

Evaluation of The Impact of Various Primers and Artificial Aging on Peel Bonding Strengths and Pattern of Bond Failure between Room Temperature Vulcanized (RTV) M511 Platinum Silicone Elastomers and Heat Polymerized PMMA

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Received February 01, 2022; Revised May 01, 2022; Accepted September 01, 2022

Abstract Aim: This laboratory research aimed to assess the impact of artificial aging and 3 dissimilar primers on peel bond strength and bond failure pattern of (RTV) M511 Platinum silicone and heat polymerizing PMMA. **Materials and Methods:** 80 heat-polymerizing PMMA samples, with dimensions 75mm×10mm×3mm were constructed, then allocated into 4 main groups I,II,III and IV based on the primers (No Primer(control), A-330-G, A 304 , and G611) utilized to adhere REF M511 Platinum silicone elastomers to acrylic resin samples. All groups were sectioned into 2 subdivisions; subgroup (a): base line (n = 10) were tested in a testing machine after 24 hours and subgroup (b) (n = 10) were tested subsequently to 360 hours of subjecting them to artificial ageing conditions. The assessments were steered at a crossheading speediness of 10 mm/min till peel and bond failure occurred. Exploration of the test readings was conducted using One-way ANOVA, Tukey's tests, and Independent t-test. **Results:** There were important influences of adhesive primers on peel resistance at both baseline and after 36h artificial aging, Peel outcomes vacillated between 0.76 and 4.75 at baseline and between 0.092 and 7.33 N/mm at after aging. At base line, (G611) Primer revealed the top peel value (4.35 N/mm), while after aging, the primer (A330-G) displayed the highest value (7.33 N/mm). Failure pattern was mainly cohesive (71.2%), and adhesive (28.8%), at baseline, while it was mainly adhesive (81.3%), and cohesive (18.7%) after 360h of artificial aging process. **Conclusion:** Best bond values achieved for peel, was achieved using the primers (G611) at the base line and (A330-G) after aging. Consequently, the most favorable combinations between silicone and primer based on bond strengths values were the combination of REF M511 Platinum silicone elastomers and G611 primer and primer A330-G.at the baseline and after aging respectively.

Keywords: silicone maxillofacial elastomers, peel bond strength, artificial aging

Cite This Article: Fahad K Alwthinani, Nouf Al Humayyani, Abdulrahman H. Alzahrani, Abdulmajeed O Alotaibi, and Mohamed Y. Abdelfattah, "Evaluation of The Impact of Various Primers and Artificial Aging on Peel Bonding Strengths and Pattern of Bond Failure between Room Temperature Vulcanized (RTV) M511 Platinum Silicone Elastomers and Heat Polymerized PMMA." *International Journal of Dental Sciences and Research*, vol. 10, no. 1 (2022): 13-19. doi: 10.12691/ijdsr-10-1-4.

1. Introduction

Maxillofacial prosthesis is defined as any prosthesis used to replace part or all of any stomatognathic and/or craniofacial structures [1]. While various substances are existing for maxillofacial applications, finding the perfect substance to replace active mobile soft tissue remains a challenge. However, silicone is noted as unique for this purpose. Silicone is favored for its biocompatibility,

durability, flexibility, and ease of manipulation. These properties make silicone suitable for creating prosthetics, implants, and other devices used in maxillofacial reconstruction. Its ability to mimic the natural texture and movement of tissues makes it particularly well-suited for applications involving living movable tissues, such as those in the face and jaw. Advancements in materials science and ongoing research may lead to the development of even more suitable materials in the future. Researchers continue to explore new materials and technologies to improve the effectiveness and safety of maxillofacial

prosthetics and reconstructions Top of Form [2]. Maxillofacial Silicone is often categorized based on the polymerization process, into: Heat-temperature Vulcanization (HTV), Room-temperature Vulcanization (RTV) Silicone. (HTV) Silicone that requires heat to initiate the vulcanization (curing) process. The material is typically mixed and then exposed to elevated temperatures to set or cure. This process results in a durable and long-lasting prosthetic. HTV silicone is known for its stability and resistance to deformation over time. RTV silicone, as the name suggests, cures at room temperature without the need for additional heat. This type of silicone is often easier to work with because it doesn't require special equipment for heating. RTV silicone is commonly used for creating facial prosthetics due to its convenience and ease of use. Both types of silicone offer advantages in terms of flexibility, durability, and biocompatibility, making them suitable for facial prosthetic applications. The choice between HTV and RTV silicone may depend on the specific requirements of the prosthetic application, the manufacturing process, and the preferences of the prosthetist or clinician creating the facial prosthesis [3]. REF M511 Platinum silicone elastomers is a commercial silicone used in medical applications; it's likely designed to meet specific requirements for medical-grade materials.

