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Effect of repair methods and materials on the flexural strength of 3D-printed denture base resin

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PURPOSE. The aim of this study was to evaluate the flexural strength of a 3D-printed denture base resin (Cosmos Denture), after different immediate repair techniques with surface treatments and thermocycling. **MATERIALS AND METHODS.** Rectangular 3D-printed denture base resin (Cosmos Denture) specimens (N = 130) were thermocycled (5,000 cycles, 5°C and 55°C) before and after the different repair techniques (n = 10 per group) using an autopolymerized acrylic resin (Jet, J) or a hard relining resin (Soft Confort, SC), and different surface treatments: Jet resin monomer for 180 s (MMA), blasting with aluminum oxide (JAT) or erbium: yttrium-aluminum-garnet laser (L). The control group were intact specimens. A three-point flexural strength test was performed, and data (MPa) were analyzed by ANOVA and Games-Howell post hoc test ($\alpha = 0.05$). Each failure was observed and classified through stereomicroscope images and the surface treatments were viewed by scanning electron microscope (SEM). **RESULTS.** Control group showed the highest mean of flexural strength, statistically different from the other groups ($P < .001$), followed by MMA+J group. The groups with L treatment were statistically similar to the MMA groups ($P > .05$). The JAT+J group was better than the SC and JAT+SC groups ($P < .05$), but similar to the other groups ($P > .05$). Adhesive failures were most observed in JAT groups, especially when repaired with SC. The SEM images showed surface changes for all treatments, except JAT alone. **CONCLUSION.** Denture bases fabricated with 3D-printed resin should be preferably repaired with MMA+J. SC and JAT+SC showed the worst results. Blasting impaired the adhesion of the SC resin. [J Adv Prosthodont 2022;14:305-14]

KEYWORDS

Dental prosthesis; Denture bases; Flexural strength; Printing; Three-dimensional; Resins

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INTRODUCTION

The digital method of manufacturing prostheses has the advantage of eliminating clinical steps, benefits in terms of costs and clinical time, digital archiving of data, elimination of problems related to conventional impressions, greater patient comfort, evaluation and previous adjustments of digitized images and better marginal sealing compared to conventional prostheses.[1]

Although many studies compare the subtractive digital fabrication technique (milling) of removable dentures to the conventional technique with poly(methyl)methacrylate (PMMA), few studies have evaluated the properties related to the additive fabrication technique (3D-printing).[2-4] In a study by Alghazzawi,[1] some of the advantages related to the digital additive technique of manufacturing removable protheses were highlighted, such as: more detailed reproduction; more economical than the subtractive technique; greater mass production (greater number of units); can produce larger objects (e.g. facial prosthesis); passive production, without force application; can reproduce complex shapes without the need for special cutting tools; unlimited geometry options; faster than the milling technique and prints exactly as designed with no waste.[1]

The denture bases are constantly subjected to repeated loads during mastication, in addition to being subject to falls during handling, especially because the users of these dentures are mostly elderly individuals with low manual dexterity.[5] In addition, the materials used to manufacture prosthetic bases are subject to biodegradation in the oral environment due to factors such as hydrolysis and salivary enzymes, mechanical stress due to thermal and chemical changes in the diet.[6] Due to these factors, denture base fractures can occur, and repair techniques must be employed to ensure the well-being of patients. The repair techniques should ideally be easy to perform and inexpensive and should guarantee good mechanical resistance to the repaired prosthesis.

Denture bases made with conventional resins based on PMMA can be repaired using simple techniques performed in the dental office by the dentist, mainly using self-curing acrylic resins.[7-13] The literature also reports the use of rigid resin for self-curing reline in repair areas,[8] associated with surface treatments in order to improve its bonding properties.[14] Among the most reported techniques are chemical surface treatments, such as conditioning with methyl methacrylate,[9,10,15-18] acetone,[7,9,15,19] chloroform,[8,20] ethyl acetate,[21] and mechanical treatments, such as surface roughening with abrasive sandpaper,[10,17] aluminum oxide particles Al_2O_3,[8,13,18,22,23] or surface treatment with a high power laser.[13,22-24] Repair techniques with reinforcement materials are also used, such as fiberglass reinforcement,[25,26] reinforcement from renewable sources such as cellulose crystals[27] and recently the incorporation of nanocomposites.[18,28,29]

