CHARACTERISATION OF DENTAL RESIN COMPOSITE WITH NATURAL AND SYNTHETIC HYDROXYAPATITE-YTTRIA STABILISED ZIRCONIA FILLERS

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ABSTRACT

This study aimed to prepare and characterise dental resin composite with hydroxyapatite-yttria stabilised zirconia (HAp-YSZ) fillers from natural and synthetic sources of hydroxyapatite (HAp). The yttria stabilised zirconia (YSZ) and HAp were used as reinforcement inorganic fillers, while the organic resin were the blended monomers of bisphenol A glycerolate dimethacrylate (Bis-GMA), triethylene glycol dimethacrylate (TEGDMA) and diurethane dimethacrylate (UDMA). The composites were thoroughly blended at ratio of 70:30 wt/wt of fillers and resin. The mixing ratios of fillers (YSZ:HAp) were fixed at 0:100; 10:90; 25:75; 50:50 and 75:25 wt/wt. The composites were moulded and crosslinked under UV-light for 60 s on the both sides of samples surfaces. The degree of conversion, flexural strength (FS), compression strength (CS), Vickers hardness (VH), surface roughness (SR), water sorption, water solubility and cytotoxicity of composites were measured. The results exhibited that the mechanical properties of YSZ-natural HAp composites with ratio of 10:90 wt/wt (FS = 65.61 MPa; CS = 160.34 MPa; VH = 50.80 HV; SR = 120.00 nm) were better performance than YSZ-synthetic HAp composites. Water sorption and solubility of this composite are 25.19 µg mm^{-3} and 5.58 µg mm^{-3}, respectively. The cytotoxicity test showed that this compositeis non-toxic. As a conclusion, HAp-YSZ dental resin composites were successfully prepared and characterised. The implication of this study is that the composite produced shows a potential to be used as dental resin composite.

PENCIRIAN KOMPOSIT RESIN PERGIGIAN DENGAN PENGISI SEMULAJADI DAN SINTETIK HIDROKSIAPATIT- YTTRIA TERSTABIL ZIRKONIA

ABSTRAK

Kajian ini bertujuan untuk menyediakan dan mencirikan komposit resin pergigian dengan pengisi hidroksiapatit-yttria terstabil zirkonia (HAp-YSZ) dari sumber hidroksiapatit (HAp) semulajadi dan sintetik. Yttria terstabil zirkonia (YSZ) dan HAp digunakan sebagai pengisi tak organik, manakala resin organik adalah monomer campuran bisfenol A gliserol dimetakrilat (Bis-GMA), trietilena glikol dimethakrilat (TEGDMA) dan diuretana dimetakrilat (UDMA). Komposit telah dicampur dengan sempurna pada nisbah 70:30 *wt/wt* pengisi dan resin. Campuran pengisi (YSZ:HAp) telah ditetapkan pada nisbah 0:100; 10:90; 25:75; 50:50 dan 75:25 *wt/wt*. Komposit telah dibentuk dan ditaut silang di bawah cahaya UV selama 60 s pada kedua-dua belah permukaan sampel. Darjah penukaran, kekuatan lenturan (FS), kekuatan mampatan (CS), kekerasan Vickers (VH), kekasaran permukaan (SR), penyerapan air, kelarutan air dan kesitotoksian komposit telah diukur. Hasil kajian menunjukkan bahawa sifat mekanik YSZ-Hap komposit semula jadi dengan nisbah 10:90 *wt/wt* (FS = 65.61 MPa; CS = 160.34 MPa; VH = 50.80 HV; SR = 120.00 nm) adalah lebih baik prestasinya daripada komposit HAp-sintetik. Penyerapan air dan kelarutan komposit ini adalah masing-masing 25.19 µg mm^{-3} dan 5.58 µg mm^{-3}. Ujian kesitotoksikan menunjukkan bahawa komposit ini tidak beracun. Sebagai kesimpulan, komposit resin pergigianHAp-YSZ telah berjaya disediakan dan dicirikan. Implikasi kajian ini adalah komposit yang dihasilkan menunjukkan potensi untuk digunakan sebagai komposit resin pergigian.

