

## **BONDING OF ZIRCONIA SUPERSTRUCTURE TO ONE-PIECE TITANIUM AND ZIRCONIA IMPLANTS WITH DIFFERENT LUTING AGENTS**

Mahy H. Hassouna\* and Lamia Dawood\*\*

### **ABSTRACT**

**Purpose:** This *in-Vitro* research was performed to determine how zirconia and titanium one-piece implants are bonded to zirconia superstructure crown using different luting cements.

**Materials and methods:** sixty-four zirconia ceramic superstructure (ceramill zolid) were bonded to thirty-two one-piece titanium implants and thirty-two one-piece zirconia implants, using four different luting agents: group GI: glass-ionomer cement, group RG: resin-modified G.I, group SA: self-adhesive resin cement and group CR: conventional resin cement. Subsequently to thermocycling (5°C to 55°C water over a 15,000-cycle span, with a dwell time of 15 seconds), the crowns were detached vertically (pull-out test) with a Universal Testing machine. Post Hoc Tukey test was used to detect pairs of comparisons. One-way ANOVA test was used for statistical analysis.

**Results:** The titanium and zirconia groups differed significantly ( $P < .001$ ). With titanium specimens the average bond strength values were higher than in the zirconia specimens. The highest reported value was for the conventional resin cement group (CR) of titanium implants (630.38±83.0 N) while the lowest one was reported for glass ionomer cement (GI) of zirconia implants (129.64±15.94 N).

**Conclusions:** The proper selection of a luting agent when bonding zirconia ceramic superstructure onto one-piece zirconia implants is important. Conventional resin cements and self-adhesive cements could be used for bonding of zirconia superstructure for both titanium and zirconia implants.

**KEYWORDS:** One-piece implant, Bonding, Adhesive cements, Titanium, Zirconia.

\* Assistant Professor, Fixed Prosthodontics Dept., Faculty of Dentistry, Mansoura University, and Delta University for Science and Technology, Egypt.

\*\*Associate Professor, Fixed Prosthodontics Dept., Faculty of Dentistry, Mansoura University, and Delta University for Science and Technology, Egypt.

## INTRODUCTION

Over the last decades titanium implants are considered the gold standard material in oral implantology.<sup>1</sup> Titanium has positive characteristics as biocompatibility, great mechanical strength and high survival rate clinically, however, the gray color of titanium may be a challenging in esthetic area.<sup>2</sup> Mainly failure of titanium implants may be a consequence of considerable titanium ions release, which was proven by studies on animals where titanium particles were found in macrophages and local lymph nodes.<sup>3-5</sup> Monolithic zirconia and yttria-stabilized tetragonal zirconia polycrystal ceramics (Y-TZP) was used as restorative materials to improve esthetics instead of metallic ones, as most patients rejected metal restorations and inquired for esthetic replacement of missed teeth. Nevertheless, zirconia was used recently for fabrication of dental implants. Zirconia was reported to have comparable biological properties and equivalent degree of survival parallel to the standard titanium alloy (Ti-6Al-4V) implants.<sup>6,7</sup> In terms of its biological and mechanical properties, low thermal conductivity, high fracture toughness (high elastic module),<sup>8</sup> and low bacterial adhesion<sup>9</sup> in addition to high esthetic, zirconia has shown to be superior to other ceramic materials. Even with small implant diameter, one-piece zirconia dental implants showed clinically acceptable mechanical properties.<sup>10</sup>

Zirconia dental implants are situated in anterior esthetic zone in cases of traditional healed edentulous position,<sup>11</sup> in immediate extraction place, and immediate temporization.<sup>12</sup> Several investigations established that osseointegration of zirconia implants is not lower in comparison to titanium implants with well-recognized coarse surfaces.<sup>1,13,14</sup> Most of currently available zirconia implants are one-piece system which exhibited a great degree of survival, constant mucosal margin levels and marginal bone integrity after 5 years in utility.<sup>15</sup> Although long-term clinical studies

are rare, the initial studies show survival rate of 95.6% after one year<sup>16</sup> and 95% five years follow up.<sup>17</sup> Nowadays, the usage of zirconia implants is expanded by the increased needs for metal-free implants and prostheses, and positive outcomes from clinical and preclinical investigations.<sup>18</sup> So, most of dental specialists expected that in the close future, zirconia oral implants will be concurrent with titanium implants.<sup>19</sup>

