

**SYNTHESIS OF URETHANE ACRYLATE MACROMER FROM PALM OIL-  
BASED POLYOL AND ITS APPLICATION AS A RESIN FOR DENTAL  
COMPOSITE**

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**2010**

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**THESIS SUBMITTED IN FULFILLMENT OF THE REQUIREMENT FOR  
THE DEGREE OF DOCTOR OF PHILOSOPHY IN CONSERVATIVE  
DENTISTRY**

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## ABSTRACT

A slightly branched urethane acrylate macromer (UAM) was synthesized by reacting palm oil-based polyol with excess amounts of polymeric methylene diphenyl diisocyanate (MDI) to form urethane prepolymer, then reacted with 2-hydroxyethyl methacrylate (HEMA) and FT-IR spectroscopy confirmed the urethane and grafted acrylate groups. 0.2% by weight of camphoroquinone (CQ) and 0.8% by weight of ethyl-4-N,N-dimethylamino-benzoate (4EDMAB) by weight were added to render the experimental resins light-curable. Their viscosity, percentage of degree of conversion (%DC) and cross-linking density (CLD), percentage of volumetric polymerization shrinkage (%VPS), water sorption and solubility, flexural strength, modulus of elasticity and toughness were determined and compared to Bis-GMA.

Light-curable experimental resin systems were also prepared by adding TEGDMA (T) and Bis-EMA (E) as reactive diluents and co-monomers. The following formulations of resin systems were investigated: BT (blending of Bis-GMA and TEGDMA); U/BT (blending of UAM and BT); U/E (3/1) (blending of UAM and Bis-EMA with mass ratio 3/1); U/E(1/1) (blending of UAM and Bis-EMA with mass ratio 1/1); and U/E/BT (blending of UAM and Bis-EMA and BT). The experimental flowable composites (FCs) were prepared by adding 60 % by weight of silanated barium borosilica glass to each of the experimental resin systems except for the U/E(3/1). The same light-initiators, 0.2% by weight CQ and 0.8% by weight 4EDMAB were used. The groups of FCs are as follows; FC-BT which acts as a control group amongst the experimental FCs (Exp-Cont); FC-U/BT; FC-U/E; and FC-U/E/BT. A commercially available Bis-GMA/TEGDMA-based flowable composite, Esthet.X flow (Dentsply, Caulk, USA) was selected as another control group (Com-Cont). The %VPS, percentage of volumetric change, water sorption and solubility, flexural strength, modulus of elasticity, toughness

and cytotoxicity (percentage of viable cell) were determined for all the experimental FCs and Esthet.X flow.

UAM exhibited lower viscosity than Bis-GMA. In addition, the UAM resin showed a higher % DC, flexural strength, toughness and % VPS than the rigid Bis-GMA, while, the water sorption and solubility of Bis-GMA was lower compared to UAM. The viscosities of U/E and U/E/BT were lower than others. When compared to the commonly used resin system BT (Cont), the U/E showed higher % DC, CLD, flexural strength and toughness, and lower water sorption and solubility. When UAM was blended with BT as U/BT resin system, the % DC, CLD, flexural strength, and toughness was higher than BT. However, the U/BT resin system showed higher % VPS, water sorption and solubility than BT resin system. When both UAM and Bis-EMA were blended with BT resin, the U/E/BT resin system showed higher flexural strength, modulus of elasticity, toughness and lower water sorption and solubility.

All experimental FCs fulfilled the requirements of ISO 4049:2000 for flexural strength and water sorption and solubility. The FC-U/BT showed higher water sorption, water solubility and volumetric change than FC-BT (Exp-Cont). On the other hand, FC-U/E and FC-U/E/BT showed lower water sorption and solubility and higher % VPS than FC-BT. UAM-based experimental FCs showed low cytotoxic activity based on the percentage of viable cell determination and the results obtained for the experimental FCs were comparable with the commercial flowable composite, Esthet.X flow.

Within the limitations of this study, it can be concluded that UAM resin showed significantly higher % DC, flexural strength and toughness than Bis-GMA. The UAM resin has potential to be used as a resin system with significantly improved DC, CLD, flexural strength and toughness. The experimental UAM-based-flowable composites fulfilled the ISO 4049 requirements and satisfactory preliminary cytotoxicity screening showed no significant difference in percentage of cell viability compared to Esthet.X

flow. However, future studies using different types of filler systems should be carried out to further enhance its properties.

