

Influence of dye and nylon fibers on microwave-cured acrylic resin properties

Influência de corante e fibras de nylon nas propriedades de uma resina acrílica polimerizada por micro-ondas

Carmen Beatriz Borges FORTES¹
Fabrício Mezzomo COLLARES¹
Vicente Castelo Branco LEITUNE¹
Juliana Gehlen WALCHER¹
Stéfani Becker RODRIGUES¹
Susana Werner SAMUEL¹
Cesar Liberato PETZHOLD²
Valter STEFANI²

ABSTRACT

Objective

The aim of this study is to analyze if both color and nylon fibers have an influence on microwave-cured acrylic resin properties.

Methods

Rectangular and disk-shaped specimens were prepared using acrylic resins; medium pink with and without nylon fibers and colorless without nylon fibers. To obtain the rectangular specimens, a stainless steel die was used with the following dimensions: 64 X 10 x 3 mm (± 0.5 mm). To obtain disk-shaped specimens, a die 50mm (± 0.5 mm) in diameter and 0.5mm (± 0.05 mm) thick was employed. They were randomized to form groups: control (colorless acrylic) and experimental (medium pink, with and without nylon fibers), with each group consisting of ten (10) specimens rectangular in shape and five (5) disk-shaped. They were analyzed in six (6) assays (izod impact strength, $n=10$; Knoop hardness, $n=10$; glass transition temperature, $n=3$; water sorption and solubility, $n=5$; degree of monomer/polymer conversion, $n=1$; flexural strength and flexural modulus, $n=10$). All variables were subjected to the analysis of variance test followed by Tukey's multiple comparison test, at a 5% level of significance.

Results

The analysis of the monomer/polymer degree of conversion did not reveal any difference between the three groups of resins (medium pink, with and without nylon fibers and colorless resin); it was approximately 88%. The results did not show significant differences between the groups for each variable ($p>0.05$).

Conclusion

The results showed that color and presence of nylon fibers in acrylic resins did not affect the properties analyzed in this study.

Indexing terms: Acrylic resins. Microwave. Nylons.

RESUMO

Objetivo

Analisar se corantes e fibras de nylon influenciam nas propriedades de uma resina acrílica ativada por energia de micro-ondas.

Métodos

Espécimes retangulares e em forma de disco foram confeccionados em resina acrílica rosa médio com e sem fibras de nylon e incolor sem fibras. Para obter os espécimes retangulares, foi utilizada uma matriz de aço inoxidável com as seguintes dimensões: 64 X 10 X 3 ($\pm 0,5$ mm) e para obter os espécimes em forma de disco, foi utilizada uma matriz com 50mm ($\pm 0,5$ mm) de diâmetro e 0,5mm ($\pm 0,05$ mm) de espessura. Os mesmos foram randomizados para formar os grupos controle (acrílico incolor sem fibras) e experimentais (rosa médio com e sem fibras), sendo cada grupo composto por dez (10) espécimes retangulares e cinco (5) em forma de disco. Foram os mesmos então submetidos a seis (6) ensaios (resistência ao impacto izod, $n=10$; dureza knoop, $n=10$; temperatura de transição vítrea, $n=3$; sorção e solubilidade, $n=5$; grau de conversão, $n=1$; resistência à flexão e módulo de flexão, $n=10$). Todas as variáveis foram submetidas à análise de variância seguida pelo teste de comparações múltiplas de Tukey, com nível de significância de 5%.

Resultados

O grau de conversão foi de 88% para os 3 grupos de resinas e os resultados não mostraram diferença estatisticamente significativa entre os mesmos para cada variável ($p>0,05$).

Conclusão

Os resultados mostraram que o corante e a presença de fibras de nylon em resina acrílica não afetaram as propriedades analisadas nesse estudo.

Termos de indexação: Resinas acrílicas. Micro-ondas. Nylons.

¹ Universidade Federal do Rio Grande do Sul, Faculdade de Odontologia, Departamento de Odontologia Conservadora, Laboratório de Materiais Dentários. Rua Ramiro Barcelos, 2492, Rio Branco, 90035-003, Porto Alegre, RS, Brasil. Correspondência para / Correspondence to: FM COLLARES. E-mail: <fabricio.collares@ufrgs.br>.

² Universidade Federal do Rio Grande do Sul, Instituto de Química, Departamento de Química Orgânica. Porto Alegre, RS, Brasil.

