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Influence of Surface Treatments on the Shear Bond Strength of Panavia 21 to a Type III Gold Alloy

Azwatee A Aziz*, Christopher J Tredwin†, Alastair Stokes‡, Daljit S Gill§ and David R Moles¶

Abstract - *Cylinders of Type III gold alloy were sandblasted with either: 50 micron Aluminium Oxide particles (Group 1); CoJet™ Sand (Group 2); the CoJet™ System - CoJet™ Sand followed by application of silane primer (Group 3). Shear testing was undertaken following apposition of Panavia 21 resin. Mean shear bond strengths (+/- standard deviation) were Group 1, 32.9 (+/-18.2) MPa; Group 2, 26.5 (+/-10.8) MPa and Group 3, 45.6 (+/- 25.8) MPa. There was no statistically difference between Group 1 and 2; and between group 1 and 3. However, there was a significant difference between Group 2 and 3 (p< 0.05). While the CoJet™ System achieved the highest mean shear bond strength it conveyed no statistical advantage over the traditional method of sandblasting with aluminium oxide.*

KEY WORDS: Gold alloy, Bonding, Sandblasting, CoJet, Panavia.

INTRODUCTION

Producing durable bonding of resin composite to metal is a desirable goal that has practical clinical application in dentistry. The potential for effective adhesion of resins to metals is not only for retaining resin-bonded retainers, but also for bonding resin veneers to metal frameworks. The resin/metal interface is at the basis of most bonding failures in resin-bonded non-precious prostheses [1,2,3] and resin-bonded precious prosthesis [4]. The factors contributing to this failure are the type of adhesive and the nature of the alloy, the treatment of the surfaces, and the stresses undergone by the prosthesis. Whatever the adhesive and treatment chosen, the best results are obtained with nickel-chrome-beryllium alloys [5,6,7]. To improve adhesion to non-precious alloys, various treatments of surfaces have been used including; macromechanical retention (drills [1]; beads [8]); micromechanical retention, selective dissolving with chemical [9]; or electrochemical [10,11,12]; etching technique and sandblasting [13].

Bonding of precious alloys such as gold is often clinically desirable. Oxidation of the surfaces of these alloys was first recommended using techniques of heating [14] or placing in oxidative liquid, and then a bond comparable to that of NiCr was obtained with a tin plating of gold alloys [15,16,17,18]. However, the electrogalvanic behaviours of the tin and gold alloys are incompatible. Today, in addition to heat treatment of gold, current practice to improve bonding to resin includes air abrasion with aluminium oxide particles, or air abrasion with silane-covered aluminium grains followed by application of a silane coupling agent. One such method of the latter is the CoJet™ System.

The CoJet™ System is reported to offer the prospect of simple enhancement of bonding resins to precious metals, for example, the manufacturer claims that pre-treatment of precious metal onlays, crowns and bridges prior to adhesive luting will increase the bond strength. Figure 1 shows a flow diagram for how the manufacturers suggest the CoJet™ System should be used. The aim of this *in-vitro* study was to investigate the effect of differing surface treatments on the shear bond strength of a type III gold alloy to chemically active resin cement. The variables considered were sandblasting with 50μm alumina; sandblasting with CoJet™ Sand alone (30μm particles); and sandblasting with CoJet™ Sand followed by the application of a silane primer.

The null hypothesis was that there is no significant difference in the average shear bond strength of Panavia 21 resin to cemented gold alloys using sandblasting with aluminium oxide, CoJet™ sand alone or CoJet™ sand with a silane coupling agent.

MATERIALS AND METHODS

Cylindrical plastic sticks with a diameter of four millimetres were cut into 5mm lengths, one hundred and twenty in total. The plastic sticks were then sprued (see figure 2), invested and cast in type III gold alloy (see figure 3). Subsequently, the gold alloy specimens were removed from the sprues and placed in an aluminium polishing jig, and wet grinding was carried out with 1000-grit silicon carbide paper for 10 minutes at 5N force and 300 rpm on a Knuth Struers Rotary Polisher, in order to ensure flat ends. The polishing jig and grub screw produced parallel surfaces on the end of each specimen.

