FERNANDA FAOT

PROPRIEDADES MECÂNICAS DE RESINAS ACRÍLICAS PARA BASE DE PRÓTESE ANTES E APÓS MÉTODOS DE REPAROS

Tese apresentada à Faculdade de Odontologia de Piracicaba, da Universidade Estadual de Campinas, para obtenção do Título de Doutor em Clínica Odontológica – Área de Prótese Dental

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RESUMO

Fraturas por impacto e flexão ainda são os principais problemas relatados tanto pelos portadores de próteses removíveis como pelos cirurgiões dentistas. Na tentativa de solucionar esse problema, alterações na composição química das resinas acrílicas como a inclusão de co-polímeros, adição de agente de ligação cruzada e a incorporação de partículas de borracha na forma de butadieno estireno têm sido propostas. Entretanto, poucos estudos avaliaram a incorporação destes aditivos modificadores de impacto no processo de fratura, deformação e microestrutura de resinas acrílicas. Além disso, devido à fragilidade dos materiais poliméricos a presença de trincas e fraturas em base de próteses removíveis ainda é alta e reparos são procedimentos comuns tanto por métodos diretos ou indiretos. Entretanto, as propriedades mecânicas de resinas acrílicas reparadas ainda não estão claramente descritas. Assim, os objetivos do presente trabalho foram: I) avaliar a resistência ao impacto e a flexão, bem como tensão de ruptura, módulo Young e deslocamento de escoamento de resinas acrílicas contendo modificadores de impacto e analisar sua microestrutura; II) determinar a resistência ao impacto e flexão de resinas acrílicas para base de prótese previamente fraturadas e reparadas com resinas fotopolimerizável, autopolimerizável e termopolimerizável. Como resultado, observou-se que a resina contendo borracha considerada de alto impacto, demonstrou alta capacidade de dissipação de energia, absorção de tensão e baixo percentual de deformação; entretanto exibiu processo de fratura frágil. Com relação às resinas utilizadas para reparo, concluiu-se que reparos realizados com a mesma resina usada para confecção dos corpos-de-prova, apresentaram melhores resultados quanto à resistência mecânica.

ABSTRACT

Fractures by impact and flexural action are the main problems described by denture users and dentists. Attempting to solve this problem, modifications on the chemical composition of the acrylic resins as co-polymer inclusion, cross linking agents addition and rubber particles incorporation in the form of butadiene styrene have been described as effective and worthwhile means to improve the impact strength and fracture propagation. However, literature lacks information regarding the incorporation of these modifying impact additives in the fracture process, plastic deformation and microstructure of acrylic resins. Furthermore, due to the brittleness of the polymeric materials, the presence of cracks and fractures in the removable prosthesis denture bases still remains high. Thus, frequent repairs are somewhat common procedures in the clinical practice, being by direct or indirect methods. Therefore, the effect of the repairs materials on the mechanical properties of acrylic resins is not clearly described yet. Front of these considerations, the purposes of the present study were: I) evaluate the impact and flexural strength, as well as, stress at yield, Young modulus and displacement at yield of acrylic resins containing impact modifiers and analyze their microstructure; II) determine the impact and flexural strength of denture base acrylic resins previously fractured and repaired with visible-light, auto and heat-polymerized acrylic resins. As results, it could be observed that the acrylic resin containing rubber particles, considered as the high impact, showed high capacity of energy dissipation and stress absorption before the fracture, lower percentage of deformation, however it exhibited brittle fracture process. Regarding to acrylic resins used as repair materials, it was concluded that repairs performed with the same resin used to fabricate the specimens showed the best results for mechanical resistance.

SUMÁRIO

1 INTRODUÇÃO

Apesar do polimetilmetacrilato ser considerado o material de eleição para a confecção de bases de prótese, a friabilidade e suscetibilidade a fratura após longos períodos de uso clínico ainda são problemas inerentes a este material (Stafford et al., 1980; Rodford, 1990; Kanie et al., 2000; Jagger et al., 2002). Como as overdentures implanto-suportadas vêm se tornando a reabilitação padrão em pacientes desdentados inferiores, além do concomitante aumento do uso das overdentures (The McGill Consensus Statement on Overdentures, 2002), a necessidade do uso de resinas mais resistentes à fratura é imperativa (Meng et al, 2005).

As fraturas das próteses ocorrem devido a fatores que levam a concentração de tensão que resultam em flexão aumentada do material durante a função, ou por queda abrupta sobre superfície dura (Franklin et al., 2005; Zappini et al, 2004). A maioria das fraturas ocorre lentamente e ao final de 3 anos de uso (Beyli, 1981; Darbar, 1994). Os fatores que desencadeiam uma fratura são difíceis de serem avaliados devido ao grande número de variáveis que podem estar envolvidas em sua origem e na propagação das trincas que incluem função no meio oral, processamento e manuseio da prótese (Franklin et al., 2005).

Diversos meios para melhorar a resistência à fratura de uma base de prótese têm sido propostos na tentativa de aumentar a capacidade de absorção de tensão pela resina acrílica tornando-a menos friável (Rodford, 1990, Jagger et al., 2001, Franklin et al, 2001). A utilização de polímeros alternativos como o policarbonato (Stafford e Smith, 1967), nylon (Stafford et al., 1986; Yunus et al., 2005), copolímeros (Rodford, 1990) bem como a utilização de agentes de reforço, como por exemplo flocos de vidro (Franklin et al., 2005), fibras (Jagger et al., 2001) e barras metálicas (Vallitu, 1996) têm sido estudados.

Entretanto, estes materiais são freqüentemente opções de alto custo quando comparadas às resinas acrílicas processadas de forma convencional.

A modificação da composição química da resina acrílica pela incorporação de borracha na forma de copolímeros de butadieno estireno tem sido uma proposta viável para melhorar a resistência ao impacto da mesma (Rodford, 1990; Rodford e Braden, 1992; Jagger 1999; Jagger 2001). Entretanto, uma das conseqüências deste tipo de reforço é a diminuição do módulo de elasticidade do material, afetando desta forma a rigidez da base de prótese (Jagger et al., 2002). Além disso, tem sido descrito na literatura que a adição de agentes de ligação cruzada de diferentes composições químicas e diluições (percentuais) a estes tipos de polímeros, também são meios alternativos de melhorar no comportamento de propagação de trincas e fraturas (Harrison et al.,1978; Price,1986; Caycik e Jagger, 1992; Kaine 2000; Memmon, 2001).

O aumento nas propriedades de resistência ao impacto e resistência à flexão, das resinas acrílicas pela adição de modificadores de impacto tem minimizado danos decorrentes de quedas acidentais ou de esforço contínuo oriundo do ato mastigatório. Entretanto, os mecanismos de propagação de fratura por concentração de tensão repentina, progressiva ou cumulativa que levam a deformação inicial per si destas resinas acrílicas ainda são pouco estudados de forma quantitativa e qualitativa (Kusy, 1975; Mecholsky, 1995; Kanie et al., 2000; Zappini et al., 2003, Faot et al., 2006). Assim sendo, a relação entre o comportamento de fratura, índice de deformação e alteração de microestrutura destes materiais nem sempre é relatada.

Em acréscimo, a fratura das próteses removíveis continua representando um problema comum e a execução de reparos diretos com resinas auto e fotopolimerizáveis vêm sendo amplamente utilizados por otimizar o tempo clínico, mesmo resultando em próteses mais friáveis (Stipho e Talic, 2001). Contrariamente, reparos realizados de forma

indireta utilizando resinas termopolimerizáveis convencionais ou de microondas (Rached e Del Bel Cury, 2001; Polyzois et al., 2001; Rached et al., 2004) tem apresentado maior resistência frente a testes mecânicos.

Entretanto devido ao alto índice de fraturas recorrentes mesmo após o processo de reparo, métodos e materiais de reparo direto sobre a superfície fraturada de próteses ainda precisam ser melhor explorados (Lin et al., 2000).

Apesar de suas limitações, as resinas autopolimerizáveis específicas para a execução de reparos ainda têm sido utilizadas na prática clínica por sua rapidez de manuseio e processamento (Polyzois et al., 1995; Dar-Odeh et al., 1997, Minami et al., 2005). Por conferirem resistência mecânica de aproximadamente 60 (Berge, 1983) a 65% (Leong, 1971) do material original, protocolos de tratamento das superfícies fraturadas com monômero de metilmetacrilato (Vallitu et al., 1994; Rached and Del Bel Cury, 2001; Sarac et al., 2005) tem sido descritos como procedimentos capazes de aumentar a adesão do material reparador às superfícies fraturadas melhorando à resistência a ruptura dos reparos. Além disso, alterações de contorno da superfície a ser reparada (Ward et al., 1992; Lin et al., 2000) também têm sido considerados meios alternativos efetivos para o aumento da resistência a fratura de resinas acrílicas para base de próteses reparadas.

Resinas acrílicas fotopolimerizávies constituídas de matriz orgânica de ligação cruzada e livre de peróxido de benzoíla e metilmetacrilato têm sido lançadas no mercado odontológico. Devido à compatibilidade destas resinas com o metilmetacrilato, estas também têm sido indicadas para a execução de reparos diretos em prótese. Mas pouco ou nenhuma informação sobre as propriedades mecânicas destes materiais quando utilizados para reparo são relatadas.

Frente ao exposto, serão propósitos desta pesquisa: 1. Avaliar a resistência ao impacto e a flexão de resinas acrílicas com modificadores de impacto e compará-las com

resinas convencionais; 2. Determinar os modelos de deformação durante o processo de fratura sobre esforços de tensão e compressão obtidos através do teste de resistência a flexão; 3. Caracterizar a morfologia da superfície fraturada e o padrão microestrutural de fraturas por impacto e flexão.

Em acréscimo, considerando que também a literatura é escassa sobre determinação do processo de fratura em resinas acrílicas reparadas mantendo-se a deformação induzida ou pré-existente por ensaios de fratura, também foi objetivo deste trabalho avaliar o efeito de três métodos de reparo sobre as propriedades mecânicas de resinas acrílicas comumente utilizadas na prática clínica.

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2 CAPÍTULO 1

Impact and flexural strength, and fracture morphology of acrylic resins with impact modifiers

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Short Title: Mechanical strength and fracture morphology of acrylic resins

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Abstract

Objectives: This study evaluated the impact and flexural strength and analyzed the fracture behavior acrylic resins. **Methods:** Eighteen rectangular specimens were fabricated of Lucitone 550, QC 20 (both unreinforced acrylic resins), Impact 1500 (extra strength impact), Impact 2000 (high impact) according to the manufacturers' instructions. The impact strength was evaluated in notched specimens (50x6x4mm) and flexural strength in unotched (64x10x3.3mm), using three-point bending test, as well as, stress at yield, Young modulus and displacement at yield data. Fragments from mechanical tests were observed by SEM. Data from impact strength, stress at yield and displacement at yield data were analyzed by 1-way ANOVA and Tukey test (α=0.05).Young modulus values were analyzed by 1-way ANOVA and Dunnett T3 multiple comparisons test (α=0.05). **Results:** Mean values of impact strength and stress at yield values were higher (P<.005) for Impact 2000 while Young modulus was higher (P<.05) for Lucitone 550; Impact 1500 and Impact 2000 showed significant values (P<.05) in the displacement at yield. Impact fractures of the all acrylic resins were brittle. Bending fractures of Lucitone 550 and Impact 2000 were brittle, QC 20 fractures were ductile and Impact 1500 showed brittle (75%) and ductile (25%) fractures. **Significance:** It is not fully understood the effect of the impact modifiers addition on the mechanical properties, fracture process and microstructure of the acrylic resins. The capacity of stress absorption and the deformation degree are determinant factors on the material selection, considering their influences on clinical performance of the acrylic resins.

Keywords: Acrylic resins, high impact, impact strength, stress at yield, fracture morphology, fracture microstructure, deformation behavior, fracture process, brittle fracture, ductile fracture, cross-linking agents.

Introduction

Denture fracture is a problem commonly encountered by denture wearers and dentists, and is related to material properties, technical features, and stresses that dentures are subject to in service or when they are dropped. In addition, considering the recommendations of the McGill Consensus Statement on overdentures [1], suggesting implant supported overdentures be the standard of care for mandibular edentulous patients and the concomitant increase in the use of overdentures, the use of acrylic resins with better quality is imperative.[2]

As a result, new and stronger acrylic resins have been developed. The modifiers introduced in acrylic denture composition include co-polymers, cross-linking agents [3-5] and rubber substances in the form of butadiene styrene.[6-8] Therefore, although high-impact denture base resins have been on the market for over 30 years ago, manufactures claim that these polymers are stronger and tougher because this type of acrylic resin is able to absorbing greater amounts of energy at a higher strain rate before fracture.[9]

There is some evidence, however, that the incorporation of rubber has not been entirely successful because it can have detrimental effects on the elasticity modulus and hence the rigidity of the denture base.[8] Furthermore, the clinical and laboratorial use of this type of acrylic resin has been limited by its high cost compared with conventional heat-cured resin. In addition, it has been shown that the high concentrations of cross-linking agents had little effect on the mechanical properties of dough-molded acrylic resins, with exception of flexural modulus.[5]

Denture fractures result in high costs to users and the social security system, considering the time and money spent on fixing dentures. Therefore, it is important to know about the mechanical aspects of high impact acrylic resins, since there is little information about the effect of adding cross-linking and rubber incorporation in acrylic

resin. The effects of these additives on toughening, microstructure and deformation behavior under the impact and flexural tests is also unknown.

The aim of this study was to determine the impact and the flexural strength of two acrylic resins with impact modifiers and compare them with conventional unreinforced denture base acrylic resins. Moreover, the stress at yield, Young modulus and displacement at yield were evaluated and the fracture processes were analyzed by stress-displacement graph. The acrylic resin microstructures in the region of fracture were also examined.

Materials and methods

The acrylic resins used in this study are listed in Table 1.

Eighteen rectangular specimens of each acrylic resin, measuring 50×6×4 mm and 64×10×3.3mm were prepared for impact and flexural strength tests, respectively. Metal master patterns were individually invested with high-viscosity silicone (Zetalabor; Zermack S.p.A, Badia Polesine, Rovigo, Italy) and used to fabricate the specimens. Patterns were invested in Type III dental stone (Herodent Soli Rock; Vigodent, Rio de Janeiro, Brazil) in metal dental flasks (Uraby; DLC, São Paulo, Brazil). Acrylic resins were mixed in accordance with the manufacturers' instructions and packed into the silicone molds at dough stage. [10]

To polymerize Lucitone 550 and Impact 2000 acrylic resins, the flasks were placed in a polymerizing unit (Termotron P-100; Termotron Equipamentos Ltd, Piracicaba, Brazil) filled with water at 74ºC water for 9 hours. Flasks containing QC-20 and Impact 1500 acrylic resins were immersed in boiling water for 20 minutes. Next, all flasks were allowed to bench cooling for 2 hours. Specimens were deflasked, and each specimen was trimmed and finished, using abrasive papers (320, 400 and 600-grit, Carbimet; Buehler, Lake Bluff, Ill) in a polishing machine (Model APL-4; Arotec, Sao Paulo, Brazil). After that, the

specimens were ultrasound cleansed (Thornton T 740, Thornton-Inpec Eletrônica LTDA, Vinhedo, Brazil) for 20 min and then immersed in distilled water at 37°C for 48 ± 02 hours before testing.

