Revista de Odontologia da Universidade Cidade de São Paulo
2008 maio-ago; 20(2): 114-121

ONE-HOUR TENSILE BOND STRENGTH OF DIFFERENT LUTING AGENTS TO A NiCr ALLOY

AVALIAÇÃO DA RESISTÊNCIA ADESIVA À TRAÇÃO DE DIFERENTES AGENTES CIMENTANTES A UMA LIGA DE NiCr APÓS UMA HORA DE ARMAZENAMENTO

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ABSTRACT

Introduction: This *in vitro* study evaluates a1-hour tensile bond strength of different luting agents to a nickel-chromium (NiCr) alloy. *Materials and Methods:* One hundred and twenty specimens of a NiCr without Be alloy (Vera Bond 2V) were submitted to airborne-particle abrasion with a 50µm aluminum oxide powder. Pairs of specimens (n=10) were luted together using: Panavia Fluoro Cement, Panavia 21, Cement-it, 3M RelyX ARC, Nexus, or Zinc Phosphate Cement (control). The luted specimens were immersed into distilled water at 37 ºC for 1-hour and, immediately after, submitted to tensile bond strength test. The data (MPa) were compared by 1-way ANOVA and Tukey´s test (*P* = .05). *Results:* Mean tensile bond strength values, in MPa, were Panavia Fluoro Cement: 36.56 (±10.74); Panavia 21: 29.53 (±4.8); Cement-it: 22.58 (±6.68); 3M RelyX ARC: 15.92 (±6.08); Nexus: 14.32 (±4.21), and Zinc Phosphate Cement: 2.40 (±0.9). These bond strengths were significantly different (*P*<.0001). *Conclusion:* All resin luting agents showed higher tensile bond strengths to a NiCr alloy after 1-hour of luting and significantly different than that of Zinc Phosphate Cement.

DESCRIPTORS: Dentin-Bending agents – Dental Cements – Tensile Strength

RESUMO

Introdução: Este estudo *in vitro* avalia a resistência adesiva à tração de diferentes agentes cimentantes a uma liga de nickel-chromium (NiCr) após uma hora de armazenamento. *Método:* Cento e vinte corpos de prova de uma liga de NiCr sem Be (Vera Bond 2V) foram microjateados com pó de óxido de alumínio de 50µm de granulação. Pares de corpos de prova (n=10) foram cimentados com: Panavia Fluoro Cement, Panavia 21, Cement-it, 3M RelyX ARC, Nexus, ou Zinc Phosphate Cement (control). Os corpos de prova cimentados foram imersos em água destilada à 37 ºC por 1 hora e imediatamente após submetidos aos testes por tração. Os resultados (MPa) foram analisados pelos testes estatísticos ANOVA e Tukey (*P* = .05). *Resultado:* As medias de resistência adesiva à tração, em MPa, foram para Panavia Fluoro Cement: 36.56 (±10.74); Panavia 21: 29.53 (±4.8); Cement-it: 22.58 (±6.68); 3M RelyX ARC: 15.92 (±6.08); Nexus: 14.32 (±4.21), e Zinc Phosphate Cement: 2.40 (±0.9). As resistências adesivas foram estatisticamente diferentes (*P*<.0001). *Conclusão:* Todos os agentes cimentantes resinosos apresentaram altas resistências adesivas à tração a uma liga de NICr após uma hora da cimentação e significativamente maiores do que aquela apresentada pelo cimento de fosfato de zinco.

