Dental Materials Journal 26(1): 89−95, 2007

Effect of Degradation of Denture Base Resin on Bond Strength to Relining Resins

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Recieved August 4, 2006/Accepted October 13, 2006

The purpose of this study was to evaluate the effect of degradation of a denture base resin on bond strength to relining resins. Denture base resin specimens were immersed under various conditions to simulate degradation. Tensile bond strength of denture base resin after immersion to relining resins was measured with or without surface grinding. The bond strength of denture base resin without grinding after immersion to relining resin was significantly lower than that before immersion. However, bond strength was improved by surface grinding before bonding. These results suggest that bonding efficiency of denture base resin to relining resins was affected by immersion condition, which caused potential degradation of the denture base resin.

Keywords: Denture base resin, Relining resin, Bond strength

INTRODUCTION

The relining of denture bases is often carried out to improve the fit and stability of dentures in the clinic[1]. In particular, the direct relining method using a chemical-cured acrylic resin is inexpensive and not time-consuming compared to indirect relining or making a new denture[1,2]. However, one common problem with the direct relining resin is bonding failure[2,3]. A weak bond between the relining resin and denture base resin could result in delamination between the two materials, a harbor for bacteria, promoting staining[1-4] and causing poor mechanical strength of the relined denture[5]. Therefore, bonding between relining resin and denture base resin is an important factor in obtaining a good prognosis.

Mechanical surface treatment by roughening the surface of resins[6] and chemical surface treatment with chloroform[1], dichloromethane[6], and methyl methacrylate (MMA) monomer[1] have been reported to have positive effects on bonding between denture base and relining resins. However, the denture base resins used in those studies were freshly prepared. In practical situations, most dentures that require relining have been used for a long time. In other words, these in-use dentures are exposed to saliva, oil, protein, *etc.*, as well as being subjected to thermal changes at mealtimes. Therefore, denture usage would inevitably lead to degradation of denture base resins and reduce the penetration ability of relining resin monomers. As a result, the bonding strength of these dentures may decrease.

The purpose of this study, therefore, was to evaluate the effects of degradation of a denture base resin on bond strength to relining resins. Moreover, changes in the physical and mechanical properties of the denture base resin after storage under various conditions were measured to investigate the degradation of denture base resin.

MATERIALS AND METHODS

Physical and mechanical properties of denture base resin after storage in various environments

Sorption, solubility, color change, tensile strength, and amounts of residual MMA and benzoyl peroxide (BPO) in the denture base resin after storage under various conditions were measured.

A heat-cured denture base resin (Acron Pink, GC, Tokyo, Japan) was used (Table 1). The powder/liquid (P/L) ratio of the denture base resin was 100 g/43 ml, and the resin was cured in a water bath according to the manufacturer's instructions.

Four different conditions were selected for simulating the oral environment: distilled water (DW) at 37℃, thermocycling (TC) in distilled water at 5 and 55℃ with a dwell time of 60 seconds, olive oil (Nisshin Oillio, Tokyo, Japan; OL) at 37℃, and milk (Nippon Milk Community, Tokyo, Japan; MI) at 4℃.

Sorption and solubility tests were performed according to ISO 1567:1999[7]. Sixteen disk-shaped specimens (50 mm in diameter and 0.5 mm in thickness) were fabricated with a stainless steel mold and placed in a desiccator at room temperature until a constant weight was reached (M_1). The weight was measured

Table 1 Materials used in the study.

Product	Manufacturer	Lot (Powder/Liquid)	Component		
			Monomer	Polymer	Primer
Heat-cured denture base resin					
Acron pink	GC	405193/0309102	MMA	PMMA	None
Acron clear	GC	10928102/03091	MMA	PMMA	None
Chemcial-cured relining resin					
Maxfit	Kuraray Medical	011193	FCMA	PEMA	Dichloromethane
RebaseII	Tokuyama Dental	F22203	AEMA	PEMA	Ethyl acetate
Kooliner	GC America	0307296	IBMA	PEMA	None

