FABRICATION AND EVALUATION OF DENTAL RESIN COMPOSITES DEVELOPED FROM BIS-GMA/TEGDMA WITH TREATED CERAMIC FILLERS

by

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Dedication

To the spirit of my father dear, my beloved mother and my family

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TABLE OF CONTENTS

ACKNOWLEDGEMENTS iii
TABLE OF CONTENTS iv
LIST OF TABLES viii
LIST OF FIGURES ix
LIST OF ABBREVIATIONS xiv
LIST OF SYMBOLS xvi
ABSTRAK xviii
ABSTRACT xx

CHAPTER 1- INTRODUCTION 1
1.1 Research background 1
1.2 Problem statement 3
1.3 Research objectives 5
1.4 Outline of the thesis 6

CHAPTER 2- LITERATURE REVIEW 7
2.1 Introduction 7
2.2 Anatomy and physiology of tooth structures 7
2.3 Saliva 9
2.4 Dental caries 10
2.5 Classification of dental caries 11
2.6 History and development of the dental resin restorations 16
2.7 Classification of dental restorations 17
2.7.1 Indirect dental restorations 17
2.7.2 Direct dental restorations 18
2.7.2.1 Amalgam 18
2.7.2.2 Compomers 19
2.7.2.3 Glass ionomers 20
2.7.2.4 Hybrid ionomers 20
2.7.2.5 Dental resin composites 21
2.8 Light curing unit 29
2.9 Classification of dental resin composites 31
2.9.1 Classification according to filler size 31

2.9.2 Classification according to method of activation 32
2.10 Biomaterials 32
2.10.1 Classification of biomaterials 34
2.10.1.1 Ceramics 34
2.10.1.2 Metals 36
2.10.1.3 Polymers 37
2.10.1.4 Composites 38
2.11 Mechanical properties of dental resin composites 39
2.12 Environmental properties of dental resin composites 42
2.13 Biological properties of dental resin composites 45
2.14 Radiopacity property of dental resin composites 46
2.15 Biocompatibility 48
2.16 Previous studies on the dental resin composites 49

CHAPTER 3- MATERIALS AND METHODOLOGY 57
3.1 Introduction 57
3.2 Materials 57
3.2.1 Resin matrix 57
3.2.2 Reinforcement fillers 58
3.2.2.1 Silica 58
3.2.2.2 Alumina 59
3.2.2.3 Zirconia 59
3.2.3 Coupling agent 59
3.2.4 Artificial saliva 60
3.3 Methodology 60
3.3.1 Characterization of raw filler particles 60
3.3.1.1 Fillers particle size and particle size distribution analysis 60
3.3.1.2 Fillers particles morphology and elemental analysis 61
3.3.1.3 Fillers elements composition analysis 61
3.3.1.4 Fillers particles phase identification 62
3.3.2 Treatment of filler particles surface 62
3.3.3 Characterization of treated filler particles 63
3.3.3.1 Fourier transform infrared spectroscopy 63
3.3.4 Preparation of the dental resin composites 64

3.3.4.1 First stage 64
3.3.4.2 Second stage 66
3.3.4.3 Third stage 67
3.3.5 Samples polymerisation process 68
3.3.6 Preparation of the artificial saliva 69
3.3.7 Characterisation of the samples of dental resin composites 70
3.3.7.1 Physical properties 70
3.3.7.2 Mechanical properties 74
3.3.7.3 Fracture surface morphology 77
3.3.7.4 Thermal characterization 77
3.3.7.5 Environmental properties 78
3.3.7.6 Biological evaluation 80
3.3.7.7 Statistical analysis 84

CHAPTER 4 - RESULTS AND DISCUSSION 85
4.1 Characterization of raw materials 85
4.1.1 Fillers particles size and particle size distribution analysis 85
4.1.2 Filler particles morphology and elemental analysis 87
4.1.3 Fillers elemental composition analysis 90
4.1.4 Fillers particles phase identification 91
4.2 Characterization of the treated filler particles 93
4.3 Effect of individual treated fillers loading on the dental resin composite 98
4.3.1 Physical properties 98
4.3.1.1 Density and porosity 98
4.3.1.2 Filler weight and volume fraction 101
4.3.1.3 Radiopacity 103
4.3.2 Mechanical properties 105
4.3.2.1 Fracture toughness 105
4.3.2.2 Flexural strength 110
4.3.2.3 Flexural modulus 111
4.3.2.4 Vickers hardness 113
4.3.3 Thermal properties 115
4.3.3.1 Thermogravimetric analysis and derivative thermogravimetry 115
4.3.3.2 Differential scanning calorimetry 120

4.3.4 Environmental properties 124
4.3.4.1 Water absorption 124
4.3.4.2 Water solubility 130
4.3.4.3 Artificial saliva absorption 134
4.3.4.4 Artificial saliva solubility 139
4.4 Effect of mixture fillers particles on the dental resin composite 143
4.4.1 Radiopacity 143
4.4.2 Fracture toughness 145
4.4.3 Flexural strength 147
4.4.4 Flexural modulus 149
4.5 Effect of optimum ratio of mixture fillers particles loading 151
4.5.1 Radiopacity 151
4.5.2 Fracture toughness 154
4.5.3 Flexural strength 156
4.5.4 Flexural modulus 158
4.5.5 Vickers Hardness 160
4.5.6 Water absorption 163
4.5.7 Water Solubility 166
4.5.8 Artificial saliva absorption 169
4.5.9 Artificial saliva solubility 172
4.6 Biological evaluation of optimum dental resin composite composition 175
4.6.1 Direct and indirect contact test 175
4.6.2 Test on extract 180

CHAPTER 5 - CONCLUSIONS AND RECOMMENDATIONS 184
5.1 Conclusions 184
5.2 Recommendations for further study 185

REFERENCES 186

LIST OF PUBLICATIONS 218

LIST OF AWARDS 219

LIST OF TABLES

Table Page

Table 2-1: Physical characteristics of alumina bioceramics (Hench, 2013). 25

Table 2-2: The main properties of zirconia (Turner, 2009). 27

Table 2-3: Medical and dental biomaterials and their applications (Davis 2003).....33

Table 4-1: Chemical composition (wt%) of the SiO_2 particles by XRF technique. ...90

Table 4-2: Chemical composition (wt%) of the Al_2O_3 particles by XRF technique. .90

Table 4-3: Chemical composition (wt%) of the ZrO_2 particles by XRF technique....90

Table 4-4: Filler weight fraction and volume fraction of the DRC reinforced with different ratios of SiO_2, Al_2O_3 and ZrO_2 particles. 102

LIST OF FIGURES

Figure Page

Figure 2.1: Human tooth structure. 9
Figure 2.2: Nursing bottle rampant caries. 12
Figure 2.3: Classification of dental caries: (a & b) class I, (c & d) class II, (e) class III, (f) class IV, (g) class V and (h) class VI. 15
Figure 2.4: Amalgam restoration. 19
Figure 2.5: Dental resin composites restoration: (a) anterior and (b) posterior teeth 21
Figure 2.6: Position of anterior and posterior teeth for upper teeth (Permanent, 2014) 40
Figure 2.7: The radiopacity of dental restorative materials (Amanda, 2014). 47
Figure 3.1: Chemical structures of: (a) Bis-GMA, (b) TEGDMA, (c) DMAEMA and (d) CQ 58
Figure 3.2: Chemical structure of γ-MPS 59
Figure 3.3: Treatment of fillers particles 63
Figure 3.4: Flow chart of the preparation of the resin matrix. 64
Figure 3.5: Flow chart of the preparation of the DRC of the first stage. 66
Figure 3.6: Flow chart of the preparation of the DRCs of the second stage. 67
Figure 3.7: Flow chart of the preparation of the DRCs of the third stage 68
Figure 3.8: Optical densities of aluminium plate (ISO 4049:2009) 73
Figure 3.9: Schematic diagram of a three point bending test. 75
Figure 4.1: Graph of particle size and particle size distribution analysis of SiO_2. 86
Figure 4.2: Graph of particle size and particle size distribution analysis of Al_2O_3. 87
Figure 4.3: Graph of particle size and particle size distribution analysis of ZrO_2. 87
Figure 4.4: SEM image and EDX spectrum of SiO_2 particles. 89
Figure 4.5: SEM image and EDX spectrum of Al_2O_3 particles. 89
Figure 4.6: SEM image and EDX spectrum of ZrO_2 particles. 89
Figure 4.7: X-ray diffraction pattern of SiO_2 particles (ICCD 01-077-1060). 91
Figure 4.8: X-ray diffraction pattern of Al_2O_3 particles (ICCD 00-010-0173). 92
Figure 4.9: X-ray diffraction pattern of ZrO_2 particles (ICCD 03-065-1022). 92
Figure 4.10: FTIR spectra of SiO_2 particles: (a) before treatment and (b) after treatment with γ-MPS. 94

