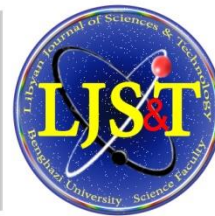




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A comparison of the shear bond strength between magnet keepers and composite resin after using two abrasive metal surface treatments; sandblasting and tribochemical silica coating (CoJet System®).

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ABSTRACT

Traditionally when magnetic attachments are used to retain overdentures, the keeper element is incorporated into gold copings, which often uses the root canal to gain additional retention to the tooth. This procedure may inevitably incur additional expense, laboratory and chair side time. The availability of magnetic attachments to patients may subsequently be limited. This study was conducted to compare the shear bond strength of magnet keepers to a commonly used composite resin following two abrasive metal surface treatments; sandblasting and tribochemical silica coating (CoJet System®). Sixty stainless steels (AUM20) keepers designed for magnetic attachments were embedded in acrylic blocks. Specimens were then divided into two groups of 30 specimens each. The first group was sandblasted with alumina and the second group was treated using the CoJet system. The composite resin was bonded to each magnet keeper and cured. Shear bond strengths were determined for each surface treatment after 24 hours storage in distilled water at 37°C (0 thermocycle), 500 thermocycling, and one-week storage in distilled water at 37°C. The shear bond strength was recorded for each specimen using a universal testing machine and statistical analysis of shear bond strength values within each group of surface treatment was compared using paired T-test. Independent T-test was used to compare the shear bond strengths of sandblasted keeper surfaces with those treated with the CoJet system®. The strongest shear bond strength was obtained with samples treated with CoJet system®. Between groups comparison showed that significant differences were found in the shear bond strength mean values at 0 thermocycle and one week water storage ($p=0.001$ and $p=0.006$). The values of shear bond strength recorded by the two treatments exceeded the maximum retentive force required to pull magnet attachment from its keeper. Most of the specimens tested exhibited adhesive failure mode. Within the limitation of this study, it is concluded that using the CoJet surface treatment on the surfaces of magnet keepers is effective for bonding the magnet keepers with the composite resin examined.

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1. Introduction

Since magnetic attachments can offer sufficient retention and bracing for overdenture prosthesis, the abutment tooth structure can be used to support the coping with the magnetic keeper. Based on the improvement of adhesive resins that provide sufficient binding force between metal and dentition, the stainless steel magnetic keepers can be adhered properly to the tooth surface through adhesive resins cements and composite resin copings. Therefore, eliminating problems of cast bonded technique used to incorporate magnet keepers into conventional gold copings. Less retentive tooth preparations rely on bonding agents that can bond to enamel and dentine (Ergin & Gemalmaz 2002).

Besides their simple application and good physical properties, adhesive resins have shown satisfactory durable bond strengths for non-precious and precious metals (Ozcan *et al.*, 1998). It has been observed in practice and concluded in literature that some

specific adhesive resins have more favourite results with specific materials and specific surface treatments (Imbery & Grant 1996).

Adhesive resins contain different adhesive monomers with different functional compounds that chemically react with precious and non-precious metal alloys (Suzuki *et al.*, 1999). Furthermore, adhesive resins provide different bond strengths when used with different metal alloys and with different surface treatment methods (Isidor *et al.*, 1991; Zhang *et al.*, 2001). The high rate of failure caused by debonding of resin bonded to metal restorations has been reduced by using improved bond systems and choosing the suitable alloy surface treatments (El-Mowafy & Rubo 2000). A reliable chemical bond at the metal restoration-resin interface can be achieved by either modifying the metal surface or reconfiguring the chemistry of the resin cement restorations (Fazi *et al.*, 2012).

Various surface treatments procedures are used to increase the bond between dental alloys and adhesive resins by increasing the surface area thus enhancing the mechanical and/or the chemical bonding of the resins to the metal. Incorporation of macro-

mechanical or micro-mechanical features can be used to improve the mechanical bond of composite resin to metal restorations (Di Francescantonio et al., 2010).

