

สมบัติเชิงกลของชีเม้นต์กราดูกเสริมแรงด้วยสารเสริมแรงชนิดผง



นาย ชัยพร เอียงศรี ธรรมชาติ

ศูนย์วิทยทรัพยากร

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต

สาขาวิชาปีตรีเคมี

บัณฑิตวิทยาลัย จุฬาลงกรณ์มหาวิทยาลัย

พ.ศ.2537

ISBN 974-584-325-3

ลิขสิทธิ์ของบัณฑิตวิทยาลัย จุฬาลงกรณ์มหาวิทยาลัย

MECHANICAL PROPERTIES OF PARTICULATE
REINFORCED BONE CEMENT

MR. CHAIYAPORN HENGSRITAWAT

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENT
FOR THE DEGREE OF MASTER OF SCIENCE

PROGRAM OF PETROCHEMISTRY

GRADUATE SCHOOL

CHULALONGKORN UNIVERSITY

1994

ISBN 974-584-325-3

Copyright of the Graduate School, Chulalongkorn University



Accepted by the Graduate School, Chulalongkorn University in
Partial Fulfillment of the Requirement for Masters' Degree

Thavorn Vajrabhaya Dean of Graduate School
(Professor Thavorn Vajrabhaya, Ph. D.)

Thesis committee

Pattarapan Prasarakich Chairman
(Associate Professor Pattarapan Prasarakich, Ph. D.)

Dawan Wiwattanada Thesis Advisor
(Assistant Professor Dawan Wiwattanada)

10 10:15

Khongkha Nongkranad.

(Phongsak Wiwattanadate, Ph. D.)

Khemchai Hemachandra, Member
(Assistant Professor Khemchai Hemachandra, Ph. D.)

Nuanphun Chantarasiri Member
(Nuanphun Chantasiri, Ph. D.)



พิมพ์ด้วยน้ำหมึกดังต่อไปนี้ภาษาไทยในกรอบสีเขียวที่เพียงแผ่นเดียว

ชัยพร เชงศรีวัช : สมบัติเชิงกลของซีเมนต์กระดูกเสริมแรงด้วยสารเสริมแรงชนิดพง
(MECHANICAL PROPERTIES OF PARTICULATE REINFORCED BONE CEMENT) อ.ที่ปรึกษา : ผศ.ดร.ดาวลักษณ์ วิวรรณะเดช, อ.ที่ปรึกษาร่วม : อ.ดร.พงศ์ศักดิ์
วิวรรณะเดช, 128 หน้า. ISBN 974-584-325-3

สมบัติเชิงกลของซีเมนต์กระดูกพอลิเมธิลเมทาไครเลต ซึ่งเป็นซีเมนต์กระดูกดังเดิมสามารถปรับปูนโดยการแทนที่พอลิเมธิลเมทาไครเลตด้วยพอลิเมอร์สมของพอลิเมธิลเมทาไครเลต กับพอลิเอธิลเมทาไครเลต ในอัตราส่วน 80 ต่อ 20 ขณะเดียวกันสมบัติเชิงกลของซีเมนต์กระดูกที่พอลิเมอร์สามารถปรับปูนขึ้นอย่างเด่นชัด เมื่อเสริมแรงด้วย แม่เรียมชัลเฟต หรือ ไฮดรอกซิอะปไทด์ที่ 5 ถึง 40 เมอร์เซนต์ โดยน้ำหนักของพอลิเมอร์ นอกจากนี้ยังพบว่าประสิทธิภาพการเสริมแรงของไฮดรอกซิอะปไทด์จะเพิ่มขึ้นอย่างเด่นชัด เมื่อไฮดรอกซิอะปไทด์มีองค์ประกอบไข聆บนพื้นผิวประมาณ 5 เมอร์เซนต์โดยน้ำหนักของไฮดรอกซิอะปไทด์

ศูนย์วิทยทรัพยากร จุฬาลงกรณ์มหาวิทยาลัย

ภาควิชา มนุสจิตศึกษา ประจำปี พ.ศ.๒๕๖๒
สาขาวิชา มนุสจิตศึกษา
ปีการศึกษา ๒๕๖๖

ลายมือชื่อนิสิต
ลายมือชื่ออาจารย์ที่ปรึกษา
ลายมือชื่ออาจารย์ที่ปรึกษาร่วม
.....

