

Weight Loss and Surface Roughness of Hard Chairside Reline Resins After Toothbrushing: Influence of Postpolymerization Treatments

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Purpose: To evaluate the effect of 2 postpolymerization treatments on toothbrushing wear (weight loss) and surface roughness of 3 autopolymerized reline resins—Duraliner II (D) (Reliance Dental), Kooliner (K) (Coe Laboratories), and Tokuso Rebase Fast (T) (Tokuyama Dental)—and 1 heat-polymerized resin, Lucitone 550 (L) (Dentsply International). **Materials and Methods:** Specimens (40 × 10 × 2mm) of each material (n = 24) were prepared and divided into 3 groups: control (no postpolymerization treatment); water bath (immersion in water at 55°C); and microwave (microwave irradiation). Specimens were dried until constant weight was achieved and the surface roughness (Ra) was measured. Tests were performed in a toothbrush machine using 20,000 strokes of brushing at a weight of 200 g, with the specimens immersed in 1:1 dentifrice/water slurry. Specimens were reconditioned to constant weight and the weight loss (mg) and surface roughness were evaluated. Data were analyzed by 2-way analysis of variance and followed by Tukey test ($\alpha = .05$). **Results:** In the control group, the weight loss of materials D and T was lower ($P < .05$) than that of L. No differences among materials were found after postpolymerization treatments ($P > .05$). The weight loss of material T (control = 0.5 mg) was significantly increased ($P < .05$) after postpolymerization treatments (water bath = 1.9 mg; microwave = 1.8 mg). For materials K and T, the toothbrushed surface roughness was higher ($P < .05$) after microwave and waterbath postpolymerization treatments. Material L showed increased surface roughness after microwave postpolymerization treatment. **Conclusion:** The toothbrushing wear resistance of L was not superior to the reline resins. The postpolymerization treatments did not improve the toothbrushing wear resistance of the materials and produced an increased surface roughness for materials L, K, and T.

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Hard chairside reline resins often have been used to improve the fit of denture bases, thus reestablishing the retention, support, and stability of removable prostheses. It has been found that differences between the composition of these materials and the conventional autopolymerizing acrylic resins are closely

related to the final physical and mechanical properties.^{1,2} Among the mechanical properties, one concern in the selection of a hard chairside reline material should be its resistance to abrasive wear. Data from previous surveys revealed that one of the most common methods of cleaning dentures was the use of toothbrushing with dentifrice.^{3–5} In addition, removable partial denture wearers tend to clean their appliance and their natural teeth with the same abrasive paste.⁶ However, the abrasive action of this method could result in removal of the denture base material, which could affect adaptation of the base to the supporting tissues. In addition, the roughness of the resulting abraded surface could be increased,⁷ thereby facilitating the adhesion of microorganisms,^{8–10} which could result in inflammatory changes frequently observed in denture wearers.¹¹ While several studies have investigated the effect of toothbrushing on the abrasion of

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Table 1 Materials Used

Product	Manufacturer	Powder/ liquid ratio	Composition		Curing cycle
			Powder	Liquid	
Duraliner II	Reliance Dental	10 mL/7 mL	PEMA	BMA	12 min at room temperature
Kooliner	Coe Laboratories	10 mL/4 mL	PEMA	IBMA	10 min at room temperature
Tokuso Rebase Fast	Tokuyama Dental	14 g/7 mL	PEMA	MAOP 1,6-HDMA	8 min at room temperature
Lucitone 550	Dentsply International	21 g/10 mL	PMMA	MMA EDGMA	90 min at 73°C and then 100°C boiling water for 30 min

PEMA = poly(ethyl methacrylate); PMMA = poly(methyl methacrylate); IBMA = isobutyl methacrylate; BMA = butyl methacrylate; MMA = (methyl methacrylate); MAOP = β -methacryloyl oxyethyl propionate; 1,6-HDMA = (1,6-hexanediol dimethacrylate); EDGMA = (ethylene glycol dimethacrylate).

different dental resins,^{12–16} a review of the literature on acrylic resins by the authors found no information regarding the toothbrushing wear resistance of hard chairside reline materials.