Impact strength test

Impact strength test was performed according to ISO standard 1567:1999/Amd.1:2003(E).[11] A type A notch was cut in the middle of each specimen using a milling machine (Model FNGJ32, INTOS Ltd., Czech Republic) and a universal milling tool (Model 1322, 45° double angle; Sandvik Coromant, Sweden). The depth was 1.2±0.1 mm leaving a residual depth beneath the notch of 4.8±0.1mm and the notch base radius of 0.25±0.05mm.

The impact strength was evaluated using plastic impact test machine (AIC - EMIC, São José dos Pinhais, Paraná, Brazil) using the Charpy method with a pendulum of 0.5J, in which the specimens were horizontally positioned, with a distance of 40 mm between the 2 fixed supports.

Flexural strength test

Flexural strength test was performed by the 3-point bending test using a universal testing machine (Instron Model 4467, Instron Industrial Products, PA, USA) calibrated with a 500kgf load cell and a crosshead speed of 5mm/min. The flexural testing device consisted of a central loading plunger and 2 polished cylindrical supports, 3.2 mm in diameter and 10.5 mm long. The distance between the centers of the supports was 50mm. The compressive force was applied perpendicular to the center of the specimens until a deviation of the load-deflection curve and the fracture of specimen occurred. The stress at yield, Young modulus and displacement at yield of the specimens were recorded and the stress-displacement graph was evaluated.

Fracture analysis

The fractures of the specimens broken by both the impact and the three point bending tests were classified as brittle or ductile. Scanning electron microscopy (SEM; LEO 435 VP, Carl Zeiss SMT, Oberkochen, Germany) was used to characterize the fracture surface microstructure around the crack tips of the specimens. A 5mm slice was sectioned from the border of the fractured under water-cooling using diamond-coated disc at 200 rpm in a precision saw (ISOMET 1000; Buhler, Lake Bluff, Ill). SEM-photomicrographs of impact specimens were taken at 100× magnification and those from three-point bending specimens were taken at 1000× magnification.

Statistical analysis

The statistical analyses were done using SAS software (SAS Institute Inc., version 9.0, Cary, NC) with the significance level fixed at 5%. As impact strength data violated the assumptions of equality of variances and normal distribution of errors, they were transformed into $\log_{10}$ (X) before they were analyzed by 1- way ANOVA. Tukey HSD test was also applied to compare impact strength, stress at yield and displacement at yield, and Dunnet T3 test to compare the Young modulus.

Results

Mean values of the impact strength (kJ/m^2), stress at yield (MPa), Young modulus (MPa) and displacement at yield (mm) are presented in Table 2. It was verified that the impact strength value was significantly higher ($P<0.05$) only for Impact 2000 acrylic resin. Significant differences ($P<0.05$) were found in the stress at yield for all acrylic resins and Impact 2000 specimens showed the highest value.

With regarding to the Young modulus, significant differences ($P<0.05$) were also observed for the studied acrylic resins, with higher values for Lucitone 550. Displacement

at yield values were significantly higher (P<0.05) for Impact 2000 and Impact 1500 acrylic resin.

The impact test specimens exhibited brittle fractures for all acrylic resins. For the flexural strength test, however, Impact 2000 and Lucitone 550 resins exhibited brittle fractures. QC 20 resin showed ductile fractures and Impact 1500 resin presented 16 ductile and 4 brittle fractures.

Fracture processes and deformation behavior of acrylic resins showed a significant right shift of the plots (Fig 1). Lucitone 550 and Impact 2000 acrylic resins showed a typical brittle failure behavior, with a low percentage of deformation (0.002% and 0.003% respectively), exhibiting only elastic deformation. QC20 and Impact 1500 presented ductile failures with lower stress concentration at yield values and higher percentage of deformation (0.032% and 0.0105% respectively), exhibiting elastic and plastic deformations during the fracture process.

SEM observations of impact fractures showed that the microstructure of the deformed regions was similar in Impact 2000 and Lucitone 550 (Fig. 2, A-C), presenting a rough surface with grain microstructure, with high density and fine striations close to the notch that dissipated into the polymeric matrix. In the QC20 and Impact 1500, a smooth surface could be observed and the striations were more concentrated near to the notch (Fig. 2, B-D). On the other hand, the three-point bending fractures showed distinct microstructures for the acrylic resins studied. Although, Lucitone 550 and Impact 2000 exhibited compact and organized surface fractures (Fig.3, A-C), it was observed that Lucitone 550 exhibited fiber morphology orientated and uninterrupted with low density and short striations while Impact 2000 disorientated fiber morphology was observed. QC20 and Impact 1500 presented disorganized and stepped fracture surfaces (Fig. 3, B-D). The

microstructure revealed the presence of crazing shown by interrupted fiber morphology with longer disorientated high density striations.

Discussion

Acrylic resin denture fracture continues to be a problem and several attempts have been made to improve the mechanical properties of denture base material. One approach is to have PMMA material strengthened and toughened by chemically modifying conventional heat acrylic resin by adding rubber graft co-polymer or cross-linking agents.

In this study, the high strength denture base acrylic resin, Impact 2000 exhibited the best results for impact and flexural strength, the impact strength being 2.4 times greater than the other acrylic resins studied, as was expected. As mentioned by Jagger et al. (2002) [8], however, it is not possible to discuss the reinforcement mechanisms of this acrylic resin, since its exact constituents are not known. Nevertheless, it is known that addition of rubber in “high impact” denture base resins, in the form of acrylate terminated butadiene styrene block copolymer, produces improved impact strength since this agent (macromers) is able of causing dispersion of the cracks.[12]

The other three acrylic resins studied showed the same impact strength values, around 1.0J, irrespective of the polymerization time used.

Moreover, it was observed that Impact 2000 showed significantly and higher values for stress at yield ($P<0.05$) followed by the conventional acrylic resin Lucitone 550 and both were polymerized for a long time by water bath. In contrast, QC20 and Impact 1500, which were polymerized for a short period in boiling water showed the lowest values ($P<0.05$).

The Young modulus of each acrylic resin was significantly different from the other, with Lucitone 550 exhibiting the highest values (2.5±204.5MPa). However, acrylic resins polymerized for a short time in boiling water showed the lowest values ($P<0.05$), almost

half that of the first mentioned. These findings are in agreement with those found by Stafford et al. (1980) [13] who showed that unreinforced acrylic resin had higher fatigue life values in comparison with some reinforced polymers.

Therefore, these results and those of the stress at yield could be attributed to the polymerization cycle. It has been shown that acrylic resins polymerized for longer periods of time provide polymers with high packing density, better interchain force and polymeric chain arrangements, resulting in acrylic resins with improved mechanical and viscoelastic properties. [5, 14, 15]

On the other hand, the higher values of Impact 2000 showed that the impact strength was improved at the expense of the Young modulus, producing a denture base with a different brittle behavior, indicating decreased ability of the polymer to flow.[13] Probably, alterations in the relaxation behavior generated by the rubber chains sections [9] could be responsible for effects on the intermolecular forces (molecular structure) affecting the chain stiffness [14] in the “high impact acrylic resin”.

Considering the displacement at yield values, no significant differences ($P>0.05$) were found between Impact 2000 and Impact 1500. Both acrylic resins were able to dissipate the crack development slowly through the poly(methyl methacrylate), possibly by different mechanisms. It is known that the rubber reinforced acrylic resin decelerates crack propagation throughout an interpenetrating network of rubber and poly(methylmethacrylate) [12] and this could happen with Impact 2000. Although Impact 1500 does not present rubber reinforcement, its crosslink agent alkyldimethacrylate could be influenced by the polymerization temperature, which could limit the geometry of the polymer network or the unreacted cross-linking agent in the form of a residual monomer, or pendant chains could act as a plasticizer. [16]

Stress-displacement curve analyses (Fig. 1) of the acrylic resins showed different fracture toughness, based on the relaxation behavior, which varied in accordance with polymerization cycles. Lucitone 550 and Impact 2000 resin showed similar curves under stress intensity, in accordance with the theory of linear elastic fracture mechanics [17] for brittle fracture. However, Lucitone 550 was brittler than Impact 2000 and started to crack before it. This probably happened because of the presence of rubber particles around the matrix polymer in Impact 2000. Differently, QC20 and Impact 1500 resins exhibited curves with ductile fracture characteristics; crack initiation and plastic deformation also differed between these resins.

Regarding to fracture process results, it is important considers that the analyses of the toughness behavior of acrylic resin with alkyldimethacrylate as impact modifier can have favorable clinical implications. The ductile fracture process and viscoelastic/relaxation ability of this material seem to be interesting to implant supported overdentures when these prosthesis are submitted to flexural loads by the masticatory forces, since the mandibular edentulous patients are always suffering a continuing no controlled resorption process that results on prosthesis desadaption and consequently recurrent fractures.

The effects of the impact fracture process on the acrylic resin microstructures observed in the SEM-photomicrographs showed a true network polymer structure with the presence of homogeneous particles for Lucitone 550 resins and Impact 2000 (Fig 2. A-C). QC20 and Impact 1500, showed a smooth and flat microstructure with some disoriented striations (Fig 2. B-D).

SEM observation of the three point bending fractures showed that Impact 2000 (Fig. 3.C) was rougher than Lucitone 550 (Fig. 3.A), which implies that massive deformation of the PMMA matrix occurred. Furthermore, it seems that the rubber particles

in the Impact 2000 resin did not adhere well to the PMMA matrix. These findings are in agreement with those observed by Cho et al. (1998) [7] in a study about toughening behavior of rubber modified PMMA, in which it was observed that the rubber particles were detached from the PMMA matrix and only part of the rubber particles were connected to the matrix. This is a reason for the brittle behavior and the unexpected low deformation (0.003%) by the multiple crazing in Impact 2000.

QC20 and Impact 1500, which exhibited a slower fracture process, had greater massive deformation (Fig. 3.B-D) and worst microstructure characteristics, evidencing that these materials did not transfer the stress far away from the crack, possibly because of their low Young modulus and low stress at yield .The micro structural differences between them, related to grain size, density, and striation lengths and thicknesses, could be explained by their different crosslinked matrixes [18, 19], since Impact 1500 contains alkyldimethacrylate, and QC20 contains ethylene glycol dimethacrylate.

The fact of the mechanical tests have not been performed in a wet conditions similar to the oral cavity could be considered as a limitations of this *vitro* study. For better understanding the fracture and deformation mechanisms future researches about the effects of the residual monomer content and the viscoelastic properties on the fracture process and microstructure of acrylic resins could be performed. Furthermore, as the alkyldimethacrylate could be responsible for a ductile fracture behavior and greater results regarding to deflection, a study of its incorporation in a rubber reinforcement polymer could be clarify if the cross-linking agents will be able to alter the fracture process improving the mechanical properties of the rubber polymers.

Conclusion

Within the limits of this investigation, it seems that there are advantages to using acrylic resin with rubber incorporation in preference to unreinforced conventional acrylic resins, since its formulation has properties comparable with those of the best proprietary materials, and it meets the requirements of impact strength with minimal decrease in Young modulus.

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Figure1.Graph of Stress-Displacement curves

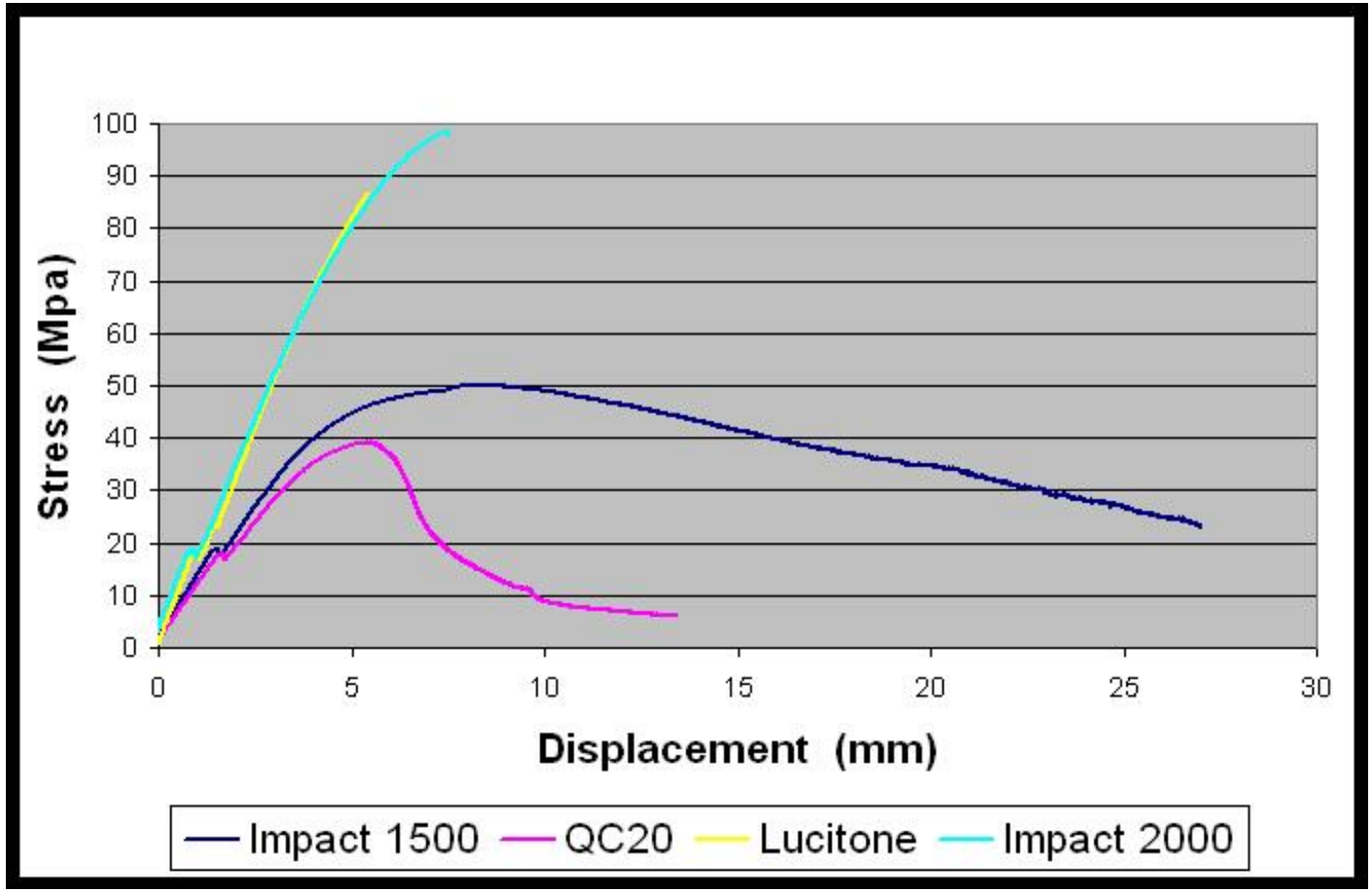


Figure 2. SEM of impact strength specimens. A. Lucitone 550 resin, B. QC20 resin, C. Impact 2000 resin and D. Impact 15000 resin. a. notch surface, b. fracture surface, c. notch-fracture junction, d. granular structure. Black arrows indicate the striations.