However, some surface treatment methods may produce a combination of mechanical and chemical bonds. In addition, changing some mechanical features on the metal surface could improve the chemical bonds. An increase in the micromechanical interlocking of the resin at the metal surface can be achieved by sandblasting non-precious and precious metal alloys followed by ultrasonic cleaning that eliminates any loose surface particles and thus making the chemical reaction more reliable without altering the composition of the metal surface (Kern & Thompson 1993).

Air abrasion of a metal surface with aluminium oxide particles of 50µm or 250 µm diameter has been used to increase the mechanical bond between composite resin and metal surface. The oxidizing agent forms an oxide film that subsequently improves wetting between the resin and the metal surface (Wiltshire 1986; Atta et al., 1990).

The important factors that control the effectiveness of air abrasion or sandblasting are particle size, sandblasting time, and pressure during sandblasting surface treatment. These factors are in turn related to the metal alloy composition. The firmly attached aluminium is strongly associated with the bonding process (Byeon et al., 2017).

Sandblasting is recommended as pre-treatment to thermal silica-coating or as part of a silica-coating process for both precious and non-precious metal alloys (Luthy et al., 1990). Acceptable results were recorded of using a silica coating (silicoater) for preparing non-precious metal alloys to bond laboratory curing composite resin veneers by composite resin and resin cements (Kolodney et al., 1992; Chang et al., 1993). Furthermore, better results provided by chemo-mechanical silica coating compared with only sandblasting for bonding resin composite to non-precious metal alloys (Ishijima et al., 1992).

The technique was first introduced in the late 1980s to improve the bond of acrylic resin to metallic frameworks as a laboratory based technique called Rocatec system (CoJet®) (Watanabe et al., 1988; Guggenberger 1989). In this system, the metal surface is airborne-particle abraded at high pressure with a special powder that contains fine alumina and colloidal silica particles.

The tribochemical coating system uses mechanical energy to forms a chemical bond to the alloy surface. It produces micromechanical retention and a site for chemical adhesion (Sun et al., 2000). The aluminium oxide particles are sprayed on the alloy surface to make it rough, and then sprayed again with aluminium oxide particles modified by silicic acid. These particles are blasted

and hit the alloy surface under pressure thereby a localized heat is produced around them. As a result of heating together with the blasting pressure, silica particles penetrate a few microns into the metal surface. A chemical bond between the metal surface and the resin through the silane is thus gained (Imbery & Grant 1996).

Silane molecules are thought to play a double role. They bond to the silica on the metal surface, and form copolymerization bonds with the resin composite (Cobb et al., 2000). The addition of silane to sandblast a non-precious alloy surface increases the bond strength with resin cement even without using a bonding agent and adding a silane during the tribochemical process results in an increase in the bond between metal surfaces and composites (Rammelsberg et al., 1993).

An increase in the concentration of Al and Si is observed on surfaces sandblasted during a tribochemical process. Changes in surface morphology and volume loss also take place during silica-coating, however, the exact mechanism by which the tribochemical process works still needs more investigations (Kern & Thompson 1993).

It has been concluded that there were no significant differences between bond strengths of resin composites bonded to non-precious metal alloys treated with tribochemical silica coating and those luted to precious metal alloys (Moulin et al., 1999). However, it has been reported that non-precious metal alloys recorded higher bond strengths with resin composite than precious ones when treated with Rocatec system (Ozcan & Vallittu 2003).

Although instant bond strength values of thermal silica coated non-precious metal alloys luted to BisGMA were higher than those of tribochemical silica coated alloys, but five months ageing made the bond strength values of tribochemical silica coated surfaces increase and those of thermal silica coated surfaces decrease.

The aim of this study was to measure the shear bond strength of magnet keepers to a commonly used composite resin following two abrasive metal surface treatments; sandblasting and tribochemical silica coating (CoJet System®).

