

# The effect of titanium dioxide nanoparticles on the bond strength between maxillofacial silicone and acrylic core

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## ABSTRACT

**Objectives.** This study was aiming to evaluate the influence of Titanium dioxide nanoparticles addition on the bonding performance of platinum primer (G611) between maxillofacial silicone and acrylic substrate.

**Materials and methods.** 180 samples were divided on two tests (shear and peel bond strength) and they were divided into two main groups (cold cure, and 3D printed) acrylic resins. Each group was subdivided into 5 subgroups (N=9) according to the concentration of nano-filler addition (0%, 0.5%, 1%, 1.5% and 2%) by weight. The data obtained were evaluated by one-way ANOVA and Tukey's post hoc tests at  $P < 0.05$ .

**Results.** Statistical analysis revealed (0.5% and 1%) concentrations had significantly the highest bond strength in both groups, whereas (2%) had the lowest bond strength in both groups.

**Conclusion.** Reinforcement of the platinum primer G611 with Titanium dioxide (TiO<sub>2</sub>) nanoparticles (1%) concentration by weight resulted in a significant improvement in the bonding performance of the platinum primer G611 larger concentrations (1.5%, and 2%) resulted in declining bonding strength.

**Keywords:** silicone elastomer, maxillofacial prosthesis, acrylic resin, nanoparticles, 3-D printing

## INTRODUCTION

A maxillofacial appliance is a prosthetic that fills in the gaps left by congenital or acquired abnormalities brought on by trauma or surgical procedures (mainly because of neoplasms). In general, maxillofacial prostheses have been held in place by spectacles, anatomical undercuts, and medical-grade adhesives. But the development of osseointegrated implants provided a better method for maintaining maxillofacial prostheses [1-6]. The housing is frequently required for an implant-retained facial prosthesis in order to keep attachments in the proper place. This housing is typically made of acrylic resin materials [5,7] Although acrylic resins and maxillofacial elastomers are all polymers, their chemical composition is different, for this reason a bonding agent is frequently required to enhance ad-

hesion between the two materials [8]. These adhesives prepare the substrates by etching them or encouraging covalent and hydrogen bonds. They also increase the substrate's wettability by enabling the polymeric substances to infiltrate the resin surface [9]. Although silicone elastomers have improved greatly in terms of their physical and mechanical characteristics, rubber delamination from the retaining substrate is still a persistent issue [10]. Previous studies have been conducted to evaluate the best method for increasing this bond strength, using shear and peel forces tests in their evaluation. These tests are nearest to clinical circumstances to measure the bond strength between the acrylic substrate and the facial elastomers [9,11,12]. The lateral displacement of the prosthesis is believed to be caused by forces with a horizontal component, the peel strength test is thought to simulate these displacing

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forces [13]. Many of these studies were focusing on the alteration of the topography of the substrate surface, either mechanically or by adding various types of primers that contain different kinds of solvents [7,14–16]. However, little attention has been given for the alteration of the physical and mechanical characteristics of the liquid primers by adding nano-fillers to increase their bonding capacity. For this reason, the aim of this study was to assess the influence of adding Titanium dioxide nano-fillers to the bonding agent of the platinum primer used for bonding platinum cured facial elastomers to an acrylic substrate. Previous studies showed that the addition of filler particles to various adhesive systems could alter their chemical or rheological properties of these adhesive liquids [17,18]. Nanoparticles in the adhesive systems can raise the bond strength by different means. Fillers could work by blocking and exhausting the crack propagation at the interface of the adhesive and substrate. Consequently, they could alter the energy level required for adhesive-substrate interface failure [19–21]. Titanium dioxide nanoparticles have excellent mechanical characteristics. The unique photoactive properties of (TiO<sub>2</sub>) nanoparticles and their excellent physical and mechanical characteristics made them an ideal additive to improve the effectiveness of polymeric and adhesive materials [22,23].

