

ORIGINAL ARTICLE

Effect of adhesives and mechanical surface treatments on the hard relining of CAD-CAM denture bases

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Abstract

Purpose: The aim of this study was to evaluate the impact of mechanical roughening, adhesive applications, and aging on the bonding between CAD-CAM denture base materials with distinct chemical contents and hard relining material.

Materials and Methods: A total of 300 denture base specimens were produced by additive, subtractive, and conventional heat-polymerization techniques ($N = 100$). The specimens have been classified into five subgroups based on the particular surface treatments administered ($n = 20$): (1) Hard relining material's adhesive application (control); (2) Tungsten carbide bur application for 1 min, and hard reline material's adhesive application; (3) Airborne-particle abrasion (APA) with $110 \mu\text{m Al}_2\text{O}_3$, and hard reline material's adhesive application; (4) Scotchbond Universal application; and (5) Visio.link application. Representative specimens from each subgroup were examined under a Scanning Electron Microscope (SEM). Subsequently, self-cure hard relining material was condensed in the center of the specimens. Half of the specimens were thermally aged with 5000 cycles at 5°C – 55°C . The shear bond strength (SBS) test was performed, and failure loads were recorded. The data was evaluated by Robust ANOVA and Bonferroni test ($p < 0.05$).

Results: No statistically significant difference was obtained between the production techniques ($p = 0.051$). The lowest SBS was observed in the control group among surface treatments, while mechanical surface treatments and universal adhesive showed the highest SBS for both aged and non-aged groups. Aging caused a significant decrease for all test groups ($p = 0.001$).

Conclusions: Mechanical surface treatments and universal adhesive applications are more effective for maintaining adhesion across all production techniques.

KEYWORDS

adhesion, computer-aided design/computer-aided manufacturing, denture bases, geriatric dentistry, hard relining material, 3D printing

Relining complete dentures effectively alleviates the discomfort and pain experienced by patients suffering from alveolar ridge resorption.¹ Individuals experiencing issues with bone undercuts, irregular bone resorption, dry mouth, thinning atrophic mucosa or immediate prostheses, and bone-healing after implantation may benefit from denture relining to reduce

symptoms.² This procedure is also recommended to improve oral health and prevent further complications.³

Conventional heat-cure polymethylmethacrylate (PMMA) resins have been the most commonly utilized material for denture bases for over 80 years.^{4,5} Nowadays, denture bases can also be fabricated by computer-aided design

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and computer-aided manufacturing (CAD-CAM) by milling (the subtractive approach) or three dimensional printing (the additive approach).^{6,7} CAD-CAM denture bases provide several benefits, such as shorter chair time, better fit, and digital archiving of the prosthesis.⁸ Utilizing CAD-CAM technology to mill denture bases from pre-polymerized acrylic resin discs results in minimal volumetric deviation and eliminates further shrinkage caused by polymerization.^{9,10} Compared to the subtractive technique, additive manufacturing offers more advantages, such as producing any object, regardless of its dimensional complexity or quantity.¹¹ Additionally, additive manufacturing generates less waste and can reproduce fine details accurately.^{11,12}

Regardless of the manufacturing technique, the relining process can be executed either chairside or in a laboratory by taking impressions. Performing a chairside relining procedure has various benefits, including being more cost-effective, time-saving, and simpler than laboratory methods.¹² Periodic hard relining of complete dentures is recommended to enhance their fit and ensure better adaptation of the denture base to the underlying structure.¹³ Conventionally produced complete denture resins and relining materials are similar in many aspects, such as mechanical properties and chemical structures. Studies have reported positive outcomes with regard to bonding these materials together.¹⁴ However, recent research highlights that relined 3D-printed resins demonstrate lower adhesion capacity than those produced by subtractive and conventional methods.^{12,14} It is paramount for dental practitioners to acquaint themselves with the relining technique for denture bases manufactured using additive techniques. This knowledge is especially crucial in the context of a fully digital workflow in clinical settings. Bond strength is influenced by various factors, including the chemical structures of denture bases and relining materials, the reaction of bonding agents, and the impact of thermal stress.^{15,16} Modifying denture bases or relining materials is not feasible due to their chemical composition. However, there is potential to enhance bond strength by optimizing the reaction between denture bases and adhesives.¹⁶ In recent years, new primers have been developed to improve the bonding of PMMA denture resins.¹⁷ Similarly, adhesives have been used to repair urethane dimethacrylate temporary restorations.¹⁸ However, there is a lack of research on improving the bonding strength between additively produced resins and relining material in the literature.

