

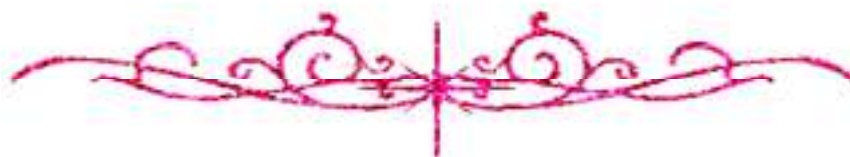
Safaa Mahmoud



بسم الله الرحمن الرحيم

مركز الشبكات وتكنولوجيا المعلومات

قسم التوثيق الإلكتروني



Safaa Mahmoud



جامعة عين شمس

التوثيق الإلكتروني والميكروفيلم

قسم

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



Evaluation of Some Properties Of A Dentin Adhesive After Adding Epigallocatechin-Gallate

**Thesis Submitted to Biomaterials Department The
Faculty of Dentistry Ain-Shams University**

**In partial fulfillment of the requirements for the
Master Degree in Biomaterials Science**

By

Nadia Taha El-Ansary

B.D.SC Ain-Shams University 2009

Biomaterials Department

Faculty of Dentistry

Ain-Shams University

2019

Supervisors

**Prof. Dr. Tarek Salah El Dine
Hussein**

Professor of Dental Materials
Biomaterials department Faculty of
dentistry Ain-shams University

Dr. Mohamed Salah Nassif

Associate Professor of Dental Materials
Biomaterials Department Faculty of
Dentistry Ain-shams University

Acknowledgement

*First, I would like to express my greatest gratitude to the Almighty **ALLAH** for giving me the strength, knowledge, ability and opportunity to undertake this research study and to complete it satisfactorily. Without his blessings, this achievement would not have been possible.*

However, conducting the research and writing this thesis required the patience, persistence and motivation of many people whom I would like to personally acknowledge. First, I would like to extend my deepest thanks to my supervisors:

***Prof. Dr. Tarek Salah El Dine Hussein**, Professor of Biomaterials, Faculty of dentistry, Ain-Shams University, for his highly appreciated guidance and endless support throughout this work. His valuable experience and honorable supervision will always be remembered with a lot of gratitude.*

***Dr. Mohamed Salah Nassif**, Associate Professor of Biomaterials, Faculty of dentistry, Ain-Shams University, for sparing no effort or time in guiding me throughout this work. The door to his office was always open for me. His constant encouragement, understanding, patience and healthy criticism added considerably to my experience.*

*These acknowledgements would be incomplete without expressing my deepest thanks and appreciation to all my professors and colleagues in the Biomaterials department for always supporting and guiding me especially **Dr. Aliaa Hazem***

***Sakr**, Assistant Lecturer of Dental Biomaterials, Faculty of Dentistry, Ain-Shams University and **Dr. Lamia Mostafa Abd El-Aziz**, Assistant Lecturer of Dental Biomaterials, Faculty of Dentistry, Ain-Shams University*

*I would also like to acknowledge **Dr. Ahmed Abdou**, Teaching Assistant of Dental Biomaterials, Faculty of Dentistry, Modern University for Technology and Information for his great help in this thesis statistics.*

Dedicated to

My dad

Who raised me to be a hard worker, my support system who always pushes me forward

My mom

My comfort zone, who always loves, supports and inspires me in every step I take

My brother

My backbone, who helps me a lot with his love, encouragement, patience and hi-tech services

My husband

My life partner and best friend who is always there for me

My children

Ibrahim and Celine who are the source of my joy and happiness

My grandmother

Who is always there for me with her love, prayers and blessings

My aunt

Who is always a source of love and support trying to make things easier for me

My cousins

Who always support and help me in every possible way

List of contents

Contents

<u>List of contents</u>	VI
<u>List of figures</u>	X
<u>List of tables</u>	XII
<u>List of abbreviations</u>	XIII
<u>Introduction</u>	1
<u>Review of literature</u>	3
<u>1 Dental adhesive:</u>	3
1.1 Components of dental adhesive:	3
1.1.1 Resin monomer:	3
1.1.2 Initiators:	6
1.1.2.1 Photo-initiators	6
1.1.2.2 Chemical initiators	7
1.1.3 Inhibitors	8
1.1.4 Solvents	9
1.1.5 Fillers	11
1.2 Classification of dental adhesives	11
1.2.1 Classification according to the interaction with smear layer	12
1.2.2 Classification based on generation systems	13
1.2.3 Classification according to mechanism of adhesion	15