Figure 4.11: FTIR spectra of Al_2O_3 particles: (a) before treatment and (b) after treatment with γ-MPS. 96
Figure 4.12: FTIR spectra of ZrO_2 particles: (a) before treatment and (b) after treatment with γ-MPS. 97
Figure 4.13: Density of the resin matrix reinforced with different ratios of the SiO_2, Al_2O_3 and ZrO_2 particles. 99
Figure 4.14: Porosity of the resin matrix reinforced with different ratios of the SiO_2, Al_2O_3 and ZrO_2 particles. 100
Figure 4.15: Optical density of resin matrix without and with differental ratio of SiO_2, Al_2O_3 and ZrO_2 particles. 103
Figure 4.16: X-ray image of aluminum plate and resin matrix with out and with differental ratio of SiO_2, Al_2O_3 and ZrO_2 particles 104
Figure 4.17: Effect SiO_2, Al_2O_3 and ZrO_2 particles loading on the K_{IC} of the DRC, respectively. 106
Figure 4.18: SEM image of the fracture surface of the resin matrix reinforced with (a) 40 wt%, (b) 50 wt%, (c) 60 wt% and (d) 70 wt% of SiO_2 particles. 108
Figure 4.19: SEM image of the fracture surface of the resin matrix reinforced with (a) 40 wt%, (b) 50 wt%, (c) 60 wt% and (d) 70 wt% of Al_2O_3 particles. 109
Figure 4.20: SEM image of the fracture surface of the resin matrix reinforced with (a) 40 wt%, (b) 50 wt%, (c) 60 wt% and (d) 70 wt% of ZrO_2 particles. 109
Figure 4.21: Effect SiO_2, Al_2O_3 and ZrO_2 particles loading on the FS of the DRCs, respectively. 111
Figure 4.22: Effect SiO_2, Al_2O_3 and ZrO_2 particles loading on the FM of the DRCs, respectively. 112
Figure 4.23: Effect of SiO_2, Al_2O_3 and ZrO_2 particles loading on the: (a) Vickers hardness, and (b) hardness improvement of the DRCs, respectively. 114
Figure 4.24: (a) thermogravimetric analysis and (b) derivative thermogravimetry curves of unfilled and filled resin matrix with different ratio of SiO_2 particles. 117
Figure 4.25: (a) thermogravimetric analysis and (b) derivative thermogravimetry curves of unfilled and filled resin matrix with different ratio of Al_2O_3 particles. 118

Figure 4.26: (a) thermogravimetric analysis and (b) derivative thermogravimetry curves of unfilled and filled resin matrix with different ratio of ZrO_2 particles. 119
Figure 4.27: DSC curves of cured resin matrix; 1st run (black line) and 2nd run (red line). 122
Figure 4.28: DSC curves of SiO_2 filled resin matrix (a) 40 wt%, (b) 50 wt%, (c) 60 wt% and (d) 70 wt%; 1st run (black line) and 2nd run (red line). 122
Figure 4.29: DSC curves of Al_2O_3 filled resin matrix (a) 40 wt%, (b) 50 wt%, (c) 60 wt% and (d) 70 wt%; 1st run (black line) and 2nd run (red line) 123
Figure 4.30: DSC curves of ZrO_2 filled resin matrix (a) 40 wt%, (b) 50 wt%, (c) 60 wt% and (d) 70 wt%; 1st run (black line) and 2nd run (red line). 124
Figure 4.31: Effect of SiO_2 particles loading at different ratios on the absorption of the DRCs: (a) absorption (%) and (b) absorption ($\mu g/mm^3$) during 28 days of immersion in water. 127
Figure 4.32: Effect of Al_2O_3 particles loading at different ratios on the absorption of the DRCs: (a) absorption (%) and (b) absorption ($\mu g/mm^3$) during 28 days of immersion in water. 128
Figure 4.33: Effect of ZrO_2 particles loading at different ratios on the absorption of the DRCs: (a) absorption (%) and (b) absorption ($\mu g/mm^3$) during 28 days of immersion in water. 129
Figure 4.34: Effect of SiO_2 particles loading at different ratios on the solubility of the DRCs: (a) weight loss (%) and (b) rate of solubility at 28 days of immersion in water. 131
Figure 4.35: Effect of Al_2O_3 particles loading at different ratios on the solubility of the DRCs: (a) weight loss (%) and (b) rate of solubility at 28 days of immersion in water. 132
Figure 4.36: Effect of ZrO_2 particles loading at different ratios on the solubility of the DRCs: (a) weight loss (%) and (b) rate of solubility at 28 days of immersion in water. 133
Figure 4.37: Effect of SiO_2 particles loading at different ratios on the absorption of the DRCs: (a) absorption (%) and (b) absorption ($\mu g/mm^3$) during 28 days of immersion in AS. 136

Figure 4.38: Effect of Al_2O_3 particles loading at different ratios on the absorption of the DRCs: (a) absorption (%) and (b) absorption (µg/mm^3) during 28 days of immersion in AS. 137
Figure 4.39: Effect of ZrO_2 particles loading at different ratios on the absorption of the DRCs: (a) absorption (%) and (b) absorption (µg/mm^3) during 28 days of immersion in AS. 138
Figure 4.40: Effect of SiO_2 particles loading at different ratios on the solubility of the DRCs: (a) weight loss (%) and (b) rate of solubility at 28 days of immersion in AS..... 140
Figure 4.41: Effect of Al_2O_3 particles loading at different ratios on the solubility of the DRCs: (a) weight loss (%) and (b) rate of solubility at 28 days of immersion in AS..... 141
Figure 4.42: Effect of ZrO_2 particles loading at different ratios on the solubility of the DRCs: (a) weight loss (%) and (b) rate of solubility at 28 days of immersion in AS..... 142
Figure 4.43: Optical density of the DRCs reinforced with ratio of 60 wt% of the (SiO_2, Al_2O_3 and ZrO_2) as mixture filler particles. 144
Figure 4.44: The X-ray image of the DRCs reinforced with ratio of 60 wt% of the (SiO_2, Al_2O_3 and ZrO_2) as mixture filler particles. 144
Figure4.45: Effect of (SiO_2, Al_2O_3 and ZrO_2) as mixture filler particles at ratio of 60 wt% on the K_{IC} of the DRCs. 146
Figure 4.46: Effect of (SiO_2, Al_2O_3 and ZrO_2) as mixture filler particles at ratio of 60 wt% on the FS of the DRCs. 148
Figure 4.47: Effect of (SiO_2, Al_2O_3 and ZrO_2) as mixture filler particles at ratio of 60 wt% on the FM of the DRCs. 150
Figure 4.48: Effect of optimum ratio of mixture filler particles loading on the OD of the DRCs..... 153
Figure 4.49: The X-ray image of the aluminum plate and resin matrix with out and with different ratios of optimum ratio of mixture filler particles. 153
Figure 4.50: Effect of optimum ratio of mixture filler particles loading on the K_{IC} of the DRCs..... 155
Figure 4.51: Effect of optimum ratio of mixture filler particles loading on the FS of the DRCs..... 157
Figure 4.52: Effect of optimum ratio of mixture filler particles loading on the

