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Research Article

Surface Hardness of Acrylic Resins Exposed to Toothbrushing, Chemical Disinfection and Thermocycling

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Abstract

The aim of this study is to investigate the effect of the toothbrushing, chemical disinfection and thermocycling treatments on the surface hardness of acrylic resins used for denture bases. The Classico (conventional), Onda Cryl (microwaved) and QC-20 (boiled) thermoactivated acrylic resins were used to prepare specimens. Prior to and after cleaning procedures, the specimens were submitted to a hardness test in an indenter (HMV-2000 Shimadzu), employing a load of 50 gf for 10 s. Three-way ANOVA and Tukey's test ($\alpha=0.05$) were used to analyze the data. Prior to thermocycling, statistical differences were found among treatments for Onda Cryl, with greater hardness values observed for Efferdent and hypochlorite; lower hardness value was obtained after toothbrushing, and

intermediate value was found for the control. Differences were found among resins for the control treatment, with higher value for QC-20 resin, lower value for the Classico, and intermediate value for the Onda Cryl. Following thermocycling, differences were observed among treatments for Onda Cryl, with control specimens presenting higher values, lower value for toothbrushing and intermediate values for Efferdent and hypochlorite. Differences were also found for QC-20, with Efferdent and hypochlorite presenting higher values, lower value for control and intermediate value for toothbrushing. Differences were found among resins for the control treatment, with higher value for Onda Cryl resin, lower value for the QC-20, and intermediate value for the Classico. The QC-20 resin showed higher value for control prior to thermocycling and Onda Cryl after thermocycling. Different effects on the surface hardness of

acrylic resins used for denture bases were observed when associating thermocycling with toothbrushing or chemical disinfections.

Keywords: Acrylic resin, toothbrushing, chemical disinfection, surface hardness, thermocycling.

Introduction

A classic study demonstrated that pathogenic microorganisms can be easily transmitted to patients after denture polishing (Kahn et al., 1982). The literature has shown that a 0.5% sodium hypochlorite solution is an effective denture cleanser for reducing microorganisms (Porta-Souza et al., 2013), and it is also indicated for disinfecting denture liners and tissue conditioners, preventing oral candidiasis (Skupien et al., 2013). Conversely, it has been suggested that alkaline peroxide tablets decrease the number of *C. albicans* colony-forming units on maxillary dentures (Uludamar et al., 2010), and this cleaning solution is also considered to be an effective remover of bacterial plaque (Rossato et al., 2011). Therefore, the choice of appropriate methods for denture cleaning is clinically important, when the

objective of the procedures is not cause surface damage of the denture base, and for daily use to prevent microbial adhesion.

It has been claimed that the correlation between insufficient cleanliness of the dentures and denture stomatitis is statistically significant. The prosthesis cleaning procedure is important since the removal of biofilm is clinically necessary to maintain the oral health of denture wearers (Kulak-Ozkan et al., 2002). A previous study indicated that denture polishing is essential for cleaning procedures to minimize the adherence and colonization of microorganisms on the denture base (Radford et al., 1998). The effects of different dentifrices differ according to the polymerization mode of the acrylic resins (Haselden et al., 1998), and fungal adherence depends on the quality of the polymeric material (He et al., 2005).

The denture base hardness is a property that is usually related to the surface characteristics of acrylic resins, and has been used to evaluate the changes resulting from toothbrush/dentifrice abrasion (Richmond et al., 2004), denture cleansers (Durkan et al., 2013), polymerization cycles (Consani et al., 2012), different systems of denture base polymerization (Ali et al., 2008), and thermal cycling (Goiato et al., 2013). Different acrylic resins present significantly lower hardness after being submitted to solutions of 4% chlorhexidine gluconate, 3.78% sodium perborate or 1% sodium hypochlorite, regardless of the disinfectant used (Neppelenbroek et al., 2005). Previous studies have shown that the hardness of a conventional denture base polymer is not significantly changed by immersion in 1% sodium hypochlorite or 4% chlorhexidine solutions for 1 minute (Azevedo et al., 2006), and in sodium perborate at 50°C for 10

