#### Appendix I

#### Preliminary study

The Table below includes three polyols-based on palm oil, FA35, AlkOA65N, and AlkOA40N, were used in the preliminary study.

POLYOL	OH-V	Equivalent weight	Molecular weight
FA35	181	309.9	930
AlkOA65N	146	397.9	1194
AlkOA40N	140	400.7	1202

During preparation of urethane polymer, one equivalent weight (133.3 g) of methylene diisocyanate needs one equivalent of polyol-based on palm oil, such as 309.9 g of the FA35 or 397.9 g of AKLOA65 or 400.7 g of AlkO40N to produce polyurethane polymer at room temperature. However, polyol AlkOA40N was very viscous compared to FA35 and AKLOA65, which created a sophisticated procedure for urethane polymer production at room temperature, such as solvent and catalyst addition. The solvent evaporation completely and catalyst cytotoxicity will be issued.

The urethane based on AlkO65N polyol was selected to be used as prepolymer for experimental urethane dimethacrylate monomer (UDMA-M). The selections of AlkO65N polyol are related to the following:

-The strength of urethane based on AlkO65N polyol showed better than that of urethane polymer based on FA35 that contain lower reactivity (OH/V =181) and aliphatic fumeric acid, however, the former contain higher (OH/V = 146) and rigid aromatic groups of phatalic anhydride;

-Moreover, the colour of urethane based on AlkO65N polyol was quite reasonable compared to yellow-brownish colour of urethane polymer based on FA35.

### Appendix II

Properties of polyol alkyd (AlkOA65N)



Cognis Chemicals (Malaysia) Sdn Bhd (Co.No. 17069-T) Cognis Rika (M) Sdn Bhd (Co.No. 93282-T)

**Analysis Report** 

Date : 20/11/2006

#### Subject: Polyol Alkyd (AlkOA65N)

B/N	Polyol Alkyd AlkOA65N		
Raw	%		
materials/Furmulation			
Oleic Acid (OL 72	65.06		
0410447)			
Phthalic Anhydride	14.43		
(Merck)			
Refine Glycerine 99.5%	20.51		
Analysis	Polyol Alkyd	Expected	Method
	AlkOA65N	Result	
Preparation, hours	5	-	
Appearance	Pale Yellow	PaleYellow	Visual
IV	66.5	-	AOCS Tg 1-64 :1997
AV, mg KOH/g	13.2	10 - 20	AOCSTe1a-64 : 1997
OH-V, mg KOH/g	146.6	130 - 140	AOCS Cd 13-60 : 1997
Viscosity @ 25°C,	980	-	AOCS Ja10-87 : 1997
mPas			
Color Lovibond 1" cell	0.2R 1.5Y	-	AOCS Cc13e-92 : 1997
Dry Residue % @	99.23	-	In-hse COM CS05
130°C			
Water, Karl Fischer %	0.10	-	ISO 760-1978 (E)

# Appendix III

# Tabulated water density

	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
20	0.998	0.998	0.998	0.998	0.998	0.998	0.998	0.998	0.998	0.998
	203	183	162	141	120	099	078	056	035	013
21	0.997	0.997	0.997	0.997	0.997	0.997	0.997	0.997	0.997	0.997
	992	970	948	926	904	882	860	837	815	792
22	0.997	0.997	0.997	0.997	0.997	0.997	0.997	0.997	0.997	0.997
	770	747	724	701	678	655	632	608	585	561
23	0.997	0.997	0.997	0.997	0.997	0.997	0.997	0.997	0.997	0.997
	538	514	490	466	442	418	394	369	345	320
24	0.997	0.997	0.997	0.997	0.997	0.997	0.997	0.997	0.997	0.997
	296	271	246	221	196	171	146	120	095	069
25	0.997	0.997	0.996	0.996	0.996	0.996	0.996	0.996	0.996	0.996
	044	018	992	967	941	914	888	862	836	809
26	0.996	0.996	0.996	0.996	0.996	0.996	0.996	0.996	0.996	0.996
	783	756	729	703	676	649	621	594	567	540
27	0.996	0.996	0.996	0.996	0.996	0.996	0.996	0.996	0.996	0.996
	512	485	457	429	401	373	345	317	289	261
28	0.996	0.996	0.996	0.996	0.996	0.996	0.996	0.996	0.996	0.995
	232	204	175	147	118	089	060	031	002	973
29	0.995	0.995	0.995	0.995	0.995	0.995	0.995	0.995	0.995	0.995
	944	914	885	855	826	796	766	736	706	676
30	0.995	0.995	0.995	0.995	0.995	0.995	0.995	0.995	0.995	0.995
	646	616	586	555	525	494	464	433	402	371
	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9

