs.

In order to exclude the possibility of any sample bias in this study, the surface hardness after erosive lesion formation was measured following the identical guidelines used for former superficial hardness measurement. This step was carried out to assure the lesion establishment and to select and randomize specimens to each of groups.

The values recorded for the human samples (Vickers) are reinforced within the literature, with studies reporting baseline human enamel Vickers microhardness from 304 to 409.

In the current investigation, the effect of initial erosive lesion was assessed. Intrinsic acid erosion needs gastroesophageal treatment and/or psychological treatment but due to the gradual and often sporadic nature of erosive tooth wear, early diagnosis is difficult. Therefore, it is important for the clinician to be familiar with and recognize the erosive wear lesions as early as possible to implement preventive measures and preserve tooth tissue.

In present study, the specimens were subjected to de-mineralization in hydrochloric acid solution simulating a moderate kind of gastric regurgitation. Mann et al., performed an in vitro study to study the impact of repetitive short erosive attacks to enamel surface over 2 min at pH 1.5 and 3 under situations imitating gastric regurgitation and they concluded that erosive lesions occurring under situations simulating gastro-oesophageal reflux disease can be discovred in its early stage, setting up chances of rapid detection and handling of this status. Their model presented an evidence of meaning for prophilometric disclosure of early erosion which provide a foundation for emergence of highly sensible clinical diagnostic tools and preventive options. Also, they emphasized the demand for quick diagnosis and handling in line with minimal intervention philosophy.

Since the pioneering work of enamel etching with phosphoric acid by Buonocore, numerous acids have been proposed for enamel etching which included citric, oxalic, maleic and nitric acid. Despite the research into alternative acids for enamel etching, the gold standard for enamel etching is phosphoric acid. While Buonocore first used phosphoric acid at a concentration of 85%, the concentration in clinical use today has progressively decreased to a range of 30–40%.

The findings of the current study revealed that the utilization of 20% lactic acid produce significant rise in the bond strength to eroded enamel compared to 35% phosphoric acid treatment for 15 s. The mechanism of conditioning the enamel with lactic acid is not purely etching but sort of an exterior etching accompanied with chelation of calcium of hydroxyapatite. The chelation creates a steady, homogeneous and insoluble chelated layer that is chemically bond to enamel surface.

This finding was supported by the results of other studies, which recorded that the mild lactic acid may be appropriate alternative enamel conditioning agents to phosphoric acid.

The results from SEM observation, present that, the enamel etching patterns of groups treated with phosphoric acid was coarser and demonstrated baggy enamel penetration bores than those treated with lactic acid. It points out that lactic acid conditioner did less harm to the previously eroded enamel surface and produced less enamel penetration bores so danger of
later disintegration of the enamel underneath or around the restoration may be considerably reduced.

Although experimental studies provide valuable information of the biomaterials and rank their performances, they do not mimic the clinical situations at oral conditions. Therefore, controlled clinical trials are recommended.

CONCLUSION
Bond strength of self-adhesive cement to eroded enamel surface significantly enhanced following application of 20% lactic acid for 30 s. Regardless the type of enamel surface treatment protocol used, Panavia SA resin cement demonstrated superior bonding, when compared to Multilink speed.

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