

# Physical Properties of a Denture Base Acrylic Resins After Incorporation of Anionic Charges

## Propriedades Físicas de uma Resina Acrílica para Base de Prótese Após a Incorporação de Radicais Aniônicos

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### ABSTRACT

This study evaluated the influence of methacrylic acid (MA) incorporation on hardness, roughness, and flexural strength of a denture base acrylic resin. Thirty-two circular and 40 rectangular specimens were divided into four groups, according to the concentration of MA substituted into the monomer component of a heat-polymerized acrylic resin, as follows: 0% (Control), 10%, 20% and 50% (v/v). The following properties were assessed: Vickers hardness, surface roughness, and flexural strength. Variables were analyzed by ANOVA/Tukey's test ( $\alpha=0.05$ ). For the four incorporated MA concentrations (0%, 10%, 20% and 50%), the following results were obtained for hardness

( $19.0\pm 1.4^A$ ,  $19.6\pm 1.3^A$ ,  $19.6\pm 0.9^A$ ,  $14.2\pm 0.6^B$  VHN), surface roughness ( $0.26\pm 0.05^A$ ,  $0.17\pm 0.01^{AB}$ ,  $0.18\pm 0.03^{AB}$ ,  $0.13\pm 0.03^B$   $\mu\text{m}$ ) and flexural strength ( $96.3\pm 8.3^A$ ,  $98.8\pm 6.2^A$ ,  $102.4\pm 8.6^A$ ,  $106.3\pm 13.7^A$  MPa). The incorporation of MA may cause slight changes in the structure of the tested material, as evidenced by the hardness test. However, the values for surface roughness reduced as MA concentration increased, suggesting that MA addition may improve the acrylic resin texture.

**KEYWORDS:** Acrylic resins, denture bases, methacrylic acid, mechanical stress, surface properties.

### INTRODUCTION

The wearing of complete dentures may have adverse effects on the health of both oral and denture supporting tissues<sup>1</sup> like denture-induced stomatitis, which is a common recurring disease for edentulous patients<sup>2,3</sup>. Despite its multifactorial etiology, great importance can be attributed to bacterial and *Candida sp.* infections, specially when associated to a poor oral hygiene<sup>4,5</sup>.

Abu-Elteen KH and Abu-Elteen RM<sup>6</sup> (1998) evaluated the prevalence of oral candidosis, the frequency of isolation of *Candida albicans* and its main sites of prevalence in the oral cavity of dentate subjects and complete denture wearers. The study comprised 190 healthy subjects and 230 complete denture wearers and it was observed a prevalence of *C. albicans* was 36.8% and 78.3% in healthy dentate and complete denture wearers, respectively. As well, the most frequently and densely colonized oral sites in complete denture wearers were the upper and lower removable dentures.

Acrylic dentures play an important role as reservoirs of micro-organisms, by increasing the risk of *Candida* colonization<sup>7,8</sup>. The adherence of these fungi to the acrylic surfaces of dentures is implicated as the first step in the pathogenesis of associated stomatitis<sup>4,9</sup>. After attached to the acrylic surface, the microorga-

nisms are capable of dividing, forming microcolonies, secreting exopolymeric material, and ultimately forming a 3-dimensional biofilm, which is defined as a complex structured microbial communities, often encapsulated within a matrix of exopolymeric material, and attached to biotic or abiotic surfaces<sup>3</sup>.

The use of surface treatment options capable to inhibit biofilm formation over acrylic resin like glow-discharge plasma<sup>18</sup>, copolymerized quaternary ammonium compounds<sup>19,20</sup> and glazes<sup>21</sup> have been studied. This adherence can also be reduced through the incorporation of polar radicals in the polymer which could be able to increase its hydrophilia. Copolymerization of methacrylic acid (MA) within conventional poly (methyl methacrylate) (PMMA) results in a relatively hydrophilic material, where a significant decrease in the adherence of *C. albicans* was observed<sup>11</sup>.

Although, after a certain ratio, the mechanical properties of the tested acrylic resin are compromised<sup>12</sup>. MA incorporation still remains as an interesting study object, as long as dentures fabricated with modified acrylic resin with this acid result in a treatment with a lower initial cost than the cited options and with a similar maintenance of a conventional prosthesis.