## MATERIALS AND METHODS

The effect of adding titanium dioxide nanoparticles (Skyspring Nanomaterials, Inc., Houston, USA) to the G611 platinum primer (Principality Medical limited, Newport, UK) on the bonding performance of the primer was evaluated using shear and peel bond strength tests to measure the bond strength between both self-cure acrylic resins (Veracril, Newstetic Co., Colombia), 3D printed denture base resin (Senertek, Senerlabs, Turkey) and a Cosmesil facial silicone (Cosmesil M511; Principality Medical limited, Newport, UK).

Titanium dioxide nanoparticles (0.0038g, 0.0076g, 0.0114g, and 0.0152g) using digital balance (with four digits precision; PG 503-S MonoBloc inside, Mettler Toledo Ltd, Switzerland) were added to (1ml) of the G611 platinum primer to form nano-filled liquid. The concentration percentages of the nanoparticles were 0.5%, 1%, 1.5%, and 2% by mass respectively. To ensure optimal distribution of the nanoparticles in the liquid primer, one hundred-eighty samples were fabricated, and they were divided to two tests (shear and peel bond tests). Ninety samples for each test were divided into cold cure resin and 3D printed resin groups which were subdivided to five subgroups (0% addition as control group), and four groups followed the concentration abovementioned (0.5%, 1%, 1.5%, and 2%). One

hundred-eighty samples were fabricated, and they were divided to two tests (shear and peel bond tests). Ninety samples for each test were divided into cold cure resin and 3D printed resin groups which were subdivided to five subgroups (0% addition as control group), and four groups followed the abovementioned concentrations (0.5%, 1%, 1.5%, and 2%). The surfaces of the samples were cleaned with acetone liquid. The G611 platinum primer were added using a brush, the primer was left to dry for 30 minutes according the manufacturer's recommendations. The facial silicone M511 (part A) was mixed with part B catalyst (ratio of 10:1), and left for 24 hours at room temperature for curing.

The fabrication method of the samples used for shear bond test was described in previous studies as follows [5,24]. An auto-polymerizing acrylic resin was mixed and packed inside PVC tubes (12.5mm external diameter, 11mm internal diameter, and 20mm height). The 3D printed resin samples were fabricated using 3D printing machine (LD-002H, Creality Co., Shenzhen, China) a disk with 10mm diameter, and 3mm thickness were fabricated and fixed at the top of the PVC tubes with aid of cold cure resin. After completing the polymerization, and 3D-printing of samples, the surface were finished and the primer filled with nanoparticles were applied on the substrate surface. Plastic rings were fixed on the top of each PVC tube with 10mm external diameter, 8mm internal diameter, and 3mm height and packed with facial silicone material under 10Kg of weight. The specimens were situated in the universal testing device (GESTER International Co., Quanzhou, China) and the shear bond strength was measured with a cross head speed 10mm/min. The shear bond strength was calculated by dividing the failure load (Newton) by the attached surface area (mm<sup>2</sup>).

For evaluation of the peel bond strength of the nano-filled modified primer a rectangular auto-polymerizing acrylic using conventional flasking procedure, and 3D printed resin samples using DLP 3D-printing method, both with (75mm length, 10mm width, 3mm thickness) were fabricated according to ASTM D 903 – 98 [2,5]. A layer of modeling wax with the same dimensions was placed over the resin one to create a room for the silicone material. After setting of the stone the twin sample was removed, and the space created was used to pack the silicone material. the primer was added as a thin layer on the area of the attachment which represents the terminal 25mm (length) and 10mm (width) from the original samples' dimensions, a separating tape was attached to the rest of the acrylic samples and the M511 silicone material was packed in the prepared flasks over the acrylic samples, pressed constantly for 24 hours at 3500psi. The peel strength of the samples was tested with the universal testing machine with crosshead speed set on 10mm/min. The

acrylic part was grasped with the lower jaw of the machine and the free end of the silicone turned upward and grasped with upper jaw. The peel strength (PS) (Newton/mm) was estimated using the following equation [13]:

$$PS = F/W (1+L/2) + 1$$

Where F is the failure force (Newton), W is the width of the sample, and L is the ratio between the stretched and non-stretched length of the silicone part. The statistical analysis was accomplished using SPSS (17) (SPSS Inc., Chicago, IL, USA), ANOVA and Tukey's tests. The significance level was defined at  $P < 0.05$ .