Considering the problem of fatigue of denture base materials and the limited literature regarding the repair of denture bases obtained by 3D-printing, with the exception of the study by Li *et al*.[30] and Neshandar Asli *et al*.,[31] little is known about the mechanical properties of the materials used in additive technique for making removable prostheses and their repair possibilities in research.[3] Therefore, the aim of this study was to evaluate the flexural strength of a denture base resin obtained through the digital additive manufacture technique (3D-printing) subjected to different repair techniques. The null hypothesis of the study is that there will be no difference on the flexural strength, irrespective of the repair technique.

MATERIALS AND METHODS

A 3D-printed type of resin (Cosmos Denture; Yller Digital, Pelotas, RS, Brazil) was used to simulate the denture base and two types of resins were used for the repair, an autopolymerized resin (Jet; Clássico, Artigos Odontológicos Ltda., São Paulo, SP, Brazil) and an autopolymerized hard reline resin (Soft Confort; Dencril Produtos Odontológicos, Vipi Ltda, Pirassununga, SP, Brazil). Thirteen groups (n = 10) were formed, according to the repair technique: control, intact Cosmos Denture resin (C); autopolymerized acrylic resin Jet (J), hard relining resin Soft Confort (SC), Jet resin monomer for 180 s + J (MMA + J), Jet resin monomer for 180 s + SC (MMA + SC) , blasting with aluminum oxide for 15 s + J (JAT + J), blasting with aluminum oxide for 15 s + SC (JAT + SC), erbium: yttrium-aluminum-garnet laser for 60 s + J (L + J), erbium: yttrium-aluminum-garnet laser for 60 s + SC (L + SC), blasting with aluminum oxide for 15 s + Jet resin monomer for 180 s + J (JAT + MMA + J), blasting with aluminum oxide for 15 s + Jet resin monomer for 180 s + SC (JAT + MMA + SC), erbium: yttrium-aluminum-garnet laser for 60 s + Jet resin monomer for 180 s + J (L + MMA + J), and erbium: yttrium-aluminum-garnet laser for 60 s + Jet resin monomer for 180 s + SC (L + MMA + SC) (Fig. 1).

Rectangular specimens (N = 130, 64 mm × 10 mm × 3.3 mm) were virtually designed with the Adobe Meshmixer v. 3.5 Software (Autodesk Inc.; San Rafael, CA, USA) (Fig. 2A) and the file was converted to a .stl file. The virtual specimens were transferred to the FlashDLPrint v. 3.28.0 (Zhejiang Flashforge3D technology, Jinhua, Zhejiang, China), and the printing layer orientation was stablished at 0 degrees to the z-axis direction without support (Fig. 2B). The Cosmos Denture resin specimens were printed on Flashforge Hunter DLP Resin 3D Printer (Zhejiang Flashforge3D technology, Jinhua, Zhejiang, China) (Fig. 2C). The curing process was activated by an ultraviolet light (LED, λ = 405 nm) with a layer thickness of 50 µm.

After printing, the specimens were cleaned with 99% isopropyl alcohol for 5 min and an additional curing with an UV light curing unit (LED, λ = 405 nm) (Curing Box; dOne 3D, Ribeirão Preto, SP, Brazil) was performed for 10 min, according to the resin manufacturer's recommendations.

All specimens were finished with 400-grit sandpaper in an Aropol 2V polishing machine (Arotec S/A Indústria e Comércio, Cotia, SP, Brazil) and polished with 600-grit sandpaper. After polishing, all specimens (N = 130) were stored in distilled water at 37°C for 50 ± 2 hours, according to ISO/FDIS 1567.[32]