Despite these reports on modification of acrylic resin for removable prostheses with polar radicals<sup>11-13</sup>, the material properties are not well explained. The mechanism of bacterial adherence on this type of material remains without elucidation, although it has an important role in denture biofilm formation<sup>14-16</sup>. The presence of polar radicals can alter physical and mechanical properties due to a larger water sorption<sup>17</sup>.

In this context, the main purpose of the present study was to investigate the effect of MA copolymerization in different concentrations on hardness, roughness and flexural strength of a heat-polymerized denture base proprietary material. The null hypotheses are that MA has no effect on these physical and mechanical properties of the tested acrylic resin.

## MATERIAL AND METHOD

The sample comprised 32 circular acrylic resin specimens for hardness test and 40 rectangular specimens for roughness and flexural strength tests. The specimens were obtained and divided according to the presence of MA (Methacrylic Acid, 99%; Sigma-Aldrich, Steinheim, Germany, Lot number: S40360-197). Concentrations of 0% (Control), 10%, 20% and 50% (v/v) were substituted into the monomer component of a heat-polymerized acrylic resin (Lucitone 550; Dentsply International Inc., York, PA, USA).

Metal master patterns were individually invested in high-viscosity silicone (Zetalabor; Zhermack S.p.A, Badia Polesine, Rovigo, Italy), and supported by type III dental stone (Herodent; Vigodent SA Ind Com, Rio de Janeiro, RJ, Brazil) within flasks. Each flask contained five circular (14.0 x 4.0mm) or two rectangular patterns (65.0 x 10.0 x 3.3mm). After the dental stone had set, the flasks were separated, and the master patterns were removed from the silicone mold. Denture base resin was mixed according to the manufacturer's recommendations. A portion of monomer (10 mL) and polymer (21 g) was mixed for each flask, thus a dough stage was reached and then placed into the molds. A pneumatic press (PM-2000; Techno Máquinas Ltda, Vinhedo, SP, Brazil) was used for packing the denture base resin initially at 250 kgf and, finally, at 1250 kgf maintained for 60 minutes. The resin was polymerized in an automatic polymerization water tank (Ribeirão Preto Dental School, Ribeirão Preto, SP, Brazil). Temperature and time were 73°C for 90 minutes, followed by 30 minutes at 100°C. Next, the specimens were bench cooled overnight before deflasking. The excess resin was trimmed with a bur (Maxi-Cut; Malleifer SA, Ballaigues, Switzerland). Each specimen was then finished using 200, 400, 600 and 1,200-grit wet/dry sandpaper (Norton; Saint-Gobain Abrasivos Ltd, Guarulhos, SP, Brazil) in a polishing machine (DPU-10; Panambra Ind. e Técn. S.A., São Paulo, SP, Brazil) at 250 rpm for 60 seconds. Specimen dimensions were confirmed with a digital caliper (Model CD-6" CSX-B; Mitutoyo Sul Americana Ltda, Suzano, SP, Brazil).

Surface hardness was determined using a hardness tester (Shimadzu HMV-2) equipped with a Vickers diamond. Testing was conducted using a 25 g load and a 30 second contact. Eight indentations were made on each specimen. The individual re-

corded value was the average of the eight values obtained. The test was conducted on the specimens after 48 hours of immersion in distilled water.

The Surface Roughness Tester SJ-201P (Mitutoyo Corp, Kawasaki, Japan) was used to measure the specimens' surface roughness. The profiler was set to move a diamond stylus across the specimen surface under a constant load. The scanning duration for each line was 10 seconds with a constant force of 4mN (0.4 gf) on the diamond stylus (5 µm radius). The surface morphology was measured with a linear variable differential transformer. The surface roughness was derived from computing the numerical values of the surface profile. The Ra value describes the overall roughness of a surface and is defined as the mean value of all absolute distances of the roughness profiles from the mean line within the measuring distance. Five measurements with a length of 4.8 mm and incremental distance of 1 mm between each scanning line were carried out for each specimen. Vertical resolution was .01 µm, which also represents the accuracy of Ra. The mean Ra was calculated from 5 lines as the mean roughness of the specimen.

