



Biodegradability and Some Mechanical Properties of Different Major Connector Materials

THESIS

*Submitted to the Faculty of Dentistry, Ain Shams
University in partial fulfillment of the requirements
for the Degree of Doctor of Philosophy in Biomaterials
Science*

By

Sherif Refat Abdelmonem

**B.D.S (Cairo University 2001)
M.Sc. (Cairo University 2010)**

**Faculty of Dentistry
Ain-Shams University
2019**

قال الله تعالى :

﴿ يَرْفَعُ اللَّهُ الَّذِينَ ءَامَنُوا مِنْكُمْ وَالَّذِينَ أُوتُوا

الْعِلْمَ دَرَجَاتٍ ۗ وَاللَّهُ بِمَا تَعْمَلُونَ خَبِيرٌ ۝۱۱ ﴾

سورة المجادلة

SUPERVISORS

Prof. Dr. Ghada Atef Alian

Professor of Biomaterials
Department of Biomaterials
Faculty of Dentistry
Ain Shams University

Prof. Dr. Mohamed Mohamed Selim

Professor of Physical Chemistry
Department of Physical Chemistry
National Research Center (NRC)

Dr. Dalia Ibrahim Sherief

Lecturer of Biomaterials
Department of Biomaterials
Faculty of Dentistry
Ain Shams University

Dedication

**To my great parents, my backbone who always
support me in every step of my life with their
love and kindness.**

To my beloved family my wife and my sons

To my brother my best friend

ACKNOWLEDGEMENT

*I would like to express my deep respect and gratitude to **Prof. Dr. Ghada Atef Alian** Professor of Biomaterials, Department of Biomaterials, Faculty of Dentistry, Ain-Shams University for her great support, inspiration, and encouragement from beginning till the last moment of this work, offering great experience, invaluable advices, effort and time supporting and guiding me to the right way.*

*I express my sincere appreciation and respect to **Prof. Dr. Mohamed Mohamed Selim** Professor of Physical Chemistry, Department of Physical Chemistry, National Research Center (NRC) for his great support and for guiding me throughout this work, especially during practical experiments and the interpretation of their results, enabling all the facilities of laboratory to be handy and teaching me a lot by his great experience and scientific knowledge.*

*Countless thanks and much gratitude to **Dr. Dalia Ibrahim Sherief** Lecturer of Biomaterials, Department of Biomaterials, Faculty of Dentistry, Ain Shams University for her invaluable advices, step by step guidance during preparation and designing of the practical work, and for her patience and great help during editing the final form of the thesis, offering me a lot of her time, effort and hard work for the optimum result of this study.*

Sherif Refat

Table of Contents

Introduction.....	1
Review of literature.....	4
1. Major connectors of maxillary removable partial dentures (RPDs).....	4
1.1. Requirements	4
1.2. Types of Major Connectors	5
2. Materials used for construction of RPD framework	6
2.1. Cobalt-Chromium (Co-Cr) alloys.....	6
2.1.1. Composition and microstructure	6
2.1.2. Properties.....	7
2.2. Thermoplastic polymers:	9
2.2.1. Acetal resins	9
2.2.1.1. Composition and microstructure	9
2.2.1.2. Properties.....	10
2.2.2. Polyetheretherketone (PEEK)	12
2.2.2.1. Composition and microstructure	12
2.2.2.2. Properties.....	13
3. Mechanical testing of denture materials	16
3.1. Flexural strength	16
3.2. Fracture strength:.....	17
4. Biodegradation of denture materials:.....	19
4.1. Evaluation methods of biodegradability of denture materials.....	21
5. Microstructure analysis of denture materials:.....	24
5.1. Scanning Electron Microscope (SEM)	24
5.2. Energy dispersive X-ray (EDX) analysis	25
Aim of study.....	27
Materials and Methods.....	28
1 Materials	28