One-piece implant design was invented to overcome the defective structure strength present in the two-piece design.<sup>16</sup> The unique design of this implant provides several advantages such as high mechanical strength, more simple surgical technique, no microgap between implant and abutment, and similar to the conventional crown and bridge procedures.<sup>20</sup>

Retention of the superstructure to one-piece implant depends on several factors, such as implant form, cement type and surface treatments.<sup>21-23</sup> As zirconia has high strength,<sup>8</sup> it can be bonded using zinc phosphate cement, glass ionomer cement or resin modified glass ionomer cement.<sup>24</sup> Nonetheless, the retention force of restorations could be improved when using resin cements.<sup>25</sup> Additional problems existed for sufficient bonding if the prosthetic material is zirconia, this is due to its high crystalline content and absence of glass,<sup>26</sup> which make it resistant to hydrofluoric acid etching.<sup>27</sup> Zirconia needs alternative surface treatments compared to other glass-matrix ceramics because retention-loss is an important problem affecting the clinical success rate.<sup>28,29</sup> Air-borne particle abrasion was used to improve the mechanical bond strengths of zirconia.<sup>30</sup>

Silane coupling is an alternative technique of enhancing bonding strength via chemical way but, it may be ineffective with zirconia as it does not contain silica.<sup>31</sup> The strength of bonding to zirconia increased after silica coating followed by silane application;<sup>32</sup> however, after thermocycling it declined in strength.<sup>33</sup>

It was concluded that the usage of primers or resin cements containing 10-methacryloyloxydecyl dihydrogenphosphate (MDP) monomer subsequent to air-borne particle abrasion improved the adhesion to zirconia.<sup>30,34</sup> MDP is an acidic phosphate monomer, which was initially used to adhere to metal oxides then, it was used for zirconia.<sup>35</sup> Its affinity to metal oxides and zirconia surface is simply shielded with a passive oxide layer, which makes zirconia like metal, as a result it can be used to improve the adhesion of resin cement to zirconia.<sup>36</sup>

Different techniques were used to calculate bonding force between materials either macro or micro specimens: tensile tests, shear tests, or push-out tests.<sup>37,38</sup> Different bonding methods to zirconia ceramic and titanium have been stated,<sup>39</sup> nevertheless, few researches were done comparing retention of zirconia crowns to both types of one piece implants using different types of luting cements.<sup>40-42</sup> So, this study aimed to estimate the impact of various luting agents on the retention force of zirconia ceramics to different one-piece implants. The postulated null hypotheses were that: (1) Cement type may influence the pull-out retention values; (2) Type of implant also might influence the pull-out retention values.

## MATERIALS AND METHODS

In the current study, 32 one-piece titanium implants (T) (Bredent® whiteSKY) and 32 one-piece zirconia implants (Z) (Bredent® whiteSKY) with 4.5 mm diameter and 10 mm length were used. The endosseous part of the implants were inserted inside transparent epoxy resin blocks (Kemapoxy 150, CMB chemicals, Egypt) using a specially designed centralizing device to hold the implant during fixation (Figure 1). It consisted of cylindrical Teflon mold (2.5 cm height and 2 cm diameter) fixed on a copper ring with a hole in its center with the same dimension of the implant head 3.4 mm

diameter and 6 mm length, two inner separate halves of Teflon ring were fixed inside the outer Teflon ring for easy separation. This device was used to guide the positioning of the implant within the center of the epoxy resin block.

