ISSN NO. 2320-5407

*Journal homepage: http://www.journalijar.com*

*INTERNATIONAL JOURNAL OF ADVANCED RESEARCH*

**RESEARCH ARTICLE**

# Effect of 37% phosphoric acid on the in-vitro shear strength between nickel chromium alloys and luting cements-An SEM Study

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## *Manuscript Info*

***Manuscript History:***

Received: 22 March 2014
Final Accepted: 25 April 2014
Published Online: May 2014

***Key words:***
phosphoric acid, nickel chromium alloys, luting cements

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## *Abstract*

**Background**: The failures at the bonding interface between the luting cement and the crown/bridge prosthesis is a major clinical problem. To address this problem, numerous surface treatments have been attempted.
**Objective**: To explore the possibility of increasing the shear bond strength of a sandblasted nickel-chromium base metal alloy with four luting agents after surface treatment with 37 % phosphoric acid.
**Design**: Wiron alloy was cast in cylindrical forms (10mm length x 8mm diameter). The base of each cylinder was surface treated (sand blasting, group 1 OR sandblasting and acid etching, group 2) and the two cylinders were joined with the help of a luting cement (Zinc phosphate/Zinc poly carboxylate/GIC type1/Resin cement) to form a bonded assembly. The INSTRON Universal Testing Machinewas used to test the shear bond strength between the two units of the bonded assembly. The surface topographic structure of the metal sample used for the formation of the bonded assembly were seen under the scanning electron microscope.
**Statistical Evaluation**: The data were analyzed using Kruskal Wallis test to establish whether there was a significant difference among the two groups. The significance was set at a P value <0.05.
**Results**: The values for all the four luting agents in both the groups were statistically non-significant.
Conclusion: 37% phosphoric acid cannot be recommended as a surface treatment for nickel chromium base metal alloys.



## INTRODUCTION:

The metal ceramic fixed dental prosthesis offers an economical esthetic option for the patient .But, the increased tooth cutting required to accommodate the ceramic and the metal can many times result in minimally retentive preparations. The phrase "minimally retentive" is used to define a tooth preparation with excessive axial convergence and short axial walls (Browning WD et al, 2002[1]). In these preparations, the mechanical retentive aspects are decreased and the retentive characteristics of the luting cements become important aspects for the crown/bridge retention (Amara Abreu et al, 2007[2])

The failures at the bonding interface between the luting cement and the crown/bridge prosthesis is a major clinical problem. To address this problem, numerous surface treatments have been attempted.

When returned from the laboratory ,the intaglio surface of a metal ceramic prosthesis has formed a surface oxide layer as a result of the elevated temperatures required for porcelain bonding to the external oxide surface. These surface oxides provide potential locations for chemical bonding and also serve to roughen the metal surface. The inner surface can be left in this state and then directly cemented **(**Amara Abreu et al, 2007[2]**).**

In addition, the surface can also be airborne-particle abraded (sandblasted) to provide for a clean and roughened surface prior to cementation. Sandblasting is routinely done by the labs before delivery of the crown or bridge to the clinician. Sand blasting is used for increasing the effective surface area for enhancing the mechanical retention strengths of the luting cements[3]. Sandblasting improves the retention between the metal and cement by cleaning oxides or any greasy materials from metal surfaces, creating very fine roughness enhancing mechanical and chemical bonding between the cements and metals.(Schneider, Powers & Pierpoint, 1992[4])**.**

Other surface treatments used for base metal alloys can be metal primers like MDP (10-methacryloyloxydecyl dihydrogen phosphate) and hydrofluoric acid.

But, MDP monomer is not routinely available in practice and hydrofluoric acid is a strong caustic agent that may create irritations of soft tissues and intraoral use of this acid gel should be seriously questioned (MacKinnon MA,1988[5])**.**

37% phosphoric acid is commonly available in all clinics as it is used for acid etching the enamel during composite restorations. It was hypothesized that, if the acid causes even minor roughness of the metal surface of the sandblasted crown/bridge, then it can potentially further increase the bonding strengths between the luting cement and the crown. So, this study was done to explore the possibility of increasing the shear bond strength of a sandblasted nickel-chromium base metal alloy with four luting agents after surface treatment with 37 % phosphoric acid. The effect of 37% phosphoric acid on the etching pattern of the sandblasted nickel chromium based base metal alloy was also examined under a scanning electron microscope.

