The Effect of Disinfectant Solutions on the Durability of the Bond between Resin Based Cement and Non-precious Metal Alloy

(Pesan Disinfektan Larutan Mengenai Ketahanan daripada Ikatan antara Resin Berasaskan Simen dan Aloi Logam Tidak Berharga)

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ABSTRACT

This study evaluated the effect of disinfectants on the tensile bond strength of Nickel-Chromium alloy bonded with resin cement. 180 pairs of Nickel-Chromium dumbbells were prepared. The dumbbells were divided into 3 groups (n=60), which received one of the following treatments: Sandblasted only (control), sandblasted and Perform®-ID or sandblasted and sodium hypochlorite (SH) before bonding with resin cement. All bonded specimens were stored in distilled water for 24 h and half of the specimens were subsequently thermocycled (500 cycles) before debonding. Tensile bond strength was recorded and each dumbbell was examined for failure mode. Two-way ANOVA analysis indicated that overall there was a statistically significant difference between 24 h and thermocycling test, but no differences between sandblasted only, sandblasted and Perform-ID or sandblasted and SH groups. Post-ANOVA contrasts indicated that only the sandblasted and SH group showed a significant difference between the 24 h and thermocycling test. Disinfectants did not significantly decrease tensile bond strength between Nickel-Chromium dumbbells bonded with resin cement.

Keywords: Adhesion; alloys; cements; disinfectants

INTRODUCTION

Mounting concerns regarding the spread of infections in a dental environment has led to increased focus on infection control procedures both in the clinic and laboratory. These procedures are designed to prevent or reduce potential transmission of infections from patient to dental health care providers (DHCP), from DHCP to patient and from patient to patient.

Dental disinfectants play a vital role in the infection control procedures essentially by destroying majority of pathogenic bacteria, thus reducing the microbial load. Its efficacy, depending on the type of disinfectant used and time of exposure, ranges from complete sterility on one end of the spectrum to reduction in microbial load at the other end (ADA Council on Scientific Affairs and ADA Council on Dental Practice 1996; Kohn et al. 2003).

A literature search has shown laboratory and clinical studies which evaluated the efficacy of various disinfectants including Perform®-ID and sodium hypochlorite (Ahmad et al. 2007; Rentzia et al 2011). In addition, dental disinfectants have also been tested for their effect on dental materials, among others, the dimensional stability and surface reproduction of impression materials (Ahmad et al. 2007; Langenwalter et al. 1990). However, to date, no research has been published on the effect of dental disinfectants on the bond strength of dental luting cement, particularly resin-based dental cement.

One of the available chemical disinfectants used in dentistry is Perform®-ID (Schülke & Mayr GmbH, Germany). The active ingredients in Perform®-ID are Pentapotassium-bis(peroxymonosulphate)-bis(sulphate), sodium benzoate and tartaric acid. The manufacturers
recommend Perform®-ID used in an immersion bath in a concentration of 2% w/v in water for 10 min to eliminate microorganisms including *Staphylococcus aureus*, *Pseudomonas aeruginosa*, and *Candida albicans*.

Another disinfectant, considered as one of the ‘traditional’ disinfectants is sodium hypochlorite. Sodium hypochlorite is a type of halogen-releasing agent and has been used on hard surfaces and reported to disinfect spillages of blood containing human immunodeficiency virus (HIV) or hepatitis B virus (HBV) effectively (McDonnell & Russell 1999). This type of disinfectant is commonly used as household bleach.

According to Pavarina et al. (2003) 1% sodium hypochlorite solution was effective in reducing microbial load including spores after 10 min of immersion. McGowan et al. (1988) tested different concentrations of sodium hypochlorite for various immersion periods and their effect on Cobalt-Chromium and Nickel-Chromium denture base materials. The result showed that 2% sodium hypochlorite for at least 5 min or 5.25% for a minimum 3 min effectively removed microorganisms and did not tarnish or corrode the base metal alloys.