C485049 : MAJOR PETROCHEMISTRY

KEY WORD: BONE CEMENT/ PARTICULATED REINFORCEMENT/ ACRYLIC POLYMER

CHAIYAPORN HENGSRITAWAT : MECHANICAL PROPERTIES OF PARTICULATE REINFORCED BONE CEMENT. THESIS ADVISOR : ASSIST. PROF. DAWAN WIWATTANADATE, Ph.D. THESIS CO-ADVISOR : PHONGSAK WIWATTANADATE, Ph.D. 128 PP. ISBN 974-584-325-3

Mechanical properties of conventional acrylic bone cement can be improved by replacement PMMA with 80/20 PMMA-Co-PEMA. Consequently, mechanical properties of the copolymer bone cement were significantly improved by reinforced with BaSO₄ or hydroxyapatite (HAP) at 5-40 % by weight of bone cement powder. Further enhancement in the mechanical properties was observed when the copolymer bone cement was reinforced with HAP having silane on the surface approximately 5 % by weight of HAP.

ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

ภาควิชาเคมีสาขาวิชากيميคอล. โพลิเมอร์

สาขาวิชา ปู.๒๖๘

ปีการศึกษา ๒๕๓๖

ลายมือชื่อนิสิต *ธน พูลวิชัย*

ลายมือชื่ออาจารย์ที่ปรึกษา *ดร. ดอนดี วงศ์สุขุม*

ลายมือชื่ออาจารย์ที่ปรึกษาร่วม *ดร. สมชาย ใจดี*



ACKNOWLEDGEMENTS

I would like to express my sincere thank to my advisor, Assistant Professor Dawan Wiwattanadate and also my co-advisor, Dr. Phongsak Wiwattanadate, for their valuable guidance, kindness and encouragement throughout the course of this thesis.

I also would like to thank Associate Professor Dr. Pattarapan Prasassarakick, Assistant Professor Khemchai Hemachandra and Dr. Nuanphun Chantarasiri for their serving on committee as chairperson and members of this thesis.

Thanks are also extended to Professor William Bonfield, Professor Mike Braden, Dr. J.C. Behiri and Dr. K.E. Tanner for their kind suggestion and coordination for materials support.

Petroleum and Petrochemical College, Metallurgy and Materials Science Research Institute, and Department of Materials Science, Faculty of Science, Chulalongkorn University are also greatly acknowledged for supporting chemicals, equipments and instruments.

Finally, I would like to thank everyone in the department, the institute and the college for their kind assistance and encouragement throughout the course of study.



CONTENTS

	PAGE
ABSTRACT (in Thai).....	iv
ABSTRACT (in English).....	v
ACKNOWLEDGMENTS.....	vi
CONTENTS.....	vii
LIST OF TABLES.....	xi
LIST OF FIGURES.....	xiii
ABBREVIATIONS.....	xvi
CHAPTER I INTRODUCTION.....	1
CHAPTER II REVIEW OF THE LITERATURE.....	4
2.1 Mechanical Properties of Cortical Bone.....	11
2.1.1 Elastic Behavior of Cortical Bone.....	11
2.1.2 Fracture Toughness of Cortical Bone.....	13
2.2 Development of New Implant Materials.....	16
2.3 Development of New Bone Cement.....	19
2.3.1 Characteristics of Acrylic Bone Cement.....	21
2.3.1.1 General and Chemical Composition....	21
2.3.1.2 Reaction Mechanism of Bone	
Cement.....	27
2.3.2 Mechanical Properties of Bone Cement.....	31
2.3.3 Factors Affecting Mechanical Properties	
of Bone Cement.....	33
2.3.3.1 Effect of Handling and Cementing	
Technique.....	33

CONTENTS (continued)

	PAGE
2.3.3.1.1 Effect of Handling.....	33
2.3.3.1.2 Effect of Cementing Technique...	34
2.3.3.2 Effect of Antibiotic and Other Inclusions.....	36
2.3.3.3 Effect of <i>In Vivo</i> Environment.....	37
2.3.3.4 Effect of Radiopacifying Materials....	38
2.3.3.5 Effect of Irradiation.....	39
2.3.3.6 Effect of Temperature.....	39
2.3.3.7 Influency of Porosity.....	40
2.3.3.8 Effect of Strain Rate.....	41
2.3.4 Modified Bone Cement.....	42
2.3.4.1 Fiber Reinforcement.....	43
2.3.4.2 Particulate Reinforcement.....	50
2.3.5 Polymer Replacement Bone Cement.....	54
2.3.6 Compatibility Enhancement with Silane Coupling Agent.....	55
CHAPTER III EXPERIMENTAL.....	59
3.1 Chemicals and Materials.....	59
3.1.1 Bone Cement Powder.....	59
3.1.2 Bone Cement Liquid.....	60
3.1.3 Silane Coupling Agent.....	60
3.1.4 Reinforcement.....	60