There were three experimental groups and in each group there were twenty pairs of specimens. The groups were treated as follows:

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*BDS, MClinDent
†BDS, BSc, MSc, MFDS RCS (Eng), ILTM
‡BDS, MSc
§BDS,BSc,MSc,FDS RCS(Eng),MOrth,FOrth
¶PhD, BDS, MSc, MSc, DDPH RCS(Eng)

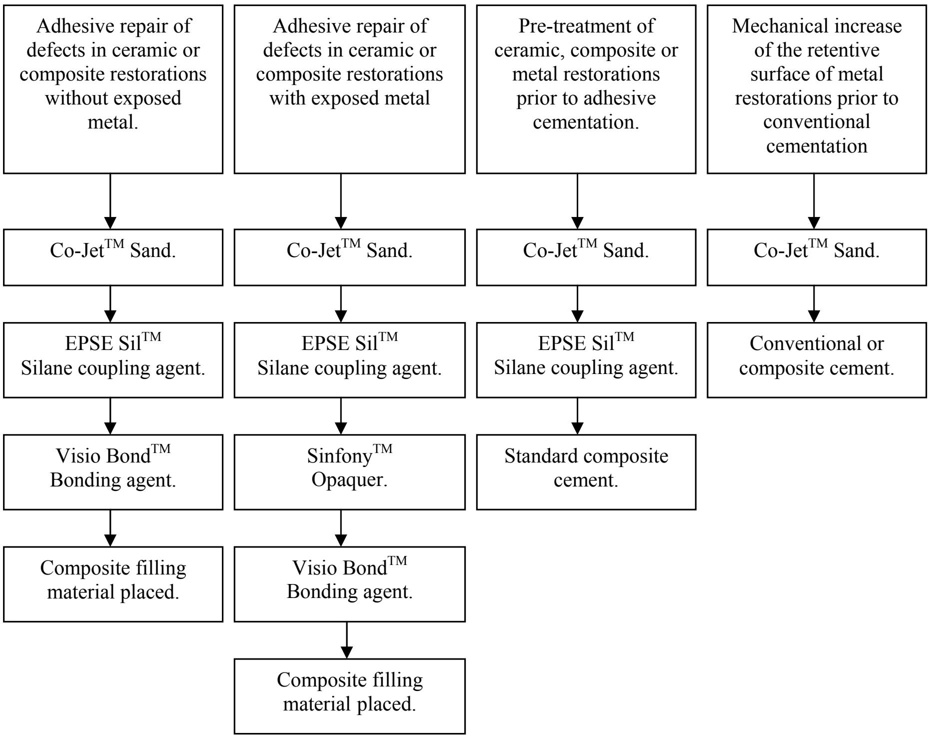


Figure 1. *Flow diagram to show manufacturer recommended protocols for the use of CoJet™*

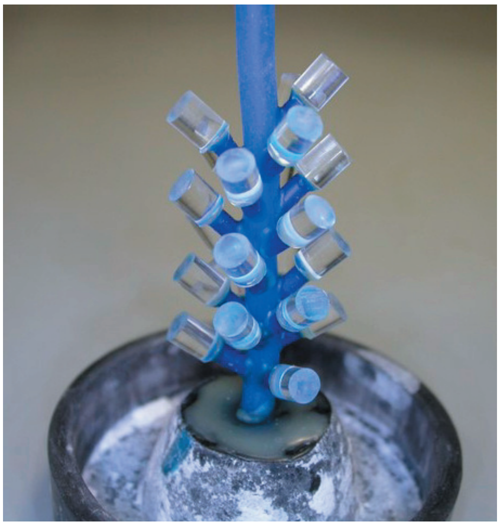


Figure 2. *Cylindrical plastic sticks cut into 5mm length, sprued prior to investing*

Figure 3. *Gold alloy castings.*

Group 1 – Sandblasted with alumina

The flat surface of each gold alloy cylinder was sandblasted with 50µm alumina particles at a pressure of 2-3 bar (35-45 psi) from a distance of 10mm for 15 seconds. The specimens were then steam-cleaned and allowed to dry for 5 minutes prior to cementation.