The purpose of the present study was to assess the effectiveness of the bonding between hard relining material and denture base materials produced through conventional heat-polymerization, subtractive, and additive techniques by considering the surface treatment method and aging. The surface treatment methods employed in the study comprised hard relining material's adhesive application alone (control group), 110 μm airborne-particle abrasion, roughening with a tungsten carbide bur, Scotchbond Universal application, and visio.link application. The null hypothesis was that the

bond strength between denture bases and relining material would remain unaffected by surface treatments, production techniques, aging, or any interactions thereof.

MATERIALS AND METHODS

This study utilized five surface treatments for conventional heat-polymerized, subtractive, and additive denture material surfaces and relined them with hard relining acrylic resin (Ufi Gel Hard C, VOCO GmbH, Cuxhaven, Germany) (Table 1) ($N = 300$, $n = 100$). Specimens were designed as 2 mm thickness and 10 mm diameter discs in the 3D design program (Autodesk Meshmixer v3.4.35, Autodesk Inc, San Rafael, CA, USA) and saved in STL format. Extra resin specimens were 3D printed to prepare the conventionally produced specimens and placed in flasks. Once the stone was set, the flasks were opened, and the printed patterns were removed. Heat-polymerized acrylic resin liquid and powder (Paladon 65, Kulzer, Hanau, Germany) were proportioned and mixed according to manufacturer instructions. The polymerization process was completed using the same procedure as the previous study by Şahin et al.¹⁹ Subtractive group specimens (Polident, Pearson Dental Supply Company, CA, USA) were produced using a dental milling device (Coritec 550i, imes-icore, Eiterfeld, Germany). Additive group specimens (V-Print Dentbase, VOCO GmbH, Cuxhaven, Germany) were fabricated using a DLP-type 3D dental printer (Solflex 650, VOCO GmbH, Cuxhaven, Germany). The layer thickness was set to 65 μm with supports opposite the testing side and printed at 0° ($n = 50$). The specimens were washed with 98% isopropyl alcohol for 4 min in an ultrasonic cleaner and dried for 30 min. Then, both sides of the specimens were treated with UV light in a curing unit (Labolight Duo, GC Europe, Leuven, Belgium).

One side of the specimens (in all test groups) was ground under running water for 1 min using sandpaper with a grain size of 800–1000–1200 to ensure standardization. The polished side of the specimens was placed facing up and then embedded in type 4 hard dental plaster. The prepared specimens were randomly divided into five subgroups based on surface treatment application ($n = 20$): (1) Hard relining material's adhesive application (control); (2) Tungsten carbide bur application for 1 min and hard relining material's adhesive application; (3) Airborne-particle abrasion with 110 μm Al_2O_3 at a 45° angle from a distance of 10 mm at 2 atm pressure for 10 s and hard relining material's adhesive application; (4) Scotchbond Universal adhesive application by rubbing it for 20 s and the specimens were light cured for 10 s with a light-emitting diode lamp (VALO™ Cordless, Ultradent, South Jordan, UT, USA); and (5) Airborne-particle abrasion with 110 μm Al_2O_3 at 2 atm pressure for 10 s, ultrasonic cleaning for 5 min then visio.link adhesive application with a brush and light cured for 90 s in a dual-mode light curing unit (Labolight Duo, GC Europe, Leuven, Belgium). The same operator (I.A.) carried out all the surface treatments.

TABLE 1 Types and compositions of the materials used in the study.

