Influence of different surface treatments on microshear bond strength of repair resin composite to two CAD/CAM esthetic restorative materials

M.M. Wahsha,*, O.H. Ghallab

*Department of Fixed Prosthodontics, Faculty of Dentistry, Ain Shams University, Cairo, Egypt  
Operative Dentistry Department, Faculty of Dentistry, Ain Shams University, Cairo, Egypt

Received 22 April 2015; revised 27 May 2015; accepted 1 June 2015

Abstract

Objective: The aim of this in vitro study was to evaluate which surface treatment and adhesive type might provide the most predictable microshear bond strength results of repair resin composite to lithium disilicate ceramic and a newly introduced resin nanoceramic (RNC).

Methods: A total of thirty six slices were prepared out of resin nanoceramic (Lava Ultimate) and lithium disilicate (IPS e.max CAD ceramic blocks) with dimensions of 14 × 12 × 3 mm (n = 18 for each material). The slices were divided into three main groups according to surface treatment: Control group (CN) received no treatment, group (DB) roughened with diamond bur and group (CJ) silica coated with Cojet system. Each group was subdivided into two subgroups according to the type of adhesive system used for repair: Subgroup (MH): Treated with Monobond plus and Heliobond adhesive system and subgroup (SBU): treated with Single bond universal adhesive system. The adhesives were applied according to the manufacturers’ instructions. Five transparent microtubules were adjusted on each substrate slice and then repair resin composite (Tetric Evoceram, shade A3) was applied and light cured (n = 15 for each subgroup). After 24 h storage in distilled water, microshear bond strength (μSBS) was measured for each specimen of tested groups.

Results: Lava Ultimate RNC showed statistically significant higher μSBS compared to IPS e.max lithium disilicate ceramic. The use of diamond bur in conjunction with Monobond plus and Heliobond adhesive system showed higher μSBS compared to all tested groups.

Conclusions: The effect of different surface treatments on the μSBS to the repair resin composite is material dependent. Combination of the abrasive action of diamond bur and adhesive with separate silane treatment modalities had a synergistic influence on μSBS of the repair resin composite under the current circumstances.

© 2015, Hosting by Elsevier B.V. on behalf of the Faculty of Dentistry, Tanta University.

Keywords: Lithium disilicate; Resin nanoceramic; Repair; Surface treatments; Microshear bond strength
1. Introduction

One of the goals of restorative dentistry is to provide functional and esthetic restorations. Ceramics are widely used clinically as indirect restorations because of their proved long lasting physical and mechanical properties. Varieties of all ceramic materials and systems are available in the market for laminate veneers, inlays, onlays and all ceramic crowns [1–3].

Lithium disilicate-based glass ceramics are of the valuable advances in CAD/CAM technology. Unfortunately, ceramics have an inherent weakness due to their brittleness. Under occlusal forces or during cementation, fracture or chipping of the material can occur especially if there was an internal defect or inadequate thickness [4,5].

Recently, 3M ESPE introduced a new CAD/CAM RNC material (Lava Ultimate™ brand) to the dental market. The material is neither a resin nor a pure ceramic; it’s a mixture of both but mainly consists of ceramic. Lava Ultimate as a RNC and in terms of material science, it belongs to the resin composite category [6]. It is developed as nanomer particles which are monodisperse, non-aggregated and non-agglomerated nanoparticles. Lava Ultimate contains two types of nanomers; silica of 20 nm diameter, and zirconia of 4–11 nm diameter. The new material is claimed to combine the merits of high flexural strength, fracture toughness, resiliency, durability and esthetics. The material is available as highly heat cured blocks through a controlled manufacturing process; thus, the firing step after milling is eliminated. It can be chair-side machined or in the dental lab [7]. The literature is short of knowledge regarding RNC, thus it is in the focus of this study.

In cases of fracture or chipping of ceramic restorations, intra-oral repair may be considered, preserving the restoration and the tooth [8]. The previously published literature reveals the merits of intra-oral repair as decrease the cost for the patient and lengthening the life span of the fractured restoration [2,9–12]. On the other hand, Tyas et al. stated that, the repair is considered as (patchwork dentistry) and it is not approved by the clinicians [13]. It also may lead to a weaker restoration [11]. At an earlier time, repair depended on macro mechanical retention by preparing grooves or undercuts. Now, the available repair kits and systems depend on micromechanical and chemical bond through different surface treatments of the substrate creating an intermediate interface [4].

