

# A comparative study of flexural strength using two composite resins fiber reinforced

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## ABSTRACT

The purpose of this study was to evaluate the mechanical properties of two composite resins Charisma (Heraeus Kulzer) and Permalute (Ultradent Products Inc.) and hence to verify the influence of a fiber addition Ribbond (Ribbond Inc.) in the flexural strength of such composite resins. Fifty-two patterns were prepared in a stainless steel template with a rectangular base in which another stainless-steel plate with nine milled spaces, measuring 15 mm length; 2 mm width and 1 mm thickness, was screwed. The patterns were divided in four groups (n=13). For Group I only Charisma; Group II Charisma + Ribbond; Group III only Permalute and Group IV Permalute + Ribbond. To all samples the three-point compression test in a MTS 810 apparatus was applied. The flexural strength until rupture was obtained and SEM analysed. Data was submitted to the non-parametric 1-way ANOVA, and Tukey test (5%). Afterwards the Levene test was applied at 5% value. The results showed that Group I obtained the mean value of 164,71 MPa (mean-SD = 21,49%); for Group II 292,28 MPa (mean-SD = 34,24%); for Group III 106,50 MPa (mean-SD 24,02%) and for Group IV 61,70 MPa (mean-SD = 24,92%). The results showed that the interlocked fibers adds greater resistance to the set, being better for

Charisma+Ribbond followed by Permalute+Ribbond. The M.E.V. analysis showed, however, that no chemical

bond occurred between the fibers and the composite resins.

## UNITERMS

Flexural strength; composite resins; interlocked fibers; three-point compression test.

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## RESUMO

A finalidade deste estudo foi de avaliar as propriedades mecânicas de duas resinas compostas Charisma (Heraeus Kulzer, Alemanha) e Permalute (Ultradent Products Inc.) e verificar a influência da adição de uma fibra (Ribbond, Ribbond Inc.) na resistência à flexão das duas resinas. Foram preparados 52

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corpos-de-prova num dispositivo de aço inoxidável com base retangular, sobre a qual é parafusada uma placa também em aço inoxidável com nove espaços usinados medindo 15mm de comprimento, 2mm de largura e 1mm de espessura, sobre os quais se aplicou as resinas compostas associadas ou não a fibras. Os corpos-de-prova foram divididos em quatro grupos (n=13), sendo o Grupo I somente de resina composta Charisma; Grupo II resina Charisma+Ribbond; Grupo III somente de resina Permalute e Grupo IV de resina Permalute+Ribbond. As amostras foram submetidas ao ensaio de compressão de três pontos para verificar a resistência flexural em um aparelho MTS-810 até à ruptura das mesmas e então avaliadas sob microscopia eletrônica de varredura. Os dados foram submetidos aos testes paramétricos 1-way ANOVA, análise de variância e de Comparação Múltipla de Tukey (5%), após o teste de homogeneidade de variância, teste de Levene, sob nível de significância de 5%. As amostras do Grupo I obtiveram média de 164,71 MPa (d.p. = 21,49%); para o Grupo II, a média de ruptura foi de 292,28 MPa (d.p. = 34,24%); para o Grupo III a média foi de 106,50 MPa (d.p. = 24,02%) e para o Grupo IV os valores médios foram 61,70 MPa (d.p. = 24,92%). Os resultados demonstram que a adição de fibras entrelaçadas aumentou consideravelmente a resistência à flexão, sendo melhor para Charisma+Ribbond do que Permalute+Ribbond. Entretanto, pela análise da região fraturada empregando-se o M.E.V., concluímos que não ocorreu reação química entre as resinas compostas e as fibras, embora não apresente completa separação entre a matriz e as fibras.

## UNITERMOS

Resistência flexural; resinas compostas; fibras entrelaçadas; teste de compressão de três pontos.

## INTRODUCTION

With the introduction of composite resins in the last two decades there was a great revolution on the restorative dentistry, and also in the replacement of dental elements, as well as in their stabilization for a long lasting period. The composite resin itself failed to maintain an adequate bonding of the pontic element with the supporting structures, a fact that led dental clinicians and researchers to investigate a possible impregnation of reinforcing materials in the composite resin. The first experiences were made using metal wires embedded in composite resins, but the material always fractured in the metal-resin interface. Other materials were also embedded in the composite resin, such as metal mesh, nylon mesh, polyethylene crossed tape, carbon fibers, straight or interlocked glass fibers and Kevlar (Du Pont).

