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Effect of surface treatment on bonding strengths of hard and soft reline materials to 3D-printed denture base resin

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Abstract

To evaluate the effects of surface treatment and thermal cycling on the tensile bond strength of hard and soft reline materials to a 3D-printed denture base resin (3DP) compared with a conventional heat-polymerized resin (HC). Specimens of 3DP and HC resins received no treatment, alumina sandblasting, dichloromethane priming, or 4-META/MMA-TBB resin (4-META) application. One hard reline material (HR), two acrylic-based soft reline materials (AC1 and AC2), and one silicone-based soft reline material (SI) were evaluated. Tensile bond strength was measured after 24 h of water storage (TC0) or 5,000 thermal cycles (TC5000) (n = 20). A significant four-factor interaction demonstrated that bond strength depended on the denture base resin, reline material, surface treatment, and thermal-cycling condition (P < 0.05). The hard reline material showed significantly higher bond strength than all soft materials (P < 0.05). For the 3DP resin, 4-META yielded the highest bond strength for AC1 under both aging conditions (P < 0.05) and improved bonding of HR and AC2 relative to untreated specimens (P < 0.05). In contrast, the silicone-based reline material exhibited limited responsiveness to surface treatment, and thermal cycling generally reduced bond strength. Bond durability between denture base and reline materials was influenced by the interaction among resin type, reline material, surface treatment, and thermal aging. Among the evaluated surface treatments, 4-META improved bonding for acrylic-based reline materials on the tested 3DP. The surface treatment protocols should be tailored to the specific denture base–reline material combination and anticipated aging condition.

1. Introduction

Digital technology has expanded the options for fabricating complete dentures, including the storage and reuse of design data as well as modifications to clinical and laboratory workflows [1–3]. CAD-CAM complete denture bases can be produced by subtractive milling or additive manufacturing [3, 4]. Studies have compared the fit of milled and 3D-printed denture bases [5–7], as well as the mechanical properties of denture base materials fabricated using these techniques [8, 9]. Milling removes material from a prepolymerized blank, whereas 3D printing builds the denture base layer by layer from a light-polymerizable resin. These fabrication methods differ in equipment requirements, material utilization, and processing steps [4, 10]. Their clinical outcomes and costs should therefore be evaluated within the context of the specific fabrication workflow [2, 11]. The accuracy of 3D-printed denture bases depends on the material and manufacturing conditions, including printing orientation [12–14]. Comparisons with milled and conventionally fabricated dentures should therefore be interpreted in relation to the tested system and measurement method. Randomized crossover trials have also compared patient-reported outcomes. Ohara et al. reported higher ratings for conventional dentures in several satisfaction domains, including general satisfaction [15]. In contrast, Iwaki et al. reported non-inferior oral health-related quality of life with 3D-printed dentures and no significant differences in patient satisfaction between the two fabrication methods [16]. These findings indicate that patient-reported outcomes may vary according to the fabrication system and clinical workflow. Residual alveolar ridge resorption continues during complete denture wear [17]. Changes in the denture-supporting tissues can compromise denture adaptation and may necessitate relining. Studies have evaluated the bonding of hard and soft reline

materials and repair resins to digitally fabricated denture bases [18–22]. Some tested combinations involving 3D-printed denture base materials demonstrated lower bond strengths than conventional or milled materials; however, the outcomes depended on the materials and experimental conditions [21–23]. Mechanical, chemical, and combined surface treatments have been investigated, and their effectiveness has also been shown to vary according to the treatment and resin system [21–24]. These differences limit the direct transferability of a surface-treatment protocol from one commercial resin to another. 4-META/MMA-TBB resin has also been investigated as an adhesive for thermoplastic denture base materials. Its use in combination with selected mechanical surface treatments improved bonding to autopolymerizing acrylic resin in studies of polyamide and other injection-molded thermoplastic resins [25, 26]. These findings provide a rationale for investigating 4-META/MMA-TBB in conjunction with 3D-printed denture base resins while recognizing differences in resin chemistry. Therefore, the purpose of this in vitro study was to evaluate the tensile bond strength of hard and soft reline materials to a 3D-printed denture base resin and to investigate the effects of surface treatment and thermal cycling. The results were compared with those obtained using a conventional heat-polymerized denture base resin.