DESCRITORES: Adesivos dentinários – Cimentos dentários – Resistência à tração

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INTRODUCTION

The use of metal-ceramic alloys for prosthodontic restorations is important because they represent the vast majority of existing tooth-colored cast restorations. (Aboush *et al.*[1] 1991, Giordano[7] 1996). These restorations have demonstrated long-term clinical success. Base metal alloys also continue to be used with metal-ceramic restorations (ADA[2], 2003). Base metal alloys are still used in the United States, Europe, Japan and studies concerning health benefits/risks determination associated with these materials are still investigated (Efeoglu *et al.*[5], 2006; Sonugelen *et al.*[16], 2006). There are high-noble, noble, and base metal alloys, each one with different indications and requiring different approaches in achieving adhesion with luting agents (Rubo *et al.*[15], 1996; Stoknorm et al.[17], 1996). As far as alloys are concerned, problems concerning failures with veneer crowns (resin/metal interface), and the development of "adhesive prostheses" (Rochette[14], 1973) in the early 1970s, resulted in studies designed to obtain more effective resin bonding to metals (Aquilino *et al.*[3], 1990).

Micro retention through airborne-particle abrasion, contributed significantly to improving the quality of adhesion to metal alloys (Imbery *et al.*[8], 1992, Kolodney *et al.*[10] 1992, Watanabe *et al.*[21], 1988) Nevertheless, adhesive luting agents are used clinically for prostheses with a lack of retention due to overly tapered preparations, which otherwise may result in treatment failure. Attempts have been made not only to develop and improve luting agents that bond to both metal alloys and tooth structures, providing more effective adhesion, but also to establish techniques to optimize results.

Zinc phosphate cement has been less used and replaced by more contemporary luting agents that provide better adhesion to metal alloys and dental structures (Imbery *et al.*[8], 1992, MCComb[12], 1996). Resin luting agents can be classified based on the mode of polymerization (chemical, light, or dual polymerization), esthetic needs, and adhesive, those containing adhesive monomers such as 10-MDP or 4-META and nonadhesive, containing only Bis-GMA or UDMA monomers. (MCComb[12], 1996, Swift[19], 1989). Effective bonding to metal surfaces with non-adhesive resin luting agents has proven to be difficult. (Aquilino *et al.*[3], 1990, Rubo *et al.*[15], 1996) In general, a clean surface and some type of adhesion-promoting primer are essential for adequate bonding (Suh[18], 1991). Some manufacturers have introduced metal primers that are specifically formulated to enhance the adhesion of nonadhesive resin luting agents to alloys through physicochemical interaction. (Aboush and Mudassir[1], 1991, Yoshida *et al.*[23], 1993) The correct choice of resin luting agents and the knowledge of how to prepare metal cast restorations (adhesion protocol) for adhesive luting have forged a new path in dentistry.

METHOD

The study consisted of evaluate and comparing the 1-hour tensile bond strength of NiCr alloy (Vera Bond 2V; Aalba-Dent Inc, Cordelia, Calif) specimens luted with different luting agents. To reduce variability, specimen preparation, surface specimen preparation and luting procedures were carried out by the same operator.

• *Specimen preparation*

Initially, one hundred and twenty solid cylindrical-shaped specimens (20mm diameter and 30mm length) were carved using an acrylic resin (Duralay; Reliance Dental Co, Worth, Ill) as a pattern, and then the metal casting was done according to the manufacturer's instructions. After divesting and removal of sprues, the NiCr cylindrical-shaped specimens were reshaped in a mechanical lathe (Xervitt; Model TPU, Xervitt Industria e Comercio de Maquinas Ltda, São Paulo, SP, Brazil) resulting in 60 pairs specimens (Fig. 1) with final sizes dimensionally standardized with a Vernier caliper (range 0-150mm, lower scale 0.05mm, Mitutoyo, Co., Suzano, SP, Brazil). The bodies of both specimens were shaped to fit into a testing machine. The luting surface of specimen A was 5mm diameter and B 10 mm, giving a final luting area of 19.635 mm^2. Specimen "B" were placed into plastic rings (30mm diameter and 10mm height) and embedded with slow-polymerizing acrylic resin (Dencor; Clássico Artigos Odontológicos Ltda, São Paulo, SP, Brazil) also to a better fit into a testing machine.