FM of the DRCs. 159
Figure 4.53: Effect of optimum ratio of mixture filler particles loading on the: (a) VH (Kg/mm^2) and (b) VH improvement (%) of the DRCs. 162
Figure 4.54: Effect of optimum ratio of mixture filler particles loading on the water absorption of the DRCs: (a) absorption (%) and (b) absorption ($\mu g/mm^3$) during 28 days of immersion in water. 165
Figure 4.55: Effect of optimum ratio of mixture filler particles loading on the solubility of the DRCs: (a) weight loss (%) and (b) rate of solubility at 28 days of immersion in water. 168
Figure 4.56: Effect of optimum ratio of mixture filler particles loading on the AS absorption of the DRCs: (a) absorption (%) and (b) absorption ($\mu g/mm^3$) during 28 days of immersion in AS. 171
Figure 4.57: Effect of optimum ratio of mixture filler particles loading on the solubility of the DRCs: (a) weight loss (%) and (b) rate of solubility at 28 days of immersion in AS. 174
Figure 4.58: Morphologies of the 3T3-L1 cell line after: (1) 24 hours, and (2) 48 hours incubated with: (a) HDP, (b) DRC, and (c) pu by the direct contact method. 176
Figure 4.59: Graphical representation of the count of 3T3-L1 viable cells after 24 hours and 48 hours incubated with HDP, DRC and pu by the direct contact method. 177
Figure 4.60: Morphologies of the 3T3-L1 cell line after: (1) 24 hours, and (2) 48 hours incubated with: (a) HDP, (b) DRC, and (c) pu by the indirect contact method. 179
Figure 4.61: Graphical representation of the count of 3T3-L1 viable cells after 24 hours and 48 hours incubated with HDP, DRC and pu by the indirect contact method. 179
Figure 4.62: Morphologies of the 3T3-L1 cell line after: (1) 24 hours, and (2) 48 hours of exposure to the: (a) HDP extract, and diluted extract of the DRC: (a) 25%, (b) 50%, (c) 75% and (d) undiluted 100% extracts, and (f) the 25% phenol. 182
Figure 4.63: Percentage of the 3T3-L1 viable cells after 24 and 48 hours of exposure to the HDP extract, the diluted extract (25% 50% and 75%) and undiluted 100%, extracts of the DRC, and 25% phenol. 183

LIST OF ABBREVIATIONS

ANOVA	:	Analysis of variance
AS	:	Artificial saliva
ASTM	:	American society for testing and material
Bis-GMA	:	Bisphenol A-glycidyl dimethacrylate
CQ	:	Camphorquinone
DMAEMA	:	Dimethylaminoethyl methacrylate
DRC	:	Dental resin composite
DSC	:	Differential scanning calorimetry
e.g.	:	for example
EDX	:	Energy dispersive X-ray
et al.	:	et alia
FESEM	:	Field emission scanning electron microscope
FM	:	Flexural modulus
FS	:	Flexural strength
FTIR	:	Fourier transform infrared spectroscopy
HDP	:	High density polyethylene
in vitro	:	Test done outside of the body
in vivo	:	Test done in or on the living tissue
ISO	:	International Organization for Standardization
LED	:	Light-emitting diode
OD	:	Optical density
PAC	:	Plasma-Arc
pH	:	Potential of hydrogen

PMMA	:	Poly (methyl methacrylate)
pu	:	Polyurethane
QTH	:	Quartz-Tungsten Halogen
rpm	:	Revolutions per minute
SEM	:	Scanning electron microscopy
TEGDMA	:	Triethyleneglycol dimethacrylate
TGA	:	Thermogravimetric analysis
VH	:	Vickers hardness
XRD	:	X-ray diffraction
XRF	:	X-ray fluorescent
γ-MPS	:	3-(trimethoxysilyl)propyl methacrylate

LIST OF SYMBOLS

%	:	Percentage
°C	:	Degree of Celsius
a	:	Notch length
b	:	Sample thickness
cm	:	Centimeter
d	:	Average length of indented diagonals
d_f	:	Density of filler
d_r	:	Density of resin matrix
g	:	Gram
Kg	:	Kilogram
K_{IC}	:	Fracture toughness
kV	:	Kilovolt
L	:	Load
m	:	Tangent gradient of the initial straight line
m_1	:	Conditioned mass, in µg, prior to immersion in water or AS
m_2	:	Mass of the sample, in µg, after immersion in water or AS
m_3	:	Reconditioned mass of the sample, in µg after dry
ml	:	Milliliter
nm	:	Nanometre
OD_b	:	Optical density of the blank
OD_n	:	Optical density of the negative control
OD_s	:	Optical density of the sample
S	:	Span length

v	:	Volume of the sample in mm^3
VH_c	:	Vickers hardness of composite
VH_m	:	Vickers hardness of matrix
w	:	Sample width
W_0	:	Weight of DRC sample
W_1	:	Weight of DRC ash
W_d	:	Dry weight of sample
W_m	:	Saturated weight of the sample
W_s	:	Suspended weight of the sample
Y	:	Geometrical correction factor
μg	:	Microgram
μm	:	Micrometer

PEMBUATAN DAN PENILAIAN KOMPOSIT RESIN PERGIGIAN DIBANGUNKAN DARIPADA BIS-GMA/TEGDMA DENGAN PENGISI SERAMIK TERAWAT

ABSTRAK

Dalam penyelidikan ini, partikel-partikel silica (SiO_2), alumina (Al_2O_3) dan zirconia (ZrO_2) telah dirawat dengan 3-(Trimethoksisilil) propil metakrilat (γ-MPS) γ-MPS dan kemudiannya disebatikan ke dalam Bis-fenol A-glisidil dimetakrilat (Bis-GMA)/trietilena glikol dimetakrilat (TEGMA) untuk menghasilkan komposit resin pergigian (DRC). Penyelidikan ini dilakukan dalam tiga peringkat. Peringkat pertama ialah penyediaan DRC berdasarkan tiga jenis pengisi, peringkat kedua ialah penyediaan DRC berdasarkan campuran tiga pengisi berlainan jenis dan peringkat ketiga ialah penyediaan DRC daripada nisbah optimum campuran ketiga-tiga jenis pengisi. DRC telah dipolimerkan dengan unit rawatan cahaya. Sampel-sampel telah diuji menurut piawaian ISO 4049:2009 untuk sifat-sifat fizikal, mekanikal dan persekitaran. Sampel-sampel juga diuji sifat termanya. Komposisi DRC optimum telah dilakukan penilaian ketoksikan menurut piawaian ISO 10993-5:2009. Keputusan menunjukkan bahawa ketumpatan DRC meningkat dengan meningkatnya muatan pengisi dan nilai ketumpatan yang tertinggi masih dianggap sesuai untuk kegunaan pergigian. Peningkatan muatan pengisi menyebabkan penurunan nilai-nilai K_{IC} dan FS. Walau bagaimanapun, nilai-nilai K_{IC} dan FS tersebut masih berada dalam spesifikasi baik pulih pergigian. Sebaliknya, peningkatan muatan pengisi menyebabkan peningkatan ketara radiopasiti, FM dan VH pada DRC berkenaan. Tambahan pula, kestabilan terma, rintangan terhadap penyerapan air, penyerapan airliur tiruan dan kelarutan telah bertambah baik dengan meningkatnya muatan pengisi. Penggunaan kandungan campuran jenis bahan pengisi juga memberikan kesan kepada sifat-sifat DRC. Radiopasiti telah meningkat dengan ketara apabila

kandungan ZrO_2 meningkat. Sementara itu, nilai K_{IC} dan FS berkurangan dengan peningkatan kandungan SiO_2. Sebaliknya, tiada perbezaan yang ketara dalam nilai FM. Dengan meningkatnya muatan campuran pengisi (nisbah optimum 70 SiO_2:10 Al_2O_3:20 ZrO_2), ketumpatan optikal telah berkurangan dari 1.84 ke 0.36, walau bagaimanapun FM dan VH telah meningkat masing-masing dari 4.94 GPa ke 12.51 GPa dan dari 14.4 ke 31.0. Tambahan pula, penyerapan dan kelarutan di dalam air telah bertambah baik masing-masing ke 18.56 $\mu g/mm^3$ dan 3.05 $\mu g/mm^3$. Kelakuan yang sama berlaku apabila didedahkan ke dalam air liur tiruan yang masing-masing bertambah baik ke 12.67 $\mu g/mm^3$ dan 2.71 $\mu g/mm^3$. Sebaliknya, nilai K_{IC} dan FS telah berkurangan masing-masing ke 1.64 $MPa.m^{1/2}$ dan 54.97 MPa. Akhirnya, komposisi optimum DRC tidak menunjukkan kesan ketoksikan ke atas sel 3T3-L1. Ringkasnya, sifat-sifat DRC yang dihasilkan boleh dilaraskan melalui gabungan yang sesuai ketiga-tiga pengisi seramik berkenaan di mana SiO_2 khusus untuk meningkatkan FM, Al_2O_3 meningkatkan VH dan ZrO_2 meningkatkan radiopasity. Oleh itu, gabungan ketiga-tiga pengisi seramik ini berjaya memberikan sinergi terbaik untuk DRC yang lebih baik.