2. Materials and methods

2.1 Specimens

Cubic specimens (20 × 20 × 20 mm) were prepared from a light-polymerized poly(methyl methacrylate) (PMMA) 3D-printed denture base resin (3DP; Dima Print Denture Base, Kulzer Japan, Tokyo, Japan) and a conventional heat-polymerized PMMA resin (HC; Acron, GC, Tokyo, Japan). The 3DP specimens were fabricated using a 3D printer (Cara Print 4.0, Kulzer Japan, Tokyo, Japan), whereas the HC specimens were polymerized at 70°C for 1 h followed by 100°C for 30 min. The bonding surfaces were polished with 600-grit abrasive paper to obtain uniform surfaces. A 0.5-mm-thick double-sided adhesive tape with a circular opening 4.8 mm in diameter was attached to one face of each specimen. The exposed bonding surface was subjected to one of four conditions, such as, no surface treatment (untreated), alumina sandblasting with 50-μm Al_2O_3 particles at 0.2 MPa (sandblasted; Hi-ALUMINA, Shofu, Kyoto, Japan), application of a dichloromethane primer (primed; Denture Primer, GC) and application of a slurry of 4-META/MMA-TBB resin (4-META; Superbond, Sun Medical, Shiga, Japan). The surface morphology after sandblasting and priming was examined by scanning electron microscopy (SEM; SM-IT210, JEOL, Tokyo, Japan) at ×300 magnification. One hard reline material (HR; Tokuyama Rebase III, Tokuyama Dental, Tokyo, Japan), three soft reline materials, namely, two acrylic-based soft reline material (AC1; Tissue Conditioner II, Shofu and AC2; Softliner, GC, Tokyo, Japan), and one silicone-based soft reline material (SI; SOFRELINER TOUGH Medium, Tokuyama Dental, Tokyo, Japan) were evaluated (Table 1). AC1 and AC2 are intended for short-term tissue conditioning and temporary reline. For SI, the dedicated ethyl acetate-containing bonding primer supplied with the product was applied to the bonding surface of the denture base material. After reline material was placed onto the bonding surface, a 6.0-mm-diameter acrylic rod (PMMA, Kasai, Kyoto, Japan) was then positioned on the reline material and held in place at room temperature for 5 minutes to allow complete setting (Fig. 1). The specimens were either immersed

in distilled water at 37.0°C for 24 h (TC0) or subjected to 5,000 thermal cycles between 5°C and 55°C using a thermal shock tester (TTS-1, Thomas Scientific, Tokyo, Japan) (TC5000). A total of 1,280 specimens were analyzed, consisting of 20 specimens for each of the 64 conditions created by combining two denture base materials, four reline materials, four surface treatments, and two thermal cycling conditions.

Table 1
Materials used in this study

Denture base materials	Product	Manufacturer	Material
3DP	Dima print Denture Base	Kulzer Japan, Tokyo, Japan	3D-printed denture base resin
HC	Acron	GC, Tokyo, Japan	Heat-polymerized denture base resin
Reline materials			
HR	Tokuyama Rebase III	Tokuyama Dental, Tokyo, Japan	Hard reline material
AC1	Tissue Conditioner II	Shofu, Kyoto, Japan	Acrylic-based soft reline material
AC2	Softliner	GC, Tokyo, Japan	Acrylic-based soft reline material
SI	SOFRELINER TOUGH Medium	Tokuyama Dental, Tokyo, Japan	Silicon-based soft reline material
Surface treatments			
sandblasting	HI-ALUMINA	Shofu, Kyoto, Japan	50-μm aluminum oxide particles
Priming	Denture Primer	GC, Tokyo, Japan	Dichloromethane primer
4-META	Superbond	Sun Medical, Shiga, Japan	4-META/MMA-TBB resin

2.2 Tensile test

Each specimen was mounted in a universal testing machine (SHIMADZU AG-15, Shimadzu Corporation, Kyoto, Japan), and tensile loading was applied at a crosshead speed of 2.0 mm/min. The maximum tensile stress at separation of the acrylic rod from the denture base specimen was recorded as the tensile bond strength (Fig. 2).

2.3 Statistical analysis

The effects of thermal cycling (TC0 and TC5000), surface treatment (untreated, sandblasted, primed, and 4-META), denture base material (HC and 3DP), and reline material (HR, AC1, AC2, and SI) on tensile bond strength were evaluated using a heteroscedasticity-robust full-factorial four-way linear model. Heterogeneity of variance was assessed using the Brown-Forsythe test. Omnibus effects were evaluated using Type III Wald F tests with an HC3 heteroscedasticity-consistent covariance matrix. Following identification of a significant four-way interaction, condition-specific pairwise comparisons were performed. Independent groups were compared using two-sided Welch t-tests with Welch-Satterthwaite degrees of freedom. Holm adjustment was applied separately to each family of comparisons for thermal cycling (32 comparisons), surface treatment (96 comparisons), denture base material (32 comparisons), and reline material (96 comparisons). Holm-adjusted P values < 0.05 were considered statistically significant. Mean differences and unadjusted 95% confidence intervals were also calculated. All statistical analyses were performed using SPSS Statistics (IBM Corp., Armonk, NY, USA).