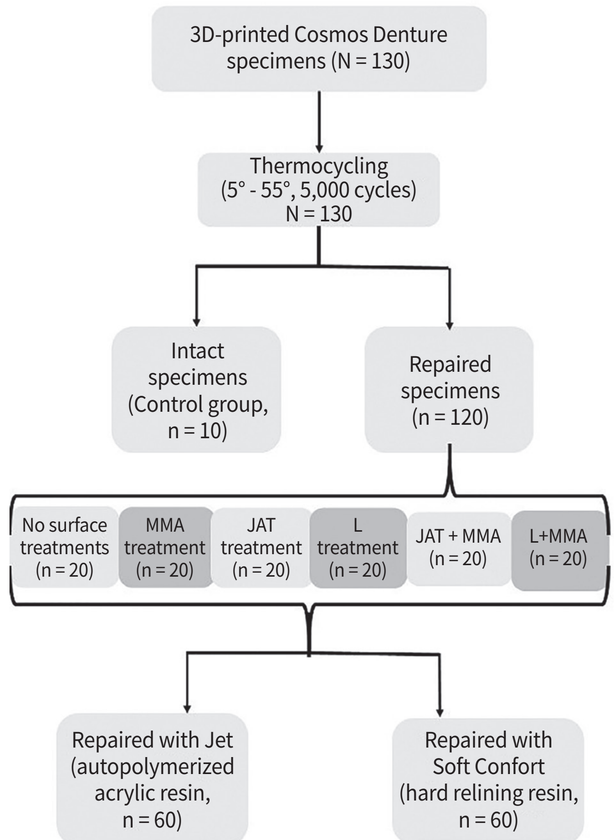

Fig. 1. Description of groups. MMA: methyl methacrylate monomer of the Jet resin for 180 s; JAT: blasting with aluminum oxide 50 µm for 15 s; L: application of an erbium: yttrium-aluminum-garnet laser for 60 s.

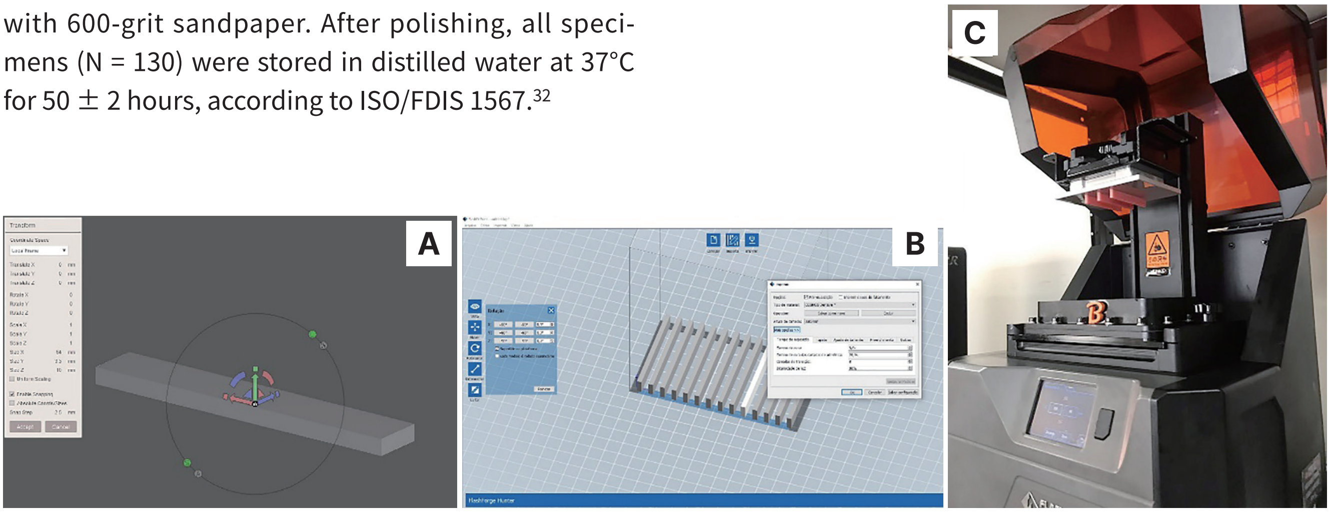

Fig. 2. (A) Layout specimens planned on Adobe Meshmixer. (B) Software FlashDLPrint v. 3.28.0, printing orientation stablished in 0 degrees without support. (C) Flashforge printer - Hunter DLP Resin 3D Printer.