It has been demonstrated that residual monomer acts as a plasticizer and could influence the properties of polymerized acrylic resins, such as creep,¹⁷ flexural strength,^{18–21} hardness,²² tensile strength,²³ and fatigue limit.²⁴ It has also been found that there is less residual monomer in heat-polymerized acrylic resins than in autopolymerized acrylic resins.^{25,26} Moreover, the level of residual monomer in autopolymerized reline material is higher in the surface layer.²⁷ The reason for this higher residual monomer content in autopolymerized acrylic resins is the low degree of conversion achieved by use of a chemical activator as opposed to that generated by heat activation.²⁸ Heat-polymerized acrylic resins have shown higher toothbrush wear resistance than autopolymerized acrylic resins.^{6,29} Therefore, it can be hypothesized that the reduction of residual monomer content could improve the overall mechanical properties of the acrylic resins, including wear resistance. Postpolymerization reactions by free radical mechanisms may contribute to the reduction of residual monomer in autopolymerized acrylic resins.³⁰ It has been observed that immersion in hot water could decrease the residual monomer content in heat-polymerized denture base materials.^{31–33} Microwave irradiation, which has been recommended for the polymerization of acrylic resins³⁴ and denture disinfection,^{35,36} could also be used as a postpolymerization treatment.³⁷ The studies of Blagojevic and Murphy³⁸ and Yunus et al¹⁸ demonstrated that microwave irradiation of a conventional acrylic resin after autopolymerization promoted a significant decrease in residual monomer content.

The aim of this study was to compare the toothbrushing abrasion resistance and surface roughness of 3 hard chairside reline resins and 1 heat-polymerizing

denture base resin. The wear abrasion resistance involved quantitative weight loss measurements, and a profilometer was used to measure the surface roughness. The hypothesis to be tested was that water bath and microwave postpolymerization heat treatments would improve the toothbrushing wear resistance of these materials without any detrimental effect on surface roughness.

Materials and Methods

Three autopolymerized acrylic resins and 1 heat-polymerized denture base resin were evaluated. The names of the resins and manufacturers, proportions of powder to liquid, composition, and polymerization conditions recommended by the manufacturers are presented in Table 1.

Twenty-four specimens of each material were fabricated from a stainless steel mold with an internal cavity of 40 × 10 × 2 mm dimensions. For the hard chairside reline resins, the mold was placed over a glass plate. The materials were proportioned and manipulated following the manufacturer's instructions, and the mix was inserted within the mold. A second glass plate was placed over the material, and pressure was applied until polymerization was complete (Table 1). For Lucitone 550 acrylic resin, silicone impression material was adapted in the stainless steel mold cavity. The specimens were then produced by investing the silicone patterns, sandwiched between 2 glass slides, in stone in a dental flask. The Lucitone 550 acrylic resin was then mixed, packed in the flasks under pressure, and processed according to the manufacturer's recommendations (Table 1). After polymerization, the specimens were bench cooled to room temperature before being removed from the mold. The sides of the specimens were then ground sufficiently to ensure close fitting in the specimen holder of the brushing ma-

chine. After finishing, the specimens were each marked with reference lines. These reference lines were used to ensure that the same area was located in the profilometer for each roughness measurement. The Lucitone 550 specimens were then immersed in water at $37 \pm 1^\circ\text{C}$ for 48 ± 2 hours.³⁹