3. Results

3.1 SEM observation

SEM images of the untreated, sandblasted and primed surfaces before bonding are shown in Fig. 3. After polishing with 600-grit abrasive paper, the untreated HC and 3DP surfaces differed slightly in morphology, and remnants of the layered printing structure were observed on the 3DP surface (a-1 and b-1). Sandblasted produced rougher, more irregular surfaces in both materials (a-2 and b-2). The primed surfaces appeared smoother than the untreated surfaces. This surface-smoothing effect was more apparent on HC surfaces, whereas residual surface irregularities remained visible on 3DP surfaces (a-3 and b-3).

3.2 Omnibus analysis

The Brown-Forsythe test indicated significant heterogeneity of variance among the 64 experimental conditions (F (63, 1216) = 20.199, P < 0.05). Accordingly, inference was based on the HC3-robust four-way model. The omnibus analysis revealed significant main effects, three-factor interactions, and a four-factor interaction (Table 2). Among the two-factor interactions, only thermal cycling × surface treatment was not significant (P > 0.05). The significant four-factor interaction indicated that the effects of each factor depended on the levels of the other factors; therefore, subsequent interpretation focused on condition-specific comparisons.

Table 2
Omnibus tests from the HC3 robust four-factor model

Thermal cycling	**df**	**F**	***P* value**
	1	**62.604**	**< 0.001**
Surface treatment	3	66.434	< 0.001
Denture base material	1	366.758	< 0.001
Reline material	3	1584.025	< 0.001
Thermal cycling × Surface treatment	3	0.678	0.5653
Thermal cycling × Denture base material	1	33.128	< 0.001
Thermal cycling × Reline material	3	31.483	< 0.001
Surface treatment × Denture base material	3	26.510	< 0.001
Surface treatment × Reline material	9	44.203	< 0.001
Denture base material × Reline material	3	132.854	< 0.001
Thermal cycling × Surface treatment × Denture base material	3	3.907	0.0086
Thermal cycling × Surface treatment × Reline material	9	1.946	0.0423
Thermal cycling × Denture base material × Reline material	3	11.119	< 0.001
Surface treatment × Denture base material × Reline material	9	15.133	< 0.001
Thermal cycling × Surface treatment × Denture base material × Reline material	9	4.388	< 0.001

Type III Wald F tests were conducted using sum-to-zero contrasts and an HC3 heteroscedasticity-consistent covariance matrix. The model included thermal cycling, surface treatment, denture base material, reline material, and all possible interactions. Denominator degrees of freedom were 1,216 for all tests. *P* values are two-sided

3.3 Effect of thermal cycling

Thermal cycling significantly decreased bond strength in 14 of the 32 experimental conditions, whereas no significant increases were observed (Table 3). For SI, bond strength decreased after TC5000 in all eight combinations of denture base material and surface treatment ($P < 0.05$). For HR, significant decreases were observed for HC with untreated, sandblasted, and primed surfaces and for 3DP with untreated surfaces ($P < 0.05$). No significant effects of thermal cycling were detected for AC1 under any condition ($P > 0.05$). For AC2, significant decreases were observed only for HC with untreated surfaces and for 3DP with primed surfaces ($P < 0.05$), whereas the remaining conditions showed no significant differences ($P > 0.05$).

Table 3
Tensile bond strength and condition-specific comparisons across thermal-cycling conditions and reline materials

Surface treatment	Denture base	Reline material	TC0 mean ± SD	TC5000 mean ± SD	95% CI of difference	*P* Value
untreated	HC	HR	13.587 ± 3.479 [a]	8.417 ± 3.675 [a]	-7.462, -2.880	0.0013
		AC1	0.838 ± 0.207 [b]	0.967 ± 0.467 [b]	-0.106, 0.364	1.0000
		AC2	0.830 ± 0.074 [b]	0.551 ± 0.250 [c]	-0.400, -0.158	0.0021
		SI	0.530 ± 0.225 [c]	0.150 ± 0.087 [d]	-0.491, -0.269	< 0.001
	3DP	HR	4.884 ± 1.422 [a]	2.954 ± 1.407 [a]	-2.836, -1.025	0.0026
		AC1	0.345 ± 0.142 [c]	0.396 ± 0.221 [c]	-0.069, 0.170	1.0000
		AC2	0.698 ± 0.187 [b]	0.672 ± 0.311 [b]	-0.191, 0.140	1.0000
		SI	0.297 ± 0.117 [c]	0.168 ± 0.075 [d]	-0.193, -0.066	0.0049
sandblasting	HC	HR	14.141 ± 3.775 [a]	9.609 ± 3.284 [a]	-6.798, -2.265	0.0052
		AC1	0.959 ± 0.212 [b]	1.015 ± 0.444 [b]	-0.170, 0.281	1.0000
		AC2	1.109 ± 0.173 [b]	0.843 ± 0.366 [b]	-0.452, -0.080	0.1198
		SI	0.301 ± 0.097 [c]	0.135 ± 0.065 [c]	-0.220, -0.114	< 0.001
	3DP	HR	8.140 ± 2.840 [a]	7.903 ± 2.029 [a]	-1.822, 1.348	1.0000
		AC1	0.796 ± 0.193 [c]	0.848 ± 0.282 [b]	-0.104, 0.207	1.0000
		AC2	1.074 ± 0.193 [b]	1.072 ± 0.459 [b]	-0.231, 0.228	1.0000
		SI	0.330 ± 0.122 [d]	0.143 ± 0.086 [c]	-0.255, -0.119	< 0.001
priming	HC	HR	21.786 ± 4.296 [a]	14.514 ± 5.306 [a]	-10.367, -4.177	< 0.001