### RESULTS

Shear and peel bond strength were tested to assess the impact of titanium dioxide nanoparticle addition to the platinum primer G611. Table 1 shows the mean and standard deviation of the shear bond strength for the main groups of self-cured and 3D printed resin. One way ANOVA statistical test showed that there was a significant statistical difference among all concentrations added per weight (0% control, 0.5%, 1%, 1.5%, and 2%) (significance 0.000) at  $P < 0.05$ . Tukey's test for group was conducted and showed that the shear bond strength for 1% addition was the highest followed by 0.5%, 1.5%, 0% control, and 2% respectively. The mean and standard deviation of the peel bond strength is presented

in Table 2 for the two main groups. One way ANOVA and Tukey's multiple comparison tests revealed that there is a significant difference among all concentrations added 0.000 at  $P < 0.05$ , with one exception, that the difference between 0% control group and 1.5% addition had insignificant difference. The highest mean of peel strength occurred with 1% addition followed by 0.5%, 0% control, 1.5% and 2% respectively.

### DISCUSSION

The adherence of different types of materials can occur due to mechanical adhesion, micromechanical adhesion, and molecular bond. The bond created by the primers depends on the mechanical and physical strength of the primers mainly because of molecular attraction and the chemical affinity of such primers to the facial silicone and the acrylic substrate [26-28]. The bonding performance of these primers can be influenced by their ability to wet the substrate surface, so they would be capable to fill the surface micro-irregularities, thereby increasing the surface area of the attachment, in addition to the micromechanical engagement of these irregularities [29,30]. The addition of titanium dioxide nanoparticles improved the bonding ability of the platinum primer G611 employed in the investigation, as evidenced by the shear and peel bond strengths assessed in this study. For both experiments, the in-

**TABLE 1.** Means and standard deviation of the shear strength of the nano-filled modified primer G611 for the cold cure and 3D printed resin groups

		Bonding strengths (shear and peel) Mean (SD)	
Titanium dioxide Nanoparticles (%)	N	Shear bond strength of the self-cure resin (N/mm <sup>2</sup> )	Shear bond strength of the 3D printed resin (N/mm <sup>2</sup> )
Control (0%)	9	3.568 (0.244) <sup>a</sup>	2.785 (0.242) <sup>a</sup>
5%	9	6.188 (0.223) <sup>b</sup>	4.330 (0.409) <sup>b</sup>
10%	9	8.371 (0.368) <sup>c</sup>	7.234 (0.332) <sup>c</sup>
15%	9	5.128 (0.273) <sup>d</sup>	2.520 (0.489) <sup>a</sup>
20%	9	2.639 (0.419) <sup>e</sup>	1.857 (0.437) <sup>d</sup>

N: Number of samples, SD: Standard deviation. Means labeled with different letters (a-e) indicate a significant difference among the means within one column

**TABLE 2.** Means and standard deviation of the peel strength of the nano-filled modified primer G611 for the cold cure and 3D printed resin groups

		Bonding strengths (shear and peel) Mean (SD)	
Titanium dioxide Nanoparticles (%)	N	Shear bond strength of the self-cure resin (N/mm <sup>2</sup> )	Shear bond strength of the 3D printed resin (N/mm <sup>2</sup> )
Control (0%)	9	5.201(0.161) <sup>a</sup>	4.874 (0.258) <sup>a</sup>
5%	9	8.183(0.319) <sup>b</sup>	8.252 (444) <sup>b</sup>
10%	9	9.440.(0.398) <sup>c</sup>	9.733 (0.344) <sup>c</sup>
15%	9	4.874(0.607) <sup>a</sup>	4.789 (0.418) <sup>a</sup>
20%	9	3.425(0.298) <sup>d</sup>	1.651 (0.336) <sup>d</sup>

