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Dentin bonding performance of experimental one-step adhesives after incorporation of POOH–SiO₂ nanoparticles

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Abstract

This study investigated the effect of adding different concentrations of phosphate-grafted-silica nanoparticles (SiO₂–POOH) on dentin bond strength of experimental one-step self-etching adhesives (1-SEAs). Phosphate functional group was grafted onto silica nanoparticles through the free radical polymerization of an acid phosphate monomer [methacryloyloxyethyl dihydrogen phosphate/bis (methacryloyloxyethyl) hydrogen phosphate—HEMA-P] in an alcoholic medium. The resulting SiO₂–POOH was characterized by FTIR spectroscopy. 1-SEA with two different acidic phosphate monomer concentrations (HEMA-P at 0 and 40% wt) were prepared and SiO₂–POOH was added in 0 (control), 0.1, 0.2, 0.5, 1, 2, 4, 6 and 8wt%. The formulated 1-SEAs were evaluated for shear bond strength to dentin. Statistical comparisons were conducted at 5% significance level. The grafting reaction of SiO₂–POOH was confirmed by FTIR. Compared with the unfilled 1-SEA, the addition of 0.1wt% of SiO₂–POOH nanoparticles resulted in a significant dentin bond strength increase for HEMA-P_{40%} concentration ($p \leq 0.001$) while the addition of higher concentration of SiO₂–POOH (0.2, 0.5, 1, 2, 4, 6 and 8wt%) reduced significantly the bond strength. The 1-SEA formulated without an acidic monomer (HEMA-P_{0%}) did not exhibit bonding to the dentin, irrespective of SiO₂–POOH concentration added. SiO₂–POOH nanoparticles were obtained through graft polymerization of acidic phosphate monomer with silanized SiO₂ nanoparticles. The addition of the 0.1wt% of SiO₂–POOH nanoparticles associated with acidic monomer to 1-SEA improves the bonding effectiveness.

Keywords: Dental adhesives, Dentin bonding agents, Filler, Graft polymerization, Acidic monomers, Bond strength

Background

The bonding effectiveness to dental tissue is related to a material's ability to promote demineralization of the substrate, resin infiltration, and form a hydrolysis-resistant hybrid layer (a polymer setting and collagen network) [1]. In self-etch adhesives, the acidic methacrylate monomer promotes physical–chemical interactions between the adhesive and dental tissue by micro-mechanical interlocking and additional chemical interaction [2]. The phosphate acidic functional groups of its monomers could interact with calcium ions of hydroxyapatite and improve the bond durability [2, 3]. Thus

a reliable bonding depends on the mechanical and chemical behavior of the polymer formed.

However, this acidic monomer affect the polymerization reaction [4, 5] and become hydrolytically unstable in one-bottle materials, [6–8] thus increasing the water sorption and decreasing the mechanical properties of the polymer [1]. Such increase in water affinity allows water movement through the adhesive layer [9, 10], and as consequence small droplets can be found at the transition layer between adhesive and lining composite [11]. This phenomenon promotes a decrease in bond strength and seems to contribute to the hydrolysis of the adhesive layer and consequently degradation over time [12]. This hybrid layer is considered the weakest spot of an adhesive restoration and, thus, is more likely to fail [1, 10, 13]. Therefore, improving the material's properties and enhancing the physical–chemical interaction of tooth–adhesive will lead to a more durable restorative treatment [1, 2].

Several commercial adhesives systems present filler particles in their constitution [14, 15]. It has previously demonstrated that the addition of nanoparticles (NPs) to adhesives increases the mechanical properties and bond strength [16–19]; however, specific nanoparticle dispersion state is necessary to optimize these properties. Grafting is an effective method to control the NPs dispersion in a polymer matrix, as well as to give specific surface characteristics [20]. Therefore, the aim of this study was to evaluate the effect of adding different concentrations of phosphate acidic functional groups grafted on silica nanoparticles surface ($\text{SiO}_2\text{-POOH}$) on dentin bond strength of experimental one-step self-etching adhesives (1-SEAs). The hypothesis tested was that the incorporation of $\text{SiO}_2\text{-POOH}$ improved the bonding strength of the 1-SEA.

