

**CHARACTERIZATION OF POLYMETHYL METHACRYLATE /
FEATHER FIBER / MONTMORILLONITE COMPOSITES FOR DENTAL
POST APPLICATION**

SITI MAIZATUL FARHAIN BINTI SALEHUDDIN

UNIVERSITI TEKNOLOGI MALAYSIA

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FIBER / MONTMORILLONITE COMPOSITES FOR DENTAL POST
APPLICATION

SITI MAIZATUL FARHAIN BINTI SALEHUDDIN

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To my beloved and greatest parents, Salehuddin bin Hanafiah and Hasinah bte Atan,
my brothers, Mohd Faizal bin Salehuddin and Muhamad Hanafi bin Salehuddin,
who are infinitely precious to me.

&

my friends, who were there for me.

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ABSTRACT

The aim of this study was to explore the potential of the goose feather fiber for the development of dental post for the treatment of pulpless teeth. In this research, polymethyl methacrylate (PMMA), feather fiber (FF), glass fiber (GF) and montmorillonite (MMT) composites were prepared using Brabender internal mixer. FF/PMMA and GF/PMMA composites were produced in the range of 1, 3, 5, 7 and 10 phr composition of feather and glass fiber respectively while FF/PMMA/MMT and GF/PMMA/MMT composites were produced in the range of 1, 3, 5, 7 and 10 phr composition of feather and glass fiber with the addition of 4 wt % of MMT. The performance of FF/PMMA was compared with GF/PMMA composites. The mechanical properties of composites were studied through the flexural test. The changes on the glass transition temperature (T_g) and thermal stability of composites was studied through differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). Morphology of the composites was analyzed using scanning electron microscope (SEM) and transmission electron microscopy (TEM). The chemical structure of composites was studied by using Fourier transform infrared (FTIR) spectroscopy. The structural characterization of silicate layer was investigated using X-ray diffraction (XRD). Biological properties were determined through in-vitro cytotoxicity test to demonstrate the biocompatibility of the composites. The result of flexural properties of FF/PMMA and GF/PMMA composites showed the addition of FF and GF significantly increased the strength and stiffness of composites. The composites containing 10 phr of both fiber had the highest flexural strength and modulus. Similar result was obtained for FF/PMMA/MMT and GF/PMMA/MMT composites. DSC results showed that the T_g of the PMMA matrix increased with increasing of fiber loading. The incorporation of MMT on FF/PMMA and GF/PMMA composites exhibited higher rigidity on the chain of the composites significantly. TGA curves exhibited a significant improvement in thermal stability for FF/PMMA/MMT and GF/PMMA/MMT composites with the incorporation of MMT. SEM analysis of the composites showed a relatively uniform distribution of the fiber in the polymer matrix and compatibility between the matrix and fiber. The TEM image showed that the presence of exfoliated structure in FF/PMMA/MMT and GF/PMMA/MMT composites. In-vitro cytotoxicity test of the composites showed no noticeable cytotoxicity indicated the excellent biocompatibility in biological properties of composites.

