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ORIGINAL ARTICLE

An In Vitro Study to Compare the Effect of Two Etching Techniques on the Tensile Bond Strength of Resin Cement Bonded to Base Metal Alloy and Enamel

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Abstract Resin-bonded retainers are being preferred for anterior restorations. To increase the retentive strength of the metal fixed to the tooth, the retainer surface has to be etched. Different etching techniques are described in the literature with different researchers expressing the superiority of one technique over the other. This study was conducted to compare electro chemical and chemical etching techniques and the mode of bond failure. Twenty human maxillary premolars with the crown portion separated from root were embedded in resin block such that mesial or distal portion of it was exposed on the top of the block. 4×5 mm area was marked on the tooth, and wax pattern was prepared to cover the exact area, with the opposite end having a hook like structure which was later attached to universal testing machine. Wiron99 Ni–Cr alloy was used for casting. Once the casting and etching procedures were finished, wax patterns were invested, casted and half the samples were etched chemically using Aqua-regia and the other half samples were etched electrochemically. The castings were cleaned and cemented to tooth structure using Rely-X ARC (3 M ESPE, USA) resin cement. Specimens were fixed to universal testing machine and de-bonded. The load required to de-bond and mode of de-bonding was noted. Results were subjected to five different statistical tests, each test specific to the variable being tested. The mean failure load was calculated as 5.95 kg for electrochemically etched samples and that of chemically etched samples was calculated as 11.15 kg. The standard deviation of the force required to debond the specimens (Kgf) was calculated and found to be 0.65 for electrochemically etched samples and 1.11 for chemically etched samples. The following conclusions have been drawn from the study. 1. Chemical etching of the samples created better retentive surfaces than electrochemical etching. 2. The results of mode of debonding show that in case of chemical etching maximum debonding occurred at resin-enamel interface and in electrochemical type it occurred at resin-metal interface.

Keywords Chemical etching · Electro chemical etching · Current density

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Introduction

Retention is defined as "That quality inherent in dental prosthesis acting to resist the forces of dislodgement along the path of placement" [1]. Full coverage metal crowns and bridges achieve retention through various factors such as crown length, taper, surface area etc. In case of conservative preparations like resin bonded bridges, only enamel portion of the tooth is prepared and metal retainer is luted using resin cement. Retention in such prostheses is attained through rough surface created on retentive wings by various methods by the clinician or the technician. Resin bonded bridges have gained popularity since Rochette [2] described the use of a resin bonded perforated cast metal framework as a periodontal splint in 1973. The resin-bonded or "Maryland" bridge has been used in the anterior and posterior regions for many years. Practitioners have used this bridge either as an interim prosthesis or an alternative to implant replacement therapy or conventional fixed prosthodontics with a success rate of

87.7 % [3] while other studies [4] have found the success rates to be as high as 93.8 %.

These restorations had two main disadvantages:

1. Greater the number of retentive holes, weaker the framework
2. Exposed resin in the holes is abraded resulting in the failure of the bonded restoration [5].

In an attempt to improve the retentive properties of metal, several methods were developed by various researchers. Some of these were, creating pitting corrosion [6], particle roughening by using salt crystals [7], porous coatings of fine metal powder [8], electrolytic etching procedure by passing current of specific density through a solution which creates rough surface in the alloy [9], and a chemical etching system using aqua regia based etchant [10, 11]. Electro chemical etching was developed in order to discover a convenient method of increasing the bond strength of dental resin to metal surface. Specific etching conditions have been developed for various base metal alloys thereby improving bond strengths tremendously [9]. Disadvantages of this electrochemical technique despite various advancements have been that the laboratory procedure required considerable time and special equipment that is not readily available to all laboratories. In addition, etch produced was not uniform especially when the restoration has appreciably curved surface [12].