2. Materials and methods

A single liquid primer designed for conditioning base metal alloys (OptiBond Solo plus®) and a light-cured, resin-based composite (Point 4®) were selected for bonding the stainless steel magnet keepers. Selection of liquid primer was based on a previous report (Matsumura et al., 1997) which compared the effect of metal conditioners on bonds formed between a stainless steel and two auto-polymerizing methacrylic resins. A summary of the materials main composition and manufacturers are presented in (Table1).

Table 1.
Materials used

Material	Composition	Manufacturer
Magnet keepers (AUM20)	Fe 78-4%, Cr19-2%, Ni, Mo 1-9%	Aichi Steel Corp., Japan
Light-cured composite (Point 4)	Bis-GMA resin matrix with Barium aluminoborosilicate glass, and silicon dioxide filler.	Kerr Italia S.P.A
Metal primer (OptiBond Solo plus)	Alkyl dimethacrylate resins, barium aluminoborosilicate glass, silicondioxyde, sodium hexafluorosilicate, ethyl alcohol.	Kerr Italia S.P.A
CoJet-System	Sand (CoJet® Sand): Silicized sand (particle size 30µm) Silane (ESPE Sil®): Silane with an attached methacrylic group, and Ethanol Bonding agent (Visio-Bond®): Bisacrylate, Aminodioldimethacrylate, Camphor quinine, Benzylidimethylketale, and Stabilizers	3M ESPE

2.1 Sample size estimation

Sample size was calculated according to previous laboratory study comparing the effect of different surface treatment of high nobel alloy on the shear bond strength of an indirect, highly filled resin composite to the alloy and on the elemental composition of the alloy surface (Petridis *et al.*, 2004). It was determined that a sample of 30 specimens for each group was needed to detect a significant change difference in bond strength. The sample size was calculated with $\alpha = 0.05$ and power = 0.8.

2.2 Preparation of acrylic blocks and mounting magnet keepers

In order to prepare solid acrylic blocks for mounting the magnet keepers; a one-piece mould made of good quality polythene provided a hollow cavity for the solid blocks with the required dimensions 30 mm in diameter and 30 mm high. A double sided tape was used to attach centrally and evenly each specimen with the bonding surface facing the base of the polythene mould. An autopolymerising clear casting acrylic resin and catalyst (Bonda, Bondaglass Voss Ltd, England) were mixed together as recommended by the manufacturer's directions (6 drops of catalyst for each 10 grams of resin) and poured into the polythene mould and left for 24 hours for complete polymerisation. A total number of 60 auto-cure acrylic resin blocks were made.

2.3 Preparation of keepers bonding surfaces for treatments

Sixty stainless steel magnet keepers 4mm diameter and 0.9 mm thickness (AUM20®) were used in this investigation for the two metal surface treatments of 30 specimens each. The following surface treatment methods were employed per experimental group:

2.3.1 Group 1. Sandblasting and metal primer application

In this group, the surfaces of the 30 specimens were subjected to perpendicular air abrasion with a sandblasting machine (Renfert Basic Master, Germany) using 50 μ m alumina particles (Al2 O3). The emission pressure was 0.5 MPa for 10 seconds and the distance of the nozzle from the metal surface was approximately 6mm. The sandblasted surfaces were then air cleaned and following the manufacturer's instructions, each treated surface received a liberal coating of metal primer (OptiBond Solo Plus) using a single fully saturated brush tip for 15 seconds. The surface was then lightly air dried for 3 seconds and light cured for 20 seconds.

2.3.2 Group 2. Tribochemical silica coating (CoJet System) application

A blast-coating agent (CoJet sand) was used to sandblast the bonding surface of each keeper for 15 seconds under a constant air pressure of 0.28 MPa using a controlled pressure intraoral blaster. The distance of the nozzle from the surface of the specimen was approximately 6mm. The bonding surface was air dried immediately following the abrasive treatment. For each sandblasted surface, a new brush was used to apply the silane coupling agent and left for 30 seconds to dry. The bonding agent of the CoJet System was then applied using another new brush and cured for 40 seconds using a light curing apparatus.

