



## Effects of temperature change and beverage on mechanical and tribological properties of dental restorative composites



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### ABSTRACT

The aim of this study was to investigate the effects of temperature change and immersion in two common beverages on the mechanical and tribological properties for three different types of dental restorative materials. Thermocycling procedure was performed for simulating temperature changes in oral conditions. Black tea and soft drink were considered for beverages. Universal composite, universal nanohybrid composite and universal nanofilled composite, were used as dental materials. The nanoindentation and nanoscratch experiments were utilized to determine the elastic modulus, hardness, plasticity index and wear resistance of the test specimens. The results showed that thermocycling and immersion in each beverage had different effects on the tested dental materials. The mechanical and tribological properties of nanohybrid composite and nanocomposite were less sensitive to temperature change and to immersion in beverages in comparison with those of the conventional dental composite.

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### 1. Introduction

Dental composite materials have different applications in dentistry such as filling the tooth cavities, veneering to mask discoloration, and correcting contour and alignment deficiencies. These materials are also used for making dental implants and bonding orthodontic brackets [1,2]. It is often desirable to find new methods for improving the mechanical properties of dental restorative composites [3–5]. In the past few years, one of the most important advancements in dental materials is related to the application of nanotechnology to dental restorative composites. For example, nano-fillers with dimensions of 5 to 100 nm have been added to the composite resins for producing dental nanocomposite. Nano-hybrid composites are also a category of dental restorative materials where in addition to nanometer particles, particles of 0.2 to 1  $\mu\text{m}$  in size are added to the composite resins. It has been shown in the past that dental nanocomposites and nanohybrid composites can be used as dental restorative materials, instead of the conventional dental composites [6–10]. The general aims for producing these new dental materials were to achieve better tensile and compressive strengths, improved fracture toughness and wear resistance, firm bonding with dental enamel surface and desired aesthetic performance [7,11–13]. A good knowledge of the mechanical and tribological properties of new dental materials in various oral conditions would help clinicians in comparing

the behavior of different dental materials and selecting the appropriate one. It could also be useful for manufacturers of dental materials for improving their products.

The variation of temperature in oral environment can affect the mechanical properties of dental restorative materials. Eating hot or cold foods and drinking beverages are the causes of most extreme temperature variation in the oral cavity. Typical minimum and maximum temperatures of tooth surface during the consumption of food stuffs are 1 °C and 50 °C [14–16]. Although the mechanical and tribological properties of commercial dental restorative materials are often measured at room or body temperature [12,14,15,17,18], the mechanical properties of polymeric materials like dental composites are often sensitive to temperature variations. Therefore, it is important to evaluate the effects of oral conditions on the mechanical and tribological properties of dental restorative composites. Thermocycling is an in-vitro process which simulates temperature variations of the oral cavity that occur through eating, drinking, and breathing [19–22]. In addition to the temperature variations in oral conditions, water or other liquids such as saliva, beverages or food components may influence their physical and mechanical properties. Global statistics show that the consumption of carbonated soft drinks in the past 50 years has increased dramatically. Consumption of acidic drinks can degrade both the teeth and the restorative materials. For example, Lussi and Hellwig [23] reported a reduction in enamel hardness after 10 and 20 min of immersion in a commercial orange juice. Aliping-McKenzie et al. [24] immersed dental restorative materials in Coca-Cola and fruit juices and found significant reduction in their surface hardness.

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In this paper, the effects of thermocycling and immersion in two types of beverages on the mechanical and tribological properties of several dental restorative composites are studied. The null hypothesis was that new dental restorative materials preserve their mechanical and tribological properties in different oral conditions compared with the conventional dental composites.

Nanoindentation and nanoscratch tests have been found to be powerful methods for measuring the mechanical properties such as modulus of elasticity, hardness and wear resistance of various materials in very small sample sizes such as polyamide-12/layered silicate nanocomposites, clay nanocomposites, bone cement and tooth enamel [25–31]. Therefore, in the current study the nanoindentation and nanoscratch experiments are employed to determine the mechanical and tribological properties of dental restorative composites.

## 2. Materials and methods

### 2.1. Sample preparation

Three types of dental restorative materials, i.e. composite, nanohybrid composite and nanocomposite were used to prepare the samples. The characteristics of these materials are presented in Table 1 according to the manufacturer's information.

Five specimens from each material were prepared in a disc-shaped mold with a diameter of 10 mm and a thickness of 4 mm. Due to the thickness of specimens, each consists of two light cure increment. According to the manufacturer's instructions of the restorative materials, each increment was light cured for 20 s using a LED light with intensity of 400 mW/cm<sup>2</sup>. During the light exposure, the light guide tip was held as close to the specimen surface as possible. Since nanoindentation and nanoscratch tests need a very smooth surface, all the specimens were smoothed by sandpapers with 400 to 2500 grits and then polished by diamond pastes with mesh sizes of 1, 0.5 and 0.25  $\mu$ m.