Product	Composition	Type	Manufacturer
Paladon 65	PMMA	Conventional heat-polymerized	Heraeus Kulzer, Hanau, Germany
Polident	PMMA	Subtractive (pre-polymerized)	Pearson Dental Supply Company, CA, US
V-Print Dentbase	UDMA (50–100%), BisGMA (25–50%), TEGDMA (5–10%), and diphenyl (2,4,6-trimethyl benzoyl) phosphine oxide (TPO) (<2.5%)	Additive	VOCO GmbH, Cuxhaven, Germany
Ufi Gel Hard C	Base: BIS-GMA, HEDMA, Urethandimethacrylate Catalyst: BIS-GMA, HEDMA, Urethandimethacrylate, Benzoyl peroxide Adhesive: Aceton, 2-Hydroxyethylmethacrylat	Hard relining material	VOCO GmbH, Cuxhaven, Germany
Scotchbond Universal	MDP phosphate monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic asit copolymer, filler, ethanol, water, initiator, silane	Universal adhesive	3 M ESPE, MN, USA
visio.link	MMA, PETIA, photoinitiators	Universal, light-curing PMMA and composite primer	Bredent, Senden, Germany

Abbreviations: Bis-GMA, bisphenol A-glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; PETIA, pentaerythritol triacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate.

An additional specimen was prepared for each surface treatment group of every production technique for SEM analysis. The prepared specimens were cleaned in an ultrasonic cleaner using distilled water for 5 min and then dried. Afterward, they were coated with a thin layer of Au-Pd (200–300 nm). The surface inspection was carried out using SEM (JSM—T220A, JOEL LTDA, Tokyo, Japan) at 1000× magnification. Subsequently, self-cure hard relining material was mixed with an automix cartilage and condensed with a silicone mold (5 mm diameter and 3 mm height). Excess material was removed 3 min after condensation. All specimens were kept in a humidifier at 37°C for 24 h. Half the specimens underwent thermal aging at 5°C–55°C in distilled water with a dwell time of 20 s for 5000 cycles. The remaining specimens, which were not aged, were loaded in a universal testing machine at a crosshead speed of 1 mm per minute. The maximum shear force was recorded before failure. The same procedure was applied to the aged specimens. The shear bond strength was calculated by dividing the fracture load by the bonded surface area ($N/mm^2 = MPa$). The failure types were assessed by using a reflected-light microscope (Digital Sight DS-Fi2, Nikon, Tokyo, Japan) at a 20× magnification. They were categorized as cohesive, adhesive, or mixed failure, as previously outlined by Younis et al.²⁰

Data were analyzed with R Project. Normal distribution was examined with the Shapiro-Wilk test. Robust ANOVA was used using the WRS2 package to compare parameters that were not normally distributed according to technique, surface treatment, and aging process. Multiple comparisons were examined with the Bonferroni test. Analysis results were presented as trimmed mean \pm standard error for quantitative variables. The significance level was set as $p < 0.05$.

TABLE 2 Results of Robust 3-way ANOVA for the three independent variables and their interactions.

	Test statistics ^a	<i>p</i>
Production technique	46.40	0.051
Surface treatment	7699.70	<0.001
Aging	8016.30	0.001
Production technique \times Surface treatment	838.40	0.001
Production technique \times Aging	51.60	0.001
Surface treatment \times Aging	1573.10	0.001
Production Technique \times Surface Treatment \times Aging	130.00	0.001

Note: Values shown in bold are statistically significant ($p < 0.05$).

^aRobust 3-way ANOVA.

RESULTS

Based on the findings presented in Table 2, the 3-way Robust method analysis outcomes suggest that although the production technique did not have a statistically significant effect ($p = 0.051$), the surface treatment and aging had significant statistical impacts, along with their interaction ($p < 0.001$). Table 3 exhibits the descriptive statistics for the SBS test, comprising production technique, surface treatment, and aging. Aging caused a statistical decrease in the SBS values of all production techniques ($p = 0.001$).

The mean and standard deviations of shear bond strength values for all test groups are shown in Figure 1, and multiple comparisons showing statistical differences are presented in Table 3. There was no statistical difference between 110 μ m airborne-particle abrasion (36.10 MPa), bur application (36.40 MPa), and Scotchbond Universal (34.90 MPa)

TABLE 3 Descriptive statistics and multiple comparison results of SBS by production technique, surface treatment, and aging.