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All specimens (N = 130) were submitted to thermocycling in a thermal cycle simulation machine - model MSCT - 3 (Marcelo Nucci - ME, São Carlos, SP, Brazil) to perform 5,000 cycles,[33,34] with temperature varying between 5°C and 55°C[33-36] and immersion time of 30 seconds in each bath.[35,37-39] This step aimed to clinically simulate a situation of a denture base already submitted to thermal aging due to the use of the denture for a period of five years.

The specimens of the experimental groups were marked at the center and another two marks were made 1 mm to the left from the center and 1 mm from the right, creating a 2 mm area of resin to be removed for the repair purpose. Then, a tape was positioned evolving the specimen on the left mark and on the right mark, leaving just the 2 mm center resin to be removed by sectioning using a diamond double-sided metal disc (KG Sorensen, Cotia, SP, Brazil) mounted on a hand piece, perpendicularly to the long axis, creating a 2 mm repair surface parallel to each other. For all specimens, the same operator manually performed this procedure.

The sectioned specimens were accommodated in rectangular metallic molds to receive the following repair techniques: repair with Jet resin (J) or Soft Confort resin (SC) without surface treatment. In the groups that received chemical surface treatment with the methyl methacrylate monomer of the Jet resin (MMA), the MMA was applied for 180 s.[10,16,40] For the standardization of the application, two drops of MMA were applied with a dropper, every 60 s on the repair surfaces and rubbed with a flexible cotton swab (Flexicotton Industria e Comércio de Produtos de Hygiene Toiletries S/A; Santo Amaro da Imperatriz, SC, Brazil), and then the groups were repaired with Jet (MMA + J) or Soft Confort (MMA + SC).

For the groups that received mechanical treatment with blasting with aluminum oxide 50 µm[8,18,23] for 15 s[41] with a pressure of 0.28 MPa[41] and a distance of 10 mm[13,18,23,41] between the repair surface and the blaster tip (Basic Classic; Renfert GmbH, Hilzingen, Germany), the loose particles were removed with a blast of air, the surfaces were washed for 10 s with a water/air spray free of oil for 3 s,[18] and then repairs were made with Jet (JAT + J) or Soft Confort (JAT+SC). The laser (L) groups received the application of an erbium: yttrium-aluminum-garnet laser (Er:YAG) (Fotona 3 Medical Lasers, Stegne, Ljubljana, Slovenia, EU) with a wavelength of 2940 nm, at a pulse frequency of 10 Hz, a pulse energy of 250 mJ, pulse duration of 100 µs,[13,23,24] for 60 seconds under water and air irrigation (setting number 6), with a distance of 7 mm between the laser tip and the surface to be repaired of the specimen.[13,23,24] After laser application, the repair was made with Jet resin (L + J) or Soft Confort (L + SC). For the groups that received the application of blasting plus monomer and laser plus monomer, the treatment techniques were the same as described, with the specimens receiving the mechanical treatment JAT or L, followed by the chemical treatment with MMA, and then the repairs were made with Jet (JAT + MMA + J and L + MMA + J) or Soft Confort (JAT + MMA + SC and L + MMA + SC).

The Jet and Soft Confort resins were mixed in a 3:1 powder/liquid ratio, accommodated in the respective repair areas with the specimens already positioned in the metallic molds fixed on a thick glass plate. A second glass plate was placed over the set and taken to a hydraulic press for 10 min at a temperature of 25° C, according to the respective manufacturer's instructions. Then, the specimens were removed from the metal molds and placed under pressure (EDG pressure chamber; EDG Equipamentos e Controles Ltda, São Carlos, SP, Brazil) at 0.2 MPa at 55°C for 15 min to complete the polymerization.[7,11,16] After the repair techniques, the repaired specimens were finished with 400 and 600 grit sandpaper and were stored in distilled water for 50 ± 2 hours at 37°C.[30]

All specimens (N = 130), intact (control) and experimental groups, were subjected to a new cycle of thermocycling of 5,000 cycles,[33,34] with temperature varying between 5°C and 55°C[33-36] and immersion time of 30 seconds in each bath.[35,37-39] This step aims to simulate a clinical situation of a denture base subjected to thermal aging for 5 years after the repair technique, which allows evaluating the longevity of the repair techniques.

