Effect of Repeated Disinfections by Microwave Energy on the Physical and Mechanical Properties of Denture Base Acrylic Resins

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The present study evaluated the effect of repeated simulated microwave disinfection on physical and mechanical properties of Clássico, Onda-Cryl and QC-20 denture base acrylic resins. Aluminum patterns were included in metallic or plastic flasks with dental stone following the traditional packing method. The powder/liquid mixing ratio was established according to the manufacturer's instructions. After water-bath polymerization at 74°C for 9 h, boiling water for 20 min or microwave energy at 900 W for 10 min, the specimens were deflasked after flask cooling and finished. Each specimen was immersed in 150 mL of distilled water and underwent 5 disinfection cycles in a microwave oven set at 650 W for 3 min. Non-disinfected and disinfected specimens were subjected to the following tets: Knoop hardness test was performed with 25 g load for 10 s, impact strength test was done using the Charpy system with 40 kpcm, and 3-point bending test (flexural strength) was performed at a crosshead speed of 0.5 mm/min until fracture. Data were analyzed statistically by ANOVA and Tukey's test (α = 0.05%). Repeated simulated microwave disinfections decreased the Knoop hardness of Clássico and Onda-Cryl resins and had no effect on the impact strength of QC-20. The flexural strength was similar for all tested resins.

Key Words: hardness, impact, flexural strength, acrylic resin, microwave disinfection.

INTRODUCTION

Prosthetic materials sent from dental clinics to the dental laboratories may be contaminated by pathogenic bacteria, which can be transferred to the prosthetic lab technicians by direct contact or during the finishing and polishing procedures. A classic study showed that microorganisms found in pumice slurry were originating from contaminated prosthetic pieces which were polished without prior cleaning and disinfection, and that these microorganisms can be transferred to one denture to another during finishing and polishing procedures (1).

Instrumental and prosthetic materials should be

properly disinfected or sterilized to avoid the cross-contamination among dentists, patients, assistants and lab technicians. In addition to the cross-contamination caused by the patients, microorganisms may contaminate the denture during procedure and manipulation stages. In the past decades, several authors have suggested chemical disinfection, using solutions of glutaraldehyde, sodium hypochlorite, iodoform, chlorine dioxide or alcohol to avoid the cross-infection by pathogenic agents. However, chemical disinfection may present disadvantages, such as denture staining and oral tissue reactions (2,3). Originally used for polymerization of the thermally cured acrylic resins (4), microwave energy

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has been indicated as a simple, practical and low-cost alternative to overcome the shortcomings of chemical disinfection methods. A recent study has demonstrated that microwave energy irradiation in a conventional domestic oven for 3 min at 650 W was capable to sterilize all complete dentures inoculated with 5 *Candida* species (5). Considering the probability of the denture base be contaminated internally and externally (2), microwave energy appears as an effective method for complete denture disinfection (6) and prevention of cross-contamination (7).

A previous study comparing chemical and microwave disinfection showed that the hardness, flexural strength and dimensional changes of the acrylic resin were not significantly altered by these disinfection procedures (8). More recent studies verified that a single microwave disinfection cycle decreased the hardness and had no effect on the impact and flexural strength of different commercial acrylic resins (9). However, it has also been shown that microwaving could cause dimensional changes or distortion of the acrylic resin denture base (10-12), which could compromise the denture stability, retention and durability.

The purpose of this study was to evaluate the effect of repeated simulated microwave disinfection on the hardness, impact and flexural strength of Clássico (long cycle), QC-20 (short cycle) and Onda-Cryl (microwave energy) thermally cured acrylic resins. The null hypothesis tested was that repeated microwave disinfections affect adversely these physical and mechanical properties of the studied acrylic resins.

MATERIAL AND METHODS

The following materials were used: Clássico (Clássico Produtos Odontológicos Ltda., São Paulo, SP, Brazil; *Powder*: polymethyl methacrylate; *Liquid*: methyl methacrylate - MMA); QC-20 (Dentsply/DeTrey, Konstanz, Germany; *Powder*: methyl/n butyl methacrylate copolymer, benzoyl peroxide and mineral pigments; *Liquid*: MMA, ethylene glycol, dimethacrylate, hydroquinone, terpinolene and n,n-dimethyl p-toluidine); Onda-Cryl (Clássico Produtos Odontológicos Ltda.; *Powder*: MMA and EADPB copolymers and benzoyl peroxide; *Liquid*: MMA monomer, topanol, ethylene glycol dimethacrylate).