Implants were scanned with an optical scanner (Ceramill map400, Amman GIRRbach, Germany). Using the special software (Ceramill Mind design software), a mandibular right first premolar crown was designed with a bar on the occlusal surface to simplify its removal after cementation.<sup>42</sup> A cement space of 50  $\mu\text{m}$  was planned for cementation, starting 1 mm away from the margins. Sixty-four zirconia ceramic crowns (ceramill zolid, Amman GIRRbach, Germany) were dry milled using the 5-axis milling machine (Ceramill Coolstream, Amman GIRRbach, Germany). Sintering of the crowns was done according to the manufacturer's recommendations using the sintering furnace (Programat P500, Ivoclar Vivadent, Liechtenstein). The ceramic glaze (Ceramill glaze Amman GIRRbach, Germany) was applied over the crowns and fired according to the manufacturer's recommendations. The fitting surface of all crowns was subjected to air-borne particle abrasion with 50  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  at right angle to the surface at distance of 10 mm and 2 bars pressure for 20 seconds. All implants and crowns were ultrasonically cleaned for 4 minutes in a 96% ethanol ultrasonic bath (TPC-15; Telsonic, Bronschhofen, Switzerland).<sup>40,43</sup> Retention of zirconia crowns to titanium and zirconia implants was examined using four different types of cements; group GI: Glass-ionomer cement (Ketac Cem, 3M-ESPE, USA), group RG: Resin-modified glass ionomer (RelyX Luting Plus, 3M-ESPE, USA), group SA: Self-adhesive resin cement (RelyX U200, 3M-ESPE, USA), and group CR: Conventional resin cement (RelyX Ultimate, 3M-ESPE, USA). With conventional resin cement groups (CR), before cementation, a universal primer containing 10-MDP (Z-PRIME Plus, Bisco Inc, Schaumburg, USA) was coated by disposable brush inside the fitting surface of the crowns, and titanium and zirconia implant

abutment portions according to manufactures' recommendations. The primer was left on the bonding surfaces for 60 seconds till reaction occurs, then, the specimens were dried using oil-free air for 5 seconds to remove any excess primer.

Each zirconia crown in each group was filled with its corresponding cement and placed on its implant under 3 kg constant load applied perpendicular to the long axis of the specimen for 10 minutes using a cementation device to ensure good seating. Light curing was used for 20 seconds (Valo Cordless, Ultradent, South Jordan, UT, USA) for resin cement groups. Subsequently, the specimens were incubated in 37°C water bath for 24 hours. After water storage, specimens were thermocycled in 5°C to 55°C water over a 15,000-cycle span, with a dwell time of 15 seconds (THE-100 SD Mechatronic Thermocycler, Germany).

#### Pull-out retention test

Each crown was attached to the upper moving holder of the Universal Testing machine (Instron, Model 3345, Norwood, MA, USA) through the occlusal bar (Figure 2) and subjected to vertically dislodging force along the apico-occlusal axis using a load cell of 5kN at a crosshead speed of 0.5 mm/min. till crown debonding occurred and load dropped. The maximum retention force at dislodgment (in Newton) was recorded.

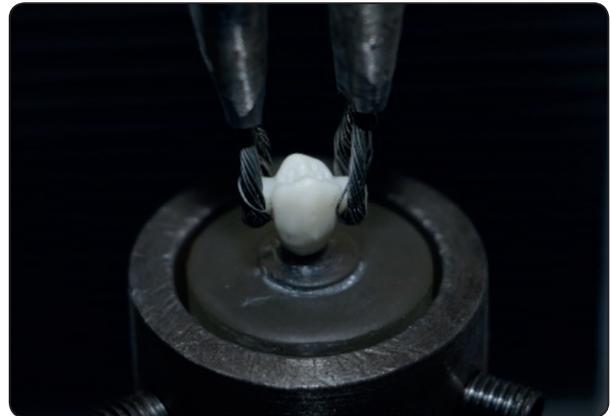


Fig. (2): Crown debonding by the Universal Testing machine.

Assessment of the debonding failure modes was done by optical examination of the abutments and crowns using a digital stereomicroscope at X25 magnification (Olympus Model SZ2-ILST, Japan) with a corresponding digital camera and software.