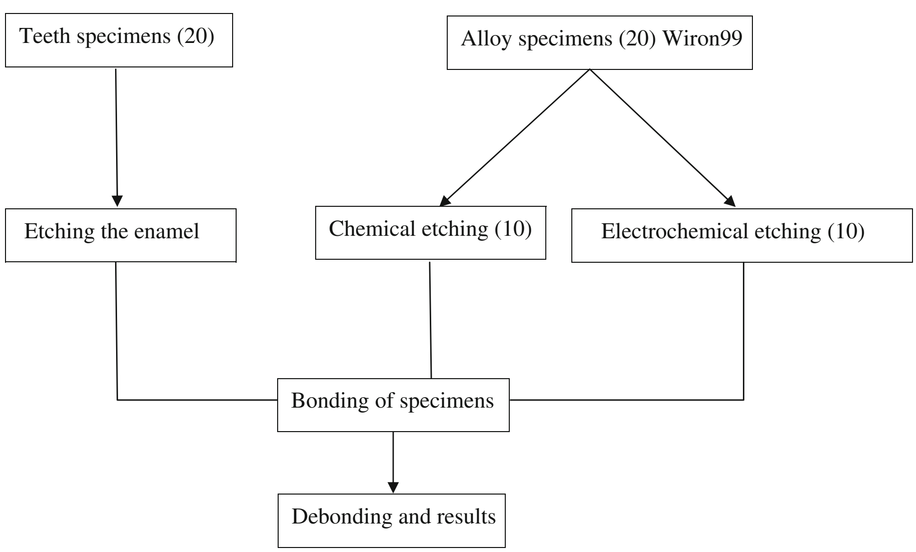
strength between metal-resin-enamel interfaces. Chemicals used for acid etching varied from 37 % phosphoric acid [13] to Aqua regia [10].

The need for the study lies in the fact that several conflicting reports have been published in the literature over many years regarding which form of etching has advantage over the other. Thompson VP claimed that electrochemical etching has created better bond strength than chemical etching [12]. Contrary to that Livaditis [11], El-sheriff et al. [1, 14] claim chemical etching to create better bonding. These controversies create confusion in the minds of clinicians. Although studies indicate etching of alloy creates better bond strength than the older methods like particle roughening or pitting corrosion [15], the question as to which type of etching creates better retention is still not conclusively answered.

Hence the present in vitro study has been undertaken to compare the effect of two etching techniques on the tensile bond strength of resin cement bonded to base metal alloy and enamel as well as to record the interface at which the bond failure occurs.

Materials and Methods

The study was carried out in the following sequence



To overcome the technical difficulties posed by electrochemical etching technique, a chemical etching technique has been discovered by Livaditis [11]. Chemical etching system was found not only to reduce the disadvantages involved in electrochemical etching but also proved to be better than the latter in terms of tensile bond

Preparation of Alloy Specimens

Twenty cylindrical wax patterns of 10 mm diameter, and 10 mm in height, each having a loop (hook) on one side and a flat rectangular side of four by five mm dimension on the other side were prepared using inlay wax (Crowax blue, Code: 023-474-0200, RENFERT, UK). Melting pot technique was used to create the wax specimens according to manufacturer's instructions. Nickel chromium alloy selected for this study was Wiron99 (Bego, Bremen, Germany). The composition of alloy was as follows:

Nickel—65 %
Chromium—22.5 %
Molybdenum—9.5 %
Nobelium—1 %
Silicon—1 %
Ferrum—0.5 %
Cerium—0.5 %
Carbon—0.02 %

Manufacturer's instructions were followed, along with ISO standardization protocol (ISO 9001/EN 29001) for casting procedure to eliminate any casting errors. The castings were recovered (Fig. 1) and sand blasted using micro sand blaster (Ivoclar AG-Type FK-2, Germany). Small imperfections on the metal cylinders were trimmed with tungsten carbide bur (Fine grit). Care was taken to ensure that the surface area of the rectangular flat surface on one end of the casting was maintained at 20 sq mm. The specimens were fired in a porcelain furnace (Ivoclar programat p-20, Ivoclar Vivadent AG, Schaan, Liechtenstein Germany) five times in order to simulate the individual firing cycles of porcelain fused to metal (Fig. 2). The samples were sand blasted and then cleaned in ultrasonic cleaner for 10 min with distilled water. Each specimen was air dried and stored in plastic packets at room temperature till they were removed and bonded to teeth.