• *Luting surface preparation*

In order to obtain a smooth and flat luting surfaces, they were manually polished with abrasive metallographic discs (Extec Co., Wallinford, Conn., USA) of decreasing grit up to #600. Then, they were submitted to airborne-particle abrasion using a micro-etcher (Micro Etcher, Danville Engineering, San Ramon, Calif) and 50 μm aluminum oxide powder (Bio-Art Equipamentos Odontológicos Ltda, São Carlos, SP, Brazil). Pressure was set at 60-80 lb/in^2 for 4-6 seconds, holding the etcher perpendicular to the metal surface, at an average distance of 1.0cm. The specimens were rinsed in running water for 1 minute and air-dried. The specimens were randomized divided into 6 groups (n=10) according to the luting agent to be tested (Table 1).

• *Luting*

Specimens A and B (10 pairs) were luted with 1 of the luting agents displayed in Table 1. Two of the resin luting agents were chemically polymerized (Panavia 21 and Cement-it), and 3 underwent dual polymerization (Panavia Fluoro Cement, 3M RelyX ARC and Nexus). For the nonadhesive resin luting agents (Cement-it, 3M RelyX ARC, and Nexus), the metal primers recommended by the manufacturers were used to improve bonding to the NiCr specimens (Table 1). The Zinc Phosphate Cement (Zinc Cement) was used as a control. Luting was performed at room temperature (22.5ºC to 24ºC), using a stainless steal apparatus developed specifically for this study (Fig. 2) to ensure constant pressure (1MPa) during luting. All materials were applied following manufacturer's recommendations. Excess marginal luting agent was removed using small brushes (Cavibrush, FGM Produtos Odontológicos Ltda, Joinville, SC, Brazil). As recommended for the dual polymerizing resin luting agents (Pfeifer et al.[13], 2003), the light polymerization unit (Curing Light XL 1500, Model 5518AA, n.X101554; 3MESPE, St Paul, Minn), with a light intensity of 500 mW/cm^2 was positioned as close as possible to the cementation margins. Each specimen was polymerized for 40 seconds at 4 positions. The luted specimen pairs were immersed into distilled water at 37 ºC for 1 hour (Mathis and Ferracane[11], 1989), and then immediately submitted to tensile bond strength test using a universal testing machine (Model FS-5; Riehle, East Moline, Ill), set at a 0.5 mm/minute crosshead speed.

• *Statistical analysis*

The data were transformed in logarithm and then statistically compared by a one-way analysis of variance (ANOVA) complemented by the Tukey´s test using a statistical software (Version 6; SAS Institute Inc., Cary, NS). The level of significance adopted was 5% (*P*=. 05).

RESULTS

The one-way Analysis of Variance (Table 2) indicated significant that the differences among the 1-hour ten-

Table 1 – *Luting agents and metal primers used.*

Luting Agent	Type	Reaction	Metal Primer	Monomer	Manufacturer
Zinc Cement	Zinc Phosphate	Crystallization		S.S. White, Rio de Janeiro, Brazil	
Cement-it	Non-adhesive resin luting agent	Chemical polymerization Metal Bond + Bond-1	Bis-GMA UDMA	Pentron, Inc, Wallinford,Conn	
Panavia 21	Adhesive resin luting agent	Chemical polymerization		10-MDP	Kuraray,Co, Ltd Osaka,Japan
Panavia F Cement	Adhesive resin luting agent	Dual polymerization		10-MDP	Kuraray,Co,Ltd
3M Rely X ARC Non-adhesive resin luting agent	Dual polymerization	Ceramic primer	Bis-GMA	3M ESPE, St Paul, Minn	
Nexus	Non-adhesive resin luting Agent	Dual polymerization	Nexus 1	UDMA Bis-GMA	SDS Kerr, Orange Calif

Table 2. *One way ANOVA results (P=0.0001)*

Source of Variation	Degrees of Freedom	Mean Square	F	Level of Significance
Luting Agents	5	1.8448	103.35	0.0001
Error	54	0.0178		
Total	59			

Table 3 – *Tukey multiple comparison tests between the means of the groups tested.*