Resin composite has the ability to bond via advanced adhesive systems capable of bonding composite to different substrates rather than enamel and dentin [14]. The bond strength of repair interface determines the longevity and serviceability of the restoration [15]. This bond integrity and durability depend on the type of repair composite as hybrid composite resins reported higher bond strength than micro filled composite resins [10] as well as the surface treatment used [4,8,15–17].

Numerous surface treatments are available for repair of lithium disilicate-based ceramics through micro-mechanical and chemical retention [18]. Micro-mechanical roughening of the substrate surface can be achieved by diamond bur, acid etching with hydrofluoric or phosphoric acid, air-borne particle abrasion (which promotes mechanical interlocking) with or without silane coupling agents and adhesive systems (which enhances chemical bonding with the repair composite) [8,11,16,19].

Cojet is a chair side tribochemical silica coating system, substrate surfaces are abraded with 30 μm AL2O3 modified with silicon acid. The surface treatment using Cojet system may increase the bond strength of repair composite to ceramics [9,20,21] and resin nano-ceramics [22].

Silanes act as mediators promoting adhesion between inorganic and organic matrices through dual molecular reactivity and increase the wettability improving the contact angle between the ceramic and resin [2,3,9,18,21]. On the other hand, Sorensen et al.,[23] considered that the use of silane coupling agent had insignificant effect on the bond strength for all ceramics used. Barghi [24], stated that, the silane coupling agents remain a weak bond. Furthermore, silane can be subjected to hydrolytic instability especially in humid conditions [10].

From all the previously mentioned, it is concluded that there is no a specific method to condition the ceramic substrate for repair with resin composite. Therefore, the aim of this study was to evaluate the effect of different surface treatments on the micro shear bond strength of repair resin composite to lithium disilicate-based ceramic and RNC using two adhesive bonding agents.

2. Materials and methods

2.1. Preparation of substrates

The materials’ description, brand names, chemical composition, manufacturers, and Lot.numbers used in this study are listed in Table 1 and are provided from the manuals of the manufacturers. All the steps were performed by the same operator following the manufacturers’ instructions. A total of thirty six slices were prepared out of Lava Ultimate and IPS e.max CAD.
ceramic blocks using diamond discs under continuous water irrigation \((n = 18)\) for each material. The dimensions of the slices were \((14 \times 12 \times 3 \text{ mm})\). The slices were polished in a circular motion using silicon carbide papers of grits 600 and 1200 under continuous water irrigation, for 20 s each \([12]\). After the polishing procedures, the substrate surfaces were ultrasonically cleaned in distilled water for 30 s and then dried.

The IPS e.max CAD slices were placed in the Programat P300 ceramic furnace\(^2\) for crystallization firing cycle with a holding time of 10 min at 840 °C.

### 2.2. Samples grouping

For each material the 18 slices were randomly assigned into three groups according to surface treatment before repair \((n = 6)\). Group CN received no further treatment and served as the control group. The other two groups received surface treatments through roughening with diamond bur \((\text{DB})\); and silica coating using Cojet system \((\text{SC})\). Each group was divided into two subgroups according to the adhesive system used \((n = 3)\): Subgroup MH: Monobond plus + Heliobond\(^1\) + TetricEvoCeram (Light-curing nano-hybrid composite)\(^3\); and subgroup SBU: Single Bond Universal adhesive\(^2\) + TetricEvoCeram (Light-curing nano-hybrid composite)\(^2\).

#### 2.3. Substrate surface treatments

Control group \((\text{CN})\) received no surface treatment. For the DB group the surface was ground using a medium grit abrasive diamond bur\(^3\) using a high speed hand piece under copious air–water irrigation in one direction for 4 s on each surface \([16]\). And for the SC group the surface was air abraded using Cojet powder, through an intraoral air abrasion device\(^4\) at 90°, at

---

1. 3M ESPE, St. Paul, USA.
2. Ivoclar, Vivadent, Liechtenstein.
3. Mani Dia-burs, Mani, Inc., Tochigi, Japan.
4. Air prophy unit, Shanghai, China.
distance of 10 mm for 20 s, and pressure of 2.8 bar [4], rinsed for 10 s using air water spray and dried using oil/water free compressed air for 3 s. The untreated surfaces were marked using a permanent marker for the ease of identification.