INTRODUCTION

The restoration of masticatory function and aesthetics is an important aim in dentistry, mainly when patients present with extensive tooth loss. For a long time polymethylmethacrylate (PMMA) has dominated the field of denture base materials¹. This material presents adequate strength to withstand masticatory forces, maintains dimensional stability, adequate resilience, biocompatibility and high polishability. It is available in both powder and liquid form for use and processing. The powders are pure polymers, which are clear and adaptable to a wide range of pigments that are used to obtain the various shades of oral tissue and include compounds such as mercury sulfide, cadmium sulfide, cadmium selenide and ferric oxide. Dyes as well as pigments, which are occasionally used, are not as satisfactory, since they tend to leach out of the plastic through the oral fluids, which results in a gradual lightening of the shade². A cross-linking agent may also be added to the monomer component to improve the solvent resistance of the cured polymer and thereby reduce its susceptibility to solvent crazing³. In terms of resin strengtheners, materials such as nylon, viscose rayon and polyester have been explored as esthetic fibers. Nylon fibers have been successfully used to match the minute blood vessels of oral mucosa⁴.

Despite the high degree of conversion achieved in dental resins, complete conversion of aliphatic carbon-carbon double bonds is, typically, not achieved. The unconverted double bonds remaining in the resin arise from at least two situations, namely unreacted monomers or pendant double bonds at the ends of polymer chains⁵. During polymerization the monomer is reduced. As temperature increases, molecular mobility speeds up, leading to more complete polymerization. With progressive polymerization of the monomer/polymer mix, the same amount of energy is absorbed by less and less monomer. All acrylic resins contain variable residual monomer levels depending on the environment.

A denture base polymer should have a smooth and glassy surface and be capable of matching the natural appearance of soft tissue. The material should be translucent for the best esthetic effect. Color and translucency should be maintained during processing and acrylic resins should not become stained or change color in clinical use⁶.

Therefore, the purpose of this study was to analyze the influence of color and nylon fibers on the acrylic resin properties.

METHODS

VIPI WAVE acrylic resin (Dental VIPI, São Paulo, SP, Brazil) and (Clássico, São Paulo, Brazil) were used. The experimental specimens were the medium pink acrylic resin (with or without fibers); and the control samples were the colorless acrylic (without dye and nylon fibers in the composition). The specimens, observing each type of acrylic resin, were randomized to form control and experimental groups, with each group consisting of ten (10) specimens of rectangular shape and five (5) disk-shaped.

Specimen preparation

Rectangular and disk-shape specimens were prepared using acrylic resins; medium pink with nylon fiber, medium pink without nylon fiber and colorless without nylon fibers. To obtain the rectangular specimens, a stainless steel mold was used with the following dimensions: 64 x 10 x 3 mm (± 0.5 mm). To obtain disk-shaped specimens, a mold 50mm (± 0.5 mm) in diameter and 0.5mm (± 0.05 mm) thick was employed. Each stainless steel matrix is reproduced through the addition of silicone, yielding a silicone matrix. This has been included in the flask with type II dental stone (common gypsum). After solidification of the cast, the resin was included in the silicone matrix and the powder/liquid proportion was measured using analytical scales. After adding the resin, the counter-balanced muffle furnace was closed and pressed with a load of 500 kg, and immediately afterwards was opened to remove the excess resin. It was then pressed with a load of 1000 kg and after 30 minutes the polymerization was initiated. A microwave oven was used (LG, São Paulo, Brazil), with the following specifications: voltage of 120V/60Hz, output power of 1400W, microwave frequency of 2450 MHz, magnetron capable of emitting waves in three dimensions, internal capacity of 32L, turntable 28 cm in diameter), for the polymerization process. The flask was then placed on the workbench for 4 hours (to cool off completely). After this, each specimen was removed from the oven and abraded using an electric motor to remove the excess resin. The abrasion was carried out using a mounted Cutter (TDV, Pomerode, Santa Catarina, Brazil). Next, the specimens were polished with 280, 400, 600 and 1000 grit wet sandpaper. After this, the samples were measured with a digital caliper: for the rectangular specimens 10 measurements of the thickness were taken, 10 measures of the width and 3 of the length. For the disks, 8 measurements were taken of the diameter and 8 of the thickness, and the average value

was calculated. This procedure was performed to obtain a more uniform measurement of the specimens. Polishing was performed using abrasive pumice and Spanish white, in order to afford smoothness and surface gloss to the material.

Izod impact strength

Ten rectangular specimens (n=10) were tested in an EMIC AIC-1 (a universal testing machine), to determine the impact energy required to fracture them. The test was performed according to the specifications of ASTM D256 (American Society Test Methods).

Knoop Hardness

Five disk specimens (n=5) were tested. MICROMET (Bueller, Munich, Germany) was used to determine Knoop micro-hardness. The values were obtained from 5 measurements on the surface of each of the specimens, one at the center and the rest at a distance of 100 micrometers from the center. A load of 25g was applied for 10 seconds.

Glass transition temperature

Three specimens (n=3) were tested. They were evaluated using the thermal analysis method known as Differential Scanning Calorimetry (DSC). The DSC-4 calorimeter was used to determine the glass transition temperature (Tg), in degrees Celsius (°C). For this analysis, samples of approximately 10 mg were embedded in aluminum capsules and subjected to a temperature range of 50 to 190 degrees Celsius, at a heating rate of 10 or 20 degrees per minute, with the Tg being calculated in the second heating cycle.