Following roughness testing, rectangular specimens were immediately submitted to the flexural strength assessment. The flexural strength of each group was measured using a three point bending test in a universal testing machine (EMIC, São José dos Pinhais, PR, Brazil) at a cross-head speed of 1 mm/min. Stress was applied until fracture by a centrally located rod connected to a 50kgf load cell. Flexural strength (S) was calculated using the formula:  $TS = 3WL / 2bd^2$ , where W is the maximum load before fracture, L is the distance between supports (50 mm), b is the specimen width, and d is the specimen thickness.

Data obtained for the tested variables were expressed as mean values and standard deviations, and differences among groups were tested by means of one-way ANOVA. Multiple comparisons were performed according to the Tukey HSD test. Significance was set at  $\alpha=.05$ , and data were analyzed with SPSS for Windows software (version 15.0.0, Chicago, Illinois, USA).

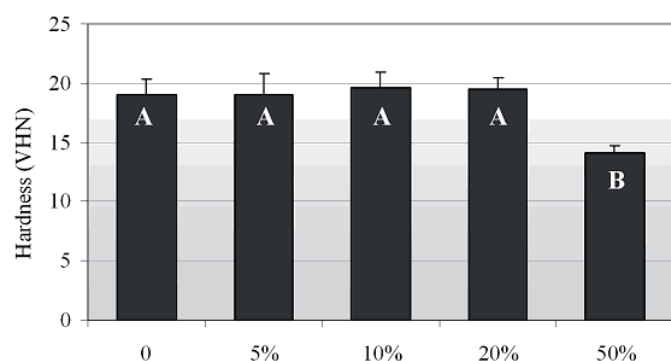
## RESULTS

Results for hardness were influenced by the tested concentrations (ANOVA,  $F=28.614$ ;  $P<0.001$ ). The mean values for 0%, 10% and 20% were similar, whereas 50% specimens presented significantly lower values (Fig. 1). Fig. 2 shows overall results for Ra. Significant difference was found among the means (ANOVA,  $F= 23.21$ ;  $P<0.001$ ), which implies that the incorporation of MA alter surface topography of the finished resin. Higher values were found for 0%, which were reducing as MA concentration increased.

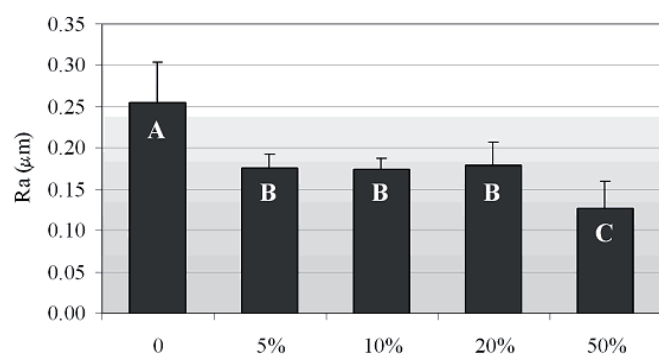
The means and standard deviations for flexural strength are displayed in Table 1. No significant difference was found among the four groups ( $F=2.045$ ;  $P=0,104$ ). This finding implies that the incorporation of MA in the studied concentrations was not able to modify the flexural strength of the finished acrylic resin tested.

**Table 1.** Mean results ( $\pm$  standard deviation) for the flexural strength assessment according to different MA concentrations (means with the same capital letter within the column are not significantly different).

Groups	Flexural strength (MPa)
0% (Control)	96.32 (8.25)
10%	97.65 (6.11)
20%	102.43 (8.61)
50%	106.34 (13.69)



**Figure 1.** Mean values for surface hardness (VHN) according to different MA concentrations. Error bars illustrate standard deviations. Means with the same letter are not significantly different (Tukey HSD test,  $\alpha=0.05$ ).



**Figure 2.** Mean values for surface roughness according to different MA concentrations. Error bars illustrate standard deviations. All means were not significantly different (Tukey HSD test,  $\alpha=0.05$ ).

## DISCUSSION

This study aimed to assess whether the physical properties of denture base acrylic resin would undergo deleterious changes. This way, the performance of a modified material can be better estimated and possible drawbacks can be weighed against a potential reduction in microbial adherence.