Tukey´s multiple comparison test between the groups tested*					Luting agents	Mean (MPa)	Logarithmical mean (Kgf)	Critical value
A					Panavia FC	36.56	2.55431	0.17590
A	B				Panavia 21	29.53	2.47421	
	B	C			Cement - it	22.58	2.34585	
		C	D		3M Rely X ARC	15.92	2.18242	
			D		Nexus	14.32	2.14982	
				E	Zinc Phosphate	2.40	1.36451	

*The groups with same letters did not show significant statistical differences (P<.05)

sile bond strengths, depending on the luting agent used (*P*<.0001). Figure 3 illustrates the 1-h bond strength of the experimental groups in MPa. Panavia Fluoro Cement showed a significant higher mean bond strength 36.56 (±10.74), when compared to the others luting agents except for Panavia 21 - 29.53 (±4.8). The bond strength

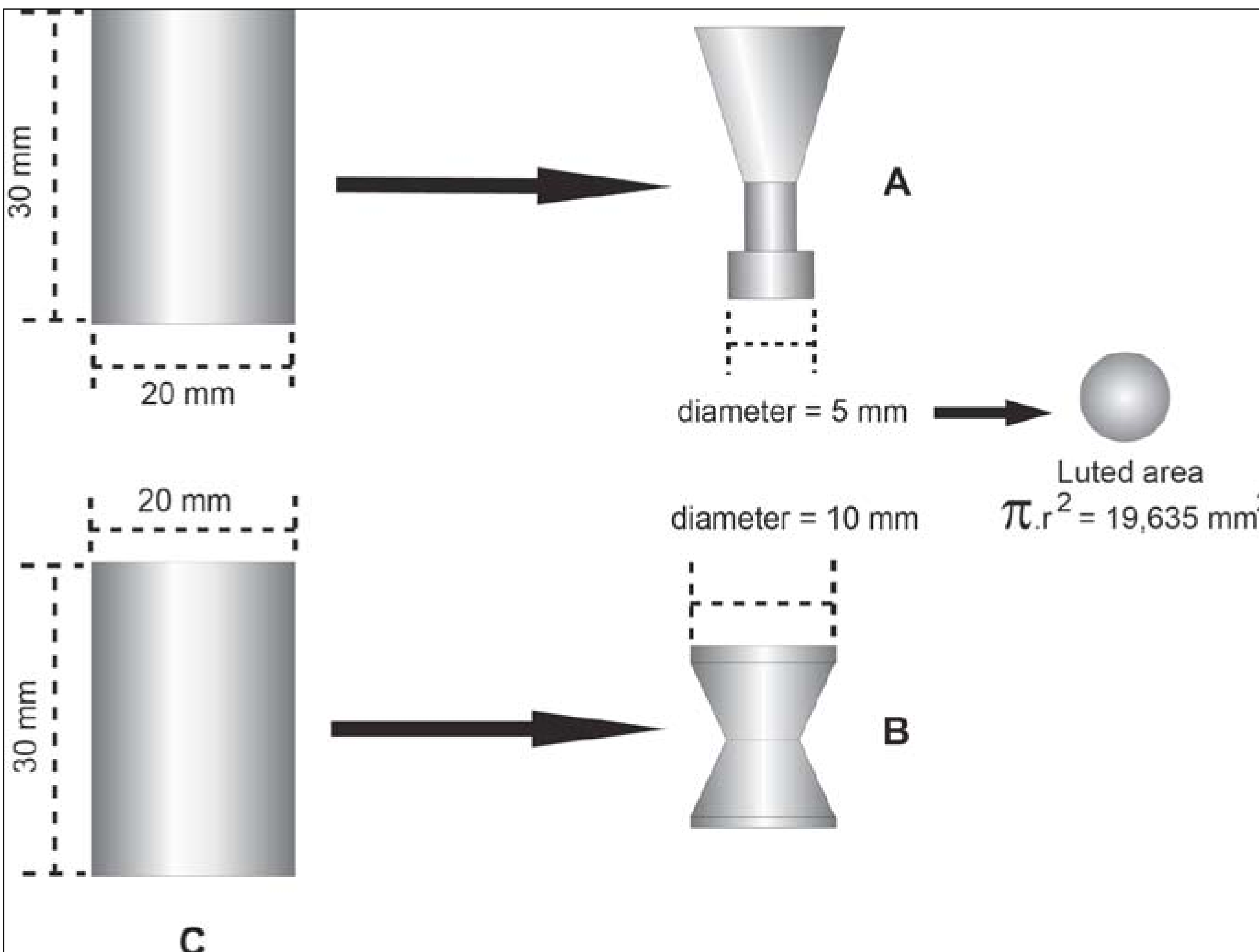

Figure 1 - *Diagram illustrating the experimental specimens. Each NiCr cylinder Specimen (C) was reshaped either resulting in specimens with 5mm diameter at the luting surface (A: arrow) or in specimens with 10mm diameter (B).*

