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ORIGINAL ARTICLE

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Evaluation of wettability characteristics and adhesion of resin composite to photo-polymerized pulp-capping materials with and without bioactive glass

Avaliação das características de molhabilidade e adesão da resina composta aos materiais fotopolimerizáveis com e sem vidro bioativo

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ABSTRACT

Objective: This study evaluated the wettability and adhesive properties of three different photo-polymerized resin-based pulp-capping materials with or without bioactive glass (BAG). **Material and Methods:** Cylindrical specimens (5 per group) were prepared from photo-polymerized pulp-capping materials (TER-TheraCal LC, BIN-Biner LC, and CAL-CalciPlus LC containing BAG). After surface finishing, contact angle measurements were made (θ)using the sessile drop method and surface free energy was calculated. For shear test, cylindrical acrylic blocks (N = 30) (diameter: 6 mm; height: 1 m) in the center were filled with the pulp-capping materials (N = 10 per group) flattened using a metal spatula and photo-polymerized. The specimens were stored at 37 °C in 100% humidity for 24 h prior to the bonding procedures. An adhesive system (CLEARFIL SE BOND, Kuraray) was applied on the material surfaces and photo-polymerized for 20 s. Then, resin composite material (Filtek Z250, 3M ESPE) was bonded on the substrate materials using translucent plastic molds (internal diameter: 2 mm; height: 2 mm). The specimens were photo-polymerized with an LED photo-polymerization unit for 20 s. After polymerization, the specimens were stored at 37 °C, at 100% humidity for 24 h. Shear force was applied at the pulp-capping material and the resin composite interface In a universal testing machine (1 mm/min).Data were analyzed using 1-way ANOVA and Tukey`s tests at the significance level of 0.05. **Results:** Contact angle values showed significant difference between the 3 materials with group CAL presenting the lowest (35.35 ± 12.89)

RESUMO

Objetivo: Este estudo avaliou as propriedades de molhabilidade e adesivas de três diferentes materiais capeadores pulpares fotopolimerizaveis à base de resina, com ou sem vidro bioativo (BAG). **Material e Métodos:** Amostras cilíndricas (5 por grupo) foram preparadas a partir de materiais capeadores pulpares fotopolimerizáveis (TER-TheraCal LC, BIN-Biner LC e CAL-CalciPlus LC contendo BAG). Após o acabamento da superfície, as medidas do ângulo de contato foram feitas (θ) usando o método de gota séssil e a energia livre da superfície foi calculada. Para o ensaio de cisalhamento, blocos de acrílico cilíndricos (N = 30) (diâmetro: 6 mm; altura: 1 m) foram preenchidos no centro com os materiais de capeamento pulpar (N = 10 por grupo) achatados usando uma espátula metálica e fotopolimerizados. Os espécimes foram armazenados a 37 ° C em 100% de umidade por 24 h antes dos procedimentos de colagem. Um sistema adesivo (CLEARFIL SE BOND, Kuraray) foi aplicado nas superfícies do material e fotopolimerizado por 20 segundos. Em seguida, o material de resina composta (Filtek Z250, 3M ESPE) foi colado nos materiais do substrato utilizando moldes de plástico translúcido (diâmetro interno: 2 mm; altura: 2 mm). Os espécimes foram fotopolimerizados com um dispositivo de fotopolimerização de LED por 20 s. Após a polimerização, os espécimes foram armazenados a 37 °C, a 100% de umidade por 24 h. A força de cisalhamento foi aplicada no material capeador e na interface da resina composta em uma máquina universal de ensaios (1 mm / min). Os dados foram analisados por meio do teste de ANOVA e teste de Tukey ao nível de significância de 0,05. **Resultados:** Os valores do ângulo de contato mostraram diferença significativa entre os 3 materiais com o grupo CAL apresentando os menores valores (35,35 ± 12,89) and group BIN the highest values (74.77 ± 13.56) ($p < 0.05$). Significantly lower surface energy was observed with BIN (36.22) ($p<0.05$) compared to those of the groups TER (44.7) and CAL (46.2)($p > 0.05$). Group BIN (9.12 ± 3.45) showed significantly lower ($p < 0.05$) mean bond strength than those of TER (11.56 ± 5.67) and CAL (12.66 ± 4.34) ($p > 0.05$). Most of the observed modes of failures in all groups were of mixed type of failures. **Conclusion:** The bioglass used in Calciplus LC did not adversely affect the wettability, surface energy and adhesive properties.