2.4 Composite resin bonding procedure

A piece of tape with a circular hole (3.2 mm in diameter and 20 μ m in thickness made using a puncture punch) was positioned over the centre of each treated surface of magnet keepers to define the bond area. Composite (Point 4 Kerr Italia) was used to fill up to 2 mm below the top of transparent gelatine capsules of 5mm diameter. Each capsule was prepared in advance and to act as a matrix for light-cured composite resin. According to the

manufacturer's instructions, a fresh composite resin (Point 4 Kerr Italia) was injected onto the remaining 2 mm section left in each capsule, and held firmly and centrally on the treated surface of a specimen. The excess resin cement was removed and according to the manufacturer's recommendations, a 3M light curing unit (Visilux-2- 3M) was used to light-polymerise each side of the resin for 40 seconds. The specimens were approximately 10 cm from the light source with an intensity of 4.75 mW/cm.

2.5 Water storage

Specimens were then subjected to a combination of water storage and thermocycling in order to give and simulate of the conditions that occur in vivo (Crim *et al.*, 1985). Therefore, all 30 bonded specimens of each surface treatment group were divided into 3 subgroups of 10 specimens each; the first subgroup were stored in distilled water at 37 °C for 24 hours (24 hours baseline or thermo-cycled 0) then tested for shear bond strength at this stage. The second subgroup was stored in distilled water at 37°C for 7 days and then tested for shear bond strength. The third subgroup was placed in a thermocycling apparatus and cycled 500 times between 5°C and 55°C water baths with a dwell time of 30 seconds in each one and subsequently upon completion of the thermocycling procedure tested for shear bond strength.

2.6 Shear bond strength measurements:

The ISO 10477: 2004 document specifies a shear test apparatus consisting of a solid block for fixation of the specimens and a connected shearing blade with a 0.5 mm blunt edge. The test was originally proposed by (Noguchi *et al.*, 1982) and later has been used for shear bond testing for resin/metal specimens (Petridis *et al.*, 1999; Watanabe *et al.*, 1999; Matsumura *et al.*, 2001).

A computer-controlled universal testing machine (Instron model 5567) with a 1.0 KN compressive load cell, at a crosshead speed of 0.5 mm/min was used to measure shear bond strength. Each specimen was positioned in a steel mould and seated in a bond test jig (ISO TR 11405). The upper platen of the machine was mounted with a stainless steel blunt edge that used to shear away the light-cured composite resin bonded to magnet keepers. Then the computers interfaced Instron machine recorded and calculated shear bond strength using series IX software version 8.15.00.

On completion of shear bond strength testing, the bonding surface of each magnet keeper was polished with a cloth wheel machine using a mixture of water and 1 micron Alpha micropolish Alumina liquid 1C or 0.5 microns Alpha micropolish Alumina liquid 3B (Buehler USA). Specimens were subsequently, washed with water and cleaned with ethyl alcohol using a clean tissue paper and dried. The previous surface treatments and composite resin bonding procedures were repeated for specimens of each subgroup, then exchanged to the other with a new water storage procedure. The sequence of each surface treatment is presented in (Fig. 1) and overview of the shear bond strength test design is illustrated in (Fig. 2).

In addition to shear bond strength, the nature of failure for every specimen was observed with the naked eye as either adhesive (occurring at the junction of the resin-metal interface) or cohesive (occurring within the body of the composite). In cases of doubt, a microscope at a magnification of 4.5 x was used to determine the failure mode.

2.7 Statistical analysis

Statistical analysis of shear bond strength values within each group of surface treatment for different storage condition was compared using paired T-test. Independent T-test was used to compare the shear bond strengths of sandblasted keeper surfaces with those treated with the CoJet system.