The specimens of all materials were divided into 3 groups ($n = 8$). In the control group, specimens were left untreated. For the water bath group, specimens were submitted to postpolymerization treatment by placing them in water bath with the temperature set at 55°C . The hard chairside reline resin specimens were held at this temperature for 10 minutes, following the Duraliner II material manufacturer's recommendation to reduce the monomer taste. In addition, the results of a previous study suggested that further polymerization could be achieved by using this heat treatment.⁴⁰ The Lucitone 550 specimens were held at this temperature for 60 minutes, following the recommendation reported by Tsuchiya et al.³² Specimens of the microwave group were given postpolymerization treatment by microwave irradiation (Sensor Crisp 38, Double Emission System, Brastemp) at 650 W for 4 minutes for Duraliner II, 550 W for 5 minutes for Kooliner, 550 W for 4 minutes for Tokuso Rebase Fast, and 500 W for 3 min for material Lucitone 550. The power/time settings used for each reline material were determined in a preliminary study, which evaluated the effect of 9 different power/exposure time combinations on the flexural strength of 4 hard chairside reline materials.³⁷ The postpolymerization treatments that produced the higher mean values for flexural strength were then established for each reline material and used in this study. For Lucitone 550, the specimens were irradiated using the power/time combination suggested by Ilbay et al.⁴¹ The hard chairside reline resin specimens were submitted to the postpolymerization treatments within 30 minutes after polymerization, whereas the Lucitone 550 specimens were treated after being immersed in water at $37 \pm 1^\circ\text{C}$ for 48 ± 2 hours.³⁹ After postpolymerization treatments, the specimens were then bench cooled to room temperature.

For determination of the amount of resin removed during brushing, it was necessary to bring each specimen to constant weight.^{3,12} Therefore, the specimens of all groups were dried in a desiccator containing silica gel at $37^\circ\text{C} \pm 1^\circ\text{C}$ for 24 hours, then removed to a similar desiccator at room temperature for 1 hour. Each specimen was weighed by means of an analytic balance with an accuracy of 0.1 mg, and was considered at constant weight when the weight loss was no more than 0.5 mg after any 24-hour period. After specimens reached constant weight, the surface roughness (Ra) was analyzed with a surface roughness profilometer (Prazis, RUG-03). Three measurements were performed

Table 2 Mean Weight Loss Values (mg) (SD)

Material	Group		
	Control	Water bath	Microwave
Duraliner II	1.2 (1.0)A ^{ab}	1.1 (0.4)A ^a	1.7 (0.6)A ^a
Kooliner	2.1 (1.0)A ^{ac}	1.5 (0.7)A ^a	1.4 (0.7)A ^a
Tokuso Rebase Fast	0.5 (0.5)A ^b	1.9 (1.2)B ^a	1.8 (0.6)B ^a
Lucitone 550	2.7 (0.9)A ^c	1.6 (1.1)A ^a	2.1 (1.3)A ^a

In each row, means with identical capital letters were not significantly different ($P > .05$). In each column, means with identical superscript letters were not significantly different ($P > .05$).

in the central area of each specimen at intervals of 2.0 mm, and the average reading was designated as the intact Ra value for that specimen. The path of the diamond stylus was perpendicular to the long axis of the specimen. Resolution was $0.01 \mu\text{m}$, interval (cutoff length) was 0.8 mm, transverse length was 2.4 mm, and stylus speed was 0.5 mm/s.

The specimens were positioned in the specimen holder of the slurry bath on the brushing machine. The toothbrushing procedure involved a mechanical cross-brushing machine equipped with 5 nylon-bristled toothbrushes (Tek, Hard, Johnson & Johnson), so that 5 specimens could be brushed simultaneously. The machine was set to brush at a rate of 60 reciprocal strokes per minute, and to provide $200 \text{ g}^{42,43}$ vertical load on each specimen. A slurry comprising 1 part by weight of dentifrice (Colgate Bicarbonato de sódio, Colgate Palmolive, Divisão Kolynos do Brasil) to 1 part of deionized water was inserted into the slurry bath. The specimens were then brushed with a total of 20,000 reciprocal strokes,¹⁶ which is representative of 2 years of denture cleansing.³ A stainless steel agitating fin was fastened to the end of the brush to ensure adequate mixing, so that settling of the abrasive material would be minimized during brushing.

After brushing, the specimens were removed from the specimen holder, thoroughly rinsed, and blot dried with soft paper tissue. Each specimen was placed back on the profilometer, oriented by the reference lines, and surface roughness was again measured (abraded Ra value). The specimens were then reconditioned at constant weight by using the previously described procedures. The wear loss by weight was determined by the difference between the weight of each specimen before and after brushing.