Group 2 – CoJet™ Sand

The flat surfaces of the gold alloy cylinders were prepared according to the manufacturers instructions. The adherend surfaces were blasted with a tribochemical silica coating (CoJet™ Sand), using 30µm silanated particles of silica coated alumina at a pressure of 2-3 bar (35-45 psi) from a distance of 10mm and perpendicular to the surface of the metal. The surfaces were blasted for 15 seconds. Any residual blast-coating agent was removed using a stream of dry, oil-free air. The samples were then allowed to stand for 5 minutes prior to cementation.

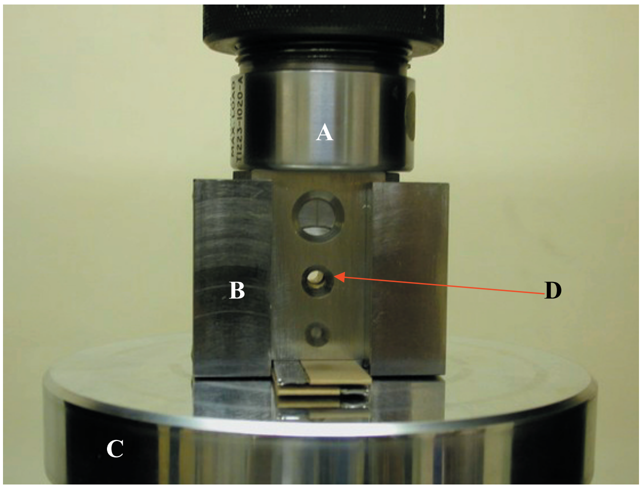
Group 3 – CoJet™ System

The flat surfaces of the gold alloy were prepared the same way as for those in group 2. After the surfaces were cleaned, they were then coated with silane-coupling agent. The silane solution was allowed to dry for 5 minutes prior to cementation.

Panavia 21 catalyst and universal paste were dispensed and mixed for 20 – 30 seconds on a mixing pad until the paste became smooth and uniform. A thin layer of the mixed cement was then applied on the adherend surfaces of the specimens. The two metal cylinders were held together in a plastic tube in order to align the specimens together while the cement was setting. A vertical static load of 5 kg was then applied for 3 minutes. Excess cement was then removed using a disposable brush and a layer of oxygen exclusion gel (Oxyguard II) was applied all around the exposed margins. The specimens were allowed to set for a further 10 minutes prior to storage in de-ionised water at 37°C. All cemented specimens were stored for 24 hours before testing.

The shear bond strength was tested using an Instron Universal Testing Machine. The shearing jig was designed according to the ISO/TR 11405:1994(E) [19]. It consisted of a solid block for fixation of the specimens and a connected shearing blade (see figures 4 and 5). The cemented specimens were placed in the shear-testing jig and aligned with a flat broad shearing edge at a 90° angle to the direction of the load. The Instron Universal Testing Machine was attached to a computer installed with a software package (Series IX Automated Materials Testing System, Version 5.34.00) that recorded the load at which the specimens debonded. The Instron machine was activated to apply compressive load to the shearing blade at a crosshead speed of 0.5mm per minute. The load at failure was recorded in kilonewton (kN) and then converted to megapascal (MPa).

The raw data were analysed using a statistical package (SPSS – Statistical Package for the Social Sciences) version 11. Analysis of variance was used to investigate whether there was any evidence of an overall difference in the means between the groups. Further post hoc comparisons between the groups were undertaken applying a Bonferroni multiple comparison correction to adjust for the effect of multiple testing.