N: Number of samples, SD: Standard deviation. Means labeled with different letters (a-d) indicate a significant difference among the means within one column

creased bonding strength was at its highest at (1%) by weight concentration. However, this effect was already noticeable at lower concentrations (0.5% and 1%), but as nanoparticle concentrations were increased continuously, the bond strength began to steadily decline starting at (1.5%) concentration, reaching its lowest shear and peel bond strength levels at (2%) concentration.

The addition of nano-fillers to various adhesive agents can change their chemical and physical properties, where, these nano-fillers can absorb some of the energy of the applied force that tend to separate the silicone from the acrylic substrate, or they could increase the bonding performance by bridging the initial crack propagation by their higher ductility consequently higher energy level would be required to break the bond between the silicone and the acrylic resin [18–20]. Also the increased bonding ability of the adhesive agents by the addition of nano-fillers could be the result of decreased viscosity of the bonding agents as long as the incorporation of nano-fillers in small concentrations can increase flow properties of the liquids, consequently, this would increase the surface wettability by the bonding agent, which is an essential property for the adhesive systems that could improve their bonding performance [9,31,32]. Because of their small size particles, more surface area would be available, they can be added in small amounts to achieve significant improvement in the bonding performance. For each individual adhesive system there is an optimal amount of nanoparticles to achieve the best effect on the bonding strength [33,34]. Higher concentration of nanoparticles can result in adverse effects due to clustering of the nanoparticles before being dispersed in the liquid, the high surface energy nanoparticles make them more vulnerable to aggregate. This agglomeration increases greatly when nano-fillers are added in higher concentration, this may reduce the distance among nanoparticles making them more prone to attract to each other, resulting in weakness of the adhesive agents, and declining bonding strength of the adhesive systems [35–37].

The results of the current study are in agreement with the results of previous studies who found that the addition of nanoparticles to an adhesive liquid can promote the strength of the bonding agent which strengthens the interface between bonded materials and can influence the degree of conver-

sion of the adhesive agent and the efficiency of polymerization [18,19,33,38]. Also, the incorporation of nanoparticles to an adhesive system can decrease the viscosity of the liquid, and also can increase the bonding capacity of the bonding agent [29–32]. Using one type of primers and one type of the silicone materials, in addition to the use of one kind of acrylic resin were the important limitations in the current study, furthermore, the surface treatment of the acrylic substrate was neglected in this study, which can be evaluated in future studies as a combined effect of nano-fillers and surface treatment, which could be a promising line for achieving optimum bonding strength of the facial silicone to the acrylic substrate. Detachment of the silicone elastomer from their acrylic substrates can lead to a critical clinical problem, the addition of 1% TiO<sub>2</sub> particles can increase the bonding performance of the platinum primer used to bond the maxillofacial elastomer to the acrylic substrate. Subsequently, it will increase the service time of the maxillofacial prostheses.

## CONCLUSIONS

Reinforcement of the platinum primer G611 with Titanium dioxide (TiO<sub>2</sub>) nanoparticles in (0.5%, and 1%) concentrations by weight resulted in a significant increase in the means of both shear and peel bond strength. Indicating that Titanium dioxide nanoparticles can be added in small concentrations (1%) to achieve better bonding performance of the platinum primer G611. The addition of the (TiO<sub>2</sub>) nano-fillers in larger amounts (1.5%, and 2%) of concentration can result in declining bonding strength. The addition of (TiO<sub>2</sub>) nano-fillers in 1% concentration had the highest means of shear and peel tests, whereas, 2% concentration means of shear and peel strength indicates that the addition of (TiO<sub>2</sub>) nano-fillers can significantly compromise the bonding performance of the platinum primer used in this study.

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