TABLE OF CONTENT

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

Å	Amstrong
ANOVA	Analysis of variance
ATCC	American Type Culture Collection
BisEMA	ethoxylated bisphenol A dimethacrylate
Bis-GMA	Bisphenol A diglicydyl methacrylate
CaO	Calcium oxide
CO_2	Carbon dioxide
CQ	Champhorquinone
DC	Degree of conversion
DCPD	Dicalcium phosphate dihydrate
DNA	Deoxyribonucleic acid
EDMAB	Ethyl-4-dimethylamino benzoate
FTIR	Fourier Transform Infrared Spectrometer
GIC	Glass ionomer cement
HAp	Hydroxyapatite

HAp-YSZ	Hydroxyapatite-yttria stabilised zirconia
HEMA	2-hydroxyethyl methacrylate
ISO	International Standard Organisation
KBr	Potassium bromide
LED	Light emitting diode
MCP	Monocalcium phosphate
MCPH	Monohydrate calcium phosphate
$NH_4H_2PO_4$	Ammonium dihydrogen phosphate
NHAp	Natural hydroxyapatite
OCP	Octacalcium phosphate
OD	Optical density
RBC	Resin based composite
SD	Standard deviations
SE	Secondary electron
SEM	Scanning Electron Microscope
SHAp	Synthetic hydroxyapatite
STEM	Scanning Transmission Electron Microscopy
TCP	Tricalcium phosphate

TEGDMA	Triethylenglycol-dimethacrylate
UDMA	Urethane dimethacrylate
UTM	Universal Testing Machine
XRD	X-ray powder diffraction
YSZ	Yttria stabilised zirconia
Zr	Zirconia

CHAPTER 1

INTRODUCTION

1.1 Biomaterials

Biomaterials in medical terminology can be defined as any materials, natural or man-made from which biomedical devices are made to replace or restore the function of traumatised or degenerated tissues or organs (heart valves, bone cement and cochlear) into the body through some pathological processes and thus improve the quality of patient's health (Patel & Gohil, 2012; Pavlovic, 2015; Sukaryo, Purnama, A., & Hermawan, 2016). According to some researchers, the study of biomaterials incorporates the element of chemistry, pharmaceutics, biochemistry, physics, medicine, biology and tissue engineering (Tathe, Ghodke, & Pratima Nikalje, 2010). The biomaterials can be used in biomedical applications such as bone plates, contact lens, heart valves, dental implants for tooth fixation and cochlear replacements

(Siraparapu, Bassa, & Sanasi, 2013; Tathe et al., 2010). These cause the application of biomaterials rise over the past decades due to the boost in life expectancy and life's style, population aging and improvement in implant technology (Holzapfel et al., 2013; Siraparapu et al., 2013; Tathe et al., 2010).

In this study of dental biomaterial, the proper materials are used in the mouth as well as those employed in dental laboratory procedures either to restore function or aesthetics caused by developmental disorders, disease or trauma. Restorative dental materials must be durable and biocompatible. Biocompatibility is a crucial factor in which the biomaterial can be accepted by the surrounding tissues and by the body as a whole, do not elicit allergic or immunologic reactions, preventing damage at the surrounding structures, do not trigger an abnormal inflammatory response and do not cause cancer (Siraparapu et al., 2013; Tathe et al., 2010). The optical characteristics of dental materials must be comparable to those of natural teeth. Their mechanical properties of strength and toughness must meet the requirement of the indication range. For example, the requirement strength of an inlay must be lower than dental bridge (Höland, Schweiger, Watzke, Peschke, & Kappert, 2008).