MMA: methyl methacrylate; PMMA: poly(methyl methacrylate);
FCMA: fluoride-containing methacrylate; PEMA: poly(ethyl methacrylate);
AEMA: (acetoacetoxy)ethyl methacrylate; IBMA: isobutyl methacrylate

with an analytical balance (AE240, Mettler Toledo, Greifensee, Switzerland), and the diameter and thickness were measured with a caliper (Digimatic caliper, Mitutoyo, Kanagawa, Japan) and a micrometer (Digimatic micrometer, Mitutoyo, Kanagawa, Japan), respectively, and the volume of the specimen (V) calculated. Sixteen specimens were divided into four groups according to immersion conditions. After immersion for 1 and 4 weeks, specimens were weighed again (M_2). Specimens were reconditioned in the desiccator at room temperature until a constant weight was reached (M_3). Sorption of immersion medium (Sp) and solubility (Sl) were calculated in $\mu g/mm^3$ using the following formulae:

$$Sp = (M_2 - M_3) / V$$
$$Sl = (M_1 - M_3) / V$$

To evaluate color changes, the CIELAB system (the standard of the Commission Internationale de L'Eclairage) was used as described previously[8]. A disk-shaped pattern (20 mm in diameter and 2 mm in thickness) was prepared using a stainless steel mold and a silicone impression material (Exafine, GC, Tokyo, Japan), and then embedded in a dental flask. Twenty specimens were fabricated by a conventional dental flasking technique. Excess flash of the specimens was trimmed with a carbide bar, and the specimens were polished with #400 silicon carbide (SiC) paper. Twenty specimens were divided into four groups as for the sorption test. Baseline $L^*a^*b^*$ values of all specimens were measured with a colorimeter (Minolta CR-13, Minolta, Tokyo, Japan) before storage. After 1 and 4 weeks' storage in each condition, the $L^*a^*b^*$ values were measured again. The total color change, ΔE, was calculated using the formula:

$$\Delta E = [(\Delta L^*)^2+(\Delta a^*)^2+(\Delta b^*)^2]^{1/2}$$

To measure tensile strength, 90 dumbbell-shaped specimens[9] (length, width, and thickness of 14, 2, and 2 mm, respectively) were fabricated in the above-mentioned manner using silicone impression material and a stainless steel mold. Specimens were divided into nine groups according to storage conditions: without storage (control), DW for 4, 12, 24, and 48 weeks (DW-04w, 12w, 24w, 48w); TC for 2 and 4 weeks (TC-02w, 04w); olive oil for 4 weeks (OL-04w); and milk for 4 weeks (MI-04w). After storage, the width and thickness of the central part of the specimen were measured with a micrometer. Tensile strength was determined by a tensile test performed with a micro material testing machine (MMT−250N, Shimadzu, Kyoto, Japan) in water at 37℃ with a crosshead speed of 1 mm/min.

To measure the amounts of residual MMA and BPO, 27 cylindrical-shaped specimens (5 mm in height and 10 mm in diameter) were fabricated in the above-mentioned manner using silicone impression material and a stainless steel mold. Three specimens without storage were used as a control, and the remaining 24 specimens were divided into four groups and stored in DW, TC, OL, or MI for 4 weeks. After storage, the surface layer of three specimens from each group was ground by〜0.5 mm (grinding group) and the weight of all specimens measured. Extraction of residual MMA and BPO was performed according to ISO 1567, and their amounts were determined using high-performance liquid chromatography (HPLC; Agilent 1100, Agilent Technologies Deutschland GmbH, Waldbronn, Germany). Specimens were

dissolved in 20 ml of acetone solution and agitated by magnetic stirring[10]. Then, 8 ml of methanol solution was added to 2 ml of the sample solution and centrifuged at 3,000 rpm for 15 minutes. After the supernatant was diluted with 50% acetonitrile + 50% H_2O, this diluted solution was applied to HPLC with diode array detection as injection volume being 20 μl. Analysis was performed at 40 ℃ using CAPCELL PAK C18 column (Shiseido, Tokyo, Japan) and a UV detector at 217 nm for MMA and 238 nm for BPO. As a mobile phase, a mixture of acetonitrile and 10 mM phosphate buffer (pH 3.5) was used and flow rate was 0.2 ml/min. Peaks for MMA and BPO were identified primarily by retention times regarding the standards, and diode array spectral match and peak purity were used to confirm identity. The quantity of residual MMA and BPO was determined using a linear regression equation obtained from calibration standards. Residual MMA and BPO were calculated in ng/mg for each specimen.

Effects of denture base resin storage before relining on bond strength

A clear shade of the same heat-cured denture base resin (Acron Clear, GC) and three chemical-cured relining resins [Maxfit (MF), Kuraray Medical, Tokyo, Japan; RebaseII (RE), Tokuyama Dental, Tokyo, Japan; Kooliner (KL), GC America, IL, USA] were used (Table 1). The P/L ratios of MF, RE, and KL relining resins were 2.9 g/2.0 g, 3.5 g/2.0 g, and 7.0 g/5.0 ml, respectively.

Three hundred and seventy-five dumbbell-shaped specimens were fabricated for the tensile strength test in the above-mentioned manner.