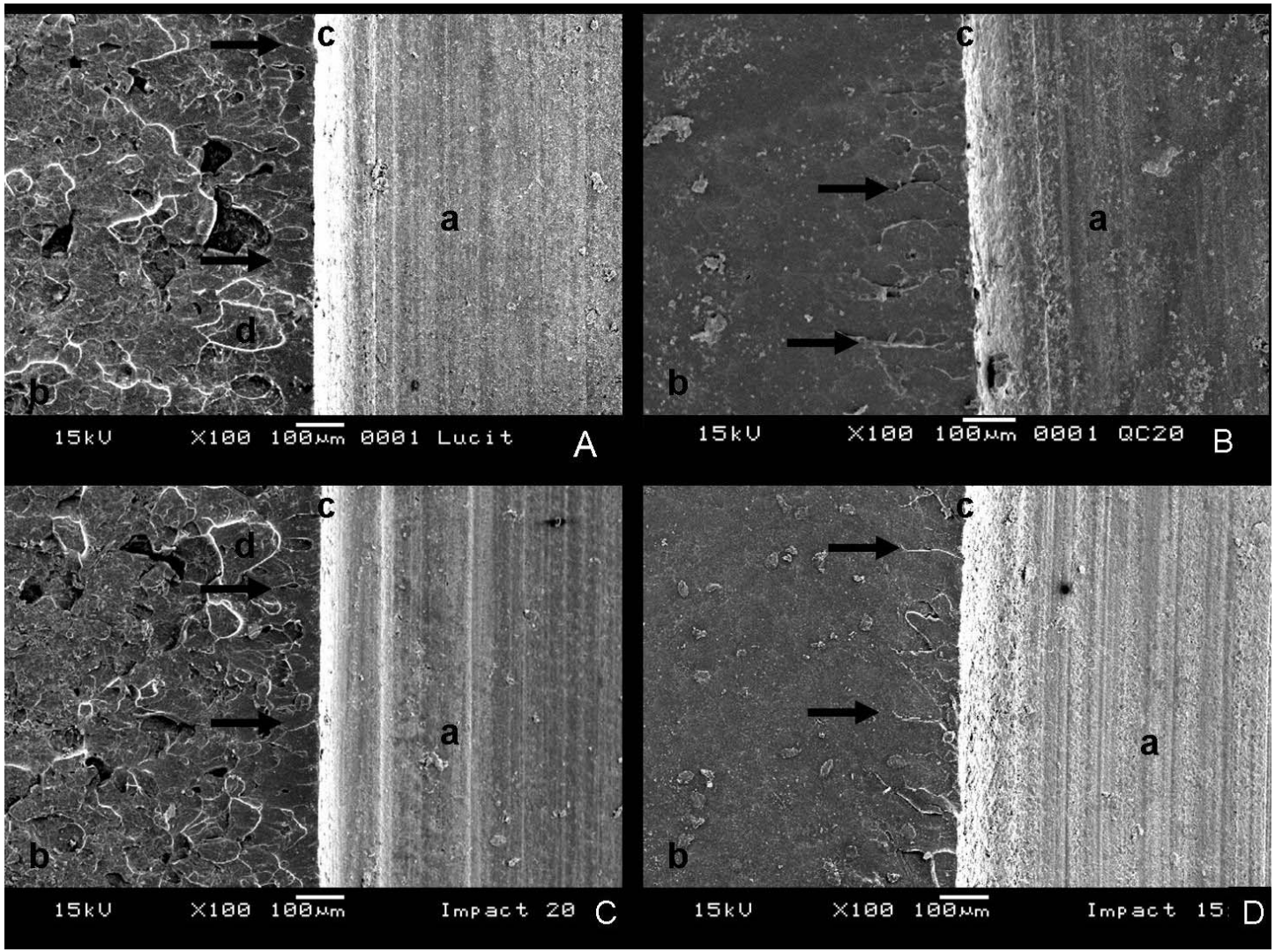
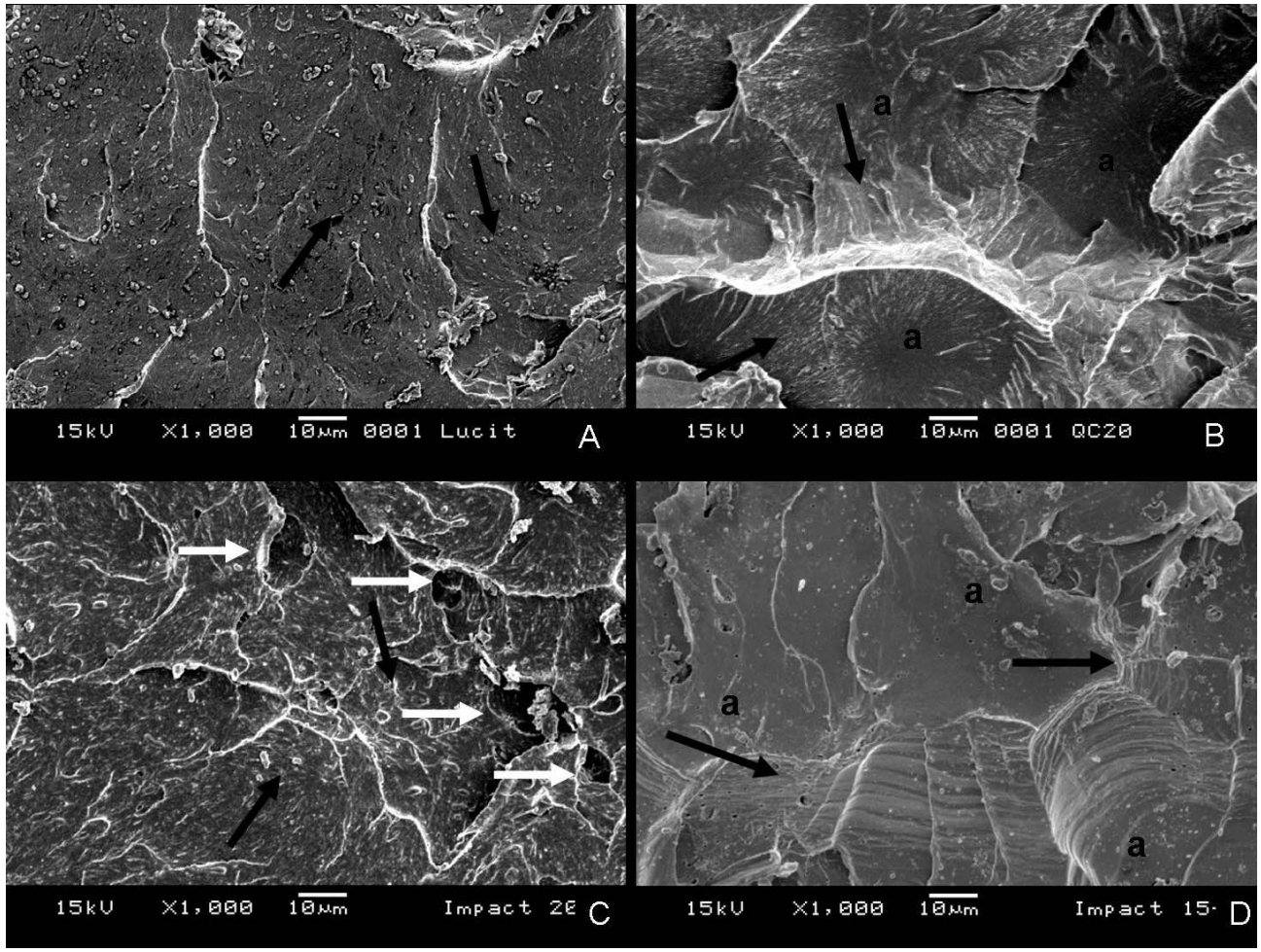


Figure 3. SEM of flexural strength specimens. A. Lucitone 550 resin, B. QC20 resin, C. Impact 2000 resin and D. Impact 15000 resin. a. Stepped surface and granules microstructure. Black arrows indicate the striations. White arrows show where the rubber particles were attached, and the delimitation indicates that they detached from the PMMA matrix



3 CAPÍTULO 2

Effects of repair methods on the mechanical properties of heat-polymerized acrylic resins

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ABSTRACT

Statement of problem. Clinicians are still confused about the choice of repair method, which depends on factors such as the length of time required for processing and mechanical strength of the repaired material. **Purpose of study.** The aim was to determine the impact and flexural strength characteristics, such as stress at yield, Young modulus and displacement at yield, of denture base resins before they were fractured and after they were repaired by three methods using heat, auto and visible light-polymerized acrylic resins. **Material and Methods.** For impact and flexural strength tests 18 rectangular specimens measuring 50×6×4mm and 64×10×3.3mm respectively, were processed using Impact 2000, Lucitone 550, Impact 1500 and QC20 acrylic resins. Fracture tests were performed according to ISO1567:1999. Afterwards, all fractured specimens were stored in distilled water at 37ºC for 7 days, and then repaired with: (G1) the same acrylic resin used for specimen fabrication (n=6); (G2) an autopolymerized acrylic resin (TruRepair, n=6) and (G3) a visible-light acrylic resin (Versyo.com, n=6). The repaired specimens were again submitted to the same fracture tests and the failures were classified as adhesive or cohesive. Data from all mechanical tests after repair by the different methods were submitted to 2-way ANOVA and means values were compared by the Tukey test. **Results.** All acrylic resins showed adhesive fractures after impact and flexural strength tests. Differences (P<.05) were found among repair methods for all acrylic resins studied, with exception of displacement at yield, which showed similar values for repairs with auto and visible-light resins. The highest values for impact strength, stress and displacement at yield were obtained when the repair was made with the same resin the specimen was made of. **Conclusion.** Denture base acrylic resins repaired with the same resin that they were made of showed greater fracture strength.

Clinical Implications. Within the limits of this in vitro study, the efficacy of the repair methods was related to the interaction between denture base acrylic resins and repair materials. Despite

some advantages, repairs made with auto and visible light polymerized acrylic resins exhibited lower mechanical properties.

KEY-WORDS

Acrylic resins, impact strength, flexural strength, repair strength, repair methods, autopolymerized acrylic resin, visible-light acrylic resin.

INTRODUCTION

Damage to acrylic resin denture bases can have many causes, including failures outside and inside the mouth, such as fractures due to dropping or excessive bite force. The most common problem patients and dentists encounter is the midline fracture, which can occur during function[1] as a result of fatigue failure.

Therefore, repairs are constantly required when there are signs of cracks and initial fracture occurs in dentures. Repairs can be made using light-polymerized, auto-polymerized or heat-polymerized acrylic resins[2-3].

However, the choice of repair material still confuses clinicians, and depends on several factors such as the length of time required for making the repair, transverse strength obtained with the repair material, and the degree to which dimensional accuracy is maintained during the repair[4]. The repair strength of heat-polymerized materials ranges from 75% to 80% of the original material[5], but the processing method is time consuming, due to laboratory packing and flasking procedures, and presents the risk of denture distortion by heat[6].

Whereas, a repair made with autopolymerized material is a fast procedure, but its strength is approximately 60%[7] to 65%[5] of that of the original material. Despite its limitations, autopolymerizing acrylic resin remains the most popular material used for denture repairs [8-11].

Visible light polymerized acrylic resins are considered an alternative material used to repair denture base acrylic resin. It is free of residual monomer and its short polymerization time eliminates the flasking procedures[12]. Furthermore, over the years, visible light acrylic resin has been improved and gained considerable attention[13-17]. The newer visible-light acrylic resin available for clinical use is a system based on a crosslinking organic matrix, applied as a single component resin. This material has a low filler/monomer ratio, and its polymerization process uses a mixture of multifunctional monomers with high molecular weight. Moreover, this acrylic resin presents greater elasticity modulus and flexural strength in comparison with conventional cold and heat polymerized resins, due to the formation of a highly cross-linked network within

the polymerized matrix. In addition, it has a high compatibility with polymethylmethacrylate and can be used for prosthesis repairs, rebasing and extensions[18].

Although this new visible light acrylic resin presents improved properties, no reports were found related to its use as a repair material. Moreover, there is no consensus about the mechanical properties of visible light acrylic resin, since Andreopoulos et al.[19] and Dixon et al.[20] reported satisfactory transverse strength, while al-Mulla et al.[21] and Ishigami et al.[22] considered its brittle nature and low impact resistance as disadvantages.

The purpose of this study was to determine the impact strength and flexural characteristics of denture base resins before they were fractured and after they were repaired with auto- and visible light-polymerized acrylic resins In addition, the fractures were classified.

MATERIAL AND METHODS

The heat-polymerized acrylic resins used to prepare all specimens as well as the acrylic resins used to repair the fractured specimens are listed in Table I.

Specimen preparation

Eighteen rectangular specimens measuring 50×6×4mm and 64×10×3.3mm were processed using Lucitone 550, Impact 2000, Impact 1500 and QC20 acrylic resins to be respectively fractured by impact and flexural strength tests. Metal master patterns were individually invested with high-viscosity silicone (Zetalabor; Zermack S.p.A, Badia Polesine, Rovigo, Italy) and used to fabricate the specimens. Patterns were invested with Type III dental stone (Herodent Soli Rock; Rio de Janeiro, Brazil) in metal dental flasks (Uraby; DLC, São Paulo, Brazil)[24]. The acrylic resins were mixed in accordance with the manufacturers' instructions and packed into the silicone mold at dough stage.

To polymerize Lucitone 550 and Impact 2000 acrylic resins, the flasks were placed in a polymerizing unit (Termotron P-100; Termotron Equipamentos Ltd, Piracicaba, Brazil) filled with water at 74ºC for 9 hours. Flasks containing QC-20 and Impact 1500 were immersed in boiling water for 20 minutes. Afterwards, all flasks were allowed to bench cool for 2 hours, then opened

and the specimens were finished using progressively smoother aluminum oxide papers (grit 320, 400 and 600) in a horizontal polisher (Arotec APL-4; São Paulo, Brazil). After finishing procedures the specimens were ultrasound cleansed (Thornton T 740, Thornton-Inpec Eletrônica LTDA, Vinhedo, Brazil) during 20 min and then immersed in distilled water at 37°C for 48 ± 02 hours. In order to simulate fractured prostheses and their defects as a result of plastic deformation, the specimens were submitted to fracture tests, and were subsequently repaired.

Impact strength test

The impact strength test was performed according to ISO standard 1567:1999/Amd.1:2003(E)[25], using impact test machine (AIC - EMIC, São José dos Pinhais, Brazil) by the Charpy method with a pendulum of 0.5J, in which the specimens were horizontally positioned, with a distance of 40 mm between the 2 fixed supports.

Flexural strength test

Flexural characteristics, such as the stress at yield, Young modulus and displacement at yield of intact and repaired specimens were determined by the 3-point bending test using a universal testing machine (Instron Model 4467, Instron Industrial Products, PA, USA) calibrated with a 500kgf load cell and a crosshead speed of 5mm/min.

The flexural testing device consisted of a central loading plunger and 2 polished cylindrical supports, 3.2 mm in diameter and 10.5 mm long. The distance between the centers of the supports was 50mm. The compressive force was applied perpendicular to the center of the intact specimens and at the midline of the repaired material, until a deviation of the load-deflection curve and the fracture of specimen occurred.