Typically, dental biomaterials are broadly classified into metal and their alloys, ceramic, polymer and composites (Siraparapu et al., 2013). Metals are extensively used as structural components for replacement of tooth structure in the posterior region where the metallic colour is not objectionable. There are two group of metal elements used in dentistry: noble and base metal. Base metal elements are a group of elements which will be oxidized when heated in the open air and quite cheaper compared with precious metals. These elements include titanium, silver, copper, zinc, indium, tin, gallium, nickel and cobalt. The example for noble metal is gold, platinum, palladium, iridium, rhodium, osmium and ruthenium. They are preeminent when used in the mouth and resistance to oxidation as well as tarnishing and corrosion in heating (Azarhoosh, 2017). Although pure metals are sometimes used, alloys often provide improvement in material properties such as strength and corrosion resistance. Low accumulation of plaque, high biocompatibility, good marginal fit and low corrosion are evaluated as it is most essential feature (Siraparapu et al., 2013).

Ceramics are defined as the materials made essentially from non-metallic, inorganic and polymeric materials usually manufactured by sintering at high temperature to achieve desirable properties. Ceramics are hard, brittle, stiff, poor thermal and electrical conductors and good resistance against compressive stresses (Azarhoosh, 2017; Sakaguchi, Ferracane, & Powers, 2018). Alumina (Al_2O_3), zirconia (ZrO_2) and carbon are termed bioinert while bioglass and glass ceramics are bioactive. Calcium phosphate ceramics are categorised as bioresorbable (Muddugangadhar, Amarnath, Tripathi, Dikshit, & Ms, 2012). In this group, hydroxyapatite (HAp) has a dominant place being used for oral and can be found in different parts of the body as a constituent of various types of calcified tissues of tooth enamel, dentin and cementum. Bioinert refers to a material that retains its structure in the body after implantation and does not induce any immunologic host reaction. Ceramics has been used in restorative dentistry as inlays, veneers, full- and partial-coverage crowns, denture teeth and even as bridges which can be made completely from high strength of ceramics (Azarhoosh, 2017).

Polymers are widely used for application including tooth restoratives, sealants, cements, root canal filling materials, denture base and others. With the advancement of material science, new materials produced have properties similar to natural biomaterial. Polymers used in dentistry are vulcanite, celluloid, phenolformaldehyde (Bakelite), polyvinylchloride (PVC) and polymethylmethacrylate (PMMA). The advantages that led to consider PMMA as the most common polymer are low cost, superior aesthetics, low solubility and low water absorption, possibility of repairment convenient and easy processing (Azarhoosh, 2017). Compared with metal, ceramic and composite, polymers have the lowest stiffness, lowest long-term stability in an aqueous environment and the lowest melting or glass transition point (Sakaguchi et al., 2018).

Composites in the term of dentistry are defined as materials of the combination polymer and ceramic where the polymer is used to bind ceramic particles. Dental composite are formable, opaque or translucent and moderate in stiffness and hardness. Polymer matrix composites also known as resin composite are employed as sealants, intracoronal and extracoronal restoration, veneers, denture teeth and cements (Sakaguchi et al., 2018). The advantages of composites include superior aesthetics, ease to bond to tooth tissue, reduce the need for extensive preparation of tooth surfaces, reduce the amount of mercury release in the environment and therefore, reduce corrosion (Azarhoosh, 2017). Composites used in dentistry are composed of organic matrix, inorganic filler components, initiator and coupling agent. The polymer organic matrix is the main organic component consist of monomers that generally are polymerised upon activation by visible light illumination, while inorganic filler particles potentially to increase the mechanical and physical properties of resin based composites (RBCs) (Ansteinsson, 2013). Examples for monomers are bisphenol A diglicydyl methacrylate (Bis-GMA), triethylene glycol dimethacrylate (TEGDMA) and diurethane dimethacrylate (UDMA). Modern RBCs nowadays contain fillers such as quartz, colloidal silica and silica glass (Dafar, 2014). Coupling agent such as silane is purposely to help in binding organic matrix with inorganic filler and to sustain the particle in RBCs in a stable mechanical properties (Khoroushi & Mansoori, 2012; St-pierre, 2011; Trautmann, 2010). Polymerisation initiator is used for chemically activated resin composite which serves as the source of free radicals. A diketone photoactivator of champhorquinone (CQ) normally is used for light-activated resin composites with the help of co-initiator such as ethyl-4-dimethylamino benzoate (EDMAB).

