

شبكة المعلومات الجامعية التوثيق الإلكتروني والميكروفيلو

بسم الله الرحمن الرحيم





HANAA ALY



شبكة المعلومات الجامعية التوثيق الإلكتروني والميكرونيله



شبكة المعلومات الجامعية التوثيق الالكتروني والميكروفيلم



HANAA ALY



شبكة المعلومات الجامعية التوثيق الإلكترونى والميكروفيلم

جامعة عين شمس التوثيق الإلكتروني والميكروفيلم قسم

نقسم بالله العظيم أن المادة التي تم توثيقها وتسجيلها على هذه الأقراص المدمجة قد أعدت دون أية تغيرات



يجب أن

تحفظ هذه الأقراص المدمجة بعيدا عن الغبار



HANAA ALY



وَمَا تَوْفِيقِي إِلَّا بِاللَّهِ عَلَيْهِ تَوَكَّلْتُ وَإِلَيْهِ أُنِيبُ

صيكة والله العظيم

Evaluation of a Newly Formulated Antibacterial Flowable Composite Material

Thesis Submitted to the Biomaterials Department Faculty of Dentistry Ain-Shams University in partial fulfillment of the requirements for the PhD Degree in Biomaterials Science

By

Mahitab Mohamed Mansour

B.D.Sc. MSA University (2009)
M.D.Sc. Cairo University (2017)
Assistant Lecturer of Dental Biomaterials Science
Faculty of Dentistry
October University for Modern Sciences and Arts
(MSA)

Biomaterials Department Faculty of Dentistry Ain-Shams University 2021

SUPERVISORS

Professor Dr. Tarek Salah El-Dine Hussein

Professor of Dental Biomaterials
Faculty of Dentistry
Ain -Shams University

Associate Prof. Dr. Haidy Nabil Salem

Associate professor of Dental Biomaterials Restorative and Dental Materials Department

National Research Center

Acknowledgment

Words cannot express my gratitude towards **Prof. Dr. Tarek Salah**, Professor of Biomaterials, Faculty of Dentistry, Ain-Shams University, for his kind supervision, valuable guidance, constructive criticism and from whom I have learnt a lot. I will always be greatly indebted to his tremendous support, unforgettable help, understanding and modesty. "Thank you for your precious trust".

I am greatly honored to express my utmost gratefulness and appreciation to **Associate Prof. Dr. Haidy Nabil Salem** researcher of Dental Biomaterials, National Research Center for her close supervision, continuous encouragement, endless support, and sincere guidance.

I also want to express my deep gratitude to **Associate Prof. Dr. Hisham El Shishtawy** associate professor of microbial genetics, for his knowledge, assistance, guidance, and support in the microbiological component of this research.

I am fortunate to express my utmost gratefulness, love and appreciation to my mother, my ultimate role model, my backbone, and mentor Mrs. Mona El Degwi who has always helped and guided me throughout my entire life and whose love and unconditional support were always with me in whatever I pursued. Thank you for always trusting and believing in me.

I would also like to extend my appreciation to my beloved grandmother **Prof. Dr. Nawal El Degwi**, for her extensive care, support, and continuous guidance. Special thanks and great love are extended to my one and only sister **Engy Mansour** and my **beloved husband Mohamed El Badawy** who have surrounded me with all the support, understanding and care I ever wished for. I also want to thank my kids **Youssef**, **Nour and Taha** for being so patient with me in this journey, and for surrounding me with all the love I needed. Moreover, I appreciate the care, encouragement and support I've always received from my best friend **Reham Aboulnaga**.

Special thanks and great appreciation are owed to my dearest friends **Dr.Mai Hesham** and **Dr.Ahmed Wagdy** for their encouragement, help, support and care throughout this journey. I also want to thank my dear friends and colleagues at **MSA university**. Finally, I would like to cordially thank all the staff members of The Biomaterials Department, Faculty of Dentistry, **Ain-Shams University**, for their friendly support and care.

Dedication



The soul of my grandfather, supporter, and God father

General Wagih El Degwi

The soul of my beloved uncle, my mentor & source of inspiration

Prof. Dr Sherif EL Degwi

And finally, to the soul of my best friend and soulmate

Dr.Miram El Fallah

I really wish you could have all been here to share with me such an accomplishment

May you rest in eternal peace

List of Contents

	Page
List of figures	II
List of tables	V
Introduction	1
Review of literature	4
I. Historical background of esthetic restorations	4
II. Chemistry and composition of dental resin composites	5
II.1. The organic matrix	5
II.1.2. The organic matrix (reinforcing phase)	7
II.1.3. Coupling agent	8
II.1.4. Initiator and activator systems	8
II.1.5. Pigments and other components	9
III. Classification	10
IV. Flowable resin composites	11
V. History of resin composites with antibacterial properties.	14
V.1.Triclosan	15
V.2. Benzalkonium chloride	15
V.3. Fluoride releasing dental composites	15
V.4. Silver containing dental composites	16
V.5. Antibacterial pre-polymerized resin fillers	17
V.6. Chlorhexidine containing dental resin composites	18
VI. Recent antimicrobial agents in dentistry	19
VI.1. Octenidine dihydrochloride	19
Aim of the study	21
Materials and Methods	22
Results	66
Discussion	107
Summary and conclusions	134
References	138
Arabic summary	