Repair procedures

After impact and flexural strength tests, the fractured specimens were randomly divided into 3 groups to be repaired using: G1) the same acrylic resin as was used to fabricate the specimen (control); G2) an autopolymerizing MMA-based acrylic resin (TruRepair, Bosworth

Company, Illinois, USA), and G3) a visible-light polymerized acrylic resin (Versyo.com, Heraeus-Kulzer, Germany).

The butt joint surface design was chosen[26] for all repair methods and the cross section of each half of fractured specimen was polished with pumice powder, and ultrasonically cleaned. The paired halves were then put back into the same preparation silicon mold, keeping a 3mm gap between the edges of each half of the specimen.

The joint surfaces of specimens in G1 and G2 were first treated with the monomer liquid of each acrylic resin for 3 minutes and the gap was filled with acrylic resin. The heat polymerized acrylic resins were then processed, as previously described, while the autopolymerized acrylic resin (Tru Repair) was considered polymerized when it had lost its glaze (10 minutes).

The joint surface of G3 was first coated with bonding agent (Versyo.bond, Heraeus-Kulzer, Germany) for 60 seconds and polymerized for 2 cycles of 90 seconds in the Heralight pre-curing unit (Heraeus Kulzer, Germany). Next, visible-light polymerized acrylic resin was carefully packed into the gap, through two increments pre-polymerized for 60-second cycles. The final polymerization was processed in the UniXS curing box (Heraeus Kulzer, Germany) for 3 minutes.

After polymerization, the surfaces of each repaired sample were finished and polished, using a polishing machine (Arotec APL-4; Sao Paulo, Brazil) and 600-grit sandpaper (CARBIMET, Buehler, Lake Bluff, USA). All repaired specimens were stored in distilled water at 37ºC for 7 days and evaluated for impact and flexural strength as previously described.

Statistical analysis

The statistical analysis was done using SAS software (SAS Institute Inc., version 8.01, Cary, NC) with a significance level fixed at $P<0.05$. ANOVA was used to test the null hypothesis of no difference among the repair methods or acrylic resins. The assumptions of equality of variances and normal distribution of errors were checked for each variable, and when

violated, the data were transformed (Box *et al.*, 1978)[27]. As mean values were not normally distributed, the impact strength and stress at yield data were transformed by exponentiation; Young modulus data by log10 (X) and displacement at yield data by square root. Tukey's test was then used for post-ANOVA comparisons.

RESULTS

All acrylic resin fractures from impact and flexural tests were classified as adhesive.

The 2-way ANOVA results for impact strength, stress at yield, Young modulus and displacement at yield after repair methods are presented in Table II. The mean values and standard deviations for impact strength, stress at yield, Young modulus and displacement at yield after repair are described in Table III.

With respect to impact strength, when the repair methods were compared for each acrylic resin, Lucitone 550 and Impact 2000 showed statistical differences among all repairs procedures *(P<.05*) while Impact 1500 was different and presented a lower value (*P<.05*) when visible-light polymerized resin was used. In contrast, QC 20 acrylic resin showed an increased value (*P<.05*) when repaired with the same resin.

Analyses of the behavior of each acrylic resin studied, when submitted to flexural strength testing, pointed out that they differed according to the repair method used.

As regards stress at yield, repairs made with the same resin that the specimen was made of showed higher and statistically significant values (*P<.05)*, except for QC 20 resin; this acrylic resin did not present differences when repaired with either autopolymerized or visible-light resin, but QC 20 exhibited better results when the same resin the specimen was made of was used. Young modulus data showed that repair methods were different only for Lucitone 550 repaired with the same or visible light resin (*P<.05*). On the other hand, with regard to displacement at yield, the best performance was obtained with the repair made with the same resin used for fabricating the specimens (*P<.05*).

DISCUSSION

Denture fracture is a problem for patients and dentists, and denture repair procedures are recurrent. Comparative studies about the efficacy of repair methods should consider evaluating fractured specimens or dentures. Differently from the other studies that used two specimens for testing the repair method, in the present study, the repair methods were done using two halves of an already fractured specimen, considering the maintenance of stress and crack propagation.

In our study, impact and flexural strengths varied according to the type of denture base resin and repair method, but no tested denture base resin was completely superior to the others. The results showed that fractured specimens repaired with the same acrylic resin as they were made of exhibited the higher strengths. Furthermore, when the acrylic resins were repaired with autopolymerized or visible light resin they differed from each other.

The higher values for impact strength exhibited by Impact 1500 when it was repaired with autopolymerized resin could be attributed to the treatment of the fractured surface with the monomer, considering that both acrylic resins, Impact 1500 and autopolymerized, have the same cross linking agent (alkyl dimethacrylate) in their composition, and this could lead to greater monomers affinity in new polymeric chains formed [10].

With regard to the effects of using visible-light resin on the impact strength of the repaired acrylic resins, its brittle nature[1,21] was more evident in the Impact 1500 and Lucitone 550 resins (Table III). Impact strength was negatively affected due to the presence of cross-linking agents with different concentrations or the filler content (as titanium dioxide, in Impact 1500 composition) because these components can reduce the bond agent penetration into the polymeric matrix, resulting in poor interaction and lack of adhesion and/or cohesion between the materials [13,16-17].

The stress at yield was affected by plastic deformation from the fracture induction test, mainly in the autopolymerized and visible-light repair methods (Table III). These findings

disagree with those of some authors[2,5, 11, 9,23] and the explanation could be attributed to different methodologies used. In the present study, the specimens used were previously fractured by the mechanical tests and then used to evaluate the repair methods, but in the majority of repair studies, the specimens used were first sectioned in the center and then repaired; this procedure avoids or reduces plastic deformation[1-2, 15, 23, 26]. Therefore, the repair processes in these studies did not represent what happens clinically, where the dentures were submitted to stress and degradation by oral fluids before they fractured, and after repair, the dentures would be in service again. Thus, is almost impossible to remove the stress of the fractured prosthesis before repair procedure.

Although the stress at yield values were higher for Lucitone 550 and Impact 2000 repaired with the same resin (Table III), it was observed that they underwent a significant reduction in the strength: 47% and 50%, respectively. The lower strength values exhibited by these denture base resins could be explained by the concentration of tension from packing and flasking procedures and also the specimen distortion by heating[2], because the second polymerization process took as long as the first one. The stress absorption decreased 30% in the Impact 1500 resin, but QC 20 was not affected by the repair method.

The results of the present study also indicated that repairs carried out in the specimen that kept the plastic deformation did not influence the rigidity of the denture base materials, considering that the Young modulus values did not differ among the repair methods (Table III) Thus, it is important to remember that this property is not affected by the plastic or elastic stress intensity induced in a material[12].

As regards the displacement at yield, when either autopolymerized or visible-light resin were used for repairs, no differences were found among denture base resins. The lower values could be due to the deflection in repaired specimens being controlled by the nature of the join, flexibility of the specimen [15-23] and stress absorption capacity. In the present study, as no interaction between acrylic resins and repair methods was found for the displacement at yield,

the lack of adhesion could be attributed to the small adhesion area provided by the butt joint, the maintenance of plastic deformation or by the different chemical composition of the resins used for repair, which result in different flexural characteristics.

The adhesive failures originated by the two mechanical tests could also be attributed to the butt joint, such as the repair surface contour [3, 23, 26]. Several factors have been described as capable of affecting the strength of repaired acrylic resin, such as the contour of repaired surface [3-26]; pre-treatment of repaired surfaces with methylmethacrylate monomer[8] and longer water storage period[1,22]. The butt joint was chosen in this study because of requiring less preparation, considering that one of the purposes of this study was to use previously fractured specimens to keep the plastic deformation.

In this study, denture base resins, polymerization cycles and repair protocols differed among the three repair methods studied. Thus, the choice of the combination between the repair method and denture base acrylic resin is of major importance for obtaining the best mechanical properties and bond strength.

CONCLUSION

The results found in this study suggest that denture base resin repaired with the same resin as the one used to fabricate it showed greater flexural and impact strengths.

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Table I. Acrylic resins and products used in this study.

Materials	Chemical Composition	Polymerization method	Manufacturer/ Batch number
Lucitone 550	Powder: Methyl methacrylate (methyl-n-butyl) co-polymer, benzoyl peroxide, mineral pigments. Liquid: Methyl methacrylate, ethylene glycol dimethacrylate[§], hydroquinone.	Water bath - 9 hours at 73°C	Dentsply International Inc., Chicago, Ill, USA/ 36898/37375
Impact 2000	Powder: Nuisance dust, benzoyl peroxide, cadmium pigments Liquid: Methyl methacrylate monomer, ethylene glycol[§].	Water bath - 9 hours at 73°C	Bosworth Company, Skokie, Ill, USA/0401-022
Impact 1500	Powder: Particulate NOC (non-cadmium), residual mononer, titanium dioxide Liquid: Methyl methacrylate monomer, alkyldimethacrylate[§].	Boiling water at 100°C for 20 min	Bosworth Company, Skokie, Ill, USA/0006-328
QC20	Powder: Methyl methacrylate (methyl-n-butyl) co-polymer, benzoyl peroxide, atoxic pigments Liquid: Methyl methacrylate monomer, ethylene glycol dimethacrylate[§], terpinolene, N-N dimethyl p-toluidine, hydroquinone.	Boiling water at 100°C for 20 min	Dentsply International Inc., Chicago, Ill, USA / 29080/60066
Trurepair	Powder: Poly (Methyl methacrylate), benzoyl peroxide, cadmium pigments Liquid: Methyl methacrylate monomer, dimethyl-p-toluidine, alkyldimethacrylate[§].	At room temperature for 10 min	Bosworth Company, Skokie, Ill, USA/0108-474
Versyo. com	Cross-linked organic matrix, photo-hardening, single-component denture base resin consisting of dimethactylate and multi-functional methacrylates.	2 cycles of 90 sec* 1 cycle of180 sec**	Heraeus Kulzer, Germany/010109
Versyo. bond	Ethyl acetate, multifunctional and monofunctional methacrylates, acrylates and photo-initiators.	2 cycles for 90 sec*	Heraeus Kulzer, Germany/010022-1

[§] Cross-linking agent.*Pre polymerization in the Heralight pre ** Final polymerization in the UniXs

Table II: Two-way ANOVA comparison for impact strength, stress at yield, Young modulus and displacement at yield values after repair procedures.

Dependent Variable	Source of Variation	DF	SS	MS	F	P
Impact strength	Acrylic Resin	3	0.506	0.169	22.490	<0.001
	Repair Method	2	2.696	1.348	179.780	<0.001
	Resin x Repair method	6	0.913	0.152	20.290	<0.001
	Residual	60	0.45	0.008		
	Total	71	4,565	0,064		
Stress at yield	Acrylic Resin	3	0.079	0.026	1.950	0.1318
	Repair Method	2	1.553	0.776	57.160	<0.001
	Resin x Repair method	6	0.197	0.033	2.420	0.0369
	Residual	59	0.801	0.014		
	Total	70	2.630	0.037		
Young modulus	Acrylic Resin	3	0.459	0.153	137.140	<0.001
	Repair Method	2	0.010	0.005	4.670	0.013
	Resin x Repair method	6	0.016	0.003	2.350	0.0422
	Residual	60	0.067	0.001		
	Total	71	0.552	0.008		
Displacement at yield	Acrylic Resin	3	1.515	0.505	9.700	<0.001
	Repair Method	2	7.891	3.945	75.780	<0.001
	Resin x Repair method	6	0.498	0.083	1.590	0.1649
	Residual	59	3.072	0.052		
	Total	70	12.98	0.186		

Table III. Mean values ad standard deviations for impact strength, stress at yield (MPa), Young modulus (MPa) and displacement at yield (mm) after repair methods (n=6).

		Repair Method		
Dependent Variable	**Acrylic Resin**	**G1**	**G2**	**G3**
Impact strength	Lucitone 550	3.3 ± 0.6 (a)	1.5 ± 0.3 (b)	0.8 ± 0.2 (c)
	Impact 2000	3.2 ± 0.3 (a)	1.2 ± 0.3 (b)	1.9 ± 0.5 (c)
	Impact 1500	1.8 ± 0.5 (a)	1.6 ± 0.4 (a)	0.5 ± 0.0 (b)
	QC 20	3.0 ± 0.6 (a)	1.0 ± 0.0 (b)	1.0 ± 0.0 (b)
Stress at yield	Lucitone 550	48.3 ± 13.5 (a)	19.0 ± 3.1 (b)	20.3 ± 8.5 (b)
	Impact 2000	47.9 ± 20.8 (a)	20.5 ± 5.3 (b)	23.5 ± 4.4 (b)
	Impact 1500	38.6 ± 5.4 (a)	19.7 ± 8.8 (b)	18.6 ± 2.5 (b)
	QC 20	31.5 ± 4.8 (a)	25.0 ± 3.6 (ab)	18.9 ± 6.0 (b)
Young modulus	Lucitone 550	2239 ± 65 (a)	1981 ± 111 (b)	2158 ± 238 (ab)
	Impact 2000	1899 ± 146 (a)	1926 ± 186 (a)	2126 ± 232 (a)
	Impact 1500	1495 ± 132 (a)	1572 ± 62 (a)	1646 ± 99 (a)
	QC 20	1278 ± 83 (a)	1364 ± 91 (a)	1373 ± 94 (a)
Displacement at yield	Lucitone 550	3.2 ± 0.7 (a)	1.6 ± 0.2 (b)	1.3 ± 0.7 (b)
	Impact 2000	3.6 ± 1.5 (a)	1.7 ± 0.5 (b)	1.9 ± 0.5 (b)
	Impact 1500	4.8 ± 1.1 (a)	2.0 ± 0.7 (b)	1.8 ± 0.3 (b)
	QC 20	5.0 ± 1.2 (a)	3.2 ± 1.1 (b)	1.8 ± 0.8 (b)

Different letters show significant differences among repair method for acrylic resin (*P<.05*).

4 DISCUSSÃO

Fraturas em resinas acrílicas para base de próteses ainda são problemas relatados tanto por pacientes usuários como por cirurgiões dentistas. Nos últimos 30 anos tem se observado esforços por parte dos fabricantes em se modificar a composição química destes polímeros, e de inúmeros estudos em desenvolver meios alternativos adicionais para o aumento da resistência a fratura destes materiais como por exemplo, a incorporação de flocos de vidro (Franklin et al., 2005), fibras (Jagger et al., 2001) e metal (Vallitu, 1996) durante o processamento dos mesmos.

Embora a adição de agentes modificadores de impacto na composição química das resinas acrílicas apresentem resultados alentadores, as propriedades relativas a resistência a fratura ainda estão longe do que se consideraria o ideal. Um dos objetivos desta pesquisa foi avaliar as propriedades mecânicas, o processo de fratura e deformação bem como a microestrutura de resinas acrílicas que apresentam em sua composição química partículas de borracha ou a adição de um agente de ligação cruzada, o alquildimetacrilato, e compará-las com resinas acrílicas convencionais.

Neste estudo, observou-se que a resina acrílica com incorporação de partículas de borracha em sua composição química apresentou resistência ao impacto 2,4 vezes superior as demais resinas acrílicas estudadas além de ter obtido valores superiores para a tensão de ruptura. Entretanto, o mesmo não foi observado para o Módulo Young que foi inferior ao de uma resina convencional não reforçada (Lucitone 550), e em acréscimo apresentou fraturas frágeis ao invés de dúctil como seria esperado.

