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Short communication

Effect of resin chemistry on depth of cure and cytotoxicity of dental resin composites

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ABSTRACT

New dental composite restorative materials are being introduced aiming to overcome the disadvantages of contemporary materials. Hence there is a need to analyze the critical properties of these composites to aid in clinical application. This study aims to comparatively analyze the degree of conversion (DC), residual reactivity (DBC/reactivity) and cytotoxicity of 2 composites based on different resin chemistry. Ceram X and Filtek P90 were used in the study to prepare disc shaped samples of 2 mm thickness and 4 mm diameter. The samples for cytotoxicity were cured for 40 s and those of Fourier Transform Infra-red Spectroscopy (FTIR) (DBC/reactivity and DC) for 5 s, 10 s, 20 s and 40 s, at an average intensity of 800 mW/cm^2 with Quartz–Tungsten–Halogen (QTH) light. DC was calculated in 60–100 μm thick and 6 mm diameter samples. Double bonds concentration/reactivity was measured in approximately 80 μm thick sections prepared from the 2 mm thick discs using a hard tissue microtome. The cell viability was scored by Trypan blue exclusion staining technique at 24 h and 48 h. Both composites showed a progressive increase in double bonds/reactivity as the distance from curing probe increased which was inversely proportional to the curing time. The DC of Filtek P90 was 20% and 96% and that of Ceram X 33% and 50% at 5 s and 40 s, respectively. Ceram X showed statistically significantly higher cell viability score at both 24 h and 48 h than Filtek P90. The results were statistically analyzed using non-parametric Kruskal–Wallis, Mann–Whitney *U* and Wilcoxon Signed Ranks tests. Though DC plays an important role in biocompatibility of dental composites, other factors like elution may play a significant role and hence need further evaluation.

1. Introduction

Dental resin composites were developed as an aesthetic and safer alternative to amalgam restorations [1–4]. Besides their advantages, clinical studies show that they indeed have much lower longevity compared to amalgam [5]. The factors contributing to their poor long term performance are polymerization shrinkage, marginal gap formation, secondary caries, low fracture toughness and adverse effects on pulpal health [5–8]. Some of these factors stem from their resin chemistry [9]. Incomplete polymerization of dental resin composites and resin-based bonding agents under clinical conditions result in unreacted resin monomers that may be released from the resin matrix into the aqueous environment of oral cavity [10]. All monomers exhibited a dose dependent cytotoxic effect, and the ranking of the cytotoxicity based on TC50 was GMA > TEGDMA > HEMA. The authors also confirmed a dose-dependent genotoxicity of the resin monomers [10]. Cases of genotoxicity without cytotoxicity can be found at various concentrations of all resin monomers. Thus it is likely that resin monomers can cause genotoxicity at the concentrations relatively lower than those for apoptotic effects [11]. Hence newer resin chemistries that overcome these potential problems are being explored [12–18].

A review of literature reveals that these newer resins do not fulfil all the essential requisites for a significantly better resin than conventional resin matrix [8,13–24]. A dendrimer–methacrylate copolymer was found to have increased degree of conversion, but poor mechanical properties [13]. Methacrylated beta Cyclo Dextrin-based composite formulations containing tri ethylene glycol dimethacrylate, 1,10-decamethylenediol dimethacrylate, or benzyl methacrylate yielded flexural strength and volumetric shrinkage values comparable to those of the Bis-GMA/tri ethylene glycol dimethacrylate formulation [14]. Appropriate ratio of Polyhedral Oligomeric Sil Sequioxane-Methacrylate, Bis-GMA and TEGDMA was found to result in improved mechanical properties [15]. Thiol-ene systems were found to have reduced shrinkage stress, increased polymerization rate, increased functional group conversion, and decreased leachable species while retaining the

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Table 1
Composition of the dental resin composites used.

Group I	Group II
Ceram X (Dentsply DeTrey, Germany) Milford, DE 19963, USA	GmbHFiltek P90 (3M, St. Paul, MN, USA)
Methacrylate modified polysiloxane	Silorane
Dimethacrylateresin	
Fluorescent pigment	
Stabilizer Camphoroquinone	
Camphoroquinone	Phenyl iodoniumhexafluoroantimonate
Ethyl 4 dimethylaminobenzoate	Ethyl 4dimethyl aminobenzoate
Barium aluminium borosilicate glass	Quartz
Silicon dioxide nanofiller	Yttrium fluoride
Iron oxide	
Titanium oxide	
Aluminiumsulfosilicate	

mechanical properties of comparable monomer in oligomeric form as well [16,17]. Novel dimethacrylate monomers with reduced reactive group densities were found to decrease the polymerization shrinkage, improve the double bond conversion and help maintain the mechanical properties of the resulting polymer, in addition to producing more homogeneous copolymer networks [18].

