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Effect of Composite Containing an Iodonium Salt on the Bond Strength of Brackets to Bovine Enamel

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This study investigated the effect of the incorporation of an iodonium salt in experimental composites, on the bond strength of metallic brackets bonded to bovine teeth. Two hundred and seventy bovine teeth were embedded in self-curing acrylic resin and divided into 18 groups (n=15), according to the experimental composite with an iodonium salt at molar concentrations 0 (control), 0.5, or 1%; the light-activation times (8, 20 and 40 s); and the storage times (10 min or 24 h). Metallic brackets were fixed on the tooth surface using experimental composites. Photoactivation was performed with a quartz-tungsten-halogen light-curing unit curing unit for 8, 20 and 40 s. The specimens were stored in distilled water at 37 °C for 10 min or 24 h and submitted to bond strength test at 0.5 mm/min. The data were subjected to three-way ANOVA and Tukey's test (α=0.05). The Adhesive Remnant Index (ARI) was used to classify the failure modes. The shear bond strengths (MPa) at 10 min for light-activation times of 8, 20 and 40 s were: G1 - 4.6, 6.9 and 7.1; G2 - 8.1, 9.2 and 9.9; G3 - 9.1, 10.4 and 10.7; and at 24 h were: G1 - 10.9, 11.1 and 11.7; G2 - 11.8, 12.7 and 14.2; G3 - 12.1, 14.4 and 15.8. There was a predominance of ARI score 3 for groups with 10 min storage time, and ARI score 2 for groups with 24 h storage time. In conclusion, the addition of iodonium salt (C05 and C1) to the experimental composite may increase the bond strength of brackets to bovine enamel using reduced light exposure times.

Key Words: shear bond strength, aging, bonding, Orthodontics, polymerization, composite.

Introduction

Metallic brackets are routinely bonded to the teeth using light-cured composite resins (1-3). The advantage of photoactivated composites is that the clinician has the control of working time to place the brackets in the correct position, easy removal of the excess composite and possibility of immediate insertion of the orthodontic archwire (4). Adequate early polymerization is necessary for brackets bond to the teeth resist the placement of the orthodontic archwire. However, complete polymerization of the composite may not be always possible when considering the clinically used light exposure times (5).

Composite material requires adequate light irradiance and wavelength (400-500 nm) to activate the photosensitizer camphorquinone (CQ), which reacts with an amine co-initiator to generate free radicals for polymerization (6,7). According to the manufacturers' instructions, light exposure times between 20 and 40 s are required to bond brackets to the teeth to obtain high bond strength (8). However, long exposure times are uncomfortable for the patient and present drawbacks for the clinician (9).

Some methods to reduce the light-curing time for bonding orthodontic brackets are proposed in the literature (2,9-12). The main method proposed is using light-curing units with high irradiance (e.g. xenon plasma arc and lasers) for fast polymerization (13,14), but these units are expensive. Another alternative is the use of an iodonium salt. It has been reported that the use of iodonium salt has shown satisfactory results for dental adhesives when used associated with CQ (15). The theory that explains the performance of iodonium salts is that the CQ, after excited by the curing light, can promote decomposition of the iodonium salt and enhance the polymerization kinetics (15,16).

Bond strength tests are usually carried out 24 h after the orthodontic brackets are bonded to the teeth. However, this is not what occurs in the clinical practice, where the insertion of the orthodontic archwire occurs minutes after the brackets are bonded. Several studies have shown that 24 h after the bonding procedure, the brackets bond strength increased in relation to the times of 5, 10, and 30 min (9,17,18). There is no consensus as regards the waiting time required before placing the brackets.

The aim of this study was to investigate the effect of the incorporation of a iodonium salt in experimental composites on the shear bond strength of metallic orthodontic brackets bonded to bovine teeth, using different storage times (10 min or 24 h). The hypotheses tested were that 1) the incorporation of an iodonium salt in experimental composites would lead to higher shear bond strengths, 2)

the storage time (10 min or 24 h) would not affect the shear bond strengths, and 3) specimens light-activated for longer periods would have higher shear bond strengths.

