

## Appendix I

### Preliminary study

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

<b>POLYOL</b>	<b>OH-V</b>	<b>Equivalent weight</b>	<b>Molecular weight</b>
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)



TG15-

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 0410447)	65.06		
Phthalic Anhydride (Merck)	14.43		
Refine Glycerine 99.5%	20.51		
Analysis	Polyol Alkyd AlkOA65N	Expected Result	Method
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 , mPas	980	-	AOCS Ja10-87 : 1997
Color Lovibond 1" cell	0.2R 1.5Y	-	AOCS Cc13e-92 : 1997
Dry Residue % @ 130°C	99.23	-	In-hse COM CS05
Water , Karl Fischer %	0.10	-	ISO 760-1978 (E)

### Appendix III

#### Tabulated water density

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

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

## Appendix IV

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

### Properties of filler

<b>Filler</b>	<b>Properties</b>
Type	Silanated barium borosilica glasses
Size of filler (%)	0.5 $\mu\text{m}$ (10%) and 1.4 $\mu\text{m}$ (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

<b>Materials</b>	<b>Viscosity (cp)</b>	<b>Specific gravity</b>
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 non-radioactive, 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® AQueous Assay can be used for the same applications as the CellTiter 96® 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® 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

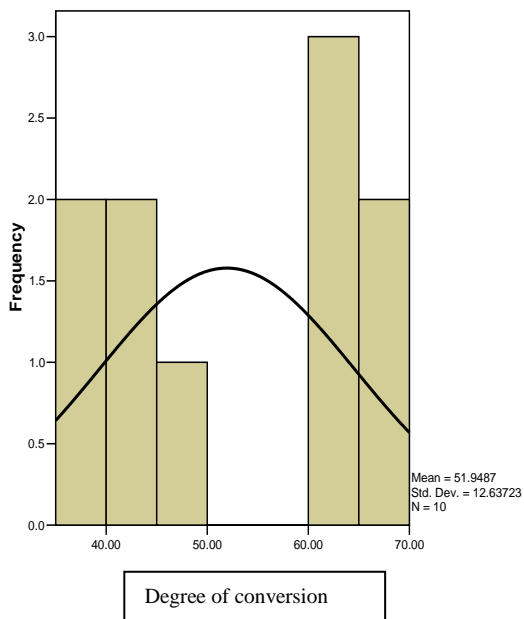
medium, samples may be returned to the incubator for further color development if desired (unlike the CellTiter® 96 Assay System) (Rhodes, 1996).

The CellTiter 96® 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® 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® 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

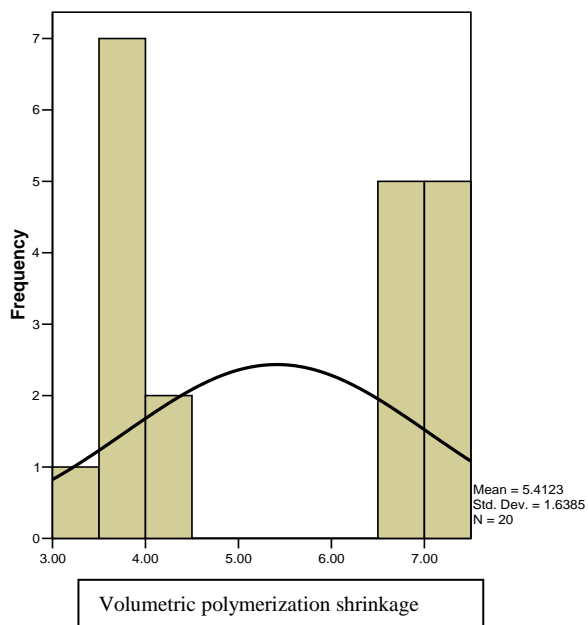
Histogram with normal distributed curve and Shapiro-Wilk test of experimental resins

### a) Degree of conversion



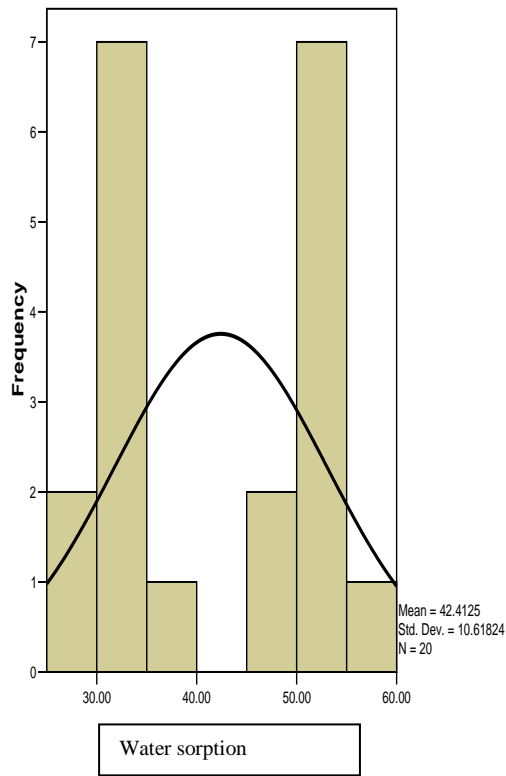
Shapiro-Wilk		
Statistic	df	Sig.
.837	10	.041