FABRICATION AND EVALUATION OF DENTAL RESIN COMPOSITES DEVELOPED FROM BIS-GMA/TEGDMA WITH TREATED CERAMIC FILLERS

ABSTRACT

In this research, silica (SiO_2), alumina (Al_2O_3) and zirconia (ZrO_2) particles were treated with 3-(Trimethoxysilyl) propyl methacrylate (γ-MPS) and then incorporated into Bis-phenol A-glycidyl dimethacrylate (Bis-GMA)/triethylene glycol dimethacrylate (TEGDMA) to produce dental resin composite (DRC). This research was conducted in three different stages. The first stage was preparation of the DRCs based on the three different types of fillers, the second stage was prepared DRCs based on the mixture of the three different types of filler, and the third stage was prepared DRCs from the optimum ratio of the mixture of the three types of filler. The DRCs were polymerised with light curing unit. The DRCs samples were tested according to ISO 4049:2009 standards, for physical, mechanical, and environmental properties. Samples were also tested for their thermal properties. The cytotoxic assessment was carried out according to ISO 10993-5:2009 standards. The results showed that the DRC density increased as filler loading increase. The fracture toughness (K_{IC}) and flexural strength (FS) were decreased as filler loading increased. On the other hand, the increase of filler loading resulted in the significantly increase of the radiopacity, flexural modulus (FM) and Vickers hardness (VH) of the DRC. In addition, the thermal stability, water and artificial saliva absorption and solubility were improved as filler loading increase. Moreover, the use of mixture filler as fillers type content has an effect on the DRC properties. The radiopacity was significantly increased as ZrO_2 increased while the K_{IC} and the FS decreased as SiO_2 increased.

On other hand, there were no significant differences in the FM values. As the mixture of fillers (optimum ratio 70 SiO_2:10 Al_2O_3:20 ZrO_2) loading increase, the optical density was significantly decreased from 1.84 to 0.36, however FM and VH were increased from 4.94 to 12.51 and from 14.4 to 31.0, respectively. In addition, absorption and solubility in water were significantly improved to 18.56 $\mu g/mm^3$ and 3.05 $\mu g/mm^3$, respectively. Similar behavior when exposed to artificial saliva which improved to 12.67 $\mu g/mm^3$ and 2.71 $\mu g/mm^3$, respectively. In contrast, the K_{IC} and FS were decreased to 1.64 $MPa.m^{1/2}$ and 54.97 MPa, respectively. Finally the cytotoxic assessment shows negligible effect on the 3T3-L1 cell line. In short, properties of the fabricated DRCs can be adjusted by the suitable combination of those three ceramic fillers used whereby specifically SiO_2 improved the FM, Al_2O_3 improved VH and ZrO_2 improved the radiopacity. Therefore, the combination of these three ceramic fillers managed to provide the best synergy for better quality of DRCs.

CHAPTER 1
INTRODUCTION

1.1 Research background

Over the past decade the provision of dental resin composites (DRCs) as an application in dentistry as a restorative material has developed at a remarkable rate. There is a widespread use of DRCs in dentistry particularly for anterior teeth, mainly for aesthetical reasons. Furthermore, DRCs can be used to repair decayed or damaged teeth as a consequence of dental caries. Characteristically, DRCs comprise a resin matrix mixed with silane-treated filler particles (Karabela & Sideridou, 2008; Miao et al., 2012; Hambire & Tripathi, 2013).

Specifically, the resin matrix usually comprises Bis-phenol A-glycidyl dimethacrylate (Bis-GMA) and triethylene glycol dimethacrylate (TEGDMA). The Bis-GMA is very high viscous at the room temperature. However, since early 1960s there has been widespread use of Bis-GMA as a base monomer whilst the TEGDMA as a low-viscosity monomer has been utilised as a diluent to achieve high filler loading of the DRCs and it is widely added in amounts ranging from 20-50 wt% (Barszczewska-Rybarek, 2009). Organosilanes, mainly 3-(Trimethoxysilyl) propyl methacrylate (γ-MPS) are used as coupling agent to provide the essential covalent connection between the filler and the polymer (Sideridou & Karabela, 2009). The bond and compatibility between the resin matrix and the fillers are vital to the dental composite's mechanical properties (Miao et al., 2012). According to Tham et al. (2010a) the improvement of the mechanical strength related to the enhancement of interfacial interaction between the polymer and the filler by the surface treatment of γ-MPS.

The loading of the inorganic filler plays a crucial role in determining the DRC's mechanical properties. Conventionally, the mass faction of inorganic fillers in commercial DRCs is as high as 75 % (Tian et al., 2007). In fact, fillers have varied from relatively large particles in the early stages of development, to micro fillers, and more recently to hybrid fillers (Hambire & Tripathi, 2013). The DRC's mechanical properties depend largely on the size of the particle, the particle's loading and the particle-matrix interface adhesion. Accordingly, the size of the particle has a noticeable impact upon the mechanical properties of the DRCs (Fu et al., 2008). Furthermore, DRCs with an increased amount of filler loading can assist to enhance some of its mechanical properties (Kim et al., 2002) and an enhanced thermal stability and glass transition temperature (Ghosh et al., 2012).

Efforts for continuous improvement are ongoing. Despite the achievements to date, further improvements to the DRC's performance are sought with respect to their aesthetic and mechanical properties. In order to further improve the DRC's properties, prior studies have focused on resin monomers (Chung et al., 2002; Atai et al., 2004) as well as the treatment of fillers (Karabela & Sideridou, 2008; Tham et al., 2010a). Furthermore, numerous studies have been conducted to evaluate the type of filler on the mechanical properties of the polymer composites; such as hydroxyapatite (Shyang et al., 2008) and zirconia (Tashkandi, 2009).

Restorations in the oral environment are subjected to mastication action stresses. These forces act upon the teeth and or on the DRCs producing different reactions that over time lead to deformation, which essentially compromises their durability. Accordingly, characterisation of the mechanical, environmental and biological properties and any other related DRC properties ought to be undertaken to establish their reliability and the applicability of DRCs.

1.2 Problem statement

In 1980, the number of teeth at risk for dental disease worldwide, for people over the age of 65 was 2.8 billion. This figure is expected to increase to the level of 5 billion teeth at risk by the year 2020 (Narayan, 2009). Due to this disease, in 2005 alone, the volume of applied DRCs (hybrid and microfiller composites) amounted to approximately 550 million Euros (Moszner & Salz, 2007). Furthermore, in smaller premise, Rahim (2011) reported that the number of tooth-restorations at Universiti Sains Malaysia dental clinic in 2009 was 9260 cavities and 930 tubes of composites were used. This is a considerably high number, which is associated with a high consumption expenditure from the government per year, given the price for a tube of a 4 gram DRCs ranges from RM 100 to RM 200 depending on the type of DRC and the brand. There is no Malaysian product of DRCs which can be commercialised for the local market. Malaysian dental clinics are utilising DRCs produced fully from others countries such as Germany, China and the United States of America.

The lifetime of an amalgam composite is higher than a resin composite. According to Sideridou et al. (2002) the life span for a restorative DRC is often no more than two to four years for posterior teeth and eight years for anterior teeth. Jablonski (1992) claimed that a permanent restoration is “a restoration designed to remain in service for not less than 20 to 30 years”. Furthermore, DRC’s have low fracture toughness which become one of the limiting factor to be used as the posterior restorations materials (Wilson et al., 1997).

Therefore, it is still seen as controversial to apply DRCs for large restorations and in that regard the fracture of posterior restorations has been found to be a common cause for the failure of restorations. In an aqueous environment DRCs absorb water and in turn release un-reacted monomers. The release of these uncured

monomers from the DRCs may well be conducive to the growth of bacteria in the region of the restoration (Hansel et al., 1998). Furthermore, the water uptake into the DRCs in the oral cavity can over time, in some patients, promote allergic reactions and can result in the degradation of the DRC's mechanical properties (Wei et al., 2011).