1.2.3.1	Etch and rinse adhesive system.....	15
1.2.3.2	Self etch adhesive.....	17
2	<u>Factors that affect the durability of resin dentin bond</u>	18
2.1	Hydrolytic degradation due to water sorption:.....	18
2.2	Incomplete resin infiltration.....	19
2.3	Collagenolysis by Endogenous MMPs and Cysteine Cathepsins.....	19
3	<u>Strategies to optimize the durability of the resin dentin bond</u>	21
3.1	Ethanol wet bond.....	21
3.2	Remineralizing the hybrid layer collagen.....	22
3.2.1	Remineralizing by fluoride containing adhesives.....	23
3.2.2	Remineralization by adhesives containing bioactive particl.....	23
3.2.3	Biomimetic remineralization.....	23
3.3	Matrix metalloproteinase inhibition and collagen cross linkin.....	24
4	<u>Epigallocatechin Gallate</u>	28
4.1	Separation and purification of EGCG.....	28
4.2	Uses of EGCG in dentistry.....	29
4.3	Effect of EGCG on bonding to dentin.....	31
5	<u>Color stability of dental adhesives</u>	32
5.1	Color stability testing.....	32
5.1.1	Colorimeters.....	33
5.1.2	Digital cameras.....	33
5.1.3	Spectrophotometers.....	33

6	<u>Water sorption and solubility of dental adhesive</u>	34
7	<u>Bond durability testing</u>	35
7.1	Shear bond strength	35
7.2	Tensile bond strength	37
8	<u>Aging protocol</u>	38
	<u>Aim of the study</u>	39
	<u>Material and methods</u> ..	40
1.	<u>EGCG/adhesive mixture preparation</u>	41
2.	<u>Testing methodology</u>	42
2.1	Color stability testing	42
2.2	Water sorption and solubility of the adhesive testing	45
2.3.	Micro-tensile bond strength testing	50
	<u>Results</u>	60
1.	<u>Results of color stability test</u>	60
a.	Effect of the adhesive of different groups on color difference	60
b.	Effect of aging on the color difference within the same group	64
2.	<u>Results of water sorption and solubility test</u>	65
a.	Water sorption test	65
b.	water solubility test	67
c.	Eluted components	68
3.	<u>Results of microtensile bond strength (Mpa)</u>	71

a. Effect of the adhesive of different groups on the microtensile bond strength.....	71
b. Effect of aging time on the microtensile bond strength within the same group.....	73
4. <u>Failure mode analysis and results of scanning electron microscope</u>	75
<u>Discussion</u>	77
<u>Summary and conclusions</u>	83
<u>References</u>	85
<u>صخلملا يبر علا</u>	١

List of figures

Figure 1: IPS emax disc.....	42
Figure 2: Agilent Cary 5000 spectrophotometer.....	43
Figure 3: The disc after being placed in distilled water for 1 year.....	45
Figure 4: Teflon mold placed on a celluloid strip and filled with adhesiv.....	46
Figure 5: Adhesive discs placed within the dessicator.....	47
Figure 6: Initial weight of an adhesive disc.....	47
Figure 7: Adhesive disc in distilled water.....	48
Figure 8: Vertex 70 FTIR spectrometer.....	49
Figure 9: Teflon mold with corresponding metal ring and paralleling screws (Top view).....	51
Figure 10: Isomet 4000.....	51
Figure 11: Flat dentin surface.....	52
Figure 12: A block of 4 mm high resin composite+tooth+acrylic.....	53
Figure 13: Gripping attachment (Front view) with perpendicular axial grooves.....	54
Figure 14: The obtained beams.....	55
Figure 15: Measuring the beam's thickness at the composite-dentine interface (0.9 mm).....	55
Figure 16: Beam mounted on Geraldeli's jig.....	56
Figure 17: Zapit glue kit.....	57
Figure 18: Jig mounted on the Universal testing machine.....	58
Figure 19: Bluehill Lite machine operating software results.....	58
Figure 20: Column chart showing mean color difference (ΔE) between the discs without adding the adhesive and 24 hours after adding it.....	63