The results of weight loss were subjected to 2-way analysis of variance (ANOVA) and Tukey test ($\alpha = .05$) to examine the influence of materials and groups. Roughness data were analyzed separately for each material using a repeated-measures 2-way ANOVA and Tukey test ($\alpha = .05$) to evaluate the influence of groups and detect differences between intact and toothbrushed surfaces.

Table 3 Mean Surface Roughness Values (μm) and SDs for All Groups

Materials	Control		Water bath		Microwave	
	Intact	Abraded	Intact	Abraded	Intact	Abraded
Duraliner II	0.13 (0.05)A	0.16 (0.07)A	0.09 (0.05)A	0.13 (0.08)A	0.12 (0.06)A	0.18 (0.07)A
Kooliner	0.05 (0.01)A	0.07 (0.04)A	0.12 (0.05)B	0.17 (0.11)B	0.10 (0.05)B	0.16 (0.13)B
Tokuso Rebase Fast	0.15 (0.02)A	0.21 (0.05)A	0.13 (0.05)A	0.34 (0.08)B	0.38 (0.06)B	0.51 (0.05)C
Lucitone 550	0.22 (0.04)A	0.41 (0.13)BC	0.29 (0.24)AC	0.55 (0.29)B	0.69 (0.31)B	1.02 (0.21)D

In each row, means with identical letters were not significantly different ($P > .05$). No comparisons were made among materials.

Results

The mean weight loss of specimens of each material after brushing is summarized in Table 2. The data relative to the influence of postpolymerization treatments on toothbrushing wear resistance indicated that for Duraliner II, Kooliner, and Lucitone 550 there were no significant differences among the 3 groups evaluated ($P > .05$). However, for Tokuso Rebase Fast, immersion in hot water and microwave irradiation resulted in increased toothbrushing abrasion ($P < .05$) compared with controls.

In comparing the results of the control group, it can be seen that the mean weight loss values of materials Duraliner II and Tokuso Rebase Fast were significantly lower ($P < .05$) than that of Lucitone 550. Table 2 also shows, for control specimens, that the mean weight loss values for Kooliner and Lucitone 550 were not significantly different ($P > .05$). After being subjected to postpolymerization treatments, there were no significant differences among the mean weight loss values for all materials evaluated ($P > .05$).

Table 3 shows the mean surface roughness values of each material before and after brushing. It can be seen that Duraliner II displayed no significant change in roughness in all groups evaluated. For Kooliner, the postpolymerization treatments resulted in a slight but significant increase ($P < .05$) in roughness compared to the control. In all groups, the surface roughnesses of Duraliner II and Kooliner were not increased after brushing. Microwave postpolymerization treatment resulted in a significant increase ($P < .05$) in surface roughness of both intact and abraded surfaces of Tokuso Rebase Fast specimens. When water bath postpolymerization was used, the abraded surfaces showed higher mean roughness values than controls ($P < .05$). In addition, the mean roughness values of the abraded surfaces were higher ($P < .05$) than those of the intact surfaces in the microwave and water bath groups. As observed for Tokuso Rebase Fast, microwave postpolymerization treatment resulted in a significant in-

crease ($P < .05$) in roughness of Lucitone 550 specimens for both intact and abraded surfaces. However, the mean surface roughness was increased by toothbrushing in all groups evaluated. Water bath postpolymerization treatment had no significant effect on the surface roughness of Lucitone 550 specimens.

Discussion

The method used in this in vitro study attempted to simulate the toothbrush/dentifrice abrasion conditions encountered in daily denture care as closely as possible by using a testing machine with well-defined parameters. The wear abrasion resistance of acrylic resins and the effect of postpolymerization treatments on this property were evaluated using quantitative weight loss measurements. This method has been used to investigate the toothbrushing wear resistance of different dental materials.^{12,13,15} Specifically for acrylic resins, the water content present in these materials can be eliminated before weighing procedures.^{3,14} Therefore, the observed weight change could be attributed to the wear occurred during brushing and related to the wear resistance of the materials evaluated. Profilometry was used to evaluate the surface roughness of the materials before and after brushing. This method produced numeric data, thus allowing comparison with previous investigations.^{8-10,13,16,43} It was expected that the postpolymerization treatments would produce an increase in the toothbrushing wear resistance of the materials evaluated. The postpolymerization treatments were performed on the basis of results reported by other studies, which indicated that heating acrylic resins by immersion in hot water^{31,32} and microwave irradiation^{18,38} promoted a reduction in the residual monomer content and an improvement in mechanical properties such as impact strength³⁸ and flexural strength.^{18-20,37} However, it was observed that the toothbrushing wear resistances of materials Duraliner II, Kooliner, and Lucitone 550 were not improved by any of the postpolymerization treatments evaluated. It seems that the

