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DOI: 10.1590/0103-6440201600733

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Effect of Polymerization Cycles on Gloss, Roughness, Hardness and Impact Strength of Acrylic Resins

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The aim of this study was to evaluate the conventional and boiled polymerization cycles on gloss, roughness, hardness and impact strength of acrylic resins. Samples were made for each Classico and QC-20 materials (n=10) in dental stone molds obtained from rectangular metallic matrices embedded in metallic flasks. The powder-liquid ratio and manipulation of the acrylic resins were accomplished according to manufacturers' instructions and the resins were conventionally packed in metallic flasks. After polymerization by (1) conventional: 74 °C for 9 h (Classico) and (2) boiled: 20 min (QC-20) cycles, the samples were deflasked after cooling at room temperature and conventionally finished and polished. The properties were evaluated after storage in water at 37 °C for 24 h. Gloss was verified with Multi Gloss 268 meter (Konica Minolta), surface roughness was measured with Surfcom SE 1700 rugosimeter (Kosaka), Knoop hardness number was obtained with HMV-200 microdurometer, and impact strength was measured in an Otto Wolpert-Werke device by Charpy system (40 kpcm). Data were subjected to Student's t-test (at $\alpha=0.05$). The results were: Gloss: 67.7 and 62.2 for Classico and QC-20 resins, respectively; Surface roughness: 0.874 and 1.469 Ra- μ m for Classico and QC-20, respectively; Knoop hardness: 27.4 and 26.9 for Classico and QC-20, respectively; and Impact strength: 37.6 and 33.6 kgf/cm² for Classico and QC-20, respectively. No statistically significant difference ($p>0.05$) were found between the resins for the evaluated properties. In conclusion, conventional and boiled polymerization cycles had similar effects on gloss, roughness, hardness and impact strength of both Classico and QC-20 resins.

Key Words: acrylic resin, polymerization cycle, physical property.

Introduction

Since the 1930's, acrylic resin based on poly (methyl methacrylate) system is the most widely used material for making conventional denture and removable partial prostheses. Several types of acrylic resins, trademarks and polymerization techniques were developed over the years, but acrylic resins can show inadequate physical properties if not properly manipulated and correctly employed. The traditional procedure for polymerization is the conventional method of hot water bath and its variations, but activation by microwave energy and visible light has also been employed (1).

Surface roughness is responsible for extrinsic pigmentation and biofilm accumulation, whereas hardness would be related to abrasiveness and wear of denture base acrylic resins. In consequence, surface roughness can enhance microorganisms adhesion, where stronger interactions occur between cells and surface features with similar dimensions, increased biofilm mass and resistance to removal. Therefore, minimization of denture abrasion during cleaning is desirable (2). The gloss is an extremely important characteristic that has effect on color perception, and it is related directly to the polishing of denture base. Thus, color stability of acrylic resins is important because

there is a significant difference between perceptibility and acceptability thresholds for denture base acrylic resins (3).

Impact strength is a significant factor because long-term changes can promote softening of the acrylic resins with a negative effect on wear, abrasion and roughness. Different polymerization techniques can also influence the impact strength of denture base resins (4). The acrylic resin roughness has been used as an index of material capacity to resist the wear and abrasion, and the surface hardness test informs the material's ability to resist the penetration of a diamond point under a specific load (1).

It has been alleged that conventional laboratory polishing is the most effective polishing technique for acrylic resin denture bases (5). Maintenance of the surface gloss of acrylic resin is an important factor for the denture hygiene, due to wear from the association between tooth brushing and abrasive materials (6). Repeated mechanical cleaning procedures change the smoothness of denture bases and provide roughness favorable to colonization by microorganisms, biofilm accumulation and staining (5).

The main disadvantages of the acrylic resins for complete denture are esthetic, physical and mechanical properties that change with time in the oral environment

and color change that affects the gloss of the denture base (7). Other variables that can change the smoothness of acrylic resins are micro porosities resulting from inadequate monomer-polymer ratio, plastic stage of the mass during packing, temperature of the polymerization cycle and viscosity of the resins (1).

Besides the factors inherent to physical properties of the acrylic resins, different polymerization protocols may influence negatively some mechanical properties (4,8,9). For this reason, it would be appropriate to verify the effect of polymerization cycles (conventional and boiled) on gloss, roughness, hardness and impact strength of acrylic resins. The hypothesis of this study was that polymerization cycles would promote different effects in each property for both acrylic resins. The aim of this study was to evaluate the effect of each conventional or boiled polymerization cycle on gloss, roughness, hardness and impact strength of Classico (conventional) and QC-20 (boiled) acrylic resins.