There are many *in vitro* studies which have looked at the bond strength of resin based cements to dental casting alloys after various surface treatments with average measurements ranging from 2.8 to 21 MPa (Abreu et al. 2009; Denizoglu et al. 2009; Fonseca et al. 2009). Air abrading the surface of the metal substrate is a widely used surface treatment which removes the oxide layer formed during casting and therefore providing micromechanical retention between resin based cement and metal substrate (Fonseca et al. 2009). Similarly, Fonseca et al. (2009) also described physicochemical bonding achieved by the presence of functional monomers found in metal primers and resin based materials, particularly resin based luting cements. The oxide layer formed on the surface of the cast metal substrate exists in a passive state and Tanaka et al. (1981) suggested that metal primers have the capability of adhering to the oxide layer better than the metal surface itself, therefore providing potential for eliminating the need for mechanical retention. These metal primers contain active monomers, which are involved in the forming of a bond between the oxide present on the metal substrate surface and the resin cement. Studies have shown that metal primers containing 4-META (4-methacryloyloxyethyl trimellitate anhydride), MDP (10-methacryloyloxydecryl dihydrogen phosphate), or MEPS (thiophosphate methacryloyloxyalkyl) derivatives monomers have been shown to increase bond strength between various dental casting alloys and resin based adhesive materials (Matsumura et al 1996; Tanaka et al 1981; Yoshida et al 1997).

Although there were *in vivo* and *in vitro* studies involving resin based adhesive cements (Abreu et al 2009; Denizoglu et al 2009; Fonseca et al 2009), no study has investigated the effect of disinfectants on the bond strength of these cements to resin bonded bridges. Therefore, the aim of this *in vitro* study was to evaluate the effect of disinfectant on tensile bond strength between resin based adhesive cement and non-precious (Nickel-Chromium) metal alloy.

The first null hypothesis for the present study was that there is no difference in tensile bond strength between aluminium oxide (Al₂O₃) blasted Nickel-Chromium dumbbells bonded with resin based adhesive cement in control group and those immersed in disinfectants prior to bonding. The second hypothesis was that thermocycling does not have any effect on the tensile bond strength between aluminium oxide (Al₂O₃) blasted dumbbells bonded with resin based adhesive cement in control group and those immersed in disinfectants prior to bonding.

**MATERIALS AND METHODS**

**PRODUCTION OF WAX PATTERN OF DUMBBELLS**

An acrylic rod measuring 5 mm in diameter was machined on a lathe to produce one uniform dumbbell shaped acrylic pattern. A laboratory silicone matrix was made of this acrylic pattern and the resulting mould was used to produce wax patterns of dumbbells. Molten modelling wax was poured into the mould and allowed to cool to produce a total of 360 wax patterns of dumbbells (Figure 1).

![Diagram showing average measurements of an individual dumbbell](image)
PRODUCTION OF CAST DUMBBELLS

Eight of the wax patterns were sprued to an investment ring which was then treated with surfactant (Tensilab Zhermack®, Italy) to reduce the irregularities on the surface of the cast metal. Subsequently, the investment ring was invested in phosphate bonded investment material (Bego, Germany). All the invested wax patterns were heated in a Carbolite CWF 1100 furnace (Carbolite Limited, London, United Kingdom) and the specimens were cast in the Heracast IQ gravity pressure casting machine (Heraeus Kulzer Ltd, United Kingdom) using Nickel-Chromium base metal alloy ingots (Heraenium NA®, Germany). All castings were subsequently devested and sandblasted with coarse aluminium oxide (Al₂O₃) particles to remove the residual investment material. The castings were separated from the sprue using carborundum discs (Bracon Limited, United Kingdom).

PRODUCTION OF TEST SPECIMENS

The test surfaces of the dumbbells were first treated with 50 micron alumina oxide particles (Bracon Limited, United Kingdom) using the Modulars SandBlaster (Silfradent®, Italy) for 30 s at a pressure of 0.03 MPa and at a distance of 10 mm in a circular motion. The 360 dumbbells were randomly divided into 6 groups of 60 dumbbells each. Specimens from 3 groups were used to test the tensile bond strength after 24 h whilst the other 3 groups were subjected to thermocycling prior to tensile testing. In the 24 h test, 60 dumbbells in each group were immersed for 10 min in 2% Perform®-ID, (Schülke & Mayr GmbH, Germany) according to manufacturer’s recommendation or, 2% sodium hypochlorite after which a continuous flow of distilled water for 30 s was used to remove the remnants of the disinfectant on the testing surface of each dumbbell.

Once dried, the dumbbells were cemented in pairs (n=30) with dual cured resin based adhesive cement, Panavia F 2.0-Opaque, (Kuraray Medical Inc, Japan). A metal primer, alloy primer (Kuraray Medical Inc, Japan) was applied on the test surface of each dumbbell prior to application of the resin based adhesive cement. During the bonding procedure, a custom made jig was used to ensure proper alignment of the dumbbell (International Standardisation Organisation 2000) and finger pressure applied for a minimum of 3 min so that the specimens would be maintained together. This was to mimic the clinical scenario during cementation of resin bonded bridges intraorally.