Fig. 1 Ni–Cr alloy sample

Fig. 2 Alloy being subjected to firing cycle

Etching of Alloy Specimens

Electrochemical Etching

Apparatus required for etching consisted of a variable low voltage direct current, rheostat, ammeter and current meter (Fig. 3). Insulated copper wire was used as anode. Another insulated wire having a stainless steel rod at its tip was used as cathode. The electrolytic solution consisted of 10 % sulfuric acid mixed in methanol in the ratio of 9:1 respectively. The surface of alloy sample that was to be etched was carefully dipped into the solution taking care only to etch the required exposed surface. Specimens were attached to the anode. The cathode was dipped into solution keeping a distance of 1.5 cm from anode which is constantly maintained by piercing holes in a thin thermocol sheet placed on top of the container. Care was taken to standardize the etching process according to established etching procedures [7]. Specimens were etched one at a

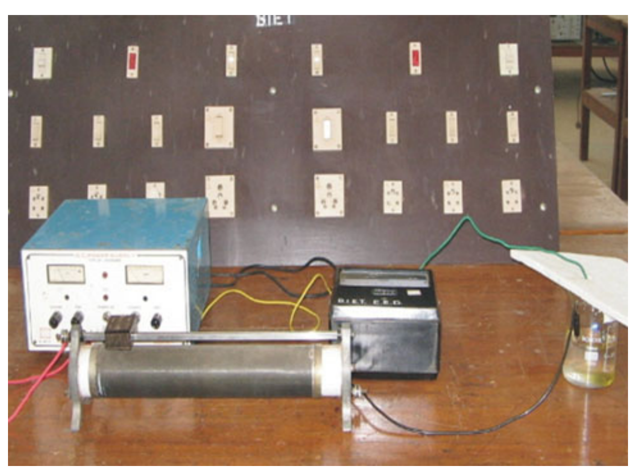


Fig. 3 Equipment used for electro chemical etching

Fig. 4 Chemical etching of alloy sample in aqua regia solution

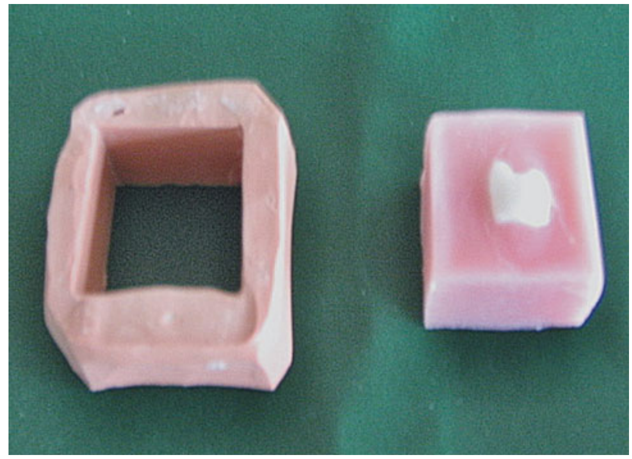


Fig. 5 Putty mould to fabricate resin block, and resin block with tooth embedded in it

time. Solution was changed after every 5 etchings as the solution tends to change the color to a slight yellow hue. The specimens were etched in 250 ml solution for 6 min at a current density of 350 mA/cm^2, at an absolute density of 70 mA (current density surface area). The surface area in this case was calculated as 0.2 cm^2. Electrode was then carefully removed from the unit avoiding contact with acid. Specimens were washed under running tap water and cleaned ultrasonically (USG 4000, Ultraschall-Dentaurum) in 18 % hydrochloric acid for 10 min. They were removed and washed in water for 2 min. Samples were stored in plastic packets at room temperature with 100 % relative humidity till they were bonded to the teeth.

Chemical Etching

Chemical etching was done using industrial grade solution of Aqua-regia, which is a combination of concentrated nitric acid (Prime Laboratories, C.A.S No: 7697-37-2) and concentrated hydrochloric acid (Prime Laboratories, C.A.S No: 7647-01-0) in the ratio of 3:1. Solution was handled very carefully under special instructions from Department of Chemical engineering. Solution was placed in a glass beaker and specimens were suspended from the center of beaker taking support from a plastic rod placed at the top (Fig. 4). Etching was done for 6 min, one specimen at a time. Solution was changed regularly at the slightest indication of change in color to a greenish hue. The etched specimen was taken out carefully and washed under tap water first, followed by ultrasonic cleansing in 18 % hydrochloric acid for 10 min. Finally the samples were washed in water for 2 min and air-dried. Samples were stored in plastic packets at room temperature with 100 % relative humidity till they were bonded to the teeth.