Numerous methods of retention incorporate adhesive, eyeglasses, magnet, implant, and grouping of them. The retaining matrix which is essential to retain bars, clips, or magnets in place is commonly made from self, heat, or light cured PMMA. The bonding forces between the retentive matrix and silicone elastomer are crucial for prosthesis functionality and durability.

Adequate bond strength ensures that the components remain securely attached during use, providing a serviceable and functional prosthetic solution. [4]. The challenge in achieving a durable and reliable maxillofacial prostheses can be overcome by combining advancements in material science, innovative design techniques, and careful consideration of patient and servicing protocols, minimizing, or eliminating the persisting problem of delamination in maxillofacial silicone prostheses. Additionally, Continue research and development efforts to identify or develop bond primers, explore different application techniques for bond primers to ensure uniform coverage and maximum effectiveness in promoting adhesion. Investigations of the chemical compatibility between the silicone and acrylic resin surfaces, develop surface treatment methods that improve adhesion and perform extensive durability testing to ensure that any surface treatment employed remains effective over the lifespan of the prosthesis [5]. The use of primers is crucial for achieving a strong bond between medical grade silicones and acrylic resin due to their dissimilar chemical structures. The adhesive primers play a vital role in facilitating adhesion between these dissimilar materials. Careful selection of the organic solvents in the primer formulation, that evaporate efficiently without leaving residues, can ensure compatibility with both the silicone elastomers and PMMA resin. Prioritize thorough cleaning of both the silicone and PMMA surfaces before applying the primer to remove any contaminants that might interfere with adhesion in addition to surface roughening techniques for both materials to enhance the mechanical

interlocking with the primer, also improve the longevity and reliability of maxillofacial prostheses [6]

Multifaceted approach to surface treatment that addresses both the physical and chemical aspects of adhesion can be achieved by combining activation techniques such as surface roughening, decrease surface tension, improving wettability, validating h2 bond, and etching with chemical components. This comprehensive strategy enhances the effectiveness of silane-containing primers, ensuring a strong and lasting bond between silicone elastomers and acrylic resin. Ongoing research and development in this area can lead to further refinements and innovations in adhesive technology for prosthetic applications [7].

The bond strength is the force required to break a bonded assembly with failure occurring in or near the adhesive/adherent interface [1]. The medical grade silicone peel values with polyurethane showed variation according to different factors such as the specific silicone elastomer used, the type of primer applied, and the conditioning procedures. The observation that the peel values was improved through the application of primers, regardless of the polymerizing technique or the period of primer use, suggests the significance of these material and procedural choices in achieving a strong adhesive bond [8]. While the combination of silicone and primers appears effective in enhancing peel-bond strength, the sensitivity of the bond to water exposure suggests the need for additional considerations in designing maxillofacial prostheses. Strategies to improve the water resistance of the adhesive bond may be explored to enhance the overall durability of the prosthetic devices. [9-12]. The curing conditions for condensation-cured and addition-cured silicones can impact their final properties. Differences in curing temperatures or times might contribute to variations in bond strength. Each silicone elastomer type has unique mechanical and chemical properties. The specific characteristics of REF M511 Platinum silicone elastomers, such as their modulus, flexibility, or surface energy, might align more favorably with the characteristics of the acrylic resins, leading to stronger adhesion [13,14]. The reported range of bonding forces between PMMA and medical grade silicone, varying from 0.026 MPa to 0.22 MPa, highlights the significance of primer composition in achieving effective bonding. Understanding the compatibility between the primer and silicone elastomer, and optimizing the primer formulation, accordingly, can contribute to consistent and robust adhesion in prosthetic applications. [15]

Artificial aging serves as a vital tool in materials testing, quality control, and product development, allowing for the assessment of long-term effects within a controlled and accelerated timeframe. It provides valuable insights into how products will perform over time and aids in making informed decisions about product design, materials, and durability. Artificial aging aims to accelerate the natural aging processes that items undergo over time. It is conducted to test subjects' items to aggravated conditions such as heat, humidity, oxygen, sunlight, and vibration to simulate real-world stressors. Artificial aging is conducted in a controlled laboratory setting where environmental conditions can be precisely regulated [16].