Material and Methods

Experimental Composites

This study is a follow up of the investigations on the use of iodonium salts to improve the polymerization of resin-based dental cements (9,19). The three experimental materials tested were obtained using diphenyliodonium hexafluorphosphate (DPIHFP; Sigma-Aldrich, Milwaukee, WI, USA) at molar concentrations of 0 (control), 0.5, or 1%. A model dimethacrylate co-monomer was obtained by mixing the monomers bisphenol-A glycidyl dimethacrylate (Esstech, Essington, PA, USA) and triethylene glycol dimethacrylate (Esstech) at a 70:30 mass ratio. The photoinitiating system consisted of ethyl-4 dimethylamino benzoate (EDAB; Sigma-Aldrich) and CQ (Esstech), each added at a molar concentration of 1%. Butylated hydroxytoluene (0.1 mol%) was added as radical scavenger. The blends were loaded with a 60 mass% of silanized strontium-based glass particles with 1 μm average particle size to control composite viscosity and render it thixotropicity.

Bonding Procedures

Two hundred and seventy bovine mandibular incisors without cracks were collected. The teeth were embedded in self-curing acrylic resin (Clássico Produtos Odontológicos Ltda., São Paulo, SP, Brazil) using polyvinyl chloride tubes as molds, leaving the buccal face perpendicular to the horizontal axis. The buccal faces of all teeth were cleaned using a rubber cups with non-fluoridated pumice-water slurry for 10 s, rinsed with air-water spray for 10 s and air-dried for 10 s. The teeth were divided into 18 groups (n=15), according to three experimental bonding materials (control, C05 and C1); light-activation time (8, 20 and 40 s) and storage time (10 min or 24 h). The middle third of the buccal vestibular face of all teeth was etched with 37% phosphoric acid gel (3M ESPE, St. Paul, MN, USA) for 30 s, rinsed with air-water spray for 30 s and air dried for 30 s. One layer of Single Bond 2 Adhesive (3M ESPE) was applied on the teeth, gently dried for 5 s, and photoactivated for 10 s with a quartz-tungsten-halogen light-curing unit (XL2500; 3M ESPE) with 800 mW/cm^2 irradiance.

Stainless steel standard maxillary central incisor brackets (slot 0.022"; Ortodontia Morelli, Sorocaba, SP, Brazil) were positioned and seated firmly on the tooth surface. Excess bonding material was removed using a microbrush (KG Sorensen, Cotia, SP, Brazil) and light-activation was carried out with one exposure on each of the four sides of the bracket, with total exposure times of 8, 20, or 40 s. The specimens were stored in distilled water at 37 °C for 10 min or 24 h.

Bond Strength Testing and Failure Analysis

The shear bond strength (SBS) test was conducted in a mechanical testing machine (Model 4411; Instron, Canton, MA, USA) using a knife-edged rod at a crosshead speed of 1.0 mm/min until failure. A mounting jig was used to align the tooth-bracket interface parallel to the testing device. The SBS values were calculated in MPa and analyzed using three-way ANOVA and Tukey's test (α=0.05). After debonding, tooth and bracket surfaces were examined with a stereomicroscope (Olympus Corp, Tokyo, Japan) under 8× magnification. The Adhesive Remnant Index (ARI) (20) was used to classify the failure modes, according to: score 0 indicates that no bonding resin remained on the tooth; score 1 indicates that less than half of the bonding resin remained on the tooth; score 2 indicates that more than half of the bonding resin remained on the tooth; and score 3 indicates that all bonding resin remained on the tooth, along with a clear impression of the bracket mesh.

Results

The results for shear bond strength of brackets to teeth are shown in Table 1. For 10 min storage time, the control group showed significantly lower ($p<0.05$) shear bond strengths than the other bonding materials for all light-activation times. After 24 h storage, the experimental composite C1 had significantly higher ($p<0.05$) SBS than the control composite when light-activated for 20 and 40 s. No statistically significant differences ($p>0.05$) were detected when light-activated for 8 s. In the comparison of C05 and C1, no significant differences ($p<0.05$) were observed for storage time or light-activation time.

When the light-activation times were compared at 10 min storage time, the control group light-activated for 8 s showed significantly lower shear bond strength ($p<0.05$) than groups light-activated for 20 and 40 s, except for the groups C05 and C1 ($p<0.05$). At 24 h, the C1 experimental composite light-activated for 40 s had significantly higher SBS ($p<0.05$) than the group light-activated for 8 s. No significant difference was detected for control group and C05 ($p>0.05$). The SBS for 24 h storage time was significantly higher ($p<0.05$) when compared to 10 min for all groups (bonding material or light-activation time). The results for ARI are shown in Figure 1. There was a predominance of score 3 was observed for groups stored for 10 min, and score 2 was the most frequent for groups stored for 24 h.