Aging	Surface treatment	Production technique			
		Conventional Median (Min–Max)	Subtractive Median (Min–Max)	Additive Median (Min–Max)	Total Median (Min–Max)
Non-Aged	Control	22.05 ±0.14 ^A	20.71 ±0.24 ^{AM}	18.06 ±0.24 ^{BDIJ}	20.30 ±0.35 ^A
	110 Al ₂ O ₃	35.73 ±0.11 ^{GH}	36.66 ±0.09 ^G	35.77 ±0.19 ^H	36.10 ±0.10 ^C
	Bur	36.50 ±0.11 ^{GH}	36.55 ±0.12 ^G	36.05 ±0.13 ^{GH}	36.40 ±0.08 ^C
	Scotchbond	32.92 ±0.21 ^L	32.81 ±0.20 ^L	39.23 ±0.16 ^O	34.90 ±0.62 ^C
	visio.link	18.39 ±0.09 ^{Bi}	18.65 ±0.10 ^{BiM}	16.99 ±0.14 ^{DEJ}	18.00 ±0.16 ^F
	Total	29.30 ±1.18 ^A	29.20 ±1.24 ^A	29.30 ±1.53 ^A	29.20 ±0.76
Aged	Control	12.63 ±0.53 ^{BCDEF}	11.15 ±0.47 ^F	5.95 ±0.20 ^N	10.40 ±0.62 ^B
	110 Al ₂ O ₃	15.93 ±0.53 ^{BCDEIJK}	15.79 ±1.11 ^{ABCDEFIJKMN}	12.33 ±1.52 ^{ABCDEFIJKMN}	14.90 ±0.70 ^{DE}
	Bur	17.66 ±0.32 ^{IJK}	16.60 ±0.28 ^{BCDEIJK}	15.51 ±0.10 ^{CK}	16.60 ±0.23 ^D
	Scotchbond	15.75 ±0.51 ^{BCDEFIJKM}	14.06 ±0.41 ^{CEF}	15.14 ±0.10 ^{CK}	14.90 ±0.25 ^E
	visio.link	11.69 ±0.74 ^{BCDEFIJKN}	10.97 ±0.30 ^F	8.45 ±1.63 ^{ABCDEFIJKMN}	10.40 ±0.65 ^B
	Total	14.80 ±0.42 ^B	13.70 ±0.45 ^B	11.50 ±0.73 ^B	13.50 ±0.34
Total	Control	17.40 ±1.22 ^{ABCDE}	15.90 ±1.23 ^{BCDE}	13.10 ±1.67 ^E	15.70 ±0.81 ^a
	110 Al ₂ O ₃	26.00 ±2.52 ^{AB}	26.50 ±2.70 ^{ABC}	24.40 ±3.07 ^{ABCDE}	25.80 ±1.54 ^b
	Bur	27.10 ±2.40 ^A	26.60 ±2.54 ^{AB}	25.80 ±2.62 ^{ABCD}	26.50 ±1.43 ^b
	Scotchbond	24.30 ±2.20 ^{ABCD}	23.50 ±2.40 ^{ABCDE}	27.20 ±3.06 ^{ABCD}	24.80 ±1.48 ^b
	visio.link	15.10 ±0.93 ^{CDE}	14.90 ±0.99 ^{DE}	12.90 ±1.35 ^E	14.40 ±0.64 ^a
	Total	21.70 ±1.01	21.20 ±1.08	20.10 ±1.30	21.10 ±0.65

^{a-b}There is no difference between the main effects with the same letter.

^{A-O}There is no difference between interactions with the same letter; trimmed mean ± standard error.

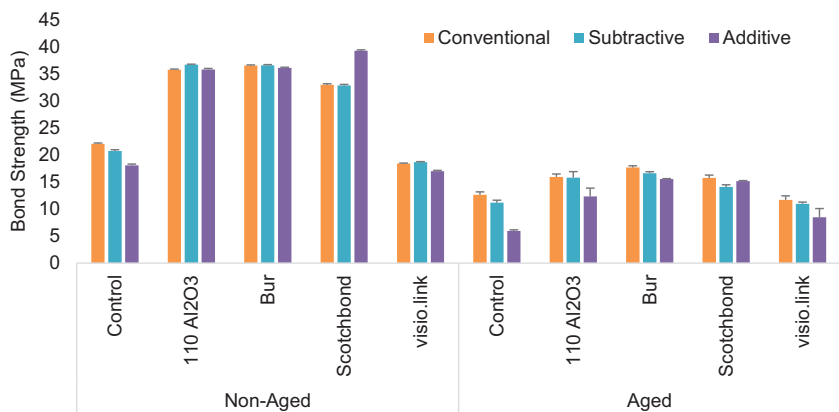


FIGURE 1 The mean and standard deviations of bond strength values for all test groups.