The problems become a main issue and being investigated by researchers. For example there are researchers that examining the use of silica particles in DRCs such as colloidal silica (Lim et al., 2002), silica with hydroxyapatite (Yücel et al., 2012), silica with zirconia (Taira et al., 1995), as well as silica glass with alumina filler particles (Nagarajan et al., 2000). Additionally, various studies have explored alumina particles as DRC fillers (Wang et al., 2007; Ellakwa et al., 2008; Thorat et al., 2013) and several previous studies have discussed zirconia particles as DRC fillers (Amirouche-Korichi et al., 2009; Chan et al., 2009). Therefore, it is obvious that the DRCs properties can be further improved by using various types of fillers or their mixtures since each filler will contribute different properties. For that reason, appropriate ratio of these filler will possibly produce the optimum properties of DRC. Therefore, their properties assessment is based on International Organization for Standardization (ISO) 4049:2009 standards such as flexural strength (FS), flexural modulus (FM), radioapacity, water absorption and solubility, and artificial saliva (AS) absorption and solubility. Fracture toughness based on ISO 13586:2000, and Vickers hardness (VH) based on American Society for Testing and Material (ASTM) 384:2008. In addition, thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC), and cytotoxicity evaluation are necessary to be qualified as a superior quality of DRCs.

According to ISO 4049:2009 standards the DRC's properties, such as the FS ought to be not less than 50 MPa, radioapacity ought to have minimum radiopacity value of the same thickness of pure aluminium, and water absorption and solubility ought to be less than 7.5 $\mu g/mm^3$ and less than 40 $\mu g/mm^3$ respectively. Based on the DRCs available on the market the FM ought to be not less than 2 GPa. In addition based on previous researches were done by Fujishima & Ferracane (1996) and Zhao et al. (1997) the K_{IC} ought to be not less than 0.7 $MPa.m^{1/2}$. Moreover, based on previous research done by Beun et al. (2007), the DRCs ought to be not less than 20 VH. In relation to in vitro cytotoxicity testing, according to ISO 10993-5:2009 standards DRCs ought to be non-cytotoxicity.

1.3 Research objectives

The objectives of this research are:

(a) To investigate the effect of γ-MPS (10 wt%) as treatment compound on the filler surface of the SiO_2, Al_2O_3 and ZrO_2 particles.

(b) To investigate the effect of individual treated SiO_2, Al_2O_3 and ZrO_2 particles as filler loading on the physical, mechanical, thermal and environmental properties of the DRCs.

(c) To investigate the effect of various mixtures of treated SiO_2, Al_2O_3 and ZrO_2 as filler type content on the physical and mechanical properties of the DRC.

(d) To investigate the effect of optimum ratio of mixture filler as filler loading on the physical, mechanical and environmental properties of the DRC.

(e) To investigate the effect of optimum DRC composition on the biological property of the 3T3-L1 cell line.

1.4 Outline of the thesis

The thesis comprises five chapters and is briefly described below.

Chapter one has provided a brief background of DRCs and outlined the problem statement, it defined the research objectives and provided the outline of the entire thesis.

Chapter two provides a review of the literature, to present an overall background for the thesis. This chapter outlines the relevant research related to this work and presents the common concepts.

Chapter three presents the experimental procedures utilised in this research endeavour. The chapter also offers a description of the laboratory equipment in use for this study.

Chapter four presents the results and provides a discussion in relation to the current research.

Chapter five presents the conclusion of this research and provide recommendations for future research.

CHAPTER 2
LITERATURE REVIEW

2.1 Introduction

This chapter reviews the topics of significance which are related to this thesis. Accordingly, there is a review in this chapter of the physiology and anatomy of tooth structures. In addition, the chapter presents a literature review regarding the development and history of dental restorations and provides a classification for dental resin composites and dental biomaterials, mechanical, environmental and biological properties of dental materials.

2.2 Anatomy and physiology of tooth structures

In relation to the importance of human teeth as a masticatory organ, teeth are closely linked to the facial aesthetics of human beings and to vital functions such as speech (Zhou & Zheng, 2008). Accordingly, teeth are not only associated with speech, but to breathing, chewing, tasting, and soft tissues and bone support and the muscles of mastication. Importantly, restorations and implants have a role in supporting the vital functions mentioned above (Lewis & Dwyer-Joyce, 2005). An individual tooth consists of an exposed crown and a root, buried in the gum and jaw.

The crown of the tooth is usually partly covered by an outer layer called enamel, which is the hardest and stiffest tissue of a tooth (Lopes et al., 2007). The principal function of enamel is to cover and, therefore, protect the softer underlying dentine. The structure of dental enamel is a composite of mineral, protein, lipid and water by volume. Furthermore, the enamel is the most mineralised tissue in the human body as it consist approximately 97 wt% mineral and 3 wt% water and organic material (Simmer & Hu, 2001; Lopes et al., 2002; Klocke et al., 2007;

Berkovitz et al., 2009;). The fracture toughness of enamel ranges from 0.4 $MPa.m^{1/2}$ for the cracks propagating in the direction parallel to the enamel rods to 1.5 $MPa.m^{1/2}$ in the direction perpendicular to the enamel rod (Hassan et al., 1981; Xu et al., 1998a; White et al., 2001; Park et al., 2008).

A material called dentine is found in an intermediate layer beneath the layer of enamel (Wang, 2007). Dentin is a mineralized, yellow-white, a vascular, elastic, sensitive, specialized connective tissue that forms the bulk of the tooth. It supports and underlies enamel, and surrounds the pulp (Geraldeli, 2012). It is comprised approximately of 20 wt% organic material, 70 wt% inorganic material, and 10 wt% water (Lewis & Dwyer-Joyce 2005; Besinis et al., 2012).

The pulp of a tooth comprises loose connective tissue which is derived from ectomesenchymal cells or neural crest cells. The dental pulp is confined within root canals and the pulp chamber of the tooth. It contains cells that provid the mature pulp with odontogenic, nutritive, sensory, and defensive functions and allow for preservation of vitality during normal homeostatic maintenance and during wound repair after injury (Avery et al., 2002).

The cementum is the calcified surface tissue covering the dentine of the root of the tooth. It has a similar structure to bone, but it has no blood supply, no nerves, does not remodel and is more resistant to resorption than bone (Noble, 2012). cementum contains on a wet-weight basis of 65% inorganic material, 23% organic material and 12% water. By volume, the inorganic material comprises approximately 45%, organic material 33%, and water 22% (Aurangabadkar, 2007). In comparison to the dentine, in general, cementum is more permeable. Any loss of cementum in these cases will result in exposed dentine (Aurangabadkar, 2007). The structure of a human tooth is presented in Figure 2.1.

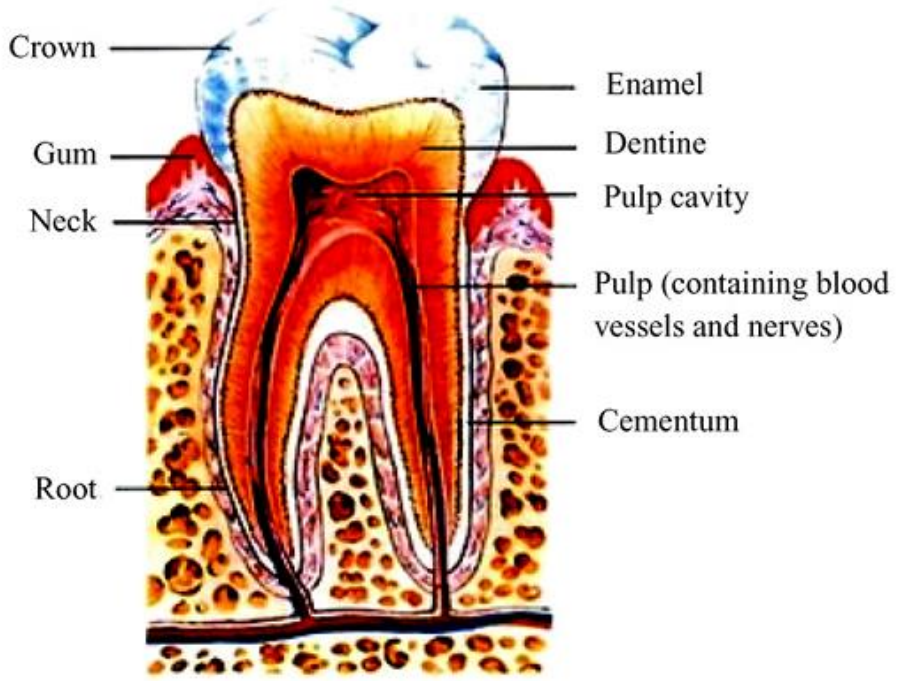

Figure 2.1: Human tooth structure.