Failure mode was recorded with the following parameters:

- I. Adhesive failure: If less than 25 % of cement content was on the surface of the abutment.
- II. Cohesive failure: If more than 75% of the cement content is on the surface of the abutment.
- III. Mixed (adhesive/cohesive) failure: If less cement material was present on the surface of the abutment.<sup>24</sup>

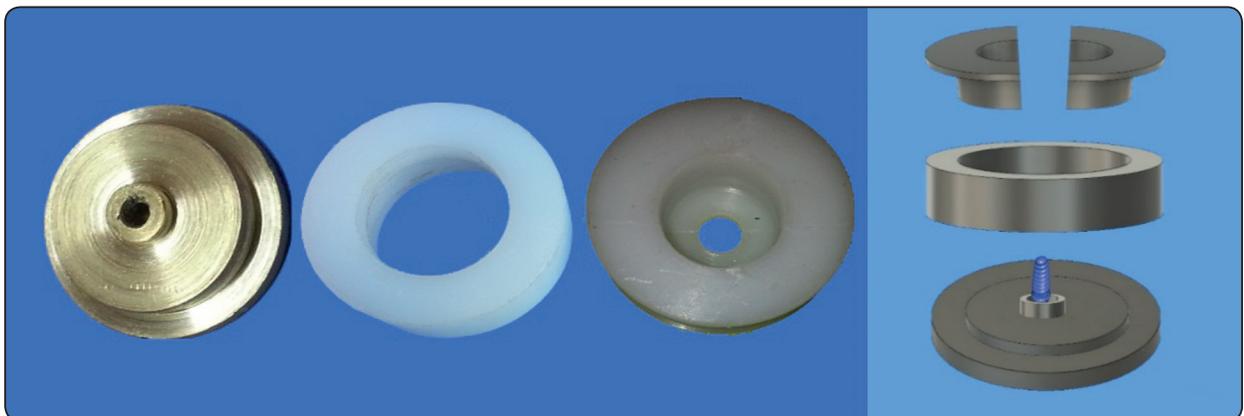


Fig. (1): Specially designed centralizing device.

**Statistical Analysis**

The statistical analysis of data was done using IBM SPSS Corp. Released 2013 (IBM SPSS Statistics for Windows, Version 22.0. Armonk, NY: IBM Corp.). Study of the data was done to test the significant difference between groups. Analysis of data was completed in three steps; two-way ANOVA test was used to detect impact of two independent factors on dependent continuous parametric variable. One-way ANOVA was conducted for the comparison between all groups. Tukey test for multiple comparisons was also performed and revealed statistically significant difference among groups. Student *t*-test was also used to compare each two independent groups.

**RESULTS**

The individual retention means and their standard deviations for all tested groups are summarized in Table 1. The maximum retention force at the time of debonding reported for titanium implants was for the conventional resin cement group (CR) (630.38±83.0 N) while the lowest one was reported for glass ionomer cement (GI) (191.25±22.33 N).

For zirconia implants, the highest reported value was for the conventional resin cement group (CR) (443.13±67.77 N) while the lowest one was reported for glass ionomer cement (GI) (129.64±15.94 N). The type of cement material significantly affected the retention force values for all implants (F= 142.990, P<.001) (Figure 3).

Two-way ANOVA revealed a statistical significant difference between both implant material type (titanium & zirconia), (F=79.33, P < .001) and between cement types(GI, RG, SA&CR) (F= 142.990, P<.001). One-way ANOVA revealed that the mean bond strength values of the titanium specimens were higher than those of the zirconia specimens. Tukey test denoted that there was no significant difference between RC and SA groups. Student *t*-test revealed that CR group displayed a higher mean bond strength than the others on both titanium and zirconia implants (t=4.94, P<.001). Glass ionomer group displayed the lowest mean bond strength than the others on both T and Z groups (t=6.35, P<.001).

Failure mode was assessed, and the result was summarized in Table 2. The most observed type was the mixed failure in both implants. Furthermore, with titanium implants cohesive failure (Figure 4) was more observed than with zirconia implants.