applications, while the control (20.30 MPa) and visio.link groups (18.00 MPa) showed statistically lower SBS in the non-aged groups. In the aged groups, there was no statistical difference between 110 μm airborne-particle abrasion (14.90 MPa) and tungsten carbide bur (16.60 MPa), and between 110 μm airborne-particle abrasion and Scotchbond Universal applications (14.90 MPa). Similar to non-aged groups, the control (10.40 MPa) and visio.link groups (10.40 MPa) showed statistically lower SBS. In relation to production technique, surface treatment, and aging interaction, the non-aged group that was additively manufactured and underwent Scotchbond Uni-

versal showed the highest mean SBS of 39.23 MPa. Conversely, the control group of the additive technique with aging displayed the lowest bond strength at 5.95 MPa.

The failure types observed in the specimens have been categorized based on aging, the production method, and surface treatment, as presented in Figure 2a,b. It was observed that in the specimens that were not aged, both the control and visio.link groups exhibited a predominance of adhesive failure. Meanwhile, in the aged specimens, all test groups showed a significant prevalence of adhesive failure. The SEM images depicting the surface treatment groups are displayed

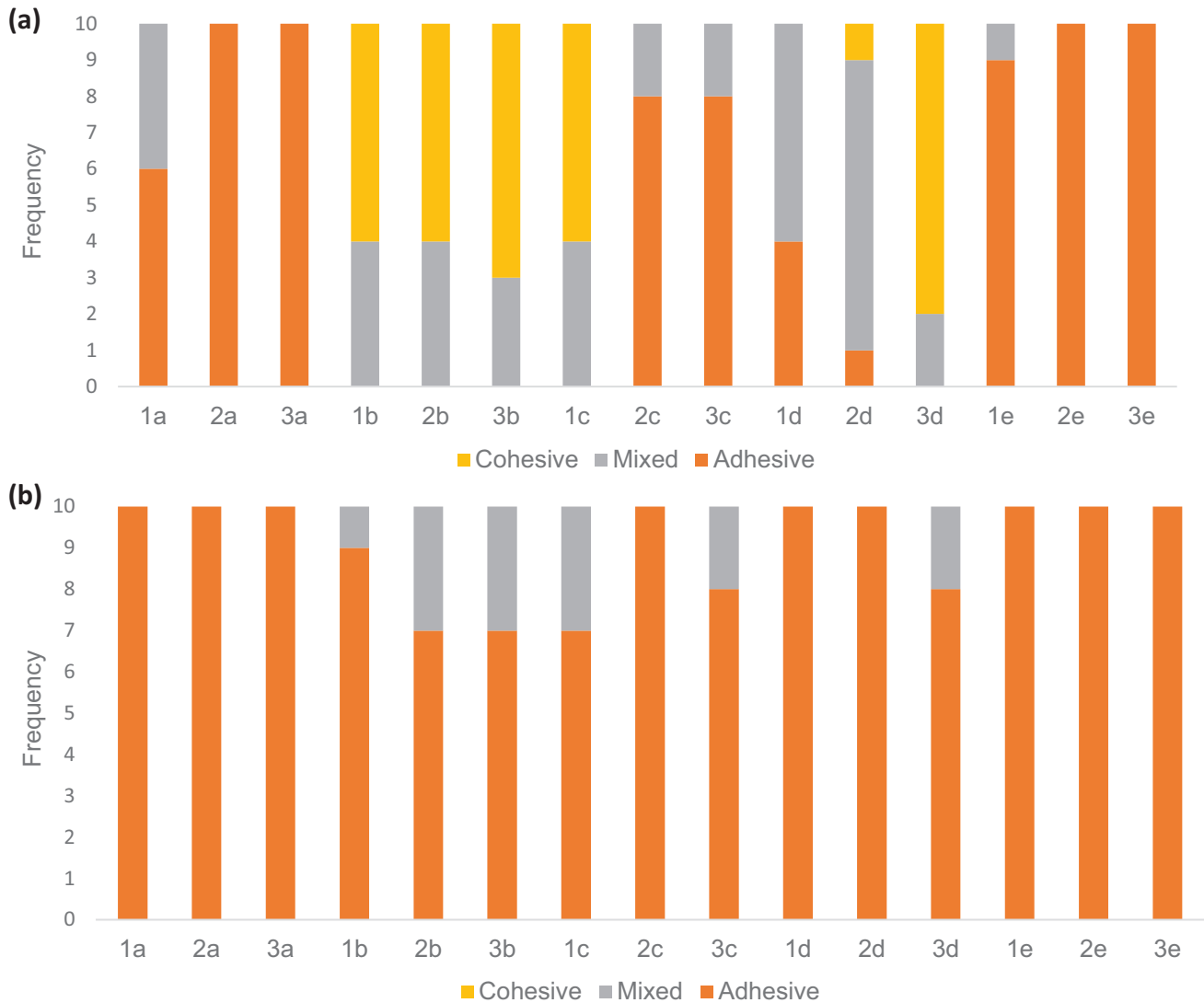


FIGURE 2 Failure types of the non-aged (a) and aged groups (b) as (1) conventional heat-polymerization, (2) subtractive technique, (3) additive technique, and surface treatments as control (a), 110 μm airborne-particle abrasion (b), carbide bur application (c), Scotchbond application (d), and visio.link application (e).

in Figure 3. The control and the visio.link groups expressed the smoothest surface, followed by the group to which tungsten carbide bur was applied. The experimental findings revealed that the groups that underwent Al_2O_3 application and Scotchbond Universal treatment exhibited a higher number of indentations and irregularities when compared to the other groups.

DISCUSSION

The study's findings have revealed that the manufacturing technique did not exhibit a statistically significant difference in bond strength. However, the surface treatments, aging, and their interactions had a statistically significant impact on bond strength. As a result, the null hypothesis was partially rejected.

Previous studies evaluating the bond strength of denture bases produced with different techniques with relining materials have reported conflicting results. Some studies have reported no difference in bond strength between conventionally heat-polymerized and subtractive techniques, while specimens produced with an additive technique exhibit lower bond strength.^{12,14} On the contrary, some studies reported no difference in bond strength between groups produced with three different techniques.²¹ A recent systematic review and meta-analysis study found that hard relining materials result in better outcomes with subtractively produced denture bases than conventionally produced ones, while there is no significant difference between 3D-printed and conventional bases.²² However, the study did not consider crucial aspects such as the type of hard relining material, surface treatment, and oral conditions. The materials used in conventional heat-polymerized and subtractive techniques typically

