Applied Surface Science 274 (2013) 248–254

Contents lists available at SciVerse ScienceDirect

Applied Surface Science

journal homepage: www.elsevier.com/locate/apsusc

Effect of surface modification of fiber post using dopamine polymerization on interfacial adhesion with core resin

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ARTICLE INFO

Article history:
Received 3 January 2013
Received in revised form 6 March 2013
Accepted 7 March 2013
Available online 13 March 2013

Keywords:
Glass fiber post
Polydopamine
Surface modification
Core resin
Interfacial adhesion

ABSTRACT

The purpose of this study is to evaluate the effects of surface modification of fiber posts using dopamine polymerization on their interfacial adhesion with core resins. The fiber posts were surface-coated with polydopamine via the oxidization polymerization of dopamine in aqueous solution. Two commercial composite resins (3M ESPE and paracore) were used to build up the cores around the post heads (modified and unmodified). Pull-out tests were conducted, and the maximum failure load (N) and the failure modes were recorded to compare the interfacial adhesion between fiber post and resin core. The results demonstrated that the tensile forces needed to damage the retention of fiber post increased from 228.6 ± 10.9 N to 276.3 ± 14.7 N in the 3M ESPE group, from 216.5 ± 17.4 N to 277.2 ± 14.3 N in the paracore group, when polydopamine-coated fiber posts were applied. No significant difference had been found between the different resin groups. The observation of the surface morphology of both fiber posts and cores after adhesive failure clearly confirmed that the presence of polydopamine interlayer had acted as a binder to bond fiber post and resin together. This study would be valuable for endodontically treatments to reduce the chances of detachment of resin core from the fiber post or dislodgement of fiber posts from the canal.

1. Introduction

Dental treatment is one of the most frequent medical treatments performed upon human beings. In case of severely damaged teeth lacking adequate structure to support restoration, a post and core is usually required for endodontically treated teeth [1]. With the root fracture has been reported to be the most severe cause of failure for endodontically treated teeth with using cast metal post-and-cores, the choice of post materials has changed to fiber-reinforced posts since the 1990s due to their optimal esthetics, metal free and mechanical characteristics similar to dentin [2–4].

Theoretically, a mechanically homogeneous unit can be created with the system of fiber posts (16–40 GPa), composite resins (5.7–25 GPa) and dentin (18.6 GPa) [5]. The fiber post can act as a shock absorber by transmitting only limited stress to the residual tooth structure to reduce the risk of root fracture [6,7]. And, the low stress in the interface area is an additional advantage to preserve the interfacial adhesion. However, tooth-threatening failures like the dislodgement of fiber posts from the canal and the detachment of resin core from the fiber post were reported occasionally [8–10].

In many cases, interfacial failure was attributed to chemical incompatibility [11]. The durability of a composite core restoration depends on the formation of a strong bond between the resin composite and the fiber post. The polymeric matrix of fiber posts is usually made of highly cross-linked epoxy resin [12]. When core resins containing methacrylate groups bonding to glass fiber posts, there is a complete lack of polymerizable groups in the epoxy resin to permit free radical polymerization with core resins.

Surface treatments are common methods for improving the general adhesion properties of a material, in which, chemical and micromechanical retention between different constituents is facilitated. To enhance the bond strength at the post-core or post-cement interface, surface pre-treatment was usually employed, involving coating of the post with a silane primer, and/or with a beforehand acid-etching of the post surface [13–16]. Their function was to improve wetting properties of fiber post and to enhance the interfacial bonding strength due to the formation of chemical ridge between the glass phase of the post and the resin matrix. However, the results were still not so satisfactory and the reports often contradictory [17–20]. The authors hypothesized that the water content and acidity of the self-etching adhesive probably might have influenced the bonding to fiber post surface [21].

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http://dx.doi.org/10.1016/j.apsusc.2013.03.027

Taking into consideration that the most frequent cause of failure of bonded fiber posts is de-bonding, the purpose of this study was to evaluate the effect of a kind of bioadhesive on enhancement in the bonding strength of fiber post to resin core.

Superior to most adhesives, bioadhesives are able to form strong and durable bonds in wet environments, especially the adhesive proteins secreted by marine mussels. Mussel adhesive proteins are able to form strong bonds with various kinds of substrates including glass, plastics and metals [22,23]. An unusual amino acid, 3,4-dihydroxy-*L*-phenylalanine (DOPA), has been identified largely responsible for the cohesive and adhesive strengths of mussel adhesive proteins [24]. With the incorporation of certain amounts of DOPA into synthetic polymers, the polymer can adsorb onto different substrate surfaces (glass, stainless steel, polycarbonate, PP, and PTFE) by virtue of the strong anchoring force of the catechol functionality [25]. Thus, the DOPA was speculated a prospective surface modification material for fiber posts to enhance the interfacial bond strength in aqueous environments. The coating process is easy and mild that substrates are soaked in a dopamine solution and mildly stirring at room temperature to result in a polydopamine layer via the oxidative polymerization of dopamine [26]. Substrates including hydroxyapatite, carbon nanotubes, glass, polytetraethylene (PTFE), polyester, silicon rubber etc. have all been surface-coated with polydopamine using the above mentioned method [27–35]. The method would not ruin any structure of the original substrate, and it is extremely useful for biomaterial applications because it does not require the time-consuming synthesis of complex linkers and the process is solvent free and non-toxic. Ku et al. showed that polydopamine surfaces resulting from the oxidative polymerization of dopamine could promote cell adhesion to various non-wetting surfaces, such as polyethylene, PTFE, and polydimethylsiloxane, which are highly resistant to cell adhesion inherently [26]. Therefore, the aim of this paper is to achieve improved bond strengths at the post-core interface with the surface modification of fiber posts using polydopamine via the oxidative polymerization of dopamine.