A – load cell
B – shearing jig
C – Instron loading table
D – cemented specimens were placed here

Figure 4. *Instron Universal Testing Machine with test jig*

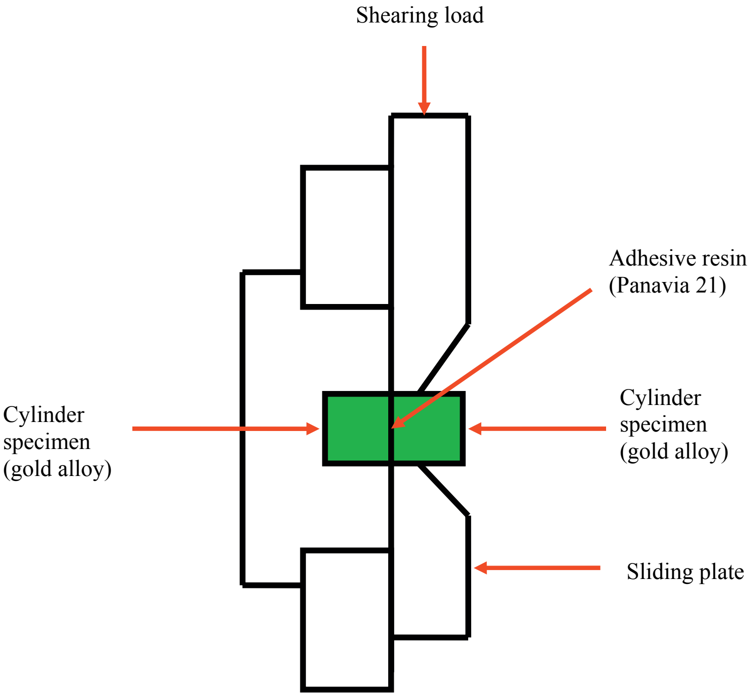


Figure 5. *Schematic diagram of jig for testing shear bond strength*

RESULTS

The mean shear bond strengths for each of the surface treatment group are presented in Table 1 with their corresponding standard deviations and 95% confidence intervals (95%CI). Table 2 shows the results of the analysis of variance for difference in mean shear bond strengths between the surface treatment groups. The post hoc comparison using the Bonferroni multiple comparison adjustment is shown in Table 3.

Treatment of the gold surface with the CoJet™ System produced the highest mean shear bond strength (45.6 +/-25.8 MPa). Blasting with CoJet™ Sand alone showed the lowest shear mean bond strength (26.5 +/-10.8 MPa). There were no significant differences between sandblasting with aluminium oxide 50μm particles and both the CoJet™ Sand and the CoJet™ System. However, the CoJet™ Sand alone had a significantly lower mean shear bond strength than the CoJet™ System group ($p<0.05$).

DISCUSSION

This study compared the effect of several surface preparations on the shear bond of type III gold cemented to Panavia 21 resin. The products and procedures tested in this *in vitro* study simulate the metal/resin part of a bonded prosthesis in which the metal adherend is type III gold alloy.

The null hypothesis that there was no difference in the mean shear bond strength of Panavia 21 resin to cemented gold alloys, following sandblasting with aluminium oxide, CoJet™ sand alone, or CoJet™ sand with a silane coupling agent was accepted between;

(1) aluminium oxide (50μm Particles) and CoJet™ sand with a silane coupling agent, and

(2) aluminium oxide (50μm particles) and CoJet™ sand alone.

However the null hypothesis was rejected for the alternative hypothesis of a difference between treatment with CoJet™ sand and CoJet™ sand with a silane coupling agent. The greater mean bond strengths occurred in the group of CoJet™ samples with the silane coupling agent.

In this study, rather than using a bulk of resin cement the gold alloy specimens were bonded together with a limited film thickness similar to their clinical application. It is assumed that a minimal cement line will encourage complete seating of the restorations and minimise the internal flaws of the cement, thus providing optimal bond strength. Thinner film thicknesses produces a better adhesive joint[20] and while film thicknesses were not determined for every sample, microscopic evaluation of selected samples indicated that film thicknesses of < 25μm were achieved.