The use of fibers has increased in several fields, such as the fabrication of airplanes, boats and automobiles 4,5,9. Therefore it could not be different in Dentistry and thus we have been observing a greater employment of these materials, with many applications in the infrastructure of bonded and conventional fixed partial prostheses. In intra root retentions and in complete dentures<sup>13</sup>, usually associated with resins. The latter can be pre-impregnated or not, and with laboratory or clinical use<sup>2,3</sup>. The employment of fiber reinforced composite resins has been extensively used as tooth contentions in the periodontal disease, and after a long orthodontic treatment, but in bonded and conventional fixed prostheses they were only indicated for long lasting temporary elements. The temporary restoration is an important step that comes before the final cementation of any fixed prostheses. Besides providing a protection of the pulp-chamber complex, reestablishing occlusion, aesthetics and phonetics, it is also important in the periodontal evaluation of the supporting teeth of futures fixed partial prostheses.

The great number of fibers that can be found in the market extended the indication of fiber reinforced composite resins, as well as the indication of fixed prostheses with greater longevity. A study of the mechanical properties regarding composite resins reinforced with fibers will favor the employment of these materials with long-lasting purposes, and hence it becomes of great interest to verify the influence of a fiber addition in the flexural strength of composite resins.

In 1990, Malquarti et al.<sup>9</sup> studied the use of carbon fiber reinforced epoxy resin in aesthetic crowns and fixed partial prostheses, with the purpose of using this material as a substitute for metal bases in removable and fixed partial prostheses, thus eliminating the problem of corrosion, allergy, toxicity and casting. After a 3-year study of DGEBA-DDM polyepoxy resin and carbon fibers in single crowns, fixed partial prostheses and contention bars, the author observed no alteration in the clinical performance of this material regarding the adjacent tissue, which were free of gingival inflammation. The author concluded that the carbon fibers had satisfactory flexural and tensile strength, as well as good biological properties, as long as it is an inert material.

In 1994, Powell et al.<sup>10</sup> compared the 0.036inch steel wire with polyamida Kevlar 49 fibers when used as a reinforcement of temporary partial prostheses of four elements and two pontics, submitted to a three point load. They concluded that there was no significant difference between the loads that created the initial fracture of the temporary, and that it depended solely on the resin tensile strength, regardless of the presence, localization or type of reinforcement. The authors also concluded that the steel wire presents greater immediate resistance to the load applied right after the fracture, a characteristic that was not observed with the Kevlar fibers. Each individual fiber did not equally resist to the load as in the initial moment of the resin fracture, probably due to the fact that the fibers are twisted together, or that there was some dislodgement of the Kevlar inside the polymethylmethacrylate.

In the same year, Viguie et al.<sup>16</sup> mentioned three types of fibers found for dental use: short and randomly distributed, long and unidirectional and interlocked. According to the authors, the mechanical function at which the prostheses will be submitted will guide the type of fiber to be chosen. The elastic modulus and resistance of the material is greater as long as a higher content of fibers is used. In fixed prostheses the interlocked fibers must be associated with longitudinal fibers, which present the best mechanical properties among the three types.

David S. Hornbrook and James H. Hastings<sup>8</sup>, in 1995, studied the use of fibers in endodontically treated teeth as a retention for crowns and fixed partial prostheses. According to these authors, a considerable advantage of the fabricated post is that it is non-rotational, passive and theoretically, able to block any possible root micro fracture not yet spread to the periodontium. The reinforcing fiber tensile strength, the flexibility and strength of the resin fiber/polyethylene set, and the high strength of the bonding provided by last generation adhesive systems allows the reconstruction of endodontically treated teeth, with the result of a flexible and strong fiber bonds to high viscosity resins. The relationship between the mechanical properties of fibers and the benefits for the root preservation are not well established yet.