A three-point flexural test was performed with a mechanical testing machine (model EMIC-DL 3000; EMIC Equipamentos e Sistemas de Ensaios Ltda, Curitiba, SP, Brazil) at a cross-head speed of 5 mm/min with a 200 Kgf load cell, applied in the center of specimens where the repair was made, until fracture. The maximum load register was used to calculate the flexural strength (MPa) using the following formula:[42,43]

$$FS = 3WL / 2.bh^2$$

Where FS = flexural strength (MPa), W = load at fracture (N), L = distance between supporting wedges (52.5 mm); b = width of the specimen (mm) and h = thickness of the specimen (mm).

For all the repaired fractured specimens, images were captured using a stereomicroscope (model M80; Leica Microsystems Ltd., Heerbrugg, Switzerland) at 1.25× magnification to identify the nature of failure: adhesive, when failure occurred at the interface between repair resin and 3D-printed resin; cohesive, if more than 75% of failure occurred within the 3D-printed resin or in the repair resin; or mixed when the failure refers to both occurring simultaneously.[44,45]

To evaluate the effects of the treatments on the repair surface of the 3D-printed denture base resin, additional Cosmos Denture specimens (n = 6) were printed, thermocycled, sectioned in half as described above and the repair surfaces were submitted to: no treatment, MMA, JAT, L, JAT + MMA and L + MMA treatments. These specimens were gold-sputtered and visually inspected at the repair surface under a field emission SEM (model JSM - 6610; Jeol, Tokyo, Japan) with a voltage acceleration of 12.0 kV. Photomicrographs were taken under a magnification of ×55 and ×190.[13,23,46]

Flexural strength data presented normal distribution (Shapiro-Wilk, $P > .05$) and heterocedasticity (Levene, $P < .05$). The means (MPa) were submitted to one-way analysis of variance with Welch's correction and Games-Howell post hoc test, with a significance level of 5%. The nature of failure was presented as a descriptive analysis. The statistical analyses were performed with IBM SPSS statistical software for Windows (SPPS v15.0; SPSS Inc., Chicago, IL, USA).

RESULTS

The one-way ANOVA with Welch's correction (Table 1 and Table 2) showed the significant effect of the different techniques ($P < .001$). Figure 3 identifies the multiple comparisons among the groups (Games-Howell post-test). The highest means of flexural strength were observed in the control group, statistically significant to the other groups (36.8 ± 6.4 MPa, $P < .001$). The comparison among the repaired groups showed that the group MMA + J had the highest flexural strength (15.5 ± 3.4 MPa), followed by the JAT + MMA + J (14.5 ± 2.2 MPa), showing the similar flexural strength. The groups that showed the lowest values of flexural strength was the SC (6.7 ± 1.0 MPa), JAT + SC (7.0 ± 1.2 MPa) and JAT + MMA + SC (9.0 ± 2.1 MPa), with no difference among these groups ($P > .05$).

Table 1. One-way analysis of variance test (α = .05)

	S S	Df	M S	Z	Sig.
Between Groups	6868.865	12	572.405	76.860	< .001
Within Groups	871.344	117	7.447		
Total	7740.209	129			

* Significance $p < .05$.

Table 2. Welch's correction, robust test of equality of means (α = .05)

	Estatistic	df1	df2	Sig.
Welch	36.144	12	45.305	< .001

* Significance $p < .05$.

Figure 4 illustrates the descriptive analysis of the failure, according to the group. The groups SC, JAT + SC and JAT + MMA + SC presented 100% of adhesive failure. For the groups repaired with J, MMA + J, L + J, JAT + MMA + J, L + MMA + J and L + MMA + SC, the prevalence of failure was cohesive within the 3D-printed denture base resin.

The SEM photomicrographs (Fig. 5) showed that all treatments resulted in noticeable surface modifications, less remarkable for the JAT group.