It has been postulated that particular restorations are subjected to different types of forces [22]. Proposing that the

Table 1. *Mean shear bond strength and corresponding standard deviation for all groups.*

Groups	*Mean shear bond strength (MPa) +/- SD*	*Min (MPa)*	*Max (MPa)*
50μm alumina	32.90 +/- 18.22	8.71	78.24
CoJet™ Sand only	26.53 +/- 10.78	9.90	51.62
CoJet™ System	45.58 +/-25.79	18.54	117.50

Table 2. *One-way analysis of variance (ANOVA) for the difference in the mean shear bond strengths between the surface treatment groups.*

	Sum of Squares	*df*	*Mean Square*	*F*	*Sig.*
Between Groups	3759.88	2	1879.94	5.07	.009
Within Groups	21144.42	57	370.96		
Total	24904.31	59			

Table 3. *Bonferroni post hoc analysis of the differences in the mean shear bond strength between the surface treatment groups.*

					95% Confidence Interval	
(I) GROUP	*(J) GROUP*	*Mean Difference (I-J)*	*Std. Error*	*Sig.*	*Lower Bound*	*Upper Bound*
Sandblast	CoJet™ Sand	6.36	6.09	0.901	-8.66	21.39
	CoJet™ System	-12.68	6.09	0.126	-27.71	2.34
CoJet™ Sand	Sandblast	-6.36	6.09	0.901	-21.39	8.66
	CoJet™ System	-19.04	6.09	0.008*	-34.07	-4.02
CoJet™ System	Sandblast	12.68	6.09	0.126	-2.34	27.70
	CoJet™ Sand	19.04	6.09	0.008*	4.02	34.07

* The mean difference is significant at the .05 level.

forces required displacing a crown would be closer to those of shear test, whereas for the resin bonded anterior bridge, the displacing forces may have a greater tensile component. Ultimately, neither tensile nor shear bond strength tests exactly simulate the true oral environment [22].

In this study, all the specimens were prepared and then stored in de-ionised water at 37°C before testing at room temperature. The ISO/TR 11405 [19] suggests that storage in water for 24 hours is normally sufficient to discriminate between those materials which may or may not withstand a wet environment. Panavia 21 was chosen for use in this study as it is a commonly used resin cement, the results of this study are solely limited to this cement and cannot be applied to other resin luting materials.

Bond strengths are usually expressed in megapascals, as in this study, however, this value is an approximation because to calculate megapascals requires the substrate's cross-sectional surface area, this is altered by surface preparation (sandblasting). This makes the determination of the actual surface area difficult to quantify with precision [23]. This effect would have been similar for all samples.

After sandblasting with CoJet™ Sand, in group 3, a silane was added to wet the surface and form a chemical bond between the silica layer and the resin composite (the CoJet™ System). Results of previous studies suggest that silica-coating of non-precious alloy, followed by silane priming, significantly improves chemo-mechanical bonding of resin to metal [23, 24].

The significant difference in shear bond strength between group 2 and group 3, in favour of group 3, may be attributed to the silane treatment in this group (CoJet™ System). Silane is a surface modifier: its hydrolysed analogues called a silanol is a bifunctional molecules. The hydrophilic part forms hydrogen bonds with the surface bound water adsorbed on the alloy surface. The hydrophilic part of the silanol molecule copolymerizes with the methacrylate group of the resin via a covalent bond to enhance adhesion between metal and resin composite [23]. It should be noted that the silane-initiated bond deteriorates with time. It has been suggested that water uptake by the resin at the interface causes hydrolysis of the silane bonds resulting in deterioration in the adhesion between resin and metal [27]. The main reason why resin adhesives fail to obtain good contact quality with metal surface is not due to high viscosity, but the fact they must compete with adsorbed water on the metal surface [27]. The manufacturers of the CoJet™ system suggest that since the cohesive strength of a set silane layer is low, that by itself it cannot create the necessary retentive strength to metal. Instead, silane provides a coating over which the adhesive resin can readily flow to attain intimate contact and subsequent micromechanical retention to the sandblasted metal surface. Published data to support this proposed mechanism is lacking and requires further investigation.

Silane can link chemically to silica tribochemically bonded to the metal surface: Both group 2 and group 3 specimens were subjected to tribochemical treatment with the CoJet™ Sand. This formed part of the post-sandblasting surfaces on the gold alloy, and its presence was confirmed by elemental microanalysis. It is therefore likely that the silane treatment in group 3 led to a link between the adherent silica and the bifunctional vinyl silane, in a manner similar to the silane-initiated resin repair of porcelain. This would provide a logical reason for the higher mean shear bond strengths achieved by group 3.