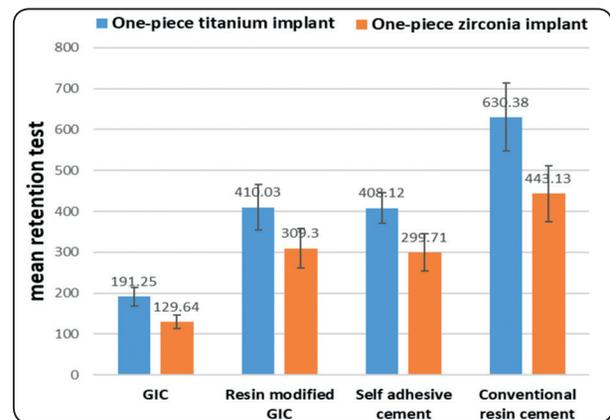


Fig. (3) Statistical summary data for pull-out retention.

TABLE (1): Means and standard deviations (in N) for bond strength of all groups (One way ANOVA and Post Hoc Tukey tests)

	GI group	RG group	SA group	CR group	test of sig.
<b>Titanium implant</b>	191.25±22.33	410.03±55.71 <sup>a</sup>	408.12±37.29 <sup>a</sup>	630.38±83.0	F=86.55 P<0.001*
<b>Zirconia implant</b>	129.64±15.94	309.30±47.87 <sup>a</sup>	299.71±46.16 <sup>a</sup>	443.13±67.77	F=56.98 P<0.001*

*Post Hoc Tukey test, the same superscripts indicate insignificant different pairs of values in same row.*

TABLE (2): Distribution of the failure mode among the studied groups.

Type	Titanium implant				Test of sig.	Zirconia implant				Test of Sig.
	GI	RG	SA	CR		GI	RG	SA	CR	
I	2 (25.0%)	2 (25.0%)	1 (12.5%)	2 (25.0%)	P=0.171	3 (37.5%)	2 (25.0%)	2 (25.0%)	1 (12.5%)	P=0.082
II	2 (25.0%)	3 (37.5%)	3 (37.5%)	4 (50.0%)		0 (0%)	2 (25.0%)	1 (12.5%)	3 (37.5%)	
III	4 (50.0%)	3 (37.5%)	4 (50.0%)	2 (25.0%)		5 (62.5%)	4 (50.0%)	5 (62.5%)	4 (50.0%)	

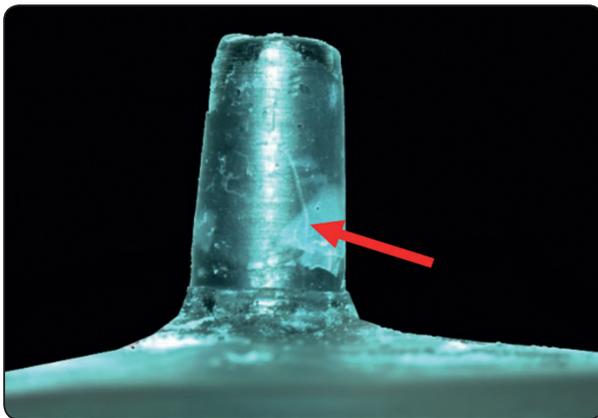


Fig. (4): Cohesive failure in titanium implants.

## DISCUSSION

One-piece implant design was used in this study to evaluate the pull-out retention with crown restoration as it represents an interesting option with the increased popularity of immediate loading.<sup>44</sup> Zirconia implants were introduced long time ago<sup>45</sup> to solve the problems of titanium implants such as dark color of titanium and its hazard of a grayish shadow of the implant through the peri-implant gingiva, mainly in the esthetic zone. Yttria-stabilized tetragonal zirconia polycrystal was used in this study to fabricate the superstructure crowns restoring the one-piece implants as it is considered the recommended restorative material

because of its high biocompatibility, mechanical properties and high esthetic properties.<sup>26</sup> Materials used for cementation of indirect restorations were classified classically as temporary or permanent luting materials; the latter includes zinc phosphate, glass-ionomer, and resin cements.<sup>29</sup> In this study, Glass ionomer cement was used in comparison with resin cements to evaluate bonding with zirconia crowns as it achieved higher bond strength as a conventional cement than zinc phosphate and zinc polycarboxylate cement in previous studies.<sup>40,42,46</sup>