2.3 Saliva

Saliva, the liquid inside the oral cavity, is comprised mainly of water (99.5%), inorganic and trace substances (0.2%) and proteins (0.3%) are also found in saliva (Zahir & Sarkar, 2006). A variety of electrolytes are found in saliva, including, potassium, sodium, magnesium, calcium, phosphates and bicarbonate. Nitrogenous products, such as ammonia and urea are also found in saliva as enzymes, proteins, immunoglobulins and mucins (Humphrey & Williamson, 2001). Six major salivary glands, produce saliva along with 200 to 400 minor salivary glands which are present in the mucosa of the palate, cheeks, lips and tongue (Thylstrup & Fejerskov, 1994; Silvers & Som, 1998).

Normally, saliva has a pH of 6 to 7, in other words it is slightly acidic. On average, the daily flow of whole saliva varies between 1 and 1.5 litres per day. The

function of saliva can be organised into many major categories which serve to create an appropriate level of ecologic balance and to maintain oral health. These categories have been identified as maintenance of tooth integrity, buffering action and clearance, lubrication and protection, taste and digestion and antibacterial activity (Mandel, 1987; Moss, 1995).

2.4 Dental caries

Dental caries, also known as tooth decay, is the localised destruction of the dental hard tissues which have become susceptible to acidic by-products through the dietary carbohydrates resulting from bacterial fermentation (Fejerskov & Kidd, 2003; Krol, 2003). Dental caries is a result of interactions of acid-producing bacteria over time, with a substrate to metabolise involving host factors including teeth and saliva (Krol, 2003; Selwitz et al., 2007). Dental caries is one of the most common diseases affecting humans. Although dental caries rarely poses a threat to life, it does account for a loss of masticatory function and substantial pain. Dental caries continues to pose a major public health problem, in spite of an increased level of understanding of the pathogenesis of caries, as the knowledge is incomplete (Martin, 2002).

The presence of dental plaque is an essential process leading to the dissolution of enamel as the dental plaque controls the diffusion of acids away from the tooth enamel surface and it limits the buffering action from saliva. It is the collapse of the inorganic matrix which results from the loss of tissue and over time a cavity is formed (Martin, 2002).

2.5 Classification of dental caries

Dental caries may be classified according to Chandra et al. (2007), as the following subsections:

2.5.1 The surface topography and environmental condition

There are four distinct clinical sites for caries initiation, accordingly they are known as follows: pits and fissures caries, smooth enamel surface caries, root surface caries, and cervical caries.

2.5.2 The rapidity of the caries progress

The rapidity of the progress of caries can be classified as follows:

2.5.2.1 Acute dental caries

Acute caries travels towards the pulp at a very fast speed. It occurs in young adults because the dentinal tubules are large and so, there is little time for secondary dentin formation. Often they occur in so-called retention points: fissures, holes and other chewing surfaces of teeth: most often in molars and premolars fissures, on the contact surface of the teeth in the neck area of the vestibular surfaces of teeth.

2.5.2.2 Rampant caries

The disease of rampant caries is pronounced by a suddenly appearing type of caries which is rapidly burrowing and results in early involvement of the pulp, for which more than 10 new lesions can emerge every year on what are ordinarily healthy teeth surfaces that are generally immune to caries. Rampant caries can be categorised into the following three types:

2.5.2.2(a) Nursing bottle rampant caries

Infants who fall asleep with a nursing bottle nipple in their mouth containing milk and sugar or a beverage containing sugar are prone to this disease. Accordingly, rampant caries result from the slowing of rate of clearance of the fluids from the oral cavity. This situation results in a number of carious lesions on what would ordinarily be described as an immune surface. As explained above, this form of rampant caries is very common in infants (Figure 2.2).

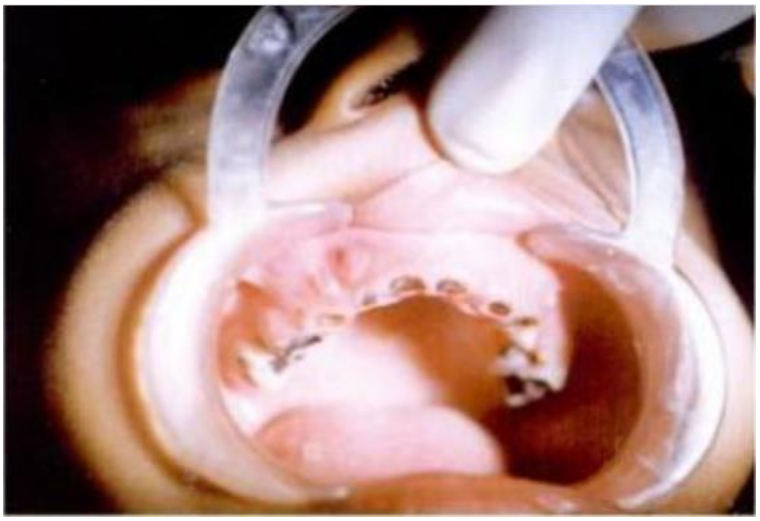

Figure 2.2: Nursing bottle rampant caries.

2.5.2.2(b) Adolescent rampant caries

This type of caries occurs at the age of adolescence. During this time in a young person's development, a high sugar diet including the consumption of confectionery at bedtime provides the ideal conditions for this type of caries to occur particularly when the adolescent falls asleep with these sugary substances in their mouth.

2.5.2.2 (c) Xerostomia induced rampant caries

Xerostomia means dry mouth. It has been commonly observed that after radiotherapy of malignant areas, or near the salivary glands, the salivary flow is very much reduced.

2.5.2.3 Chronic dental caries

Chronic caries travels very slowly towards the pulp. Usually the lesions of chronic dental caries are very large cavities, and wide open. The cavities appear after tooth eruption and located near the pulp therefore, less food is retained and the acids produced by the microorganisms are neutralized by saliva. It commonly occurs in adults.

2.5.3 Primary caries, secondary or recurrent caries

Whether caries attacks have been noted previously at the margin of restoration, or on an intact surface it is classified as either: primary caries; secondary; or recurrent caries. Furthermore, the proximity of the position of the caries to the pulp is also noted.

In addition, Chandra et al. (2007) reported that G. V. Black at the end of nineteenth century presented a form of classification to identify a simple cavity as listed as follows:

Class I Cavities

Pit and fissure caries lead to cavities occurring in the occlusal surface of molars and premolars, and lingual surface of molars, the lingual surface of incisors

and any other abnormal position as well as the occlusal two-third of the buccal surface. These are referred to as class I cavities (see Figure 2.3 a & b).

Class II Cavities

These cavities are the result of caries in the proximal surface of molars and premolars (see Figure 2.3 c & d).

Class III Cavities

These cavities have resulted from caries in the proximal surface of the anterior teeth (canine and incisors) and it does not involve the incisal angle (see Figure 2.3 e).

Class IV Cavities

Class IV cavities have resulted from caries in the proximal surface of anterior teeth also involving the incisal angle (see Figure 2.3 f).

Class V Cavities

These gingival cavities have resulted from the impact of caries on the gingival third, on the lingual and facial or palatal surfaces of all teeth (therefore these are not cavities in relation to pit and fissure) (see Figure 2.3 g).

A modification was made to Black's classification to provide more specific localisation of cavities as indicated below.

Class II Cavities

The classification was updated to include cavities involving proximal surfaces in that the single or both proximal surfaces of premolar and molar teeth involved a proximal surface; it is then referred to as a mesio-occluso-distal cavity.

Class VI Cavities

Cavities on the cusp tips of posterior teeth and the incisal edges of anterior, without involvement any other surface (see Figure 2.3 h).