## MATERIALS AND METHODS:

The materials used for the study are:

| TYPE | BRAND NAME | MANUFACTURER |
|---|---|---|
| Base metal alloy | Wiron. 99 Alloy | BEGO-WILCOS BRASIL |
| Zinc phosphate cement | De Trey Zinc Crown and Bridge Fixodont Plus | DENSPLY |
| Zinc poly carboxylate cement | GC LIVCARBO | GC CORPORATION |
| Glass Ionomer type I | GC Gold Label Luting & Lining Cement | GC CORPORATION |
| Resin cement | Rely X U 100 | 3M ESPE |
| Etchant | Scotchbond Multipurpose | 3M ESPE |

**Specimen preparation:**

Wiron alloy was cast in cylindrical forms (10mm length x 8mm diameter). The base of each cylinder was surface treated and the two specimens were joined with the help of a luting cement to form a bonded assembly. (Fig 1) The INSTRON Universal Testing Machine was used to test the shear bond strength between the two units of the bonded assembly.

The samples made were divided into two groups with twelve bonded assemblies in each group:

**Group 1:**

The surface treatment done before bonding with the luting cement was sand blasting. Casted metal pellets were sand blasted in sandblasting machine (Mine Technology Private Limited) with alumina particles (particles size 110µm).

**Group 2:**

The surface treatments done before bonding with the luting cement,were sandblasting and acid etching with 37% phosphoric acid.Casted metal pellets were sand blasted and then etched for 2 min with phosphoric acid.

Each group was divided into four sub groups A, B, C, and D.

- Sub group A: Bonded assemblies were made using zinc phosphate cement.
- Sub group B: Bonded assemblies were made using zinc poly carboxylate cement.
- Sub group C: Bonded assemblies were made using Glass Ionomer Type I luting cement.
- Sub group D: Bonded assemblies were made using self adhesive universal resin cement.

Cast metal cylinders were bonded with the respective luting cements.The cement was mixed as specified by manufacturer. Mixed cement was applied over the surface of the metal pellet held in a cementing zig (Fig 2) (Manufactured at Hebich Engineering, Mangalore).A load of 500g was applied over it to obtain uniform film thickness.

In the case of ***Rely X U 100,*** following the 500g load the cement was allowed to self cure for 2-3 min. After this, the material becomes like a gel and excess was removed. Then the material was light cured for 20 sec from all the surfaces using a 3M LED light cure unit.

The excess cement was then removed from the surfaces of the joints after 10 min.Two layers of a dental varnish (GC Fuji varnish, G-C Dental Industrial Corp., Tokyo, Japan) were applied around the joints of all the samples cemented with any of the four luting cements.. Samples are stored in humidifier in a beaker with 100ml water at 37 $^{0}$C and 50% relative humidity for 24 hours.

**Shear test:**

The shear tests were conducted on an Instron Universal Testing Machine (Instron Model 4206, Germany). The cross-head speed was set at 2.1 x $10^{-6}$ m/s. The samples were held in a shear device (Manufactured at Hebich Engineering, Mangalore) (Fig 3) designed to apply a pure shear force to the joints.

**Scanning Electron Microscope (SEM) study:**

To see the surface topographic structure of the metal sample used for the formation of bonded assembly, surface characteristics of a casted metal without any treatment (Fig 4), with sandblasting(Fig 5) and sandblasting with acid etching (Fig 6) was seen under the Scanning Electron Microscope(JOEL Scanning Electron Microscope).Samples were kept in vaccum chamber of scanning electron microscope and vaccum was created. After that electrons were allowed to scan the surface and image was reflected in the monitor. Magnification used was 500X.

**Statistical Evaluation:**

The data were analyzed using Kruskal Wallis test establish whether there was a significant difference among the two groups. The significance was set at a P value <0.05.

## RESULTS:

The mean and standard deviation values obtained are shown in Table 1(A, B, C, D).The values for all the four luting agents in both the groups were statistically non-significant.

**TABLE 1: Mean and standard deviation values**

***A: Zinc phosphate cement***

| | Mean | Standard deviation |
|---|---|---|
| Sand blasting | 3.6367 | 1.09436 |
| Acid etching + sand blasting | 3.8533 | 0.36556 |

***B: Zinc polycarboxylate cement***

| | Mean | Standard deviation |
|---|---|---|
| Sand blasting | 4.1167 | 0.41861 |
| Acid etching + sand blasting | 4.39 | 0.6844 |

***C: Glass Ionomer luting cement***

| | Mean | Standard deviation |
|---|---|---|
| Sand blasting | 6.0433 | 0.41041 |

| | | |
|---|---|---|
| Acid etching + sand blasting | 5.97 | 0.73137 |

**D:** ***Self adhesive universal resin cement***

| | Mean | Standard deviation |
|---|---|---|
| Sand blasting | 9.7467 | 1.46466 |
| Acid etching + sand blasting | 9.6133 | 0.89473 |