KEYWORDS

Adhesion; Biocompatible materials; Bioglass; Dental pulp capping; Shear bond strength; Bioactive glasses.

e o grupo BIN os maiores valores (74,77 ± 13,56) ($p < 0,05$). Uma energia de superfície significativamente menor foi observada com o BIN (36,22) ($p <0,05$) em comparação com os grupos TER (44,7) e CAL (46,2) ($p> 0,05$). O grupo BIN (9,12 ± 3,45) apresentou resistência de união média significativamente menor ($p <0,05$) do que a de TER (11,56 ± 5,67) e CAL (12,66 ± 4,34) ($p> 0,05$). A maioria dos modos de falhas observados em todos os grupos eram de tipo misto de falhas. **Conclusão:** O biovidro utilizado no Calciplus LC não afetou negativamente a molhabilidade, energia superficial e propriedades adesivas.

PALAVRAS-CHAVE

Adesão; Materiais biocompatíveis; Biovidro; Capeamento de polpa dentária; Resitência ao cisalhamento; BAG.

INTRODUCTION

Pulpal vitality is important for continuity of structural integrity and normal physiological function of teeth. In this regard, vital pulp therapy aims for protecting the exposed dental-pulp complex and maintain pulp vitality following carious removal or traumatic injury [1-3]. Pulp capping materials used for such therapies protect the pulp complex from thermal, chemical, and other noxious stimuli (i.e. bacterial toxins) [4].

Photo-polymerizable resin-based pulp capping materials are indicated for use as liners under resin composite restorations through which adhesion could be achieved between multiple material layers, thus reducing microleakage. Such materials could be considered as ideal pulp-capping materials as they allow for co-polymerization with the subsequent resin composite filling material. The Photo-polymerized systems proposed so far are characterized with alkaline pH, calcium ion release, formation of apatite and functional groups able to chelate calcium ions. In addition, therapeutic re-mineralization of dentine has also been observed [5-7].

Biomaterials used in pulp-capping therapy should possess characteristics such as biocompatibility, pulp vitality preservation, adherence to dentin and restorative material, and resistance to forces during restoration placement and function [8]. Bioactive glasses (BAG) are one of the most highly investigated biomaterials and include SiO_2, Na_2O, CaO and P_2O_5. BAGs have shown to have antimicrobial effects on oral bacteria and promising effects on hard/pulp tissue regeneration [9,10]. The antimicrobial and potential re-mineralizing effects of BAGs are attributed in part to the release of ions (i.e. calcium and phosphate) [9,10] and are added to various dental materials including fissure sealants, resin composites, bone or dental tissue scaffolds, and regenerative endodontic materials such as photo-polymerized pulp-capping materials [11,12].

Adhesion of restorative materials to pulp-capping agents is essential for the long-term success of the restoration following pulp-capping. Optimal wettability is important to enable the adhesive system to spread across the entire surface of pulp-capping material and to establish durable adhesion [13]. The strength of the bond formed between the resin-based pulp-capping materials and resin composite depends on several factors, including the characteristics and wetting of the adherent surface. For this purpose, contact angle measurements on hard surfaces provide information about surface-free energy in relation to bonding characteristics of the solid materials [14,15]. Surface free-energy of a solid is defined as an indicator of surface wettability and comprises dispersion forces, polarity forces, and hydrogen-bonding forces [16,17]. While dispersion forces are one aspect of van der Waals forces, polarity forces represent electric/metallic interactions and dipolar interactions, and hydrogen-bonding forces indicate surface hydrophilicity [16,17].