Fifteen control specimens were cut with a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) at the center, and the sectioned surface was polished with #400 SiC paper. Deposits on the specimens were removed by ultrasonic cleaning in distilled water for 5 minutes, the specimens were wiped with a clean dry towel and air-dried for 1 minute. The sectioned dumbbell-shaped specimens were placed into the dumbbell-shaped stainless steel mold. Mixed relining resin was inserted into the remaining space of the mold, covered with a slide glass, and left for 10 minutes at room temperature. Regarding MF and RE, the sectioned surface was primed with the corresponding primer before bonding. After setting of the relining material, excess flash was trimmed. In this manner, 10 bonded specimens were prepared for each relining resin.

The remaining 360 specimens were divided into eight groups. Thirty from each group of 45 specimens were cut and polished in a manner similar to the control group. Each group of 60 sectioned dumbbell-shaped and 15 dumbbell-shaped specimens was immersed in one of the storage conditions, which were identical to the tensile strength test. Thirty from 60 sectioned dumbbell-shaped specimens were used as the no mechanical treatment group, while the remaining 30 sectioned dumbbell-shaped specimens were used as the ground group. The ground group specimens were ground from the bonding surface by 0.5 mm with #400 SiC paper to remove the affected surface area before bonding with the relining resin. Then, the 15 dumbbell-shaped specimens of each group were cut and polished similar to the controls after storage and used as the cut group. Tensile bond strength was determined with the tensile strength test.

Statistical analysis

The data were statistically analyzed using one-way or two-way analysis of variance (ANOVA), and Tukey's HSD and Dunnett's multiple comparison tests at a significance level of 0.05.

Table 2 Sorption and color change of denture base resin after storage under various conditions.

Storage condition	Sorption (μg/mm^3)	Solubility (μg/mm^3)	Color difference (ΔE)
DW-01w	18.14±0.42^{a}		0.43±0.17^{d}
DW-04w	18.05±0.33^{a}	0.17±0.15^{c}	0.55±0.21^{d,e}
TC-01w	18.55±0.23^{a}		0.57±0.25^{d,e}
TC-04w	19.04±0.31^{a}	0.18±0.20^{c}	0.73±0.27^{d,e}
OL-01w	5.68±1.00^{b}		3.10±0.76^{f}
OL-04w	5.21±1.38^{b}	0.26±0.28^{c}	6.13±1.48^{g}
MI-01w	17.81±0.49^{a}		1.58±0.68^{d,e}
MI-04w	18.86±0.48^{a}	0.19±0.25^{c}	1.89±0.70^{e,f}

Data are presented as Mean ± SD.
Values with same superscript letters (a, b, c, d, e, f, g) are not significantly different (P>0.05).

Table 3 Tensile strength of denture base resin after storage under various conditions.

Storage condition	Tensile strength (MPa)
Control	47.27±2.45
DW-04w	42.83±2.24*
DW-12w	48.50±1.77
DW-24w	41.79±2.95*
DW-48w	42.63±2.54*
TC-02w	45.95±3.36
TC-04w	47.59±2.98
OL-04w	43.20±5.70*
MI-04w	44.15±2.53

Data are presented as Mean ± SD.
Values with the asterisk are significantly different from the control ($P<0.05$).

Table 4 Residual MMA and BPO of denture base resin after storage under various conditions.

Storage condition	MMA (ng/mg)	BPO (ng/mg)
Control	2033 ± 23	3134 ± 29
DW-04w (N)	2041 ± 11	2853 ± 105
DW-04w (G)	2090 ± 18	3051 ± 483
TC-04w (N)	2174 ± 103	3275 ± 173
TC-04w (G)	2329 ± 432	3109 ± 280
OL-04w (N)	1987 ± 20	2642 ± 315
OL-04w (G)	2043 ± 44	3353 ± 371
MI-04w (N)	2038 ± 16	2923 ± 142
MI-04w (G)	2039 ± 46	3681 ± 395

Data are presented as Mean ± SD.
N: Without grinding the surface; G: With surface grinding by 0.5 mm.

RESULTS

Physical and mechanical properties of denture base resin after storage in various environments

Sorption, solubility, color change, tensile strength, and amounts of residual MMA and BPO are shown in Tables 2, 3, and 4.

Sorption ranged from 5.21 to 19.04 $\mu g/mm^3$. Two-way ANOVA indicated that the storage medium had a significant effect. Sorption in OL was significantly lower than in the other media. Solubility ranged from 0.17 to 0.26 $\mu g/mm^3$. However, storage medium and period for solubility were not significant. The ΔE ranged from 0.43 to 6.13. Two-way ANOVA indicated that storage medium, storage period, and their interaction had a significant effect. OL showed a significantly greater ΔE than the other media, and a longer storage period showed higher ΔE. However, storage period did not significantly affect ΔE for the other media. Only OL showed a significantly higher ΔE, but MI showed a greater ΔE than DW and TC.