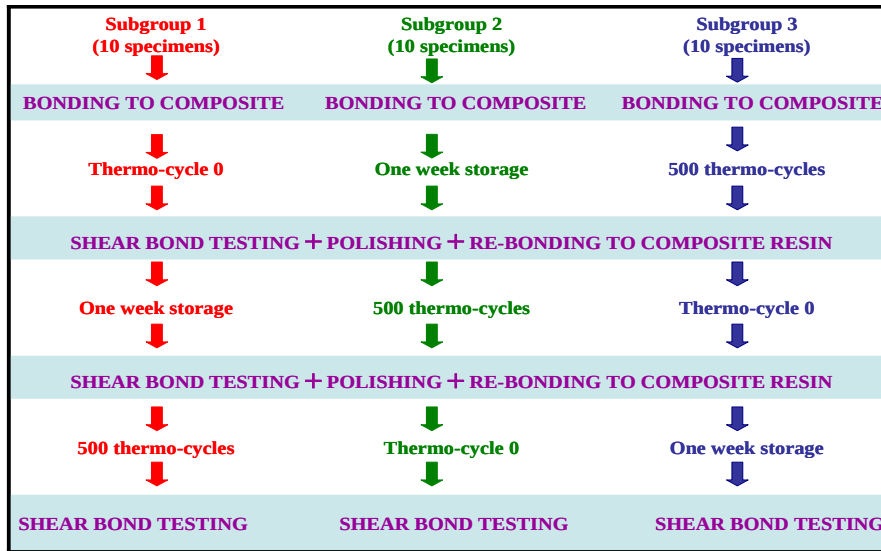


Fig. 1. Illustration of experiment sequences for 30 specimens of each surface treatment.

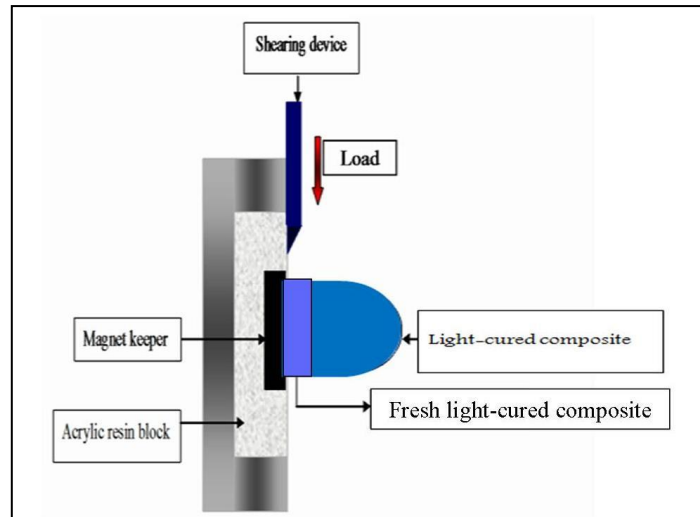


Fig. 2. Shear bond strength test design.

3. Results

According to statistical analysis there were no significant differences within each metal surface treatment subgroups (Table 2). The strongest shear bond strength was obtained with samples treated with CoJet system whereas, sandblasted samples with thermocycle 0 showing the lowest strength values.

Between groups, comparison showed that significant differences were found in the shear bond strength mean values at thermocycle 0 and one week water storage ($p=0.001$ and $p=0.006$) (Table 3 & Fig. 3).