Surface treatment	Denture base	Reline material	TC0 mean ± SD	TC5000 mean ± SD	95% CI of difference	*P* Value
		AC1	1.483 ± 0.371 [b]	1.219 ± 0.454 [b]	-0.530, 0.002	0.7201
		AC2	1.068 ± 0.346 [c]	1.108 ± 0.432 [b]	-0.211, 0.291	1.0000
		SI	0.317 ± 0.124 [d]	0.170 ± 0.086 [c]	-0.215, -0.078	0.0028
	3DP	HR	4.652 ± 2.170 [a]	6.862 ± 2.806 [a]	0.601, 3.819	0.1357
		AC1	0.388 ± 0.192 [c]	0.528 ± 0.365 [b]	-0.048, 0.329	1.0000
		AC2	0.711 ± 0.203 [b]	0.450 ± 0.284 [b]	-0.420, -0.102	0.0385
		SI	0.255 ± 0.090 [c]	0.134 ± 0.063 [c]	-0.170, -0.070	< 0.001
4-META	HC	HR	18.613 ± 4.280 [a]	14.424 ± 5.100 [a]	-7.207, -1.173	0.1325
		AC1	1.536 ± 0.515 [b]	1.776 ± 0.625 [b]	-0.127, 0.607	1.0000
		AC2	1.115 ± 0.409 [b]	1.344 ± 0.494 [b]	-0.062, 0.519	1.0000
		SI	0.547 ± 0.210 [c]	0.159 ± 0.088 [c]	-0.493, -0.284	< 0.001
	3DP	HR	12.522 ± 3.436 [a]	9.415 ± 4.147 [a]	-5.548, -0.666	0.2102
		AC1	1.290 ± 0.250 [b]	1.406 ± 0.411 [b]	-0.103, 0.336	1.0000
		AC2	1.346 ± 0.413 [b]	1.343 ± 0.380 [b]	-0.256, 0.252	1.0000
		SI	0.285 ± 0.083 [c]	0.190 ± 0.083 [c]	-0.149, -0.042	0.0164

Values are presented as mean ± standard deviation (MPa) (*n* = 20 per group). Within each fixed combination of surface treatment, denture base material, and thermal-cycling condition, reline materials sharing the same lowercase letter in brackets do not differ significantly (Welch's *t*-tests with Holm adjustment across all 96 reline-material comparisons). Letter annotations should not be compared across thermal-cycling conditions, surface-treatment groups, or denture-base materials. The 95% confidence intervals represent unadjusted confidence intervals for the mean difference calculated as TC5000 − TC0. Adjusted *P* values are based on two-sided Welch's *t*-tests with Holm adjustment across

all 32 thermal-cycling comparisons. TC0 = without thermal cycling, TC5000 = with thermal cycling, HC = conventional heat-polymerized denture base resin, 3DP = 3D-printed denture base resin, HR = hard reline material, AC1 and AC2 = acrylic-based soft reline materials, SI = silicone-based soft reline material

3.4 Effect of surface treatment

Surface-treatment effects depended on the denture base material, reline material, and thermal-cycling condition. All differences described below were significant after Holm adjustment across the 96 surface-treatment comparisons (adjusted $P < 0.05$).