### b) Volumetric polymerization shrinkage



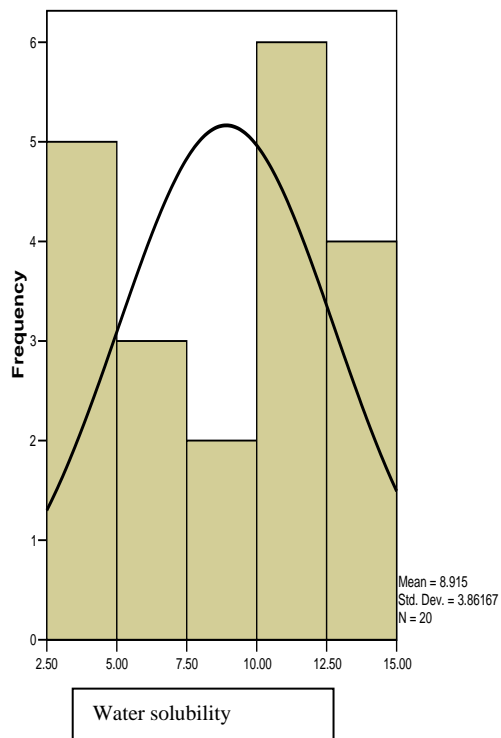
Shapiro-Wilk		
Statistic	df	Sig.
.762	20	.000