### 2.2. Thermocycling procedure

In order to study the effect of cyclic thermal stresses that occur in the mouth during the service life of the dental restorative materials, two samples for each type of material were thermocycled. The thermocycling apparatus consists of two stainless steel water baths filled with distilled water and a mechanical arm that transports the specimens from one bath to another. The ISO TR 11450 standard indicates that a thermocycling procedure of 500 cycles in water between +5 °C and +55 °C is an appropriate artificial aging test [32]. However, long-term aging process could be selected for long-lasting restorative materials such as materials used in this study [33–37]. Therefore, two samples from each material were subjected to 2000 cycles between the temperatures of +5 and +55 °C, with a dwell time of 30 s in each bath per cycle and a transport over time of 15 s. A total number of 2000 cycles of thermocycling in this study can be considered to be equal to maintaining the dental materials for 200 days in the mouth conditions.

### 2.3. Immersion in beverages

For investigating the effect of beverage alone and also simultaneous effect of thermocycling and beverage on the dental restorative materials, a non-thermocycled sample and a thermocycled sample for each type of material were immersed in carbonated soft drink (Coca-Cola) and another pair was immersed in black tea for 48 h. Each specimen was placed in a separate container. The temperature was kept at 55  $\pm$  5 °C for tea and at 5  $\pm$  2 °C for soft drink to reach more realistic evaluation of beverage effects.

All in all, five samples were prepared for each type of the three investigated dental materials: (1) control or intact sample (non-thermocycled and non-immersed in any beverage), (2) non-thermocycled and immersed in tea, (3) non-thermocycled and immersed in soft drink, (4) thermocycled and immersed in tea, and (5) thermocycled and immersed in soft drink.

### 2.4. Nanoindentation experiment

The basic mechanical properties of the test specimens, i.e. hardness, elastic modulus and plasticity index were determined by a Triboscope nanoindentation test system (Hysitron Inc., USA) based on ISO 14577 [38]. A Berkovich indenter was used in all experiments. This type of indenter is usually used for bulk samples and for thin films thicker than 100 nm. The test setup and the Berkovich indenter tip are shown in Fig. 1. Oliver and Pharr method [39] was used to calibrate the device and also to analyze the test results.

An indentation load of 1950  $\mu$ N was applied to the surface of each sample in 30 s with a constant loading rate. To take account of likely creep effects, the indenter was kept on the sample at the maximum load for 10 s [40]. Finally, the unloading stage of the test was performed by removing the tip from the sample at the same rate.

For each sample, the nano-indentation tests were repeated at least 5 times in different randomly selected sites, and the curves of force versus indenter displacement were recorded through the test instruments. According to the simple power and sample analysis, when the experiment repeats 5 times in each of the 5 groups, the 0.05 level of difference in means will be detected by 90% power, assuming that the common standard deviation is 0.500 [41].

Before and after the experiments, atomic force microscopy (AFM) images were taken from the sample surfaces to analyze the indentation hole. In the AFM process, the same indenter tip as the nano-indentation test, scans the surface of specimen without applying any load.

### 2.5. Nanoscratch experiment

Each nano-scratch test was performed in three main stages. First, a pre-scan of each sample was carried out and AFM images were taken to investigate the roughness and tilt angle of the sample surface. Then, the nano-scratch test was performed on the sample by using the same indenter as that of the nano-indentation test. The normal load was 1950  $\mu$ N and the scratch length was 4  $\mu$ m. The indenter penetrated the

**Table 1**  
Dental restorative materials used for experiments.

Type	Material	Type of resins	Filler particles	Filler content	Shade	Manufacturer
Composite	Filtek Z250	Bis-GMA <sup>1</sup> , UDMA <sup>2</sup> , Bis-EMA <sup>3</sup>	Zirconia/silica	60%	B1	3M ESPE, USA
Nanohybrid composite	Filtek Z250 XT	Bis-GMA, UDMA, PEGDMA <sup>4</sup> , TEGDMA <sup>5</sup>	Silica, Zirconia/silica	67.8%	B1	3M ESPE, USA
Nanocomposite	Filtek Z350 XT	Bis-GMA, UDMA, TEGDMA, PEGDMA, Bis-EMA	Zirconia, silica	63.3%	WE	3M ESPE, USA

<sup>1</sup> Bis-GMA: bisphenol A glycidyl methacrylate.

<sup>2</sup> UDMA: diurethane dimethacrylate.

<sup>3</sup> Bis-EMA: ethoxylatedbisphenol A dimethacrylate.

<sup>4</sup> PEGDMA: polyethylene glycol dimethacrylate.

<sup>5</sup> TEGDMA: triethylene glycol dimethacrylate.

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