Based on the findings of this study, the first hypothesis that cement type may influence the pull-out retention values was accepted. The cement type influenced the pull-out retention values of crowns cemented to both titanium and zirconia implants as both conventional and adhesive resin cements possessed higher retention force than did glass ionomer and resin modified glass ionomer ones. This may be attributed to the action of phosphate monomer present in the composition of the self-adhesive resin cement investigated in this analysis and the application of adhesive primer containing MDP with conventional resin cement. This monomer has a longer  $-(CH_2)_2$  chain than other monomers of phosphate acid,<sup>47</sup> which produces higher bond strength values to both implants than other cements.<sup>48</sup> These results were in agreement

with earlier investigations<sup>29,49</sup> which have also confirmed extra promising adhesive bond force of conventional resin cement comparative to self-adhesive sorts to Y-TZP, particularly after artificial aging. However, it disagreed with Blender et al<sup>40</sup> who reported that self-adhesive resin cement has higher retention force than conventional resin cement, this may be due to the use of different types of cements with zirconia implants only.

In the present study, the second null hypothesis was also accepted, as the titanium implants possessed higher bond strength values than did the zirconia implants which agreed with other studies<sup>50,51</sup>. These results may be attributed to the surface hardness of the zirconia material, which is much higher comparing with that of titanium, therefore, surface treatments have less effect on the zirconia surface.<sup>52</sup>

Although it is problematic to accomplish a satisfactory bond to zirconia<sup>27</sup> the tested self-adhesive and conventional resin cements exhibited retentive forces  $299.71 \pm 46.16$  N and  $443.13 \pm 67.77$  N respectively. Resin modified glass ionomer cement produced relative retentive value near to those of self-adhesive resin cements despite it lacks monomers with phosphate group. This may be due to the micromechanical holding in the retentive irregularities of zirconia, or it may result from the vacuum effect which is produced by the close-fitting of the polymerized cement layer to the implant. In this study, glass ionomer cement has slightly lower retention values as it depends on carboxyl groups which make weak bond with zirconia, as previously stated.<sup>41</sup> These findings were agreed with the results of Sellers et al<sup>53</sup> although they used lithium disilicate crowns bonded to zirconia abutments. This was refused by other study<sup>40</sup> which compared different types of luting agents for cementation of zirconia crowns to zirconia implant and the bond strengths were ranked as the following: glass ionomer cements > self-adhesive resin-based luting agents > resin-based adhesive luting agents > phosphate cement > polycarboxylate cement > temporary cements. This

difference may be due to the use of different types of cements with zirconia implants only.

Bonding of superstructure crown to implant depends on three mechanisms, in addition to preparation method and crown fit. Firstly, mechanically through micro-retentive undercuts on the roughened surfaces of crown and implant. Secondly, a close-fitting of the superstructure crown to the implant abutment which produces a vacuum effect. Thirdly, by chemical bonding of the cement to the ceramic through hydroxyl groups<sup>54</sup> or phosphate groups.<sup>55</sup>

Failure mode was easily determined by evaluation of the de-bonded surfaces, mixed type of failure was commonly observed in this current study. Nonetheless, it was observed that adhesive mode of failure decreased with conventional resin cements. Also, it was detected that most of titanium groups displayed cohesive failure which pointed to a more stable attachment of cement material to the titanium implants comparable with zirconia implants which agreed with other studies.<sup>40,41,50</sup>

Subsequently, the general concurrence with investigation findings allowed determining that the ordering of the cements in the existing study is valid. At that point, the present and earlier studies offer a scenario of the bonding manners of different cements to one-piece implants that may be used as a guide until clinical studies are accompanied.

## CONCLUSIONS

The following conclusions have been drawn within the restrictions of the present study:

1. Adhesive resin cements produced the greatest bond strength with both titanium and zirconia implants than did glass ionomer cements and self-adhesive one, moreover, the conventional glass ionomer cement showed the lowest bond strength.
2. Titanium implants had greater bonding strength with zirconia superstructure than did zirconia implants regardless the type of luting cement.

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