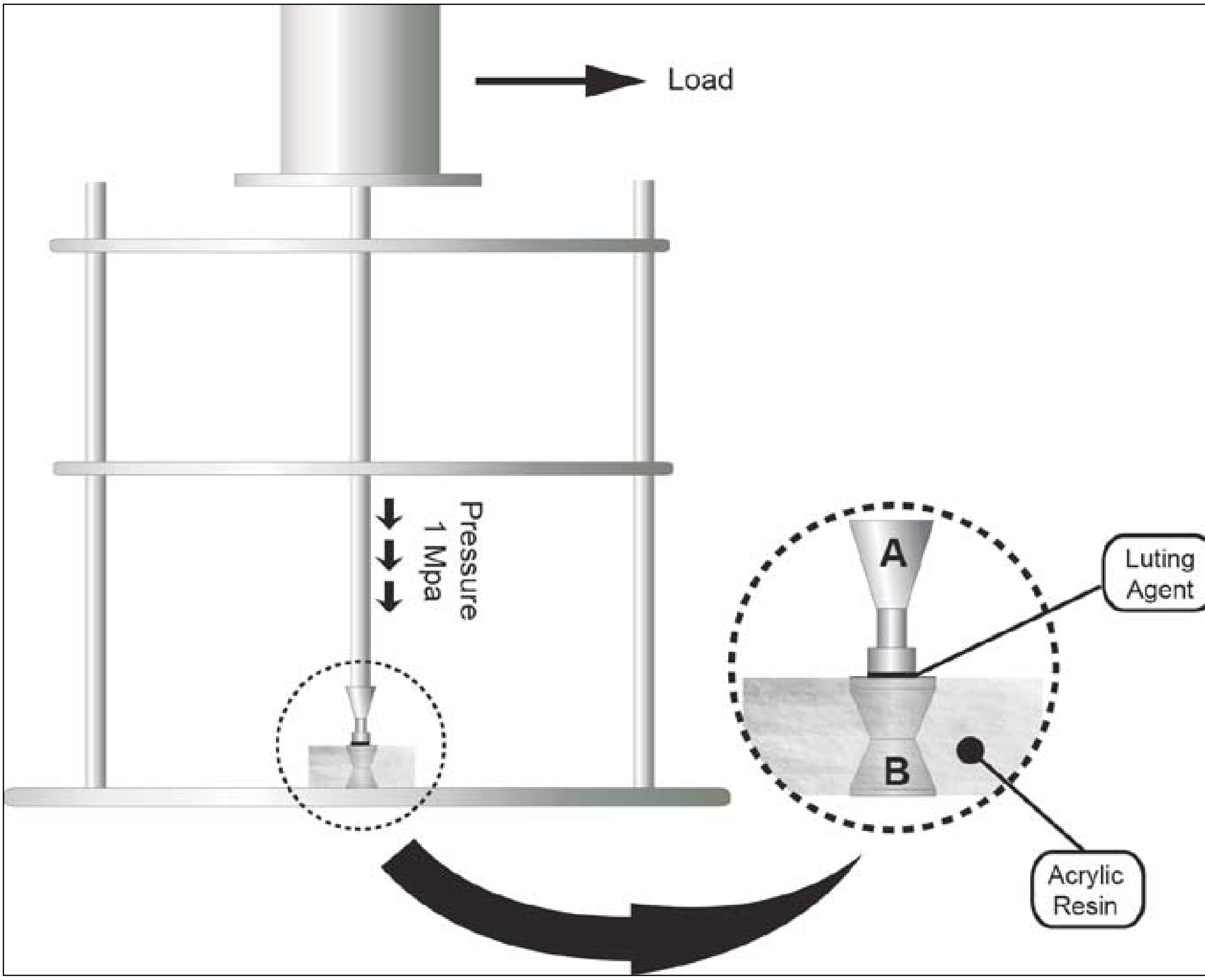

Figure 2 - *Diagram illustrating the stainless steal apparatus especially designed to ensure constant pressure on the luted specimens (A+B)*

of the other groups were, as follows: Cement-it: 22.58 (±6.68); 3M RelyX ARC: 15.92 (±6.08); Nexus: 14.32 (±4.21). The Zinc Cement, used as control, presented mean bond strength significantly lower than those of all other tested luting agents 2.40 (±0.9). Table 3 shows the Tukey test results on the transformed (logarithm) reported means values of the materials tested.

DISCUSSION

The comparison of the 1-hour tensile bond strength of different resin luting agents and zinc phosphate cement to a NiCr alloy indicated significant highest bond strength when either the luting agent Panavia Fluoro Cement or Panavia 21 was used, whereas the smallest bond strength was observed at the zinc phosphate cement group.

The tensile bond strength analysis was done 1-hour after luting because in many clinical situations the newly luted restorations can be prematurely placed in function jeopardizing the treatment, especially when the patient does not follow the clinician´s recommendations.

Panavia Fluoro Cement and Panavia 21 showed the highest bond strengths to the NiCr alloy because they probably generated chemical adhesion in addition to micro-mechanical retention (Atta *et al.*[4] 1990). This chemical adhesion was only possible with the development of bonding monomers to metal such as 10-MDP, found in Panavia 21 and Panavia Fluoro Cement. The adhesive monomer 10-MDP has a chemical affinity with the metal oxides found in base metal alloys (MC-Comb[12], 1996). Resin luting agent bond strength can be high, especially after airborne-particle abrasion to base