ABSTRAK

Tujuan kajian ini adalah untuk meneroka potensi terhadap gentian bulu angsa untuk menghasilkan pos pergigian bagi rawatan gigi tanpa pulpa. Dalam kajian ini, komposit adalah berasaskan polimetil metakrilat (PMMA), gentian bulu (FF), gentian kaca (GF) dan montmorilonit (MMT) disediakan dengan menggunakan pencampur dalaman *Brabender*. Komposit FF/PMMA and GF/PMMA dihasilkan dengan julat komposisi 1, 3, 5, 7 dan 10 phr daripada gentian bulu dan kaca manakala komposit FF/PMMA/MMT dan GF/PMMA/MMT pula dihasilkan dalam julat komposisi 1, 3, 5, 7 dan 10 phr daripada gentian bulu dan kaca dengan penambahan 4 peratus berat daripada MMT. Prestasi antara komposit FF/PMMA dan GF/PMMA telah dibandingkan. Ciri-ciri mekanikal komposit telah dikaji melalui ujian lenturan. Perubahan dalam suhu peralihan kaca (T_g) dan kestabilan terma komposit telah dikaji menggunakan pengimbasan kalorimeter pengkamilan (DSC) dan analisis termogravimetri (TGA). Morfologi komposit telah ditentukan dengan menggunakan mikroskop imbasan elektron (SEM) dan mikroskop elektron transmisi (TEM). Struktur kimia komposit telah dikaji dengan menggunakan spektroskopi sinar inframerah Fourier (FTIR). Pencirian struktur lapisan silikat telah dikaji dengan menggunakan pembelauan sinar-X (XRD). Sifat biologi telah ditentukan dengan menggunakan kaedah ujian sitotoksik in-vitro untuk menunjukkan keserasian bagi komposit. Hasil daripada sifat lenturan komposit FF/PMMA dan GF/PMMA menunjukkan penambahan FF dan GF dengan ketara meningkatkan kekuatan dan kekukuhan komposit. Komposit yang mengandungi 10 phr gentian mempunyai kekuatan lenturan dan modulus lenturan yang paling tinggi. Hasil yang sama juga diperoleh daripada komposit F/PMMA/MMT dan GF/PMMA/MMT. Keputusan DSC menunjukkan T_g telah meningkat dengan penambahan komposisi gentian. Penambahan MMT pada komposit FF/PMMA dan GF/PMMA didapati mempamerkan ketegaran yang lebih tinggi dalam rangkaian komposit. Keluk TGA menunjukkan peningkatan yang ketara dalam kestabilan terma untuk komposit FF/PMMA/MMT dan GF/PMMA/MMT komposit dengan penambahan MMT. Analisis SEM menunjukkan taburan gentian yang agak seragam di dalam matrik polimer dan mempunyai keserasian antara matrik dan gentian. Imej TEM pula menunjukkan kehadiran struktur exfoliasi dalam komposit FF/PMMA/MMT dan GF/PMMA/MMT. Ujian sitotoksik in-vitro bagi komposit tidak mempamerkan sitotoksik ketara, ini menunjukkan komposit mempunyai keserasian-bio yang baik dalam sifat biologi.

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LIST OF ABBREVIATIONS

Au-Pt	-	Gold-Platinum
ASTM	-	American standards test method
Bis-GMA	-	Bisphenol-A glycidyl methacrylate
CF	-	Carbon fiber
CEC	-	Cation exchange capacity
CFF	-	Chicken feather fiber
DSC	-	Differential scanning calorimetry
DTG	-	Derivative thermogram
FDT	-	Final decomposition temperature
FF	-	Feather fiber
FRC	-	Fiber reinforced composite
FTIR	-	Fourier transform infrared
GF	-	Glass fiber
GPa	-	Giga pascal
HDPE	-	High density polyethylene
HB	-	Horizontal burning
IPN	-	Interpenetrating polymer network
ISO	-	International standard organization
KBr	-	Potassium bromide
LDPE	-	Low density polyethylene
MMA	-	Methylmethacrylate
MMT	-	Montmorillonite
MTT	-	Tetrazolium
NaOH	-	Sodium hydroxide
Ni-Cr	-	Nickel- Chromium
OLS	-	Organically modified layered
PBS	-	Phosphate buffered saline

PLS	-	Polymer layered silicate
PMSO	-	Dimethyl sulfoxide
PMMA	-	Polymethylene methacrylate
PVOH	-	Polyvinyl alcohol
pH	-	Potential hydrogen
phr	-	Parts per hundred
r-HDPE	-	Recycle high density polyethylene
RGR	-	Relative growth rate
SEM	-	Scanning electron microscopy
TCPS	-	Tissue culture polystyrene
TEM	-	Transmission electron microscopy
Ti	-	Titanium
TGA	-	Thermogravimetric analysis
UHMWPE	-	Ultra high molecular weight polyethylene
UL	-	Underwriters laboratories
UP		Unsaturated Polyester
XRD	-	X-ray diffraction

LIST OF SYMBOLS

cm	-	Centimeter
cm ⁻¹	-	Per centimeter
g	-	Gram
g/mol	-	Gram per mol
g/10 min	-	Gram per 10 minutes
g/cm ³	-	Gram per centimeter cubic
mg	-	Miligram
mm	-	Milimeter
mm ³	-	Milimeter cubic
mm min ⁻¹	-	Milimeter per minute
M	-	Molarity
mequiv/g	-	Miliequivalent per gram
N	-	Newton
nm	-	Nanometer
kg/cm ²	-	Kilogram per centimeter square
kg.cm/cm	-	Kilogram centimeter per centimeter
kV	-	Kilo voltage
rpm	-	Revolutions per minute
T _g	-	Glass transition temperature
T _{50%wt}	-	Temperature at 50 % weight loss
T _{max}	-	Maximum temperature rate weight loss
w _i	-	Weight fraction
wt %	-	Weight percent
° C/min	-	Degree celsius per minute
° C	-	Celsius
° F	-	Fahrenheit
%	-	Percent