There is a gap in the existing research related to the

impacts of dissimilar primers on the silicone peel bonding strength with PMMA, so current research designed to contribute in understanding of medical grade silicone-acrylic bond strengths by examining the influences of dissimilar primers, aging conditions. Focusing on accelerated daylight-aging adds a practical dimension to the investigation, and the null hypothesis sets clear expectations for potential outcomes.

2. Materials and Methods

75 × 10 × 3 mm rectangular shaped hard wax blocks¹ were manufactured, and flaked [Figure 1](#). Following dental stone² set, the flasks were placed in washing machine for wax burnout. Then cold mold seals were painted on the mold cavity and heat cure acrylic resins³ were mixed and packed into the mold. Ordinary techniques of curing, deflasking, finishing and polishing of acrylic resin samples were followed. After 24h of construction, 60grit silicone carbide waterproofed abrasive papers were lapped over the surfaces of acrylic samples which were then wiped with acetone and permitted to be dried in air. sticky tapes were utilized to mark the region on which the REF M511 Platinum silicone elastomers⁴ were added to the PMMA samples. A region of (50mm×10mm×3mm) were masked by tapes exposing a region of (25mm×10mm) of silicone elastomer attached to the PMMA samples. The unattached silicones were clutched during the peel test. Teflon disks (18x8x3mm) were employed to specify the section throughout which primers were applied. Additional group of wax blocks of larger thickness (75x10×6mm.) were utilized to create stone moulds as defined before. The PMMA samples were located in the new moulds. Preceding to placing the M511 Platinum silicone, uniform films of primers were painted with brushes on the surfaces [Figure 2](#) and kept for half an hour at temperature not more than 25°C and moisture of 50%. The final thicknesses of the specimens were six millimeters (three for PMMA and three for M511 Platinum silicon). Silicones were fused to the PMMA at one end (25x10x3mm) and unattached to the other (50x10x3 mm). All unattached slips were bent backside at 180° so that the PMMA and silicone slips can be clutched. 80 overall combinations between silicone and PMMA were made and categorized to four groupings according to the primer applied; Group I (n=10; control): No primer, Group II (n=10): A-330G primer⁵, Group III(n=10): A 304 Primer⁶, and Group IV(n=10): G611 primer⁷.

Following polymerization, samples were kept for 24h at 37±1°C and the 180° peel test standards were ensued. Inside each group, half of the samples (n=5) rolled as baseline samples and put to the test after 24h of manufacturing and the residual samples (n= 5) were exposed to accelerated light aging for 360h in aging

chamber⁸ [Figure 3](#). Faster simulated daylight was created with 150 klx streamed Xenon light. The entire weathering sequences persisted for 120 min, involving 18 minutes of drenched by monitored stream of 30°C filtered aqueous solution, after that subjected to dry weather at 36°C for 102 minutes. The humidity inside the device was 70%, under 700–1060 hPa air pressure. The Xenon illuminations were used for the entire period of ageing (360 h). Universal testing devices⁹ were utilized to accomplish the 180° peel bonding experiments at crossheading rate of 10mm/min [Figure 4](#). All samples were strained to strip the silicone from the PMMA, and the highest forces needed for bonding loss were documented. The peel bonding strengths for all samples were detected via the dedicated equation: "P.G = F/W" where P.G = Peel bonding value (N/mm), F = Maximum loa at the initial failure point (N), W = specimen width (mm). The test was accompanied matching the American Standards for Testing and Materials (ASTM) D-903 [10,17].



Figure 1. wax blocks flaked in the dental flask



Figure 2. Adhesive primer painted on acrylic resin samples prior to addition of maxillofacial silicone