Table 2.
Within group comparisons of bond strengths at different storage conditions

SANDBLASTING						COJET SYSTEM				
	Mean $\pm$ SD	Mean differ $\pm$ SD	95% CI		P-value	Mean $\pm$ SD	Mean differ $\pm$ SD	95% CI		P-value
Thermocycle 0 One week storage	13.9 $\pm$ 5.3	1.1 $\pm$ 6.9	-1.5	3.8	0.392	19.7 $\pm$ 6.3	1.6 $\pm$ 10.8	-2.7	5.8	0.449
	12.8 $\pm$ 5.1					18.1 $\pm$ 7.9				
Thermocycle 0 500 thermocycle	13.9 $\pm$ 5.3	2.4 $\pm$ 8.8	-6.3	1	0.143	19.7 $\pm$ 6.2	3.3 $\pm$ 9.2	-0.7	6.9	0.106
	16.3 $\pm$ 7.0					16.4 $\pm$ 6.1				
One week storage Thermocycling	12.8 $\pm$ 5.0	3.5 $\pm$ 8.1	-6.3	0.4	0.083	18.1 $\pm$ 8.0	1.7 $\pm$ 10.6	-2.1	6.3	0.315
	16.3 $\pm$ 7.0					16.4 $\pm$ 6.1				

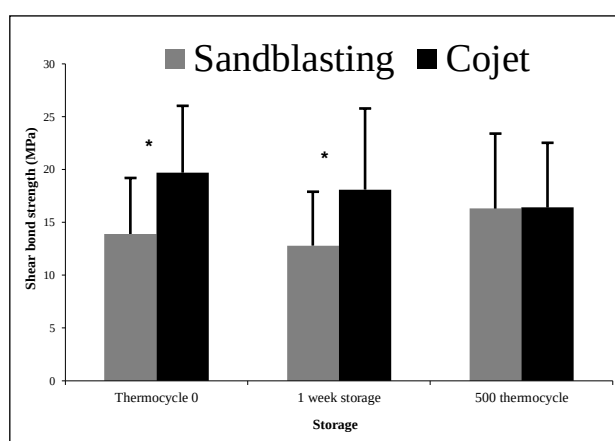
Table 3.

Between groups comparisons of shear bond strengths at different storage conditions.

	SANDBLASTING	COJET SYSTEM	Mean difference.±SD	95% CI		P-value
	Mean ±SD	Mean ±SD				
Thermocycle 0	13.9 ±5.3	19.7 ±6.33	5.8± 1.6	-9.1	-2.9	0.001
One week storage	12.8 ±5.1	18.1 ±7.68	5.3± 1.7	-8.4	-1.5	0.006
500 thermocycle	16.3 ±7.1	16.4 ±6.13	0.1± 1.9	-2.8	4.7	0.617

Most of the specimens of all subgroups tested in this study exhibited adhesive failure mode at the metal-composite resin interface (adhesive), whereas thirteen specimens in total showed mechanical failure (composite along with magnet keeper detached

from their holding acrylic blocks). A possible explanation for the mechanical failures recorded for both treatment groups could be due to the influence specimens fixation into acrylic blocks.

**Fig.3.** Significant shear bond strength between metal surface treatments.

4. Discussion

Bonding composite resins to metal may be obtained through mechanical, chemical bond or a combination of the two. Chemical bonding is based on the alteration of the metal surface by creation of oxidized layers or with the use of adhesive monomers. Various studies indicate that using chemical methods to bond resin to metal produce a stable bond at temperature variations (Oweis et al., 2017).

However, chemical bonding is technique sensitive with the possibility to contaminate the adhesive layer resulting in decrease of the composite-metal bond strength (Yoshida et al., 2001).

Studies into the mechanism by which sandblasting treatment improves the bond strength between the resin and the precious and non-precious metal alloys indicate that the sandblasting increases surface area in the range of 1.3-1.9 times. Furthermore, the sandblasting treatment made the alloy-water contact angle smaller and the wet ability greater. The bond strength records were 2-3 times greater than those of untreated metal surfaces (Kawamura 1989). The use of 50 µm alumina for sandblasting non-precious metal surfaces resulted in bond strength values higher than those produced by either etching or salt particle inclusion (Harley & Ibbetson 1991). Sandblasting with 50 µm alumina produced higher bond strength values than with 250 µm alumina and furthermore, higher than with etched non-precious metal alloys (Peutzfeldt & Asmussen 1988; Van der Veen et al., 1988). In addition, poor retention is achieved if sandblasting is not used at all (Seon et al., 2017).