Density of Water (g/cm<sup>3</sup>) at Temperatures from 20°C (liquid state) to 30°C by 0.1°C inc.

# Appendix IV

Properties of fillers and monomers (Esstech. Materials data sheet)

# Properties of filler

Filler	Properties
Туре	Silanated barium borosilica glasses
Size of filler (%)	0.5 µm (10%) and 1.4 µm (90%)
Silane coupling agent	3-(methacryloyloxypropyl) trimethoxysilane (MPTMS)
Solubility in water	Insoluble
Specific gravity	2.2-3.6
appearance	Fine white powder

### Properties of monomers

Materials	Viscosity (cp)	Specific gravity
Bis-GMA	484.000 (300.000-1.100.000)	1.14 (1.12-1.16)
TEGDMA	(5-30)	1.072 (1.065-1.075)
Bis-EMA	836 (800-1200)	1.122 (1.118-1.124)

#### Appendix V

# CellTiter 96® Aqueous One Solution Cell Proliferation reagent composition and properties

Promega currently offers several systems to non-radioactively monitor cell proliferation and cytotoxicity. A new system, the CellTiter 96® AQueous One Solution Cell Proliferation Assay, was recently introduced as a more convenient alternative to the existing CellTiter 96® AQueous Systems. The CellTiter 96® Assay is a nonradioactive, colorimetric assay for measuring the number of viable cells in proliferation, attachment and agent-mediated cytotoxicity assays. Both adherent and suspension cells may be analyzed with this system. The Dye Solution, containing the tetrazolium salt MTT, is added to the cells and is internalized and reduced into an insoluble blue formazan product by cellular metabolism. Only those cells which are living at the time the dye is added will significantly reduce the MTT. The Solubilization Solution is added to lyse the cells and dissolve the formazan dye product. The samples are then read in a 96 well plate reader at 570nm. The intensity of the blue color that appears is directly proportional to the number of viable cells (Rhodes, 1996).

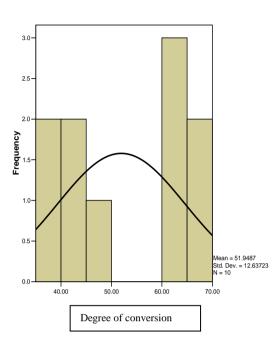
The CellTiter 96<sup>®</sup> AQueous Assay can be used for the same applications as the CellTiter 96<sup>®</sup> Assay. Both systems measure the conversion of a tetrazolium salt into a colored formazan product by the metabolic activity of living cells. The main difference between the two systems is that the CellTiter 96<sup>®</sup> AQueous Assay utilizes MTS\* rather than MTT as the tetrazolium reagent. The system also includes PMS, an electron coupling reagent, which facilitates the reduction of MTS. During the assay, MTS is converted into a soluble formazan product, eliminating the need for addition of Solubilization Solution. After incubating the samples for 1-4 hours, they are quantitated using a 96 well plate reader at 490nm. Since the final product is soluble in culture

medium, samples may be returned to the incubator for further color development if desired (unlike the CellTiter® 96 Assay System) (Rhodes, 1996).