The specimens were fabricated according to a previous study (9). Three aluminum rectangular pattern

dies (65.0 x 12.0 x 3.5 mm) were invested in metallic (Safrany; J. Safrany Metallurgy, São Paulo, Brazil) or plastic (GC; GC Dental Products, São Paulo, Brazil) flasks with type III dental stone (Herodent; Vigodent, Petrópolis, RJ, Brazil) mixed according to the manufacturer's instructions with a powder to liquid ratio of 100 g powder to 30 mL water.

After bench press for 1 h, the aluminum pattern dies were deflasked and the reproduction accuracy was verified in the stone molds. Ten specimens were made for each acrylic resin according to the manufacturers' mixing instructions, and the following groups were formed: Group 1: Clássico acrylic resin pressing, polymerization in water bath at 74°C for 9 h (Termotron; Termotron Laboratory Products, Piracicaba, SP, Brazil) and deflasking after bench cooling at room temperature; Group 2: QC-20 acrylic resin pressing, polymerization in boiling water in steam press (Termotron) and deflasking after bench cooling at room temperature; Group 3: Onda-Cryl acrylic resin pressing, polymerization in a domestic microwave oven (Continental; Manaus, AM, Brazil) set at 900 W power (3 min at 40% power; 4 min at 0% power; and 3 min at 90% power), and deflasking after bench cooling at room temperature.

Clássico and Onda-Cryl acrylic resins were prepared with a monomer/polymer ratio of 1:3 by volume, corresponding to 37.5 g powder to 15 mL liquid. A powder/liquid ratio of 23 g powder to 10 mL liquid was used for QC-20 resin. The prepared dough was packed in the dough-like stage in a hydraulic press (Linea H; Linea, São Paulo, SP, Brazil), with a load of 850 kgf. In the metallic flask procedure, the final closure was performed under a load of 1,250 kgf for 5 min and the flasks were placed in traditional spring clamps. The final closure of the plastic flasks was under a load of 1,000 kgf for 5 min and the screws tightened the plastic flasks before press release.

Atotal of 30 specimens (10 of each material) were made and deflasked by routine laboratorial technique. Finishing of the specimens was carried out using stones for acrylic resin abrasion and sandpaper of decreasing abrasiveness. Pumice slurry was used with white and black brushes and felt tip for polishing in a bench lathe. After final polishing with flannel wheel and universal paste (Kota Manufacturing and Trade Co., São Paulo, SP, Brazil), the specimens had 65.0 x 10.0 x 3.3 mm dimensions, which are in accordance with to the ISO 1567 standard (13). All specimens were stored in distilled

water at 37°C for 24 h.

Fifteen control specimens (5 of each material) were not subjected to microwave disinfection. The other 15 specimens (5 of each material) were subjected to a 3-min microwaving cycle at 650 W in a domestic microwave oven (Continental) (14). For this procedure, the specimens were immersed individually in glass vials containing 150 mL of distilled water (12). Each specimen was subjected to 5 simulated disinfections, being 1 per day, during 5 consecutive days. The specimens were stored in distilled water at 37°C between disinfections. All control and microwaved specimens were subjected to the following tests:

Hardness: Specimens were subjected to Knoop hardness test in a microindenter (Shimadzu HMV-2000; Shimadzu Corporation, Kyoto, Japan) calibrated with 25 g load during 10 s. Three indentations were made in each specimen, one in the center and one at each end. The average of the 3 indentations was considered as the specimen hardness.

Impact strength: Specimens were subjected to an impact strength test in the Charpy system (Otto Wolpert Werke, Germany) with 40 kpcm impact load. The impact load obtained at the moment of the specimen failure was changed to impact strength (kgf/cm²) using the following equation: IS=I/LH, where: IS= impact strength (kgf/cm²); I= impact load (kpcm); L= specimen width in the impact region (cm); and H= specimen height in the impact region (cm).

Flexural strength: Specimens were subjected to a 3-point bending test in a universal testing machine (Instron; Canton, MA, USA) running at a crosshead speed of 5 mm/min until failure. Flexural strength value was calculated using the following equation: F=3 WL/2 bd², where: F=flexural strength (kgf/cm²); W= ultimate load before the failure; L= distance between the support points (20 mm); b= specimen width (10 mm); d= specimen thickness (3 mm). Results in kgf/cm² were changed to MPa by multiplying the value by the constant 0.098.

Data from the control and microwaved specimens were analyzed statistically by ANOVA and Tukey's test at 5% significance level, considering the factors acrylic resin and disinfection and their interactions.

RESULTS

For Knoop hardness, comparing the acrylic resins within the same disinfection condition, there was

statistically significant difference (p<0.05) among the materials for both non-disinfection and microwave-disinfection conditions, except for the microwaved Clássico specimens. When the resins were evaluated separately, microwaved Clássico and Onda-Cryl specimens presented significantly lower hardness values than the control specimens (Table 1).