The shear bond strength in the current study was tested after applying two different metal surface treatments, sandblasting and Cojet system, recommended for bonding composite to metal alloys. In general, Cojet system produced higher shear bond strength values than sandblasting and the absence of statistically significant difference between Cojet abrasion and sandblasting at 500 cycles

does not change the overall performance of both treatments. In relation to the clinical applications of these metal treatment systems and according to the requirements of the revised ISO 10477, the minimum acceptable shear bond strength values at least 5 MPa. However, it is suggested that the resin to-metal shear bond strength necessary for the achievement of clinically satisfactory results should exceed 10 MPa (Matsumura et al., 2001). The bond strength values recorded in the present study for all resin-magnet keeper surface treatments, even with 500 thermal-cycles exceed the requirements of ISO 10477. Restorations in the oral environment are continuously subjected to temperature changes. Thermocycling is a common laboratory technique to simulate aging of materials in the oral environment. Although it has been cited that the major deteriorating effects of thermo-cycling on a heterogeneous interface appear up to the first 200 cycles (Luthy et al., 1990). In the present study, 500 thermal-cycles were used. The observed decrease of mean shear bond strength in the thermocycling subgroups after 500 thermal-cycles was not statistically significant although, the revised ISO 10477 recommends 5000 thermal cycles for testing these materials. Optical observation of the debonded specimens revealed that the mode of failure was adhesive. This indicates that the cohesive strength of the composite resin used in the study exceeded the metal-resin bond strength for all surface treatments and thermal cycles. Considering the different experimental conditions (type of alloy and adhesive resin, number of thermal cycles, airborne-particle abrasion with different sizes particles), the values reported in the present study are within the range of those in the revised ISO 10477.

A study measured the Shear bond strengths before and after thermocycling, using AUM20 alloy as one of the substrate material, which recorded mean shear bond values of 16.3 MPa for unprimed post-thermocycling bond strengths and 30.3 MPa for those of the

conditioned specimens. These values were higher than the corresponding values of the present study (15.9 MPa) (Matsumura et al., 2000). This could be due to differences in the number of thermal cycles used; therefore, the relatively lower bond strengths observed in the present study could be attributed to the lower number of 500 thermal cycles used. There is strong evidence that retention of any prosthesis is of great importance for a patient's satisfaction (Burns et al., 1995). However, it is assumed that forces of 8 to 20 N are sufficient for overdentures in the edentulous mandible (Setz et al., 1998). The commercially available magnet attachments possess a range of 5.9 to 10.8 N retentive forces (Minoru & Shiao 2004). With the maximum force of 4.7N is required to pull magnet attachment from its keeper (Rutkunas & Miztani 2004). The design of the present study did not consider factors existing in the oral environment, such as pH changes; therefore, careful application of the results in clinical situations is suggested. The efficacy of the tested systems in providing reliable bond strength should be confirmed by further research studies.

5. Conclusion

Considering the limitations of the present laboratory study, the shear bond strength values of bonding composite resin to magnet keepers' surfaces by the two treatments exceed both the requirements of ISO 10477 and the retentive force of magnets. The bond strength values between the AUM20 alloy and the composite resin evaluated was higher for CoJet system compared to sandblasting. There was no significant effect of the thermal cycles on the two surface treatments on the resin-metal bond strength despite the decrease observed in bond strength values.

In summary, the development of a stable bond between magnetic keepers and composite resin copings may provide a reliable and cost-effective alternative to conventional gold copings. The results of this laboratory study indicate that the use of CoJet system effectively enhanced bond strength between magnetic keepers and composite resin copings.

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