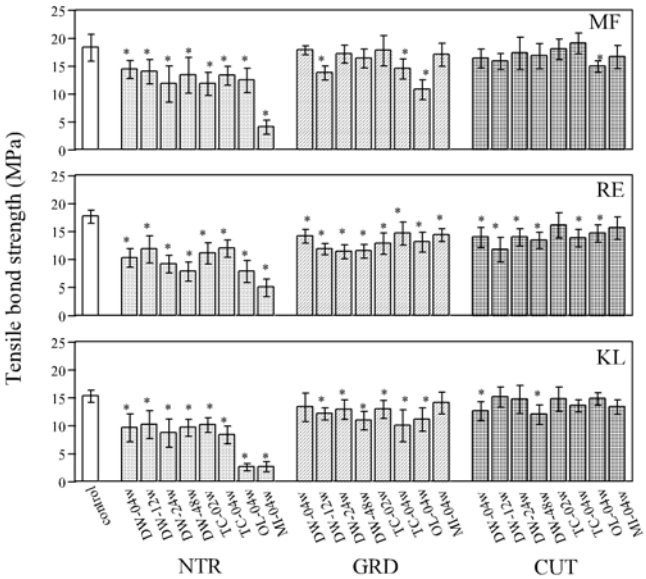

Fig. 1 Tensile bond strength of each relining resin. NTR: No mechanical treatment group; GRD: Ground group; CUT: Cut group.

Tensile strength ranged from 41.79 to 48.50 MPa. The tensile strength in the DW-04w, DW-24w, DW-48w, and OL-04w groups were significantly lower than that of the control.

The amounts of residual MMA and BPO ranged from 1987 to 2090 ng/mg and 2642 to 3681 ng/mg, respectively. MMA and BPO in groups with grinding were greater than those without grinding. Two-way ANOVA, except for control, and selecting factors as the storage medium and mechanical treatment, revealed that residual BPO was significantly affected by mechanical treatment, but which was not so for residual MMA.

Effects of denture base resin storage before relining on bond strength

The fracture surface of all specimens showed adhesive failure by macroscopic examination. The results of the tensile bond test are shown in Fig. 1. The bond strength of MF, RE and KL relining resin was 4.1—19.09, 4.97—17.65 and 2.61—15.29 MPa, respectively. In the group without mechanical treatment, the bond strength of all storage conditions for each relining resin was significantly lower than that of the control group. Then, for all storage conditions, bond strength was improved by grinding, but nonetheless still significantly lower than the control group. In the cut group, bond strength was improved and closer to the control group. However, for some

storage conditions, bond strength was still significantly lower than the control group for RE, MF, and KL.

DISCUSSION

To evaluate the effect of degradation of a denture base resin on bond strength to relining resins, we first investigated degradation of denture base resin by evaluating changes in the physical and mechanical properties of the denture base resin after immersion in various conditions. Distilled water, olive oil, milk, and thermocycling were selected as the simulated oral environments in this study, because the dentures in use are exposed to saliva, oil, protein, *etc.*, as well as being subjected to thermal changes.

Physical and mechanical properties of denture base resin after storage in various environments

Water sorption and solubility of denture base resin immersed in water for 1 week have been reported as 24.98 and 0.12 μg/mm^3 [11] respectively. The solubility in our study was similar to previous values, but water sorption was slightly lower. If immersed in water, denture base resin absorbs water and residual MMA and BPO and benzoic acid, degradation product of BPO may migrate into the water. It has been reported that water sorption mainly occurs during the initial 14 days[12], and most of the residual MMA leaches out within a few days[13]. Water absorbed into the resin acts as a plasticizer and decreases its mechanical properties[14].

ΔE values after storage in olive oil and milk were significantly greater than those in the other conditions. These results indicated that olive oil and milk were adsorbed onto the denture base resin surface. However, further study will be required to clarify how adsorbed olive oil and milk affect the surface of the denture base resins. Milk consists of ≧90% water and contains protein and lipid. Sorption of milk was the same as that of water, suggesting that the non-water components of milk have difficulty penetrating the denture base resin because of their large molecular weight. The effects of olive oil seem to be different from the other three conditions because olive oil is not hydrophilic but lipophilic. Sorption of olive oil was lower than that of the other conditions. This will be due to the large molecular weight and lipophilicity of olive oil. Specimens were cleaned by ultrasonic cleaning in distilled water, although olive oil might not be removed by this cleaning. However, further examination will be necessary to clarify how deep the non-water components of olive oil and milk penetrate into the denture base resin.