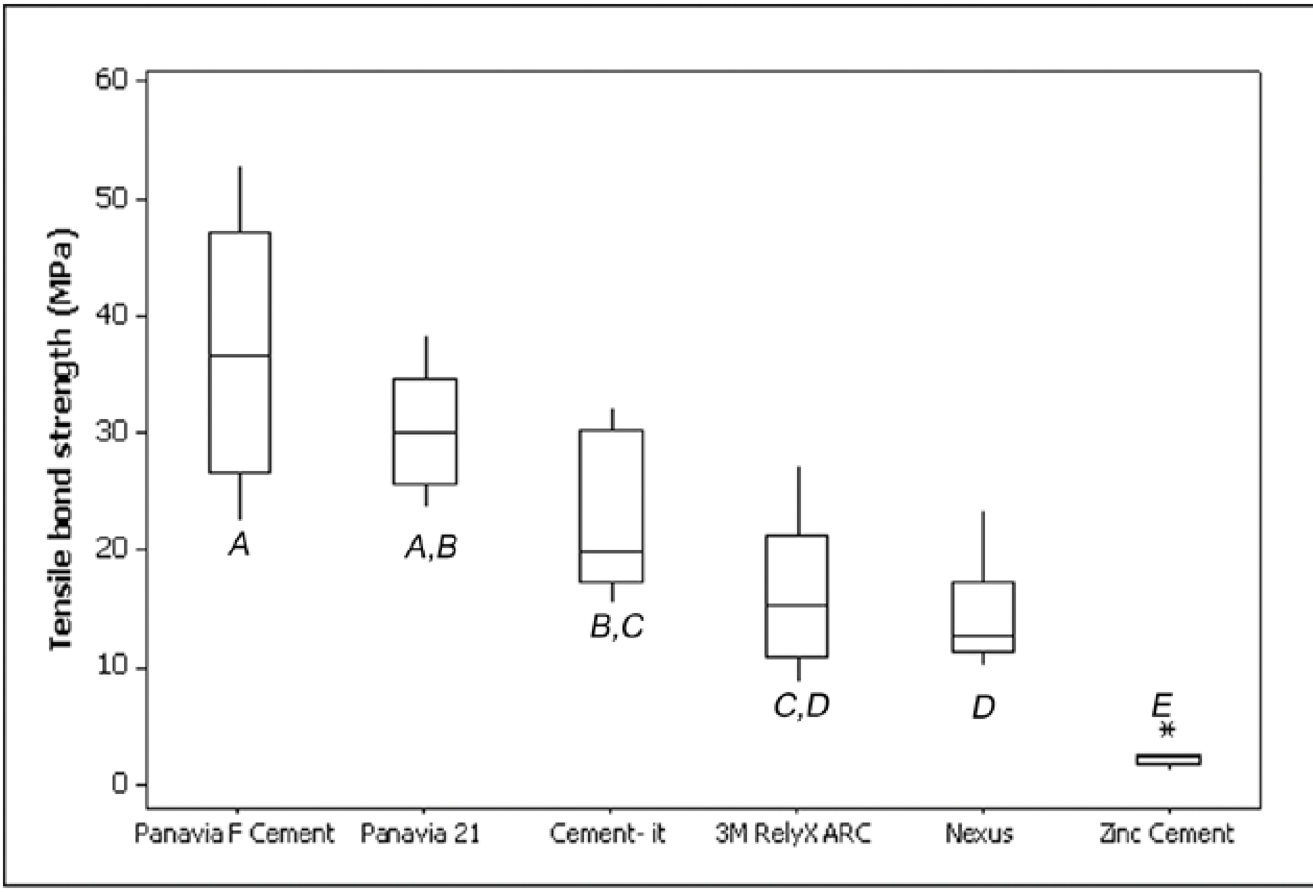

Figure 3 – *Graphic representation of the 1-hour tensile bond strength test. Data are presented as mean + standard deviation, in MPa, of 10 replicas. Different letters indicate statistical differences (p < .05). Observe that Panavia Fluoro Cement presented the highest values, whereas the Zinc Cement presented the smallest values.*

metal alloys. (Atta *et al.*[4] 1990, Flood *et al.*[6], 1989, Knobloch *et al.*[9], 1996) Probably Panavia Fluoro Cement (a new version of Panavia 21) has a better adhesive capacity and final polymerization due to it chemical and photo activation (dual polymerization) which raises its monomers into polymer conversion, resulting in a better mean value. High bond strength values of adhesive resin luting agent (with chemical polymerization) to an airborne-particle abraded NiCr metal alloy have been reported (Flood *et al.*[6], 1989).

The bond strength of the nonadhesive resin luting agent, Cement-it, although lower, was not significantly different from that of Panavia 21 (Aquilino *et al*[3] and Suh[18], 1991) had already reported problems in developing a resin luting agent that would adhere to metal. Thus, for a nonadhesive resin luting agent with Bis-GMA or UDMA based monomers, the metal primer placed between the metal and the nonadhesive resin luting agent creates a chemical bond that is critical for increasing bond strength (Suh[18], 1991; Yoshida *et al.*[22], 1996; Yoshida *et al.*[23], 1993). The different adhesive monomers found in metal primers have chemical affinity with the oxides present in base metal alloys (Suh[18], 1991, Yoshida et al.[23], 1993) and they appear to improve the bonding of the luting agent to the metal substrate (Suh[18], 1991, Swift[19], 1989 , Tjan *et al.*[20], 1987; Yoshida *et al.*[22], 1996) although their efficiency, according to some authors, is not fully evidenced (Atta *et al.*[4] 1990, Tjan *et al.*[20], 1987). The application of Metal Bond and Bond-1 with Cement-it caused a mean bond strength not statistically different to that of Panavia 21.