1 Technowax®-Baseplate, Protechno, Girona, Spain

2 Durguix, Protechno, Girona, Spain

3 ECO-CRYL HOT, Protechno, Girona, Spain.

4 Technovent REF M511 Platinum silicone elastomers, Factor II Inc., Lakeside, Ariz., USA.

5 primer A-330G, Factor II, Inc., Lakeside, Ariz., USA

6 primer A-304, Factor II, Inc., Lakeside, Ariz., USA

7 primer 611, Factor II, Inc., Lakeside, Ariz., USA

8 Heraeus Suntest CPS Accelerated Light Fastness Tester, Hanau, Germany

9 5 ST, Tinius Olsen India Pvt Ltd, India



Figure 3. Heraeus Sun test CPS Accelerated Light aging chamber

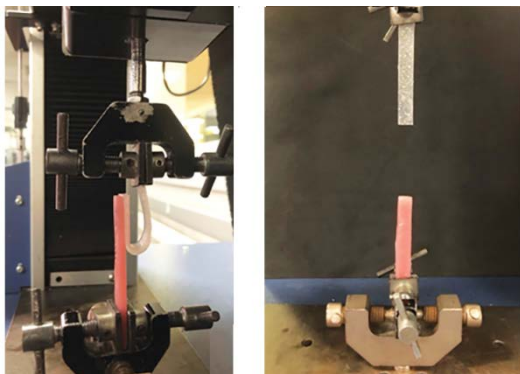


Figure 4. peel bond test of the samples

The breakdown of the bond or the bond failures were labelled under cohesive, adhesive, or miscellaneous through inspecting interfaces in-between acrylic resin and elastomers. complete splitting of PMMA and elastomers termed as adhesive failure while cohesive failure is defined by way of breakdown arise only within the elastomeric material. Both failures synchronized in miscellaneous failure.

analytical evaluations of experiment results were performed using One-way ANOVA and Tukey’s multiple comparison test, were utilized to verify the pair

differences. primer that exhibited the highest bond strengths were noted. The influences of aging on the bond strengths were explored throughout the independent t-test.

3. Results

Table 1 displayed the peel bonding strengths values of studied samples. Both at baseline, and after artificial aging, primers significantly improved the bonding strengths of M511 Platinum silicone elastomers to PMMA. The highest peel bond strength values were realized with G611 Primer (4.35) at baseline, followed by A - 330G primer(2.83), A 304 Primer(2.08) and the least in control Group (0.76). while after 360h of light-aging, A - 330G primer showed the highest values(7.33) followed by A 304 Primer(1.75) and the least values were founded with G611 primer(1.09). The impacts of artificial aging on peel bonding strengths were founded to be unpredictable as It was increased for primer A330-G and diminished for the control, G611, and A304 primers.

Regarding failures patterns (Table 3), peeling was deemed adhesive failure, while tearing is cohesive. The results revealed that the mode of failures at baseline were mostly cohesive (71.2%), and adhesive (28.8%), while following artificial aging, were essentially adhesive (81.3%), and cohesive (18.7%).

Table 1. Peel bonding strength values od study groups (N/mm)

Groups	Peel bonding strength (MPa)		
	Primer	Base line	After artificial aging
Group I (Control)	No primer	0.76 (0.42)	0.092
Group II	A - 330G primer	2.83(0.53)	7.33(2.41)
Group III	A 304 Primer	2.08(0.89)	1.75(1.62)
Group IV	G611 primer	4.35(0.84)	1.09(0.82)

Table 2. bond strength differences between Groups

Groups	Base line			After artificial aging		
	Difference	SE	Sig.	Difference	SE	Sig.
Control vs A - 330G primer	-2.074500	0.001488	0.000	-7.2384500	0.0003	0.000
Control vs A - 304G primer	-1.327200	0.001395	0.000	-1.658100	0.0002	0.000
Control vs G-611primer	-3.593500	0.001395	0.000	-0.998600	0.0002	0.000
A - 330G primer vs A - 304G primer	0.751600	0.001488	0.000	5.580500	0.0003	0.000
A - 330G primer vs G-611 primer	-1.527600	0.001395	0.000	6.245700	0.0002	0.000
A - 304G primer vs G-611 primer	-2.271700	0.001488	0.000	0.331700	0.0003	0.000

Table 3. Modes of failures of Peel bond tests

Failure mode	Peel bond strength (N/mm)	
	Base line	After artificial aging
Cohesive	29 (71.2%)	7 (18.7%)
Adhesive	10 (28.8%)	33 (81.3%)
Mixed	0	0
Total	39h	40h

4. Discussion

The bond between medical grade silicone and PMMA is a critical issue for the overall performance and durability of maxillofacial prosthesis. Peel strength is a measure of the force required to peel apart two materials that are bonded together. This study focused specifically on the impacts of primers on peeling strengths in-between The REF M511 silicone elastomers and PMMA.