Excess cement was removed prior to setting and a layer of Oxyguard (Kuraray Co. Ltd., Japan) was applied at the metal-cement junction to provide an anaerobic environment to allow complete polymerization and setting of the resin cement. All bonding procedures were performed by one operator. The dumbbells, which were sandblasted but not treated with any disinfectant, were labelled as the control group.

One hundred and eighty pairs of the cemented dumbbells were stored at 37°C (RH: 40%) in the incubator (Sanyo Electric Company Ltd, Japan) for 24 h to allow complete polymerization of the resin cement. Half of the cemented dumbbells (90 pairs) were subjected to 500 thermal cycles between 5 and 55°C in a thermocycling machine (Julabo Labortechnik GmbH, Germany).

SPECIMEN TESTING

All the bonded dumbbells were tested for tensile bond strength with the Instron Universal Testing Machine (Instron Limited, High Wycombe, United Kingdom). A tensile load was applied at a crosshead speed of 0.5 mm per min. One cell was used, capable of detecting load up to 1000 N. The specimens were loaded in tension until separation of the dumbbells occurred and the resulting tensile bond strength in MPa was submitted for statistical analysis.

SURFACE ANALYSIS OF SEPARATED TEST SPECIMENS

Each pair of de-bonded dumbbell surfaces was analysed using the Meiji VM 1000 Binocular Dissecting Microscope (Meiji Techno Company, Ltd. Japan) at 30x magnification to determine the mode of failure.

Three types of failure modes were established: Cohesive failure within resin cement, adhesive failure between metal and resin cement, a mixed failure showing a combination of both modes.

SCANNING ELECTRON MICROSCOPY OF SEPARATED TEST SPECIMENS

A pair of dumbbells from the control group and one, which had not been subjected to any sandblasting was randomly selected for surface topography analysis under the scanning electron microscope. The specimens were viewed at 15, 30, 100, 250 and 500x magnifications at an accelerating voltage of 15 Kv using the Hitachi S-3500N Scanning Electron Microscope (Hitachi High-Technologies Corporation, United Kingdom).

A pair of dumbbells, which have undergone cohesive and combination failure were also randomly selected for surface topography analysis under similar standardized magnifications as described previously.

DATA ANALYSIS

Data collected was analysed using the statistical programme STATA 11 (Statcorp LP, USA). A two-way analysis of variance (two-way ANOVA) was performed on the data and post-ANOVA contrasts were conducted using a Scheffe test. The frequency of each mode of failure for all three disinfectant groups (sandblasted only, sandblasted and Perform®-ID, sandblasted and sodium hypochlorite) by each test condition (24 hours and thermocycling) was analysed using a chi-square (X²) test. For all statistical analysis, the significance level was set at p<0.05.
RESULTS

TENSILE BOND STRENGTH

Table 1 presents the results of disinfectant (sandblasted (SB) only (control), SB and Perform®-ID, SB and sodium hypochlorite) and test condition (24 h and thermocycled) on the tensile bond strength of resin based adhesive cement.

The median tensile bond strength of the SB and disinfected dumbbells were greater than the SB only dumbbells, but the differences were not significant. In the SB only group, which was the control group, the median tensile stress values recorded for 24 h test (0 thermal cycles) and thermocycling test were 29.3 and 18.7 MPa, respectively. Dumbbell specimens sandblasted and immersed in Perform®-ID demonstrated a decrease in tensile bond strength between the 24 h (0 thermal cycles) test (31.2 MPa) and thermocycling test (20.9 MPa). A similar trend was exhibited by specimens, which were sandblasted and immersed in sodium hypochlorite whereby the median tensile stress for samples in 24 h test was 36.5 MPa and specimens in the thermocycling test recorded a reduction of 12.6 MPa.

Two-way ANOVA analysis indicated that overall there was a statistically significant difference between the 24 h and thermocycling test (p=0.012), but no differences between SB only (control), SB and Perform®-ID or SB and sodium hypochlorite groups (p=0.272). In addition, there was no interaction between the factors in the two-way analysis (p=0.247). Post-ANOVA contrasts based on this finding indicated that only the SB and sodium hypochlorite group showed a significant difference between the 24 h (0 thermal cycles) and thermocycling test (p=0.012).