postpolymerization treatments are more effective in improving the bulk strength of the specimens rather than their surface abrasion resistance. This is supported by another study³³ in which higher hardness values were recorded at greater specimen depths after an additional cycle of polymerization via microwave energy or hot water. Another possible explanation is that because all specimens were dry stored at 37°C until they reached constant mass before toothbrushing, during this period (approximately 14 days) the residual monomer molecules of the control specimens could have diffused to the sites of active radicals, resulting in further polymerization reaction.³⁰ It can be speculated that the fall of residual monomer levels could be to the extent that, after desiccation, the control specimens then had a degree of monomer conversion in polymer similar to that induced by water bath and microwave irradiation postpolymerization treatments. As a consequence, the toothbrushing wear resistance was not significantly different among the 3 groups evaluated. Surprisingly, the mean weight loss of Tokuso Rebase Fast was increased after specimens were submitted to both water bath and microwave postpolymerization treatments. Abrasion wear is the removal of a softer material by harder asperities of a counterface. In the abrasion wear mechanism, an asperity causes ploughing resulting in plastic deformation. The deformed surface may fracture to yield wear debris. Materials that maintain a smooth surface and minimize surface friction appear to have the best wear characteristics.⁷ The postpolymerization treatments likely resulted in changes of the surface texture, thereby causing increased friction between the abrasive particles of the dentifrice and Tokuso Rebase Fast. It should be emphasized, however, that the mean weight loss values of this material were not significantly different from the other materials after the postpolymerization treatments were performed.

When control specimens were compared, Duraliner II and Tokuso Rebase Fast showed lower mean weight loss values than the heat-polymerized resin Lucitone 550, which in turn was not significantly different from Kooliner reline resin. A common assumption seems to be that autopolymerizing reline resins present lower resistance to plastic deformation than heat-polymerizing resins.²¹ This could be related to the contents of autopolymerizing acrylic resins. The liquids of Kooliner and Duraliner II materials contain isobutylmethacrylate and butylmethacrylate, respectively.¹ These monomers act as plasticizers because they increase the backbone separation of the polymer molecules through pendant groups and decrease the intermolecular interactions.² Tokuso Rebase Fast liquid contains β -methacryloyl oxyethyl propionate (39.8%), which is a monofunctional monomer with 2 esoteric bonds and

which forms long flexible polymer chains.¹ Hence, the resulting surface deformation of the autopolymerizing resins caused by the dentifrice abrasive particles would be more resilient in nature. The higher resilience of autopolymerizing resins would lead to friction reduction of the abrasive particles against the resin surface, thus resulting in less removal of material. This would help explain the lower weight loss observed for Duraliner II and Tokuso Rebase Fast compared to Lucitone 550, when the materials were not postpolymerized. Although this was not observed for Kooliner, its mean weight loss was not significantly different from that of Lucitone 550.

The mean weight loss values observed in this study were small, ranging from 0.5 mg to 2.7 mg. Sexson and Phillips,³ using similar method as in this study, evaluated various denture cleansers and observed that dentifrice promoting a mean weight loss of 3.1 mg caused a surface loss of only 0.057 mm. This change was thought to be small and may not be great enough to affect the adaptation of the denture bases, because it is expected that the tissues would compensate for such a discrepancy. Therefore, it seems reasonable to assume that all materials evaluated in the present study exhibited mean weight loss values that likely would not be clinically significant.