2.4. Repair using Heliobond adhesive

Silane coupling agent (Monobond plus) was applied on the treated surfaces of the samples for 1 min using a brush. The plates were dried for 10 s with oil/water free compressed air. Adhesive resin (Heliobond) was applied using a brush, lightly thinned with compressed air. Light emitting diode curing unit of high intensity 1500 mW/cm² was used to cure the bonding agent for 20 s. The intensity of the light curing unit was checked using a radiometer before curing of each group.

2.5. Repair using Single Bond Universal adhesive

The adhesive was applied using a brush for 20 s, air-dried using oil/water free compressed air for 5 s and light cured for 10 s using the same light curing unit.

2.6. Application of the repair resin composite

Each substrate surface received 5 resin micro-tubules (n = 15 in each subgroup). Small transparent microtubules were cut from polyvinyl tube with inner diameter 0.9 mm and height 1 mm. After curing of the previously mentioned adhesives, each micro-tubule was adjusted over the cured adhesive, filled with light-curing nanohybrid composite TetricEvoCeram (Shade A3) and light cured for 20 s.

2.7. Microshear bond strength (µSBS) testing

After 24 h storage in distilled water, the microtubules were sectioned to expose the composite micro-cylinders using sharp scalpel blade number 11. Excess adhesive around each micro-cylinder was scrapped out using the same surgical blade to limit the bonding surface area. The micro-cylinders were examined by a magnifying lens for any defects at the interface.

Each specimen with the bonded composite micro-cylinders was secured with tightening screws to the lower fixed compartment of Universal testing machine⁵ with a load cell of 5N. A loop prepared from an orthodontic wire (0.014” in diameter) was wrapped around the bonded micro-cylinder assembly as close as possible to the base of the micro-cylinder and aligned with the loading axis of the upper movable compartment of the testing machine.

A shearing load with tensile mode of force was applied via the testing machine at a crosshead speed of 0.5 mm/min. The relatively slow crosshead speed was selected in order to produce a shearing force that resulted in debonding of the micro-cylinder along the substrate/adhesive interface. The load required for debonding was recorded in Newton and the data were recorded using computer software.⁷ The load at failure (Newton) was divided by bonding area (mm²) to express the bond strength in MPa. The results were collected, tabulated and statistically analyzed.

2.8. Statistical analysis

Statistical analysis was performed using SPSS (version 21st edition).⁸ Two-way ANOVA was used to evaluate the effect of material, treatment and their interaction on micro shear bond strength. One-way ANOVA followed by Tukey HSD Post hoc test were used to evaluate the effect of material with each treatment. Student T —Test was used to assess the effect of material within each treatment at P value = 0.05.

3. Results

Table 2 showed that, both the type of material and the surface treatment applied had a statistically significant effect. Additionally, the interaction between them had a statistically significant effect.

Regardless of the surface treatment, Lava Ultimate RNC showed statistically significant higher microshear bond strength compared to IPS e.max lithium disilicate ceramic.

Regardless of the material used, the group (DBMH) subgroup showed statistically significant higher µSBS value compared to all treatment groups, except for group (CNMH), where the increase in µSBS was insignificant (Table 3).

In Table 4, Lava Ultimate RNC, (DBMH) subgroup showed statistically significant higher µSBS value compared to (CJSBU) subgroup, and statistically non-significant with the other groups of treatments.

While for IPS e.max, (DBMH) subgroup showed statistically significant higher µSBS value compared to other groups of treatments and statistically non-significant with (CNMH) group.

Please cite this article in press as: Wahsh MM, Ghallab OH, Influence of different surface treatments on microshear bond strength of repair resin composite to two CAD/CAM esthetic restorative materials, Tanta Dental Journal (2015), http://dx.doi.org/10.1016/j.tdj.2015.06.001

⁵ Radii plus, SDI dental limited, Australia.
⁶ Model LRX-plus; Lloyd Instruments Ltd., Fareham, UK.
⁷ Nexygen-MT Lloyd Instruments Ltd., Fareham, UK.
⁸ IBM Corporation, New York, USA.
On comparing the two tested materials within each treatment, Lava Ultimate RNC subgroups (CNSBU), (DBSBU) and (CJSBU) showed statistically significant higher $\mu$SBS values compared to the same IPS e.max subgroups. There was no statistically significant difference between the subgroups (CNMH), (DBMH) and (CJMH) for both tested materials.