TABLE 1. Median (inter-quartile range) of tensile bond strength of resin based adhesive cement for disinfectant (SB only (control), SB and Perform®-ID, SB and sodium hypochlorite) by test condition (24 h and thermocycled)

<table>
<thead>
<tr>
<th>GROUP</th>
<th>TEST CONDITION</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>24 h (0 thermal cycles) (MPa)</td>
</tr>
<tr>
<td>Sandblasted (SB) only</td>
<td>29.3 (16.1-41.7)</td>
</tr>
<tr>
<td>SB and Perform®-ID</td>
<td>31.2 (10.5-38.5)</td>
</tr>
<tr>
<td>SB and sodium hypochlorite</td>
<td>36.5 (26.4-42.2)</td>
</tr>
</tbody>
</table>

TABLE 2. Frequency of each mode of failure for all three disinfectant groups (SB only, SB and Perform®-ID, SB and sodium hypochlorite) at 24 h and after 500 thermal cycles

<table>
<thead>
<tr>
<th>GROUPS</th>
<th>24 h test Mode of failure</th>
<th>Thermocycling test Mode of failure</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Adhesive</td>
<td>Cohesive</td>
</tr>
<tr>
<td>Sandblasted (SB) only</td>
<td>0</td>
<td>2</td>
</tr>
<tr>
<td>SB and Perform®-ID</td>
<td>0</td>
<td>6</td>
</tr>
<tr>
<td>SB and sodium hypochlorite</td>
<td>0</td>
<td>5</td>
</tr>
</tbody>
</table>

FAILURE MODE ANALYSIS

For both the 24 h and thermocycling test, all three disinfectant groups recorded only cohesive and combination failure. No adhesive failure at metal and resin cement interface was recorded. In the 24 h test, the control group recorded the highest combination failure at 28 pairs followed by SB and sodium hypochlorite as well as SB and Perform®-ID groups at 25 and 24 dumbbell pairs, respectively. Chi-square (X²) analysis showed no significant difference in the mode of failure between the disinfectant groups (p=0.413).

For the dumbbells which were thermocycled, 27 of the 30 pairs in the control and SB and Perform®-ID group had combination type. Eight of the dumbbell pairs in the SB and sodium hypochlorite group exhibited cohesive failure within the resin cement. X² analysis did not show any difference between the two modes of failure for all the three types of disinfectant groups (p=0.154).

SCANNING ELECTRON MICROSCOPY

In Figure 2(iv) which exhibits combination failure, ‘A’ represents an area which has undergone adhesive failure, exposing the Nickel-Chromium alloy. Adjacent to ‘A’, is remnants of the resin based adhesive cement, indicating cohesive failure and represented by ‘B’.

DISCUSSION

After 24 h of water storage, the results showed there was no significant differences with regards to the tensile bond strength. There was also no difference in the type of failure with the majority of the dumbbells exhibiting a mixed of cohesive and adhesive failures (combination type failure).
as shown in Figure 2(d). These findings indicate that there were no significant differences between the three types of surface treatment subjected to the dumbbells. Dumbbells which recorded combination failure predominantly exhibited cohesive failure indicating that the weaker link was in the cement and not the bond to the oxide layer of the metal alloy. The cohesive failure experienced by some specimens suggests the resin cement may be affected by either the presence of water, probably leading to hydrolysis of the chemical bond formed, or repeated thermal stresses. In the thermocycling test group, a similar trend was observed with no significant differences in the tensile bond strength and the type of failure mode between the three disinfectant groups.

However, with regards to durability testing, further statistical analysis indicated that only the dumbbells in the sandblasted and sodium hypochlorite group showed significant reduction in the tensile bond strength between 24 h and thermocycling test conditions. These findings indicated that compared to Perform®-ID, sodium hypochlorite negatively influenced the durability of the resin based cement – Nickel-Chromium bond. However, further research is necessary to investigate the effect of disinfectants on the surface characteristics of Nickel-Chromium dumbbells.

Base metal alloys, Cobalt and Nickel-Chromium, have good corrosion resistance provided by the thin layer of chromic oxide, which protects the bulk of the alloy. This oxide layer is replenished immediately if the surface of the alloy is damaged, rendering these alloys to be permanently resistant to corrosion. In their study, McGowan et al. (1988) concluded that base metal alloys soaked in various concentrations of sodium hypochlorite for short duration (below 15 min) did not tarnish or corrode the metal alloys. However, in 1983, Sarkar et al. wrote that sodium hypochlorite selectively removes nickel from surface of Nickel-Titanium (NiTi) alloy creating micropits. Similarly, O’Hoy et al. (2003) reported significant corrosion of NiTi endodontic instruments, which were exposed to 1% sodium hypochlorite overnight. The immersion time in these studies were much longer then the 10 min employed in the present study. In the present study, the higher bond strength values in the sodium hypochlorite groups may possibly have been due to increased bonding surface and micropit retention provided by the micropits caused by selective removal of nickel from the surface of Nickel-Chromium alloy.