minutes (Machado et al., 2009). Conversely, the hardness of other commercial types of denture base acrylic resins are reported to be altered by solutions of 1% sodium hypochlorite, 2% glutaraldehyde or 4% chlorhexidine (Carvalho et al., 2012). It is possible that decreased surface hardness of acrylic resins due to cleaning and disinfecting treatments can contribute to the formation of biofilm and microorganism adherence on the dentures.

Based on these considerations it would be appropriate to evaluate the effect of the toothbrushing and chemical disinfection (Efferdent or 0.5% sodium hypochlorite) on the surface hardness of acrylic resins (conventional, microwaved or boiled), submitted to thermocycling (5°C and 55°C). The hypothesis of this *in vitro* study was that thermocycling associated with mechanical or

chemical cleanings can cause different effects on the surface hardness of acrylic resins used for denture bases.

Materials and Methods

Conventional (Classico; Classico Dental Products, Sao Paulo, SP, Brazil), microwaved (Onda Cryl; Classico) and boiled (QC-20; Dentsply, Petropolis, RJ, Brazil) heat-polymerized acrylic resins were used to manufacture rectangular samples (25 mm-length, 14 mm-width and 3 mm-height). Silicone molds were conventionally included with type III dental stone (Herodent; Vigodent/Coltene, Rio de Janeiro, RJ, Brazil) in metallic and plastic flasks, while the acrylic resins were conventionally pressed in the silicone molds.

A Classico resin ratio of 21 cm² polymer to 7 mL monomer was manipulated following the manufacturer's recommendations. The resulting mass was included in the dough-like phase in metallic flasks (Safrany Metallurgical, Sao Paulo, SP, Brazil) and polymerized at 74°C for 9 h in a thermopolymerizing unit (Termotron Dental Appliances, Piracicaba, SP, Brazil) using a conventional hot water bath cycle. Onda Cryl resin ratio of 21 cm² polymer to 7 mL monomer was manipulated following the manufacturer's recommendations, and included in the dough-like phase in plastic flasks (Classico). Following the manufacturer's recommendations, the Onda Cryl resin was made using the following steps: 30% potency for 3 min, 0% potency for 4 min, and 70% potency for 3 min using a microwave domestic oven (Continental; Manaus, AM, Brazil) at 900 W. A QC-20 resin ratio of 21 cm² polymer to 7 mL monomer was manipulated following

the manufacturer's instructions, and included in the stringy phase in metallic flasks (Safrany). QC-20 resin polymerization was achieved using boiled water for 20 min in a thermopolymerizing unit (Termotron).

The specimens were deflasked after cooling at room temperature. Subsequently specimens were submitted to finishing and polishing with 320- 400- and 600-silicon carbide paper in an APL-4 mechanical polisher (Arotec; Sao Paulo, SP, Brazil). Finishing and polishing procedures were carried out for 8 s under a load of 20 g. The specimens were then water stored at 37°C for 24 h. Forty specimens of each resin were divided into four groups (n=10): G1- Control, G2- Toothbrushing, G3- Efferdent disinfection, and G4- Sodium hypochlorite disinfection.

All specimens were submitted to surface hardness evaluation prior to and after thermocycling.

Oral B toothbrushes (P&G Co, Louveira, SP, Brazil) with soft rounded bristles were used for specimen brushing in a MSet mechanical device (Nucci ME; Sao Carlos, SP, Brazil) with a dentifrice (Smile; Kolynos, Sao Paulo, SP, Brazil) dilution of 4.6 g to 6 mL of distilled water. The manufacturer claims that this dentifrice is somewhat abrasive and is composed of calcium carbonate, sodium bicarbonate, sodium lauryl sulfate, sodium monofluorophosphate and fluoride. A load of 250 gf and speed of 350 strokes per minute at room temperature was developed for the toothbrushing procedure (Lira et al., 2012). A linear brushing of 20,000 strokes was accomplished on each specimen (Sexson & Phillips, 1951), which was submitted to 3 mL of diluted dentifrice

injected automatically per minute. After washing with running water, the specimens were stored in distilled water at $37^{\circ}\text{C} \pm 1^{\circ}\text{C}$ for 24 h.