When surface roughness of the materials Kooliner, Tokuso Rebase Fast, and Lucitone 550 was evaluated, it was observed that the postpolymerization treatments generally increased the surface roughness of these materials. Considering that the level of residual monomer is higher in the surface layer,²⁷ some of this residual monomer could be reduced by further polymerization at the sites of active radicals during the postpolymerization treatments.^{30,31} This reduction might have occurred in localized areas, thus resulting in a heterogeneous surface on areas with higher and lower degrees of conversion. This could affect the surface condition after toothbrushing because of the selective abrasion of the material. Consequently, toothbrushing after postpolymerization treatments resulted in a more uneven abraded surface compared to controls. Despite these differences, it should be noted that no significant differences were observed in mean weight loss for Kooliner and Lucitone 550. This indicates that these 2 properties are almost certainly affected by other factors.¹⁶ The weight reduction expressed total loss of material, while roughness measurements provided information about the topography of the abraded surface.¹³ The results also demonstrated that for Lucitone 550 and Tokuso Rebase Fast, the toothbrush-abraded areas were rougher than intact areas, showing the abrasive effect of toothbrushing. This effect was more evident after microwave postpolymerization heat treatment, which promoted an

increase in surface roughness even before toothbrushing. Most likely, the rougher intact surface increased friction of the abrasive particles, thus resulting in a more uneven surface. On the contrary, the surface roughnesses of Duraliner II and Kooliner were not affected by toothbrushing. Therefore, the loss of substance was generally uniform, producing an even abrasive wear pattern. This promotes a smooth surface and reduces the effect of abrasive wear on surface topography.

Extraction of clinical relevance from an in vitro investigation is very difficult. The results of in vitro studies may not correlate well with the abrasion of denture bases with in vivo variables, such as the type of toothbrush used, toothbrushing technique, and frequency.⁶ Although the surface roughness profilometry used in the present investigation provides important information about surface characteristics of denture acrylic resins as a result of toothbrush/dentifrice abrasion,^{6,16,29,43} future studies should consider the use of a scanning electron microscope to evaluate surface texture. A more thorough understanding of how denture base and hard reline resins are affected by toothbrushing could be achieved. Nonetheless, with these obvious limitations in mind, there are a few generalities that can be stated. The clinical significance of denture base surface roughness has been emphasized by Verran and Maryan,⁸ who evaluated the retention of *Candida albicans* and observed that a significantly higher number of microorganisms were found on rough surfaces. Bollen et al⁹ reviewed the literature, including articles that evaluated surface roughness of several intraoral hard materials, and reported that some in vivo studies suggested a threshold surface roughness for bacterial retention ($R_a = 0.2 \mu\text{m}$) below which no further reduction in bacterial accumulation could be expected. The results of the present study revealed that the control specimens of the reline materials showed mean surface roughness values lower than this limit. This finding suggests that the reline resins exhibited favorable surface characteristics, with mean roughness values of such magnitude that plaque accumulation could be minimized, thus facilitating good denture hygiene. These favorable results were observed for Duraliner II and Kooliner even after they were subjected to postpolymerization treatments and toothbrushing abrasion. Conversely, the surface roughness of the material Tokuso Rebase Fast was increased by the postpolymerization treatments, with mean values higher than the threshold of $0.2 \mu\text{m}$. In addition, the denture base resin Lucitone 550 showed the highest mean surface roughness values in all groups evaluated. This indicated that the higher surface roughness of these materials should be taken into consideration when denture hygiene instructions are given to patients.

Nevertheless, the range of mean surface roughness values observed in this study is smaller than reported by Zissis et al,¹⁰ who found that the hard reline resins exhibited mean values ranging from 0.7 to $4.4 \mu\text{m}$.

Conclusions

Within the limits of this in vitro study, the following conclusions can be drawn:

1. The toothbrushing wear resistance of the materials evaluated was not improved by water bath or microwave irradiation postpolymerization treatments.
2. The toothbrushing wear resistance of the hard chairside reline resins Kooliner, Duraliner II, and Tokuso Rebase Fast was not different from that of the denture base Lucitone 550.
3. For Kooliner, Lucitone 550, and Tokuso Rebase Fast, the postpolymerization treatments resulted in increased surface roughness.

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