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## Research Paper

# Thermal cycling effects on adhesion of resin–bovine enamel junction among different composite resins

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## ABSTRACT

Thermal cycling is used to mimic the changes in oral cavity temperature experienced by composite resins when used clinically. The purpose of this study is to assess the thermal cycling effects of in-house produced composite resin on bonding strength. The dicalcium phosphate anhydrous filler surfaces are modified using nanocrystals and silanization (w/NP/Si). The resin is compared with commercially available composite resins Filtek Z250, Z350, and glass ionomer restorative material GIC Fuji-II LC (control). Different composite resins were filled into the dental enamel of bovine teeth. The bond force and resin–enamel junction graphical structures of the samples were determined after thermal cycling between 5 and 55 °C in deionized water for 600 cycles. After thermal cycling, the w/NP/Si 30 wt%, 50 wt% and Filtek Z250, Z350 groups showed higher shear forces than glass ionomer GIC, and w/NP/Si 50 wt% had the highest shear force. Through SEM observations, more of the fillings with w/NP/Si 30 wt% and w/NP/Si 50 wt% groups flowed into the enamel tubule, forming closed tubules with the composite resins. The push-out force is proportional to the resin flow depth and uniformity. The push-out tubule pore and resin shear pattern is the most uniform and consistent in the w/NP/Si 50 wt% group. Accordingly, this developed composite resin maintains great mechanical properties after thermal cycling. Thus, it has the potential to be used in a clinical setting when restoring non-carious cervical lesions.

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## 1. Introduction

Composite resins have replaced silver amalgams in being the dominant material used in dental fillings because it can be adjusted to fit natural teeth color (Chen and Wu, 2014). Dentists are also able to closely match the material according to the esthetics and functional needs of patients. Furthermore, composite resin has better coloring and gloss without releasing ions or heavy metals from alloys (Lavigne and Zhu, 2012). Contemporary dentists have many therapy treatment options for clinically defective restorations (Kuper et al., 2012). Among the different types of defects, clinicians must carefully evaluate enamel cavity preparation and material choice for difficult-to-treat cervical caries. Although composite resin also has better adhesion than alloys due to its flowable property, which allows it to penetrate into dentinal tubules, the repeated changes in temperature within the oral cavity can cause microleakages in restorative material margins and retention failure (Helvatjoglu-Antoniades et al., 2004; Sarrett, 2005; Lavigne and Zhu, 2012).

Much effort has been put into developing new composite resins by improving adhesion and restorative techniques (Bayne, 2005; Lavigne and Zhu, 2012). These challenges and the criteria used for evaluating restorative substances depend on many factors, including polymerization contraction, thermal changes, and occlusion forces that result in debonded stress at the marginal interfaces (Xu et al., 1999, 2002; Silva et al., 2013). Currently, commercially available composite resins suffer from microleakage because the hydrophilic monomers polymerization often used for composite resins are easily affected by the water content of the oral cavity in patients, causing crevices and leakages (Xu et al., 1999; Bayne, 2005). Furthermore, thermal cycling from changes in oral temperature provides conditions for degraded bond strength in an aqueous environment as well as microleakages (Xu et al., 2002). The formation of microleakages between the margin of restorative sites and teeth causes complications due to bacterial growth. Eventually, bacterial growth may acidify dentin and restorative substances, thereby causing the resin interface to dissolve. Secondary caries may result, and, if untreated, may cause restoration fracture (Sarrett, 2005; Miglani et al., 2010).

Thus, composite resin is continuously being researched and improved upon to meet different clinical needs and dental restoration purposes (Dickens-Venz et al., 1994; Skrtic et al., 1996; Dickens-Venz et al., 2003; Chen, 2010; Vouvoudi and Sideridou, 2012). For example, the basic reinforced fillers formula and types for coupling agents are being studied. Most especially, changing reinforced fillers have a significant effect on the properties of composite resin and is the commonly used strategy (Klapdohr and Moszner, 2005; Cramer et al., 2011). For example, when fillers are modified with whiskers capped on its surface, the adhesion between the composite resin matrix and the fillers increases significantly, overcoming the shrinkage caused by polymerization (Xu et al., 1999, 2002; Klapdohr and Moszner, 2005). Furthermore, the ions automatically released by calcium phosphate fillers may be beneficial for decalcified dentin from remineralization and controls microleakage from thermal cycling

effects (Skrtic et al., 1996; Dickens-Venz et al., 2003). Although the aforementioned calcium phosphate bioceramic has the ability to induce remineralization of dentin in vitro (Thomann et al., 1990; Chen et al., 2013a), relevant studies have not supported the expected results of restorative materials against thermal cycling.

The current research uses technology previously developed in-house by capping a layer of nanocrystals and salinization with resin matrix to form a compound of composite resin (Chen et al., 2013b; Chen and Wu, 2014). In vitro studies have shown that reinforced fillers with treated with nanocrystals increase the mechanical strength of composite resins. The hydroxyl group within the phosphate composite increases the chemical bond between the resin and dentin. Furthermore, the release of calcium and phosphate ions induces apparent remineralized precipitates on the sample surfaces in vitro (Klapdohr and Moszner, 2005; Chen, 2010; Cramer et al., 2011).

Although initial studies are promising, more research must be performed to study the clinical applications of composite resins and prove that such resins could be quantified for treating cervical carious lesions. To simulate fillings in the clinical setting, composite resins including Filtek Z250 composite resin (A3shade, 3 M/ESPE), Filtek Z350 flowable resin (A3 shade, 3 M/ESPE) as the comparison group, and a clinically-available glass ionomer Fuji-II filling (GIC) as the control group were filled into bovine teeth and put through thermal cycling. The mechanical properties and SEM observations were studied. This study hypothesizes that, after thermal cycling, composite resins using calcium phosphates as fillers have less debonding force decay than other fillers such as  $ZrO_2$  and glass ionomers. This study is based on the aforementioned premises that study how thermal cycling processes cause shear force decay and the bonding differences between the composite material and dentin to simulate composite resins used clinically.

## 2. Materials and methods

### 2.1. Preparation of composite resins

The DCPA filler ( $CaHPO_4$ , Alfa Aesar GmbH & Co. KG, Karlsruhe, Germany) powder ranged from  $1\ \mu m$  to  $3\ \mu m$  in particle distribution size, and powder with 98% was used. Nanocrystal formation behavior was determined during the monitored treatment according to previous studies (Chen et al., 2013b; Chen and Wu, 2014). Briefly, 5 g of DCPA powder was mixed in 40 mL solution with a constant calcium-to-phosphate atomic ratio of 2.0 and a pH of 5.0 for 20 min at room temperature. The particles were filtered, washed, and then dried. A 100 mL colloidal solution of cyclohexane solvent with 4% (v/v) (3-Mercaptopropyl) trimethoxysilane and 2% (v/v) n-propylamine from Alfa Aesar GmbH & Co., KG (Karlsruhe, Germany) was used to cap the particle surface. Up to 5 g per batch of DCPA powder with nanocrystal modification was added to the colloidal solution under rapid agitation for 30 min at room temperature and heated to  $60\ ^\circ C$ . Then, the solvent was removed by drying the samples in a vacuum for 2 h.

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