Alkaline peroxide effervescent tablets (Efferdent; Warner-Lambert, Morris Plains, NJ, UA) or 0.5% sodium hypochlorite (Proderma Handling Pharmacy; Piracicaba, SP, Brazil) solutions were used for chemical disinfection. The disinfection of each specimen was carried out at room temperature for 20 min, and repeated eight times daily for 90 days (Lira et al., 2012). Specimens were washed with running water and then stored in water at $37^{\circ}\text{C} \pm 1^{\circ}\text{C}$ for 24 h following each daily disinfection.

Specimens of all groups (G1, G2, G3 and G4) were submitted to 20,000 thermal cycles (Pinto et al., 2004) using a MSCT-3 Plus

thermocycler unit (Nucci ME) with alternative water baths at $5^{\circ}\text{C} \pm 1^{\circ}\text{C}$ and $55^{\circ}\text{C} \pm 1^{\circ}\text{C}$ for a 60-second dwell time. The number of thermal cycles was based on the supposition that, within two years, the oral temperature alters 10 times daily due to the ingestion of hot or cold foods and beverages, and that each procedure constitutes three thermal cycles (Pinto et al., 2004).

Specimens of the G1, G2, G3 and G4 groups were submitted to the surface hardness test in a HMV-2 Knoop indenter (Shimadzu, Tokyo, Japan) with an indentation load of 25 gf for 5 sec. Three penetrations were made along the specimen surface, and the average of these values was considered as the hardness value for each specimen.

Data were analyzed by three-way ANOVA for repeated measures and comparisons by Tukey's test ($\alpha=0.05$). The factors considered were acrylic resin, treatment and thermocycling. Interactions among these factors were also considered.

Results

Three-way ANOVA (Table 1) revealed significant differences in the surface Knoop hardness only for the treatment ($p<0.0001$) and thermocycling-resin-surface treatment interaction ($p<0.0330$).

Table 1 - Results of Three-Way ANOVA Statistical Analysis for Surface Knoop Hardness

| Variation cause | <i>df</i> | Sum of squares | Mean Square | F | <i>P</i> |
|--------------------|-----------|----------------|-------------|--------|----------|
| Thermocycling (Tc) | 1 | 9.217253 | 9.21725 | 1.8289 | 0.17422 |
| Resin (R) | 2 | 25.20954 | 12.60477 | 2.5010 | 0.08236 |
| Treatment (Tr) | 3 | 117.60738 | 39.20246 | 7.7786 | 0.00017* |
| Tc x R | 2 | 14.83803 | 7.41901 | 1.4721 | 0.23020 |
| Tc x Tr | 3 | 6.84551 | 2.28183 | 0.4528 | 0.71972 |
| R x Tr | 6 | 54.31744 | 9.05290 | 1.7963 | 0.10036 |
| Tc x R x Tr | 6 | 70.46860 | 11.74476 | 2.3304 | 0.03309* |
| Residue | 216 | 1088.59747 | 5.03980 | | |
| Total | 239 | 26601.691 | | | |

General mean = 17.7075; variation coefficient = 12.67%; (*) significant.