DISCUSSION

Denture bases are susceptible to fractures due to falls or as a result of material degradation over time, which is constantly subjected to chewing loads, temperature and pH fluctuations in the oral environment. In this context, information about the longevity of different repair techniques of a 3D-printed denture base resin becomes relevant. The null hypothesis that there would be no difference on the flexural strength among the different repair techniques was rejected.

Flexural strength is a suitable property to predict

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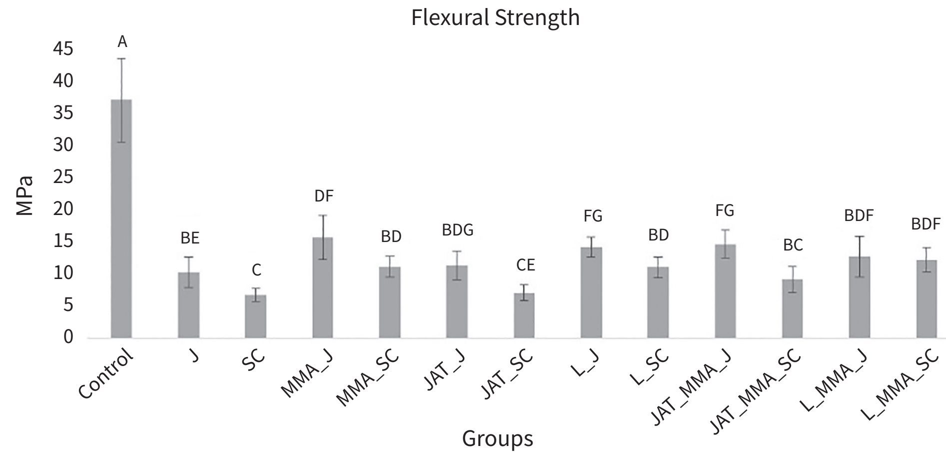

Fig. 3. Means of flexural strength (MPa) and standard deviations, according to the group (Games-Howell test, α = 0.05). Similar capital letters represent statistical similarity between different groups.

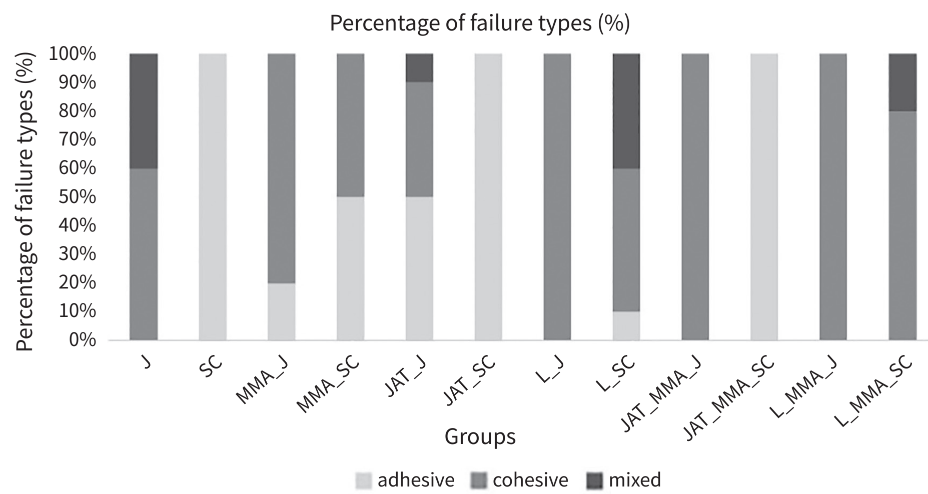

Fig. 4. Percentage of types of failure, according to the group.