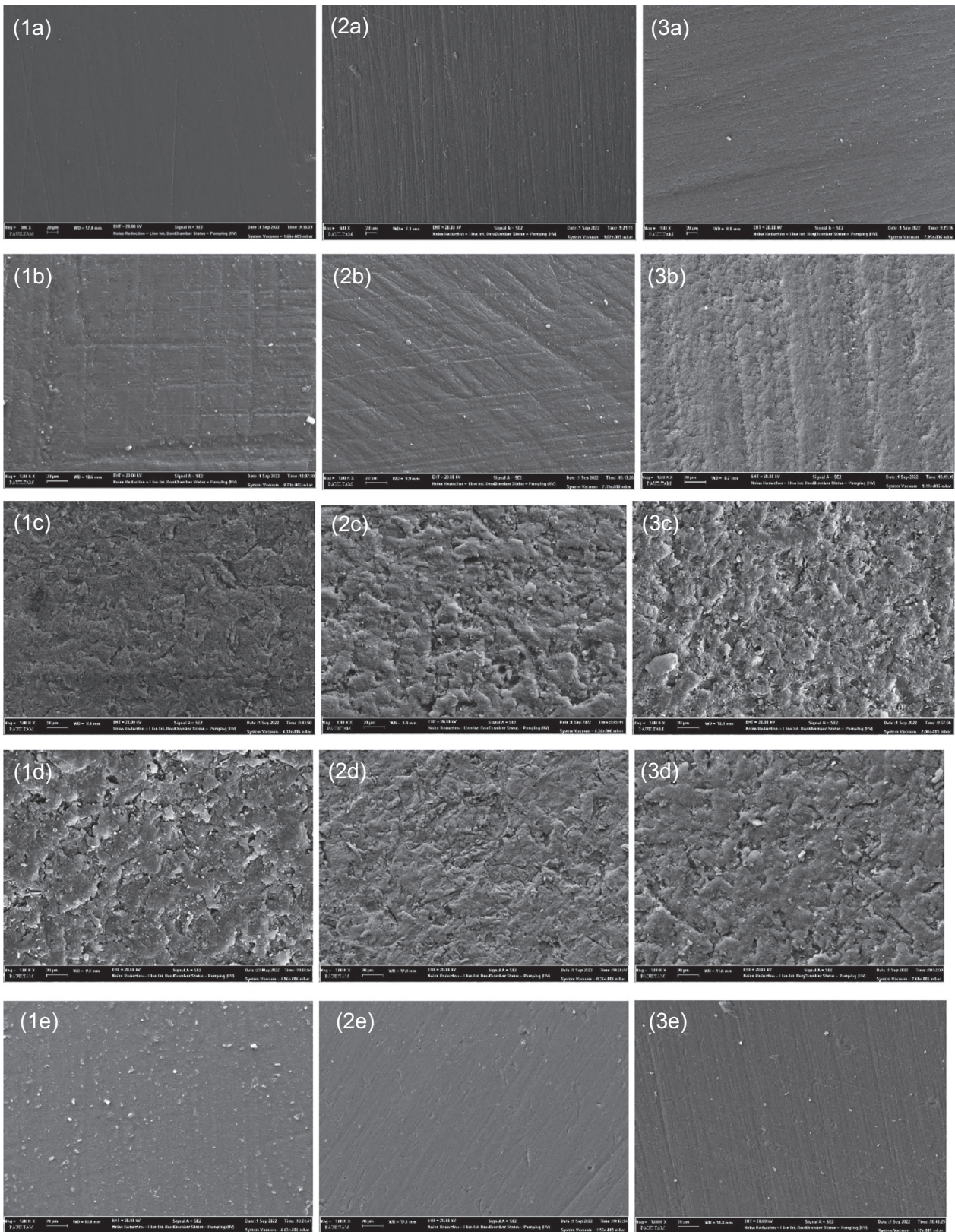


FIGURE 3 SEM images of production techniques as (1) conventional heat-cure, (2) subtractive, (3) additive and surface treatments as control (a), tungsten carbide bur application (b), 110 μm airborne-particle abrasion (c), Scotchbond application (d), and visio.link application (e). 1000 \times magnification.

comprise PMMA. Conversely, materials used in additive techniques and hard relining materials may exhibit variations in their chemical composition. The variations in bond strength observed in previous studies can be attributed to disparities in surface treatments, relining materials, testing procedures, and the extent of replication of the oral environment.

Wemken et al.²³ investigated the bond strength of denture bases made through conventional and CAD-CAM methods to both soft and hard relining materials. The same chair-side hard relining material as in the present study was tested alongside an additively manufactured denture base material with a similar chemical composition. Unlike the control group in the present study, they reported almost twice the bond strength of additively produced denture bases compared to the other two production techniques. As per their statement, the observed outcome could be attributed to using acetone as a solvent in the adhesive. Using acetone leads to greater swelling in the material and facilitates deeper penetration of the monomers. However, it should be noted that this theory is not entirely conclusive, as additively produced specimens did not exhibit any surface changes upon exposure to acetone, while the surface integrity of conventional and subtractive specimens was affected. The polymerization of methacrylates follows a linear pattern in conventional and subtractive techniques. Conversely, crosslinked polymers form from multifunctional monomers, and the linear bonded polymethyl methacrylate (PMMA) is more soluble than the crosslinked additively manufactured resin.^{16,23} In the present study, the conventionally produced group showed higher bond strength than the subtractively produced group, similar to the aforementioned study. The CAD-CAM PMMA blocks undergo a process of pre-polymerization under optimized parameters to achieve maximum conversion from monomer to polymer.²⁴ The subtractive technique may provide a more uniform surface structure, while conventional denture bases may have more residual monomers on their surfaces. As a result, acetone may significantly affect conventional denture bases, leading to higher bond strength.