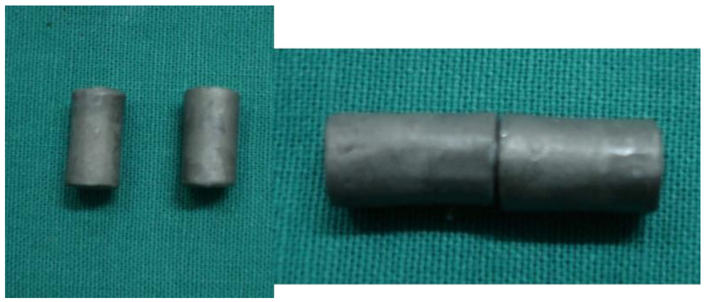

**Fig 1: The cast cylindrical alloys and the bonded assembly.**

**Fig 2: Cementing zig.**

Fig 3: Shear device placed in the Instron testing machine

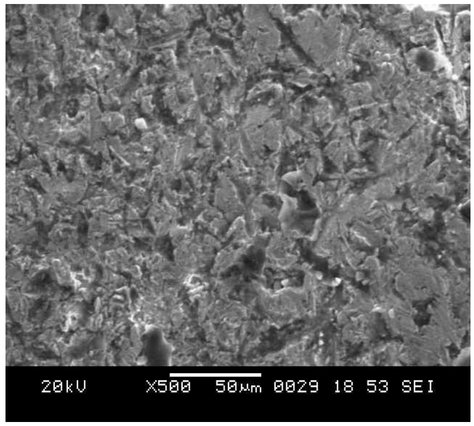


**Fig 4: SEM picture of an untreated sample.**

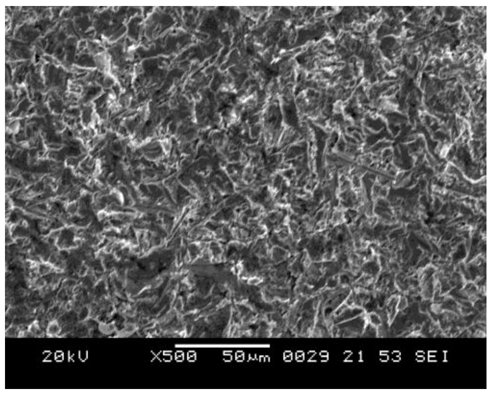


**Fig 5: SEM picture of a sand blasted sample.**

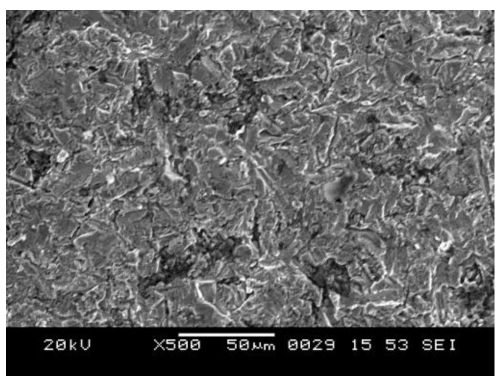


**Fig 6: SEM picture after sandblasting and acid etching**

## DISCUSSION:

This study evaluated the effect of 37 % phosphoric acid on the shear bond strength of a nickel-chromium based base metal alloy with four luting agents.

A shear stress or a tensile stress applied to a butt joint is usually the most common bond tests used in dental research. A pure tensile stress is difficult to achieve. A very small deviation in the direction of the load can concentrate stress in a particular area of the joint and lower the resistance to fracture. Moreover, even in a perfect experimental setting, the stress may not be distributed evenly along the interface. As an alternative to tensile testing, the shear bond test is probably more representative of the stress applied on dental restorations. (LeesWA,1985[6]; Van Noort R,1989[7])

In this study the samples which were acid etched following sandblasting showed no major increase in the mean values compared to the samples which were only sandblasted. This can be explained on the mild nature of the acid and is adequate only for etching the tooth and not a metal surface.

The SEM analysis also supported the result obtained wherein, the untreated samples showed only small porosities which would have occurred during casting (Fig 4).The sandblasted samples showed definite surface roughness and irregularities which would have enhanced micro mechanical retention (Fig 5). The sandblasted sample treated with 37% phosphoric acid showed very small areas of corrosion which would not be adequate in enhancing bond strengths (Fig 6).

## CONCLUSION:

Under the conditions of the study:

37% phosphoric acid cannot be recommended as a surface treatment for nickel chromium base metal alloys due to their mild nature. Higher concentration of phosphoric acidor other promising avenues like metal primers or should be considered to increase the metal cement bond strength.

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