Material and Methods

Sample Preparation

Samples (n=10) were prepared for each acrylic resin by wax rectangular matrices (65 mm long, 5 mm wide and 2 mm high) conventionally included in metallic flasks (Safrany; Metalurgia Safrany, São Paulo, SP, Brazil) with type III dental stone (Herodent; Vigodent, Petrópolis, RJ, Brazil) at the ratio of 100 g powder to 30 mL water, vacuum mixed for 30 s and poured with mechanical vibration.

The flasks remained in hydraulic press (Linea Delta; Delta, São Paulo, SP, Brazil) at 850 kgf for 1 h. After this, the flask was opened, the wax matrices removed and the resulting stone molds cleaned with a water-domestic detergent (Ypê; Amparo Produtos Químicos, Amparo, SP, Brazil) solution to remove the wax residues and petroleum jelly used as dental stone isolator and washed with hot water.

The volumetric polymer-monomer ratios for Classico (Clássico Dental Products, São Paulo, SP, Brazil) and QC-20 (Dentsply, Petrópolis, RJ, Brazil) acrylic resins was according to manufacturers' instructions, using 21 g powder to 7 mL liquid. In the dough-like stage, the mixed resins were inserted in the dental stone molds isolated with a water-soluble alginate solution (Cel Lac; S.S. White, Rio de Janeiro, RJ, Brazil). A polyethylene sheet was placed over the resin, the flask reassembled and placed in a hydraulic press (Linea Delta). Pressures of 850 kgf for 5 min and 1,250 kgf for 20 min applied incrementally were used for initial and final pressings, respectively. Classico acrylic resin polymerization procedure was in an automatic apparatus (Thermotron; Thermotron Dental Products, Piracicaba, SP, Brazil), set for a heated water cycle at 74 °C for 9 h. QC-20

acrylic resin polymerization procedure was performed in hot water for 20 min.

Sample deflasking was accomplished after flask cooling at room temperature. Afterwards the sample surface was abraded with silicon carbide sandpapers (Norton, São Paulo, SP, Brazil) with decreased granulation (200, 400 and 600), and polishing was made with magnesium and calcium carbonate slurry (Pasom; Pasom Dental Materials, São Paulo, SP, Brazil) followed by a universal polishing slurry based on aluminum oxide (Kota, Cotia, SP, Brazil).

Gloss Evaluation

The gloss-meter measurement principle is based on a light beam incident on the sample surface. The intensity of the reflected light is measured and compared with a reference value. The gloss-meter calibration was made with a highly polished black glass pattern supplied by manufacturer. Gloss values for Classico and QC-20 acrylic resins were measured with gloss-meter (Multi Gloss 268; Konica Minolta, Ramsey, NJ, USA) using a light incidence of 60°. Four measurements were made on each sample and the arithmetic mean was considered the gloss value (Gloss Unit - GU).

Roughness Evaluation

Classico and QC-20 samples were submitted to a profilometer (Surfcorder SE 1700; Kosaka, Tokyo, Japan) for surface roughness evaluation. The considered value was the arithmetic mean (Ra) of the highest peak and the deepest valley in a 2.4 mm measuring path, a 0.8 mm wavelength and a 0.5 mm/s speed. Three measurements were made in each sample and the arithmetic mean was considered the roughness value (Ra-μm).

Knoop Hardness Evaluation

Indentations were accomplished in Classico and QC-20 samples with a durometer (HNV-2; Shimadzu, Tokyo, Japan) using a 50 gf load for 10 s. Knoop hardness value was considered as the arithmetic mean of three indentations made on each sample, located at the center and at each end (16 mm distance among them).

Impact Strength Evaluation

Classico and QC-20 samples were submitted to impact strength test in an Otto Wolpert-Werke device (Frankfurt, Germany) by Charpy system (40 kpcm). The value of impact obtained for the fractured sample was changed for impact strength using the formula

$$IS = Ir/wh,$$

where: IS= impact strength; Ir= impact realized (kpcm); w= sample width in the impact region (cm); h= sample height at the impact region (cm).