The CellTiter 96<sup>®</sup> AQueous Assay includes the electron carrier phenazine methosulfate (PMS), which mediates the reduction of the tetrazolium salt MTS. The MTS and PMS solutions must be mixed prior to the addition of the dye solution to the cell culture medium. The CellTiter 96<sup>®</sup> AQueous One Solution Assay contains a single solution of MTS and phenazine ethosulfate (PES). The PES component is an alternative electron carrier which is more stable in solution than PMS. The solution containing MTS and PES is supplied pre-mixed, sterile and ready to add to cell culture medium. The formazan product of the CellTiter 96<sup>®</sup> AQueous One Solution Assay is also soluble in tissue culture medium. Comparing the relationship of cell number to color formation, the performance characteristics of these two systems are nearly identical (Rhodes, 1996).

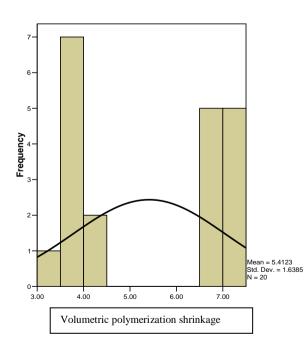
# Appendix VI

Histogram with normal distributed curve and Shapiro-Wilk test of experimental resins



Shapiro-Wilk				
Statistic	df	Sig.		
.837	10	.041		

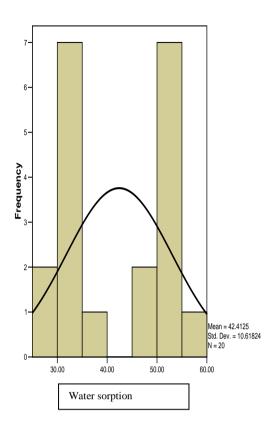
# b) Volumetric polymerization shrinkage



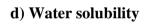
Shapiro-Wilk			
Statistic df Sig.			
.762	20	.000	

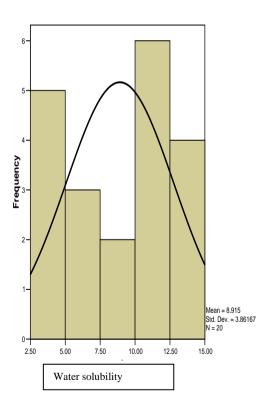
### a) Degree of conversion

# c) Water sorption



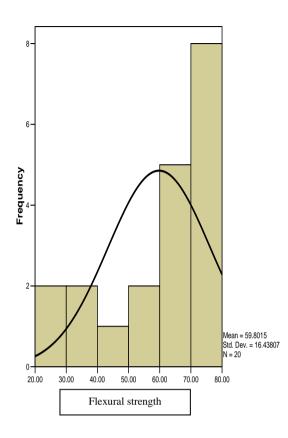
Shapiro-Wilk				
Statistic df Sig.				
.836	20	.003		





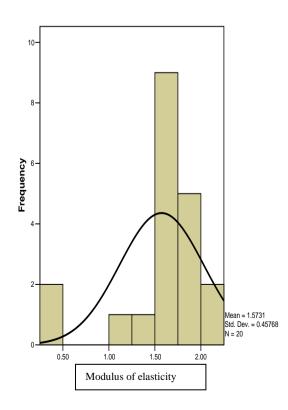
Shapiro-Wilk				
Statistic df Sig.				
.923	20	.115		

# e) Flexural strength



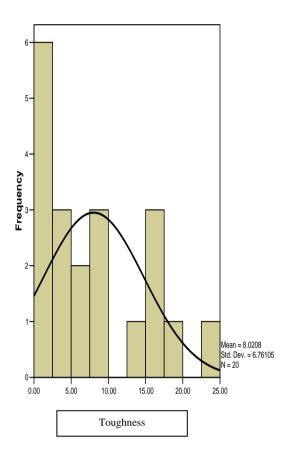
Shapiro-Wilk				
Statistic df Sig.				
.848	20	.005		

f) Modulus of elasticity



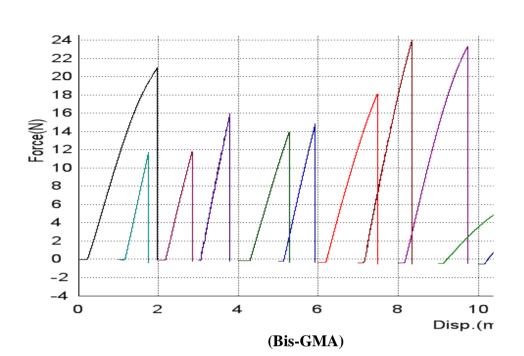
Shapiro-Wilk			
Statistic df Sig.			
.822	20	.002	