Of the new resin chemistries being studied, noteworthy are the ones like ormocers and silorane [8,19–24]. Ormocer is an organically modified ceramic. It has functionalized fillers and a polysiloxane matrix. The polymerization of the matrix is through the common free-radical induced addition polymerization reaction. Silorane is a silicone based resin with an oxirane coupling. The polymerization of the matrix is through the cationic ring opening addition polymerization reaction. These are claimed to have low volumetric shrinkage, greater stability and less elution of questionable moieties. A volumetric shrinkage ranging from 0.12% to 4.2% was reported for 8 different oxirane based composites [12]. Though these materials are commercially available, it is worthwhile studying their critical properties to have an insight into their clinical applicability. Extensive review of literature [8,13–24] indicates that resin chemistry, concentration of unreacted double bonds/reactivity, degree of conversion, polymerization shrinkage and biocompatibility are the key parameters in deciding the long-term survival and success of resin based dental restorative materials.

The aim of this study is to comparatively analyze the remaining double bonds concentration (DBC for Ceram X) or the residual reactivity of the material (for Filtek P90), degree of conversion (DC) and cytotoxicity of 2 composites based on different resin chemistry, namely, ormocer (Ceram X) and silorane (Filtek P90).

The null hypothesis is (i) that there will not be any significant difference between the materials in the DBC/reactivity under identical polymerization protocol, (ii) that there will not be any significant difference between the materials in the DC under identical polymerization protocol, and (iii) that there will not be any significant difference between the materials in the cytotoxicity under identical polymerization protocol.

2. Materials and methods

Dental composites Ceram X based on ormocer chemistry (Group I) and Filtek P90 based on silorane chemistry (Group II) were used for the present study. The composition of the materials is provided in Table 1. Fourier Transform Infrared Spectroscopy (FTIR) was done for measuring the degree of conversion and double bonds concentration or reactivity from the top surface to the bottom surface of the specimens (surface closest vs. surface farthest from the light source) as a function of curing time. Cytotoxicity was measured by direct contact test on human cervical cancer cell-line He La using Trypan blue exclusion dye staining technique. The choice of the cell line was based on the permanent (immortal) nature of the cells, ability to correlate apoptosis and robustness.

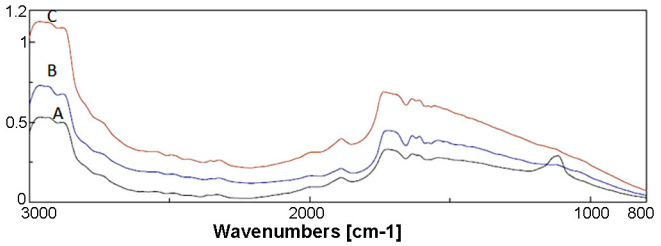

Fig. 1. Absorption Mode FTIR of Group I (Ceram X): A – non-polymerized sample; B – sample polymerized for 5 s; C – sample polymerized for 40 s.

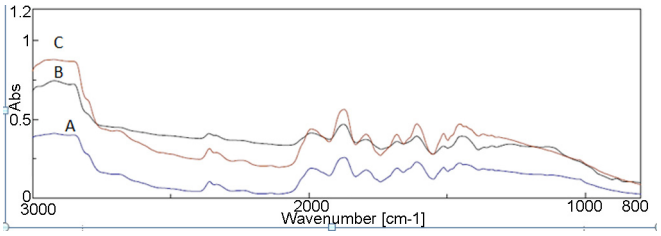

Fig. 2. Absorption Mode FTIR of Group II (Filtek P90): A – non-polymerized sample; B – sample polymerized for 40 s; C – sample polymerized for 5 s.

2.1. FTIR

Custom made polyethylene moulds were used to prepare 16 samples of 2 mm thickness and 4 mm diameter for each material. The number of specimens was arbitrarily chosen, as advanced analytical techniques require only one-four samples to accurately assess their repeatability and reproducibility. The specimens were light cured for 5 s, 10 s, 20 s and 40 s and thus sub-grouped accordingly, using QTH (QHL 75 Dentsply) at an average intensity of 800 mW/cm^2. The specimens were cut (cross section) to yield on an average 7 sections from each of approximately 80 μm thick sections using hard tissue microtome. Two microscope slides were used to prepare 20 samples of approximately 60 μm thickness and 6 mm diameter for each material. The specimens were light cured for 5 s and 40 s and thus sub-grouped accordingly, using the same QTH as above with the same energy parameters. FTIR was done in absorbance mode in the 400–4000 cm^{-1} range at a resolution of 4 cm^{-1} (Jasco FTIR 4100) for the seven sections from each disc sample and the 60 μm film sample for both the materials. Apart from this, unpolymerized sample of each material was also subjected to FTIR. Sodium chloride crystals were used to hold the specimens during IR and 32 s scan was obtained in duplicate for each specimen and the peaks of various functional groups were analyzed.

Area under the 1638, 1608, 1722 peaks were measured and compared for DC and double bond concentration in Ceram X (Fig. 1). Areas under 880 and 1254 peaks were measured and compared for DC and reactivity in Filtek P90 (Fig. 2). The beginning and end of the peaks were considered as the baseline for the area calculation. The DC was calculated using the following formulae (24, 26–28, 30–32):

The DC was calculated using the following formulae:

For Group I:

$$DC\% = 1 - \left\{ \frac{\frac{C{=}C\ \text{aliphatic}(1638)}{C{=}C\ \text{aromatic}(1608)}\ \text{in polymerized resin composite}}{\frac{C{=}C\ \text{aliphatic}(1638)}{C{=}C\ \text{aromatic}(1608)}\ \text{in unpolymerized composite}} \right\} \times 100$$