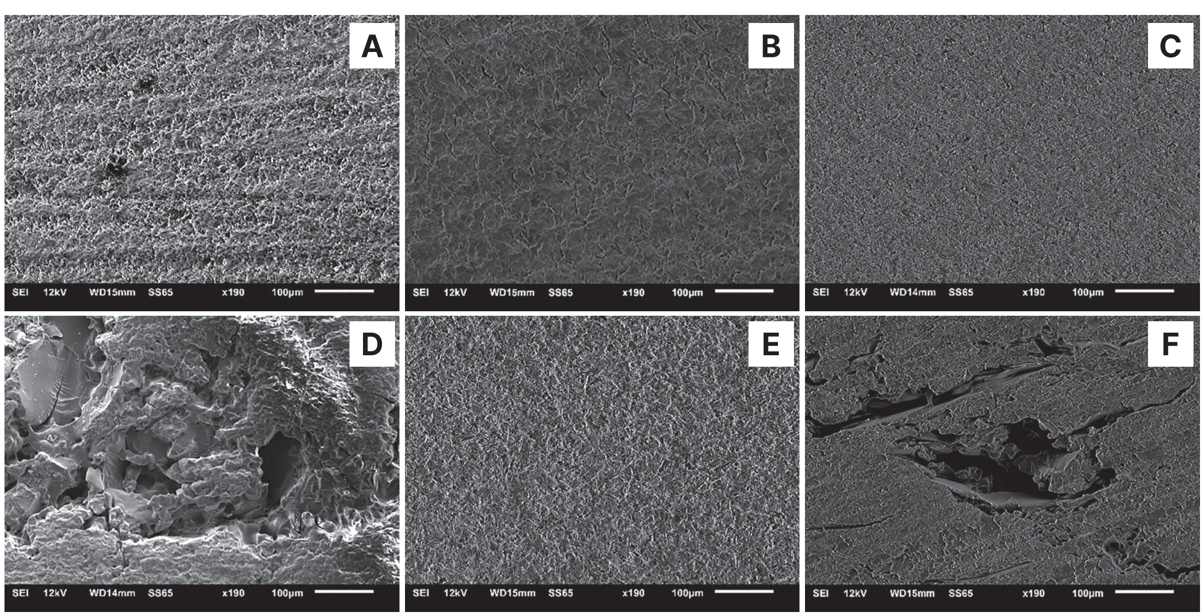

Fig. 5. SEM photomicrographs of surface treatments with 12 kV and magnification of × 190. (A) no treatment, × 190; (B) Monomer for 180 s, × 190; (C) Blasting, × 190; (D) Laser, × 190; (E) Blasting + monomer, × 190; (F) Laser + monomer, × 190.

the mechanical resistance of a denture base material. It was standardized by ISO/FDIS 1567 that a conventional denture base resin intact and not subjected to thermal aging should reach at least 65 MPa.[32] When submitted to repairs, the flexural strength of conventional denture base resins (thermopolymerized in a water bath or by microwave energy) varies according to the repair technique. Arioli Filho *et al*.[11] observed an average flexural strength of 6.7 MPa when repairing a water-bath polymerized resin with an autopolymerized resin without any surface treatment and without thermal aging. In the present study, the 3D-printed specimens were thermocycled before and after the repair and reached flexural strength of 10.2 MPa when an autopolymerized resin was used without any surface treatment (group J).

The repair flexural strength depends on good adhesion between the repair material and the base resin.[41] The groups that showed the highest flexural strength means had the autopolymerized resin Jet as the repair material, and the surface treatment with methyl methacrylate monomer for 180 s significantly improved the flexural strength. According to Vallitu *et al*.,[16] the application of the monomer is capable of dissolving a resin based on PMMA; this dissolution allows new polymer chains to be chemically formed at the bonding interface when repairing this PMMA resin with an autopolymerized resin.

Neshandar Asli *et al*.[31] recently evaluated the roughness and flexural strength of a repaired 3D-printed denture resin after different mechanical surface treatments: laser Er:YAG, blasting with 250 µm aluminum oxide particles, and bur grinding. The authors did not evaluate any chemical surface treatment and they found out that the bur grinding treatment provided the highest flexural strength (58.4 ± 2.4 MPa) in comparison with the others treatments. The present study did not show a flexural strength as high as those found by Neshandar Asli *et al*.[31] in any of the groups, although the treatment methods were different among mechanical treatments with laser and blasting, and the chemical treatment with an autopolymerized monomer alone and combined with mechanical treatment. The higher flexural strength in the treatment groups was observed on MMA + J (15.5 ± 3.4 MPa) and it should be taken into account that all of the specimens were thermocycled by 10,000 cycles.