Figure 21: Column chart showing mean color difference (ΔE) between the discs without adding the adhesive and 6 months after adding it.....	63
Figure 22: Column chart showing mean color difference (ΔE) between the discs without adding the adhesive and 1 year after adding it.....	63
Figure 23: Column chart showing mean weight difference over volume in $\mu\text{g}/\text{mm}^3$ for different groups (ANOVA test).....	66
Figure 24: Column chart showing mean weight difference over volume in $\mu\text{g}/\text{mm}^3$ for different groups (ANOVA test).....	68
Figure 25: Infrared spectrum of distilled water.....	69
Figure 26: Infrared spectrum of distilled water after removing the disc of group 1.....	69
Figure 27: Infrared spectrum of distilled water after removing the disc of group 2.....	70
Figure 28: Infrared spectrum of distilled water after removing the disc of group 3.....	70
Figure 29: : Column chart showing mean microtensile bond strength values in Mpa for different groups in different aging periods.....	72
Figure 30: Line chart showing mean microtensile bond strength values in Mpa for each adhesive group in different observation times.....	74
Figure 31: Image of the fractured beam using Stereomicroscope and showing mixed mode of failure	75
Figure 32: SEM image 1200 x showing fractured surface having remnants of composite and adhesive with open dentinal tubules showing mixed mode of failure.....	76
Figure 33: SEM image 1200x showing fractured composite surface indicating cohesive failure in the composite.....	76

List of tables

Table 1: Materials, brand name, manufacturer, main constituents, lot number and expiry date.....	40
Table 2: The mean color difference (ΔE) of the emax discs before and after adding the adhesive for 24 hours in different groups.....	61
Table 3: The mean color difference (ΔE) of the emax discs before and after adding the adhesive for 6 months in different groups	61
Table 4: The mean color difference (ΔE) of the emax discs before and after adding the adhesive for 1 year in different groups	62
Table 5: The mean color difference (ΔE) between the discs without adding the adhesive and after adding it for different adhesive groups in different aging periods	64
Table 6: Mean weight difference over volume ($W2-W1/V$) in $\mu\text{g}/\text{mm}^3$ for different groups (ANOVA test).....	65
Table 7: Table (7) Mean weight difference over volume ($W1-W3/V$) in $\mu\text{g}/\text{mm}^3$ for different groups (ANOVA test).....	67
Table 8: Mean microtensile bond strength values in Mpa for different adhesive groups in different aging periods (ANOVA test).....	72
Table 9: Mean microtensile bond strength values in Mpa at different aging periods for different adhesive groups.....	74

List of Abbreviations

ACP: Amorphous calcium phosphate

Bis-GMA: Bisphenol A diglycidyl methacrylate

BHT: Butylated hydroxy toluene

BAC: Benzalkonium chloride

BPDM: Biphenyl dimethacrylate

CHX: Chlorhexidine

CQ: Camphroquinone

HEMA: 2-hydroxyethyl methacrylate

EDC: 1-ethyl-3-(3-dimethylaminopropyl) carbodiimide

EDTA: Ethylene diamine tetraphosphonic acid

EGCG: Epigallocatechin gallate

EGDMA: Ethylene glycol dimethacrylate

FEA: Finite element analysis

GAGs: Glucosaminoglycans

GTase: Glucosyltransferase

GBR: Guided bone regeneration

H₃PO₄: Phosphoric acid

MDPB: 12-methacryloyloxydodecylpyridinium bromide

MEP: 2-methacryloyloxyethyl dihydrogen phosphate

MEHQ: Monomethyl ether hydroquinone

MMA: Methyl methacrylate

Mpa: Mega pascal