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

DR. FADHEL A.A. ALSANABANI

DEPARTMENT OF CONSERVATIVE DENTISTRY

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DR. FADHEL A.A. ALSANABANI

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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.

DR. FADHEL A. A. AL-SANABANI

Date:

SUPERVISOR:

ASSOCIATE PROFESSOR DR. NOOR HAYATY ABU KASIM

DEPARTMENT OF CONSERVATIVE DENTISTRY

FACULTY OF DENTISTRY

CO-SUPERVISOR:

PROFESSOR DR. GAN SENG NEON

DEPARTMENT OF CHEMISTRY

FACULTY OF SCIENCE

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ABBREVIATIONS

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

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

CHAPTER ONE:

INTRODUCTION AND OBJECTIVES

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LITERATURE REVIEW

CHAPTER THREE:

METHODOLOGY

CHAPTER FOUR:

RESULTS

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CONCLUSIONS AND SUGGESTIONS

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