1.2 Dental Restorative Materials

Dental restorative materials are the substances used to repair, replace or restore the function of the patient's teeth and their surrounding structures. Patients may need dental restorative procedures covered by the dental specialty of prosthodontic dentistry, including fillings, veneers, crowns, bridges, full and partial dentures and dental implants due to disease, trauma and aesthetics purposes. The most common dental disease is caries which is treated by the removal of bacteria and the placement of a restorative material.

Restorative materials can be classified into two types which are direct and indirect restorations. Direct restorations are fillings placed directly into a prepared tooth including dental amalgam, RBCs, glass ionomer cements (GICs) and resin-modified glass ionomer cements (RMGICs). They are usually soft and are hardened by a chemical reaction. Once the tooth is prepared by the dentist, the fillings will be placed and adjusted during a single visit. The dentist may choose from a variety of filling options based on the type and location of the filling. Most often these fillings are made of metal or resin. Indirect restorations restoration are the fillings involve customised tooth replacements in the form of inlays, onlays, veneers, crowns and bridges. These fillings often require two or more visits to complete the restorations and are usually made of metal, resin or ceramic materials. They include ceramic, metal ceramic, cast-gold (high noble) alloys and base metal (non-noble) alloys. These indirect materials are used to invent the restorations in the dental laboratory which then are placed in or on the teeth (ADA Council on Scientific Affairs, 2003). Table 1.1 and Table 1.2 show the comparison of some direct and indirect restorative dental materials that mimic the appearance of natural tooth colour.

Table 1.1

Comparison of direct restorative dental materials (ADA Council on Scientific Affairs, 2003).

Factors	Resin based composite	Glass ionomer cements	Resin-modified glass ionomer cements
General description	A mixture of submicron glass filler and acrylic.	A mixture of fluoride containing glass powder and organic acid.	A mixture of sub-micron glass filler with fluoride containing glass powder and acrylic resin.
	Self- or light-hardening at mouth temperature.	Self-hardening and able to release fluoride.	Self- or light-hardening and able to release fluoride.

Principal uses	Esthetic dental fillings and veneers.	Small non-load bearing fillings, cavity liners and cements for crowns and bridges.	
Leakage and recurrent decay	Low leakage when properly bonded to underlying tooth and recurrent decay depends on maintenance of the tooth-material bond.	Low leakage and recurrent decay is comparable to other direct materials. The release of fluoride provide beneficial at high risk for decay.	Low leakage when properly bonded to the underlying tooth and recurrent decay is comparable to other direct materials. The release of fluoride provide beneficial at high risk for decay.
Overal durability	Good in small to moderate size restorations.	Moderate to good in non-load bearing restorations or poor in load bearing	
Cavity preparations considerations	Adhesive bonding permits removing less tooth structure.		
Clinical considerations	Must be placed in a well-controlled field of operation; very little tolerance to presence of moisture during placement.		
Resistance to wear	Resistance is moderate but less so than amalgam.	High wear when placed on chewing surfaces.	
Resistance to fracture	Moderate resistance to fracture in high load restorations.	Low resistance to fracture.	Low to moderate resistance to fracture.
Biocompatibility	Well-tolerated with rare occurrences of allergenic response.		
Post-placement sensitivity	Occurrence of sensitivity highly depends on the ability to adequately bond to the underlying tooth.	Low	Occurrence of sensitivity highly depends on the ability to adequately bond the restoration to the underlying tooth.
Relative Cost to Patient	Moderate and actual cost of fillings depends on their size and technique.		