Tensile strength in the present study was higher than that previously reported for another polymethyl methacrylate-based denture base resin (36.4 MPa)[15]. Tensile strength decreased after storage in water or olive oil. Absorbed water acts as a plasticizer. Olive oil is lipophilic, thereby absorption and solubility of the denture base resin in olive oil may show different behavior from these in water, which will cause a decrease of tensile strength of the denture base resin. However, further study will be necessary to clarify the behavior of the denture base resin immersed in olive oil. Regarding thermocycling, water at 55℃ might have caused post-polymerization and compensated for a decrease in tensile strength caused by water sorption. Water absorption has been reported to reach saturation earlier with a higher water temperature[16] and in thinner specimens[17]. Milk was kept at 4℃ to prevent deterioration in this study. If the storage period with thermocycling or in milk were longer, tensile strength might indeed decrease. It should also be highlighted that although the tensile strength of denture base resin decreased after storage, it was greater than the bond strength to the relining resins. In other words, decrease in tensile strength of denture base resin will not affect the bonding strength of denture base resin to relining resins.

Effects of denture base resin storage before relining on bond strength

Tensile bond tests have the advantage that the tensile load can be distributed evenly on the bonding area between both materials[18]. The tensile bond strength between the denture base resin and KL has been reported as 20 MPa[2], which was similar to the value obtained in this study. The results of this study indicate that storage condition affected bond strength. In particular, bond strength dramatically decreased after storage in olive oil and milk, which could be attributed to olive oil or milk ingredients adsorbed on the specimen surface. The bond strength of MF and RE after storage in olive oil was higher than that in milk; but the bond strength of KL in olive oil was the same as that in milk. It should be highlighted that MF and KE were applied with their primers before bonding, which could be effective in olive but not in milk. This was because olive oil consisted of triglyceride which is lipophilic, whereby these primers might easily penetrate. On the other hand, proteins in milk might interfere with the penetration of these primers. It was speculated that adsorption of olive oil and milk occurred mainly on relatively shallow surface areas. As a result, the effect of olive oil and milk could be dramatically diminished by removing the affected surface.

Bond strength also decreased after storage in water and TC, but was improved by removing the affected surface. This could be a result of the combined effects of residual MMA elution, BPO

decomposition, and uptake of water from the surface to the inside. After removing the affected surface, amounts of residual MMA and BPO were slightly greater than that without surface removal. The difference may be clearer, if the amount of MMA and BPO were to be compared directly between the inside and the surface area of the denture base resin. Greater amount of BPO, polymerization initiator, would have enhanced the polymerization performance of the relining resin when the affected denture base resin surface was removed.

If the depth of the affected area after storage reached a uniform 1 mm from the surface, then the bond strengths of both ground and cut groups would be similar because the size of the cross-sectional area of specimen was 2×2 mm. However, the bond strength was more improved in the cut group than in the ground group. This result indicates that the immersion effects did not reach to deep areas. Nonetheless, further study is required to clarify the extent of immersion effect into the denture base resin.

Three kinds of relining resin were used in this study, all were composed of different monomer, polymer, and primer components. Bond strength of MF and KL were improved by removing the affected surface but that of RE was not improved to such a great extent. Further research is needed to elucidate the effect of components of resins and primers.

During the relining of denture bases in a clinical setting, dentists remove the surface of the denture base resin with a carbide bar to expose the fresh (non-affected) resin to eliminate contamination and to gain a rough surface. Our results suggested that the surface layer of the denture base resin was indeed deteriorated due to water absorption and adsorption of food components. Hence, removal of the surface layer of denture base resin proved to be effective in diminishing the effects of surface degradation, and thereby obtaining improved bonding with the relining resin.

Results of this study suggested that immersion in water, olive oil, and milk as well as thermocycling caused absorption of water or ingredients of the storage medium. This then led to a decrease in the tensile strength of denture base resin. Moreover, bonding efficiency of denture base resin to relining resins also decreased following immersion in these media.

CONCLUSION

Within the limitations of this study, bonding efficiency of denture base resin to relining resins was affected by immersion in water, olive oil, and milk as well as thermocycling, which caused potential degradation of the denture base resin. Immersion in these conditions caused absorption of water or ingredients of media and decrease of tensile strength of denture base resin. Removing the surface layer of the denture base resin before bonding was effective for obtaining improved bonding.

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