The REF M511 Platinum silicone elastomers and the primers (A-330G, A 304, and G611) selected for current research are already established and widely accepted within the field of maxillofacial prosthetics and how these materials respond to different types of stress and strain are inspected in various studies [18,19].

The study's focus on testing methods and the insights gained into bonding and debonding characteristics, as well as the forces involved in prosthetic removal, contributes valuable information to the field. Researchers and practitioners can use these insights to enhance the design and performance of implant-retained maxillofacial prostheses, ultimately improving patient comfort and the longevity of prosthetic devices.

Patients typically remove implant-retained prostheses by employing rotational or peeling movements. Understanding how patients interact with the prostheses is crucial for designing materials that can withstand such forces and movements. The study employed test methods, such as the peel test, to gain insights about the adhesive properties of REF M511 Platinum silicone elastomers adhered to PMMA. This experimental approach helps researchers and clinicians understand the behavior of the materials in real-world scenarios and aids in refining prosthesis design. The peel test is particularly relevant as it simulates the horizontal displacement forces prevalent during removing the prosthesis from its position. This is crucial information for assessing material's performance and mechanical properties of any material used for construction of facial prosthesis, providing insights into material's ability to withstand forces associated with prosthetic removal. This information is valuable for addressing concerns related to material durability and designing solutions that can mitigate or prevent silicone stripping during prosthesis removal.

The study highlights the dynamic nature of the bonding in-between M511 Platinum silicone elastomers and a rigid acrylic base, outlines changes in bond strengths between silicone and a rigid acrylic resin base under various conditions and emphasizing the impact of aging. Initially, at baseline conditions, the combination of REF M511 Platinum silicone elastomers and 611 primer displayed the highest peel strength, measuring at 4.35N/mm. This implies that, under standard or initial conditions, this combination provided the strongest bond. Conversely, after artificial aging, there was a change in the optimal combination. The REF M511 Platinum silicone elastomers and A330-G primer combination confirmed a wonderful peel strength of 7.33N/mm after aging. This suggests that the performance of the REF M511 Platinum silicone elastomers and A330-G primer combination became more favorable over time, indicating improved bond strength. The shift in optimal combinations after aging suggests that

different combinations can prove to be serviceable under prolonged use conditions. This is important for understanding the long-term durability and stability of facial prosthesis.

The variations in bond strengths were due to chemical structures differences between both silicone and primer[20]. The primer act as an adhesive medium in-between the REF M511 Platinum silicone elastomers and PMMA [16,20].

In this study, along with other studies [21,22] considered the peeling strengths as the maximum peel force recorded per unit of width. However, it's noted that in these cases, the extension ratio was not accounted for.

The absence of consideration for the extension ratio in such calculations warrants caution in interpreting results. The extension ratio reflects the degree of elongation or stretching during the peeling process.

In another study [10], the peel bond strengths were calculated considering the elastic deformation of silicones that is persuaded by aspects such as sample stiffness and dimensions. Compliance refers to the ability of a material to undergo elastic deformation in response to an applied force. Hardness, on the other hand, is a measure of a material's resistance to permanent deformation.

The area of bonding is crucial, as it determines the extent of the bonded interface contributing to the overall strengths of the adhesion bonding. This emphasizes the importance of understanding the specific conditions under which peeling occurs and tailoring calculations accordingly [23].

The effects of light-aging on silicones are complex and can vary based on factors like material composition. Recognizing these variations and understanding the influence of aging is pivotal for advancing the development and assessment of silicone elastomers in applications where durability and long-term performance are critical, such as in maxillofacial prosthetics. Light aging is described as enhancing silicone resistance to tear. This suggests that the aging process, involving exposure to both heat and light, contributes to positive changes or improvements in the material properties of silicone elastomers. Silicone elastomers continue polymerization during light-aging [24,25]. Polymerization is the process by which monomeric molecules join together to form a polymer. In this context, ongoing polymerization could cause modifications in silicone properties and structure. The effect of aging impact was observed to be different and dependent on the bond test used. Peel-bond strengths were specifically mentioned to increase for the A330-G group but decrease for the control (unaged), G611, and A304 groups. For the A330-G group, an increase in peel-bond strengths implies that light-aging has a positive effect on the adhesive bond between the silicone and the substrate. In contrast, a decrease in peel-bond strengths for the control, G611, and A304 groups suggests that these materials may experience a reduction in adhesive strength under the aging conditions. The information emphasizes that the aging effect on bond strengths was dependent on the type of bond test conducted. This highlights the need for careful consideration of the testing method when evaluating the impact of aging on adhesive properties. The reduction in bond strengths after severe conditioning regimes is a multifaceted phenomenon. It involves