# g) Toughness

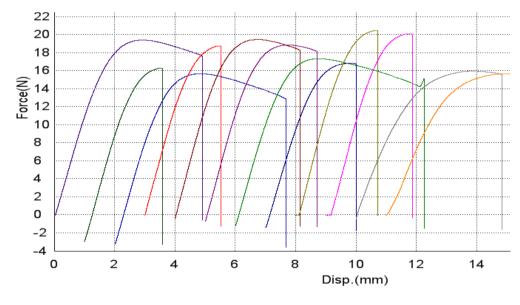


Shapiro-Wilk			
Statistic df Sig.			
.890	20	.027	

# Appendix VII



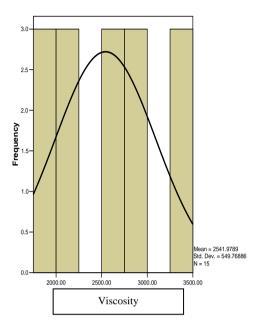
Stress/strain curve showed the toughness of Bis-GMA and UAM



(UAM)

# Appendix VIII

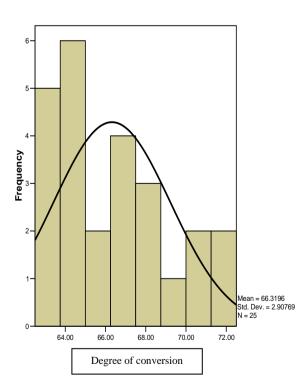
Histogram with normal distributed curve and Shapiro-Wilk test of experimental resin systems



a) Viscosity

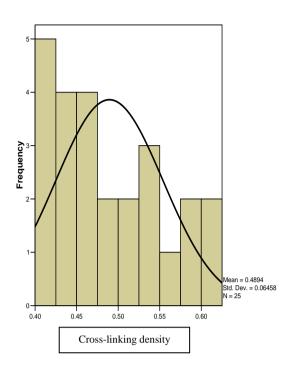
Shapiro-Wilk						
Statistic df Sig.						
.902	15	.103				

# b) Degree of conversion



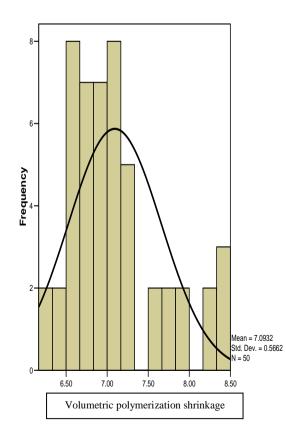
Shapiro-Wilk					
Statistic df Sig.					
.898	25	.017			

# c) Cross-linking density



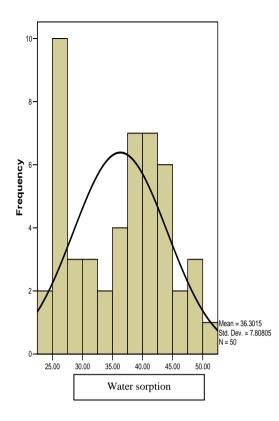
Shapiro-Wilk				
Statistic df Sig.				
.906	25	.025		

# d) Volumetric polymerization shrinkage

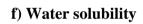


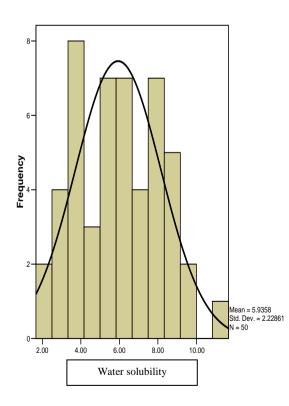
Shapiro-Wilk					
Statistic df Sig.					
.903	50	< .001			