The findings of this study also demonstrated that, in general, repairing a 3D-printed denture base resin with the hard reline resin Soft Confort (SC) resulted in the lowest flexural strength values, which may have occurred due to the incompatibility of adhesion between the SC and the 3D-printed denture base resin (Cosmos Denture). Soft Confort resin is composed of an ethylmethacrylate polymer powder and an iso-butylmethacrylate monomer liquid, whereas Cosmos Denture resin is composed of oligomers, monomers (acrylates and methacrylates), photoinitiators, stabilizers and pigment (manufacturer's information). The evaluation of the type of failure corroborates with these findings since a higher percentage of adhesive failure was found in the groups SC, JAT + SC, and JAT + MMA + SC, which are in agreement with the study by Alkurt *et al*.[13]

The images of SEM displayed irregularities on the surface repair submitted to laser application, which could explain the improvement of the flexural strength for the specimens repaired with Jet resin. This surface change may have favored the adhesion of the Jet resin, allowing it to flow and enter the irregularities formed on the repair surface. According to Sari *et al*.,[46] the laser promotes a surface melting of the resin by increasing the temperature in the areas where it was applied.

The results of this study also demonstrated that laser application had a similar effect to MMA conditioning on the flexural strength data. These findings are in agreement with the study by Alkurt *et al*.,[13] which found similarity on the flexural strength of a denture base resin when comparing laser treatment and MMA application followed by repair with an autopolymerized resin.

Blasting was insufficient to promote surface changes, as demonstrated by SEM images. For the JAT + SC group, blasting impaired the flexural strength, as reported by Gundogdu *et al*.,[23] and the flexural strength was similar to those that of the repaired group with SC and J without treatment. In contrast, Alkurt *et al*.[13] and Neshandar Asli *et al*.[31] found a significant improvement on the flexural strength of a denture base resin previously blasted with 250 µm aluminum oxide and repaired with an autopolymerized resin. The size of the aluminum oxide particles might explain these contrasting results.

For the MMA treatment, SEM images showed a noticeable surface modification with more exposure of the resin matrix, which might imply that more carbon chains are free to make new bonds with the repair material. In contrast, Li *et al*.[30] observed that wetting the surface of a 3D-printed denture base resin with MMA did not promote visible morphological changes on the surface of the 3D-printed resin.

In the present study, the specimens were submitted to thermocycling to simulate the ageing of the material for five years before the repair and after this procedure. It is well-known that thermocycling impact the flexural strength of denture base materials[44,47] due to plasticizer effect of the water on the bond interface, promoting a solubilization and deterioration by hydrolysis of the methacrylate groups. In addition, thermocycling promotes cyclic stress due to the water sorption and the different thermal expansion coefficients of the materials used in the repair techniques and the denture base.[48,49]

The 3D-printing technology applied to prosthetic dentistry opens several possibilities for the development of new researches in clinical and laboratory areas, since the biological, mechanical, physical and chemical behavior of these materials are still being investigated. It has been shown that any changes made in the process of manufacturing this 3D specimen can change its properties.[30,31,49-51]

The results of this study suggest the longevity of a 3D-printed denture base resin repaired under different conditions, as a clinical condition of thermal aging was simulated before and after repair. It should be considered that other clinical factors are involved in the failure of materials in the oral environment, such as hydrolysis by salivary enzymes, pH, diet and masticatory loads. Furthermore, specimens had a different shape from a denture base, just as a denture is subjected to a variety of loads in addition to the transverse resistance load.[6]

CONCLUSION

Within the limitations of this study, it was possible to conclude that none of the repairs was able to achieve the flexural strength of the intact 3D resin. For the repair of the denture bases fabricated with 3D-printed resin, an autopolymerized resin in the repair area associated with the chemical surface treatment with monomer should be considered. The repair with hard relining resin did not show good flexural strength results when used as a repair material alone without blasting. Blasting the surface with aluminum oxide did not improve the bond between the 3D-printed denture base resin and repair resin, showing a higher percentage of adhesive failures when associated with the repair made with hard relining resin.

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