For HR bonded to HC at TC0, primed produced higher bond strength than untreated and sandblasted specimens, and 4-META produced higher bond strength than untreated specimens ($P < 0.05$). After TC5000, primed and 4-META produced higher bond strength than untreated specimens ($P < 0.05$). For HR bonded to 3DP at TC0, 4-META produced higher bond strength than all other treatments, and sandblasted produced higher bond strength than untreated and primed specimens ($P < 0.05$). No significant difference was detected between primed and untreated ($P > 0.05$). After TC5000, sandblasted, primed, and 4-META produced higher bond strength than untreated specimens ($P < 0.05$), with no significant differences among these three treatments ($P > 0.05$). For AC1 bonded to HC at TC0, primed and 4-META produced higher bond strength than untreated and sandblasted specimens ($P < 0.05$). After TC5000, 4-META produced higher bond strength than untreated and sandblasted specimens ($P < 0.05$). For AC1 bonded to 3DP at TC0, 4-META produced higher bond strength than all other treatments, and sandblasted produced higher bond strength than untreated and primed specimens ($P < 0.05$). After TC5000, 4-META again produced higher bond strength than all other treatments, whereas sandblasted produced higher bond strength than untreated specimens ($P < 0.05$). No significant difference was detected between primed and untreated at either thermal-cycling condition ($P > 0.05$). For AC2 bonded to HC at TC0, sandblasted produced higher bond strength than untreated specimens ($P < 0.05$). After TC5000, primed and 4-META produced higher bond strength than untreated specimens, and 4-META also produced higher bond strength than sandblasted specimens ($P < 0.05$). For AC2 bonded to 3DP at both TC0 and TC5000, 4-META produced higher bond strength than untreated and primed specimens, and sandblasted produced higher bond strength than primed specimens ($P < 0.05$). Sandblasted also produced higher bond strength than untreated specimens at TC0 ($P < 0.05$). No significant difference between 4-META and sandblasted was detected at either thermal-cycling condition ($P > 0.05$). For SI bonded to HC at TC0, untreated and 4-META showed higher bond strength than sandblasted and primed specimens ($P < 0.05$). No significant difference was detected between untreated and 4-META or between sandblasted and primed ($P > 0.05$). No significant surface-treatment differences were detected for SI bonded to HC after TC5000 or to 3DP at either thermal-cycling condition ($P > 0.05$).

3.5 Effect of denture base material

HC exhibited significantly higher bond strength than 3DP in 15 of the 32 corresponding conditions ($P < 0.05$). The influence of denture base material was most pronounced for HR and least evident for SI. For HR, HC showed significantly higher bond strength than 3DP under all surface-treatment conditions at

TC0 ($P < 0.05$). After TC5000, HC maintained higher bond strength in the untreated, primed, and 4-META groups ($P < 0.05$), whereas no significant difference was observed in the sandblasted group ($P > 0.05$). For AC1, HC exhibited significantly higher bond strength in the untreated and primed groups regardless of thermal cycling ($P < 0.05$). No significant differences were observed in the sandblasted or 4-META groups ($P > 0.05$). For AC2, HC showed significantly higher bond strength only in the primed groups at both TC0 and TC5000 ($P < 0.05$). No significant differences were detected under the remaining surface-treatment conditions ($P > 0.05$. For SI, HC demonstrated significantly higher bond strength than 3DP only in the untreated and 4-META groups at TC0 ($P < 0.05$). No significant differences were observed in the remaining conditions ($P > 0.05$).

3.6 Effect of reline material

Of the 96 reline-material comparisons, 84 were significant after Holm adjustment ($P < 0.05$). Overall, HR exhibited the highest bond strength, whereas SI exhibited the lowest. HR showed significantly higher bond strength than AC1, AC2, and SI in all 16 combinations of denture base material, surface treatment, and thermal cycling ($P < 0.05$). In addition, AC2 showed significantly higher bond strength than SI in all 16 conditions ($P < 0.05$), whereas AC1 exceeded SI in 14 of the 16 conditions ($P < 0.05$). No significant differences between AC1 and SI were observed for untreated or primed 3DP specimens at TC0 ($P > 0.05$). Differences between AC1 and AC2 depended on the denture base material. For HC, AC1 showed significantly higher bond strength than AC2 with primed specimens at TC0 and untreated specimens at TC5000 ($P < 0.05$), whereas no significant differences were observed in the remaining six conditions ($P > 0.05$). For 3DP, AC2 showed significantly higher bond strength than AC1 with untreated, sandblasted, and primed specimens at TC0 and with untreated specimens at TC5000 ($P < 0.05$), whereas no significant differences were observed in the remaining four conditions ($P > 0.05$).

