ACP
AMERICAN COLLEGE OF PROSTHODONTISTS®

Influence of Double Flask Investing and Microwave Heating on the Superficial Porosity, Surface Roughness, and Knoop Hardness of Acrylic Resin

Celia Marisa Rizzatti-Barbosa, DDS, MSc, PhD[1] & Margarete C. Ribeiro-Dasilva, DDS, MSc, PhD[2]

[1] Department of Prosthodontics and Periodontology, Piracicaba Dental School, State University of Campinas, Piracicaba, Brazil
[2] University of Florida College of Dentistry, Gainesville, FL

Keywords
Acrylic resin; flask; surface roughness; Knoop hardness; superficial porosity; microwave energy.

Correspondence
Celia M. Rizzatti-Barbosa, Departamento de Protese e Periodontia, Faculdade de Odontologia de Piracicaba, Unicamp, Av. Limeira 901 Bairro Areiao, Piracicaba SP 13414 018, Brazil. E-mail: rizzatti@fop.unicamp.br

Accepted July 7, 2008

doi: 10.1111/j.1532-849X.2009.00469.x

Abstract

Purpose: Simultaneous polymerization of maxillary and mandibular complete dentures with teeth in occlusion through investing in a double special flask has been described as a more rapid and efficient method to polymerize prostheses than the conventional method; however, no study has been done to verify important properties of resin, including superficial porosity, surface roughness, and hardness, when processed by this technique. The purpose of this study was to verify if the simultaneous polymerization associated with microwave heating may alter the superficial porosity, surface roughness, and Knoop hardness of acrylic resin.
Materials and Methods: Resin specimens processed in single and double dental flasks were compared using microwave energy and warm water methods. Four groups were tested according to the investing flask and the method of resin cure: Group I control specimens (n = 15) were invested in single metal flasks and cured by warm water at 74°C for 9 hours. Group II (n = 15) specimens were invested in single polyvinyl chloride flasks and cured by microwave energy at 90 W for 20 minutes plus 450 W for 5 minutes. Group III (n = 30) and Group IV (n = 30) specimens were processed by simultaneous polymerization in double flasks and cured by the same warm water and microwave energy protocols, respectively.
Results: No significant differences were found in mean superficial porosity (8.06 ± 2.28 pore/cm^2), surface roughness (0.14 ± 0.03 μm), or Knoop hardness (19.66 ± 2.25 kgf/mm^2) between the control group (GI), and the other three experimental groups ($p > 0.05$).
Conclusion: Processing acrylic resin in a double flask heated by either warm water or microwave energy does not alter the resin's superficial porosity, surface roughness, or Knoop hardness; however, other properties of resin should be analyzed using this denture processing technique.

Changes in denture occlusion caused by processing resin may result in traumatic occlusion, irregular distribution of occlusal stresses on underlying tissues, and alterations of oral function, denture comfort, and chewing efficiency.[1] Occlusal tooth harmony is important in complete dentures,[2] mainly in the total rehabilitation of prostheses supported by implants.[3] Occlusal interferences can take place due to errors introduced in clinical or laboratory procedures during the fabrication of the prosthesis.[4] Dimensional changes and distortion of the denture due to the investing stone mold and the heating of acrylic resin can promote tooth movement and, consequently alterations in the occlusal contacts and occlusal vertical dimension (OVD).[5,6]

Simultaneous polymerization of maxillary and mandibular complete dentures with the teeth in occlusion by means of a special double flask (DF), has been described as a more rapid and simple method for investing and polymerizing prostheses.[7] The first designed DF was a metal copper–aluminum flask (DMF) for simultaneous polymerization of both maxillary and mandibular prostheses in a warm water bath (Dental VIPI Ltd, Pirassununga, Brazil). The double polyvinyl chloride flask (DPVCF) (Dental VIPI Ltd) was developed following the same principles for simultaneous processing of both dentures in occlusion through microwave energy heating.[7] This new technique associating acrylic curing with microwave energy can be considered a clean method[8–10] that saves time,[11]