4. Discussion

Replacement of defective restorations may not necessarily be the most practical solution because it weakens the tooth structure, in addition to further trauma to the pulp. Repair of a failed restoration, when possible and appropriate, offers many merits over replacement as:

- reduced chair time, lower cost and ease of application [25,26].

The integrity of the bond between the substrate and the repair resin composite determines the clinical success of the ceramic repair system. This bond is achieved either by chemical or mechanical pre-treatment of the ceramic surface or by combination of both [19,25]. The selection of diamond burs in this study as a mechanical treatment to enhance the bond between repair resin composite and ceramics [28], as it is an easily applicable and cost-effective method for abrasive conditioning of the ceramic surface [1]. The surface roughening which result from Cojet system is thought to provide a large surface area for increased wettability, micro-retentive structure for micromechanical luting of the bonding material, thus enhancing the bond strength [8,25,28,29].

Single bond Universal adhesive system (SBU) used in this study, is claimed to have a unique chemistry containing silane coupling agent and MDP in addition to other components which allows the adhesive to chemically bond to glass ceramic surfaces without using a separate ceramic primer [30]. Ceramic repair system with separate silane step (Monobond plus + Heliobond adhesive) was also selected for the purpose of comparison representing a commonly used repair modality.

Tensile and shear bond strength tests are the most commonly used by the researchers to evaluate the adhesion properties of the adhesive systems. For evaluation of the bond strength between repair resin composite and ceramic surface, microshear bond strength test was used in this study as the specimens might be easily prepared [27] and to avoid the cohesive fracture on numerous samples [31].

Regarding the effect of surface treatment, the results of the current study revealed that the subgroups treated with (DBMH) showed higher statistically significant microshear bond strength value ($12.4 \pm 4.5$ MPa) regardless of the material used. The use of diamond

| Source                           | Type III sum of squares | df | Mean square | F      | Sig.  |
|----------------------------------|-------------------------|----|-------------|--------|-------|
| Corrected model                  | 2516.098$^a$            | 11 | 228.736     | 14.113 | .000  |
| Intercept                        | 10551.142               | 1  | 10551.142   | 651.007| .000  |
| Material                         | 277.107                 | 1  | 277.107     | 17.098 | .000  |
| Treatment                        | 1644.278                | 5  | 328.856     | 20.290 | .000  |
| Material*Treatment               | 598.080                 | 5  | 119.16      | 7.380  | .000  |
| Error                            | 2739.055                | 169| 16.207      |        |       |
| Total                            | 15880.135               | 180|             |        |       |
| Corrected total                  | 5255.153                |    |             |        |       |

$^a$ R squared = .479 (Adjusted R squared = .445).

Table 3
Means ± SD for the effect of different surface treatments regardless the materials used on $\mu$SBS in MPa.

| Subgroup | Symbol | Mean ± SD |
|----------|--------|-----------|
| DBMH     | A      | 12.4 ± 4.5|
| CNMH     | AB     | 10.5 ± 4.4|
| CJMH     | BC     | 7.5 ± 4.2 |
| CNSBU    | CD     | 6.1 ± 6.1 |
| DBSBU    | CD     | 5.7 ± 5.0 |
| CJSBU    | D      | 3.5 ± 2.2 |

Means with the same subscript small letters in the same column and the same superscript capital letters in the same row are not statistically significant at $P = 0.05$.

On comparing the two tested materials within each treatment, Lava Ultimate RNC subgroups (CNSBU), (DBSBU) and (CJSBU) showed statistically significant higher $\mu$SBS values compared to the same IPS e.max subgroups.

There was no statistically significant difference between the subgroups (CNMH), (DBMH) and (CJMH) for both tested materials.

4. Discussion

Replacement of defective restorations may not necessarily be the most practical solution because it weakens the tooth structure, in addition to further trauma to the pulp. Repair of a failed restoration, when possible and appropriate, offers many merits over replacement as:
bur might present a more irregular and rough surface than those subjected to abrasion with silica coated particles (CJMH) (7.5 ± 4.2 MPa). This was in accordance with Phoenix and Shen [32], who stated that the mechanical interlocking has an effective influence on the formation and maintenance of ceramic-to-resin bond [4].