Methods

Reagents

The silica nanoparticles (SiO_2 -Aerosil[®] 380/7 nm average size, Degussa, Germany) were silanized by gamma-methacryloxypropyltrimethoxysilane (γ -MPTS—Aldrich Chemical Co., USA) and grafted by acidic phosphate monomer (methacryloyloxyethyl dihydrogen phosphate/bis (methacryloyloxyethyl) hydrogen phosphate—HEMA-P) that was synthesized as previously described [21]. Acetic acid, benzoyl peroxide (BPO), acetone, and ethanol were purchased from Vetec, Brazil and used as received. Distilled water was used throughout all of the experiments. The experimental 1-SEAs were formulated by monomers 2,2-bis[4-(2-hydroxy-3-methacryloyloxy propyl)phenyl]-propane (Bis-GMA), (di)methacrylate monomers urethane dimethacrylate (UDMA), 2-hydroxyethyl methacrylate (HEMA) and trietilenglicol dimethacrylate (TEGDMA) which were supplied by Esstech Inc., USA, and used as received. The photosensitizer, camphorquinone (CQ-Esstech), as well as the co-initiator ethyl 4-(dimethylamino) benzoate (EDAB) and diphenyliodonium hexafluorophosphate (DPIHFP), were purchased from Aldrich Chemical Co, USA, were used to render the materials photocurable.

Graft polymerization of phosphate onto the SiO_2 nanoparticle

In a 500-mL round-bottom vessel connected to a reflux condenser, γ -MPTS and acetic acid (10 and 15% wt of the nanoparticles wt, respectively) were dissolved in 15 mL of distilled water and 150 mL of ethanol. 5 g of SiO_2 was added to this mixture. The

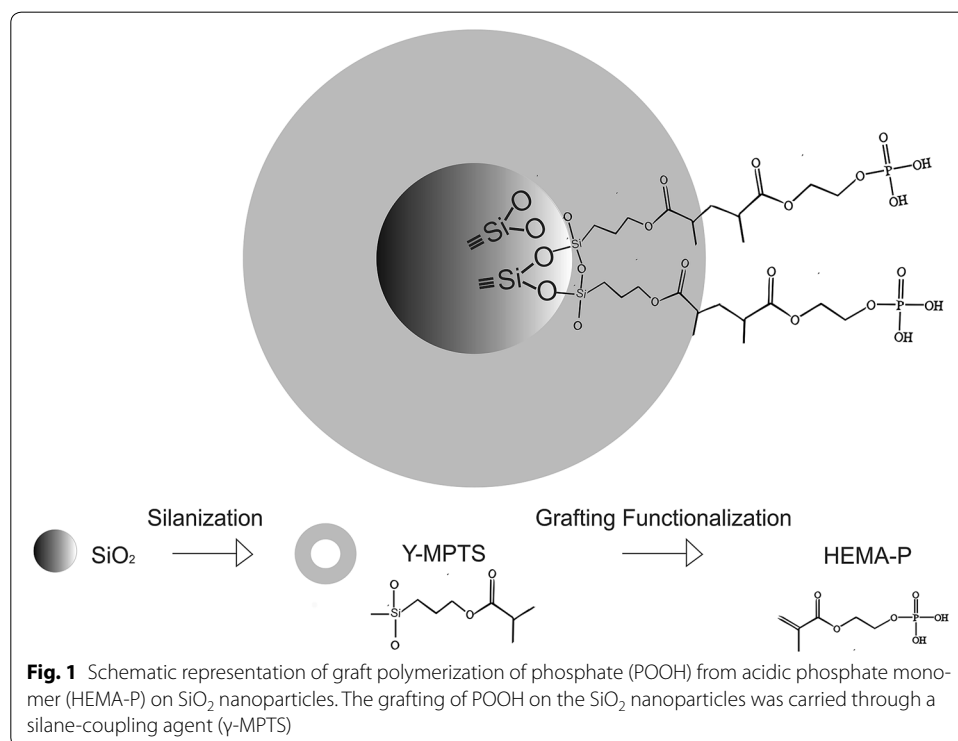
temperature was held at 80 °C for 24 h while being vigorously stirred. SiO₂ silanization was confirmed through appearing the C=C signal at 1636 cm⁻¹ in the Fourier transform mid-infrared (FTIR) spectrum. The acidic phosphate monomer HEMA-P and BPO as radical initiator (15 and 1% wt of the nanoparticles wt, respectively) were then added to the reaction medium. The temperature was maintained at 80 °C while stirring. The polymerization reaction was left to take place for 24 h. The product was washed with acetone three times to remove the unreacted monomers and dried at room temperature for 24 h, resulting in silica powder with phosphate-functional groups at their surface (SiO₂-POOH). Schematic representation of graft polymerization of phosphate onto the silanized SiO₂ nanoparticle is summarized in Fig. 1.