# e) Water sorption



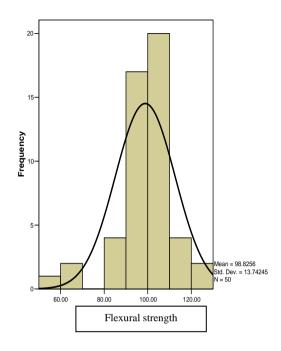
Shapiro-Wilk				
Statistic df Sig.				
.942	50	.016		





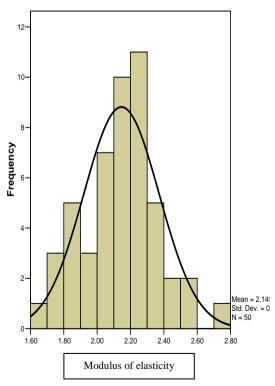
Shapiro-Wilk					
Statistic df Sig.					
.974	50	.339			

# g) Flexural strength



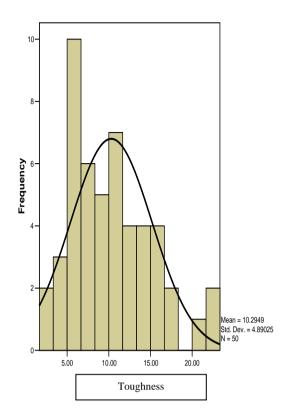
Shapiro-Wilk					
Statistic df Sig.					
.926	50	.004			

h) Modulus of elasticity



Shapiro-Wilk				
Statistic df Sig.				
.983	.668			

# i) Toughness



Shapiro-Wilk				
Statistic df Sig.				
.951	50	.038		

#### **Appendix IX**

#### Homogeneity of variances test

Levene Statistic	df1	df2	Sig.
2.294	4	10	.131

a) Test of homogeneity of variances of viscosity for experimental resin systems

**b**) Test of homogeneity of variances of water solubility for experimental resin systems

Levene statistic	df1	df2	Sig.
.962	4	45	.438

c) Test of homogeneity of variances of water solubility for experimental resin systems

Levene statistic	df1	df2	Sig.
4.075	4	45	.007

d) Test of homogeneity of variances of volumetric change for flowable composites

Levene statistic	df1	df2	Sig.
2.994	4	45	.028

\_

\_

e) Test of homogeneity of variances of water solubility for flowable composites

Levene statistic	df1	df2	Sig.
1.024	4	45	.405

	Levene Statistic	df1	df2	Sig.
Flexural strength	1.861	4	45	.172
Toughness	2.191	4	45	.085

f) Test of homogeneity of variances of flexural strength and toughness for flowable composites

# g) Test of homogeneity of variances of viable cells for flowable composites

Levene statistic	df1	df2	Sig.
1.609	5	48	.176

#### Appendix X

Calculations of familywise error and familywise alpha value

Familywise error (FWE) represents the probability that any one of a set of comparisons or significance is a Type I error. The FWE can be estimated with the following formula:

$$\propto FWE \leq 1 - (1 - \propto EC)c$$

where by

Alpha<sub>FEW</sub> = familywise error rate  $\propto EC$  = alpha rate for an individual test (.05) <sup>C</sup> = exponent (where C is the total number of pairwise comparisons) In this study, the calculation is shown as:

$$\propto FWE \leq 1 - (1 - .5)10$$

$$\propto FWE \leq 1 - (.599)$$

 $\propto FWE \leq .40$ 

The Bonferroni simply calculates a new pairwise alpha to keep the familywise alpha value at .05 (or another specified value). The formula for doing this is as follows:

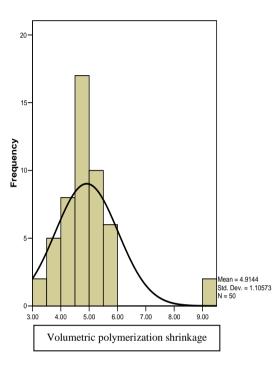
$$\alpha_{B} = \frac{\alpha_{FWE}}{c}$$

where  $\alpha_{\rm B}$  is the new alpha based on the Bonferroni test that should be used to evaluate each comparison or significance test,  $\alpha_{\rm FWE}$  is the familywise error rate as computed in the first formula, and *c* is the number of comparisons (statistical tests).

