

METHODOLOGIES



Polymerization cycles on hardness and surface gloss of denture bases

Rafael Leonardo Xediek Consani, Maria Giulia R. Pucciarelli, Marcelo F. Mesquita, Moises C. F. Nogueira, Valentim A. R. Barão

Department of Prosthodontics and Periodontology, Piracicaba Dental School, State University of Campinas, Piracicaba, SP, Brazil

Correspondence

Dr. Rafael Leonardo Xediek Consani,
Piracicaba Dental School, State
University of Campinas, 901 Limeira
Ave., 13414-903 Piracicaba, SP, Brazil.
Phone: +55 19 2106-5296,
E-mail: rconsani@fop.unicamp.br

Received 12 November 2014;

Accepted 14 December 2014

doi: 10.15713/ins.ijcdmr.8

How to cite the article:

Rafael Leonardo Xediek Consani, Maria
Giulia R. Pucciarelli, Marcelo F. Mesquita,
Moises C. F. Nogueira, Valentim A. R. Barão,
"Polymerization cycles on hardness and
surface gloss of denture bases," Int J Contemp
Dent Med Rev, vol. 2014, Article ID 041114,
2014. doi: 10.15713/ins.ijcdmr.8

Abstract

The aim of this study was to evaluate different polymerization cycles on the hardness and surface gloss of acrylic resins for denture bases. Classico and Vip Cril Plus acrylic resins samples were made in dental stone molds. Powder-liquid ratio and resin manipulation were according to the manufacturers' instructions, and the resulting mass pressed in metallic flasks. The polymerization cycles were A - hot water bath at 74°C for 9 h; B - hot water bath at 74°C for 8 h + 100°C for 1 h, and C - hot water bath at 74°C for 2 h + 100°C for 1 h. After polymerization, the samples were deflasked and submitted to finishing and polishing procedures, and stored in water at 37°C for 24 h. A hardness indenter with load of 25 gf for 10 s evaluated the Knoop hardness values. A gloss meter evaluated the surface gloss using a light incidence of 60°. Data were submitted to ANOVA and Tukey's test ($\alpha = 0.05$). Hardness: There were significant differences between resins regardless of polymerization cycles (Classico = 22.28 and Vip Cril Plus = 25.83). Significant differences occurred among polymerization cycles regardless of resins (A = 25.83, B = 24.64 and C = 21.73). There was similarity for the resin and cycle interaction (Classico: A = 24.51, B = 22.68 and C = 19.65; Vip Cril Plus: A = 27.15, B = 26.53 and C = 23.81). Surface gloss: Significant differences were shown between resins regardless of polymerization cycle (Classico = 57.26 and Vip Cril Plus = 49.38) and between polymerization cycles regardless of resin (A = 48.82, B = 53.46 and C = 57.68). Statistical differences were also found for the resin and cycle interaction (Classico: A = 52.32, B = 63.79 and C = 55.67; Vip Cril Plus: A = 45.32, B = 43.14 and C = 59.69). Different polymerization cycles showed similar effects on hardness and different effects on the surface gloss of denture base acrylic resins.

Keywords: Acrylic resins, curing cycles, hardness, surface gloss

Introduction

Acrylic resins have been used for complete denture bases due to their several favorable mechanical conditions. Although considered rigid, acrylic resin is a relatively soft material that can be easily finished and polished with low abrasive materials commonly used in prosthetic laboratories.^[1]

The hardness of acrylic resins is an index of the ability of the material to resist wear and abrasion and used to compare materials of the same category. There are several tests used to determine surface hardness; however, the majority is based on the capacity of the material surface to resist the penetration of a diamond point under the action of a specific load.^[1]

In recent decades, a classic study showed that the surface of complete denture bases must be polished to provide comfort to patients, as well as adequate aesthetics, prosthesis hygiene and

low level of biofilm retention.^[2] However, repeated mechanical cleaning procedures can change the denture base smoothness and promote a rough surface that favors the adhesion and colonization of microorganisms existing in the oral environment.