For the impact strength comparing the acrylic resins within the same disinfection condition, there was statistically significant difference (p<0.05) among the acrylic resins only in the microwaved specimens. When the resins were evaluated separately, the microwaved Clássico specimens presented significantly higher (p<0.05) impact strength than the non-disinfected specimens, while the microwaved QC-20 specimens presented the lowest values of material/condition interaction (Table 2).

For the flexural strength, comparing the acrylic resins within the same disinfection condition, there was no statistically significant difference (p>0.05) among the non-disinfected materials or the materials subjected microwave disinfection. When the resins were evaluated separately, no statistically significant difference (p>0.05) was between control and microwaved specimens (Table 3).

DISCUSSION

This study characterized and compared the effect of repeated simulated microwave disinfections on the hardness, impact and flexural strength of thermally activated acrylic resins. The tested hypothesis that repeated microwave disinfection cycles affect adversely

Table 1. Knoop hardness mean values for Clássico, QC-20 and Onda-Cryl acrylic resins subjected or not to repeated simulated microwave disinfection.

Acrylic resin	No disinfection	Simulated microwave disinfection
Clássico	$14.59 \pm 1.45 \text{ b,A}$	$12.18 \pm 0.75 \text{ ab,B}$
QC-20	$11.53 \pm 0.67 \text{ c,A}$	$11.34 \pm 1.90 \text{ b,A}$
Onda-Cryl	$18.53 \pm 0.32 \text{ a,A}$	$13.61 \pm 0.59 \text{ a,B}$

Same lowercase letters in columns and uppercase letters in rows indicate no statistically significant difference (Tukey's test; p>0.05).

these physical and mechanical properties of the resins was partly accepted.

Although classic finding has shown that hardness is not influenced by the acrylic resin type (15), in the present study, Onda-Cryl presented higher hardness, QC-20 presented lower hardness, and Clássico had intermediary Knoop hardness values in the non-disinfected condition (Table 1). In the same way as observed in a previous study, different results of surface nanohardness were obtained when different commercial types of denture base acrylic resins were evaluated (16).

Microwave-polymerized acrylic resins show higher hardness when compared to those cured by conventional and fast cycles (17). This fact is due to differences in the residual monomer levels in the different polymerization cycles, considering that the hardness should establish an inversely proportional relation with the amount of residual monomer (18). Knoop hardness values for conventional resins have been shown to differ from those obtained for resins polymerized by boiling water and microwave energy (9). Although these resins have similar chemical composition, it is possible that the degree of conversion to change monomer in polymer was different for each material evaluated before microwave disinfection in the mentioned study (9).

In the present study, Onda-Cryl and QC-20 specimens subjected to repeated microwave disinfections presented significantly lower Knoop hardness than the non-microwaved QC-20 specimens (Table 1). It may be speculated that repeated microwave disinfection cycles caused different plasticizing effects on the polymer chains, resulting in different hardness values for these materials. When control and microwave-disinfected specimens were compared, there was statistically sig-

Table 2. Impact strength mean values (kgf/cm²) for Clássico, QC-20 and Onda-Cryl acrylic resins subjected or not to repeated simulated microwave disinfection.

Acrylic resin	No disinfection	Simulated microwave disinfection
Clássico	$7.25 \pm 1.91 \text{ a,B}$	$9.19 \pm 0.88 \text{ a,A}$
QC-20	$8.19 \pm 0.55 \text{ a,A}$	$4.33 \pm 0.55 \text{ c,B}$
Onda-Cryl	$8.13 \pm 1.44 \text{ a,A}$	$7.86 \pm 0.80 \text{ b,A}$

Same lowercase letters in columns and uppercase letters in rows indicate no statistically significant difference (Tukey's test; p>0.05).

nificant difference for the Clássico and Onda-Cryl resins, both with lower Knoop hardness when microwaving was done (Table 1). It has been claimed that the decrease of the hardness of these acrylic resins is due to disarrangements in the polymer chains, which probably occur under the effect of the first disinfection cycle (9) and thus was not changed by the repeated disinfection cycles performed in the present study. This fact indicates that the first microwave disinfection cycle caused the main change in the resin hardness. In addition, QC-20 has components for an additional chemical activation to thermal activation, which could be responsible for the lack of statistically significant difference between treatments, after repeated microwave disinfection cycles.