2	Methods.....	30
2.1.	Designing of specimens	30
2.1.1.	Design of palatal straps specimens for fracture resistance and biodegradability tests	30
2.1.2.	Design of rectangular bars specimens for flexural strength and micro-hardness tests	36
2.2.	Preparation of specimens	37
2.2.1.	Preparation of acetal polymer palatal straps and bars.....	37
2.2.2.	Preparation of granular BioHPP polymer palatal straps and bars	39
2.2.3.	Preparation of cast cobalt-chromium Co-Cr alloy specimens.....	47
2.3.	Grouping of specimens	52
2.4.	Storage conditions.....	56
2.5.	Testing procedures	59
2.5.1.	Fracture resistance test (maximum fracture load).....	59
2.5.2.	Flexural strength test.....	61
2.5.3.	Microhardness test	63
2.5.4.	Tests for biodegradation	64
2.5.4.1.	Fourier-transform infrared spectroscopy (FTIR)	65
2.5.4.2.	UV-Vis Spectrophotometer	66
2.5.4.3	High performance liquid chromatography (HPLC) ..	67
2.5.4.4	Atomic absorption spectroscopy (AAS)	68
2.5.5.	Microstructural analysis.....	68
2.6.	Statistical analysis.....	70
	Results	72
1	Statistical analysis of mechanical testing results	72
1.1.	The effect of water storage on mechanical properties	72
1.1.1.	Maximum compression load (MCL) of palatal straps	72
1.1.2.	Flexural strength of bar specimens.....	74
1.1.3.	Microhardness of bar specimens	75

1.2.	The effect of different materials on mechanical properties	77
1.2.1.	Maximum compression load (MCL) of palatal straps	77
	No water storage subgroups.....	78
	After 6 months of water storage.....	78
1.2.2.	Flexural strength of bar specimens.....	79
	No water storage subgroups.....	80
	After 6 months water storage.....	80
1.2.3.	Microhardness of bar specimens	81
	No water storage subgroups.....	82
	After 6-months water storage.....	82
2.	Results of biodegradation tests	83
2.1.	Fourier-transform infrared spectroscopy (FTIR).....	83
2.1.1.	Taking chamber air spectrum as background.....	83
2.1.2.	Taking de-ionized water spectrum as background..	88
2.2.	UV-Vis Spectrophotometer	91
2.3.	Results of high-performance liquid chromatography (HPLC).....	93
2.4.	Results of atomic absorption spectroscopy (AAS).....	94
3.	Results of Field Emission Microscope (FEM) Scanning and Energy Dispersive Spectroscopy (EDAX).....	96
3.1.	Specimens of microstructure study group	96
3.1.1.	Pink acetal specimen	96
3.1.2.	BioHPP specimen.....	98
3.1.3.	Co-Cr alloy specimen.....	102
3.2.	Fractured or deformed palatal straps	106
3.2.1.	Pink acetal palatal straps	106
3.2.1.1.	Fractured pink acetal palatal straps without water storage (A _p PS1).....	106
3.2.1.2.	Fractured pink acetal palatal straps after water storage (A _p PS2).....	109

3.2.2. Deformed BioHPP palatal straps.....	112
3.2.2.1. Deformed BioHPP palatal straps without water storage (BPS1)	112
3.2.2.2. Deformed BioHPP palatal straps after water storage (BPS2)	115
3.2.3. Fractured Co-Cr alloy straps	118
3.2.3.1. Fractured Co-Cr alloy straps without water storage (CPS1)	118
3.2.3.2. Fractured Co-Cr alloy straps after water storage (CPS2)	123
Discussion.....	130
Summary and Conclusions	150
Appendix.....	155
I-Mechanical testing graphs:.....	155
References.....	168

List of figures

Figure 1. Duplicated stone cast.....	31
Figure 2. Desktop structured-light 3D scanner SHERA eco-scan 7 .	32
Figure 3. Dental wings designing device	32
Figure 4. SHERA eco-mill 5x CAD/CAM milling machine	33
Figure 5. SHERA eco-mill 5x milling machine loading table after milling	34
Figure 6. SHERA CAD/CAM software application	34
Figure 7. Adjusting milling parameters	35
Figure 8. Positions of milling in CAD/CAM blocks.....	35
Figure 9. Supports between specimens to prevent their falling.....	36
Figure 10. Acetal palatal strap on the scanned stone cast.....	38
Figure 11. Outside caliper (top), Vernier caliper (bottom)	38
Figure 12. Acetal bars milled from CAD/CAM blocks as shown in the lower part of the block.....	39
Figure 13. CAD/CAM wax milling blanks used for milling of BioHPP palatal straps wax patterns	40
Figure 14. Wax pattern of BioHPP bar milled by CAD/CAM machine	40
Figure 15. BioHPP granules to be loaded in the loading tubes of..	41
Figure 16. Injection molding system Bredent thermopress 400	41
Figure 17. Flasking of BioHPP wax patterns using hard stone	42