It has long been claimed that denture-base irregularities are reservoirs for microorganism adhesion and consequent biofilm accumulation.^[3,4] As such, an increased surface roughness increases the levels of microorganism adhesion.^[5,6] The influence of the surface roughness on plaque accumulation is more important than the surface free energy,^[7] and a roughness of 0.2 μm is the threshold at which microbial infestation occurs on acrylic resins.^[8]

Microporosities resulting from an inadequate polymer-monomer ratio, distinct stages of the mass during the packing process, and inadequate pressure on the mass inside the flask

during the polymerization procedure are factors that influence the surface smoothness of the acrylic resins.^[1] A previous study has shown that different polymerizing cycles affect the linear dimensional change, hardness and impact strength of acrylic resins.^[9] In addition to factors inherent to the physical properties of the acrylic resins and different protocols of procedures,^[10-14] it is possible that a positive correlation exists between hardness and the surface gloss of acrylic resins for denture bases.

The purpose of this study was to verify the effect of polymerization cycles on the hardness and surface gloss of thermopolymerized acrylic resins for denture bases (Classico and Vipi). The work hypothesis was that different polymerization cycles would not affect these properties.

Materials and Methods

Materials and samples preparation

Classico (Classico Dental Products, Sao Paulo, SP, Brazil) and Vipi Cril Plus (Vipi Dental Products, Pirassununga, Sao Paulo, SP, Brazil) commercial acrylic resins were evaluated in this study. According to the manufacturers, the products are thermoactivated and presented the following basic chemical composition: Polymer consisting of poly-methylmetacrylate (PMMA) and monomer of methylmetacrylate (MMA). Hydroquinone as inhibitor. Crosslink agents for improving the mechanical strength and pigments to achieve a pink coloration are present in the materials.

Rectangular wax patterns (66.0 mm × 13.0 mm × 4.5 mm) were included in metallic flasks (Safrany; Safrany Metallurgy, Sao Paulo, SP, Brazil) with Type III dental stone (Herodent; Vigodent, Petropolis, RJ, Brazil), prepared according to the manufacturer's instructions (100 g powder to 30 mL water). The metallic flask pressing was at room temperature for 1 h. After this time, the wax patterns were deflasked and the stone molds cleaned with an aqueous solution of household detergent (Ype; Amparo Chemical Products, Amparo, SP, Brazil). The stone molds were filled with silicone rubber (Zetaflex; Zhermack, Rovigo, Italy), and in which metallic dies (65.0 mm × 12.0 mm × 3.5 mm) were included.^[9] The metallic dies removed from the flasks were after silicone polymerization, and the impression quality carefully evaluated in each silicone mold.

The acrylic resins were proportioned and manipulated according to the manufacturers' instructions and inserted into the silicone molds. The polymer-monomer ratio for both acrylic resin types was 3:1 in volume. In the initial packing, the resin pressing was in the dough like stage using a hydraulic press (Delta; Delta Dental Press, São Paulo, SP, Brazil). After excess resin removal, the definitive flask closure was with a load of 1,250 kgf. Afterward, the flask was transferred to a conventional flask carrier.

Thirty samples were prepared for each acrylic resin type ($n = 10$) according to the protocols established for polymerization cycles (Anusavice, 2003). They were the following: A - hot water bath at 74°C for 9 h, B - hot water bath at 74°C for 8 h + 100°C

for 1 h, and C - hot water bath at 74°C for 2 h + 100°C for 1 h. The polymerization of the acrylic resins was in an automatic unit (Termotron; Termotron Dental Equipaments, Piracicaba, SP, Brazil), and the samples deflasked after the flask cooling at room temperature.

Samples were conventionally finished using an abrasive stone and abrasive papers in decreasing granulations (Norton; Norton Abrasives, Guarulhos, SP, Brazil). Initial polishing was made using a bench lathe (MM; Metallium Electric Engenes, Marilia, SP, Brazil), employing pumice slurry sequentially with white and black rotating brushes, and a felt cone. Definitive polishing was with a flannel brush and universal slurry (Kota; Kota Trade and Manufacture, Sao Paulo, SP, Brazil).

Knoop hardness measurement

An indenter (Shimadzu HMV 200; Shimadzu Co., Kyoto, Japan) with load of 25 gf for 10 s assessed the samples for the Knoop hardness test. Three measurements in each sample at different sites along the diametrical line (center, and left and right ends) pondered the values for statistical purposes.