Table 1.2

Comparison of indirect restorative dental materials (ADA Council on Scientific Affairs, 2003)

Factors	Ceramic (Porcelain)	Metal-ceramic
General description	Porcelain, glass-like fillings and crowns.	Porcelain is fused to provide strength to a filling, crown or bridge.
Principal uses	Inlays, onlays, crowns, strong dental fillings and veneers.	Crowns and fixed bridges.
Leakage and recurrent decay	Sealing ability depends on materials and underlying tooth structure. Recurrent decay depends on maintenance of the tooth material bond.	To provide a good seal against leakage, the common methods are used for placement. The occurrence of recurrent decay is similar to other restorative procedures.
Durability	Excellent durability, brittle and may fracture under heavy biting loads.	Very strong and durable.
Cavity preparation considerations	Requires more aggressive tooth reduction during preparation since strength depends on adequate porcelain thickness.	Creates a stronger restoration than porcelain alone and more tooth reduction is required.
Clinical considerations	Most restorations need multiple appointments and laboratory fabrication because various step procedures require highly accurate clinical and laboratory processing.	
Resistance to wear	Highly resistant to wear but porcelain can rapidly wear opposing teeth if its surface becomes rough. .	
Resistance to fracture	Prone to fracture when placed under tension.	Prone to impact fracture as the metal has high strength.
Biocompatibility	Well tolerated.	Well tolerated but low quality base may cause allergic reactions.
Post-placement sensitivity	Low thermal conductivity reduces the possibility of discomfort from hot and cold.	High thermal conductivity may result in early post-placement discomfort from hot and cold.
Relative cost to patient	Higher and it requires at least two office visits and laboratory services.	

In this study, the RBC is selected to investigate as it is a conservative, economical and simple procedure needed. This direct RBC filling is aimed to focus on the anterior teeth region for restoration treatment. Anterior teeth (Figure 1.1) comprise of incisors and canines which located at the maxillary (upper jaw) and mandibular (lower jaw). Incisor teeth play a role in cutting while canine teeth work for cutting and tearing (Le Révérend, Edelson, & Loret, 2014). Researchers studied that the RBC is used for about 92 % of the anterior restorations as every patient requires a functional and superior aesthetically teeth appealing smile (Nascimento et al., 2010). The RBC on anterior teeth gives positive impact not only to patient's smile but also patient's appearance, self-confidence and overall mental health. This can be observed where patients today frequently visit their dentist as they wish to look more beautiful in social interaction. In fact, the attention appears mainly on mouth and eyes of the face of the person speaking. The aesthetic appearance of the oral region during smiling is obvious part of facial attractiveness (Demir, Oktay, & Topcu, 2017; Gouveia, Theobaldo, Vieira-Junior, Lima, & Aguiar, 2017).

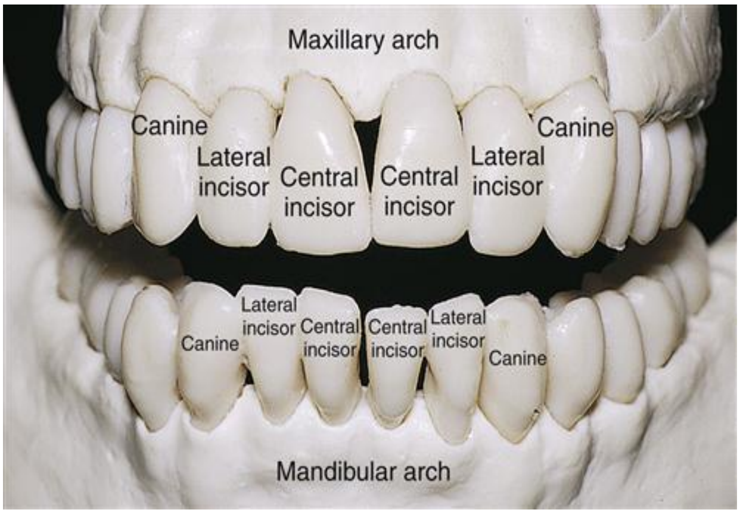

Figure 1.1. The anterior teeth including incisors and canines. Adapted from https://pocketdentistry.com/16-permanent-anterior-teeth/#s0010