### c) Water sorption



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

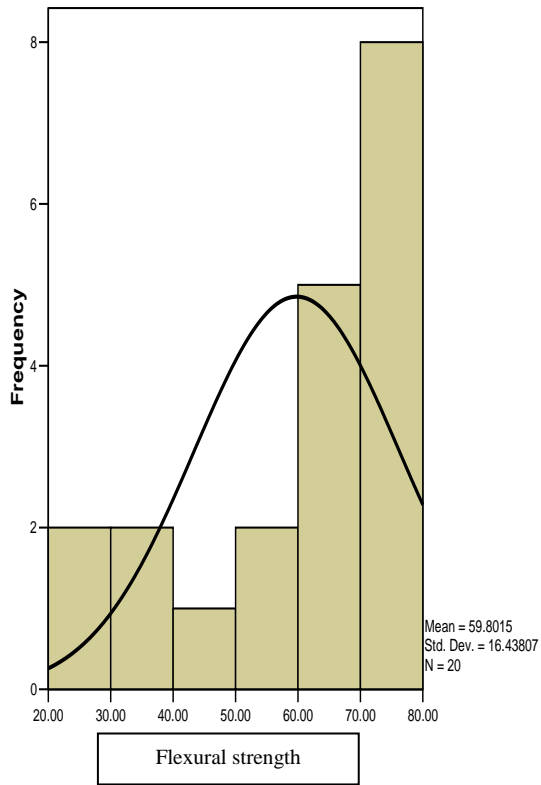
### d) Water solubility



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

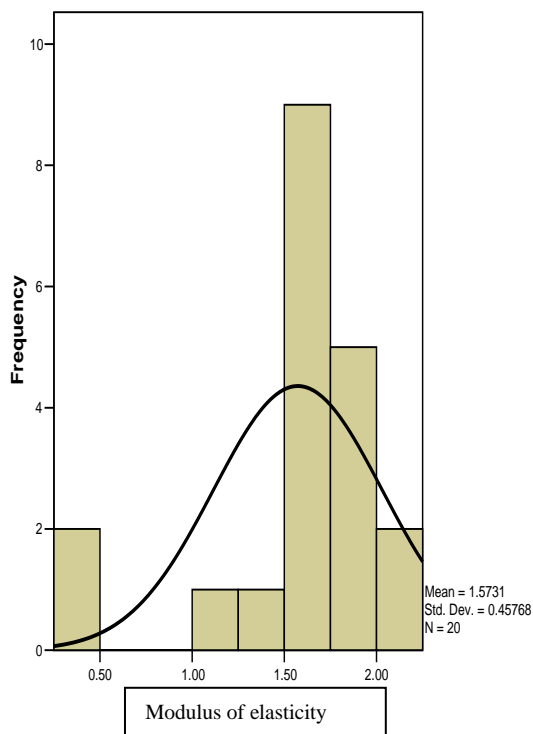


**e) Flexural strength**



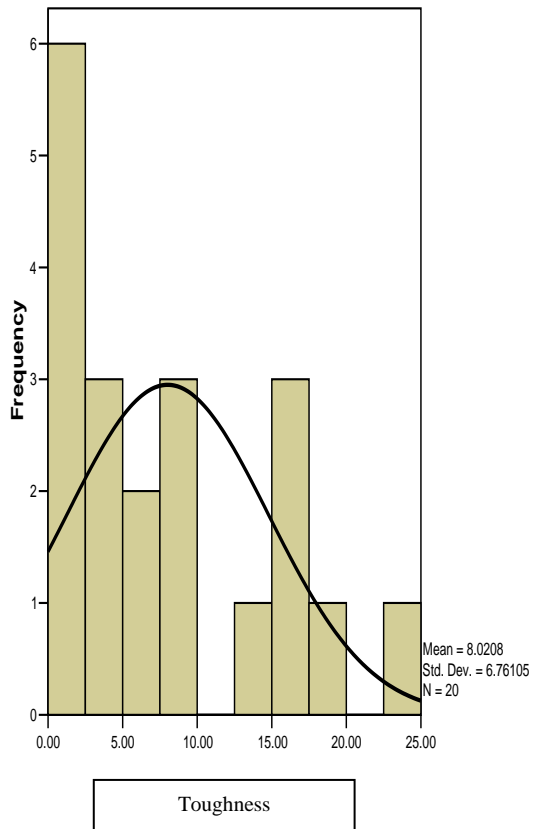
Shapiro-Wilk		
Statistic	df	Sig.
.848	20	.005

**f) Modulus of elasticity**



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

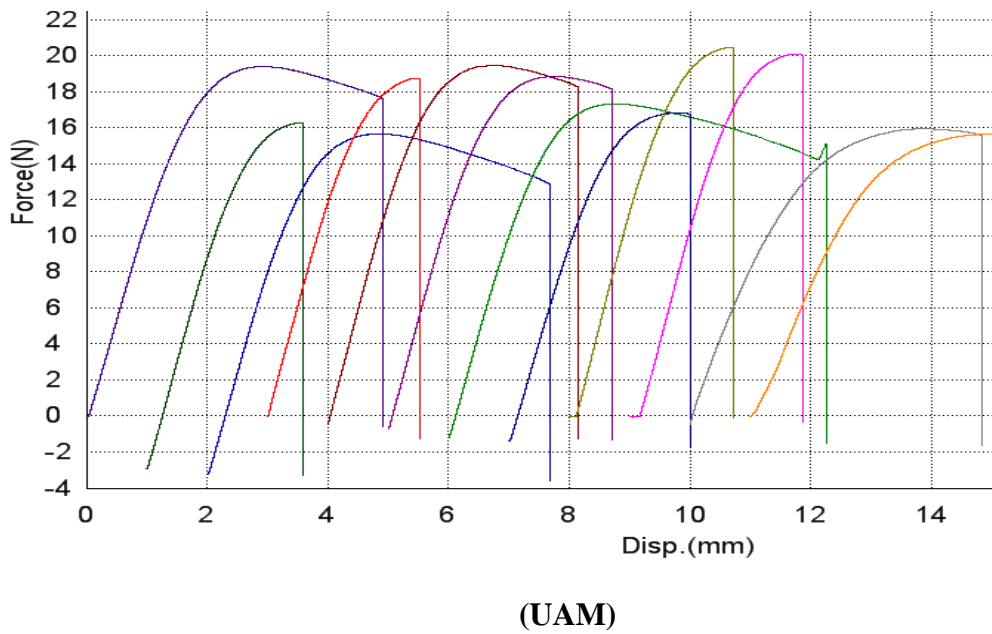
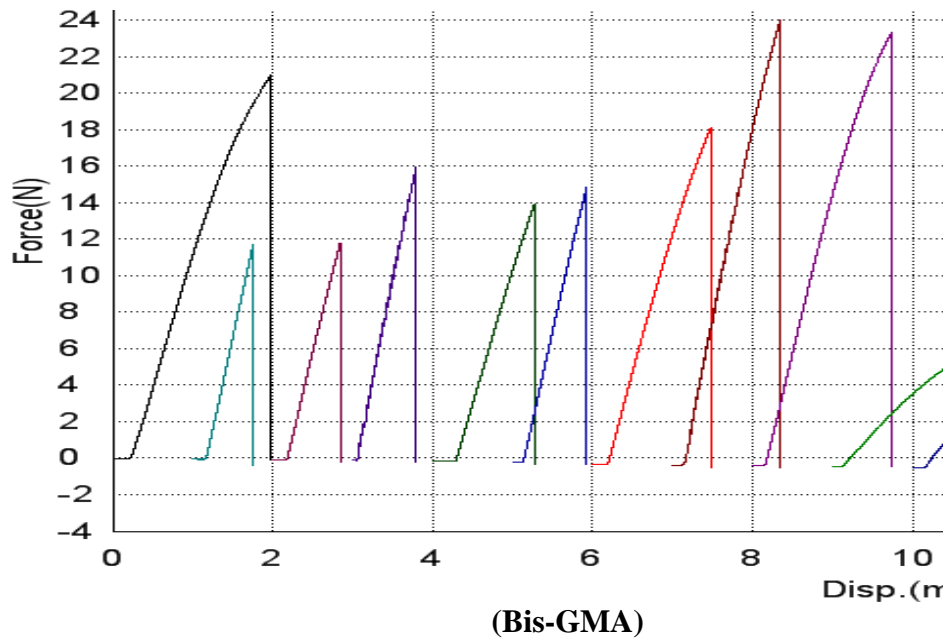
### g) Toughness



