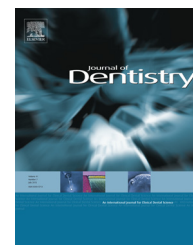


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Effect of temperature on post-cure polymerization of bulk-fill composites

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ABSTRACT

Objectives: To investigate the 24 h post-cure polymerization and the effect of temperature on the post-cure polymerization of one conventional and three bulk-fill composite materials.

Methods: A conventional composite GrandioSO (GR) and three bulk-fill composites: Tetric EvoCeram Bulk Fill (TECBF), Quixfil (QF) and X-tra fil (XF) were investigated. The samples were cured for 20 s with irradiance of 1090 mW/cm². Composite samples were divided into two groups: the “room-temperature” group (RT, n = 5) and the “body-temperature” group (BT, n = 5) and they were stored in dark at 20 °C and 37 °C, respectively. Measurements of degree of conversion (DC) were made immediately after curing (0 h) and 24 h post-cure (24 h). To analyse the extent of post-cure DC increase, the DC values of 0 h-RT/24 h-RT and 0 h-BT/24 h-BT were compared. To analyse the difference in DC between RT and BT, the DC values of 0 h-RT/0 h-BT and 24 h-RT/24 h-BT were compared.

Results: DC increase 24 h post-cure was significant for all composites and ranged between 6.3% and 8.2% in RT and between 12.5% and 15.7% in BT. All composites demonstrated a higher DC in 24 h-BT compared to 24 h-RT. The difference was statistically significant for GR, TECBF and QF.

Conclusions: All composites demonstrated a significant post-cure effect after 24 h. Post-cure temperature increase from 20 °C to 37 °C yielded a higher post-cure DC increase.

Clinical significance: Due to the temperature effect on the final DC, studies performed at composite samples at room temperature may record more inferior properties than these attained in the oral cavity.

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1. Introduction

Contemporary composite materials are typically based on dimethacrylate resins which harden via free-radical polymerization. Activation by visible light is the primary mode of

triggering the polymerization.^{1,2} Polymerization occurs by subsequent addition of dimethacrylate monomer molecules, resulting in rapid growth and crosslinking of polymeric chains. During polymerization, free radicals are located on growing polymeric chains and represent reactive sites that must be reached by monomeric molecules for addition to take

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place.³ Since polymeric chains are relatively immobile due to high molecular mass, the reaction is controlled by the mobility of monomer molecules. In the initial stages of polymerization, monomer is free to flow to reactive sites and addition proceeds rapidly. However, as degree of conversion (DC) increases, it becomes increasingly difficult for monomer to reach reactive sites and therefore the rate of reaction decreases.⁴

Free radical polymerization features complex kinetics, with phenomena known as autoacceleration and autodeceleration.³ Autoacceleration occurs in early stages of polymerization because limited mobility of free radicals located on growing polymeric chains inhibits termination. This results in the increase of reaction rate, despite the fact that the amount of reactants is decreasing. Autodeceleration occurs in later stages of polymerization when increasing viscosity limits the mobility of monomer molecules, thus inhibiting propagation. In other words, kinetics of polymerization reaction is largely affected by mobility of reactants during both early and late stages of reaction.⁵ Due to the increasing diffusivity limitations, the polymerization rate slows down significantly and reaction extends for prolonged periods of time.

Most of the polymerization reaction occurs during the first minutes after irradiation; however a significant portion of reaction may take place after curing.⁶ The extent of post-cure reaction depends on initial DC,⁷ resin composition,⁸ availability of free radicals⁹ and the temperature of the sample.¹⁰

Bulk-fill composites are designed for placement in layers up to 4 mm. In order to achieve properties that enable such placement, manufacturers have addressed issues of shrinkage stress and depth of cure by various composition modifications.¹¹ These modifications may alter polymerization kinetics, especially because slower polymerization is sometimes used as a strategy to reduce shrinkage stress.^{12,13}

The aims of this study were: (I) to assess the post-cure DC increase for one conventional and three bulk-fill composites,

and (II) to examine the influence of temperature on the post-cure DC increase. The null-hypotheses were: (I) there is no difference between the DC values obtained immediately after curing and 24 h post-cure, and (II) there is no difference in the post-cure DC increase between the samples stored at 20 °C and 37 °C.

2. Materials and methods

Composite materials are listed in Table 1. Three bulk-fill composites were investigated and a conventional composite GrandioSO (Voco, Cuxhaven, Germany) was used as a control.

Cylindrical samples with diameter of 3 mm and height of 6 mm were prepared using a solid stainless steel mould with polished and thus reflective surface. The outside shape of the mould was cuboid, with dimensions of 20 × 6 × 8 mm. Uncured composite material was applied in bulk into the mould, both mould apertures were covered with a polyethylene terephthalate (PET) film and curing was performed with a LED curing unit Bluephase G2 (Ivoclar-Vivadent, Schaan, Liechtenstein) for 20 s in the wavelength range of 380–515 nm. The irradiance of Bluephase G2 in “high” mode, as stated by the manufacturer, is 1200 mW/cm² while the value of 1090 mW/cm² was measured using a single CureRite radiometer (Caulk Dentsply, Konstanz, Germany, model no. 644726). Taking into account the variability of the irradiance measurements obtained by some hand-held radiometers,^{14,15} in the present study the radiometer was used for monitoring the constancy of the curing unit irradiance before the polymerization of each composite material. The variations were under 10%, and value of 1090 mW/cm² represents the arithmetic mean of ten measurements. The curing time of 20 s corresponded to or exceeded manufacturer’s recommendations for all tested materials. The light probe of the curing unit

Table 1 – Manufacturers’ information about the composite materials used.

Type	Composite material/ code (Manufacturer)	Shade/LOT (EXP)	Composition	Filler amount wt%/vol%
Conventional	GrandioSO/GR (Voco, Cuxhaven, Germany)	A2/1222126 (2014/11)	Inorganic fillers in a methacrylate matrix (Bis-GMA, TEGDMA)	89/73
Bulk-fill	Tetric EvoCeram Bulk Fill/TECBF (Ivoclar Vivadent, Schaan, Liechtenstein)	IVA/P82299 (2015/12)	Dimethacrylates: Bis-GMA, Bis-EMA, UDMA, barium glass, ytterbium trifluoride, mixed oxide and prepolymer; additives, catalysts, stabilizers, pigments	81/61
	Quixfil/QF (Dentsply, York, Pennsylvania, USA)	Universal/121200233 (2014/05)	UDMA, TEGDMA, di- and tri-methacrylate resins, carboxylic acid modified dimethacrylate resin, butylated hydroxy toluene (BHT), UV stabilizer, camphorquinone, ethyl-4-dimethylamino-benzoate, silanated strontium aluminium sodium fluoride phosphate silicate glass	86/66
	X-tra fil/XF (Voco, Cuxhaven, Germany)	U/1311472 (2015/03)	Inorganic filler in a methacrylate matrix (Bis-GMA, UDMA, TEGDMA)	86/70

Bis-GMA: bisphenol-A-glycidylmethacrylate; TEGDMA: triethylene glycol dimethacrylate; Bis-EMA: ethoxylated bisphenol-A-dimethacrylate; UDMA: urethane dimethacrylate.

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