4. Discussion

This study evaluated the tensile bond strength of one hard reline material and three soft reline materials to a conventional heat-polymerized denture base resin and a 3D-printed denture base resin after four surface treatments, with and without thermal cycling. The significant four-factor interaction indicated that the influence of each factor varied according to the levels of the remaining factors. Consequently, condition-specific comparisons were considered more informative than interpretation of the main effects alone. Therefore, the main effects should not be interpreted in isolation. HR consistently produced the highest bond strength, the benefit of surface treatment was concentrated mainly in HR and selected acrylic-based soft reline materials, and thermal cycling reduced bond strength particularly for SI. Continued residual ridge resorption provides a clinical context for the need to maintain denture adaptation [17]. Previous studies have reported lower bond strengths for some combinations involving 3D-printed denture bases than for conventional or milled denture bases [18–23]. The present results were broadly consistent with previous observations, although several differences were also identified. Differences between HC and 3DP were evident primarily in HR and less consistently observed in the soft reline materials. Differences in polymer-network structure and surface chemistry may contribute to the

variation in bonding performance among the tested materials. However, the composition and network structure of the tested resin were not characterized in this study. Studies using other printable denture base systems have shown variable responses to chemical treatment, mechanical roughening, and artificial aging depending on the material tested [18, 21–23, 27]. Surface treatment generally improved HR bonding; however, no single treatment was consistently optimal across all denture base materials and aging conditions. The response depended on both the substrate and the thermal-cycling condition, which is consistent with previous reports describing material-specific effects of mechanical and chemical surface modification [21–24]. The effectiveness of a given primer should not be assumed across different printable resin systems. The SEM observations supported these findings. Sandblasting produced a rougher and more irregular surface morphology in both HC and 3DP, suggesting that mechanical roughening may have contributed to the increased bond strength observed in several material combinations. In contrast, priming produced a more pronounced surface-smoothing effect on HC, whereas residual surface irregularities remained visible on 3DP. This difference may partly explain why priming tended to be more effective for HC than for 3DP in several reline-material combinations. The use of 4-META as an adhesive for denture base materials is supported by previous studies of thermoplastic resins. Shimpo et al. reported improved tensile bonding of autopolymerizing PMMA to alumina-blasted polyamide after application of 4-META resin, although bond strength decreased after thermocycling [25]. Hamanaka et al. found that tribochemical silica coating followed by 4-META produced the highest post-thermocycling shear bond strengths to the tested polyamides, whereas alumina abrasion combined with 4-META was most effective for the tested polyethylene terephthalate copolymer and polycarbonate [26]. These results support the use of 4-META as part of a substrate-specific bonding approach. However, those studies evaluated thermoplastic denture base materials rather than photopolymerized printable resins. For the tested 3DP resin, 4-META improved bonding performance for HR and the acrylic-based reline materials, with the most consistent effect observed for AC1. Although surface infiltration may have contributed to the observed increase in bond strength, the present study did not evaluate viscosity, wetting behavior, or penetration depth. HR consistently demonstrated superior bonding performance relative to the tested soft reline materials. However, comparisons among reline materials should be interpreted cautiously because of their different compositions, mechanical properties, and clinical indications. AC1 and AC2 are tissue-conditioning materials intended for short-term use rather than long-term soft denture liners. Therefore, their results after 5,000 thermal cycles describe their response to this experimental aging protocol and should not be taken as evidence of suitability for long-term clinical service. This distinction also matters when comparing them with HR and SI. In contrast, SI showed limited responsiveness to the evaluated surface treatments. Because the manufacturer's dedicated primer was applied to all SI specimens, the present findings reflect the additional contribution of surface treatment within an existing bonding protocol rather than the isolated effect of denture-base surface modification. Therefore, these results should not be generalized to other silicone liners or primer systems. Without failure-mode data, these values cannot establish whether failure occurred at an interface, within the liner, or by a combination of these mechanisms. Thermal cycling exerted a material-dependent influence on bond strength. The greatest reductions were observed for SI, whereas AC1 appeared comparatively resistant to the aging protocol

employed in the present study. Similar effects of hydrothermal aging on bond strength have been reported previously [27]. However, Janyaprasert et al. found that silicone-based soft reline material had the highest tensile bond strength and allylic-based soft reline material the lowest among their tested liners [28], whereas SI had lower values than AC2 in the present study. Differences in denture base resin, specimen configuration, liner thickness, primer application, loading rate, and failure mode may contribute to this discrepancy; their individual contributions cannot be determined by comparing the two studies. The present SI results should therefore not be generalized to all silicone liners or bonding protocols. From a clinical perspective, Among the evaluated surface treatments, 4-META demonstrated the most consistent positive effect for the tested 3D-printed denture base resin, particularly when combined with acrylic-based reline materials. For HR, several surface-treatment strategies, including priming, sandblasting, and 4-META application, may improve bonding performance; however, their effectiveness depends on the denture base material and aging condition. Protocols established for conventional PMMA should be verified before use with a 3D-printed denture base. The SI findings support further evaluation of the complete bonding protocol, including the dedicated primer, rather than attributing the results to the denture-base surface alone. The findings for the short-term tissue conditioners should be interpreted according to their intended duration of use. Whether the observed differences in bond strength are associated with long-term clinical outcomes remains unclear. This study has several limitations. Only one heat-polymerized resin, one 3D-printed denture base resin, and four reline materials were evaluated. The findings may vary depending on resin formulation, printing orientation, layer thickness, post-polymerization protocol, and the primer system associated with each product. Furthermore, the use of flat specimens subjected to uniaxial tensile loading does not replicate the complex geometry or the combined tensile, shear, peel, and fatigue stresses encountered in clinical dentures. Failure modes were not analyzed; therefore, the relative contributions of interfacial and cohesive failure to the measured bond strength could not be determined. Although thermal cycling simulated one aspect of material aging, other clinically relevant factors, including cyclic mechanical loading, denture cleansers, saliva, pH fluctuations, and long-term intraoral service, were not considered. Consequently, the present bond-strength values should not be interpreted as direct predictors of clinical performance.