Uma razão apontada para o desenvolvimento de fratura frágil poderia ser o fato de que uma porcentagem de partículas de borrachas poderia ter se desprendido da matriz polimérica durante o processo de fratura (Cho et al., 1998). Esta afirmação foi corroborada pela encontrada na análise microestrutural dessa resina ,realizada em MEV com os espécimes resultantes da resistência a flexão. Nesta análise, uma microestrutura granular e cavidades bem

delimitadas puderam ser observadas. Desta forma pode-se supor que as partículas de borracha não se incorporaram de forma eficiente a matriz polimérica durante o processo de polimerização.

Por outro lado, quando o gráfico tensão/deformação foi analisado, não foram encontradas diferenças significantes (P>0,05) para o deslocamento de escoamento entre as resinas com incorporação de borracha (Impact 2000) ou com adição de um agente de ligação cruzada alquildimetacrilato (Impact 1500); entretanto ambas apresentaram valores maiores e significantes quando comparadas com as resinas convencionais (P<0,05). Dessa forma, comprova-se que ambas foram capazes de dissipar a tensão lentamente através das cadeias do polimetilmetacrilato, mas por mecanismos diferentes, pois segundo Jagger et al. (1999, 2002) uma resina acrílica reforçada com borracha é capaz de desacelerar a propagação de trincas e fraturas na interface entre a rede polimérica e as partículas borrachosas.

As resinas QC20 e Impact 1500 exibiram comportamento característico de fratura dúctil, e quando da comparação entre estas duas resinas de ciclo de polimerização curto (100°C por 20 minutos), Impact 1500 obteve um processo de fratura mais estável e com maior potencial de resistir a tensões de ruptura. Uma justificativa para tais diferenças poderia ser o fato das mesmas possuírem agentes de ligação cruzada diferentes, e neste caso o alquildimetacrilato presente no Impact 1500 poderia ter sofrido menor influência da temperatura de polimerização, que por sua vez pode ser capaz de limitar a geometria da rede polimérica. Além disso, agentes de ligação cruzada na forma de monômero residual ou cadeias pendentes poderiam ter agido como plasticizante (Harrison et al., 1978).

O objetivo do segundo estudo foi determinar o processo de fratura em resinas acrílicas reparadas, mantendo-se a deformação pré-existente provenientes dos ensaios mecânicos executados no primeiro estudo. Os resultados desta pesquisa mostraram que todos os corpos de prova que foram reparados com as mesmas resinas que foram confeccionadas apresentaram valores superiores (P<0,05) para todas as propriedades mecânicas estudadas,

com exceção do Módulo Young que apresentou valores similares aos valores obtidos antes dos corpos de prova ser reparados (Anexo 4.1). Estes resultados podem ser explicados pela presença de radicais livres de metilmetacrilato nas resinas acrílicas processadas, na forma de monômero residual e/ou agentes de ligação cruzada não reagidos, que associados a inserção de uma nova camada de material e um segundo ciclo de polimerização, promovem um alto grau de conversão (Kupiec and Barkmeier, 1996, César et al., 2001)

Neste estudo, a tensão de ruptura foi a propriedade que mais sofreu o efeito da deformação plástica inerente ao corpo de prova, principalmente quando os mesmos foram reparados pelos métodos autopolimerizável e fotopolimerizável. Estes achados discordam de alguns autores (Leong et al., 1971; Stafford et al., 1955; Polyzois et al., 1996; Rached et al., 2004; Jagger et al., 2003) provavelmente porque estas diferenças poderiam ser explicadas pelas diferentes metodologias usadas. Na maioria dos estudos de reparo os corpos-de-prova são sempre seccionados no centro para se evitar a incidência de deformação plástica e logo após reparados (Polyzois et al., 2001; Lin et al., 2000; Dar-Odeh et al., 1997; Polyzois et al., 1996; Rached et al., 2004; Andreopoulos et al., 1994). Assim sendo, o processo de reparo nestes estudos não representa a real performance clínica dos métodos de reparo em próteses fraturadas, uma vez que efeitos intrínsecos como a degradação do material pela tensão oriunda por danos provenientes de fraturas por impacto e flexão, não podem ser totalmente removidas.

O efeito dos métodos de reparo na resistência mecânica dos corpos-de-prova reparados pode ser interpretado e discutido através da análise dos resultados da ANOVA de dois critérios, que mostraram a existência de interação entre resina acrílica e métodos de reparo para resistência ao impacto, tensão de ruptura e Módulo Young. Assim, a combinação entre resina acrílica e métodos de reparo pode ser responsável pela alteração de tenacidade do material reparado e absorção de energia durante a fratura por impacto, mostrando que reparos com o mesma resina que foi confeccionado o corpo-de-prova são capazes de minimizar os efeitos da deformação plástica mantida. Com relação queda significativa da tensão de ruptura, esta pode

ser atribuída aos diferentes comportamentos mecânicos das resinas acrílicas utilizadas como materiais reparadores, uma vez que com esta finalidade permanecem confinadas a uma menor área e portanto com menor capacidade de dissipar uma carga flexural. Além disso, diferentes técnicas de processamento podem interferir na eficácia da adesão do material reparador ao material de base.

Quanto ao Módulo Young, os valores similares obtidos pelos corpos-de-prova antes e após a execução de reparos nos métodos estudados nas quatro resinas acrílicas confirmaram que efeitos da intensidade de tensão induzida no material, seja ela plástica ou elástica, não são capazes de alterar a rigidez do material (Anuavice, 2003).

Diferentemente, para o deslocamento de escoamento os valores inferiores obtidos pelos reparos auto e fotopolimerizáveis poderiam ser atribuídos ao fato de que durante o processo de fratura em espécimes reparados a deflexão é controlada pela natureza da junção, pela flexibilidade do espécime e pela capacidade de absorção de tensão (Andreopoulos e Polyzois, 1994; Polyzois et al.,1996). Como em nosso estudo, não foi observada interação entre resina e métodos de reparo para o deslocamento de escoamento, a deficiência de adesão dos reparos pelos métodos auto e foto poderiam ser atribuídos aos seguintes fatores: pequena área de adesão fornecida pela junção em topo (Lin et al, 2000; Polyzois et al., 1994, Ward et al., 1992) manutenção (não remoção) da deformação plástica e pelas diferentes composições e afinidades químicas das resinas reparadoras, que por sua vez, possuem diferentes características flexurais (Stipho et al.,1999; Andreopoulos e Polyzois, 1994; Soderholm e Roberts, 1991; Pontes et al., 2005; Andreopoulos et al., 1991; al-Mulla et al., 1988; Ishigami et al., 1987).

Ainda, nesta pesquisa materiais, ciclos de polimerização e protocolos de reparos foram diferentes entre os três métodos de reparo estudados. Entretanto, os diferentes resultados observados entre as várias associações entre resina acrílica e métodos de reparo tendem a confirmar achados anteriores (Grajower R. and Goultschin J., 1984; Moradians et al., 1982;

Andreopoulos et al., 1991; Shen et al., 1984; Berge et al., 1983) que concluem que a escolha da combinação entre o método de reparo e a resina acrílica para base de prótese é de grande importância na manutenção das propriedades mecânicas e da resistência a adesão de reparos. Em complementação, estudos adicionais deveriam ser realizados com o objetivo de se avaliar a influência da quantidade de monômero residual na eficácia de reparos, a estabilidade do reparo em função do tamanho e localização da área a ser reparada, bem como sua associação com os efeitos deletérios na cavidade oral.

5 CONCLUSÃO

De acordo com os resultados obtidos e dentro das limitações desse estudo concluí-se que:

- A resina acrílica contendo partículas de borracha e dessa forma considerada de alto impacto apresentou alta capacidade de dissipação de energia, absorção de tensão e baixo valor de deformação.

- A incorporação de borracha na composição química de uma resina acrílica não desencadeou um comportamento de fratura dúctil apesar de ter apresentado módulo de elasticidade inferior a uma resina acrílica não reforçada.

- Reparos realizados com a mesma resina acrílica utilizada para a confecção do corpo-de-prova, sejam elas reforçadas ou não, apresentaram melhores resultados quanto à resistência mecânica.

- Para se estudar reparo de resinas acrílicas termopolimerizáveis, a manutenção da deformação pré-existente oriunda dos testes de fratura por impacto e flexão demonstrou ser um método capaz de mensurar a eficácia de reparos nos espécimes de todas as resinas estudadas.

- Segundo este estudo, se métodos indiretos de reparos forem utilizados, sugere-se a adoção de resina acrílica autopolimerizável.

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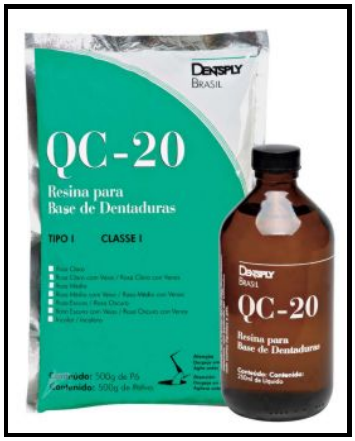
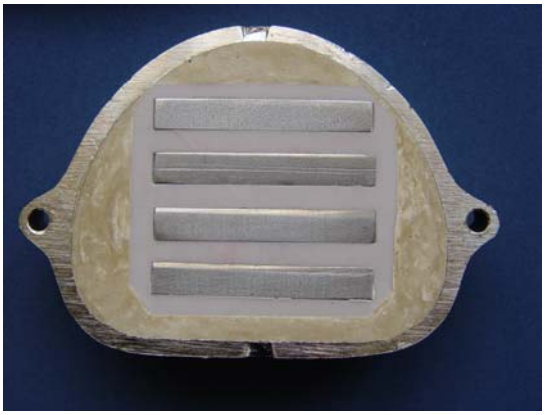
ANEXOS

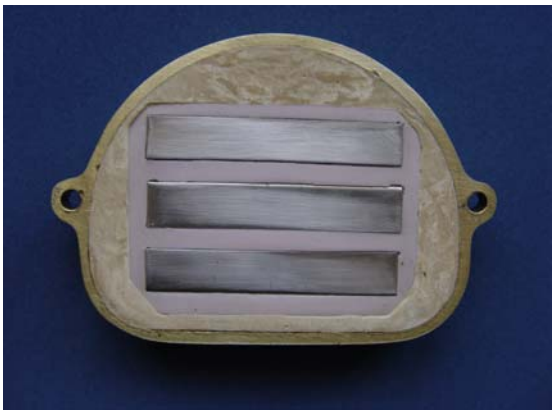
ANEXO 1 – Figuras do Capítulo 1

1. Resinas acrílicas utilizadas neste estudo.

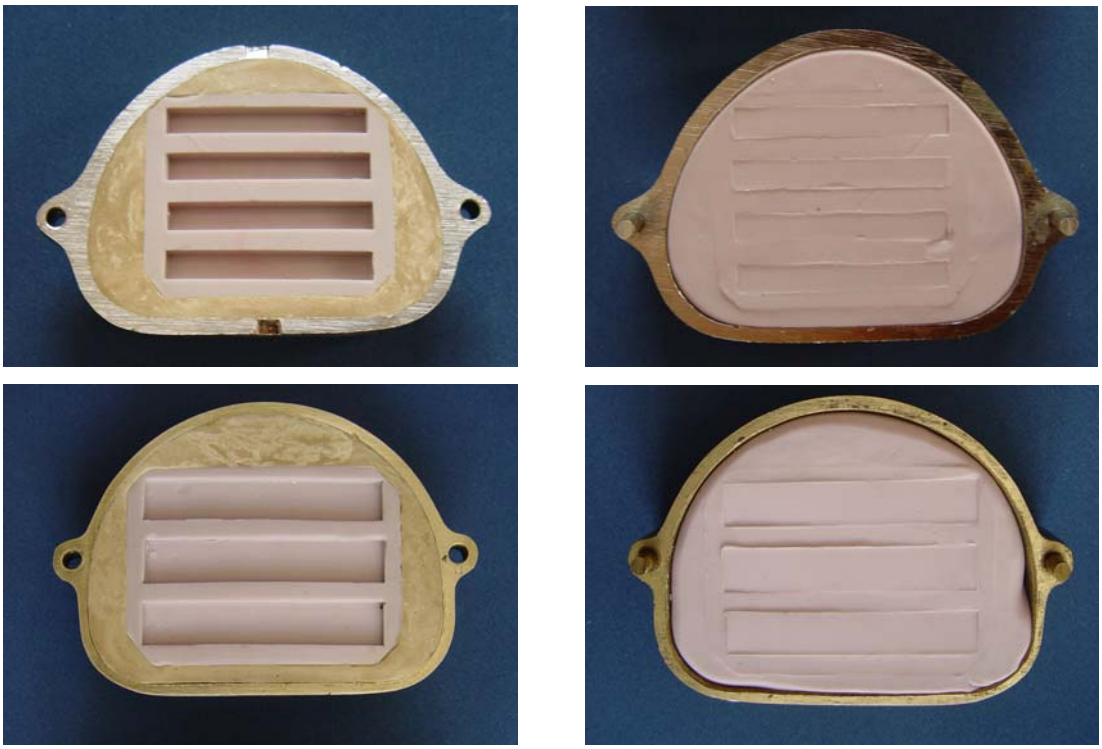


2. Matrizes metálicas dos corpos de prova para o teste resistência ao impacto e flexão.

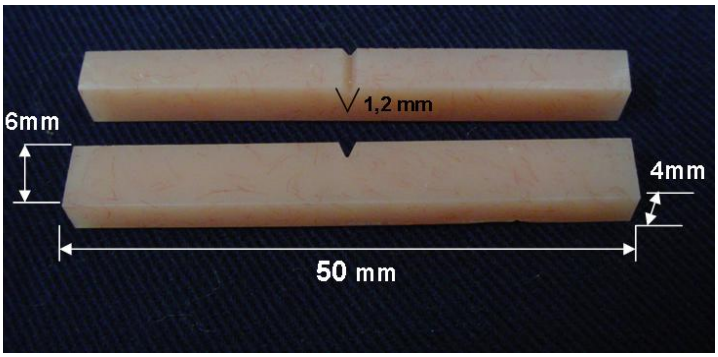




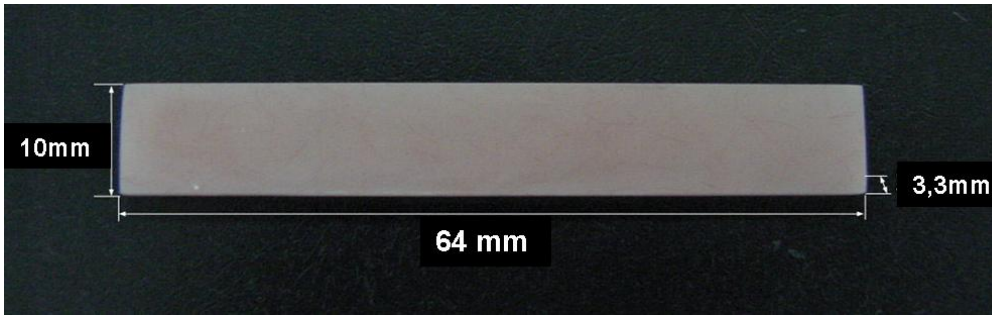
3. **Moldes dos corpos de prova para o teste de resistência a flexão.**