Therefore the bonferroni of alpha in this study is .04:

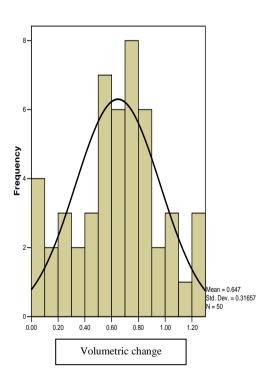
# Appendix XI

Histogram with normal distributed curve and Shapiro-Wilk test of flowable composites



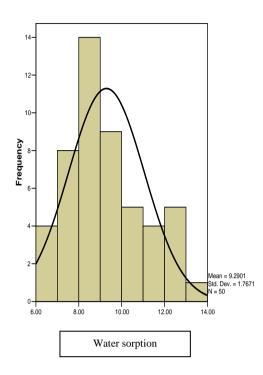
Shapiro-Wilk			
Statistic df Sig.			
.771	50	< .001	

b) Volumetric change



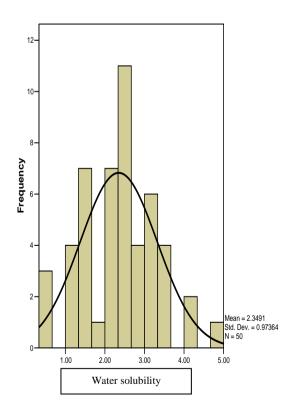
Shapiro-Wilk			
Statistic	df	Sig.	
.972	50	.289	

# c) Water sorption

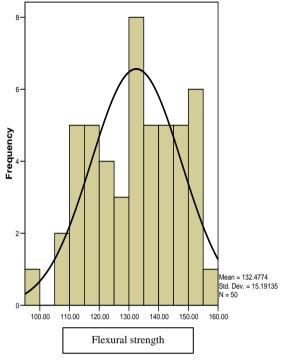


Shapiro-Wilk			
Statistic	e df	Sig.	
.942	50	.016	

# d) Water solubility

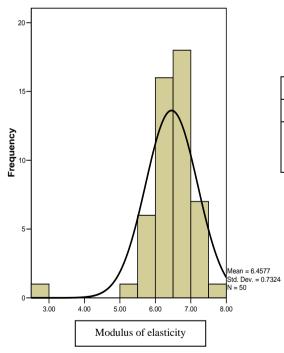


Shapiro-Wilk			
Statistic df Sig.			
.982	50	.658	



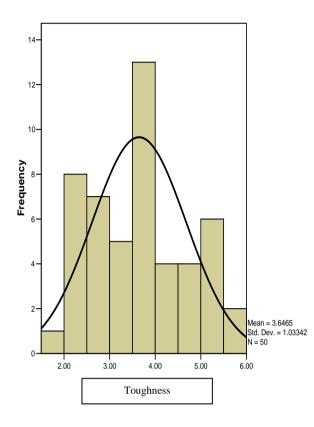
Shapiro-Wilk			
Statistic	df	Sig.	
.969	50	.220	

# f) Modulus of elasticity



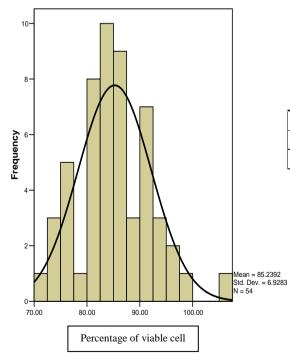
Shapiro-Wilk		
Statistic	df	Sig.
.736	50	.000