	-	Beta
μm	-	Micrometer
μL	-	Microliter
α	-	Alpha
	-	Angstrom

CHAPTER 1

INTRODUCTION

1.1 Background of Research

Endodontically treated teeth are known to present a higher risk of biomechanical failure than vital teeth. Posts are generally indicated to restore missing tooth structure and pulpless teeth (Sorensen and Martinoff, 1984). Post which will be act as an anchor for the placement of crown is used to reinforce the remaining tooth structure, when the amount of coronal tooth structure remaining is small (Hoag and Dwyer, 1982 ; Lovdahl and Nicholls, 1977). The choice of an appropriate restoration for endodontically treated teeth is guided by the strength and esthetics.

Endodontically treated teeth have a little coronal tooth tissue remaining that requires a post, core and crown. Traditionally, these posts have been cast or machined from metal and it is acknowledged that such posts weaken the roots and lead to root fracture. Stainless steel, brass, zirconium oxide and titanium alloys posts have been used (Burgess *et al.*, 1992). It is based on the assumption that the post should be rigid, high strength, excellence in ductility and resistance to wear. In 1990, Duret *et al.* (1990) described a non-metallic material for the fabrication of posts based on the carbon fiber reinforced principle. Laboratory-based studies have shown that these posts have a high tensile strength (King and Setchell, 1990) and modulus of elasticity, similar to dentine (Asmussen *et al.*, 1999). Previously, rigid metal posts resisted lateral forces without distortion and this resulted in stress transfer to the less rigid dentine causing potential root cracking and fracture.

In 1992, glass fiber reinforced resin post systems were introduced (Goldberg and Burstone, 1992). The posts are composed of unidirectional glass fibers where it is embedded in a resin matrix. Commonly polymer used is epoxy polymers with a high degree of monomer conversion and a highly cross-linked structure (Goldberg and Burstone, 1992). An advantage of glass fibers is that they distribute stress over a broad surface area. The post begins to show an evidence of micro-fractures when the load threshold is increased. (Pest *et al.*, 2002). Consequently, fiber reinforced posts are reported to reduce the risk of tooth fractures and display higher survival rates than teeth restored with rigid zirconia posts (Manocci *et al.*, 1999). Lippo *et al.* (2004) have found that in vitro studies, FRC-posts might possess some benefits over metal posts due to their modulus of elasticity being closer to that of dentin.

Polymers are widely used in a large of number in various medical product applications due to variety of compositions, properties and forms such as solids, fibers, gels, films and can be fabricated readily into structure and complex shapes. Besides, from the beginning of biometric development, a nondegradable polymer as polymethyl methacrylate (PMMA) were used in various biomedical applications (Whitaker III, 1996) and one of the best choices for implant materials intended to perform a function for an extended period of time. The first use of PMMA as a dental device was for the fabrication of complete denture bases, but its qualities of biocompatibility, reliability, relative ease of manipulation, and low toxicity were soon seized upon and incorporated by many other medical fields. The popularity of PMMA is associated with its favorable working characteristics, processing ease, accurate fit, stability in the oral environment, superior esthetics, and the ability to be used with inexpensive equipment. Yuhong *et al.* (2010) study on dental plastic interpenetrating polymer network (IPN) post composite by the method of step-by-step polymerization with bisphenol A-glycidyl methacrylate (Bis-GMA)/polymethyl methacrylate (PMMA) found that the composite material has no irritations to the oral mucosa, has no short toxicity, it does not lead to acute haemolysis, and has good biological properties.

Nowadays, the use of feather fiber as reinforcement materials for polymer matrix has an attraction to industry of engineering. A few studies have been investigated on feather fibers (FF), for bio composite material applications, they found that the materials which derived from feathers can be used as the reinforcing materials in polymer matrix composites and give much advantageous. These fibers act as an interesting replacement of conventional synthetic fiber, such as carbon, boron, glass and aramid fiber in composite product design. Because of its recyclable, renewable resources characteristic, this reinforcement has emerged gradually as a new class of reinforcement for bio composite of polymer. So, an exploration made in the use of FF in the development of bio composite dental posts. Thus, FF inclusion in a composite dental post would potentially lower the overall density, compared with the density in a composite of synthetic reinforcement. The manufacturing process is an essential in developing the engineering design, product application of FF based composite. Hence, the full understanding on mechanical properties, thermal stability behavior, surface morphologies, nature bonding characteristic exist between FF and polymer matrix also an environmental influences due to moisture and chemical attacks must be explored in order to achieve an excellent combination of final product at the end.