## DECLARATION

I certify that this research report is based on my own independent work, except where acknowledged in the text or by reference. No part of this work has been submitted for degree or diploma to this or any other university.

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## ACKNOWLEDGEMENTS

All praise and thanks are due to Allah (S.W.T) the most gracious and merciful who has given me the ability to complete this study.

First of all, I would like to express my profound gratitude and deep appreciation to my supervisory committee. My deepest appreciation and infinite gratitude to my first supervisor Associate Professor Dr. Noor Hayaty Abu Kasim for her guidance and encouragement, feedback, insightful remarks, unforgettable support, magnanimity, research behaviour, and advises. My heartfelt appreciation also goes to my second supervisor Professor Dr. Gan Seng Neon who has a wealth of knowledge and experience in chemistry. His guidance in the chemistry field, which is new to me, helped me build my confidence. I am really grateful and appreciative of his understanding, guidance and patience.

I would also like to express my gratitude to Dr. Shamsul from Institute of Medical Research (IMR) for his guidance and help in the cytotoxicity test and for his permission to use the laboratory at IMR.

Many thanks to my colleague, Ms. Shafiza (Chemistry department), and gratitude to the conservative department's secretarial staff; Yati, Florence, and Halimah for being cooperative and friendly.

Financial support from MOSTI (grant 03-01-03SF019), University of Malaya (grant PS281/2007B and PS323/2009A), and Yemen government (Scholarship-Dhamar

University) are gratefully acknowledged. I would also like to thank ESSTECH for donating the chemicals and fillers.

I would like to express my special thanks and deep gratitude to my family (Al-sanabani), first of all my brother Hani for his encouragement and financial support, also Dr Maher, and Professor Dr. Jaber.

Finally, I cannot imagine going through graduate life without my lovely wife Belqes and my sweet sons Ali and Rian and lovely daughter Dona. They provided me with love, support and patience.

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## ABBREVIATIONS

<b>Abbreviation</b>	<b>DESCRIPTION</b>
<b>μl</b>	Micro-liter
<b>μm</b>	Micrometer
<b>4EDMAB</b>	Ethyl-4-N,N-dimethylamino-benzoate
<b>AlkOA65N</b>	Alkyd polyol
<b>Bis-EMA</b>	Ethoxylated Bisphenol A methacrylate
<b>Bis-GMA</b>	Bisphenol A glycidyl methacrylate
<b>CLD</b>	Cross-linking density
<b>cm<sup>-1</sup></b>	Centimeter per minus one
<b>cm<sup>2</sup></b>	Centimeter per meter square
<b>Com-Cont</b>	Commercial control
<b>cp</b>	Centipoise
<b>CQ</b>	Camphoroquinone
<b>DBTDL</b>	Dibutyltin dilaurate
<b>DC</b>	Degree of Conversion
<b>DMEM</b>	Dulbecco's modified Eagle's medium
<b>Exp-Cont</b>	Experimental control
<b>FT-IR</b>	Fourier Transform Infrared Spectroscopy
<b>g</b>	Gram
<b>GPa</b>	Giga Pascal
<b>HEMA</b>	2- hydroxyethyl methacrylate
<b>HQ</b>	Hydroquinone
<b>ISO</b>	International organization for standardization.
<b>MDI</b>	Polymeric methylene diphenyl diisocyanate
<b>LDH</b>	Lactate dehydrogenates
<b>ml</b>	Milliliter
<b>mm</b>	Millimeter
<b>μg/mm<sup>3</sup></b>	Micrograms per cubic millimeter
<b>MPa</b>	Mega Pascal
<b>Mw</b>	Molecular weight
<b>mW/cm<sup>2</sup></b>	Milliwatts per square centimeter

<b>NaCl</b>	Sodium chloride
<b>PBS</b>	Phosphate buffered saline
<b>rpm</b>	round per minute
<b>SOC</b>	spiro orthocarbonate
<b>SD</b>	Standard deviation
<b>SPSS</b>	Statistical package for the social science
<b>TEGDMA</b>	Triethylene glycol dimethacrylate
<b>UAM</b>	Urethane acrylate macromer
<b>UDMA</b>	Urethane dimethacrylate
<b>UV</b>	Ultra violet
<b>VPS</b>	Volumetric polymerization shrinkage

**CHAPTER ONE:**

**INTRODUCTION AND OBJECTIVES**

**CHAPTER TWO:**  
**LITERATURE REVIEW**

**CHAPTER THREE:**

**METHODOLOGY**

## **CHAPTER FOUR:**

### **RESULTS**

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