List of Figures

	Page
Figure 1: Jenway 3505 bench pH meter	27
Figure 2: TEGRA SPEED electric fast sintering dental furnace	28
Figure 3: White clusters of the sintered nano-silica powder	28
Figure 4a: Grounding particles using agate mortar and pestle	29
Figure 4b: Powder after sieving	29
Figure 5: Sigma 3-16KL centrifugation machine	29
Figure 6: Deposition of nano fillers after 30 minutes centrifugation	30
Figure 7: The precipitate in the petri dish	30
Figure 8: BINDER hot air drying and heating oven	31
Figure 9: The petri dish inside the desiccator	31
Figure 10: Sputter coater	33
Figure 11: Scanning Electron Microscope (SEM)	34
Figure 12: SCHIMADZU FTIR device	35
Figure 13: Split Teflon mold used for diametral tensile strength test	37
Figure 14: Specimens of the diametral tensile strength test	38
Figure 15: Specimen under diametral tensile testing in the universal testing machine	39
Figure 16: Split Teflon mold used for compressive strength test	40
Figure 17: Specimens of the compressive strength test	41
Figure 18: Specimen under compressive strength in the universal testing machine	41
Figure 19: Teflon mold used for flexural strength test	42
Figure 20: Specimens of the flexural strength test	43
Figure 21: Specimen under flexural strength testing in the universal testing machine	44
Figure 22: Mold used for water sorption test	45
Figure 23: Specimens of the water sorption test	45
Figure 24: Mold used for water solubility test	47
Figure 25: Specimens of the water solubility test	48
Figure 26: Mold used for degree of conversion test	50
Figure 27: Specimens of the degree of conversion test	50
Figure 28: Polymerized powder mixed with the KBr salt	51
Figure 29: SCHIMADZU pelleting device	52
Figure 30a: Powder before obtaining the pellet	52
Figure 30b: Powder after turning to a pellet	52
Figure 31: Thirty-two freshly extracted sound human permanent premolars	54
Figure 32: Top view of the premolars embedded in the colored acrylic blocks	55
Figure 33: Teflon mold used for resin-dentin shear bond strength test	56
Figure 34: Resin composite after curing on the teeth for the resin-dentin shear bond	57
strength test	
Figure 35a: Bacterial wells in the BHI media	59

Figure 35b: Bacterial wells in the MRS media
Figure 36: Fisher scientific incubator
Figure 37: Applied Biosystems thermal cycler
Figure 38: UVP transilluminator system
Figure 39: BIO-RAD electrophoresis apparatus
Figure 40: SEM image of (group 2) with 800X magnification
Figure 41: SEM image of (group 2) with 1500 X magnification
Figure 42: EDX of (group 2) showing the main elements Si, O, and C
Figure 43: SEM image of (group 3) with 800X magnification
Figure 44: SEM image of (group 3) with 1500X magnification
Figure 45: EDX of (group 3) showing the main elements Si, O, C and Cl
Figure 46: SEM image of (group 4) with 800X magnification
Figure 47: SEM image of (group 4) with 1500 X magnification
Figure 48: EDX of (group 4) showing the main elements Si, O, C and Cl
Figure 49: FTIR spectrum for all the experimental groups
Figure 50: XRD of all the experimental groups
Figure 51: Bar chart illustrating mean diametral tensile strength in different groups
Figure 52: Bar chart illustrating mean compressive strength in different groups
Figure 53: Bar chart illustrating mean flexural strength in different groups
Figure 54: Bar chart illustrating mean water sorption in different groups
Figure 55: Bar chart illustrating mean solubility in different groups
Figure 56: Bar chart illustrating mean degree of conversion % in different groups
Figure 57: Bar chart illustrating mean dentine bond strength in different groups
Figure 58: Representative photograph for adhesive failure
Figure 59: Representative photograph for cohesive failure
Figure 60: Representative photograph for mixed failure
Figure 61: Bar chart illustrating mean antibacterial effect against <i>Streptococcus mutans</i>
in different groups at 0, 30, 60, 90 and 180 days
Figure 62: Line chart illustrating effect of time on mean antibacterial effect against
Streptococcus mutans in different groups
Figure 63: Control groups of the BHI media showing no antibacterial activity
Figure 64: Plates of group 3 and 4 showing antibacterial activity against <i>Streptococcus</i>
mutans at time 30 days
mutans at time 180 days
Figure 66: Images of the specimens at the start of the test and after 180 days showing
dissolution and surface disintegration after being removed from BHI media
Figure 67: Bar chart illustrating mean antibacterial effect against <i>Lactobacillus casei</i> in
different groups at 0, 30, 60, 90 and 180 days
Figure 68: Line chart illustrating effect of time on mean antibacterial effect against
Lactobacillus casei in different groups
Figure 70: Plates of group 3 and 4 showing antibacterial activity against <i>Lactobacillus</i>
casei at time 30 days
Figure 71: Plates of group 3 and 4 showing antibacterial activity against <i>Lactobacillus</i>
casei at time 180 days

