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Effects of temperature on mechanical and tribological properties of dental restorative composite materials

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ABSTRACT

The purpose of this study is to evaluate the effects of temperature on the mechanical and tribological properties of three commercial posterior direct filling resin based restorative composite materials, Synergy (Coltene/Whaledent, Altstätten, Switzerland), Surefil (Dentsply/Caulk, New York, USA) and Alert (Pentron Clinical, Wallingford, USA). The aim of this paper was to evaluate how temperature influences; the hardness, flexural modulus and strength, elastic modulus, and wear of the listed commercial restorative materials.

Three commercial resin composites were tested at several temperatures. A closed control system was used to ensure constant test conditions that simulated the temperatures found in an oral environment. For each of the tests, the following mechanical and tribological properties were determined, microhardness (Vickers micro-indentation), elastic modulus (determined both by dynamical methods and by bending tests), flexural resistance and work of fracture parameter (bending tests), and wear (reciprocating tests).

Following the tests, the evaluation of the mechanical and tribological properties of the resins suggests that their performance in an oral environment can be significantly affected by temperature, especially the wear resistance. Surefil is the resin least sensitive to temperature variation, while Synergy exhibited the best resistance to wear with respect to temperature variation.

1. Introduction

Direct filling dental resin composites, reinforced with inorganic particles, are a category of dental restorative materials specifically developed as an alternative to amalgam, with a greater aesthetic benefit.

The properties of a restorative composite material are dependent on externally imposed variables, namely; temperature, the oral medium, and mechanical loads. It would seem reasonable to assume that ingesting hot and cold substances is the cause of the most extreme temperature variation in the oral cavity. The typical mean maximum and minimum tooth surface temperature during the consumption of hot foodstuffs varies roughly from 1 to 50 °C [1,2]. The maximum temperature measurement *in vivo* during the consumption of a hot beverage, measured between the lower incisors, reached a maximum value of 76.3 °C (mean maximum temperature of 46.4 ± 7.51 °C) and for hot food, the highest temperature was measured on the upper incisor, reaching a value of 53.61 °C (mean maximum temperature was 41.6 ± 4.31 °C [3]. It is important that the temperature range used for testing dental materials be appropriate and reflects the *in vivo* intraoral temperature conditions [2]. As the properties of many polymeric materials are sensitive to temperature of this magnitude, it is important to assess the effects of the operating temperature on the mechanical behaviour of these materials. However, mechanical and tribological testing of commercial dental restorative material is commonly done at room temperature (21–25 °C), or more rarely, at nominal body temperature (37 °C) [1,4].

Temperature sensitivity in the dental resin composites is a subject that lacks evaluation regarding the mechanical and tribological characterisation, in order to apply this knowledge into products for clinical service. There are few studies regarding the effect of temperature on mechanical properties [5]. Flexural tests were performed with water immersion temperatures ranging from 5 to 55 °C. Musanje and Darvell [1] studied the effects of strain rate and temperature (12, 24 and 37.8 °C) on some of the mechanical properties of resin restorative composite materials with three-point bend tests, testing flexural strength, elastic modulus and total energy to failure. They also investigated the construction of temperature-strain rate equivalence 'master curves' to determine property values under conditions other than those tested. Both the studies concluded that resin composites are sensitive to temperature and the importance of temperature effect on the general behaviour of the composites, and to the

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damage accumulation on the surface, or subsurface microstructure due to transient thermo-mechanical loads, need to be taken into account in the correlation of *in vitro* and *in vivo* behaviour [5]. Xu et al. [6] investigated the effects of thermal cycling on whisker-reinforced composites, through the measurement of the mechanical properties, or the breakdown of the whisker–resin interface after thermal cycling. The filler mass fraction ranged from 0% to 70%, and the specimens were thermal cycled from 5 to 60 °C in water baths, and then fractured in three-point bending tests to measure strength; a nano-indentation was used to measure modulus and hardness.

The effect of temperature on wear is a poorly studied subject. A great majority of studies have focused mainly on the storage or degree of conversion of resin composites.

A wider clinical acceptance of polymer composite materials is still limited by unanswered questions related to their behaviour during long term exposure in the oral environment; specifically, in terms of their competence of mechanical properties like wear resistance, hardness, flexural modulus, flexural strength, and toughness [7]. Composite restorative materials, when placed in the oral environmental, are constantly subjected to challenges of chemical [8] and thermal nature [2,9], due to the intake of food and fluids at different temperatures. Such thermal variations, if significant, would affect the mechanical and tribological properties of the restorative materials. Dental materials are prone to fracture and abrasion during normal masticatory function; thus, fracture and wear are the two main failure modes which occur in posterior restorations [10]. Therefore, mechanical characterisation is a key element in understanding the nature of the dependency between temperature and the expected performance in an intraoral environment.