Particles' characterization

The SiO₂ and SiO₂-POOH were analyzed by Fourier transform mid-infrared (FTIR) spectroscopy (Prestige-21; Shimadzu, Japan) at a resolution of 4 cm⁻¹ and 24 scans in the range of 700–2500 cm⁻¹. The SiO₂ was used as background before the SiO₂-POOH analysis to avoid overlapping the peaks between 500 and 1500 cm⁻¹.

Experimental one-step self-etch adhesive (1-SEA) formulations

A model 1-SEA was prepared through mixing of Bis-GMA (42.5% wt), HEMA (38% wt), TEGDMA (8.5% wt), UDMA (8.5% wt); and a ternary initiation system CQ (0.5% wt), EDAB (1% wt) and DPIHFP (1% wt). 1-SEA with two different acidic phosphate monomer concentrations (HEMA-P at 0 and 40% wt) were prepared. SiO₂-POOH was added to the 1-SEA in 0 (control), 0.1, 0.2, 0.5, 1, 2, 4, 6 and 8 wt%. To perform monomer photoactivation, a light-emitting diode unit (LED-Radii; SDI, Australia) was used. An



irradiation value of 1400 mW/cm^{-2} was confirmed with a power meter (Ophir Optronics, USA).

Shear bond strength to dentin

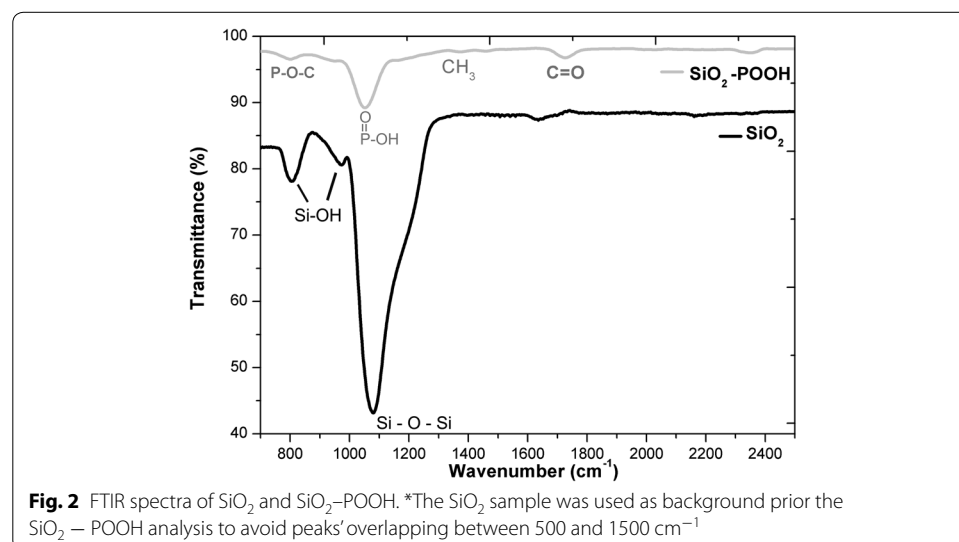
Sixty bovine incisors were embedded in epoxy resin and their buccal faces were wet-ground to expose the dentin ($n = 10$). The dentin surfaces were wet-polished with 600-grit SiC abrasive papers for 60 s. Dentin moisture was controlled with absorbent paper until no surface water was observed. The 1-SEAs were vigorously applied to the prepared dentin surfaces for 20 s by using a microbrush; elastomer molds (thickness 0.5 mm) with a cylindrical orifice (diameter 1.5 mm) were placed onto the adhesive layer and light-activated for 30 s. The orifices were filled with the resin composite (Filtek Z250, 3 M ESPE, USA) to produce cylindrical specimens with bonded area of 1.77 mm^2 ; the molds were covered with a polyester strip and a glass slide and the resin composite was photoactivated for 40 s. After storage in distilled water at 37°C for 24 h, a thin steel wire (diameter 0.2 mm) was looped around the cylinder and aligned with the bonding interface. The shear test was conducted at a crosshead speed of 0.5 mm min^{-1} until failure in a universal testing machine (EMIC DL 500, Brazil). Shear bond strength (SBS) values were calculated in MPa.