Within these limitations, bonding performance was influenced by the combined effects of denture base material, reline material, surface treatment, and thermal aging, highlighting the complexity of achieving durable bonding to both conventional and 3D-printed denture base resins. Because no surface-treatment strategy was consistently optimal across all material combinations and aging conditions, treatment selection should be tailored to the specific clinical situation. Additional studies using different printable resin systems and more clinically relevant test conditions are needed to confirm the generalizability of the present findings.

5. Conclusions

Within the limitations of this in vitro study, bond strength was influenced by the interaction among denture base material, reline material, surface treatment, and thermal cycling. For the tested 3D-printed

denture base resin, 4-META generally improved the bond strength of HR, AC1, and AC2 compared with untreated specimens under both thermal-cycling conditions, with the most pronounced effect observed for AC1. Thermal cycling significantly reduced bond strength in 14 of the 32 conditions, including all SI conditions, whereas no significant increases were observed.

Declarations

Funding

No funding was received for conducting this study.

Competing Interests

The authors declare no competing Interests related to this study.

Ethics Approval

Ethics approval was not required because this in vitro study did not involve human participants or animals.

Informed Consent

Informed consent was not required because no human participants were involved.

Data Availability

Data generated or analyzed in this study are available from the corresponding author upon reasonable request.

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Author Contribution

Shimpo H conceived the study, performed the experiments, analyzed the data, and wrote the manuscript. Shibata S, Yamazaki M, Masumoto M, Shirai M performed the experiments, analyzed the data. Ohkubo C supervised the study and critically revised the manuscript. All authors read and approved the final manuscript.

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Figures



Figure 1

Specimen preparation

After surface treatment, a hard or soft reline material was applied within the circular opening, and a 6.0-mm-diameter acrylic rod was positioned and held until the material had set