1.2 Problem Statement

Metal posts which are custom and prefabricated have been the standard for many years. However, the metal posts, create a negative impact include: high rigidity (high E modulus) with resulting concomitant risk of a rising hypercritical stress peaks and the problem of corrosion. Therefore, to address the need for a more esthetic material in the anterior region, an effort is invested in making use of nonmetallic posts which have been introduced. There have been significant advances on last several years in the development of bond able, esthetic post, fiber reinforcement to reinforce treated teeth of endodontical (Ferrari *et al.*, 2000). The soft tissues adjacent to the root surface can cause shadowing with the presence of metal post, in which adversely affect the esthetic results required of ceramic restoration and bonded resin in the anterior region (Takeda *et al.*, 1996). An allergic

reaction exhibited to many people with the presence of metallic devices in the body. The devices made of polymer composites eliminate such allergic reaction. In the United States in 1995, the earliest fiber reinforced composite posts were introduced and were fabricated with carbon fibers. They had excellent physical properties (Sidoli *et al.*, 1997) but because of the carbon, the fibers turned black. The glass fiber posts were introduced for use after endodontic therapy instead of ceramic post and metal alloy.

In the last few decades, feather fibers provide many advantages which give an attraction to many researchers over the conventional reinforcement. FF is considered as a significant waste material and economical disposal of FF is a problem of growing concern to the poultry processing industries. Therefore, a study of the properties of polymethyl methacrylate / feather fiber composites for dental post application was done in an attempt to enhance the properties of composites. Incorporating feather fibers which are treated using an alkali solution for compatibility and improving the properties of the composites are the principal problems that paving the track of this thesis work. In this study, sodium hydroxide will be used to treat the feather fiber that can modify FF/PMMA and FF/PMMA/MMT composites with a variation of PMMA and FF contents as a comparison to GF/PMMA and GF/PMMA/MMT composites. A series of tests were conducted to investigate the respective formulation properties.

1.3 Objectives of Research

The overall objective of this study is to develop and investigate the performance of dental post product made of FF/PMMA, GF/PMMA, FF/PMMA/MMT and GF/PMMA/MMT with focus on mechanical properties, thermal stability, the morphology, the chemical structure and the structural characterization analysis of the composite with various composite compositions. The principal objectives of this study were as follows:

- (i) To study the effect of fiber loading on the FF/PMMA and GF/PMMA composites on flexural, thermal analysis, morphological behavior and chemical structure.
- (ii) To investigate the incorporation of MMT on the FF/PMMA and GF/PMMA composites on flexural, thermal analysis, morphological behavior, chemical structure and structural characterization analysis.
- (iii) To analyze the biological properties of the FF/PMMA, GF/PMMA, FF/PMMA/MMT and GF/PMMA/MMT composites.

1.4 Scope of Research

In order to achieve the objectives of the research the following activities were carried out:

1. Sodium hydroxide (NaOH) and distilled water are used in the preparation of feather fiber treatment. Molarity of NaOH were used 0.5 M. The fiber lengths received ranging from 0.5 – 4.0 cm. Uniform lengths are obtained after grinding and sieving.

2. Composite fabrication was conducted via the Brabender internal mixer process of compounding. Followed by compression molding to produce the composite film.
3. Flexural testing was carried out to evaluate the strength and modulus of the composites.
4. The characterization of feather fiber was evaluated by fourier transform infrared spectroscopy. Thermogravimetric analysis (TGA) and differential scanning calorimeter (DSC) was carried out to study thermal stability of the composites. Scanning electron microscopy (SEM) was carried out to study the microstructure by using flexural fracture specimen and transmission electron microscopy (TEM) to investigate the morphology and the actual structure or pattern of dispersion in the composites. Fourier transform infrared (FTIR) spectroscopy was carried out to identify the chemical structure of the composites. X-ray diffraction (XRD) was carried out to investigate the structural characterization analysis of the composites. Biological properties behavior was carried out to demonstrate that the composites are compatible.

1.5 Significance of Research

The research is expected to explore the utilization of feather fiber (FF) as reinforcement in the development of dental post for treatment of pulpless teeth. The potential commercialization of feather fiber is expected as a potential replacement, alternative or substitute material for currently used conventional reinforcement such glass fiber (GF) reinforced composite and primarily due to its lower cost and environmental friendly. The incorporation of montmorillonite has significantly improved the properties of composites.

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