Statistical Analysis

Data of gloss, hardness, roughness and impact strength were evaluated for normality by Kolmogorov-Smirnov and the heterocedasticity of variances by Levene's test. Data distribution was normal and the variances were homogeneous, except for hardness. The comparison of gloss, roughness and impact strength was performed by Student's t-test. The comparison of hardness values was made by Mann-Whitney's test. A significance level of 5% was set for all analyses.

Results

Table 1 shows the mean values and standard deviations (SD) for gloss, roughness, hardness and impact strength of the Classico (conventional) and QC-20 (boiled) thermo-activated acrylic resins. The effects of the polymerization cycles on the evaluated properties were statistically similar for both acrylic resins.

Discussion

The current study evaluated the effect of polymerization cycles on gloss, roughness, hardness and impact strength of each Classico (conventional) or QC-20 (boiled) acrylic resins. Table 1 shows that there were no statistically significant differences for polymerization cycle-resin interaction when these properties were evaluated. In these conditions, the hypothesis of this study that polymerization cycles would promote different effects in each property for both acrylic resins was not accepted.

This result is not similar to a previous study showing that linear dimensional change, hardness and impact strength of acrylic resins were differently affected by four different polymerization cycles, when the samples were deflasked immediately or after water storage for 7 and 30 days (8). In addition, there were significant differences in surface roughness between conventional (9 h at 74 °C) and short (1 h at 74 °C + 30 min at 100 °C) curing cycles (9). Different effects on the linear dimensional change, hardness and impact strength of acrylic resins were observed under the influence of different polymerization cycle-water storage

associations (10). When a high impact heat-cured acrylic resin was polymerized in autoclave and conventional methods, there were significant differences between techniques in transverse, impact, hardness and porosity values. The slow (long) curing cycle improved the physical and mechanical properties compared with the fast (short) curing cycle (11).

Despite the conventional polymerization cycle showing greater numerical values compared to the boiled method (except for roughness), there were no statistically significant differences between the acrylic resins. It is also interesting to stress the disinfection effects on the mechanical properties of acrylic resins. Thus, decreased roughness values were shown in previous studies; however, no effect was observed on the impact or flexural strength of acrylic resins submitted to single (12) or repeated (13) simulated microwave disinfections, and no significant difference in roughness was observed for acrylic resins disinfected with 1% sodium hypochlorite or 1% peracetic acid (9). In addition, flexural strength of denture base acrylic resins was significantly affected by immersion in 1% sodium hypochlorite, 2% glutaraldehyde or 10% Micro 10 solutions for 30 min (14).

Another interesting fact when mechanical and biological properties were compared was that manipulation techniques (mass or saturation) and polishing methods (mechanical or chemical) showed more influence on the cytotoxicity in cell cultures than on flexural strength of denture base acrylic resins for periods of 24 to 168 h (15). The addition of nanostructured silver vanadate (β -AgVO₃) may provide the acrylic resins antibacterial activities but reduces the impact strength (16).

It is alleged that the similarity of fracture strength values of acrylic resins is due to material resilience. This mechanical property has been considered similar for different acrylic resin materials (12,17). This was also observed in the current study, as there was no statistically significant difference for impact strength values in both polymerization cycles. Conversely, it has been shown that short and long curing cycles by water bath, pressure cooker and microwave technique produce different effects on the impact strength of denture base acrylic resins. There was statistically significant difference in the impact strength for denture base resins polymerized using long cycle and short curing cycle in each technique, with better results for the long curing process. In addition, denture base resin polymerized by microwave activation technique showed the highest impact strength value when related to short and long water bath curing cycles

Table 1. Means and standard deviations for Classico and QC-20 acrylic resins related to evaluated properties

	Gloss		Roughness		Hardness		Impact strength	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Classico	67.7	10.8 a	0.8742	0.4280 a	27.4	3.6 a	37.6	10.4 a
QC-20	62.2	5.1 a	1.4694	0.8852 a	26.9	0.9 a	33.6	9.7 a
	p= 0.161		p=0.072		p=0.910		p=0.391	

Same lowercase letters in columns do not differ by Student's t-test (p<0.005).

or pressure cooker (4).