Discussion

The first hypothesis tested was accepted. Generally, an increased SBS was observed when DPIHFP-modified agents were incorporated in experimental composites for all light-

activation times. This finding is in agreement with a previous study with experimental composites, which also found significantly higher SBS in relation to the control group, but using diphenyliodonium chloride (DPC) as iodonium salt (9). The higher bond strength of experimental composites using DPIHFP-modified agents may be explained by the fact that the salt, even in low concentration, can promote higher polymerization reactivity. According to Ogliari et al. (15) the introduction of DPIHFP as co-initiator showed significant effect on the monomer conversion. In the ternary photo-initiator system, formed by CQ, EDAB and DPIHFP, the DPIHFP may react with inactive CQ radicals formed during polymerization to decrease the termination rate and improve the initiation rate by producing new radicals from the salt fragmentation, improving the monomer reactivity in relation to two-component initiator systems (16,21,22).

Table 1. Mean shear bond strength values (S.D.) in MPa

Storage time*	Bonding material**	Light-activation time		
		8 s	20 s	40 s
10 min	Control	4.6 (1.5)^{b,B}	6.9 (2.2)^{b,A}	7.1 (2.5)^{b,A}
	C05	8.1 (2.4)^{a,A}	9.2 (2.1)^{a,A}	9.9 (2.8)^{a,A}
	C1	9.1 (2.91)^{a,A}	10.4 (3.9)^{a,A}	10.7 (3.3)^{a,A}
24 h	Control	10.9 (2.8)^{a,A}	11.1 (3.2)^{b,A}	11.7 (3.1)^{b,A}
	C05	11.8 (2.7)^{a,A}	12.7 (4.0)^{ab,A}	14.2 (4.5)^{ab,A}
	C1	12.1 (4.2)^{a,B}	14.4 (4.4)^{a,AB}	15.8 (4.7)^{a,A}

*Significant differences were observed between 10 min and 24 h for all light-activation time and bonding material combinations ($p<0.05$). **C05 and C1 were formulated containing 0.5 and 1 mol% of DPIHFP. For each storage time, means followed by different lowercase letters in the same column indicate significant differences for bonding material, and by different uppercase letters in the same row indicate significant differences for light-activation time (Tukey's test, $p<0.05$).

An increase of the polymerization rate is advantageous to the development of experimental composites by reducing curing time. When the two concentrations of DPIHFP were compared, no statistically significant difference in shear bond strength was found for all light-activation times. In this study, increased molar concentration of iodonium salt did not increase the curing reactivity of the experimental composite. These results agree with those of a previous study where lower concentrations of iodonium salt had minor influence on the polymerization rate (15).

The second hypothesis was rejected, since significant differences were observed between shear bond strength for storage time at 24 h in relation to 10 min for all groups (bonding material or light-activation time). This result is in agreement with other studies, where the shear bond strength increased after 24 h in relation to 10 min (9,17,18). According to Leung et al. (23), post-polymerization of the composite, mainly in the first 24 h, may be responsible for increase in SBS. For other authors (24,25), any alteration in conversion in the latter curing stages may increase polymer crosslinking and improve polymer properties.

When light-activation times were compared, the SBS of the groups light-activated for 20 and 40 s at 10 min was significantly higher than for 8 s of the control group. No statistically significant differences were found among the light-activation times for the other groups tested at 10 min or groups tested at 24 h. Therefore, the third hypothesis was partly accepted. According to Rueggeberg (5), polymerization of resin materials is dependent on the radiant exposure, which is the product of irradiance and exposure time. In this study, approximately 6.4, 16 and 32 J/cm^2 energy doses were used for specimens light-activated for 8, 20 and 40 s. These differences in energy doses may

