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A resin composite material containing an eugenol derivative for intracanal post cementation and core build-up restoration

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ABSTRACT

Objectives. To formulate and evaluate new dual cured resin composite based on the inclusion of eugenyl methacrylate monomer (EgMA) with Bis-GMA/TEGDMA resin systems for intracanal post cementation and core build-up restoration of endodontically treated teeth.

Methods. EgMA was synthesized and incorporated at 5% (BTEg5) or 10% (BTEg10) into dual-cure formulations. Curing properties, viscosity, T_g , radiopacity, static and dynamic mechanical properties of the composites were determined and compared with Clearfil™DC Core-Plus, a commercial dual-cure, two-component composite. Statistical analysis of the data was performed with ANOVA and the Tukey's post-hoc test.

Results. The experimental composites were successfully prepared, which exhibited excellent curing depths of 4.9, 4.7 and 4.2 mm for BTEg0, BTEg5 and BTEg10 respectively, which were significantly higher than Clearfil™DC. However, the inclusion of EgMA initially led to a lower degree of cure, which increased when measured at 24 h with values comparable to formulations without EgMA, indicating post-curing. The inclusion of EgMA also lowered the polymerization exotherm thereby reducing the potential of thermal damage to host tissue. Both thermal and viscoelastic analyses confirmed the ability of the monomer to reduce the stiffness of the composites by forming a branched network. The compressive strength of BTEg5 was significantly higher than the control whilst flexural strength increased significantly from 95.9 to 114.8 MPa (BTEg5) and 121.9 MPa (BTEg10). Radiopacity of the composites was equivalent to ~3 mm Al allowing efficient diagnosis.

Significance. The incorporation of EgMA within polymerizable formulations provides a novel approach to prepare reinforced resin composite material for intracanal post cementation and core build-up and the potential to impart antibacterial properties of eugenol to endodontic restorations.

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1. Introduction

The restoration of endodontically treated teeth (ETT) remains a challenge in clinical practice, especially under conditions of extensive root canal flaring [1,2]. Factors such as caries, trauma to immature permanent teeth, anomalies, internal resorption, and over preparation may result in flared root canals with thin dentinal walls and open apices which make root canal debridement difficult and complicate the endodontic and restorative procedures [3,4]. In such cases, prefabricated fiber posts are often used to provide retention for the final coronal restoration. For luting procedures, the use of resin composite core materials with high modulus of elasticity is highly recommended because it can increase the fracture resistance of these weakened teeth and is an alternative to resin cements for one-stage post placement and core build-up restoration [5,6]. The modulus of elasticity of current luting cements are far lower than that of posts and dentine, which may create a zone of high stresses especially when a thick layer of cement is present in a wide or flared canal, leading to inefficient bonding [5,7].

More recently, dual cured resin composite materials with different viscosities have been used in combination with fiber posts to restore structurally compromised ETT [8]. Most of these materials are methacrylate resin based with high filler content and superior mechanical properties than those of resin cements. Previous studies have shown that incorporation of high amounts of filler improve the rigidity of the luting agent but increase stress development during polymerization, which in turn affects the integrity of adhesive interface, reducing bond strength and increasing microleakage [9,10]. The higher viscosity that is associated with higher filler load [11] also impedes the injection of the material into the root canal producing gaps and voids that may provide a site for recurrent caries to develop. The composition of the matrix [12] also has an effect on both viscoelastic and rheological properties, which influence the contraction stress and microleakage of the direct restoration [13,14]. Consequently, the incorporation of low molecular weight monomers within methacrylate resin composite materials can enhance the flexural properties and lower viscosity [15].

On the other hand, numerous efforts have been made recently on the development of new monomers to be added into the formulation of dental resin composites with the aim of improving their functionality, quality and durability. Several low viscosity ionic mono and dimethacrylate monomers containing quaternary ammoniums groups such as 1,2-methacryloyloxydodecylpyridinium bromide (MDPB) and bis(2-methacryloyloxyethyl) dimethylammonium bromide (IDMA) imparting antimicrobial properties in conjunction with existing dental dimethacrylate-based monomers have been reported [16,17]. However, adverse effects on mechanical properties associated with high monomethacrylate content were found. In addition, some of the quaternary ammonium based monomers exhibit miscibility problems with hydrophobic dimethacrylates [17].

Eugenyl methacrylate monomer (EgMA), a low molecular weight monomer obtained by modifying the chemical structure of eugenol was reported by Rojo et al. [18], which has

a polymerizable methacrylate group that allows facile free radical polymerization reaction while impair desired functionalities [18]. Furthermore, previous studies on rheological properties of the EgMA copolymers confirmed the formation of branching structures with a range of degree of crosslinking that were responsible for the elastic or viscoelastic properties of these systems. In addition, this monomer also demonstrated intrinsically bactericidal properties against different microorganisms including *Streptococcus mutans* [19], which is involved in composite failures associated with secondary caries [20,21].

The purpose of this study was to formulate and characterize new dual cure resin composite materials based on EgMA monomer and Bis-GMA/TEGDMA resin systems for endodontic post cementation and core build-up restoration. The addition of this monomer was expected to enhance the viscoelastic properties, the mechanical response of the composites and potentially impart some antibacterial property to the resin system by virtue of the EgMA residues [19]. The influence of this monomer on curing kinetics, viscosity, physical and mechanical properties of the experimental composites are reported and the results compared with those of a commercially available dual cured resin composite core material.

2. Materials and methods

2.1. Materials

2, 2-Bis [4-(2-hydroxy-3 methacryloyloxypropyl)-phenyl] propane (Bis-GMA) and tri-ethylene glycol dimethacrylate (TEGDMA) were purchased from Esschem Europe Ltd (Durham, UK). Benzoyl peroxide (BPO) and A-174 silane coupling agent (3-Trimethoxysilyl propylmethacrylate) were supplied by Merck (Frankfurt, Germany). Methacryloyl chloride (95%) was purchased from Alfa Aesar, UK. Camphorquinone (CQ), N,N-dimethyl-p-toluidine (DMPT), eugenol and trimethylamine were purchased from Sigma-Aldrich, Company Ltd, Dorset, UK. The fillers used in this study were hydroxyapatite (HA, Plasma Biotol Ltd., Tideswell, Derbyshire, UK) and zirconium oxide (ZrO₂, Fisher Scientific Ltd., Loughborough, UK) with a mean particle size diameter of 3–5 µm and 18 µm respectively, which were silanized according to the method described elsewhere. [22] Solvents used were of HPLC grade from Acros-Organics UK. All other reagents were purchased from Sigma Aldrich and used as received, except BPO that was purified by fractional crystallization from ethanol. A commercially available resin composite material (Clearfil™DC Core plus, Kuraray, Tokyo, Japan) was used as a commercial reference.

2.2. Synthesis and characterization of Eugenyl Methacrylate

EgMA monomer (MW = 232.23 g/mol) was synthesized as reported previously by Rojo et al. [18]. In brief, eugenol (0.061 mol) and triethylamine (0.061 mol) were dissolved in 50 ml of dichloromethane. Methacryloyl chloride (0.076 mol) was dissolved in 10 ml of dichloromethane and then added drop wise whilst the reaction mixture was kept in an ice bath

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