Surface gloss and surface roughness are time-dependent characteristics influenced by polishing procedures and applied force (18). A study analyzing six mechanical and chemical polishing techniques (Resilit high-luster polishing liquid, Universal polishing paste, Abraso-star K50, Pumice, Jet seal liquid and Acrypoint) on the surface roughness of heat- and light-cured denture base acrylic resins showed that the mechanical polish produced the smoothest surface, while the surface roughness values produced by these procedures were not influenced by the type of acrylic resin (19). In addition, the difference between smooth polished and rough unpolished resin surfaces was statistically significant, and it was also found that the gloss of heat-polymerized denture base resin was affected by immersion in four beverage solutions, showing noticeable gloss changes (7).

Surface gloss seems to be more consistent than roughness, despite the close association between these properties (18). Considering that the polishing time and load applied on the samples were standardized in the study, this may possibly explain the statistically similar results. As the surface smoothness and gloss levels are considered dependent on material hardness (20), the results observed in the present study followed the same pattern in relation to these acrylic resin properties. In other words, less rough surfaces showed greater hardness and improved gloss values. It is perhaps why a previous study (21) examined the clinical appearance (color, gloss, and surface roughness) of TiO₂ coating on polymethyl methacrylate resin dentures. The surface roughness increased following TiO₂ coating and afforded high levels of gloss while maintaining the color of the denture base material.

Regardless the activation mode of the acrylic resin, a stress will occur during the procedure whatever the polymerization method chosen. This fact is due to polymerization shrinkage inherent to polymeric materials (1). However, the different polymerization temperatures that occur for different curing cycles could compromise some mechanical properties, but this is a subject for further researches.

In addition, with the intention that the polymerization shrinkage promotes the least effect on the mechanical properties, denture base misfit and change in position of the teeth, some authors have developed methods and techniques in an attempt to compensate this unwanted effect. Thus, acrylic resins processed in double flask using hot water bath or microwave energy did not alter the porosity, roughness and hardness values (22). A new tension packing system was associated with decreased dimensional changes in the simulated maxillary denture bases processed by heat-polymerization (23). Different flask

systems did not cause change in the distances between teeth during denture processing, and there is no evidence that different metal flask systems would promote tooth displacement with clinical significance (24). Bimaxillary flask associated to two different investing materials did not promote first molar inclination in dentures processed by microwave energy (25). In conclusion, conventional and boiled polymerization cycles produced similar effects on gloss, roughness, hardness and impact strength of both Classico and QC-20 acrylic resins.

Resumo

O propósito neste estudo foi avaliar os ciclos de polimerização convencional e por fervura sobre o brilho, rugosidade, dureza e resistência ao impacto de resinas acrílicas. Amostras foram confeccionadas para cada resina Clássico ou QC-20 (n=20) em moldes de gesso obtidos de matrizes metálicas retangulares incluídas em muflas metálicas. A proporção monômero/polímero das resinas e manipulação foram de acordo com as recomendações dos fabricantes e a massa convencionalmente incluída em muflas metálicas. Após polimerização nos ciclos (1) convencional: 74 °C por 9 horas (Clássico) e (2) fervura: 20 min (QC-20), as amostras foram demufladas após esfriadas em temperatura ambiente e convencionalmente acabadas e polidas. As propriedades foram avaliadas após armazenagem das amostras em água a 37 °C por 24 h. O brilho foi verificado com medidor Multi Gloss 268 (Konica Minolta), a rugosidade avaliada com rugosímetro Surfcometer SE 1700 (Kosaka), a dureza Knoop foi obtida com microdurômetro HMV-200 (Shimadzu) e a resistência ao impacto determinada pelo sistema Charpy (Otto Wolpert Werke). Os dados submetidos ao teste t de Student ($\alpha=0.05$) mostraram que Os resultados foram: brilho: 67,7 e 62,2 para Clássico e QC-20, respectivamente; rugosidade: 0,874 e 1,469 Ra- μ m para Clássico e QC-20, respectivamente; dureza: 27,4 e 26,9 para Clássico e QC-20, respectivamente; e resistência ao impacto: 37,6 e 33,6 kgf/cm² para Clássico e QC-20, respectivamente. Não houve diferença estatisticamente significativa entre as resinas para as propriedades avaliadas. Conclui-se que os ciclos de polimerização convencional e por fervura promoveram similares efeitos sobre o brilho, rugosidade, dureza e resistência ao impacto para ambas as resinas Clássico e QC-20.

Acknowledgements

The authors thank the support of CNPq/UNICAMP/PIBIC for the development of this research at Piracicaba Dental School, UNICAMP, SP, Brazil.

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Received November 6, 2015
Accepted February 17, 2016