Water sorption and solubility

Five disk-shaped specimens from each group (n=5) were weighed using an analytical scale and were then conditioned to constant mass in a desiccator placed in an oven at 37°C for 24 hours. The specimens were weighed and reweighed until the weight loss was no greater than 0.0002 g in any 24 hour period. They were immersed in 50 ml of deionized water at 37°C for 7 days. Afterwards, the specimens were removed from the water and dried with paper towels, weighed and reconditioned to constant mass in the desiccator.

Degree of monomer/polymer conversion

The degree of conversion was measured via Raman micro-spectroscopy. One specimen from each

group was used (n = 1). The spectrum peaks of double bonds between carbon atoms unreacted (C=C) at around 1650 cm⁻¹, and C=O bonds at around 1750 cm⁻¹ were analyzed. The percentage of double bonds was determined based on peak areas in the monomer and in the polymer spectra.

Flexural strength and flexural modulus

Ten specimens (n=10) from each group were tested. The deflection of the specimens was evaluated after immersion in distilled water for 50 (± 2) hours at 37°C. This test consists of applying an increasing load from zero at a constant speed of 5 (± 1) mm/min in the center of the specimen, which is supported at both ends. The load is applied until the specimen ruptures.

Statistical analysis

All variables were subjected to analysis of variance followed by Tukey's multiple comparison test, at a level of significance of 5%. The results showed no significant difference between the groups for each variable (p>0.05).

RESULTS

All data are reported in table 1. The monomer/polymer degree of conversion revealed no difference among the three groups of resins (medium pink with and without nylon fibers and colorless resin); it was approximately 88%.

Table 1. Experimental groups.

PROPERTIES	FR	NFR	CR
Flexural strenght (MPa)	98±9	100±10	99±9
Flexural modulus (MPa)	2555±95	2581±87	2586±91
Izod strenght (J/m)	99±9	99±9	98±9
Microhardness Knoop (KHN)	21±1	21±1	21±1
Glass transition temperature (°C)	104±1	104±1	104±1
Sorption (µg/mm ³)	26±1	26±1	26±1
Solubility (µg/mm ³)	0.9±0.1	0.8±0.1	0.8±0.1

DISCUSSION

Porosities and residual monomer content have been shown as important factors influencing the flexural strength. The processing technique used to polymerize the denture base resin has been found to be an important factor, which can induce stress in the denture base during processing and ultimately lead to fatigue failure⁷. Porosity has been attributed to a variety of factors that include the following: trapped air during mixing, monomer contraction during the polymerization, monomer vaporization associated with exothermic reaction, the presence of residual monomer, insufficient mixing of monomer and polymer, processing temperature higher than 74°C, inadequate compression in the flask may also cause porosity in denture base resin⁸.

The advantages of polymerizing denture base resin by microwave energy are a greatly reduced polymerization time, a cleaner method of processing and a denture base with superior adaptation to the dental cast⁹. But porosity in acrylic resin is a complex phenomenon of multifactorial origin. It appears to depend partly on the material/polymerization combination method and the press technique used. Once acrylic resin has been polymerized and pores form in its mass, it is very difficult to determine whether the porosity resulted from the material itself, the polymerization cycle, or handling during the pressing procedure¹⁰.

Adding the monomer to the polymer causes a resin polymerization reaction that occurs without the formation of by products. Nevertheless, conversion of monomer into polymer is generally not complete¹¹. The residual monomer has the potential to create irritation, mucosal inflammation and allergic reaction. Clinical symptoms include erythema, erosion and a burning sensation in the oral area¹². Furthermore, the pigment added to the powder may be another causative factor of hypersensitivity¹³.

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CONCLUSION

Taking into consideration that the color and the presence of fibers in the acrylic resins do not influence the degree of conversion, we could infer that the quantity of residual monomer is similar also. So, the presence of nylon fibers and color do not appear to decrease the acrylic resin properties.

The plasticizing effect of absorbed water is a well-known process and has been implicated in the reduction of mechanical properties of denture base polymers submitted to microwave disinfection¹⁴. It is known that the unreacted monomer may act as a plasticizer, thereby increasing the resilience¹⁵ and decreasing the strength of the polymerized resin¹⁶. It may be concluded that color and the presence of nylon fibers in acrylic resins did not affect the properties analyzed in this study.

Collaborators

CBB FORTES participated in the study design development, laboratory tests and manuscript preparation. FM COLLARES participated in the study design development, manuscript preparation and final review; VCB LEITUNE participated in the study design development, data interpretation and manuscript final review; JG WALCHER participated in the data interpretation and manuscript preparation; SB RODRIGUES participated in the study design development, data interpretation and manuscript preparation; SM SAMUEL participated in the study design development and manuscript final review; CL PETZHOLD participated in the study design development, data interpretation and manuscript final review and V STEFANI participated in the study design development, data interpretation and manuscript final review.

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