Preparation of Teeth Specimens

Twenty freshly extracted human maxillary premolar teeth with no carious lesions, attrition or enamel hypoplasia (cleaned and disinfected in 2 % glutaraldehyde solution) were selected. The crown portion of teeth were sectioned apart from root portion near the cemento-enamel junction. A hole was made from the occlusal surface of tooth through the pulp similar to access opening in case of root canal treatment. This hole helped in retention of the tooth after it was embedded in a resin block. A 222 cm block of plaster was prepared. Putty was adapted around it such that a hollow cube was formed as shown in the image (Fig. 5). Self cure resin (DENTSPLY) was added by sprinkle on method till the brim. The tooth was immersed in it such that either the mesial or distal surfaces show onto the top of the block. Resin was allowed to set in warm water. The surface of tooth that was exposed was flattened and an area of 45 mm was marked on all the specimens to confirm to the same amount of area that was present on the etched surface of castings. Two horizontal grooves were placed about 2 mm deep on two sides of the resin block which was to serve as retentive grooves for the universal tensile testing machine.

Etching of Teeth

Surface of tooth beyond the area to be etched was blocked with wax. The enamel portion of teeth that was marked was etched using 37 % phosphoric acid gel (Uni-etch, 3 M ESPE-USA) present in the Scotch bond kit (3 M ESPE-USA) for 15 s, washed and air-dried. Wax was removed and specimens were ready to be used.

Bonding of Specimens

Before bonding the etched alloy specimens to teeth, a wedge was fabricated in order to help in positioning the bonding of specimens without slippage. Enamel and metal interface was checked for close approximation to remove the error of irregular cement thickness. Specimens which were not in close approximation were discarded and replaced with new ones. Surface of enamel was conditioned using Scotch-bond kit (3 M ESPE, USA). Manufacturer's instructions were followed carefully to achieve best results. A silane coupling agent (3 M ESPE, USA) was applied to the metal surface as per manufacturer's instructions to improve bond between metal and resin. Since it was applied to both chemically etched and electrochemically etched samples, the chance of error was reduced. The resin cement used for the study was RELY-X ARC (3 M ESPE, USA) which is a dual cured resin. Resin cement was dispensed using the dispensing gun and mixed on a mixing pad. It was applied first on the etched tooth followed by carefully placing the etched surface of the alloy sample on to the tooth. A static load of 1 kg was applied on the sample by creating a notch on the metal weight at the centre. The load was applied for 5 min. Excess cement was removed and the specimens were light cured. Specimens (Fig. 6) were carefully stored in a box filled with cotton gauze in between the samples to prevent breaking of specimens before testing them under Hounsfeld universal testing machine.

Testing of Bond Strength

Specimens were tested for tensile bond strength by fixing to the Hounsfeld universal testing machine (Fig. 7). The grooves on the resin block were used to hold the specimen tightly while the hook portion of specimen was attached to the hook of the machine. The cross head speed used was 0.5 mm/min. Readings were noted separately for both types of specimens. Since the readings were electronically recorded, the sensitivity of the readings was in the order of grams. Readings were recorded when the specimens debond completely, with the alloy separating from the tooth. The mode of de-bonding (whether it occurred at the metal-resin interface or resin–enamel interface) was also noted.

Results

The results obtained were subjected to five kinds of statistical tests to compare the two etching techniques. Analysis of tensile bond strength was done by Anova test, Tukey's test and Dunkan's multiple range test. Fischer exact test and χ^2 test were used to analyse mode of bond failure Graphs 1, 2. A master chart was prepared showing the tensile bond failures of two groups of specimens. The electrochemical group was designated as group A, and the chemical group was designated as group B. The mean failure load was calculated as 5.95 kg for group A and that of group B as 11.15 kg (Tables 1, 2). The standard deviation of the force required to debond the specimens (Kgf) was calculated and found to be 0.65 for group A and 1.11 for group B (Table 3). Mode of bond failure was also tabulated (Table 4).