Furthermore, for the nonadhesive resin luting agents, the mean bond strength of Cement-it and 3M RelyX ARC did not differ significantly, whereas those of Cement-it and Nexus did. The bond strength did not differ significantly for the nonadhesive and dual polymerizing resin luting agents, 3M RelyX ARC and Nexus. The tensile bond strength of a dual polymerizing, nonadhesive resin luting agent to an airborne-particle abraded NiCr alloy, when light-activated, has been reported as approximately 15 MPa (Ju and Xu[24], 1987). Although the bond strength of 3M RelyX ARC did not differ significantly from that of Cement-it, its mean value was lower. Despite the fact that 3M RelyX ARC was used with Ceramic Primer, and that Nexus was used with Nexus 1, the resulting values were lower than those of all other resin luting agents tested, which leads to the assumption that nonadhesive, dual polymerizing resin luting agents are not as effective as the other resin luting agents, even when they are combined with the metal primer systems recommended by the manufacturer. The dual polymerizing, nonadhesive resin luting agents 3M RelyX ARC and Nexus did not attain the mean tensile bond strength suggested in the Literature for clinical success (20.00 MPa; Ju and Xu[24], 1987), but their tensile bond strength were much higher and significantly different from the Zinc phosphate Cement (Zinc Cement). The mean tensile bond strength registered for Zinc Cement was significantly lower than that of all others tested luting agents. Although the Zinc Phosphate Cement presented lower adhesion in this experiment, when appropriate crown resistance and retention form are clinically present this cement may employed as well as the resin luting agents that lead to clinical success in fixed prosthodontics. However, when a clinical condition of lack retention is present, using luting agents with better adhesive capability could be priority.

Limitations and Suggestions for Future Studies

There are few scientific data about bond strength of resin luting agents at initial periods of setting reaction. First hours of setting reaction are critical, where the luting agent resistance must be analyzed because involuntarily showed strength may start at a short period after luting involving restorative treatment, jeopardizing the treatment success. Nowadays there is a wide range of commercially available luting agents. The majority of them are resin luting agents, and adhesion procedures are complex. Chemically polymerizing resin luting agents are the better choice for metal cast restorations, however, dual polymerizing resin luting agents can also be used as a recommended by many manufacturers. But the passage of the light through the cast specimen being luted can be impaired during light polymerization. Then, when a dual polymerizing resin luting agent is used clinically with metal cast restorations, light polymerization of the luted margins must be reinforced in order to reach higher bond strength. This *in vitro* study allowed an immediate assessment of the tensile bond strength between the luting agent and the NiCr alloy used. However, *in vitro* tests cannot simulate clinical handling factors of preparing cast metal restorations for adhesive luting. On the contrary, regarding to adhesive and nonadhesive resin luting agents, a correct application of metal primers and light activation (when necessary) may be crucial for achieving appropriate bonding between NiCr alloy and teeth. New studies must be developed in order to approximate this *in vitro* model to the *in vivo* clinical situation. However, the results obtained in this study can be interesting to the clinicians that deal with situations where a newly luted restoration will be prematurely placed in function.

CONCLUSION

1 - Panavia Fluoro Cement showed the highest 1-hour tensile bond strength but not significant different from that of Panavia 21.

2 - The 1-hour tensile bond strength registered for Zinc Cement was significantly lower than that of all others tested luting agents.

ACKNOWLEDGMENTS

The authors thank Dr Washington Steagall Jr. for drawing the experimental stainless steal apparatus specially designed for this study and, Professor Márcia Marques for the critical review of the manuscript.

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Recebido em:06/07/2007

Aceito em: 08/12/2007