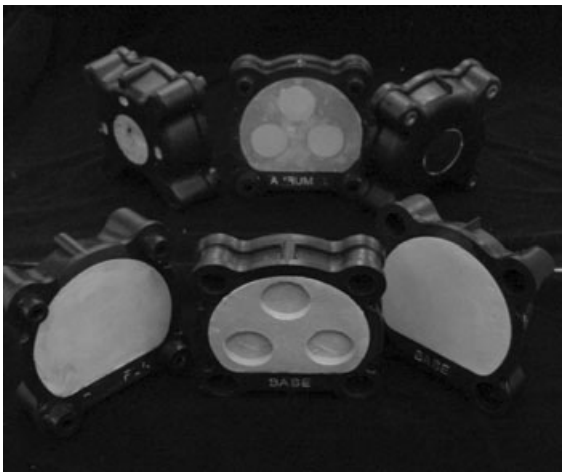

Figure 1 Front and back (in the mirror) views of group IV patterns invested in the double polyvinyl chloride flask.

reduces occlusal interferences, preserves the teeth occlusion, and maintains the OVD;[7] however, to date, no investigation into the effects of this simultaneous double processing technique on the porosity, roughness, and hardness of the acrylic resin dough when using either a warm water bath or microwave energy heating methods has been made. Therefore, the aim of this work was to evaluate the effect of DF investing on the superficial porosity, surface roughness, and Knoop hardness of acrylic resin cured by warm water and microwave energy.

Materials and methods

Water bath-cured (Vipi-Cril®) and microwave-cured (Vipi-Wave®) resins were used to make the specimens in this study (Dental VIPI Ltd). Silicone-shaped disc patterns (30.0 mm diameter, 3.0 mm thickness) were used to make 90 resin specimens. The silicone patterns were randomly divided into one control group and three experimental groups. The patterns of the control group (Group I; n = 15) were invested in single metal flasks (SMF), and the specimens of Vipi-Cril® resin were cured by warm water in a curing tank at 74°C for 9 hours. The silicone patterns of Group II (n = 15) were invested in single polyvinyl chloride flasks (SPVCF) developed for microwave irradiation, and the specimens of Vipi-Wave® resin were cured by microwave energy in a domestic microwave oven at 90 W for 13 minutes plus 450 W for 5 minutes. The patterns of Group III (n = 30) and Group IV (n = 30) were invested in special double metal flasks (DMF) and DPVCF, respectively (Fig 1). The resin specimens were cured by warm water (Vipi-Cril®) and microwave energy (Vipi-Wave®), respectively, through the same heating protocol used to invest SMF and SPVCF specimens. Three patterns were invested simultaneously in each single flask (SF), and six patterns were invested in each DF, three in the lower half of the flask and three in the upper half. The investing of patterns was done with type III dental stone, according to the instruction of the manufacturer presented in previous publications.[7] The flasks were compressed (0.5 ton), and were opened; the silicone patterns were removed and the cavities inspected for integrity. All molds were washed with warm water and a neutral detergent and coated with a separating medium (Al-Cote®, Dentsply Ind. e Com. Ltda, São Paulo, Brazil).

The resin specimens were prepared at room temperature ($21 \pm 2°C$) following the manufacturer's guidelines. After filling the molds with the dough resin, the flasks were fitted and maintained under compression (1.25 ton) in a hydraulic bench press for 10 minutes. After compression, the flasks were processed according to the experimental protocols, and the flasks were allowed to cool at room temperature after processing. After deflasking, excess material was removed from the specimens with 320-grit sandpaper in a polishing machine (Model APL-4, Arotec, Sao Paulo, Brazil). Specimens were finished with progressive abrasive papers (400, 600, 1200 grit) and cleaned in distilled water for 2 minutes in an ultrasound bath.

Hardness tests were performed by a hardness tester (Shimadzu HMV-2000, Shimadzu Scientific Instruments, Columbia, MD) equipped with a Knoop diamond penetrator. A 25-g load was applied for 10 seconds. Three penetrations were obtained for each specimen (one on the center, two on the border), and the average hardness was calculated.