# g) Toughness



Shapiro-Wilk			
Statistic	df	Sig.	
.956	50	.061	

h) Percentage of viable cell



Shapiro-Wilk		
Statistic	df	Sig.
.976	54	.354

# Appendix XII

Esthet.X flow liquid micro hybrid flowable composite composition and properties (Dentsply Caulk data sheet)

### Composition and properties of Esthet.X flow flowable composite

Resin	Bis-GMA/TEGDMA	
	Barium fluoroboroaluminosilicate glass and silica nanofiller.	
Type, Size and % filler	The particles size range is 0.01 to 5 $\mu$ m and average approximately 0.9 $\mu$ m.	
	The filler load approximately 61 % by weight	
Flexural strength	112 MPa	
Modulus of elasticity	5.622	
Fluoride release	From ~ 50 $\mu$ g/gm in the first week to 200 $\mu$ g/gm in the twenty fifth week in de-ionized water.	

#### Appendix XIII

#### Contributions of this study

#### **Published contributions**

- Radzi, Z., Abu Kasim, NH., Yahya, NA., Gan, SN, A. Fadhel. (2007). Impact strength of an experimental polyurethane-based polymer. Annal Dent Univ Malaya; 14: 46–51.

- Al sanabani, F., Abu Kasim, N. H., Gan, S.N. (2008). Conversion of a new monomer for dental resin application. Abstract; International Association Dental Research (IADR), Manila-Philippine, 8-10 October.

- Al sanabani, F., Abu Kasim, N. H., Gan, S.N. (2009). Mechanical properties of BP-UDMA and its copolymer for dental resin. Abstract; International Association Dental Research (IADR), Wuhan-China, 22-24September.
- Al sanabani, F., Abu Kasim, N. H., Gan, S.N. (2010). Sorption and solubility of BP-UDMA-based resins for dental composite. Abstract; International Association Dental Research (IADR), UKM-Malaysia, 5-6 February.
- Al sanabani, F., Abu Kasim, N. H., Gan, S.N. (2010). Conversion and mechanical properties of dental resin system base don a new monomer derived from palm oil-based polyol. international Conference on Functional Materials & Devices (ICFMD), Terengganu-Malaysia, 13-17 June.
- Abu Kasim, N. H., Al-sanabani, F., Muhamed, S. and Gan, S.N. (2010). Cytotoxicity of polyurethane dimethacrylate derived from palm oil. Abstract; International Association Dental Research-IADR, Barcelona-Spain, 14-17 July.

#### **Conferences and Exhibitions**

- Gan, SN., Abu Kasim, N. H., Al-sanabani, F. (2008). Development of polyurethane oligomer derived from palm oil polyol for application in restorative dentistry. Malaysian Technology Exhibition (MTE), Silver Medal. Kuala Lumpur-Malaysia, 21-23 February.
- Abu Kasim, N. H., Gan, SN., Al-sanabani, F. (2009). A novel resin system based on palm oil polyol for dental composites. Malaysian Technology Exhibition (MTE), Silver Medal. Kuala Lumpur-Malaysia, 19-21 February.
- Abu Kasim, N. H., Gan, SN., Al-sanabani, F. (2009). A novel polymer for restorative dentistry. University of Malaya EXPO (UMEXPO), Gold Medal. Kuala Lumpur-Malaysia, 13-15 January.
- Abu Kasim, N. H., Gan, S.N., Al sanabani, F. (2008). Properties of novel polyurethane dimethacrylate based on palm oil for dental application. Invention

& New Product Exposition (INPEX). Gold Medal (Therapeutic Category). Pittsburg-USA, 11-14 June.

### Patent

- Polyurethane oligomers for use in restorative dentistry. Malaysia Patent Application; PI 20092415. Inventors; Gan Seng Neon, Noor Hayaty Abu Kasim, Fadhel Alsanabani, Zamri Radzi, and Noor Azlin Yahya.