Surface gloss measurement

A Multi Gloss 268 meter (Konica Minolta, Ramsey, NJ, USA) calibrated for an illumination of 60° evaluated the surface gloss of the samples. Four measurements in each sample at different sites along the diametral line pondered the values for statistical purposes.

Statistical analysis

Two-way ANOVA analysis (resin and polymerization cycle) evaluated the results of hardness and surface gloss. Tukey's test compared differences with a probability level set at $\alpha = 0.05$.

Results

Knoop hardness

Two-way ANOVA analysis revealed significant differences in the hardness values for the Classico and Vipi Cril Plus materials in relation to resin ($P = 0.00001$) and polymerization cycle ($P = 0.00002$). The interaction between resin and cycle ($P = 0.53455$) was not significant.

Regardless of polymerization cycle, Tukey's test showed that there was a statistically significant difference in the hardness of the resins, when the Vipi resin demonstrated higher hardness [Table 1].

Regardless of resin type, Tukey's test showed that there was statistically significant difference in the hardness values due to the polymerization cycles. The A and B cycles promoted statistically similar values between them and higher when compared with the C cycle [Table 2].

There was no statistically significant difference in the hardness values when comparing each resin in the different polymerization cycles, and between resins in each cycle [Table 3].

Table 1: Knoop hardness means (SD) for acrylic resins, regardless of polymerization cycle

Resin	Hardness
Classico	22.28 (1.70) ^b
Vipi Cril Plus	25.83 (2.07) ^a

Means followed by different letters differ statistically by Tukey's test (5%).
SD: Standard deviation

Table 2: Knoop hardness means (SD) for polymerizing cycles, regardless of resin

Polymerization cycle	Hardness
A (74°C/9 h)	25.83 (2.07) ^a
B (74°C/8 h+100°C/1 h)	24.64 (2.07) ^a
C (74°C/2 h+100°C/1 h)	21.73 (1.59) ^b

Means followed by different letters differ statistically by Tukey's test (5%).
SD: Standard deviation

Table 3: Knoop hardness means (SD) in relation to resin and polymerization interaction

Resin	Polymerization cycle		
	A (74°C/9 h)	B (74°C/8 h+100°C/1 h)	C (74°C/2 h+100°C/1 h)
Classico	24.51 (2.98) ^{aA}	22.68 (1.70) ^{aA}	19.65 (2.72) ^{aA}
Vipi Cril Plus	27.15 (2.07) ^{aA}	26.53 (2.07) ^{aA}	23.81 (1.59) ^{aA}

Means followed by same lowercase letters in each column and capital letters in each row do not differ statistically by Tukey's test (5%)

Surface gloss

Two-way ANOVA analysis revealed significant difference in the gloss values for Classico and Vipi Cril Plus materials in relation to resin ($P = 0.00032$) and polymerization cycle ($P = 0.00218$). The interaction between resin and polymerization cycle ($P = 0.00007$) was also statistically significant. Regardless of polymerization cycle, Tukey's test shows that there was statistically significant difference in the gloss between resins, with Classico showing higher value [Table 4].

Table 5 shows that there was statistically significant difference in surface gloss when the polymerization cycles were compared, regardless of resin. The C cycle presented a statistically lower value than the A cycle. The B cycle showed an intermediate value.

There was statistically significant difference in the surface gloss values in the resin and polymerization cycle interaction. For Classico, the B cycle presented statistically higher value than the A and C cycles, both with similar values each other. For Vipi Cril Plus, the C cycle showed significantly greater gloss than the A and B cycles, which presented similar values each other. The values were without significant difference between resins only for the C cycle [Table 6].

Discussion

This study evaluated the influence of polymerization cycles (74°C/9 h, 74°C/8 h+100°C/1 h and 74°C/2 h+100°C/1 h)

Table 4: Surface gloss means (SD) for acrylic resins, regardless of polymerization cycle

Resin	Gloss
Classico	57.26 (9.64) ^a
Vipi Cril Plus	49.38 (6.58) ^b

Means followed by different letters differ statistically by Tukey's test (5%).
SD: Standard deviation

Table 5: Surface gloss means (SD) for polymerization cycle, regardless of resin

Polymerization cycle	Gloss
A (74°C/9 h)	48.82 (7.30) ^b
B (74°C/8 h+100°C/1 h)	53.46 (4.37) ^{ab}
C (74°C/2 h+100°C/1 h)	57.68 (7.30) ^a