To evaluate superficial porosity, the specimens were immersed in a solution of permanent black ink for 12 hours, washed for 10 seconds, and dried with absorbent paper. Three equidistant surface areas of 10 mm^2 were randomly delimited in each specimen and observed under 40× magnification in a stereo light microscope (Leica CLS 100×, Leica Microsystems, Heerbrugg, Switzerland). The number of pores per area was determined for each specimen, and an average value was calculated for each group.

Surface roughness (R_a) of the acrylic specimens was measured using a profilometer (Surfcorder SE 1700, Kosaka Laboratory Ltd, Kosaka, Japan) with a 0.01 μm resolution, calibrated at a specimen length of 0.8 mm, 2.4 mm percussion of measure, and 0.5 mm/sec. Three readings were made for each specimen, and a mean value was calculated.

Results

The Knoop hardness, superficial porosity, and surface roughness means and standard deviations for each group are presented in Table 1. Statistical analysis was performed by comparison of the means of hardness, superficial porosity, and surface roughness scores by ANOVA, and are presented in Tables 2, 3, and 4, respectively. No statistical differences were found among the groups ($p > 0.05$), and the values were within the limits of the American Dental Association (ADA).[12]

Discussion

Superficial porosity and roughness have a strong relationship to the colonization of resin by oral microorganisms, because surface defects and porosities provide favorable niches for microbial colonies' development and differentiation.[13] This was confirmed by a laboratory study[14] showing the relationship between surface roughness of denture resin and microorganism infection. In vivo studies made by Bollen et al[15] and Quirynen et al[14] noted that clinically acceptable roughness in the

Table 1 Means and standard deviations of the Knoop hardness (kgf/mm^2), superficial porosity (pore/cm^2), and surface roughness (μm) tests

	Group I		Group II		Group III		Group IV	
Test	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Knoop hardness	19.66[a]	±2.25	18.43[a]	±1.56	20.90[a]	±2.00	20.07[a]	±2.28
Superficial porosity	8.06[a]	±2.28	8.14[a]	±1.18	7.13[a]	±2.20	7.83[a]	±2.11
Surface roughness	0.14[a]	±0.03	0.13 [a]	±0.02	0.13[a]	±0.04	0.12[a]	±0.03

[a]No significant difference among groups (ANOVA); $p = 0.05$.

oral environment should not exceed 0.2 μm. These properties may be associated with the conversion of acrylic monomers in polymers, where a lack in this conversion can negatively affect hardness and superficial porosity.[11,16]

In the present study, the surface roughness of specimens varied between 0.12 and 0.14 μm, and was insufficient for retaining most bacteria (Table 1), regardless of the flask investing or the heating method used. Therefore, one explanation is that a reasonable polymerization of acrylic mass on the surface could have occurred, leading to acceptable values in the analyzed properties.

The incomplete polymerization of monomers can be associated with various conditions. Mistaken proportions of polymer and monomer, inadequate agglutination of powder particles to the liquid, application of resin at an improper stage of the reaction, heating temperature and a too-short curing cycle can be associated with a lack in hardness, superficial porosity and surface roughness of resin.[18] Specifically, roughness can be attributed to the monomer vaporization associated with the exothermic reaction.[18]

Due to the larger amount of resin in the same flask when investing resin in DF, especially in DFPVC, the result expected was an increase in the alteration of resin promoted by the higher exothermal heating from the polymerization reaction associated with the abrupt heating of the microwave processing.[19] Instead of the expected results, no difference was found in any of the analyzed properties (porosity, Knoop hardness, and superficial roughness) among the specimens, independent of the heating or investing technique. Ilbay et al did not associate porosity and modification in hardness when using microwave to process resin;[10] however, Bafile et al found moderate to severe porosity in the resin specimens polymerized by microwave.[20] These variations in results probably are due to the different methodologies used.