In 1997, Ashkan Samadzadeh<sup>12</sup> studied the fracture resistance of a polymethylmethacrylate temporary restoration (Coldpac) and a dual-cure temporary restorative material (Provipont DC), whether reinforced or not with interlocked polyethylene fibers, and treated with plasma (Ribbond, Ribbond Inc. USA). It was concluded that polymethylmethacrylate restorations reinforced with polyethylene treated with plasma showed no significant increase in the fracture strength when compared to the non-reinforced restorations, whereas the restorations reinforced with resin revealed significantly higher fracture strength than the non-reinforced polymethylmethacrylate reinforced restorations. Regarding the Providont D material, the Ribbond reinforcement is efficient in increasing the fracture strength. The reinforcement with Ribbond alters the mechanism of fracture of the Provipont DC under a compressive load from a catastrophic failure (the complete separation of the set occurs in a partial fracture pattern in which the pontic-retainer connection remains intact) to a partial separation, thus leaving the set in a single piece. Hence, the authors concluded that the use of interlocked polyethylene fibers treated with plasma is an effective method for the internal reinforcement of temporary partial fixed prostheses.

P. K. Vallittu<sup>14</sup> (1998) studied the use of glass fibers as a reinforcement in temporary partial prostheses to determine the necessary load to obtain a fracture on a three-element temporary fixed partial prostheses. He used a n-butylmethacrylate monomer polymethylmethacrylate to prepare temporary prostheses and compared the same material reinforced with glass fibers of one, two or three unidirectional or interlocked fibers. According to the author, in spite of the limitations found in his study, it could be concluded that the use of glass fibers as a reinforcement of temporary partial prostheses considerably increased the fracture strength, although the adequate position of the glass fibers was not obtained in terms of physical properties of the materials. The use of an interlocked fiber as reinforcement avoids the crown fracture.

In 1999, Göhring et al<sup>6</sup> studied 20 fixed partial prostheses in 15 patients, and reported no fracture in the surfacing resin visible clinically or

under a scanning electron microscope after one year of masticatory function. There was moderate formation of bacterial biofilm after a 1-year clinical examination.

Uzun et al.<sup>13</sup> emphasized that besides increasing the resistance to impact in complete dentures, when a fracture occurred, the fragments remained close, thus facilitating the repair.

Vallittu<sup>15</sup> also studied the flexural properties of acrylic resin polymers reinforced with unidirectional interlocked glass fibers in temporary fixed partial prostheses and denture bases made with self- and heat-curing resin, and verified that the major problem is the impregnation of the fibers with the resin. The inadequate impregnation of the fibers mainly occurs with heat-curing polymers, which form a high viscosity and poor wettability step, a fact that does not occur with self-curing resins. However, some areas present spaces between the fibers, which provoke a considerable reduction on the tensile strength and elastic modulus of the resin. Theoretically, with the reduction in viscosity there should be an increase in the fiber impregnation with resin, but it was proved that the higher content of monomers in the mixture increases the polymerization contraction of the resin, leading to a gap between the fibers and the matrix. When compared to a metal reinforcement, we verify a slight distortion provoked by the polymerization contraction of the resin inside the reinforcing fiber. The greater water absorption is an additional problem in poorly impregnated areas, as well as the discoloration that occurs as a result of microorganism penetration between the empty spaces of the fibers. It is proved that the spaces are oxygen reserves which inhibit the polymerization radicals of the acrylic resin, leading to a reduction on the fiber strength.

Another factor that should be taken into consideration regarding the fiber strength is their quantity inside the matrix, orientation and bonding to the matrix. The silanated glass fibers have been an option due to the better bond to the polymer matrix as opposed to the fibers treated with plasma (Ribbond). The bonding of the fibers to the resin matrix is an important factor under a clinical point of view, because it highly affects the composite strength.

The evolution on techniques and the use of fiber reinforced composite resins can not be solely evaluated. The search for new materials was stimulated with the development of more conservative preparations, thus preserving noble structures of enamel and dentin. Systems in which there are fibers with different characteristics depending on the mechanical necessities of each clinical situation have been introduced in the market with the purpose of overcoming the deficiencies regarding mechanical properties. Hence, clinical and laboratory investigations are necessary to allow the indication of these reconstruction systems, also enhancing their longevity.