Therefore, given the expectation of temperature sensitivity of dental resin composites and the lack of published information on this matter, as well as the need to characterise products for clinical service, this study aims to investigate the general mechanical and tribological behaviour of dental composite materials, in order to understand and predict their performance in the mouth. This research investigates the mechanical and tribological behaviour of three commercially available dental restorative materials; Synergy (Coltene/Whaledent, Altstätten, Switzerland), Surefil (Dentsply/Caulk, New York, USA), and Alert (Pentron Clinical, Wallingford, USA), with respect to temperature variation.

2. Experimental procedures

The experimental procedures of this study are divided into mechanical and tribological tests. Tests undertaken for mechanical evaluation include the following: Vickers hardness, four-point bending, and impulse excitation. These tests facilitate the determination of several material properties, namely: hardness, flexural resistance, static elastic modulus, work of fracture, and dynamic elastic modulus. Tribological tests are used to determine the wear resistance of the composite material and also the wear produced in the oral environment. Every test, mechanical and tribological, was executed at temperatures of: 5, 10, 20, 30, 40 and 50 °C for each of the three specimens of the studied materials. Thus, each property value presented represents the average value of the three test specimens.

2.1. Materials and specimens

These restorative direct posterior composites selected for test, are the most suitable for posterior restorations and are in commercial use. Table 1 summarises the information regarding matrix composition and reinforcement filler, the latter being subdivided into type, average particle dimension and percentage of fraction, in volume and/or weight (information provided by material manufacturers).

Although these composites are the so-called packable or condensable composites, they form a special category of hybrid composites used for posterior restoration, with very different types of reinforcement, regarding filler type (particles and/or fibres) and size distribution of the reinforcement particles. Surefil has an average particle size of 0.8 μm, but the scatter is significant and some particles measure up to 20 μm. In fact, the criterion for the selection of these materials was to examine a wide range of filler parameters, types of filler, and average particle dimension. Synergy represents a nanofilled composite, while Surefil is microfilled, and Alert is a hybrid reinforced composite. Fig. 1 shows SEM images of the microstructures of the three tested composites.

An aluminium mould was used to produce parallelepipedic specimens of composite, 60 mm length, 6 mm width and 2 mm thickness. The mould was placed on a transparency film, supported on a glass, filled manually with a slight excess of resin composite and covered in the same manner as the underneath. Before curing, the composite samples were compacted manually by applying light finger pressure on the upper glass. A Kerr polymerisation unit, the Optilux 501, was used to perform this task in accordance with the manufacturer's specifications and appropriate curing mode. The output wavelength range of the curing light varied from 400 to 510 nm, with a minimum light intensity of 850 mW/cm^2. The curing time for all specimens was 40 s, which is compatible with the curing time recommended by the composite manufacturers. The tip of the light guide (11 mm in diameter) was placed in contact with the upper glass and the specimens irradiated along its length in order to ensure effective curing of the surface. All specimens were cured at 6 locations spaced 10 mm apart, for a duration of 40 s per location, thus ensuring the complete cure of the entire surface. Hardness tests were made along the specimen's length to ensure similar characteristics among specimens of the same material. Specimens with mean values of hardness greater than 10% of the average of the material (studied in detail elsewhere [11]) were rejected. The manufacturer of the polymerisation unit ensured a curing depth greater than the thickness of the specimens, and the composite manufacturers only recommended a 20 s curing time for the actual thickness of the specimens.

2.2. Equipment description and test conditions

2.2.1. Hardness

A Vickers micro-indentation test was performed using a Struers-Duramin equipment, applying a load of 1.962 N for a period of 40 s. Ten indentations on the surface directly exposed to the curing light were made on each specimen and for each temperature. To control the test temperature, a circuit with an immersed pump and a thermostatic controller was used in order to ensure a constant temperature (Fig. 2). The water circuit is comprised of two vessels: a larger one to ensure the thermal inertia, and a smaller one in which the specimen was placed to measure the hardness. The thermocouple was placed in the upper surface of the specimens and was connected to the thermostatic controller to verify the temperature at which the composite was under test, and to control the circuit pump, which guaranteed an operational temperature increment ± 1 °C. For tests with temperatures higher than 20 °C, warm water was used as a thermo fluid; whilst for test temperatures below 20 °C, a mixture of ethanol and liquid nitrogen were used.