The similarity in the results observed in the properties analyzed in the present study could be explained by the efficient diffusion of heat through the surrounding materials (dental stone mold) in both heating polymerization methods and by the efficacy in the monomer polymerization, decreasing exothermic heat production.

The advantage of using DF investing over SF investing is that this new technique makes it possible to polymerize both prostheses in dental occlusion in one investment. This procedure is efficient and simple, maintaining the dental occlusion of the artificial teeth,[7] and is also associated with the absence of changes in such properties of resin as surface roughness, hardness, and superficial porosity, as demonstrated in the present experiment. When associated with microwave energy processing, the main advantage is that microwave heating can quickly and efficiently polymerize the resin, and it has the potential for saving time in processing dentures, maintaining the properties evaluated within the ADA recommendations.[12] These associations of investing methods are relatively new in dentistry, despite the use of microwave processing. The disadvantage is that it involves using the special flask made specifically for this technique.

Confirming the use of microwave energy in the processing of acrylic resin is based on classic studies examining the use of microwave energy to polymerize acrylic resin. Shlosberg et al[21] tested hardness in resins processed by microwave energy. No statistical differences were noted when microwave or water-bath curing was used. Reitz et al[22] compared porosity and hardness of microwave and water-bath cured specimens and found no significant differences in this and other properties. According to the authors, the frequency and size of porosity in thick specimens could be reduced to 30% by a longer polymerization time at a lower wattage. Smith et al[23] investigated hardness of resins using a water bath, microwave energy, and visible light. They proved that microwave curing has little

Table 2 One-way ANOVA for Knoop hardness test

Variation source	*df*	SS	MS	F	*p*-value
Heating method*	1	0.00000018	0.00000017	0.01	0.93$^{(ns)}$
Flask**	2	0.0000033	0.000016	0.69	0.51$^{(ns)}$
Heating method and flask	2	0.0000020	0.0000010	0.04	0.95$^{(ns)}$

*Warm water or microwave energy.
**Single or double investing flask.
$^{(ns)}$No significant difference; $p = 0.05$.

Table 3 One-way ANOVA for superficial porosity test

Variation source	*df*	SS	MS	F	*p*-value
Heating method*	1	0.01409	0.01590	0.91	0.34$^{(ns)}$
Flask**	2	0.00052	0.00026	0.02	0.98$^{(ns)}$
Heating method and flask	2	0.02241	0.01120	0.72	0.48$^{(ns)}$

*Warm water or microwave energy.
**Single or double investing flask.
$^{(ns)}$No significant difference; $p = 0.05$.

Table 4 One-way ANOVA for surface roughness test

Variation source	*df*	SS	MS	F	*p*-value
Heating method*	1	0.00013	0.000134	0.17	$0.68^{(ns)}$
Flask**	2	0.00898	0.004493	5.59	$0.53^{(ns)}$
Heating method and flask	2	0.00014	0.000071	0.09	$0.91^{(ns)}$

*Warm water or microwave energy.
**Single or double investing flask.
$^{(ns)}$No significant difference; $p = 0.05$.

effect on the properties of resins evaluated. Recently, Lai et al[24] reported that there were no significant differences in the surface hardness and the domain size distribution of the effective rubber phase when using microwave polymerization of resin. The choice of a suitable power and polymerization time is important to reduce porosity to a minimum level. Both the length of polymerization and the proportion of the resins used in the present experiment were according to manufacturer's recommendation.

Therefore, this new technique of investing associated with microwave energy should be further investigated to determine other properties of resin. Because it offers some important physical properties as good as conventional processing, along with the advantage of being a quicker and easier method, it should also be considered in processing removable partial dentures and complete dentures.

Conclusions

According to the experimental protocol used in this study and within the limitations of this controlled laboratory study, it can be concluded that DF investing heated by either warm water or microwave energy is not a factor that alters surface roughness, superficial porosity, or Knoop hardness of acrylic resin. Additionally, a translational study should be done to prove our finding.