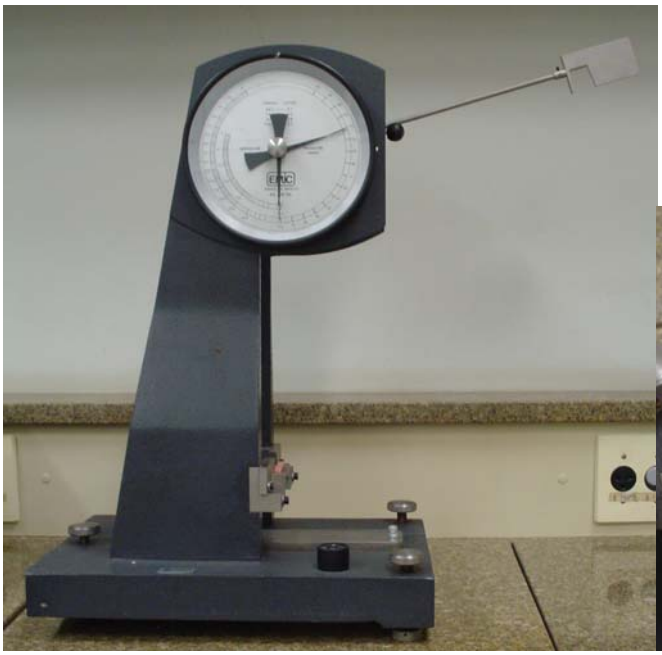
4. **Corpos de prova para o teste de resistência ao Impacto (entalhado).**

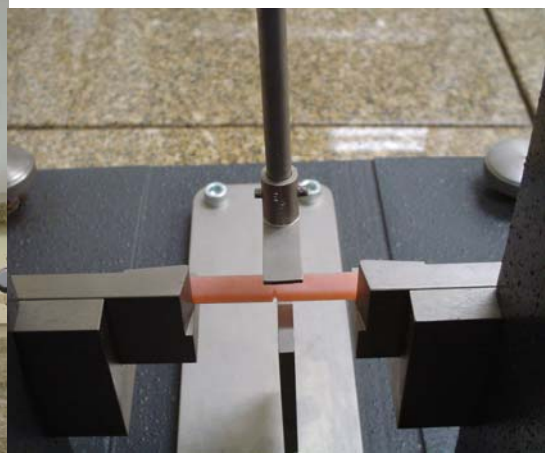


5. **Corpos de prova para o teste de resistência a flexão.**

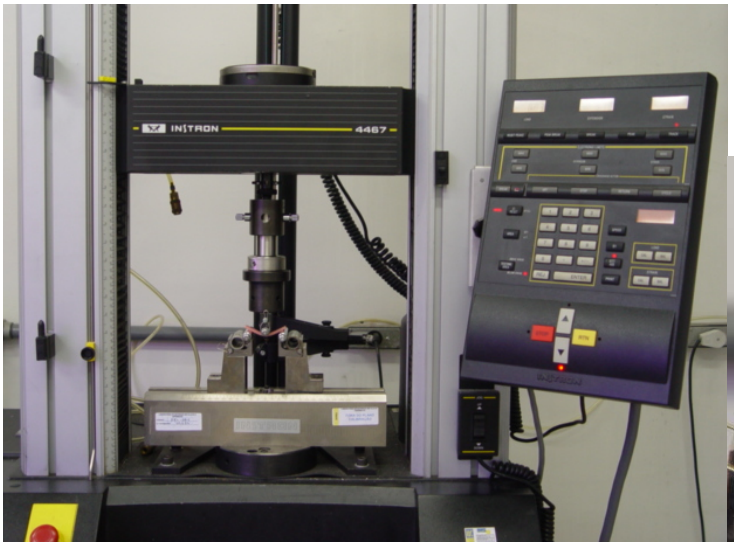


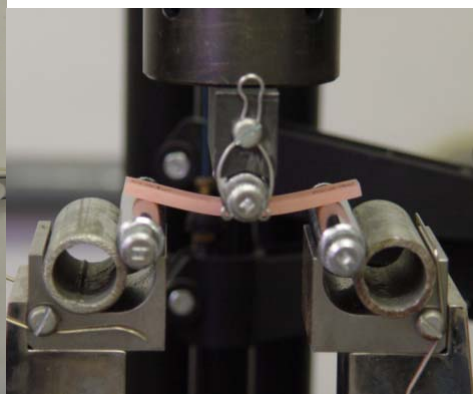
6. Máquina de ensaio de resistência ao impacto em polímeros – método Charpy.



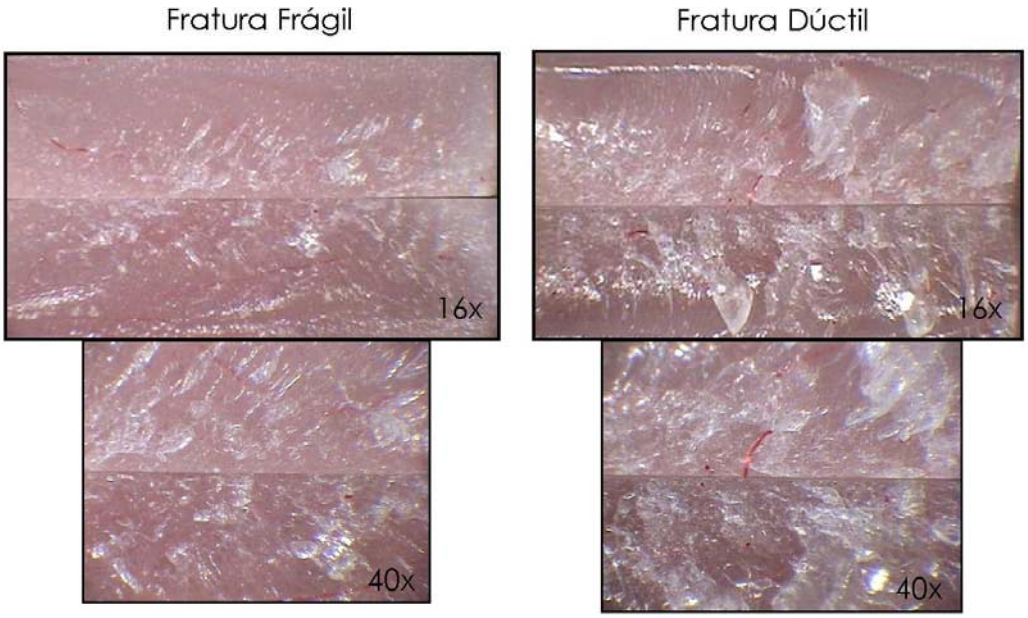


7. Máquina de ensaio universal – Teste de resistência à flexão em 3 pontos.

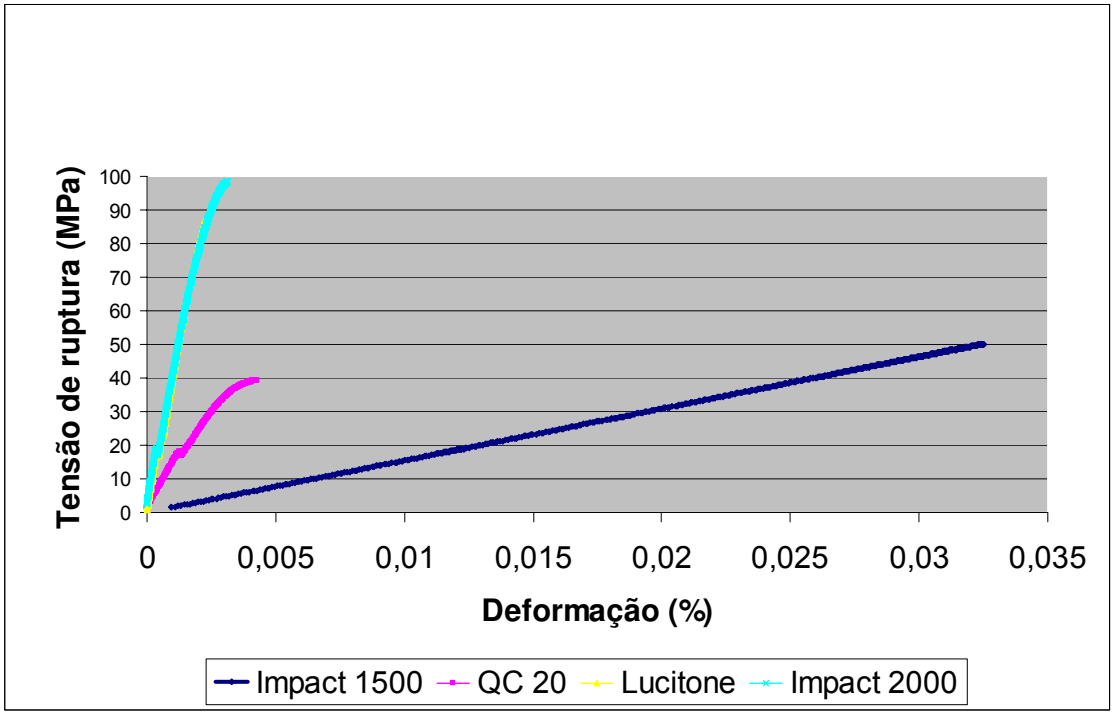




8. Modelo de superfície das fraturas frágeis e dúcteis dos corpos de prova fraturados. Observação em lupa estereoscópica em aumento de 16× ou 40×.



9. Gráfico de Tensão-Deformação.



ANEXO 2 – Figuras do Capítulo 2

1. Corpos de prova fraturados pelo teste de resistência ao impacto e flexão posicionados nos moldes demonstrando a obtenção do gap de 3mm para reparo.

Corpos-de prova de resistência ao impacto

Corpos-de-prova de resistência à flexão

2. Resina acrílica autopolimerizável TruRepair para confecção de reparos.

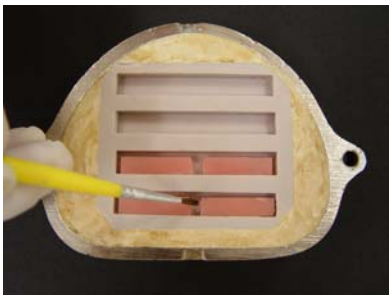
3. Sistema Versyo.com – confecção do reparo com resina fotopolimerizável.

Sistema de injeção pneumático e resina VersyoHD.

Agente de união

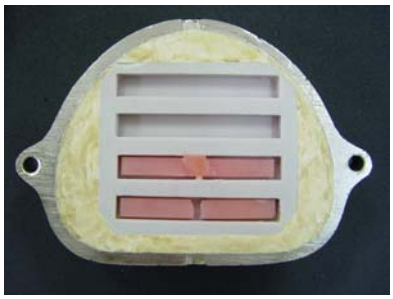
Pré-polimerizadora Heralight

4. Demonstração da execução do reparo nos corpos de prova de resistência ao impacto.



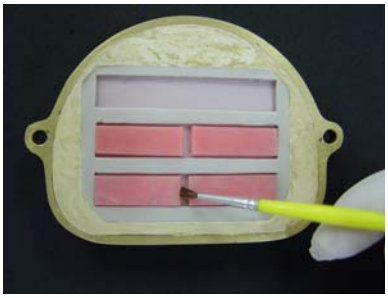
Interfaces tratadas com monômero

Reparo resina termopolimerizável

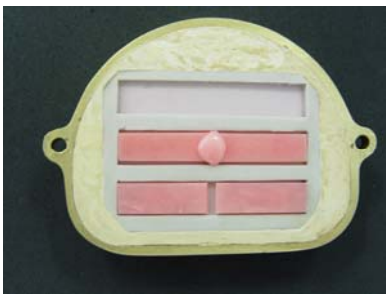


Reparo resina autopolimerizável

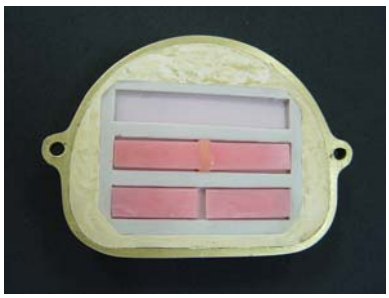
5. Demonstração da execução do reparo nos corpos de prova de resistência a flexão.



Interfaces tratadas com monôme

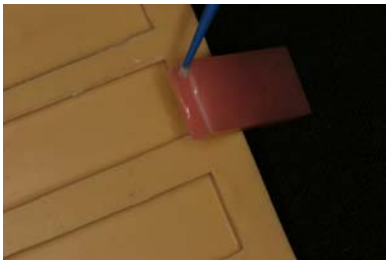


Reparo resina termopolimerizável



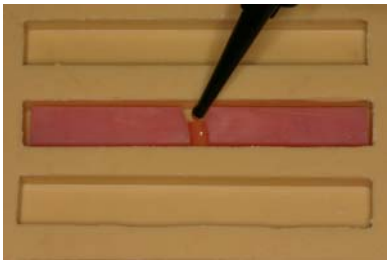
Reparo resina autopolimerizável

6. Demonstração da execução do reparo com resina fotopolimerizável (Versyo.com).



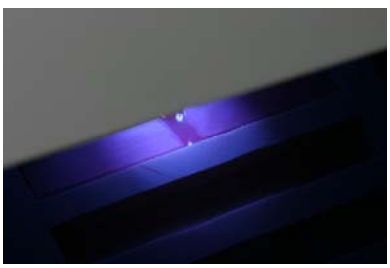
Aplicação do Versyo.bond

Fotopolimerização do sistema adesivo



Aplicação do 1ºincremento de resina

Aplicação do 2ºincremento de resina



Demonstração da Pré-polimerização

Fotopolimerização final

ANEXO 3 – ANÁLISE DOS RESULTADOS E TESTES ESTATÍSTICOS -CAPÍTULO 1

3.1 Análise dos resultados de Resistência ao Impacto

Tabela 1 – Estatísticas descritivas

Resina	Média	Erro-padrão	Mínimo	Máximo	N
QC20	1,022	0,0147	0,771	1,041	18
Lucitone	1,023	0,0328	0,781	1,302	18
Impact 2000	2,491	0,0750	2,057	3,101	18
Impact 1500	1,009	0,0195	0,781	1,041	18
Total	**1,386**	**0,0785**	**0,7716**	**3,101**	**72**

Tabela 2 – ANOVA (LOG RI)

Fonte de Variação	Soma de Quadrados	Graus de Liberdade	Quadrado Médio	Teste F	Significância
Resinas	3.42786350	3	1.14262117	497.13	<0,0001
Resíduos	0.26661940	68	0.00229844		
Total	3.69448290	71			

R^2= 0.927833

Tabela 3 - Teste de Comparações Múltiplas (Tukey HSD) para LOG RI

Resina	Média – (Teste de Tukey)	Significância
QC 20	0.00698 (B)	0,0323
Lucitone 550	0.00467 (B)	
Impact 2000	0.39380 (A)	
Impact 1500	-0.00102 (B)	

3.2 Análise dos resultados de Tensão de ruptura

Tabela 1 – Estatísticas descritivas

Resina	Média	Erro padrão	Mínimo	Máximo	N
QC 20	35,31	1,72	24,88	50,39	18
Lucitone	86,30	1,76	73,97	100,37	18
Impact 2000	97,32	1,14	85,46	104,89	18
Impact 1500	56,88	1,47	48,15	70,10	18
Total	**68,95**	**2,99**	**24,88**	**104,89**	**72**