Knoop hardness values for resins under the effect of toothbrushing or chemical disinfections are shown in Table 2. Prior to thermocycling, statistical differences were found among treatments for Onda Cryl, with greater hardness values observed for Efferdent and hypochlorite; lower hardness value was obtained after toothbrushing, and intermediate value was found for the control. Differences were found among resins for the control treatment, with higher value for QC-20 resin, lower value for the Classico, and intermediate value for the Onda Cryl. After thermocycling, significant differences were observed for the Onda Cryl resin, according to treatment, with a lower value for toothbrushing compared to the control, while Efferdent and hypochlorite treatment produced intermediate values. For QC-20, the lowest value was observed for the control specimen, while higher values were found for the

Efferdent and hypochlorite treatments, while toothbrushing was intermediate. There were significant differences among resins only for the control specimens, with the highest value for Onda Cryl and lowest for QC-20, while Classico presented the intermediate value. Significant differences were found in the control specimens only for the Onda Cryl and QC-20 resins prior to and after the thermocycling procedures.

Table 2 - Knoop Hardness Means (SD) for Acrylic Resins Relative to prior to and after Thermocycling Treatments

| Treatment | Prior thermal cycle | | |
|---------------|---------------------|-----------------|-----------------|
| | Classico | Onda Cryl | QC-20 |
| Control | 15.9±1.9 aB | 17.3±1.6 abAB | 18.4±1.8 aA (*) |
| Toothbrushing | 16.4±1.9 aA | 15.2±2.0 bA | 17.0±1.2 aA |
| Efferdent | 17.5±1.7 aA | 18.9±0.9 aA | 18.2±1.2 aA |
| Hypochlorite | 18.2±2.2 aA | 18.2±1.5 aA | 18.3±1.7 aA |
| Treatment | After thermal cycle | | |
| | Classico | Onda Cryl | QC-20 |
| Control | 17.4±1.6 aAB | 19.7±2.3 aA (*) | 15.7±1.9 bB |
| Toothbrushing | 16.3±2.4 aA | 16.9±6.8 bA | 18.0±2.0 abA |
| Efferdent | 17.5±1.8 aA | 18.5±1.7 abA | 18.3±1.8 aA |
| Hypochlorite | 18.4±1.2 aA | 18.6±3.0 abA | 18.9±2.4 aA |

Means followed by different small letters in each column and by different capital letters in each row differ significantly by Tukey's test ($\alpha=0.05$). (*) Asterisk means statistical differences for the same treatment when prior to and after thermocycling are compared. (SD) = Standard deviation.

Discussion

Acrylic resin-based materials display differences in their surface hardness levels (Parr & Rueggeberg, 2002; Azevedo et al., 2005; Bindo et al., 2009 and Pinto et al., 2010). Denture base surfaces with different hardness levels, in turn, may be influenced differently by mechanical and chemical cleaning procedures.

Based on these considerations, this study evaluated the effect of toothbrushing and chemical disinfections on the surface hardness of Classico (conventional), Onda Ceryl (microwaved) and QC-20 (boiled) denture acrylic resins, submitted or not, to thermocycling. Surface hardness data was analyzed by three-way ANOVA (Table 1) and revealed significant differences for treatment and thermocycling-resin-treatment interaction. As

thermocycling, when associated or not, with mechanical and chemical cleanings caused different effects on the surface hardness of denture base acrylic resins, the working hypothesis was accepted.

It is difficult to understand and to explain why the effects of these cleaning and disinfection treatments were not similar for all the acrylic resins studied. Considering that acrylic resins have similar chemical compositions, the polymerization mode of each polymer may be a factor in these results or, in other words, different polymerization temperatures may influence the different polymerization modes. A traditional study showed that surface hardness values were similar for acrylic resins polymerized by ebullition water and microwave energy (Truong & Thomasz, 1998). In addition, different polymerization methods (Bartolini et

al., 2000) and water sorption/solubility levels (Tuna et al., 2008) could be considered significant factors in influencing the surface characteristics of acrylic resins.

For this reason, for the prior to thermocycling condition, only the Onda Cryl resin presented significant differences when the control and the Efferdent and sodium hypochlorite disinfection treatments were compared to toothbrushing, as well as for the when the resins. It may be speculated that another important factor that may influence hardness constitutes the chemical characteristics inherent to each polymeric material, which could promote different effects on the sample surface or due also to the associated effects caused by these same factors. More *in vitro* studies need to be conducted to corroborate this supposition.