An important finding was a decrease in hardness associated with the 50% concentration. When compared to the control group, mean VHN was similar for 10 and 20% ratios and lower for the 50% group. It seems that MA molecules interfere on the entanglement of polymer chains and thereby change the physical characteristics of the resultant polymer. The presence of hydrophilic radicals probably increased water sorption. Water is a complex solvent with possible strong interaction with the polymer, due to its polarity and ability to form hydrogen bonds. Thus, there is a tendency for it to cluster and cause plasticization of the material matrix<sup>17</sup>. The dilution of other components of the

liquid, such as the cross-linking agent ethylene glycol dimethacrylate (EGDMA), cannot be discarded as a possible cause for the reduced hardness.

For all groups, roughness was near to 0.2  $\mu\text{m}$ , which is an indicative of minimal susceptibility to microorganism colonization<sup>23</sup>. Interestingly, the incorporation of MA affects the surface roughness of acrylic resin. Higher values for roughness were found for control group and they were reducing as MA concentration increased.

This reduction can be due to MA influence over EGDMA mentioned above allowing the material to be polished easily. Although the reduction promoted by MA in surface topography may not be clinically important for bacterial colonization itself, it could represent a difficulty for the deleterious effects of other agents over acrylic resin, such as denture hygiene methods or diet. The significance of lower roughness after the incorporation of MA should be further investigated under the influence of those agents, however.

When compared to other methods used to change energy surface in denture base acrylic resin, such as the substitution of monomer with phosphate-containing monomer<sup>13</sup>, silver-zinc zeolite<sup>24</sup> and apatite-coated  $\text{TiO}_2$  photocatalyst<sup>25</sup>, no decline in flexural strength was found as the MA concentration increased, which could result in greater fracture incidence by impact or occlusal forces<sup>24</sup>. The results of the mechanical tests suggest that MA can be added to acrylic resins without distorting these properties and this addition may not affect the degree of conversion of PMMA. Although alterations were observed for the superficial properties, they were probably not strong enough to cause changes like high water sorption and solubility of denture base acrylic resins. If so, serious impact on their mechanical properties in reducing flexural strength and fatigue limit could have been observed.

Some limitations should be stated. Firstly, literature is scarce about the subject, and this was an important limitation for this study. Only a few reports were found describing incorporation of MA in polymers for dental applications<sup>11,12</sup>. Our results point out that the incorporation of MA is possible but other physical, chemical and biological properties must be tested before application in clinical research.

## CONCLUSIONS

The present study showed that topographical changes were detected after MA copolymerization in the heat-polymerized denture base acrylic resin tested. A decreasing in surface roughness was found for all used concentrations; in hardness, it was found only for 50% ratio group. The addition MA may not lower the flexural strength, regardless of the tested concentration.

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## RESUMO

Este estudo avaliou a influência da incorporação do ácido metacrílico (AM) sobre a dureza, rugosidade e resistência flexural de uma resina acrílica para base de próteses removíveis. Trinta e dois espécimes circulares e 40 retangulares foram divididos em quatro grupos, conforme a concentração de AM substituído no componente monomérico de uma resina acrílica termopolimerizável: 0% (Controle), 10%, 20% e 50% (v/v). As seguintes propriedades foram avaliadas: dureza Vickers, rugosidade superficial e resistência flexural. As variáveis foram analisadas por meio de ANOVA seguida pelo teste de Tukey ( $\alpha=0,05$ ). Para as concentrações de AM incorporadas (0%, 10%, 20% e 50%), os seguintes resultados foram obtidos para a dureza

( $19,0\pm 1,4^A$ ,  $19,6\pm 1,3^A$ ,  $19,6\pm 0,9^A$ ,  $14,2\pm 0,6^B$  VHN), rugosidade superficial ( $0,26\pm 0,05^A$ ,  $0,17\pm 0,01^{AB}$ ,  $0,18\pm 0,03^{AB}$ ,  $0,13\pm 0,03^B$   $\mu\text{m}$ ) e resistência flexural ( $96,3\pm 8,3^A$ ,  $98,8\pm 6,2^A$ ,  $102,4\pm 8,6^A$ ,  $106,3\pm 13,7^A$  MPa). A incorporação do AM pode causar mudanças discretas nas propriedades estruturais do material testado, como evidenciado pelo teste de dureza. No entanto, os valores de rugosidade diminuíram em proporção ao acréscimo de AM, sugerindo que essa incorporação pode melhorar a textura da resina acrílica para base protética.

**PALAVRAS-CHAVE:** Resina acrílica, bases de prótese, ácido metacrílico, propriedades mecânicas, propriedades de superfície.



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