The objectives of this study were to evaluate the wettability and adhesion potential of different photo-polymerized resin-based pulp capping materials with or without BAG in their composition and classify failure types after debonding. The null hypothesis tested was that resin-based pulp-capping materials with or without BAG would not show significant difference in terms of wettability and adhesion.

MATERIAL AND METHODS

Specimen preparation

Fifteen specimens (n=5 per group) were form three different photo-polymerized resin-based pulp-capping materials (20 x 2 mm) (TER-TheraCal LC, BIN-Biner LC, and CAL-CalciPlus LC). The chemical composition and manufacturers of the materials tested are listed in Table 1. All materials were prepared following manufacturer instructions, and photo-polymerized for 40 s (Elipar, 3M ESPE, St. Paul, USA).

Table 1 - Brands, abbreviations, types, manufacturers and chemical composition of the materials used in the study

Brand	Material Type	Manufacturer	Chemical Composition
TheraCal LC (TER)	Photo-polymerized resin-modified calcium silicate	Bisco Inc, Schaumburg, USA	Aerosol 8%, biocompatible hydrophilic resin 42.5% (bis-GMA 20%, biocompatible resin-77.25%, modifying agent 2.4%, initiating agent 0.32%, stabilizer for the initiating agent 0.032%), active ingredients in MTA 44.5%, and barium sulphate 5%
Biner LC (BIN)	Photo-polymerized calcium hydroxyphosphate	Meta Biomed Inc, Horsham, USA	Diurethanedimethacrylate, hydroxyapatite, triethyleneglycoldimethacrylate, aerosol, barium aluminium borosilicate
CalciPlus LC (CAL)	Photo-polymerized calcium hydroxyphosphate	Imicryl Inc, Konya, Turkey	Resin Matrix: Urethane dimethacrylate, ultra lowshrinkage monomer, photoinitiator, stabilizers Filler: Ultrafinebioactive glass (BAG), fluoroaluminasilicate glass, antibacterial nano composite filler

Contact angle and surface free energy measurements

Contact angles (θ) were determined with the sessile drop method using three test liquids: distilled water, 1-bromonaphthalen and ethylene glycol, each of which has previously identified surface free energy values [18]. The contact angle measurement device (Drop Master DM500, Kyowa Interface Science Co., Saitama, Japan) was fitted with a charge-coupled camera that allowed automatic measurements of contact angles. For each test liquid, the equilibrium contact angle (θ) between the droplet and the solid surface was measured ata specific period time (10 s) at 23 ± 1 °C for each specimen. The value at which the water droplet first touches the surface was recorded as the maximum contact angle (θ) and the value after spreading at the end of 10 s was recorded as the minimum contact angle (θ), and the mean of these measurements was calculated. The procedure for measuring contact angle was the same for all groups and was performed by the same calibrated operator.

The surface free energy was then Measured based on the fundamental concepts of wetting. The Young-Dupré equation describes adhesion of a solid (S) and a liquid (L) in contact (WSL) where the interfacial free energy between the solid and the liquid (γSL), and the surface free energy of the liquid and the solid (γL and γS, respectively) are calculated as follows:

$$WSL= \gamma L+ \gamma S-\gamma SL = \gamma L\ (1+ \cos\theta)$$

By extending the Fowkes equation, γSL was expressed as follows:

$$\gamma SL= \gamma L+ \gamma S - 2\ (\gamma L^{d}\bullet\gamma S^{d})^{1/2}-2\ (\gamma L^{p}\bullet\gamma S^{p})^{1/2} - 2\ (\gamma L^{h}\bullet\gamma S^{h})^{1/2}$$

$$\gamma L= \gamma L^{d} + \gamma L^{p} + \gamma L^{h},\ \gamma^{S} = \gamma S^{d} + \gamma S^{p} + \gamma S^{h},$$

where γLd, γLp, and γLh were components of the surface free energy (γ) arising from dispersion forces, polar (permanent and induced) forces, and hydrogen bonding forces, respectively. θ values between the droplet and the solid surface were determined for the three test liquids, and surface free energy parameters were calculated from the measured θ values, using add-on software and an interface measurement and analysis system (FAMAS, Kyowa Interface Science, Niiza-City, Japan) [19].