There may be a difference between the reported contribution of unbonded alumina particles to long term adhesive failure of resin to metal bonds [28, 29] and the potentially more stable tribochemical bonding inherent in the CoJet™ System.

While it is interesting to note that the mean bond strength found were as follows:

CoJet System™ > Aluminium Oxide (50µm particles) > CoJet™ Sand (30µm particles).

Surface treatment with Aluminium oxide did not yield statistically significantly different results from either of the other two treatments. There was large variability in the data obtained and this may account for the lack of statistically significant differences found and requires further investigation with a larger sample size.

This study only evaluates the early shear bond strengths in which the samples were not exposed to thermal (thermocycling) and (prolonged) mechanical loads. The values found may be therefore considered as an indication of 'maximum' bond strength achievable. While initial strength is an obvious requirement for clinical function, the durability of the bond under oral environmental stresses is equally important. It may be expected that all bonds will show deterioration as a result of thermal and mechanical load. However, it has been emphasised that an acceptable bonding system should not be affected by either of these clinically relevant aging parameters [30], which are often used to simulate aging of the resin bonds [31].

CONCLUSIONS

Within the limitations of this study, the following conclusions may be drawn:

- The CoJet™ System which included the application of the silane bonding agent after the gold alloy surfaces have been sandblasted with CoJet™ Sand, achieved the highest mean shear bond strength.
- There was a statistically difference between the shear bond strength of resin to alloy surfaces treated with CoJet™ Sand only and those treated with CoJet™ System (p=0.008).
- Sandblasting with alumina particles size 50µm produced a slightly higher mean shear bond strength compared to treatment with CoJet™ Sand alone, but the difference was not statistically significant (p=0.901).

MANUFACTURERS DETAILS

- Aluminium Oxide (50µm) powder. Chaperlin and Jacobs Ltd, Sutton, Surrey, UK.
- CoJet™ Sand (Blast coating agent 30µm). Batch 0005. 3M/ESPE Dental Products, St Paul, MN 55144-1000, USA.
- ESPE Sil™ (Silane coupling agent). Batch 0132. 3M/ESPE Dental Products, St Paul, MN 55144-1000, USA.
- Panavia 21. Batch Number 41257. Kuraray Company Ltd, Japan.
- Oxyguard II. Batch Number 41257. Kuraray Company

Ltd, Japan.
- EC830 - Type III Yellow Gold Casting Alloy - 73% Au, 15% Ag, 6.5% Cu, 2.2%Pt, 2.8% Pd). Engelhard Sales Ltd., Dental Department, Chessington, Surrey, UK.
- Cylindrical plastic sticks (4mm diameter). Metrodent Ltd, Lowergate, Paddock, Huddersfield, UK.
- Sprue Wax (2.5mm, 4mm). Renfert GmbH, Shulty Dental, Germany.
- Jelenko Accu-Therm 150 Heat-Soaking Furnace. Jelenko Dental Health Products, Armonk, New York, USA.
- Heraeus Hanau Combilabor CL-G77. High frequency casting machine. Heraeus Edelemtalle, Hanau, Germany.
- Polishing jig. Eastman Dental Institute, London, UK.
- Shear test jig. Eastman Dental Institute, London, UK.
- 1000 grit silicon carbide paper. Struers Ltd., Glasgow, UK.
- Knuth Rotor. Struers Ltd., Glasgow, UK.
- Aquaclean 3 (Steam cleaner). Degussa, Degussa AG, D-63403, Hanau, Germany.
- Instron Universal Testing Machine. Instron Corporation, Buckinghamshire, UK.

ADDRESS FOR CORRESPONDENCE

Christopher J Tredwin, Clinical Lecturer / Honorary Specialist Registrar in Restorative Dentistry, UCL Eastman Dental Institute, University College London, 256 Grays Inn Road, London, WC1X 8LD. Email: c.tredwin@eastman.ucl.ac.uk

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