However, similar values of surface hardness were found for the resins after toothbrushing and both the chemical treatments. It is probable that factors such as poly-methylmethacrylate copolymers reinforced with cross-linking, more efficient cycles of polymerization and lower quantities of residual monomer improve the surface hardness of resins and nullify the deleterious effects of cleaning and disinfection. However, regardless of these associated factors, it may be suggested that the hardness of acrylic resins is not be influenced differently by the water absorption in these solutions, since these materials present similar chemical compositions (Anusavice, 2003).

Conflicting surface hardness findings have also been reported in the literature when other types of resin-based materials were submitted to chemical disinfection. Different hardness was

shown for heat- and auto-cured acrylic resins, whose values were increased following long-term exposure to disinfectants (Mese, 2007). Conversely, there was a significant decrease in hardness after the chemical disinfection of acrylic resins that were polymerized by a conventional short cycle and boiled water method (Neppelenbroek et al., 2005), and a significant decrease in hardness was observed for acrylic resins treated with 30 soak cycles for 24 h when the water temperature was increased from 40 to 100°C (Devlin & Kaushik, 2005).

However, previous clinical studies have shown that neither increased hardness (Makila & Honka, 1979) nor decreased resilience (Schmidt & Smith, 1983), due to disinfection, occurred for silicone based-material submitted to chemical disinfections. Conversely, soft-lining acrylic resin-based materials contain

plasticizers in their composition, and the softness and modulus of elasticity for polymers of similar composition will depend upon the concentration of the plasticizer (McCabe, 1976).

Consequently, the plasticizer may cause deleterious effects on the softness of the material when submitted to thermocycling (Qudah et al., 1991), an event that does not occur in the acrylic resin denture bases.

Interestingly, thermocycling promoted significant differences among treatments for the Onda Cryl and QC-20 resins. In addition, when prior-to and after thermocycling procedures were compared there were significant differences only for the control of the Onda Cryl and QC-20 resins (Table 2). The surface hardness of denture acrylic resins can change in function of chemical disinfectants and long-term water immersion

(Neppelenbroek et al., 2005). With the exception of the Onda Cryl and QC-20 resins, cleaning and disinfection treatments made for Classico were not influenced by the thermocycling. Similar results were also found in the current study for the treatments prior to thermocycling.

Increased hardness values due to the use of chemical solutions were only evident for Onda Cryl prior to thermocycling and for QC-20 after thermocycling. It is possible that polishing increases the stiffness of the specimens, consequently increasing the surface hardness. However, other studies would be necessary to confirm this supposition and it may be important to dissociate conjugated effects that could influence the hardness level of the acrylic resins.

Thermal shock due to thermocycling causes successive shrinkage and expansion of the polymeric material (Anil et al., 2000). Based on the results of the present study and from the standpoint of general understanding, the thermal shock due to thermocycling did not cause sufficient mechanical hardening to increase the surface hardness of the acrylic resins studied. This supposition is supported by the fact that only the Onda Cryl and QC-20 resins presented statistically different hardness prior to and after thermocycling procedures.

Results from this study show the importance of effects produced by cleaning and disinfection methods and their interactions with thermocycling on the surface hardness of denture base acrylic resins. These factors in association may influence the quality, longevity and satisfaction of complete-denture wearers.

Future studies are necessary to evaluate other factors that depend upon the hardness of polymers, such as impact strength and tooth-denture base bond strength, which are all important when accidental falls of the prosthesis occur. Furthermore, different disinfectants can produce a marked color loss and slight surface gloss loss in resin-based materials (Davenport et al., 1986).

Conclusion

Different effects on the surface hardness of acrylic resins for denture bases were observed when associating thermocycling with toothbrushing or chemical disinfections.

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