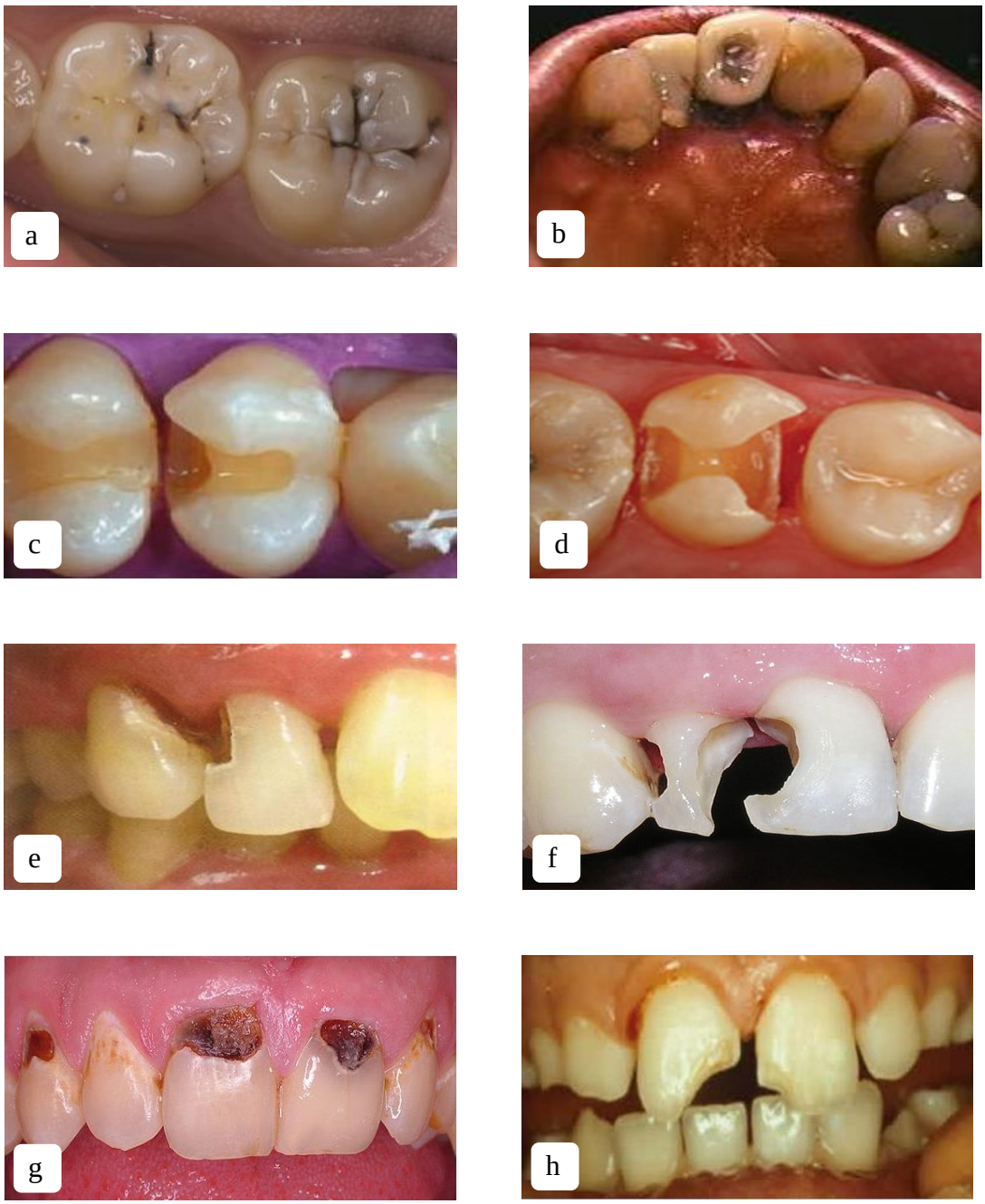

Figure 2.3: Classification of dental caries: (a & b) class I, (c & d) class II, (e) class III, (f) class IV, (g) class V and (h) class VI.

2.6 History and development of the dental resin restorations

Acrylic resins which were self-curing were developed in the 1940's in Germany. This innovation led to the introduction of acrylic filling materials in 1948. The monomer Bis-GMA was developed by Bowen in 1956 by the introduction of attaching the epoxy monomer to methylmethacrylate groups. To acknowledge Bowen's innovation in dentistry, resins which contained Bis-GMA became known as "Bowen's resins" Furthermore, in 1962 Bis-GMA resin-based composites were introduced (Matos et al., 1997). The production of an innovative methacrylate particle-filled resin matrix, which was tooth-coloured, has formed the cornerstone of what is known as modern restorative dentistry with aesthetic and functional restorations (Bowen, 1964).

However, the first of these resin-based composite materials did not yield any significant improvement in relation to its mechanical properties as a result of a lack of reinforcement between the resin matrix and the inorganic filler particles. Later, in relation to filler particles, the use of silane treatments is recommended to enhance the bonding of the filler particles with the resin matrix. DRCs were adopted for use for the anterior teeth as the DRCs were vulnerable and unable to tolerate the pressure imposed by the posterior teeth. Accordingly, there was a need to improve DRCs and great improvements have been made (Alsharif et al., 2010). During the late-twentieth and early twenty-first centuries an ensuing development and enhancement of DRC materials for aesthetic restorative dentistry has led to a wide ranging diversity of materials. These developments included DRCs intended for universal use for both posterior and anterior placement (Cobb et al., 2000), highly viscous 'packable' DRCs (Manhart et al., 2001) and also 'flowable' DRCs (Braga et al., 2005). By the 1970s, further advancements had been made with the development of polycarboxylate and

glass ionomer cements. Additional advances were made in dentine adhesives, composites and ceramics by the 1980s. Furthermore, during this period sealants for the surfaces of restorations, light-cured liners were introduced, and advances in orthodontics including alterations to orthodontic wires were made (Ferracane, 2001).

2.7 Classification of dental restorations

Dental restorations can be placed into two broad types; direct and indirect restorations. The term “direct” restoration relates to the placement in situ of the DRCs and the term “indirect” restoration relates to the machined- or laboratory-made ceramics.

2.7.1 Indirect dental restorations

The indirect dental restorations are fabricated in a laboratory. There are four types of materials that are used for indirect restorations. These are metals-ceramics, porcelain, gold and indirect composite restorations. The common types of indirect restorations are represented by crowns, onlays and inlays. A crown covers the entire chewing surface of a tooth, an inlay lies within the cusps of the tooth, and an onlay covers and one or more cusp tip, including part of the chewing surface.

The indirect dental restorations can be made from a variety of materials. Gold is still used for some inlays, but for someone concerned about white teeth, porcelain and tooth-colored composite resins are other options.

2.7.2 Direct dental restorations

Currently, two types of materials are in use for direct dental restorations. The first non-aesthetic dental restorations (amalgam) and the second are aesthetic dental restorations (compomers, glass ionomers, hybrid ionomers, and DRCs) (Powers & Wataha, 2008).

2.7.2.1 Amalgam

An amalgam is an alloy containing mercury. In dentistry, amalgam refers to a specific result of mixing a silver-based alloy, in the form of small particles, with mercury into a paste which then sets hard (Darvell, 2009). After mixing, the reaction of the alloy and mercury is called an amalgamation reaction. This reaction produces a restorative material which is hard and silver-grey in colour (McCabe & Walls, 2009). These restorations, due to their grey colour, are used for posterior teeth where there is no requirement to address clinical aesthetics (see Figure 2.4).

Amalgams have been used in dentistry for over 200 years. Currently, the content of dental amalgam alloys comprises a higher copper amount (10 to 30 wt%) in comparison to the old ones. Furthermore, in general these modern dental amalgam alloys comprise 12 to 30 wt% tin and the silver content is 40 to 70 wt% and they are mixed with 43 to 50 wt% mercury (Hatrick et al., 2003). In 1845, as a consequence of problems attributed to a lack of regulation, and poor techniques and toxicity concerns regarding mercury, the American Dental Association prohibited the use of amalgams (Eley, 1997).

Today, the governments of Norway, Sweden, and Denmark have already banned the use of mercury fillings in dentistry. Germany and Canada have limited

their use for pregnant women. France, Finland, and Austria have recommended that alternative dental materials be used for pregnant women.

The demand from patients for restorations which are aesthetic and mimic the look of natural teeth has dramatically increased (Burke 2004; Roeters et al., 2004; Opdam et al., 2007). As a result, the use of amalgam worldwide is decreasing annually, albeit at different rates (Roeters et al. 2004; Opdam et al. 2007).

Figure 2.4: Amalgam restoration.

2.7.2.2 Compomers

For restorations for use in low-bearing areas, compomers are often chosen. These are composites which have been modified with polyacid groups. Compomers are suitable for those patients who carry a medium caries risk. They contain monomers, formulated without water, which have been modified by polyacid groups with silicate glasses which are fluoride releasing. Furthermore, some comprise modified monomers which provide supplementary fluoride release. They release fluoride by mechanisms similar to that of glass and hybrid ionomers. As a consequence of the lower levels present of glass ionomer in compomers, the amount of fluoride release and its subsequent duration is at a lower level in comparison to hybrid and glass ionomers (Powers & Wataha, 2008).