1.3 Problem Statement

A wide variety of dental resin based composite materials are available in the market today due to the unlimited revolution of composite especially at the anterior teeth region which involve class III and class IV restoration. There are three examples available commercial composite for clinical use including charisma (HeraeusKulzer, Hanau, Germany), Filtek Z250 (3M/ESPE), and Filtek Z350 XT (3M/ESPE). The charisma commercial composite consist of Ba-Al-B-F-Si glass and pyrogenic SiO_2 while Filtek 250 and Filtek Z350 XT composes of silica nanoparticles and silane-coated zirconia prior to incorporation into resin matrix (Abuelenain & Neel, 2015; Baldissera et al., 2013). Even though these commercial composites exhibit high mechanical properties in term of compression strength and flexural strength, they are popular with the aesthetic problem appearance such as staining or discolouration, high water sorption, shrinkage problem and inadequate degree of conversion (DC) (Schneider, Cavalcante, & Silikas, 2010; E. M. da Silva, Almeida, Poskus, & Guimarães, 2008). The staining or discolouration of commercial composites occur due to the insufficient polymerisation time, surface roughness, contact time with or immersion in colouring environments and the type of resin composite used (Ceci et al., 2017; Duc, Betrisey, Di Bella, Krejci, & Ardu, 2018).

Furthermore, the commercial composites are lack of hydroxyapatite (HAp) content that can be found in enamel and dentine of teeth. About 95 % of HAp content in enamel is biocompatible and able to release the calcium ions which provide the remineralisation of teeth (Ganss, Lussi, & Schlueter, 2012; Okulus, Buchwald, Szybowicz, & Voelkel, 2014). This inorganic material of HAp is much cheaper than commonly used glass fillers, gives an appearance of bright white that mimic the natural of tooth colour and thus satisfy the aesthetic aspect (Calabrese et al., 2016; Okulus et al., 2014). However, the low mechanical strength and fracture toughness of HAp has limits its usage for hard tissues. The improvement of the mechanical properties of HAp can be extending its scope of applications by adding a second phase of yttria stabilised zirconia (YSZ) (Balazsi, Gergely, Sahin, & Goller, 2011; Guo, Khor, Boey, & Miao, 2003).

Adding YSZ in HAp has been improved fatique resistance and strength compared HA alone. YSZ is a biocompatible material with better mechanical properties which makes it suitable as a structural material in dentistry. Transformation toughening is the mechanism behind the mechanical behaviour of YSZ. At the above 1000 °C in pure zirconia, the tetragonal phase is maintain to room temperature by adding a suitable amount of yttria which employ as a stabilizer. With 3 mol.% of yttria in zirconia, a fair combination of bending strength and fracture toughnesscan be accomplished (Camposilvan, Marro, Mestra, & Anglada, 2015).

For this research, the use of HAp with the combination of YSZ in fabricating dental composite is a challenge. This is because the RBCs using HAp from natural source of fish scale has never been studied by any researcher. Therefore, the properties of the fish scale in RBCs are unknown. It perhaps could contribute in the effort to improve the performance of restorative composite filling materials that potentially to improve the mechanical properties of teeth and overcome the biological concern in mouth such as chronic inflammation in pulp tissues.

1.4 Objectives of Study

The objectives of the study are as follows:

i) To prepare the resin composite of HAp-YSZ from natural and synthetic sources at different ratios of Hap and YSZ.
ii) To characterise and compare the mechanical properties and morphology of resin composite in both natural and synthetic sources of HAp.