Shear Bond Strength Test

For this test, cylindrical acrylic blocks (N = 30) were prepared with holes of 6 mm diameter and 1 mm height in the center of the cylinders. The blocks were filled with the pulp-capping materials (N=10 per group) following manufacturers` instructions. The surfaces were flattened using a metal spatula and photo-polymerized with an LED photo-polymerization unit for 20 s (Elipar).

The specimens were stored at 37°C in 100% humidity for 24 h prior to the bonding procedures. An adhesive system (CLEARFIL SE BOND, Kuraray, Okayama, Japan) was applied on the material surfaces and photo-polymerized for 20 s. Following application of the adhesive system, a restorative resin composite material (Filtek Z250, 3M ESPE) was bonded on the substrate materials using translucent plastic moulds (internal diameter: 2 mm; height: 2 mm). The specimens were photo-polymerized with an LED photo-polymerization unit for 20 s (Elipar). After polymerization, the plastic moulds were removed carefully, and the specimens were stored at 37 °C, at 100% humidity for 24 h.

The bonded specimens were mounted in the jig of the Universal Testing Machine (Instron Model-8500 Plus Dynamic Testing System-1341 Instron-Instron Corp., Canton, MA, USA). Shear force was applied using a knife-edge blade between pulp-capping material and the resin composite at a cross-head speed of 1 mm/min. The force required to debond was measured in Newtons (N) (1 MPa=1 N/mm^2) and the bond strength was calculated by dividing the maximum load value by the restorative material base area (3.14 mm^2).

Failure type analysis

Failure modes in each group were evaluated by one operator using polarized light microscope (NexiusZoom, Euromex, Arnhem, Germany) at x40 magnification and categorized as follows: Adhesive failure: Failure between the pulp-capping material and the resin composite with no resin remnants on the substrate; Cohesive failure: Failure within the pulp-capping material or resin composite; Mixed failure: Combination of both adhesive and cohesive failure.

Statistical analysis

The data were statistically analyzed using the software SPSS (SPSS Version 19.0, IBM, Chicago, USA). The Shapiro-Wilk test was used to analyze normality distribution of data. One-way analysis of variance (ANOVA) and Tukey`s post hoc tests were used to determine significant differences among the material groups. $P < 0.05$ was considered as statistically significant in all tests.

RESULTS

Mean contact angle values showed significant difference between the 3 materials with group CAL presenting the lowest (35.35 ± 12.89) and group BIN the highest values (74.77 ± 13.56) ($p < 0.05$) (Table 2). Significantly lower surface energy was observed with BIN (36.22) ($p < 0.05$) compared to those of the groups TER (44.7) and CAL (46.2) which were not significantly different from one another ($p > 0.05$).

Group BIN (9.12 ± 3.45) showed significantly lower ($p < 0.05$) mean bond strength than those of TER (11.56 ± 5.67) and CAL (12.66 ± 4.34) (Table 3). No significant difference was observed between TER and CAL ($p > 0.05$). Most of the observed modes of failures in all groups were of mixed type of failures.