Statistical analysis

The SBS data were submitted to Kruskal–Wallis one-way Analysis of Variance and the Student–Newman–Keuls test ($p < 0.05$).

Results

Figure 2 shows the FTIR spectra of the SiO_2 -POOH illustrating the characteristic peaks of the POOH functional groups which confirms the grafting process. SiO_2 -POOH spectrum shows the presence of peaks at about 801 and 1053 cm^{-1} , which are assigned to the POOH functional group [21]. Also, spectrum shows peaks at 1730 cm^{-1} , which



corresponds to the methacrylate C=O stretching vibrations and peaks at 1374 and 1379 cm^{-1} , which are assigned the CH_3 bending deformation of the polymer grafted backbone.

The effect of the SiO_2 -POOH nanoparticles concentration on the dentin bond strength of 1-SEA is shown in Table 1. Compared with the unfilled 1-SEA, the addition of 0.1wt% of SiO_2 -POOH nanoparticles resulted in a significant dentin bond strength increase for HEMA-P_{40%} concentration ($p \leq 0.001$). SiO_2 -POOH-containing adhesives in higher concentration (0.2, 0.5, 1, 2, 4, 6 and 8wt%) showed a significantly lower bond strength as compared with the HEMA-P_{40%}/ SiO_2 -POOH_{0.1%} and unfilled 1-SEA. The 1-SEA formulated without an acidic monomer (HEMA-P_{0%}) did not exhibit bonding to the dentin, irrespective of SiO_2 -POOH concentration added, and hence it was not represented in table. Special interpretation of the SBS-values was performed, in case of a pre-testing failure, shear bond strength value of zero MPa was assigned to specimen, and this data was included into the statistical analysis.

Discussion

The grafting of POOH on the SiO_2 nanoparticles was carried through a silane-coupling agent. The SiO_2 was first functionalized with a silane-coupling agent (γ -MPTS) where the hydrolyzed alkoxy silane groups (Si-OH) from γ -MPTS reacts with the hydroxyl groups (OH) from SiO_2 to form covalent siloxane bonds (Si-O-Si-). Remaining methacrylate end functional groups provides active sites for graft copolymerization [22]. The polymerization of the acidic phosphate monomer (HEMA-P) in the presence of silanized SiO_2 particles, and a free radical initiator, PB, leads to graft polymerization involving the acrylate groups of γ -MPTS and HEMA-P.

The covalent linkage between acrylates groups of γ -MPTS and HEMA-P occurs by breaking of the double bond C=C during the free radical polymerization. The percentage of HEMA-P used (15% wt) correspond a 2:1 molar ratio of γ -MPTS linked to SiO_2 nanoparticles, which ensures complete graft polymerization between both monomers. The absence of the peak at 1636 cm^{-1} due to stretching vibration of the C=C [23] is an evidence of this polymerization process in SiO_2 -POOH. Other characteristic peaks appears at 801 and 1053 cm^{-1} , which correspond to aliphatic binding of P-O-C and

Table 1 Median (95% of confidence interval) for shear bond strength varying SiO_2 -POOH nanoparticles concentration (wt%) of 1-SEA with HEMA-P_{40%}

HEMA-P _{40%} / SiO_2 -POOH concentration, wt% (%)	Dentin bond strength, MPa
0	6.01 (1.54) ^b
0.1	8.63 (1.45) ^a
0.2	5.56 (2.13) ^c
0.5	2.37 (2.55) ^d
1	2.53 (0.90) ^e
2	5.56 (1.80) ^c
4	1.07 (1.49) ^e
6	3.54 (2.19) ^d
8	4.19 (1.88) ^d

Values followed by distinct superscript letters indicate significant differences for 1-SEAs tested ($p < 0.05$)

Note: It was not possible to test any specimens for HEMA-P_{0%} as all pre-testing failure

P–OH functional groups of HEMA-P structure [21]. Representative peaks at 1730, 1374 and 1379 cm^{-1} which corresponds to free carbonyl stretching vibration (C=O) and to CH_3 bending deformation can confirm the presence of a polymer backbone grafted to the NPs. Also, as SiO_2 sample was used as background prior the SiO_2 –POOH analysis, any of Si–O–Si at 1098 cm^{-1} and Si–OH at 793 cm^{-1} linkages could not appear into the SiO_2 –POOH spectrum and therefore, it is expected that only linkages corresponding to the –POOH backbone grafted to the SiO_2 surface could be observed [24].