Means followed by different letters differ statistically by Tukey's test (5%).
SD: Standard deviation

Table 6: Surface gloss means (SD) in relation to resin and polymerization interaction

Resin	Polymerization cycle		
	A (74°C/9h)	B (74°C/8 h+100°C/1 h)	C (74°C/2 h+100°C/1 h)
Classico	52.32 (4.37) ^{aB}	63.79 (6.49) ^{aA}	55.67 (9.64) ^{aB}
Vipi Cril Plus	45.32 (6.58) ^{bB}	43.14 (8.98) ^{bB}	59.69 (7.30) ^{aA}

Means followed by different lowercase letters in each column and capital letters in each row differ statistically by Tukey's test (5%). SD: Standard deviation

on the hardness and surface gloss of Classico and Vipi Cril Plus thermoactivated acrylic resins.

Knoop hardness

Surface hardness is an important property for the comparison of restorative materials. Hardness is the resistance to permanent penetration, and the most common concept of hard and soft materials is the relative resistance that they offer to indentation.^[15]

In the current study, although the interaction between resin and polymerization cycle was not statistically significant ($P = 0.53455$), Tukey's test showed that the hardness values were different for resins, regardless of polymerization cycle. The Vipi Cril Plus resin presented greater hardness value [Table 1]. A previous study evaluating the effect of polymerization cycles on the linear dimensional change, hardness and impact strength of acrylic resins showed that these properties were affected differently after deflasking and water storage at 74°C for 7 and 30 days.^[9] In addition, while differences were shown in the hardness values of acrylic resins activated by conventional cycle, boiling water or microwave energy,^[16] studies from past decades reported that the surface hardness was not influenced by the acrylic resin type.^[17,18]

Conversely, the hardness values were not significantly different in relation to resin and polymerization cycle interaction [Table 3]. Since no statistical difference occurred for this interaction, the working hypothesis that different polymerization

cycles would not differently affect the acrylic resin hardness was accepted. It is possible that these results occurred because the resins evaluated in the current study have similar chemical basic compositions. This fact could cause the same degree of conversion to transform monomer molecules into polymer molecules. Despite of heat-polymerized acrylic resins compositions be similar, a previous report showed that longer curing cycles produced samples with the highest degree of cure; however, it is not important in determining the extent to which the polymer beads dissolve in the monomer. The conversion of monomer to polymer is time-dependent, and greater conversion rate occurs by raising the curing temperature up to 100°C. However, there were no consistent differences between samples obtained by different curing cycles.^[19] These findings suggested that polymerization procedures, with different temperatures and shorter times, did not cause significant effects on the degree of conversion and polymerization rates of resins and, consequently, on the surface hardness.

Another interesting observation was that regardless of resin [Table 2], the longer polymerization cycles (A and B) promoted higher hardness values than the short cycle (C). If we disconsider the similarity of the hardness values between resins in the interaction [Table 3], this finding may reflect the different residual monomer levels resulting from each polymerization cycle. A classic study showed that the residual monomer affects adversely the mechanical properties due to their plasticizing effect on the polymeric chain, allowing greater resin deformation under loading.^[20] Another work revealed that shorter polymerization cycles promote higher levels of residual monomer.^[21] Given these facts, it is reasonable to assume that the residual monomer rate may have an inversely proportional relationship to hardness of acrylic resins.

Temperature and time of polymerization affect the residual MMA content of heat-polymerized denture base polymers.^[22] However, the similar roughness of acrylic resins may explain why the polymerization methods does not influence the adherence level of *Candida* species on acrylic resin samples,^[23] a finding that may be clinically significant for the oral health of complete denture wearers.

Thermoactivated acrylic resins (conventional, chemical or microwaved) showed significant differences in hardness before the procedure of accelerated artificial aging^[24] or prior to the addition of glass fibers used to improve the mechanical resistance of resins.^[25] Another interesting finding was that the microwaved and hot water bath polymerization methods in association with a special double flask did not change the hardness of acrylic resins.^[26] A recent study showed that, although differences exist in modulus and flexural strength, there was the similarity in the surface microhardness of acrylic resins formulated with PMMA pearls of different molecular weights.^[27]

The conflicting results obtained by different studies about surface hardness reflect the mechanical behavior of acrylic resins. In the agreement with this supposition, a recent study showed that unfilled PMMA and microfilled acrylate polymers presented lower Martens hardness values after abrasive toothbrushing than

aesthetic restorative materials performed by CAD/CAM.^[28] These findings show the complexity of the variables involved in the discussion of this study. Given the complexity of the results of this investigation, other studies would be appropriate to verify the effects of polymerization cycles on the adaptation of denture bases, and the bond and displacement of artificial teeth.