Perform®-ID disinfectant was chosen for this study as it is commonly used in dental clinics. The active ingredients in Perform®-ID are pentapotassium-bis (peroxymonosulphate)-bis(sulphate), which is an oxidizing agent and therefore has bacteriocidal properties and sodium benzoate, a sodium salt of benzoic acid which has antimicrobial features and prevents bacterial growth by interfering with their ability to generate energy. In this study, bond strength values for dumbbells immersed in Perform®-ID were higher than sandblasted only (control group) but lower than sandblasted and sodium hypochlorite group. It is possible that these dumbbells had higher surface reactivity compared to the control group and thus forming a better bond with the resin cement. Unpublished data by the manufacturer has reported that Perform®-ID solution was compatible with stainless steel, copper, brass and aluminium when immersed in 0.5% solution for 24
h (Gregson, personal communication, March 2011). However, this data does not provide any information on the effect of Perform®-ID on non-precious metal alloys.

Alloy primer, a type of metal primer containing both 10-methacryloxy decyl dihydrogen phosphate (MDP) and 6-(4-vinylbenzyl-n-propyl)amino-1,3,5-triazine-2,4-dithione (VBATDT), was used in the present study as a surface pretreatment and as an adjunct to sandblasting for all three groups. Alloy primer was used in the current study based on a previous study which reported that metal primers increased the bond strength of resin cement to base metal alloy (Abreu et al. 2009). This is possibly due to metal primers increasing wetting of the surface and consequently increasing contact between resin based cement and the metal alloy surface. However, the high cohesive failure (both individually and in combination) suggests that the VBATDT primer may interfere with the bonding mechanism between MDP primer and metal oxide layer leading to weaker bond strength (Fonseca et al. 2009).

In the current study, metal dumbbells were bonded to similar surfaces and the single effect of tensile bond strength between resin based cement and non-precious metal alloy was determined. Such an assembly eliminates other factors such as the bond strength between resin based cement and enamel, which may have an effect on the bond strength between resin based cement and metal alloy. However, such an assembly has the limitation of not being able to predict the clinical outcome of the bonding system.

In relation to the variation in bond strength values, all three test groups which were evaluated in this study showed high tensile bond strength at 24 h despite recording a wide variety of bond strength values in each group ranging from 15.8 to 28.0 Mpa. The large variation in bond strength values could be due to the variation in alignment of dumbbells during cementation procedure. Kohli et al. (1990) reported similar variation in tensile bond strength in their study which evaluated the effect of three surface treatments on bond strength of resin cement bonded to Nickel-Chromium-Beryllium alloy. In their study too, metal specimens were bonded to each other. The authors also described that the unequal thickness of resin cement in some specimens may have contributed to the increased stress caused by polymerization shrinkage. These stresses acting at the interface between resin cement and metal could have weakened the bond, thus resulting in variation of bond strength.

Based on the results of the present study, it appears that the use of dental disinfectants according to manufacturer’s recommendations should be continued as part of the infection control practice in dental clinics and laboratories. The current regime, which recommends 10 min of immersion in disinfectants is applicable to resin retained bridges bonded with resin based adhesive cements. Nonetheless, other factors which could possibly influence the result of this study, such as intraoral pH or fatigue loading, have not been evaluated. Therefore, it is necessary for careful interpretation of clinical implication of the results in this study.

CONCLUSION

Within the limitations of this study, it can be concluded that: There was no difference in tensile bond strength between the Nickel-Chromium dumbbells which were sandblasted only and those immersed in Perform®-ID and sodium hypochlorite after sandblasting; it appears that the tensile bond strength of Nickel-Chromium dumbbells which were sandblasted and immersed in sodium hypochlorite decreased significantly after thermocycling; and failure mode analysis did not show any significant difference between Nickel-Chromium dumbbells in all three test groups at 24 h and after 500 thermal cycles.

REFERENCES


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Received: 25 September 2013
Accepted: 9 September 2014

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Received: 25 September 2013
Accepted: 9 September 2014