Tabela 2 – ANOVA

Fonte de Variação	Soma de Quadrados	Graus de Liberdade	Quadrado Médio	Teste F	Significância
Resinas	42.905,08	3	14.301,69	331,19	0,00
Resíduos	2.936,34	68	43,18		
Total	45.841,42	71			

R^2= 93,6%

Tabela 3 - Teste de Comparações Múltiplas (Tukey HSD)

(I) Resina	(J) Resina	Diferença Média (I-J)	Significância
QC 20	Lucitone	-50,99	0,00
	Impact 2000	-62,02	0,00
	Impact 1500	-21,57	0,00
Lucitone	QC 20	50,99	0,00
	Impact 2000	-11,03	0,00
	Impact 1500	29,42	0,00
Impact 2000	QC 20	62,02	0,00
	Lucitone	11,03	0,00
	Impact 1500	40,44	0,00
Impact 1500	QC 20	21,57	0,00
	Lucitone	-29,42	0,00
	Impact 2000	-40,44	0,00

3.3 Análise dos resultados de Módulo Young

Tabela 1 – Estatísticas descritivas

Resina	Média	Erro padrão	Mínimo	Máximo	N
QC 20	1.172,79	44,98	907,86	1.533,29	18
Lucitone	2.460,70	48,19	2.056,70	2.772,38	18
Impact 2000	2.079,74	35,19	1.808,74	2.351,07	18
Impact 1500	1.459,03	26,63	1.231,97	1.635,98	18
Total	13793,06	63,11	970,06	2.772,30	72

Tabela 2 – ANOVA

Fonte de Variação	Soma de Quadrados	Graus de Liberdade	Quadrado Médio	Teste F	Significância
Resinas	18.436.390,09	3	6.145.463,36	216,97	0,00
Resíduos	1.925.992,06	68	28.323,41		
Total	20.362.382,15	71			

R^2= 90,5%

Tabela 3 - Teste de Comparações Múltiplas (Dunnett T3)

(I) Resina	(J) Resina	Diferença Média (I-J)	Significância
QC 20	Lucitone	-1.287,91	0,00
	Impact 2000	-906,96	0,00
	Impact 1500	-286,24	0,00
Lucitone	QC 20	1.287,91	0,00
	Impact 2000	380,95	0,00
	Impact 1500	1.001,67	0,00
Impact 2000	QC 20	906,96	0,00
	Lucitone	-380,95	0,00
	Impact 1500	620,71	0,00
Impact 1500	QC 20	286,24	0,00
	Lucitone	-1.001,67	0,00
	Impact 2000	-620,71	0,00

3.4 Análise dos resultados de Deslocamento de escoamento

Tabela 1 – Estatísticas descritivas

Resina	Média	Erro padrão	Mínimo	Máximo	N
QC 20	6,08	0,154	4,65	7,05	18
Lucitone	5,20	0,185	3,68	6,79	18
Impact 2000	8,67	0,311	6,60	10,69	18
Impact 1500	8,87	0,189	6,80	10,21	18
Total	**7,21**	**0,217**	**3,68**	**10,69**	**72**

Tabela 2 – ANOVA (LN Deslocamento)

Fonte de Variação	Soma de Quadrados	Graus de Liberdade	Quadrado Médio	Teste F	Significância
Resinas	3,78	3	1,26	71,64	0,00
Resíduos	1,19	68	0,01		
Total	4,98	71			

R^2= 76%

Tabela 3 - Teste de Comparações Múltiplas (Dunnett T3)

(I) Resina	(J) Resina	Diferença Média (I-J)	Significância
QC 20	Lucitone	0,161	0,007
	Impact 2000	-0,348	0,000
	Impact 1500	-0,378	0,000
Lucitone	QC 20	-0,161	0,007
	Impact 2000	-0,509	0,000
	Impact 1500	-0,540	0,000
Impact 2000	QC 20	0,348	0,000
	Lucitone	0,509	0,000
	Impact 1500	-0,030	0,980
Impact 1500	QC 20	0,378	0,000
	Lucitone	0,540	0,000
	Impact 2000	0,030	0,980

ANEXO 4 – ANÁLISE DOS RESULTADOS E TESTES ESTATÍSTICOS -CAPÍTULO 2

4.1 Médias e desvio padrão da resistência ao impacto (J), tensão de ruptura (Mpa), módulo Young (Mpa) e deslocamento de escoamento (mm) das resinas acrílicas estudadas antes e após os reparos.antes e após os métodos de reparo. Dados na íntegra.

		Tensão de ruptura					Modulo Young					Deslocamento de escoamento					Resistência ao impacto				
Resina	Reparo	Antes	Depois	Result Médio	Desvio-padrão	%	Antes	Depois	Result Médio	Desvio-padrão	%	Antes	Depois	Result Médio	Desvio-padrão	%	Antes	Depois	Result Médio	Desvio - padrão	%
QC20	Ppresina	30,62	31,51	0,89	3,46	3%	1.061,54	1.277,62	216,08	59,01	20%	5,90	5,04	-0,86	0,40	-15%	1,04	3,02	1,99	0,25	192%
QC20	Auto	32,69	24,96	-7,73	2,39	-24%	1.100,97	1.363,61	262,64	87,55	24%	6,13	3,19	-2,94	0,58	-48%	0,99	1,04	0,04	0,04	4%
QC20	Foto	32,78	18,90	-13,88	4,02	-42%	1.355,86	1.373,21	17,35	44,87	1%	6,24	1,84	-4,40	0,51	-71%	1,04	1,04	0,00	0,00	0%
Lucitone	Ppresina	90,55	48,30	-42,25	6,09	-47%	2.538,07	2.238,65	-299,41	57,71	-12%	5,14	3,18	-1,96	0,39	-38%	1,04	3,33	2,29	0,25	221%
Lucitone	Auto	81,16	18,97	-62,19	1,99	-77%	2.509,02	1.980,62	-528,39	109,66	-21%	4,99	1,56	-3,43	0,30	-69%	1,04	1,47	0,43	0,09	42%
Lucitone	Foto	87,20	20,30	-66,90	3,80	-77%	2.335,02	2.158,25	-176,78	119,84	-8%	5,49	1,27	-4,22	0,41	-77%	1,00	0,78	-0,22	0,04	-22%
Impact 2000	Ppresina	96,46	47,89	-48,57	9,63	-50%	2.132,05	1.899,09	-232,96	119,82	-11%	8,65	3,62	-5,03	0,99	-58%	2,34	3,16	0,82	0,19	35%
Impact 2000	Auto	96,65	20,51	-76,14	2,55	-79%	2.040,49	1.926,21	-114,28	48,89	-6%	9,14	1,73	-7,42	0,57	-81%	2,55	1,21	-1,34	0,10	-53%
Impact 2000	Foto	98,88	23,47	-75,41	1,55	-76%	2.066,71	2.125,85	59,14	94,94	3%	8,23	1,87	-6,36	0,54	-77%	2,59	1,94	-0,65	0,09	-25%
Impact 1500	Ppresina	56,30	38,59	-17,71	4,43	-31%	1.404,04	1.494,83	90,79	81,84	6%	8,92	4,79	-4,13	0,50	-46%	1,04	1,81	0,78	0,21	75%
Impact 1500	Auto	56,86	19,71	-37,15	4,34	-65%	1.476,46	1.571,54	95,08	22,92	6%	9,14	1,96	-7,18	0,42	-79%	0,95	1,56	0,61	0,19	64%
Impact 1500	Foto	57,49	18,55	-38,94	2,80	-68%	1.496,60	1.645,58	148,98	62,78	10%	8,56	1,77	-6,79	0,44	-79%	1,04	0,52	-0,52	0,00	-50%

4.2 Médias e desvio padrão da resistência ao impacto (J), tensão de ruptura (Mpa), módulo Young (Mpa) e deslocamento de escoamento (mm) das resinas acrílicas estudadas antes e após os reparos.antes e após os métodos de reparo. G1 – reparos com a própria resina, G2- reparos com resina autopolimerizável e G3- reparos com resina fotopolimerizável.

			Métodos de reparo		
Variável dependente	**Resinas acrílicas**		**G1**	**G2**	**G3**
Tensão de ruptura	Lucitone 550	A	48.3 ± 13.5 (a)α	19.0 ± 3.1 (b)β	20.3 ± 8.5 (b)β
	Impact 2000	A	47.9 ± 20.8 (a)	20.5 ± 5.3 (b)	23.5 ± 4.4 (b)
	Impact 1500	A	38.6 ± 5.4 (a)	19.7 ± 8.8 (b)	18.6 ± 2.5 (b)
	QC 20	A	31.5 ± 4.8 (a)	25.0 ± 3.6 (ab)	18.9 ± 6.0 (b)
Módulo Young	Lucitone 550	A	2239 ± 65 (a) α	1981 ± 111 (b) α	2158 ± 238 (ab) β
	Impact 2000	B	1899 ± 146 (a)	1926 ± 186 (a)	2126 ± 232 (a)
	Impact 1500	C	1495 ± 132 (a)	1572 ± 62 (a)	1646 ± 99 (a)
	QC 20	D	1278 ± 83 (a)	1364 ± 91 (a)	1373 ± 94 (a)
Deslocamento de escoamento	Lucitone	A	3.2 ± 0.7 (a) α	1.6 ± 0.2 (b) β	1.3 ± 0.7 (b) β
	Lucitone 550	A	3.6 ± 1.5 (a)	1.7 ± 0.5 (b)	1.9 ± 0.5 (b)
	Impact 2000	BC	4.8 ± 1.1 (a)	2.0 ± 0.7 (b)	1.8 ± 0.3 (b)
	Impact 1500	C	5.0 ± 1.2 (a)	3.2 ± 1.1 (b)	1.8 ± 0.8 (c)
Resistência ao impacto	Lucitone 550	A	3.3 ± 0.6 (a) α	1.5 ± 0.3 (b) β	0.8 ± 0.2 (c)γ
	Impact 2000	B	3.2 ± 0.3 (a)	1.2 ± 0.3 (b)	1.9 ± 0.5 (c)
	Impact 1500	C	1.8 ± 0.5 (a)	1.6 ± 0.4 (a)	0.5 ± 0.0 (b)
	QC 20	A	3.0 ± 0.6 (a)	1.0 ± 0.0 (b)	1.0 ± 0.0 (b)

Letras diferentes mostram as diferenças significantes. Letras maiúsculas na linha vertical mostram as diferenças entre as resinas acrílicas; letras minúsculas mostram as diferenças entre os métodos de reparo dentro de cada resina acrílica. Símbolos mostram as diferenças entre os métodos de reparo. (P<.05).

4.3 Médias e desvio padrão das diferenças (depois-antes) para resistência ao impacto (J), tensão de ruptura (Mpa), módulo Young (Mpa) e deslocamento de escoamento (mm) das resinas acrílicas estudadas após os métodos de reparo. G1 – reparos com a própria resina, G2- reparos com resina autopolimerizável e G3- reparos com resina fotopolimerizável.

			Métodos de reparo		
Variável dependente	**Resinas acrílicas**		**G1**	**G2**	**G3**
Tensão de ruptura	Lucitone 550	A	-42.2 ±14.9 (a) α	-62.2 ± 4.9 (b) β	-66.9 ± 9.3 (b) β
	Impact 2000	A	-48.6 ±23.6 (a)	-76.1 ± 6.2 (b)	-75.4 ± 3.8 (b)
	Impact 1500	B	-17.7 ±10.8 (a)	-37.2 ± 10.6 (b)	-38.9 ± 6.9 (b)
	QC 20	C	0.9 ±8.5 (a)	-7.7 ± 5.8 (ab)	-13.9 ± 4.0 (b)
Módulo Young	Lucitone 550	A	-299.4 ± 141.4 (a) α	-528.4 ± 268.6 (a) α	-176.8 ± 293.5 (a) α
	Impact 2000	AB	-233 ± 293.5 (a)	-114.3 ± 119.8 (a)	59.1 ± 232.5 (a)
	Impact 1500	B	90.8 ± 200.5 (a)	95.1 ± 56.2 (a)	149.0 ± 153.8 (a)
	QC 20	B	216.1 ± 144.5 (a)	262.6 ± 214.4 (a)	17.3 ± 109.9 (a)
Deslocamento de escoamento	Lucitone 550	A	-1.96 ± 0.96 (a) α	-3.43 ± 0.74 (b) β	-4.22 ± 1.00 (b) β
	Impact 2000	B	-5.03 ± 2.42 (a)	-7.42 ± 1.38 (a)	-6.36 ± 1.33 (a)
	Impact 1500	B	-4.13 ± 1.23 (a)	-7.18 ± 1.03 (b)	-6.79 ± 1.07 (b)
	QC 20	A	-0.86 ± 0.97 (a)	-2.94 ± 1.42 (ab)	-4.40 ± 1.24 (b)
Resistência ao impacto	Lucitone 550	A	2.29 ± 0.62 (a) α	0.43 ± 0.21 (b) β	-0.22 ± 0.11 (b) β
	Impact 2000	AB	0.82 ± 0.48 (ab)	-1.34 ± 0.25 (a)	-0.65 ± 0.22 (b)
	Impact 1500	A	0.78 ± 0.52 (a)	0.61 ± 0.45 (a)	-0.52 ± 0.00 (a)
	QC 20	B	1.99 ± 0.60 (a)	0.04 ± 0.10 (b)	0.00 ± 0.00 (-)

Letras diferentes mostram as diferenças significantes. Letras maiúsculas na linha vertical mostram as diferenças entre as resinas acrílicas; letras minúsculas mostram as diferenças entre os métodos de reparo dentro de cada resina acrílica. Símbolos mostram as diferenças entre os métodos de reparo. (P<.05).

5.4 Médias e desvio padrão dos percentuais (diferença entre depois-antes) para resistência ao impacto (J), tensão de ruptura (Mpa), módulo Young (Mpa) e deslocamento de escoamento (mm) das resinas acrílicas estudadas após os métodos de reparo. G1 – reparos com a própria resina, G2- reparos com resina autopolimerizável e G3- reparos com resina fotopolimerizável.