Table 2 - The minimum, maximum, mean contact angle and surface free energy results. Different lower superscript letters in one column indicate significant differences ($p < 0.05$). See Table 1 for group abbreviations

Pulp Capping Materials	Contact Angle			Surface Energy
	Min	Max	Mean	(Ytotal)
TER	51.72±12.67	82.35±9.78	55.14±11.12b	44.70^{a}
BIN	74.39±10.45	77.07±14.77	74.77±13.56a	36.22^{b}
CAL	28.9±12.56	79.12±12.43	35.35±12.89c	46.20^{a}

Table 3 - The mean±standard deviation of shear bond strength values of rein composite to photo-polymerized pulp capping materials and distribution of failure types. Adhesive failure: Failure between the pulp-capping material and the resin composite with no resin remnants on the substrate;Cohesive failure: Failure within the pulp-capping material or resin composite;Mixed failure: Combination of both adhesive and cohesive failure. Different lower superscript letters in one column indicate significant differences ($p<0.05$). See Table 1 for group abbreviations

Pulp Capping Materials	Shear Bond Strength (MPa)	Type of Failures		
	Mean±SD	Adhesive	Cohesive	MIxed
TER	$11.56±5.67^{a}$	2	3	5
BIN	9.12±3.45b	3	3	4
CAL	12.66±4.34a	4	2	4

DISCUSSION

This study investigated the wettability and adhesion potential of different photo-polymerized resin-based pulp capping materials with or without BAG in their compositions.

Contact angle and surface free energy can be correlated to specific properties of resin-based materials including ability to adhere to dental tissues or dental materials and bacterial colony adhesion to their surface [20]. The contact angle reflects interactions of fluids with solid surfaces, which depends on surface topography, hydrophobicity and wettability, surface tension of the liquid, surface energy of the substrate, and level of interaction between liquid and solid in the components involved. Measuring contact angle at the solid-air-liquid interface is one of the fundamental parameters used to investigate wettability of solid substrates [21,22].

The contact angle for liquids tends to be high if the substrate is hydrophobic. When the surface is hydrophilic, the droplet quickly dissipates, and the measured contact angle becomes low. Similar to other methods, the contact angle measurement method provides an average value of hydrophobicity [23]. Hydrophobicity, an important characteristic of dental resin-based materials, affects liquid absorption and adhesion of oral bacteria [24]. Breakdown of margin areas between enamel and restorative materials can provide potential pathways for bacterial invasion and recurrence of caries. Resin-based materials may be covered with an organic biofilm, that usually consists of host and bacterial elements. Differing chemical properties and surface topographies of various materials may play a role in wettability, bond strength, or biofilm formation, and is directly affected by the composition along with other properties [21,25].

Bond strength is one of the most important parameters in adhesive dentistry since stability of adhesion between the resin-based materials and tooth structure/resin materials contributes directly to long term clinical success [26]. On the other hand, wettability is important in order to enable adhesive materials to spread across the entire solid surface and establish adhesion. Low contact angle and high surface energy are associated with maximum wetting of the adherent surface by an adhesive system in solid substrates [27].

Takimoto et al., investigated the influence of temporary cement contamination on surface free energy of dentin surfaces [28] where application of both eugenol-containing and eugenol-free temporary cements reduced surface free energy of dentin. Temporary cement cannot be completely removed from the dentin surface, and such remaining residue might alter wettability of the dentine surface leading to changes in surface free energy. Depending on the reduction in surface energy of dentin, its wettability decreases and bond strength of the adhesive systems with dentine decreases [28]. Tjisumoto et al., on the other hand reported that a thin layer of contaminant remains on the zirconia ceramic surface after exposure to saliva, significantly impeding bonding of the resin cement and reducing surface free energy of the ceramics [19]. Likewise, Kondo et al. investigated surface properties of PMMA-containing resin composites and reported that contact angle of the experimental materials tended to increase with incorporation of PMMA fillers in the resin composite, thus increasing surface hydrophobicity of the material [29]. According to published literature [19,28-30], materials with contact angle above 65° are considered highly hydrophobic. High hydrophobic character of the tested materials in the present study was also reflected in surface energy values in which were above 30 mJ m^{-2}.