considerations such as the stability of adhesive primers, the impact of aging on silicone resistance, the effects of water storage and continued lighting, and the influence of residual stresses and thermal expansion coefficients. Understanding these factors is crucial for developing resilient bonding systems, especially in applications like maxillofacial prosthetics where the materials are exposed to various environmental conditions and mechanical stresses [26].

The bonding breakdown pattern was evaluated and categorized into adhesive, cohesive, or miscellaneous. This indicates an examination of how the bonding in-between the silicone elastomers and PMMA responds under peel forces. Cohesive failure refers to the breaking or separation of the silicone material itself, rather than at the interface with the denture base. After light-aging, there was a shift in the predominant failure type. Peel forces exhibited a predominant adhesive failure pattern (81.3%). Adhesive failure implies that the bonds in-between M511 Platinum silicone elastomers and PMMA were weaker than the bonding inside the M511 Platinum silicone elastomers itself. For cohesive failures, it's noted that the peel bonding strengths in-between M511 Platinum silicone elastomers and PMMA were greater than the M511 Platinum silicone elastomers strength. This suggests that the bond to the PMMA was more robust over the inherent silicone strengths. This information suggests caution in interpreting cohesive peel-bond test failures. While the peel test has advantages, such as a controlled rate of failure, cohesive failures would be approached cautiously in terms of their implications. The assessment of bond failure types provides insights into how bonding in-between M511 Platinum silicone elastomers and the denture base responds to peel forces. The shift from predominantly cohesive failures to adhesive failures after aging indicates changes in the adhesive properties over time. Additionally, the cautionary note on interpreting cohesive failures emphasizes the complexity of adhesive behaviors and the need for careful analysis in studies related to maxillofacial prosthetics.

The study found that the selection of primers significantly influenced bond strengths within peel bond tests. This underscores the importance of primer choice in determining the adhesive performance of the silicone material to a substrate. For REF M511 Platinum silicone elastomers, it was observed that the baseline 611 primer samples displayed the greatest bonding strengths. However, this bond strength was noted to decrease after aging. This suggests that the effectiveness of primer 611 was affected by the aging process. After light-aging, the study found that combination of REF M511 Platinum silicone elastomers and primer A330-G displayed superior and durable bonding strength. This implies that, in the context of light-aging, the A330-G primer was more effective in maintaining or even enhancing bond strength compared to primer 611. The term "serviceable bond strengths" suggests that the bond strengths achieved after light-aging were considered suitable or acceptable for the intended application. This indicates that, despite the aging process, the bond between silicone MDX4-4210 and the substrate remained effective and reliable when using A330-G primer.

The methodology used in the present research in order to investigate bonding strengths, emphasizing use of peel

bond tests and the decision not to conduct tensile tests. Tensile tests typically measure the material's resistance to a force pulling it apart. While valuable for assessing the intrinsic strength of a material, this method may not specifically address the adhesive strength between the material and a substrate. It's mentioned that all bond tests, including peel bond tests, have been exposed to practical reviewing [24]. This highlights an awareness of the limitations and challenges associated with bond testing methodologies. The limitations of bond tests are acknowledged, particularly in the context of representing the authentic loads applied during prosthetic applications. Bonding assessments typically use only one force in a single path, whereas prosthetic devices in real-world scenarios are subjected to numerous forces in various paths. The complexity of bonding process adds a layer of struggle in explaining outcomes from any experiment. Despite the acknowledged challenges and limitations, bond tests are considered helpful in judging and grading the bond between silicone elastomers and PMMA. They provide a controlled environment for assessing the relative performance of various adhesive primers and surface treatments.

5. Conclusions

Regarding study limits, peel bond strength was applicable and appropriate test for investigating the efficiency of primer type to bond REF M511 Platinum silicone elastomers to PMMA. The peel bond strength ranged from 0.09-7.33N/mm. initially (baseline), the failure patterns were cohesive in nature and adhesive after artificial aging. Maximum peel bonds were accomplished using REF M511 Platinum silicone elastomers and A330-G primer.

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