For RNC material, the remarkable increase in μSBS value of (DBMH) subgroup (12.6 ± 6.0 MPa) compared to the (CNMH) subgroup (8.8 ± 4.6 MPa) proved the efficiency of created retentive surface with diamond bur that might have exposed the hydroxyl groups of RNC (high silica content) which was compatible with the silane. Thus it led to increase the wettability and resin impregnation of Heliobond adhesive into the micro-retentive areas [9,25]. While IPS e.max subgroups treated with (CJMH) (6.3 ± 4.5 MPa) and (CJSBU) (2.7 ± 1.7 MPa) showed statistically significant lower μSBS values compared to the subgroup treated with (DBMH) (12.6 ± 5.0 MPa). This may denote that Cojet system could not roughen the surface to provide reliable bond strength between repair resin composite and ceramic. Silica coating is not the choice of treatment for silica-based ceramics [4,33] because the silica content of lithium disilicate ceramics is approximately (57–80%) as purported by the manufacturer, which does not need added silica coating as the integral silica content is adequate for the reliable chemical bond. Also Rathke et al. [34] found that the use of silica coating had no advantage over common bonding systems when used in repair of microhybrid composite.

However, Frankenberger et al., [35] stated that, fatigue resistance of the repaired resin composite after using Cojet system or carbide burs used for roughening gave similar results. While Bouschlicher et al. [36] and Zaghloul et al. [29] reported better results when using Cojet system.

Regardless of the substrate material, the results of this study showed that all the groups treated with (SBU) adhesive system had significant lower μSBS values compared to the groups treated with separate silane step. Bonding to ceramic substrate seems to be dependent on the presence of silica on their surfaces. Silica which is well incorporated to the ceramic surface has a great affinity to silane coupling agent [3,4,27]. The decrease in μSBS values suggested that, the silane might not be effective to form siloxane bridges with the substrate surfaces [2,10,14,18,21]. In turn, it impaired the wettability and resin impregnation leading to failure of bond. This may be due to the lower silane concentration in (SBU) than that of Monobond plus. There should be balance between the amount of the hydroxyl groups of inorganic substrates exposed and the hydrolysable functional groups present in the silanes [37]. This balance might have been lost, which might have led to the presence of unreacted hydrolysable functional groups with existence of weak siloxane bond. These results were in accordance with Zaghloul et al. [29] who showed that, the additional silanization step enhanced the chemical bonding to the exposed hydroxyl groups and wettability with the resin composite.

An overall comparison between the two ceramics repaired; Lava Ultimate RNC showed statistically higher significant μSBS values with the groups treated with (SBU) compared to the same treated groups of IPS e.max lithium disilicate ceramic. This may be due to the difference in micro-structure of the tested substrate materials. When treated RNC groups with (SBU) adhesive which contains MDP phosphate monomer that chemically reacts to zirconia in the RNC providing a higher bond specially after mechanical roughening of the latter's surface [6]. Borges et al., [3] concluded that, the efficiency of the surface treatment is highly dependent on the composition of the ceramic substrate rather than the treatment itself. In addition to the different properties of flexural strength, resilience that might allow the RNC to have higher μSBS values before failure [6,8]. These findings suggested that, the effect of surface treatment on μSBS of repair composite to CAD/CAM restoratives is material dependent.

5. Conclusions

Within the limitations of this study the following conclusions could be drawn for both tested materials:

1. The effect of surface treatment on μSBS of the repair resin composite is material dependent.
2. Combination of the abrasive action of diamond bur and adhesive with separate silane treatment had a synergistic influence on μSBS of the repair resin composite under the current circumstances.

Disclosure statement

It is hereby stated that each author has no conflict of interest.

References

[1] Leibrock A, Degenhart M, Rosentritt M, Handel G. In vitro study of the effect of thermo- and loading-cycling on the bond strength of porcelain repair systems. J oral Rehab 1999;26:130–7.
Please cite this article in press as: Walsh MM, Ghallab OH. Influence of different surface treatments on microshear bond strength of repair resin composite to two CAD/CAM esthetic restorative materials, Tanta Dental Journal (2015). http://dx.doi.org/10.1016/j.tdj.2015.06.001