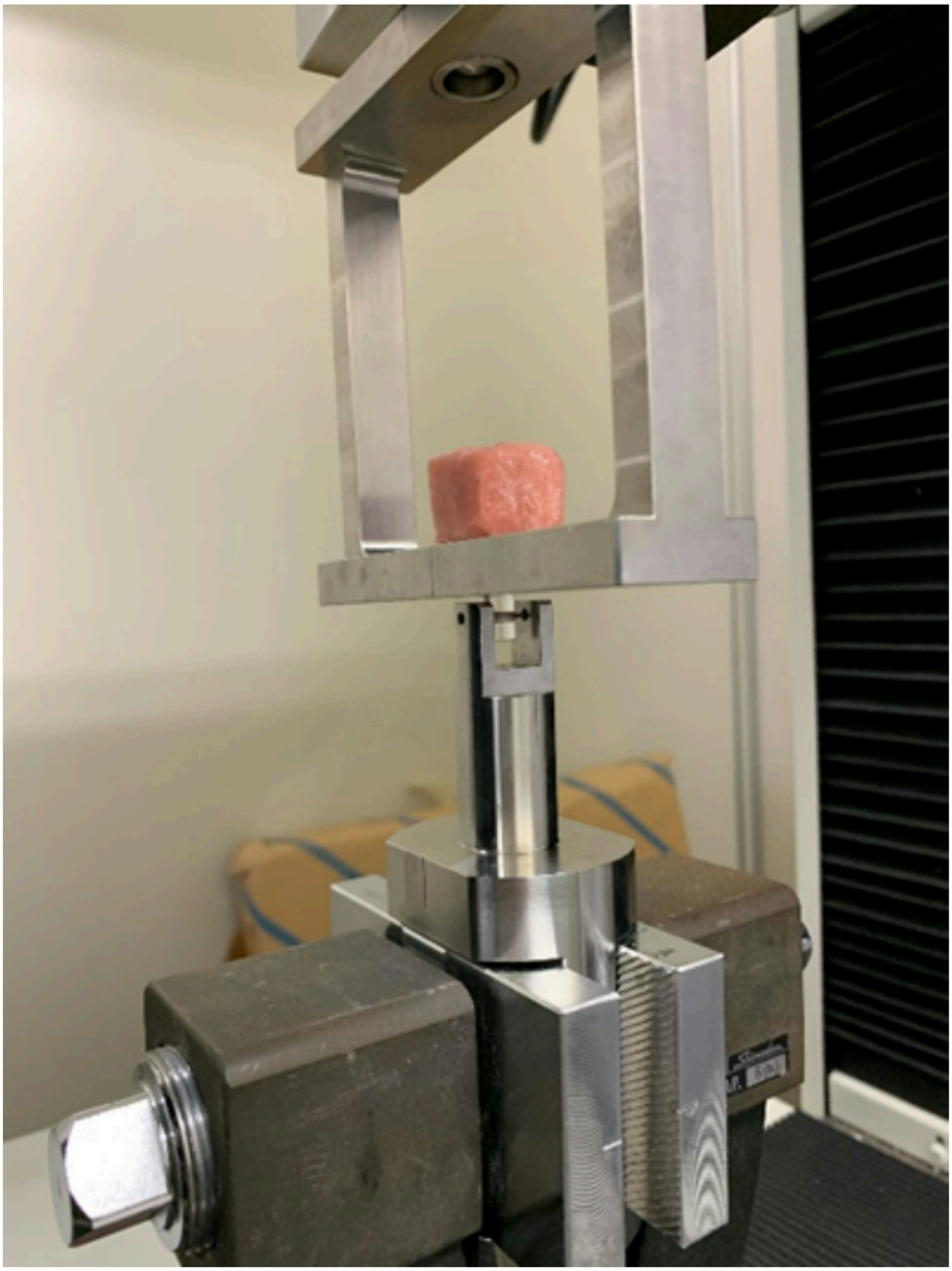

Figure 2

Measurement of tensile bonding strength

After the specimens were placed in a universal testing machine, a tensile test was performed at a crosshead speed of 2.0 mm/min

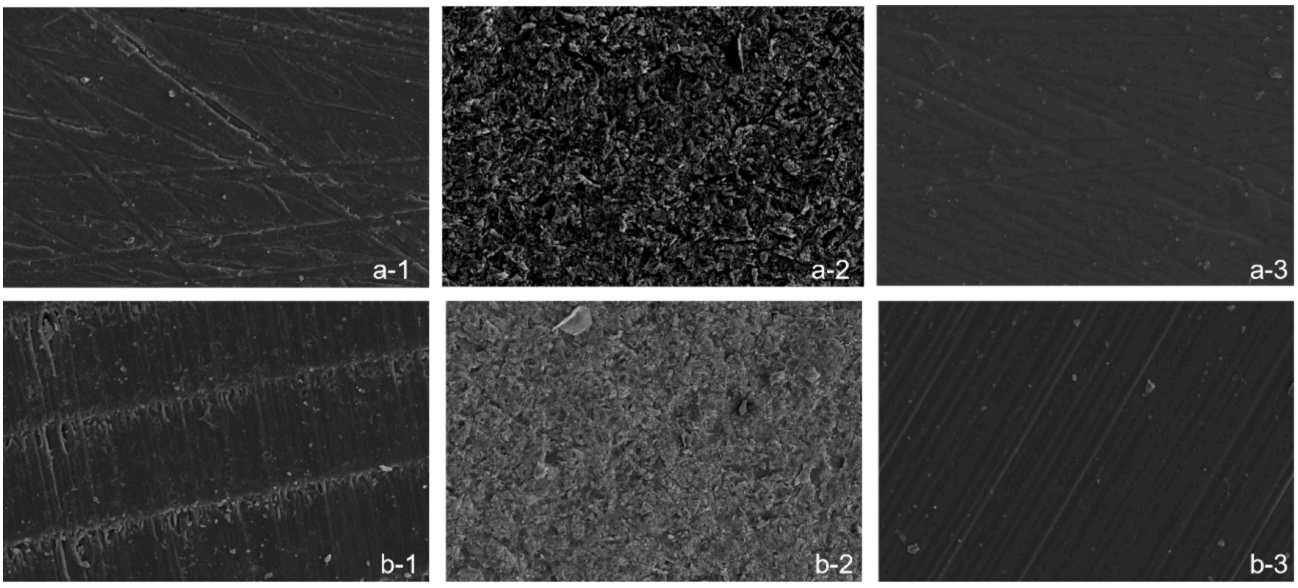

Figure 3

SEM observations after surface treatments (×300)

a-1: HC-untreated, a-2: HC-sandblasted, a-3: HC-primed, b-1: 3DP-untreated, b-2: 3DP-sandblasted, b-3: 3DP-primed

Supplementary Files

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