The addition of 0.1wt% of SiO_2 –POOH nanoparticles to 1-SEA improved the bonding strength to the dentin, except for 1-SEA formulated without an acidic monomer (HEMA-P_{0%}) did not exhibit bonding to the dentin, irrespective of SiO_2 –POOH concentration added (data no showed). Therefore, the tested hypothesis should be partially accepted. Self-etch adhesives require acidic functional monomers, such as HEMA-P, to simultaneously condition and prime the dental substrate [2]. In this study, experimental groups without HEMA-P did not exhibit any bonding to dentin because there was not any acidic functional monomer in these 1-SEA's which could promote demineralization and infiltration of the adhesive system. Also, it was possible to confirm that phosphate functional groups grafted on SiO_2 nanoparticles does not act as a self-etching agent.

The addition of 0.1wt% of SiO_2 –POOH nanoparticles in 1-SEA with HEMA-P, in contrast, resulted in an increased adhesion to dentin. The HEMA-P_{40%}/ SiO_2 –POOH_{0.1%} showed significantly higher bond strengths ($\approx 50\%$) than the correspondent unfilled 1-SEA HEMA-P_{40%}. Previous studies have already been proven that the addition of an optimum amount of filler into adhesives increases the mechanical properties and bond strength [18, 19, 25]. The increase in these properties is due an increment of the elastic modulus of the adhesive layer between dentin and restoration. This adhesive layer acts as an elastic buffer which offers the resin–dentin interface a sufficient strain capacity to resist higher debonding forces [26]. Also, it could be theorized that phosphate functional groups attached to solid SiO_2 particles could probably promoted an additional chemical interaction with Ca^{2+} ions of the hydroxyapatite from the dentin [2]. In addition, since it's well known the high hydrophilicity of phosphate acid groups [27], improved dentin bond strength could be achieved due an increase in dentin wettability by 1-SEAs.

The present results indicate that the maximum increases of shear bond strength occurred only when 0.1wt% of SiO_2 –POOH was added in 1-SEA with HEMA-P, the increase of filler above this concentration lead to a decrease of dentin bond strength. Correlation for the decreasing of the bond strength to the increasing of SiO_2 –POOH nanoparticles concentration, however, was not found. Surprisingly some groups e.g. 8wt% of SiO_2 –POOH presented higher results than 1 and 4wt% of SiO_2 –POOH. Two possible phenomena could explain this behavior, first the agglomeration of the nanoparticles and subsequently accumulation of these filler agglomerates on the top of the conditioned dentin prevent the resin infiltration into the dentinal tubules; and on the other hand, higher filler concentrations increase significantly the viscosity of the adhesive systems, which also reduces the penetration of the 1-SEA [15]. Both mechanisms seem to prevent optimal adaptation of the adhesive into the dentin surface, and consequently bond strength and marginal integrity is compromised [15]. Also, it was shown that fillers into adhesive systems yield thicker adhesive layers, which can affect its capacity to relief contraction stresses produced by the restorative composite [26]. This is in agreement

with other studies that also showed a high level of bond strength to dentin with the addition of a small amount of filler to the adhesive [16, 18, 19, 28]. These effects, however, should be addressed in future studies. The improvement bonding efficacy of 1-SEA's modified with SiO₂-POOH nanoparticles, under clinical conditions, could improve the clinical service of restorations, as well as render a clinical procedure simpler, more user-friendly, and less technique-sensitive.

Conclusion

SiO₂-POOH nanoparticles were obtained through graft polymerization of acidic phosphate monomer with silanized SiO₂ nanoparticles. The addition of the 0.1wt% of SiO₂-POOH nanoparticles associated with acidic monomer to experimental one-step self-etching adhesives improves the bonding effectiveness.

Authors' contributions

CTWM, CECS, SMA and CMA confectioned the specimens, performed the mechanical test, conducted the statistical analysis and write the manuscript. FMC, EP and FAO delineated the experiment and help in the manuscript written. All authors read and approved the manuscript.

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Competing interests

The authors declare that they have no competing interests.

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