Figure 72: Images of the specimens at the start of the test and after 180 days showing	105
dissolution and surface disintegration after being removed from the MRS media	103
Figure 73: Gel electrophoresis of Streptococcus mutans	106
Figure 74: Gel electrophoresis of <i>Lactobacillus casei</i>	106

List of Tables

	Page
Table I: Classification of resin composite based on type of resin composite, filler particle size and clinical use	10
Table II: Material, commercial name, manufacturer, and batch number of the commercially available materials used in the study	22
Table III: Chemical name, presentation, manufacturer, and batch number of chemicals used in preparation and testing in this study	22
Table IV: Factorial design	25
Table (Va): Common IR assignments for BIS-GMA and TEGDMA	72
Table (Vb): Characteristic peaks of FTIR spectrum of Octenidine dihydrochloride	72
Table (VIa): Descriptive statistics of diametral tensile strength and comparison between groups (ANOVA)	74
Table (VIb): Detailed results of Tukey's post hoc test for comparison of diametral tensile strength	74
Table (VIIa): Descriptive statistics of compressive strength and comparison between groups (ANOVA)	. 76
Table (VIIb): Detailed results of Tukey's post hoc test for comparison of compressive strength	76
Table (VIIIa): Descriptive statistics of flexural strength and comparison between groups (ANOVA)	. 78
Table (VIIIb): Detailed results of Tukey's post hoc test for comparison of flexural strength	78
Table (IXa): Descriptive statistics of water sorption and comparison between groups (ANOVA)	. 80
Table (IXb): Detailed results of Tukey's post hoc test for comparison of water sorption Table (Xa): Descriptive statistics of solubility and comparison between groups (ANOVA)	80 82
Table (Xb): Detailed results of Tukey's post hoc test for comparison of solubility	82
Table (XIa): Descriptive statistics of degree of conversion % and comparison between groups (ANOVA)	84
Table (XIb): Detailed results of Tukey's post hoc test for comparison of degree of conversion %	84
Table (XIIa): Descriptive statistics of resin-dentin shear bond strength and comparison between groups (ANOVA)	86
Table (XIIb): Detailed results of Tukey's post hoc test for comparison of resin-dentin shear bond strength	86
Table (XIIIa): Distribution of failure modes within each group after the shear test	88
Table (XIIIb): Percentage of mode of bond failure found in each group after the shear test	88
Table (XIVa): Descriptive statistics of antibacterial effect against Streptococcus mutans and comparison between groups (ANOVA)	91
Table (XIVb): Detailed results of Tukey's post hoc test for comparison of antibacterial effect against Streptococcus mutans at 30 and 60 days	92

Table (XIVc): Detailed results of Tukey's post hoc test for comparison of antibacterial	93
effect against Streptococcus mutans at 90 and 180 days	93
Table (XV): Effect of time on antibacterial effect against Streptococcus mutans within	94
the same group (Repeated measures ANOVA)	94
Table (XVIa): Descriptive statistics of antibacterial effect against Lactobacillus casei	99
and comparison between groups (ANOVA)	99
Table (XVIb): Detailed results of Tukey's post hoc test for comparison of antibacterial	99
effect against lactobacilli at 30 and 60 days	99
Table (XVIc): Detailed results of Tukey's post hoc test for comparison of antibacterial	101
effect against lactobacilli at 90 and 180 days	101
Table (XVII): Effect of time on antibacterial effect against Lactobacilli within the same	102
group (Repeated measures ANOVA).	102

Introduction

Dental caries is a very common localized and transmissible pathological infectious disease that results in the destruction of hard dental tissues ⁽¹⁾. The different treatment modalities of dental caries include the removal of decayed dental tissues and restoring them with various types of dental restorations, such as dental amalgam, resin composites, ceramics, and gold.

The scientific developments and advances in restorative dental materials, have made resin composites one of the most commonly used materials worldwide for different classes of restorations. Owing to its ability to bind readily to the tooth structure with adhesives, and most importantly having a greater range of shades that aid in close matching to the natural teeth allowing the restoration to look imperceptible ⁽²⁾.

Resin composite restorations are characterized by a high compressive strength relative to most of the other restorative materials ⁽³⁾. Properties of resin composites can be altered to suit a wider range of uses by modifying several factors such as the size of the filler particles, and the activation process. Depending on these properties, resin composites can be used in different parts of the oral cavity.

Flowable resin composites with improved mechanical and chemical characteristics have been widely used in the clinical practice. These resin composites are low-viscosity materials with a reduced amount of inorganic filler particles and a higher percentage of resinous components. Flowable resin composites with their low elastic modulus and minimal stress contraction, compete with stress development potentially helping in maintaining the marginal seal of the restorations. The flowable resin composites are readily workable and adaptable to cavity walls and their use can reduce marginal defects, cuspal deflections and polymerization shrinkage in restorations (4).