2. Materials and methods

2.1. Materials

The glass fiber reinforced epoxy resin composite posts were supplied by Beijing Oya Biomaterials Sci. & Tech. Corp., Ltd. The glass fibers were SE8400LS from Owens Corning. The resin matrix was Epoxy-828 from Shell. The diameter of the prefabricated fiber posts was 1.3 mm. Dopamine hydrochloride was purchased from Sigma and used directly for the surface coating modification. The two core resins used in this study came from 3M and Coltène/Whaledent AG, respectively. One was a kind of self-adhesive universal resin cement (Rely X^{TM} U100, 3M ESPE), containing acrylic salt, phosphorylation of acrylic ester and inorganic filler. The other was a kind of dual cured core & resin cement (paracore), containing methyl acrylate, polyene acid ester, glass filler and amorphous silica.

2.2. Coating fiber posts with polydopamine by oxidative polymerization

The polished fiber posts were ultrasonically cleaned in ethanol for 0.5 h before use (50 Hz). To perform the polydopamine coating, the fiber posts were immersed into a dopamine solution (2 mg/ml in 10 mM Tris, pH 8.5) at 25 °C for 14 h under continuous stirring [26,29]. The treated posts were then retrieved and washed with deionized water to remove excess monomer, and then air-dried.

2.3. Characterizations of polydopamine-coated fiber posts

The polydopamine coating was characterized by Fourier Transform Infrared Spectroscopy (FT-IR, Perkin-Elmer, System 2000), X-ray photoelectron spectroscopy (XPS, Thermo V6 ESCALAB 250, Britain) and water contact angle measurement (Surface Electro Optics Co., Korea). FTIR spectra were obtained by accumulating 10 scans with a resolution of 4 cm^{-1} in the range of 650–4000 cm^{-1}. The XPS spectra were obtained by using AlKα radiation at a power of 300 W under vacuum (3×10^{-7} Pa) at an incidence angle of 90°. The water contact angles were determined by automatically dropping 0.25 μL of deionized water onto the sample, and five samples were used for each test. The morphological observations of the samples were conducted by scanning electron microscope (SEM, S-4700, Hitachi) at an accelerating voltage of 20 kV after being sputter-coated with platinum (30 mA, 20 s) using a sputter coater (Polaron E5600, USA).

2.4. Evaluation of bonding strength between fiber post and core resin

2.4.1. Specimens preparation

To prepare the specimens for pull-out test, fiber posts were all cut into 40 mm in length, which was facilitated to the jig size for performing pull-out test (Fig. 1). Resin cores, 6 mm in diameter and 6 mm in height, were built up using commercial 3M ESPE (3M Company, USA) or paracore (Coltène/Whaledent, Switzerland) composite resins. The specimens were prepared with the aid of a special designed mold made of PTFE. The mold was composed of two halves which was convenient for taking out the specimens.

Briefly, as shown in Fig. 1a–d, the post-and-core structure was built through steps as: (a) injecting the core resin into the mold; (b) inserting the fiber post into the core resin; (c) being light-cured for 30 s (450 mW/cm^2 output; QHL75; Dentsply; USA); (d) finally splitting the mold, taking out the specimens and being light-cured for an additional 40 s to ensure polymerization.

With two kinds of fiber posts (modified and unmodified) and core resins (3M ESPE and paracore), a total of 48 specimens were divided into four groups of twelve each ($n = 12$). For each group, two specimens were randomly selected to SEM observation and the others were subjected the pull-out test. To observe the cross-section of the post-and-core structure, the specimens were transected from the middle of the resin core, and mechanically polished using wet silicon carbide paper in increasing grits of No. 600, 800 and 1000. The polished specimens were then ultrasonicated for 10 min in deionized water, and gently air-dried before SEM observation.

2.4.2. Pull-out test

The prepared post-and-core specimens were fixed to the jig as shown in Fig. 1. A universal testing machine (Model 1121, Instron Corp.) was used to apply a tensile force along the long axis of the post at the crosshead speed of 0.5 mm/min until failure. The maximum failure load of each specimen was recorded, at which point the post-and-core structure was damaged by post dislodgement or core breakage. After the pull-out testing, every fractured piece was examined by SEM to determine the failure mode.

2.5. Statistic analysis

The results of the pull-out test were represented as mean ± standard deviation for $n = 10$. Statistical analysis was made based on *t*-test and differences between groups were considered as significant for $p \leq 0.05$.