			Métodos de reparo		
Variável dependente	**Resina acrílica**		**G1**	**G2**	**G3**
Tensão de ruptura	Lucitone 550	A	-46.48 ± 15.24 (a)α	-76.63 ± 3.52 (b) β	-76.81 ± 8.84 (b) β
	Impact 2000	A	-49.74 ± 23.23 (a)	-78.80 ± 5.27 (b)	-76.31 ± 4.19 (b)
	Impact 1500	B	-30.02 ± 16.02 (a)	-65.08 ± 14.80 (b)	-67.43 ± 5.56 (b)
	QC 20	C	5.66 ± 25.46 (a)	-21.93 ± 15.32 (a)	-42.84 ± 12.21 (b)
Módulo Young	Lucitone 550	A	-11.65 ± 5.39 (a) α	-20.31 ± 9.19 (a) β	-7.17 ± 11.88 (a) β
	Impact 2000	A	-9.96 ± 13.64 (a)	-5.69 ± 5.79 (a)	3.02 ± 11.30 (a)
	Impact 1500	A	7.42 ± 13.83 (a)	6.54 ± 3.92 (a)	10.42 ± 10.90 (a)
	QC 20	A	21.91 ± 16.22 (a)	26.43 ± 21.76 (a)	1.62 ± 8.19 (a)
Deslocamento de escoamento	Lucitone 550	A	-37.41 ± 15.29 (a)α	-68.29 ± 5.01 (b)αβ	-76.56 ± 11.49 (b) β
	Impact 2000	A	-56.07 ± 21.42 (a)	-80.69 ± 6.42 (b)	-77.17 ± 5.10 (b)
	Impact 1500	A	-46.12 ± 12.02 (a)	-78.43 ± 8.50 (b)	-79.03 ± 4.19 (b)
	QC 20	C	-14.95 ± 17.44 (a)	-46.98 ± 19.12 (b)	-69.61 ± 15.92 (b)
Resistência ao impacto	Lucitone 550	A	220.80 ± 60.02 (a)α	42.22 ± 20.29 (b) β	-22.70 ± 12.33 (b) β
	Impact 2000	B	37.09 ± 23.53 (a)	-52.58 ± 9.91 (a)	-25.55 ± 8.79 (a)
	Impact 1500	A	74.72 ± 50.28 (a)	68.05 ± 57.15 (a)	-50.01 ± 0.02 (a)
	QC 20	C	191.66 ± 58.51 (a)	5.55 ± 13.59 (b)	0.00 ± 0.00 (-)

Letras diferentes mostram as diferenças significantes. Letras maiúsculas na linha vertical mostram as diferenças entre as resinas acrílicas; letras minúsculas mostram as diferenças entre os métodos de reparo dentro de cada resina acrílica. Símbolos mostram as diferenças entre os métodos de reparo. (P<.05).

5.5 Tabela de comparações da ANOVA de duas vias do método de reparo e resina acrílica na resistência ao impacto, tensão de ruptura, Módulo Young e deslocamento de escoamento.

Variável dependente	Source of Variation	DF	SS	MS	F	P
Resistência ao impacto	Resina acrílica	3	0.506	0.169	22.495	<0.001
	Método de reparo	2	2.696	1.348	179.783	<0.001
	Resina x Método de reparo	6	0.913	0.152	20.287	<0.001
	Resíduo	60	0.450	0.008		
	Total	71	4.565	0.064		
Tensão de ruptura	Resina acrílica	3	0.085	0.028	2.079	0.113
	Método de reparo	2	1.570	0.785	57.798	<0.001
	Resina x Método de reparo	6	0.197	0.033	2.421	0.037
	Resíduo	59	0.801	0.014		
	Total	70	2.609	0.037		
Módulo Young	Resina acrílica	3	0.459	0.153	137.139	<0.001
	Método de reparo	2	0.010	0.005	4.674	0.013
	Resina x Método de reparo	6	0.016	0.003	2.346	0.042
	Resíduo	60	0.067	0.001		
	Total	71	0.552	0.008		
Deslocamento de escoamento	Resina acrílica	3	1.515	0.505	9.698	<0.001
	Método de reparo	2	7.864	3.932	75.524	<0.001
	Resina x Método de reparo	6	0.498	0.083	1.594	0.165
	Resíduo	59	3.072	0.052		
	Total	70	13.01	0.186		

5.6 Tabela de comparações da ANOVA de duas vias do percentual representativo (depois-antes) da resistência ao impacto, tensão de ruptura, Módulo Young e deslocamento de escoamento entre resinas acrílicas e métodos de reparo.

Variável dependente	Source of Variation	DF	SS	MS	F	P
Resistência ao impacto	Resina acrílica	3	10.015	3.338	44.264	<0.001
	Método de reparo	2	8.942	4.471	59.286	<0.001
	Resina x Método de reparo	6	12.526	2.088	27.680	<0.001
	Resíduo	59	4.450	0.0754		
	Total	70	36.177	0.517		
Tensão de ruptura	Resina acrílica	3	306757.373	102252.458	38.832	<0.001
	Método de reparo	2	202596.462	101298.231	38.469	<0.001
	Resina x Método de reparo	6	36583.725	6097.287	2.316	0.045
	Resíduo	60	157992.566	2633.209		
	Total	71	703930.127	9914.509		
Módulo Young	Resina acrílica	3	3.370	1.123	1.231	0.306
	Método de reparo	2	6.558	3.279	3.593	0.034
	Resina x Método de reparo	6	8.951	1.492	1.635	0.153
	Resíduo	60	54.754	0.913		
	Total	71	73.633	1.037		
Deslocamento de escoamento	Resina acrílica	3	7629.436	2543.145	15.297	<0.001
	Método de reparo	2	17928.911	8964.455	53.920	<0.001
	Resina x Método de reparo	6	1890.371	315.062	1.895	0.096
	Resíduo	60	9975.260	166.254		
	Total	71	37423.978	527.098		

5.7. Tabela de comparações da ANOVA de duas vias das diferenças representativas (depois-antes) da resistência ao impacto, tensão de ruptura, Módulo Young e deslocamento de escoamento entre resinas acrílicas e métodos de reparo.

Variável dependente	Source of Variation	DF	SS	MS	F	P
Resistência ao impacto	Resina acrílica	3	1.166	0.389	9.349	<0.001
	Método de reparo	2	1.363	0.681	16.396	<0.001
	Resina x Método de reparo	6	2.374	0.396	9.519	<0.001
	Resíduo	59	2.452	0.0416		
	Total	70	7.230	0.103		
Tensão de ruptura	Resina acrílica	3	2229.278	743.093	116.395	<0.001
	Método de reparo	2	338.739	169.370	26.529	<0.001
	Resina x Método de reparo	6	48.305	8.051	1.261	0.289
	Resíduo	60	383.054	6.384		
	Total	71	2999.376	42.245		
Módulo Young	Resina acrílica	3	480.618	160.206	4.696	0.005
	Método de reparo	2	204.012	102.006	2.990	0.058
	Resina x Método de reparo	6	308.509	51.418	1.507	0.191
	Resíduo	60	2047.085	34.118		
	Total	71	3040.224	42.820		
Deslocamento de escoamento	Resina acrílica	3	182.786	60.929	36.429	<0.001
	Método de reparo	2	86.876	43.438	25.972	<0.001
	Resina x Método de reparo	6	15.126	2.521	1.507	0.191
	Resíduo	60	100.351	1.673		
	Total	71	385.139	5.424		

Anexo 5.8 - Testes estatísticos realizados pela comparação entre os valores médios dos corpos-de-prova reparados (Programa SAS) segundo as variáveis dependentes.

Dependent Variable: RI_D_T

Source	DF	Sun of Squares	Mean Square	F Value	Pr > F
Model	11	4.11507342	0.37409758	49.89	<.0001
Error	60	0.44991963	0.00749866		
Corrected Total	71	4.56499305			

R-Square	Coeff Var	Root MSE	RI_D_T Mean
0.901441	50.81683	0.086595	0.170406

Source	DF	Type II SS	Mean Square	F Value	Pr > F
REPARO	2	2.69626750	1.34813375	179.78	<.0001
RESINA	3	0.50604305	0.16868102	22.49	<.0001
REPARO*RESINA	6	0.91276286	0.15212714	20.29	<.0001

The GLM Procedure

Tukey's Studentized Range (HSD) Test for RI_D_T

NOTE: This test controls the Type I experimentwise error rate, but it generally has a higher Type II error rate than REGWQ.

Alpha	0.05
Error Degrees of Freedom	60
Error Mean Square	0.007499
Critical Value of Studentized Range	3.39867
Minimum Significant Difference	0.0601

Means with the same letter are not significantly different.

Tukey Grouping	Mean	N	REPARO
A	0.43222	24	pp_res
B	0.10852	24	aut_po
C	-0.02952	24	foto_p

Tukey's Studentized Range (HSD) Test for RI_D_T

NOTE: This test controls the Type I experimentwise error rate, but it generally has a higher Type II error rate than REGWQ.

Alpha	0.05
Error Degrees of Freedom	60
Error Mean Square	0.007499
Critical Value of Studentized Range	3.73709
Minimum Significant Difference	0.0763

Means with the same letter are not significantly different.

Tukey Grouping	Mean	N	RESINA
A	0.28283	18	Im_2000
B	0.18405	18	Lucitone
B			
B	0.16797	18	QC20
C	0.04678	18	Im_1500

Dependent Variable: STRESS_D_T

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	11	1.80804009	0.16436728	12.10	<.0001
Error	59	0.80127378	0.01358091		
Corrected Total	70	2.60931387			

R-Square	Coeff Var	Root MSE	STRESS_D_T Mean
0.692918	8.323188	0.116537	1.400151

Source	DF	Type II SS	Mean Square	F Value	Pr > F
REPARO	2	1.55266652	0.77633326	57.16	<.0001
RESINA	3	0.07933284	0.02644428	1.95	0.1318
REPARO*RESINA	6	0.19729623	0.03288270	2.42	0.0369

The GLM Procedure

Tukey's Studentized Range (HSD) Test for STRESS_D_T

NOTE: This test controls the Type I experimentwise error rate.

Alpha	0.05
Error Degrees of Freedom	59
Error Mean Square	0.013581
Critical Value of Studentized Range	3.40013

Comparisons significant at the 0.05 level are indicated by ***.

REPARO Comparison	Difference Between Means	Simultaneous 95% Confidence Limits		
pp_res - aut_po	0.30536	0.22360	0.38711	***
pp_res - foto_p	0.32165	0.23989	0.40340	***
aut_po - pp_res	-0.30536	-0.38711	-0.22360	***
aut_po - foto_p	0.01629	-0.06459	0.09717	
foto_p - pp_res	-0.32165	-0.40340	-0.23989	
foto_p - aut_po	-0.01629	-0.09717	0.06459	

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Tukey's Studentized Range (HSD) Test for STRESS_D_T

NOTE: This test controls the Type I experimentwise error rate.

Alpha	0.05
Error Degrees of Freedom	59
Error Mean Square	0.013581
Critical Value of Studentized Range	3.73889

Comparisons significant at the 0.05 level are indicated by ***.

RESINA Comparison	Difference Between Means	Simultaneous 95% Confidence Limits	
Im_2000 - Lucitone	0.04166	-0.06254	0.14586
Im_2000 - QC20	0.06374	-0.04046	0.16794
Im_2000 - Im_1500	0.07591	-0.02829	0.18011
Lucitone - Im_2000	-0.04166	-0.14586	0.06254
Lucitone - QC20	0.02208	-0.08062	0.12478
Lucitone - Im_1500	0.03425	-0.06845	0.13695
QC20 - Im_2000	-0.06374	-0.16794	0.04046
QC20 - Lucitone	-0.02208	-0.12478	0.08062
QC20 - Im_1500	0.01217	-0.09053	0.11487
Im_1500 - Im_2000	-0.07591	-0.18011	0.02829
Im_1500 - Lucitone	-0.03425	-0.13695	0.06845
Im_1500 - QC20	-0.01217	-0.11487	0.09053

Dependent Variable: MOD_D_T

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	11	0.48534606	0.04412237	39.53	<.0001
Error	60	0.06696891	0.00111615		
Corrected Total	71	0.55231497			

R-Square	Coeff Var	Root MSE	MOD_D_T Mean
0.878749	1.032598	0.033409	3.235414

Source	DF	Type II SS	Mean Square	F Value	Pr > F
REPARO	2	0.01043281	0.00521641	4.67	0.0130
RESINA	3	0.45920196	0.15306732	137.14	<.0001
REPARO*RESINA	6	0.01571129	0.00261855	2.35	0.0422

The GLM Procedure

Tukey's Studentized Range (HSD) Test for MOD_D_T

NOTE: This test controls the Type I experimentwise error rate, but it generally has a higher Type II error rate than REGWQ.

Alpha	0.05
Error Degrees of Freedom	60
Error Mean Square	0.001116
Critical Value of Studentized Range	3.39867
Minimum Significant Difference	0.0232

Means with the same letter are not significantly different.

Tukey Grouping	Mean	N	REPARO
A	3.252432	24	foto_p
B	3.227303	24	aut_po
B			
B	3.226508	24	pp_res

Tukey's Studentized Range (HSD) Test for MOD_D_T

NOTE: This test controls the Type I experimentwise error rate, but it generally has a higher Type II error rate than REGWQ.

Alpha	0.05
Error Degrees of Freedom	60
Error Mean Square	0.001116
Critical Value of Studentized Range	3.73709
Minimum Significant Difference	0.0294

Means with the same letter are not significantly different.

Tukey Grouping	Mean	N	RESINA
A	3.32599	18	Lucitone
B	3.29525	18	Im_2000
C	3.19495	18	Im_1500
D	3.12546	18	QC20

Dependent Variable: DISP_D_T

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	11	9.93454084	0.90314008	17.35	<.0001
Error	59	3.07174583	0.05206349		
Corrected Total	70	13.00628667			

R-Square	Coeff Var	Root MSE	DISP_D_T Mean
0.763826	14.45939	0.228174	1.578035

Source	DF	Type II SS	Mean Square	F Value	Pr > F
REPARO	2	7.89092953	3.94546476	75.78	<.0001
RESINA	3	1.51463350	0.50487783	9.70	<.0001
REPARO*RESINA	6	0.49797467	0.08299578	1.59	0.1649

Tukey's Studentized Range (HSD) Test for DISP_D_T

NOTE: This test controls the Type I experimentwise error rate.

Alpha	0.05
Error Degrees of Freedom	59
Error Mean Square	0.052063
Critical Value of Studentized Range	3.40013

Comparisons significant at the 0.05 level are indicated by ***.

REPARO Comparison	Difference Between Means	Simultaneous 95% Confidence Limits		
pp_res - aut_po	0.62801	0.46794	0.78809	***
pp_res - foto_p	0.77574	0.61566	0.93582	***
aut_po - pp_res	-0.62801	-0.78809	-0.46794	***
aut_po - foto_p	0.14773	-0.01064	0.30609	
foto_p - pp_res	-0.77574	-0.93582	-0.61566	***
foto_p - aut_po	-0.14773	-0.30609	0.01064	

The GLM Procedure

Tukey's Studentized Range (HSD) Test for DISP_D_T

NOTE: This test controls the Type I experimentwise error rate.

Alpha	0.05
Error Degrees of Freedom	59
Error Mean Square	0.052063
Critical Value of Studentized Range	3.73889

Comparisons significant at the 0.05 level are indicated by ***.

RESINA Comparison	Difference Between Means	Simultaneous 95% Confidence Limits		
QC20 - Im_1500	0.14779	-0.05329	0.34888	
QC20 - Im_2000	0.24123	0.03721	0.44524	***
QC20 - Lucitone	0.40386	0.20278	0.60495	***
Im_1500 - QC20	-0.14779	-0.34888	0.05329	
Im_1500 - Im_2000	0.09343	-0.11058	0.29745	
Im_1500 - Lucitone	0.25607	0.05499	0.45715	***
Im_2000 - QC20	-0.24123	-0.44524	-0.03721	***
Im_2000 - Im_1500	-0.09343	-0.29745	0.11058	
Im_2000 - Lucitone	0.16264	-0.04138	0.36665	
Lucitone - QC20	-0.40386	-0.60495	-0.20278	***
Lucitone - Im_1500	-0.25607	-0.45715	-0.05499	***
Lucitone - Im_2000	-0.16264	-0.36665	0.04138	