CONTENTS (continued)

	PAGE
3.2 Apparatus.....	62
Mould for Tensile Testing.....	62
3.3 Machines and Instruments.....	63
3.3.1 Particle Size Analyzer.....	63
3.3.2 Scanning Electron Microscopy (SEM).....	63
3.3.3 FT-IR Spectrophotometer.....	64
3.3.4 Universal Testing Machine.....	64
3.4 Sample Preparation and Testing Procedure.....	64
3.4.1 Particle Size Analysis.....	64
3.4.2 Microstructural Analysis.....	64
3.4.3 Silane Surface Treatment on Hydroxyapatite....	65
3.4.4 Silane Content Determination.....	65
3.4.5 Preparation of Tensile Specimens.....	66
3.4.6 Tensile Testing.....	67
CHAPTER IV RESULT AND DISCUSSION.....	69
4.1 Shape of PMMA, PMMA-Co-PEMA, BaSO₄, HAP investigated by SEM.....	69
4.2 FT-IR Characterization of Silane Coupling Agent on Hydroxyapatite.....	76
4.3 Silane Coupling Agent Content on Hydroxyapatite.....	78
4.4 Mechanical Properties of BaSO₄ Reinforced PMMA Bone Cement.....	78
4.5 Mechanical Properties of BaSO₄ Reinforced PMMA-Co-PEMA Bone Cement.....	81

CONTENTS (continued)

	PAGE
4.6 Mechanical Properties of Hydroxyapatite Reinforced PMMA-Co-PEMA Bone Cement.....	84
4.7 Mechanical Properties of Silane Treated HAP Reinforced PMMA-Co-PEMA Bone Cement.....	86
4.8 Void Content in the Bone Cement Specimens.....	94
CHAPTER V CONCLUSION.....	101
SUGGESTION.....	102
REFERRENCE.....	103
APPENDIXES	
APPENDIX A Mechaincal Properties Test by Instron Universal Testing Machine	118
APPENDIX B Calculation Works.....	121
APPENDIX C Basic of Instruments.....	123
VITA.....	128

TABLES

	PAGE
Table 2.1 Some previous investigations of the fracture mechanics parameters for bone.....	15
Table 2.2 Comparison of mechanical properties of current implant materials with those of cortical bone.....	17
Table 2.3 Current clinical applications of acrylic resin implants.....	21
Table 2.4 Composition of mouldable room temperature curing(a) acrylic resin.....	29
Table 2.5 Effect of mechanical properties for radiopacifiers added PMMA cement.....	38
Table 2.6 Mechanical properties of human bone, PMMA, and prosthesis materials.....	45
Table 2.7 Mechanical properties of carbon fiber reinforced PMMA.....	47
Table 2.8 The mechanical properties of typical bioactive materials.....	53
Table 3.1 Characteristic of powders and reinforcements.....	59
Table 3.2 Chemical composition of bone cement.....	67
Table 4.1 Silane coupling agent content inhydroxyapatite.....	78
Table 4.2 Tensile properties of BaSO ₄ reinforced PMMA.....	79
Table 4.3 Comparison of tensile properties of PMMA-Co-PEMA and PMMA	81

TABLES (Continued)

	PAGE
Table 4.4 Tensile properties of BaSO ₄ reinforced PMMA-Co-PEMA.....	82
Table 4.5 Tensile properties of untreated hydroxyapatite reinforced PMMA-Co-PEMA.....	84
Table 4.6 Tensile properties of PMMA-Co-PEMA bone cement reinforced with hydroxyapatite having 3.72 weight percent silane content	87
Table 4.7 Tensile properties of PMMA-Co-PEMA bone cement reinforced with hydroxyapatite having 5.21 weight percent silane content	89
Table 4.8 Tensile properties of PMMA-Co-PEMA bone cement reinforced with hydroxyapatite having 7.83 weight percent silane content	91
Table 4.9 Void content of bone cement specimens.....	95

ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

FIGURES

	PAGE
Figure 1.1 Schematic diagram showing Charnley's total hip replacement	7
Figure 1.2 Schematic diagram showing factors effecting characteristic of joint.....	8
Figure 2.1 Chemical structure of organic components in the powder.....	23
Figure 2.2 A characterized tacticities of bone cement powder.....	25
Figure 2.3 The chemical structure of 3 liquid component.....	26
Figure 2.4 Bonding through silanes by interdiffusion : open circles indicate of regions of coupling agent; solid circles indicate of regions of polymer	56
Figure 2.5 Monolayer model for bonding in mineral-polymer composites.....	57
Figure 3.1 A size distribution curve of BaSO ₄	61
Figure 3.2 A size distribution curve of hydroxapatite	61
Figure 3.3 Standard dimensions mould of ASTM D638 type V (microtension) designer for tensile testing.....	62
Figure 3.4 Standard dimensions mould of ISO/DIS 6239/1 designer for tensile testing.....	63
Figure 4.1 SEM photograph of BaSO ₄ particles (a) x 1.0 k (b) x 20 k.....	70
Figure 4.2 SEM photograph of hydroxyapatite particles (a) x 1.0 k (b) x 50 k.....	71

FIGURES (Continued)

	PAGE
Figure 4.3 SEM photograph x 100 of PMMA powder.....	72
Figure 4.4 SEM photograph x 150 of PMMA-Co-PEMA powder....	73
Figure 4.5 SEM photograph of fracture sample of untreated hydroxyapatite reinforced PMMA-Co-PEMA bone cement (a) x 800 (b) x 1.0 k.....	74
Figure 4.6 SEM photograph of fracture sample of silane treated hydroxyapatite reinforced PMMA-Co-PEMA bone cement (a) x 1.0 k (b) x 3.0 k.....	75
Figure 4.7 FT-IR spectra of hydroxyapatite before (a) and after (b) silane surface treatment.....	77
Figure 4.8 Tensile properties of PMMA dependence with increasing weight percent of BaSO ₄ (a) Stress at maximum load (b) Strain to failure (c) Young's modulus.....	80
Figure 4.9 Tensile properties of PMMA-Co-PEMA dependence with increasing weight percent of BaSO ₄ (a) Stress at maximum load (b) Strain to failure (c) Young's modulus.....	83
Figure 4.10 Tensile properties of PMMA-Co-PEMA dependence with increasing weight percent of untreated hydroxyapatite (a) Stress at maxximum load (b) Strain to failure (c) Young's modulus.....	85

FIGURES (Continued)

	PAGE
Figure 4.11 Tensile properties of PMMA-Co-PEMA dependence with increasing weight percent of hydroxyapatite having 3.72 weight percent silane content (a) Stress at maximum load (b) Strain to failure (c) Young's modulus.....	88
Figure 4.12 Tensile properties of PMMA-Co-PEMA dependence with increasing weight percent of hydroxyapatite having 5.21 weight percent silane content (a) Stress at maximum load (b) Strain to failure (c) Young's modulus.....	90
Figure 4.13 Tensile properties of PMMA-Co-PEMA dependence with increasing weight percent of hydroxyapatite having 7.83 weight percent silane content (a) Stress at maximum load (b) Strain to failure (c) Young's modulus.....	92
Figure 4.14 Comparison between Young's modulus of the PMMA-Co-PEMA increased with increasing weight percent of hydroxyapatite reinforcement.....	93

ABBREVIATIONS

min	minute
mm	millimeter
msec	millisecond
mJ	millijoule
ppm	part per million
μm	micrometer
ν	Poisson's value
%	percent
ASTM	The American Standard Test Methods
BaSO_4	barium sulphate
n-BMA	normal butyl methacrylate
BPO	benzoyl peroxide
$^{\circ}\text{C}$	Celsius degree
DHSS	Department of Health and Social Service
DMPT	N,N-dimethyl-p-toluidine
E	Young's modulus
E_c	Young's modulus of composite
E_f	Young's modulus of fiber
E_m	Young's modulus of matrix
FDA	Foods and Drugs Agency
FT-IR	Fourier transform infrared spectroscopy
G	shear modulus
GPa	gigapascal

ABBREVIATIONS (continued)

GPC	gel permeation chromatography
G _c	critical strain energy release rate
HAP	hydroxyapatite
IPN'S	interpenetrating polymer networks
ISO	International Organization for Standardization
K _c	critical stress intensity factor
Mg	megagram
MMA	methyl methacrylate monomer
MPa	megapascal
NMR	nuclear magnetic resonance
PEMA	polyethylmethacrylate
PMMA	polymethylmethacrylate
PMMA-Co-PMMA	polymethylmethacrylate-Co-polyethylmethacrylate
SEM	scanning electron microscope
STEM	scanning-transmission electron microscope
TEM	transmission electron microscopy
V _f	volume of fiber
V _m	volume of matrix