The BIN group presented the highest contact angle value (74.77 ± 13.56°) and the lowest surface free energy (γ: 36.22). These results indicate that this material has highly hydrophobic as per Vogel's et al. interpretation [31]. Unlike other photo-polymerized resin-based materials used in this study, BIN contains hydroxyapatite particles (10 w%) as active material and diurethanedimethacrylate (32 w%) as resin matrix monomer. It could therefore be anticipated that these differences in the composition led to increased hydrophobicity compared with the other materials tested (TER and CAL) with which more hydrophilicity was observed. Changes in contact angles and surface free energy could have resulted from the polymer (aromatic structure) and/or from the inhomogeneous surface caused by the fillers [32]. Bis-GMA monomers used in TER and ultra-low shrinkage monomers used in CAL confer hydrophilic properties of these materials. Hydrophilic property increases wettability, allowing the adhesive system to spread better on the solid surface causing stronger binding between the resin material and the pulp-capping agent. Thus, the results support our null hypothesis that surface free energy and bond strength of resin-based pulp-capping materials would not be affected by addition of BAG. Despite major advances in adhesive technologies, bonding interface in tooth structure remains the weak point in some adhesively bonded restorations. Quality and durability of bonding between pulp-capping materials and restorative materials is of clinical significance in terms of longevity and predictability of final restoration [33].

The most commonly used test to assess bond strength is the macro-shear test [34]. Although testing assembly has great influence on stress distribution, and the use of a knife-edge chisel may cause stress concentration at the load application area, compared to wire loop, in order to make comparisons with similar studies using the materials tested in this study, a knife-edge chisel was used. The obtained results should be verified using the wireloop in future studies.

Alzraikat et al. studied the bond strength of TER, MTA and GIC with a resin composite (Filtek-Z 250) where TER displayed significantly the highest bond results which was in part attributed to the resin content in TER that resulted in chemical bonding with the resin composite [35]. In another study, also TER showed significantly higher microshear bond values compared to Biodentine [36] where higher bond strength of TER was attributed to the presence of dimethacrylate monomer in its composition and thereby the adhesion to the resin composite. Likewise, Cantekin et al. evaluated the bond strength of methacrylate- and silorane-based composites in comparison with TER and MTA where TER significantly increased the bond strength for the methacrylate-based composite [37]. The silorane-based composite is based on ring-opening polymerization of silorane molecules containing both hydrophobic siloxane and low shrinkage oxirane, rather than free radical polymerization of dimethacrylate monomers in methacrylate-based composites. This hydrophobic structure may have yielded to weak bond strength [37].

In this study, bond value of CAL (12.66 ± 4.34 MPa) with resin composite was the highest among all tested groups with no significant difference to TER. Studies have reported that TER contains a hydrophilic resin monomer that makes it an excellent adhesion promoter enhancing bond strength [5,38,39]. TER and CAL have in fact similar resin matrix monomer. Therefore, the higher bond strength could be due to the matrix monomer (hydrophilic structure) when compared to BIN.

Studies have estimated that bond strength ranging from 17 to 20 MPa may sufficiently resist contraction forces and produce gap-free restoration margins [35,37,40]. In this regard, the photo-polymerized resin-based materials used in this study did not demonstrate adequate bonding strength. For all materials, the failure modes were mostly mixed or adhesive also indicating insufficient bond between the materials tested.

The hydrophilic nature of TER and CAL resulted in better bond strength in these materials and the presence of BAG in the composition of the latter did not impair adhesion but future studies should focus on employing surface conditioning methods in conjunction with other adhesives aiming for improved adhesion after aging conditions.

CONCLUSION

From this study, the following conclusions were drawn:

1. Hydrophilic nature of Theracal LC and Calciplus LC, verified with lower contact angle values, resulted in higher bond strength compared to Biner LC;

2. The bioglass used in Calciplus LC did not adversely affect the wettability, surface energy and adhesive properties;

3. The less incidence of cohesive failures still do indicated potential for improvement in adhesion between the tested pulp-capping materials and the resin composite.

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CONFLICT OF INTEREST

All authors declare no conflict of interest in this study.

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