2.7.2.3 Glass ionomers

Glass ionomers cements were invented in an English laboratory in the early 1970s (Wilson & McLean, 1988). In various areas of dental science they have a proven ability to be effective due to their continual fluoride production and marginal seal which is long lasting and eliminates secondary caries. They are also effective in preventing decay on adjacent tooth material. In addition, they used as bone grafting materials, due to their biocompatibility, and it has been evidenced that glass ionomers improve bone growth (Ribeiro et al., 2006). The advantages of glass ionomer restorations is that they have properties of low solubility, a FM which is not dissimilar to dentine, have a fairly high level of opacity, and an expansion coefficient which is similar to that of the tooth structure. Although the bond strength of composites to dentine is higher than that of glass ionomers, clinical studies have indicated that in areas of cervical erosion the retention of glass ionomers is considerably better than composite retention (Powers & Wataha, 2008).

2.7.2.4 Hybrid ionomers

Patients with a high risk of caries are recommended to select hybrid ionomers. These are preferred aesthetically to glass ionomers. They have an advantage of being able to bond to tooth structure without the requirement of an agent of a dentine bond. Prior to the placement of a hybrid ionomer, it is necessary to prepare the tooth by using polyacrylic acid which etches the tooth surface. An advantage of hybrid ionomers is that they set instantly when light-cured and can be finished directly. They leach less fluoride in comparison to glass ionomers, however hybrid ionomers leach more fluoride than compomers and composites do (Powers & Wataha, 2008).

2.7.2.5 Dental resin composites

It is common to use DRCs to restore anterior teeth. They are also used to restore posterior teeth (Ramakrishna et al., 2004). An example of DRC restoration is shown in Figure 2.5: DRCs are complex, and consist of inorganic fillers as a dispersed phase and a polymer matrix as an organic phase, with a coupling agent, and minor additives which includes initiators, accelerators, stabilizers as well as colouring pigments (Sakaguchi & Powers, 2013). The definition of a DRC is a compound of two or more materials which are specifically different which comprise properties that are said to be in-between those of the individual constituents or components (Bhat & Nandish, 2007).

The properties of DRCs, according to (García et al., 2006; Powers & Sakaguchi, 2006), are dependent upon a number of factors including the amount of resin matrix, filler particles type and size, and the adhesion between the filler particles and the resin matrix (Ferracane, 2001).

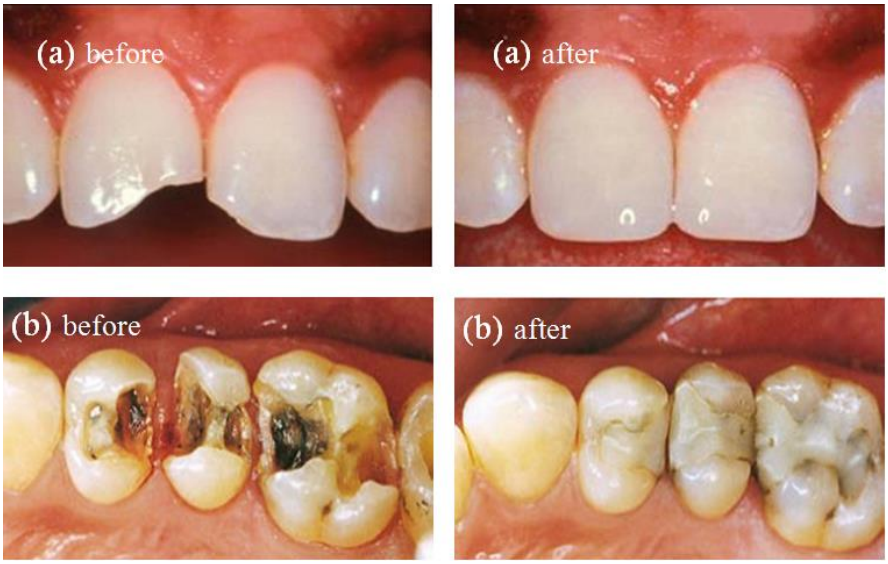

Figure 2.5: Dental resin composites restoration: (a) anterior and (b) posterior teeth

2.7.2.5.(a) Resin matrix

Most DRCs use monomers that are aromatic or aliphatic diacrylates. The common dimethacrylates utilised for DRCs are Bis-GMA and TEGDMA (Anusavice et al., 2013). Bis-GMA monomer, also known as Bowen's monomer, which is the name attributed to the inventor of the monomer (Noort, 2013). The application of Bis-GMA remains the choice of the main monomer for the majority of DRC systems for a period of over 50 years. This is due to its higher molecular weight in comparison to methyl methacrylate. Bis-GMA monomer can have a polymerisation shrinkage value of 7.5% by volume in comparison to methyl methacrylate monomer which have a polymerisation shrinkage value of 22% by volume (Noort, 2013).

Bis-GMA possesses many advantages, such as high molecular weight, lower polymerization shrinkage, more rapid hardening and production of stronger and stiffer polymer matrix (Du & Zheng, 2008). However, its desirable properties are partially negated by a relatively high viscosity and low mobility that could affect to the degree of conversion (Du & Zheng, 2008; Filho et al., 2008). Accordingly, to thin down the viscosity of the polymer and to increase the degree of conversion and for incorporation of the filler, the composite is supplemented with the addition of low viscosity diluent monomer (Kim & Shim, 2001).

2.7.2.5.(b)Filler

The DRCs have a number of important properties which are enhanced with the addition of the filler particles to resin matrix such: reinforcement of the resin matrix resulting in increased hardness, compressive strength, tensile strength, stiffness, abrasive resistance, and improves its modulus of elasticity if the filler particles is chemically bonded to the resin matrix. The reinforcement of the resin

matrix also limits polymerisation shrinkage, and reduces the thermal expansion coefficient. It also allows for an improvement in handling, and other factors such as staining and softening, reducing the absorption of water and increasing the diagnostic sensitivity and radiopacity (Bhat & Nandish, 2007). The reinforcement particular of composite can be enhanced in the matrix's properties, and it is dependent upon the filler particle size, content and the interaction of one filler particle with another, and also the high-quality adhesion at the interface surface and the filler particles' distribution within the resin matrix (Maiti & Lopez, 1992). The two important factors ought to be considered when developing DRCs properties which are the interface adhesion between filler particles and resin matrix (Fu et al., 2008).

To provide improved levels of stiffness and strength a polymer composite is strengthened with fillers particles and other matrix reinforcement. The properties of the DRC are dependent upon the original properties of the filler particle and the resin matrix, the interface between the filler particles and the resin matrix and the concentration and the dispersion of the filler particles in the resin matrix, as well as the geometrical quality of the filler particles, and in particular its shape and size (Ranganathan et al., 2010).

Silica is the common name for materials composed of silicon dioxide and occurs in crystalline and amorphous forms (Napierska et al., 2010). Quartz is the most commonly found form of silica in nature and comprises 12% of the crust of the earth. Furthermore, there are at least eight different polymorphas that silica can crystallise into: α-quartz, moganite, coesite, tridymite, cristobalite, β-quartz, stishovite and keatite. By far the most abundant is α-quartz is as it is the most stable thermodynamically under ambient conditions (Guthrie & Heaney, 1995).

As mentioned previously, silica can be crystalline or amorphous, and it can also be hydroxylated or anhydrous, non-porous (dense) or porous (Ralph, 1979). It is mainly synthesized from an aqueous solution, with dissociated monomeric silicic acid or from a vapour of a silicon compound such as silicon tetrachloride. Until recently, toxicological research into silica particles focused mainly on "natural" crystalline silica particles of 0.5 to 10 μm (coarse or fine particles) (Napierska et al., 2010). Silica can also be found naturally in an amorphous non-crystalline form, including biogenic silica, volcanic glass, diatomaceous earth and opal. Importantly, silica in its amorphous forms is not known to produce any carcinogenic effects (Shy, 2000).

Aluminium oxide (Al_2O_3) or alumina is an inert ceramic has been in use as components for prostheses since 1970, and is often used in the medical field for surgical implants (Lee, 2008). Alumina ceramics have been clinically tested for prostheses for areas in the human body such as fingers, wrists, shoulders and elbows with success equalling or bettering other types of material systems (Heimke, 1990; Hulbert, 1993).

Alumina is suited to biomaterial applications due to a number of factors including the following properties: excellent wear resistance; a high degree of chemical inertness particularly under physiological conditions; its excellent hardness and its ability to attain a surface finish which is at a level high of polish. Furthermore, alumina's properties can be utilised for the purposes of implants, where it can be used for surgeries for hip and knee joint replacements as an articulating surface (Heimke, 1990; Hench, 2013).