## **MATERIAL AND METHODS**

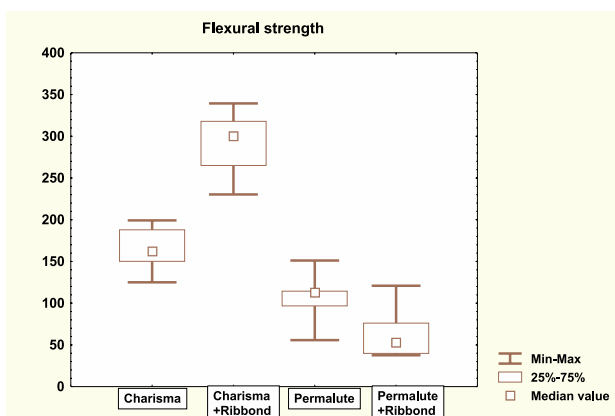
To prepare the specimens in this study we used a stainless-steel template with a rectangular base in which another stainless-steel plate with nine milled spaces was screwed. These spaces had 15mm of length, 2mm of width and 1mm of thickness, and were filled with composite resin either reinforced or non-reinforced with fibers, as it can be seen in Figure 3.

Fifty-two specimens were divided in four groups (n=13). For Group I, specimens consisted in hybrid composite resin (Charisma, Heraeus Kulzer, Germany) condensed with a resin insertion spatula (#12, Thompson, USA), and light-cured using an Optilux 400 unit (Demetron) for 40 s. For Group II, a stripe of interlocked fiber (Ribbond, Ribbond Inc, USA) with 13mm of diameter was initially applied to the template base using a bonding agent (Denthadesive, Heraeus Kulzer, Germany), which was then light-cured for 20s. After that, the hybrid composite resin was placed as described for Group I until the total filling of the milled space was achieved, then being light-cured for 40s. For Group III it was used only the hybrid composite resin (Permalut, Ultradent Products, Inc.), inserted with "TwoSpence" syringe provided by the manufacturer, light cured for 40s. For Group IV a stripe of interlocked fiber (Ribbond, Ribbond Inc. USA), with 13mm width was applied to the template base using a bonding agent (PermaQuick, Ultradent Products, Inc.), light cured for 5s and afterwards the dual resin Permalute was applied

with "TwoSpence" syringe, which was also light cured for 20s. Finally, samples were submitted to the three-point compression test in a MTS-810 apparatus to evaluate the flexural strength until rupture was obtained, and also analyzed under a scanning electron microscope (SEM). The data were submitted to the parametric test, 1-way ANOVA and Tukey test (5%).

## RESULTS

The specimens of Group I obtained a mean flexural strength value of 164,71 MPa (d.p. = 21,49%); for Group II, the mean value up to the rupture was 292,28 MPa (d.p.= 34,24%); for Group III, was 106,50 MPa (d.p. = 24,02%) and for Group IV 61,70 MPa (d.p. = 24,92%). In Figures 1 and 2 it can be observed a different behaviour between the composite resin material, this is, Charisma+Ribbond had a higher flexural strength than Permalute+Ribbond. In terms of mean values, Charisma+Ribbond showed a mean value superior to all other groups (292,28 MPa), whilst Permalute+Ribbond showed a lower value (61,70 MPa) (Figure 2).