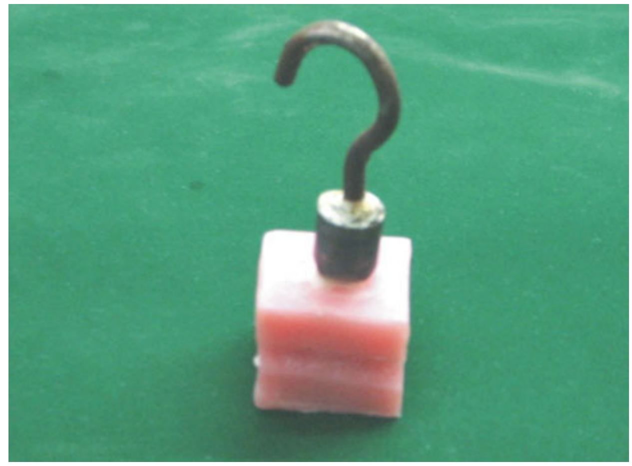


Fig. 6 Sample with alloy bonded to tooth in the resin block

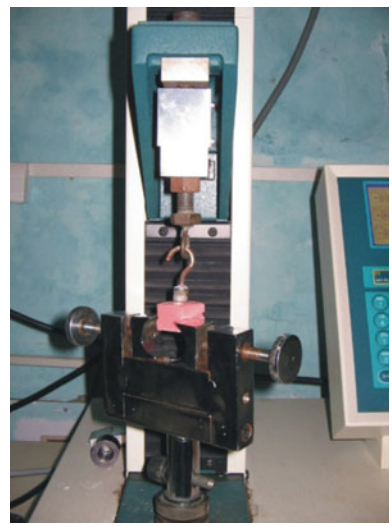
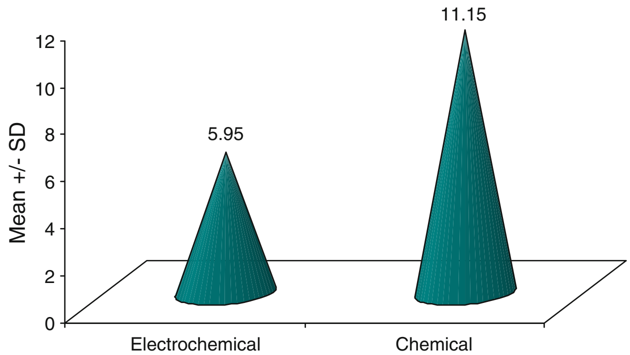


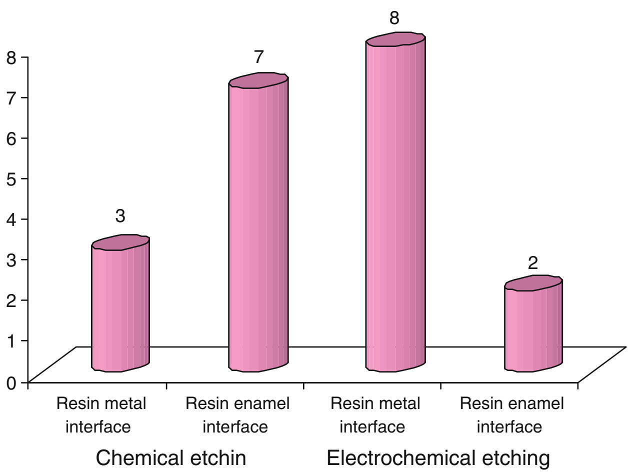
Fig. 7 Sample being tested in Universal testing machine

Discussion

Method of etching to be followed for better bonding between metal-resin-tooth interface has been a much



Graph 1 Showing mean debond value for group A and group B, as well as standard deviation for the two groups



Graph 2 Showing mode of bond failure

Table 1 Showing load at debond for electrochemically etched samples

Sample No.	Load at debond (Kgf)
1	5.91
2	6.7
3	5.83
4	6.61
5	5.67
6	4.77
7	5.81
8	5.91
9	5.37
10	6.9