Regarding the impact strength, there was no significant difference in the control and microwave-disinfected Onda-Cryl specimens, but significant differences were found for the other acrylic resins (Table 2). This finding does not confirm those of previous investigations evaluating non-disinfected specimens (18) and specimens subjected or not to a single microwave disinfection procedure (9); both of these studies showed no statistically significant difference among resins.

Poly(methyl methacrylate) with monomer addition to permit copolymers with cross-linking is the basic composition of these resins. During impact load application, the non-disinfected specimens absorbed the same amount of energy, probably due to their similar resilience, which resulted in statistically similar fracture strength. It is also claimed that acrylic resins polymerized by large cycle show better energy absorption due to long polymer chains with high molecular weight, when compared to microwave polymerization, with short polymer chains with low molecular weight (19).

Table 3. Flexural strength mean values (MPa) for Clássico, QC-20 and Onda-Cryl acrylic resins subjected or not to repeated simulated microwave disinfection.

Acrylic resin	No disinfection	Simulated microwave disinfection
Clássico	$5.96 \pm 0.27 \text{ a,A}$	$5.88 \pm 0.08 \text{ a,A}$
QC-20	$5.96 \pm 0.26 \text{ a,A}$	5.92 ± 0.29 a,A
Onda-Cryl	$6.12 \pm 0.49 \text{ a,A}$	$6.28 \pm 0.32 \; a,A$

Same lowercase letters in columns and uppercase letters in rows indicate no statistically significant difference (Tukey's test; p>0.05).

In addition, microwave-polymerized acrylic resins have better physical and mechanical performances than conventionally cured resins (15,17).

When the resins were subjected to repeated microwave disinfections, significant differences were found among the impact strength values (Table 2). These results differ from those of a previous study in which disinfection by microwave energy had no effect on the impact strength of Clássico, Onda-Cryl and QC-20 acrylic resins (9). It is likely that the resilience of the specimens was changed due to the post-polymerization caused by the repeated disinfection procedures. The different levels of residual monomer in each type of resin might also have contributed to these results.

Regarding the flexural strength, no significant differences were found between the tested conditions (control or microwave disinfection) or among the acrylic resins (Table 3).

Flexural strength of microwave-cured denture baseplates in a previous study showed significant differences among the tested acrylic resin types, although the microwaved resin did not absorb much more energy before failure than the water bath-polymerized acrylic resin (20). In the present study, the repeated microwave disinfection cycles did not alter the amount of energy absorbed by the non-disinfected specimens during the 3-point bending test, resulting in similar flexural strength values for all studied acrylic resins. Comparable results have been observed in acrylic resin specimens subjected to a single microwave disinfection cycle (9).

Although attempts were made to characterize the effect of repeated microwave irradiation disinfections on the tested physical and mechanical properties, the present study was limited to predicting the effect of other variables. Further studies are needed to investigate whether the effect of microwave energy may be deleterious to denture base distortion and stability in oral use. Within the limitations of an *in vitro* study, the results showed that microwaving of the polymerized resins had an adverse effect on the tested properties. In conclusion, repeated simulated microwave disinfections decreased the Knoop hardness of Clássico and Onda-Cryl resins and had no effect on the impact strength of QC-20. The flexural strength was similar for all tested resins.

RESUMO

Este estudo verificou o efeito de repetidas desinfecções por

microondas sobre a dureza, impacto e resistência à flexão das resinas acrílicas Clássico, Onda-Cryl e QC-20. Matrizes de alumínio foram incluídas em muflas metálica ou plástica com gesso pedra tipo III, de acordo com a técnica de inclusão tradicional. A proporção polímero/monômero usada foi de acordo com as recomendações dos fabricantes. Depois da polimerização em água à temperatura de 74°C por 9 h, água em ebulição por 20 min ou por energia de microondas a 900 W por 3 min, os corpos-de-prova foram demuflados após esfriamento da mufla em temperatura ambiente e submetidos ao polimento convencional. Testes de dureza, impacto e resistência à flexão foram feitos nos corpos-de-prova controle e experimentais. Cada corpo-de-prova foi submetido individualmente a 5 desinfecções por microondas em forno doméstico com 900 W de energia por 3 min, imersos em 150 mL de água destilada. O teste de dureza Knoop foi realizado com carga de 25 g por 10 s, de resistência ao impacto pelo sistema Charpy com 40 kpcm e de resistência à flexão por três pontos com velocidade de 0,5 mm/min até fratura. Os dados obtidos foram submetidos à análise de variância e ao teste de Tukey (α =0.05%). Repetidas desinfecções por microondas diminuíram a dureza das resinas Clássico e Onda-Cryl, não promoveram nenhum efeito sobre a resistência ao impacto da resina QC-20. A resistência à flexão foi similar para todas as resinas.

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