References

1. Ruffino AR: Improved occlusal equilibration of complete dentures by augmenting occlusal anatomy of acrylic resin denture teeth. J Prosthet Dent 1984;52:300-302
2. Dubojska AM, White GE, Pasiek S: The importance of occlusal balance in the control of complete dentures. Quintessence Int 1998;29:389-394
3. Garg AK: Analyzing dental occlusion for implants: Tekscan's TScan III. Dent Implantol Update 2007;18:65-70
4. Zakhari KN: Relation of investing medium to occlusal changes and vertical opening during denture construction. J Prosthet Dent 1976;36:501-509
5. LaBarre E, Giusti L, Pitigoi-Aron G: Addressing problems in complete dentures. Compend Contin Educ Dent 2007;28: 538-540
6. Anusavice KJ: Philips' Science of Dental Materials (ed 11). St. Louis, Elsevier, 2004, pp. 739-741
7. Rizzatti-Barbosa CM, Machado C, Joia FA, et al: A method to reduce tooth movement of complete dentures during microwave irradiation processing. J Prosthet Dent 2005;94:301-302
8. Harrison A, Huggett R: Effect of the curing cycle on residual monomer levels of acrylic resin denture base polymers. J Dent 1992;20:370-374
9. Sanders JL, Levin B, Reitz PV: Comparison of the adaptation of acrylic resin cured by microwave energy and conventional water bath. Quintessence Int 1991;22:181-186
10. Ilbay SG, Güvener S, Alkumru HN: Processing dentures using a microwave technique. J Oral Rehabil 1994;21:103-109
11. de Oliveira VMB, Léon BLT, Del Bel Cury AA, et al: Influence of number and position of flasks in the monomer release, Knoop hardness and porosity of a microwave-cured acrylic resin. J Oral Rehabil 2003;30:1104-1108
12. American Dental Association: Council on dental materials and devices. Specification # 12 for denture base polymers. J Am Dent Assoc 1975;90:451
13. Moura JS, da Silva WJ, Pereira T, et al: Influence of acrylic resin polymerization methods and saliva on the adherence of four *Candida* species. J Prosthet Dent 2006;96:205-211
14. Quirynen M, Marechal M, Busscher HJ, et al: The influence of surface free energy and surface roughness on early plaque formation. An in vivo study in man. J Clin Periodontol 1990;17:138-144
15. Bollen CM, Lambrechts P, Quirynen M: Comparison of surface roughness of oral hard materials to the threshold surface roughness for bacterial plaque retention: a review of the literature. Dent Mater 1997;13:258-269
16. Compagnoni MA, Barbosa DB, de Souza RF, et al: The effect of polymerization cycles on porosity of microwave-processed denture base resin. J Prosthet Dent 2004;91:281-285
17. Jerolimov V, Huggett R, Brooks SC, et al: The effect of variations in the polymer/monomer mixing ratios on residual monomer levels and flexural properties of denture base materials. Quintessence Dent Technol 1985;9:431-434
18. Wolfaardt JF, Cleaton-Jones P, Fatti P: The occurrence of porosity in a heat-cured poly(methyl methacrylate) denture base resin. J Prosthet Dent 1986;55:393-400
19. De Clerck JP: Microwave polymerization of acrylic resins used in dental prostheses. J Prosthet Dent 1987;57:650-658.
20. Bafile M, Graser GN, Myers ML, et al: Porosity in denture resin cured by microwave energy. J Prosthet Dent 1991;66:269-274
21. Shlosberg SR, Goodacre CJ, Munoz CA, et al: Microwave energy polymerization of poly (methyl methacrylate) denture base resin. Int J Prosthodont 1989;2:453-458
22. Reitz PV, Sanders JL, Levin B: The curing of denture acrylic resins by microwave energy: physical properties. Quintessence Int 1985;8:547-551
23. Smith LT, Powers JM, Ladd D: Mechanical properties of new denture resins polymerized by visible light, heat, and microwave energy. Int J Prosthodont 1992;5:315-320
24. Lai CP, Tsai MH, Chen M, et al: Morphology and properties of denture acrylic resins cured by microwave energy and conventional water bath. Dent Mater 2004;20:133-141