Surface gloss

In the current study, there were significant differences between resins regardless of cycles [Table 4], between cycles regardless of resin [Table 5], and in the resin and cycle interaction [Table 6]. Considering the occurrence of statistical differences, the working hypothesis that different polymerization cycles would not affect the acrylic resin gloss was not accepted. The roughness and surface gloss are material-dependent and influenced by the polishing time and force applied. The patterns of improvement in roughness and gloss vary considerably among materials; however, gloss appears to be more consistent than roughness, despite their close association.^[29]

Based on these considerations, it is reasonable that a similar effect would occur with the denture base acrylic resins since the smoothness of the material would depend on the surface hardness and, therefore, the surface gloss. This assumption becomes more evident when decreased brightness and color values of thermoplastic resins occurred by the immersion in cleaning solutions. However, the different values of reduction (ΔE^*) were clinically unacceptable for water and peroxide solutions.^[30] In addition, different beverages cause different changes in the gloss of cold- and heat-polymerized acrylic resins. Statistically significant effects occurred between polished and unpolished surfaces.^[31] The cleaning solutions and beverages evaluated by these authors promoted, probably, different chemical effects on the gloss in relation to the resin hardness. Thus, the resin hardness depends on the degree of conversion and consequent levels of roughness/smoothness, which are closely associated to the degradation process.

Undoubtedly, significant differences can exist among polishing techniques used for acrylic resins. Conventional laboratory polishing produces smoothest surfaces in denture base acrylic resins.^[32] In the current study, the conventional laboratory polishing promoted the smoothest surface and consequently, the brightest surface. In addition, the ranked surface gloss for resin-based composites was according to polishing systems,^[33] and there was an interaction between direct composite resins and the polishing systems for the gloss value.^[34] As previously reported, it is reasonable to assume that similar findings would occur for acrylic resins since smoothness and surface gloss depend on the surface hardness.

Possibly, for this reason, the gloss values were significantly different for the interaction between resin and polymerization cycle in both resins. For Classico, the B cycle resulted in greater surface gloss when compared to the A and C cycles. For the Vipi Cril Plus resin, the C cycle presented a significantly greater value in relation to the A and B cycles. Values were statistically similar

only for the C cycle when the resins were compared [Table 6]. Analyzing the interaction, there were different values for each resin, and these findings are difficult to understand and discuss. Probably, these results occur due to influence of unknown factors that affect the surface brightness. In addition, a recent study showed that unfilled PMMA and microfilled acrylate polymer presented less gloss retention after abrasive toothbrushing than aesthetic restorative materials performed by CAD/CAM.^[28] The finding that brightness and color of thermoplastic resins are reduced differently when immersed in cleaning solutions emphasizes this result; these differences in values of reduction (ΔE^*) for acrylic resins were clinically unacceptable.^[30]

The scattering coefficient that determines the color and translucency is dependent of the surface gloss of translucent materials. The inverse relationship between gloss/roughness and scattering coefficient is due to increased diffuse reflection of the rougher surface or due to the higher scattering index or both,^[35] reinforcing the findings of the current study.

Based on these considerations, the surface gloss does not closely relate to the level of residual monomer resulting from each polymerization cycle as occurs for hardness. The measurement of the brightness is due to the light incident at certain angulation, and consequent absorption and reflection. Thus, the different polymerization cycles appear to cause little effect on these properties. For better understanding of the results, future studies are necessary to investigate other variables that affect the brightness of acrylic resins.

Conclusion

Based on the statistically analyzed and discussed results, it was possible to draw the following conclusion: The different polymerization cycles promoted similar hardness and different surface gloss on thermoactivated acrylic resins for denture bases.

Acknowledgments

The authors gratefully acknowledge the support provided by the PIBIC/CNPq/UNICAMP at Piracicaba Dental School, State University of Campinas, Brazil.

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