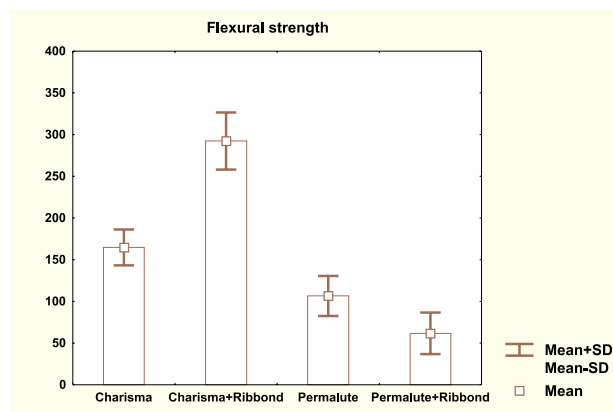


**FIGURE 1** - Box plot referring to flexural strength values (MPa) according to the composite material.

From the Figures above it is outstanding the variability, this is, the mean SD rate value is in between 21,49 MPa - 34,29 MPa. Such difference has no significance in the Levene test ( $P = 0,369$ ). The mean values when compared by the ANOVA (one-way) show that there is a statistical significant difference ( $f_{3,48} = 183,56$ ;  $p = \text{value} = 0,0001$ ) and the Tukey test (5%) showed that the materials have statistical difference when comparing one with another. However, the scanning electron microscope (SEM) analysis demonstrated that a chemical bonding between the composite resin and the fiber did not occur, and the presence of incorporated air blisters was also observed in this interface.

## DISCUSSION

The results obtained in this study demonstrated that the addition of interlocked fibers considerably increased the flexural strength of composite resins when compared to the non-reinforced material. The mean deformation until rupture occurred was equally increased, what probably may be attributed to the fiber deformation and its resistance before the set ruptured. However, by observing the



**FIGURE 2** - Mean values and SD Mean (MPa) referring to flexural strength of the composite material.

aspect using a scanning electron microscope, we verify that this increased resistance may not be attributed to a chemical adhesion, but probably only to the fiber resistance itself.

The interlocked fibers have an elastic modulus with intermediate magnitude between the short and long fibers, thus becoming indicated for temporary fixed partial prostheses and tooth contentions<sup>14,15,16</sup>. In the case of long lasting fixed pros-

theses, the addition of long and unidirectional fibers is recommended, as they have better mechanical properties<sup>7</sup>. However, there are reports in literature of clinical success for a longer period of time with the employment of interlocked fibers in fixed partial prostheses<sup>1</sup>, or even the indication of multidirectional fibers, due to the clinical performance of these fibers where there is little movement between teeth (as in periodontal contentions),



**FIGURE 3** - Stainless steel screwed matrix developed for specimen preparation.



**FIGURE 4** - SEM aspect of Charisma (Heraeus-Kulzer, Germany) composite resin under 500x magnifications.



**FIGURE 5** - Ribbon fiber and Charisma upon fracture under a 2000x magnification.



**FIGURE 6** - Remaining composite resin observed between the interlocked fibers in the fracture area (SEM, 75x).



**FIGURE 7** - No chemical bonding occurred between the composite resin and the interlocked. Fibers, but only mechanical union (SEM, 50x).



**FIGURE 8** - SEM aspect of Permalute+Ribbond under a 500x magnification.



**FIGURE 9** - SEM aspect of Permalute + Ribbond under 50x magnification.



**FIGURE 10** - SEM aspect of Permalute + Ribbond under 500x magnification.

that could lead to the fracture of more rigid materials<sup>11</sup>.

Regarding the fracture type, we agree with Samadzadeh<sup>12</sup>, once the addition of reinforcing fibers alters the fracture characteristics (it becomes partial and not catastrophic) without a bonding loss by the infrastructure, thus simplifying the replacement and repair of the prostheses.

## CONCLUSION

Based on the flexural strength values obtained for all the groups, under the scope of this study, the assemble Charisma+Ribbond has the highest flexural, followed by Charisma alone; Permalute composite resin and at last Permalute+Ribbond. Although the reinforcing fibers have, with no doubt, added greater strength to the set, the composite resin by itself showed that it plays an important

role in the choice of a material to be used for fixed partial prostheses. Therefore, it should be considered that when using a more flow resin for intra-root retention, it may not concur to a more resistant core, regardless the fiber reinforcement that is being used. According to the results of this study it can be concluded that:

1 - group III has higher flexural strength than Group I, followed by Groups II and IV, respectively;

2 - the addition of interlocked fibers contributed to a greater resistance, thus being indicated for the reinforcement of temporary fixed prostheses, as well for tooth contentions;

3 - however, after a SEM analysis of the fractured areas, we concluded that no chemical reaction occurred between the composite resin and the fibers, resulting in greater flexural strength for specimens of Group II, when compared to those prepared with non-reinforced resin (Group I).

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