Various mechanical methods such as plasma and laser applications, airborne-particle abrasion, and bur roughening have been used in studies to enhance the bonding of base materials.^{25–27} While previous studies,^{27,28} have shown that plasma and laser also improve adhesion, bur roughening, and Al_2O_3 applications were used in the current study due to the unavailability of plasma and laser application in every clinic and small laboratories. The study's results indicate that mechanical surface treatments produced higher outcomes for all denture bases except for the additive group treated with Scotchbond. Akin et al.²⁹ found that increasing the bond strength of PMMA surface can be achieved through airborne-particle abrasion with Al_2O_3 particles or grinding with a tungsten carbide bur. According to previous studies, using Al_2O_3 airborne-particle abrasion is a viable technique for augmenting the bond strength of subtractively produced PMMAs.^{27–30} In a study conducted by Li et al.,³¹

the shear bond strength of denture base materials produced through additive manufacturing was evaluated. As a result, they stated roughening with 600-grained sandpaper (simulating the homogeneous roughness produced by dental burs) and airborne-particle abrasion with $125\ \mu\text{m}\ \text{Al}_2\text{O}_3$ at 2 bar pressure resulted in similar outcomes as observed in the current study.

Studies evaluating the bond strength of universal adhesives and PMMA and urethane dimethacrylate materials focus on temporary restorations. The phosphate monomer 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) in the universal adhesive can bond to various substrates, such as resin bis-GMA matrix via a terminal double bond group.^{32–34} Filokyprou et al.³⁵ evaluated the retention force of temporary restorations consisting of urethane dimethacrylate. The researchers examined the impact of various surface treatments, including sandblasting, tribochemical silica coating application, and combinations. The study results indicate that applying MDP containing silane produced the highest retention force. They reported that the reason for this may be the chemical reaction of the silane with the urethane dimethacrylate resin. In the present study, higher SBS values were observed between additively produced specimens and universal adhesives, similar to the mentioned study. On the other hand, universal adhesive showed higher SBS for PMMA groups than the control group even aged. Goracci et al.³⁶ also found higher bond strength with Scotchbond Universal and PMMA resins, and they indicated that could be due to the chemical bond between the 10-MDP and methacrylates of the substrate. However, more studies should be revealed to understand the mechanism of these reactions.

Visio.link is a resin primer specifically formulated to enhance the bond between PMMA denture and composite resin materials. However, to our knowledge, no research has been conducted to evaluate its efficacy in improving the bond strength of PMMA denture bases. Chemical priming is an effective method to enhance adhesion between PMMA and resins. This technique introduces a polymerized layer that acts as an intermediary and significantly improves the bonding strength.³⁷ The visio.link contains two main components: pentaerythritol triacrylate (PETIA), which acts as a crosslinking agent, and methyl methacrylate (MMA). The MMA component increases the viscosity and fluidity of the primer and, after photopolymerization, forms a polymerized layer.³⁸ Peng et al.³⁹ reported that the process of polymerization of MMA into PMMA resulted in the formation of bubbles. This phenomenon was attributed to a heterogeneous wetting state during the polymerization shrinkage. The current study has revealed that conventional denture base surfaces exhibit partial bubbles, whereas denture bases produced with the subtractive technique demonstrate a more uniform surface. Although the difference in SBS between the groups was not statistically significant, this situation could result in inferior bonding of conventionally produced denture bases compared to subtractively produced ones. Also, forming a different chemical layer between materials with similar

chemical structures may have negatively affected the bonding for the additive group.

The current study's findings indicate that the bond strength of all test groups decreased with aging. Fluctuations in intraoral temperature and oral fluids can significantly impact the junction interface between denture base materials and repair materials. This interface is particularly susceptible to thermal stress during temperature changes, given the varying coefficients of thermal expansion present in these materials.²⁷ Additionally, water absorption can cause the polymer chains in the denture base to separate from each other.⁴⁰

The current study aimed to simulate oral conditions through thermal aging only. However, it did not account for the impact of repetitive chewing force stresses and pH changes. Furthermore, the study only tested one 3D printing technique and hard relining resin along with one universal adhesive. Therefore, it is recommended that future studies explore the bond strength of 3D-printed denture bases and universal adhesives from different brands in combination with various relining materials.

CONCLUSION

The adhesion between the hard relining resin and denture base materials can vary and can be enhanced by various surface treatment methods. According to the study results, the groups that underwent mechanical surface treatments and universal adhesive application exhibited higher bond strength. However, regardless of the production techniques used, the bond strength decreased with aging.


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CONFLICT OF INTEREST STATEMENT

The authors did not have any commercial interest in any of the materials used in this study.

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