Figure 18. Pink wax for making sprue	42
Figure 19. The chosen program for BioHPP injection	43
Figure 20. Thermo paste 400 coated on the outer surface of the BioHPP granules loading tubes.....	43
Figure 21. Acrylic sep separating medium coated on investment stone	44
Figure 22. Loading flasks in thermopress device	45
Figure 23. Granular BioHPP® granules.....	45
Figure 24. The BioHPP specimens after flasks disassembling with sprue in the middle.....	46
Figure 25. Finished BioHPP palatal straps.....	46
Figure 26. CAD/CAM PMMA blanks	47
Figure 27. CAD/CAM milled PMMA patterns of palatal strap and bar specimens.....	48
Figure 28. Sprue wax for PMMA palatal strap specimen	48
Figure 29. Boxing of PMMA specimen.....	49
Figure 30. Investing the PMMA specimen.....	49
Figure 31. Wax burnout furnace.....	50
Figure 32. The used metal alloy in casting	51
Figure 33. Cast Co-Cr alloy palatal strap	52
Figure 34. Storage de-ionized water	56
Figure 35. Bar specimens stored in glass flasks	56

Figure 36. Storage of specimens in incubator	57
Figure 37. Each palatal strap stored separately	58
Figure 38. Instron testing machine.....	59
Figure 39. BioHPP palatal strap after performing fracture resistance test.....	60
Figure 40. BioHPP palatal strap marked at the loading point	60
Figure 41. BioHPP bar on supports before 3 point bending test	62
Figure 42. Acetal bar during 3 point bending test	62
Figure 43. Microhardness tester	64
Figure 44. Field emission microscope (Quanta FEG 250)	70
Figure 45. Bar chart of effect of water storage on means of maximum compression load for the tested straps materials.....	73
Figure 46. Bar chart of effect of water storage on means of flexural strength of the three tested bar materials.....	75
Figure 47. Bar chart of the effect of water storage on means of microhardness for the three tested bar materials.....	76
Figure 48. Bar chart of the effect of palatal strap material type on the means of maximum compression load	78
Figure 49. Bar chart of effect of bar specimens material type on the means of flexural strength.....	80
Figure 50. Bar chart to explore the effect of bar material type on the means of microhardness.....	82
Figure 51. ATR FTIR spectrum graph of fresh as-received deionized water sample taken directly from water package (W before)	83

Figure 52. ATR FTIR spectrum graph of stored de-ionized water without specimens (W)	85
Figure 53. ATR FTIR spectrum graph of pink acetal storage water (A).....	86
Figure 54. ATR FTIR spectrum graph of BioHPP storage water (B)	87
Figure 55. ATR FTIR spectrum graph of Co Cr alloy storage water (C)	88
Figure 56. Spectral analysis graph of storage water of pink acetal (A*).....	89
Figure 57. Spectral analysis graph of storage water of BioHPP (B*)	89
Figure 58. Spectral analysis graph of storage water of Co Cr alloy (C*)	90
Figure 59. Spectral analysis graph of de-ionized water stored without specimens (W*)	90
Figure 60. UV-Vis spectral analysis graph of pink acetal storage water (AUV).....	91
Figure 61. UV-Vis spectral analysis graph of BioHPP storage water (BUV).....	92
Figure 62. UV-Vis spectral analysis graph of Co Cr alloy storage water (CUV)	92
Figure 63. UV-Vis spectral analysis graph of stored deionized water without specimens (WUV)	93
Figure 64. Pink acetal surface 500X micrograph (microstructure study group)	96
Figure 65. EDAX of random point area of pink acetal surface with elemental composition (microstructure study group)	97