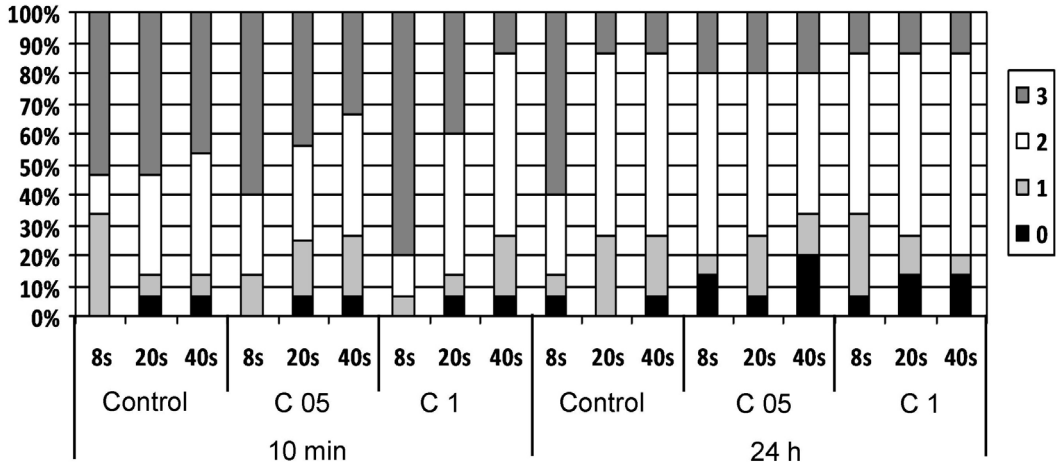

Figure 1. Frequency distributions (%) of the Adhesive Remnant Index (ARI) scores for all groups. Score 0 indicates that no bonding resin remained on the tooth; Score 1 indicates that less than half of the bonding resin remained on the tooth; Score 2 indicates that more than half of the bonding resin remained on the tooth; and Score 3 indicates that all bonding resin remained on the tooth, along with a clear impression of the bracket mesh.

explain the difference among the light-activation times for the control group at 10 min storage time.

The results of ARI scores indicated that the majority of debonding failures left all bonding resin on the tooth, along with a clear impression of the bracket mesh (score 3) at 10 min storage time and prevalence of score 2, where more than half of the bonding resin remained on the tooth, at 24 h storage time. Similar results were found in a previous study using iodonium salt-modified agents (9). According to Costa et al. (9), failures of modified materials tended to include fracture of the bonding material, indicating a better mechanical interlocking of the experimental composite with the bracket mesh, probably by improvements in the polymerization due to the addition of iodonium salt. In conclusion, the present study demonstrated that the addition of iodonium salt in the experimental composites may increase the bond strength of brackets bonded to bovine enamel using reduced light exposure times.

Resumo

Estudo investigou o efeito da incorporação de um sal de iodônio em compósito experimental, na resistência de união de braquetes metálicos fixados em dentes bovinos. Duzentos e setenta dentes bovinos foram embutidos e divididos em 18 grupos (n=15), de acordo com o compósito experimental com sal de iodônio na concentração molar de 0 (controle), 0,5 e 1 %; tempo de fotoativação (8, 20 e 40 s); e, tempo de armazenagem (10 min e 24 h). Braquetes metálicos foram fixados na superfície do dente usando compósitos experimentais. A fotoativação foi efetuada com o aparelho XL 2500 por 8, 20 e 40 s. As amostras foram armazenadas em água destilada a 37º C por 10 min e 24 h e submetidos ao ensaio de resistência de união à velocidade de 0,5 mm/min. Os dados foram submetidos à Análise de variância de 3 fatores e ao teste de Tukey (α=0,05). O Índice de Remanescente do Adesivo (IRA) foi usado para classificar os modos de falhas. Os valores de resistência de união ao cisalhamento (MPa) no período de 10 min para os tempos de fotoativação de 8, 20 e 40 s foram: G1 - 4,6; 6,9 e 7,1; G2 - 8,1; 9,2 e 9,9; G3 - 9,1; 10,4 e 10,7; e no período de 24 h foram: G1 - 10,9; 11,1 e 11,7; G2 - 11,8; 12,7 e 14,2; G3 - 12,1; 14,4 e 15,8. Houve predominância de escore 3 para os grupos armazenados por 10 min e escore 2 para os grupos armazenados por 24 h. Em conclusão, a adição do sal de iondônio (C05 e C1) em compósitos experimentais pode aumentar a resistência de união de braquetes ao esmalte bovino usando reduzidos tempos de fotoativação.

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