Table 2 Showing load at debond for chemically etched samples

Sample No.	Load at debond (Kgf)
1	10.21
2	12.27
3	11.71
4	9.31
5	10.57
6	10.93
7	11.67
8	11.07
9	10.5
10	13.21

Table 3 Showing mean debond value for group A and group B, as well as standard deviation for the two groups

Groups	No. of observations	Mean ± SD
Electro chemical	10	5.95 ± 0.65
Chemical	10	11.15 ± 1.11

Table 4 Showing mode of bond failure

Chemical etching		Electrochemical etching	
Resin metal interface	Resin enamel interface	Resin metal interface	Resin enamel interface
3	7	8	2

debated topic amongst prosthodontist. Kuyinu and Levine [5] quoted electrochemical etching procedure created better retentive strengths than chemical method. Livaditis [1, 11] and Shillingburg et al. [14] have quoted chemical etching as better than electrochemical etching. This study was performed to check the facts and to establish within the limits of available resources as to which method of etching is better, and the nature of debonding in the two methods. The variable factors like bonding agent, etchant, film thickness of luting cement, luting cement and most importantly, mechanical considerations of tooth preparation which determine the outcome of the study have been standardized to limit the defects that may creep into the study. Studying the nature of debonding helps us indirectly to determine which method of etching creates rougher surface.

Materials were selected according to availability without compromising in the quality of materials. Results of the study were subjected to statistical analysis. It was found that the mean bond strength of samples etched chemically was almost double that of samples etched electro-chemically. Values were self explanatory and showed high clinical significance. The mode of debonding was also subjected to statistical analysis. It was found that most of the failures occurred at the resin–metal interface in case of electro-chemical etching and at resin-enamel interface in case of chemical etching. These findings were in accordance with previous studies [12]. Electro chemical etching is a procedure that requires meticulous care in the preparation of electrolytic solution. Care also needs to be taken to record etching time. There are well established procedures in the literature that explain the criteria for a successful etching procedure using electrochemical etching technique [9]. Comparative studies done to check the efficacy of various techniques [15] have conclusively proved that etched cast restorations provide better retention. Comparative studies of the two etching techniques have not been able to prove conclusively as to which method is better [5, 11, 14].

The results of the current study were in absolute harmony with almost similar studies conducted by previous researchers [11, 14]. Chemical etching produced better retentive features than electrochemical etching. Various factors can be cited in support of the outcome. Electrochemical etching was found to produce a layer of metal coating which tends to obstruct the penetration of resin into the etched surface thereby decreasing the bond strength. On the other hand electron microscopic studies [16] have shown that electrolytic etching produces lesser depth of etched surface than chemical etching. These factors combined with more etching time in case of chemical etching would contribute to better bond strength achieved by chemical etching. It was concluded in previous studies that increase in etching time in the electrochemical method produced more retention [6]. Hence it may have been possible to get a value of bond strength through electrochemical etching comparable to that of chemical etching if the time of etching was increased. But both the procedures were performed according to well framed rules and specifications by previous studies [9, 10]. Previous studies which concluded opposite to that of this study may have come to such a conclusion because the composition of the chemical etchant what they used was not specified and also the exact procedure in which they did the electrochemical etching was not described [2]. Thus it is debatable whether those values secured in previous studies can really oppose the values secured in the present study. Since this is an in vitro study the specimens were tested immediately after fabrication and the conditions do not exactly coincide with those seen in a clinical situation [17–19]. It can be safely said that chemical etching is better than electrochemical etching when one particular methodology is followed which may not be in harmony with results that could be obtained when another method is followed. Aqua regia solution has been used instead of a gel in the study. Electron microscopic study was not done to perceive the depth of etching in the two methods [16]. A measure of depth of etching may have further supported the results of study.

Conclusion

The study enumerates various etching techniques that are prevalent among clinicians and lab technicians while etching base metal alloys. Two main techniques utilized in the study are chemical etching and electro chemical etching. Within the limitations of the study it was established conclusively that:

1. Chemical etching of the samples created better retentive surfaces than electro-chemical etching.
2. The results of mode of de-bonding show that in case of chemical etching maximum debonding occurred at resin-enamel interface and in electrochemical type it occurred at resin-metal interface.

The above conclusions will make the process of providing quality etched cast restorations by the dentists more predictable.

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