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

## Appendix VII

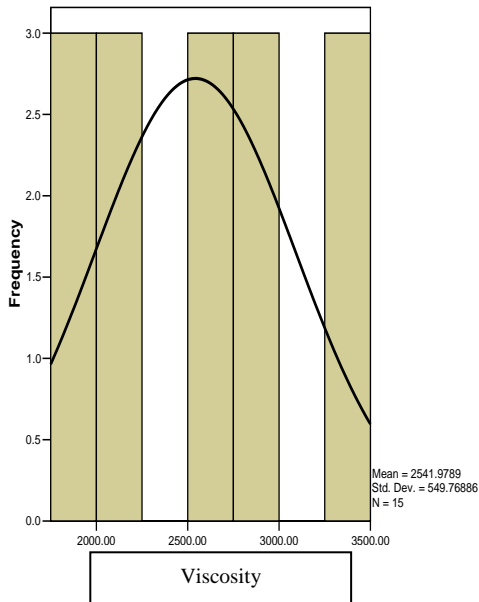
Stress/strain curve showed the toughness of Bis-GMA and UAM



## Appendix VIII

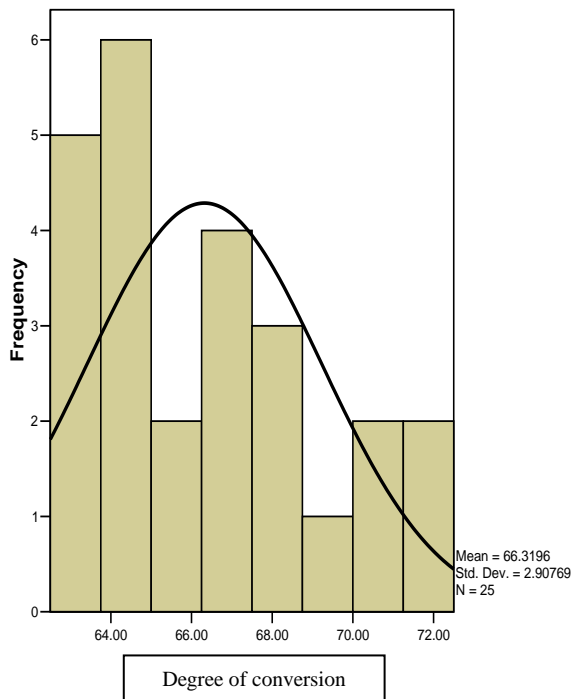
Histogram with normal distributed curve and Shapiro-Wilk test of experimental resin systems

### a) Viscosity



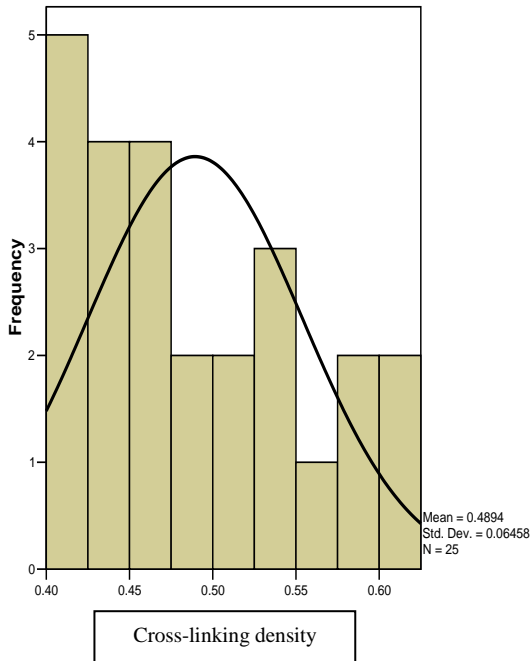
Shapiro-Wilk		
Statistic	df	Sig.
.902	15	.103

### b) Degree of conversion



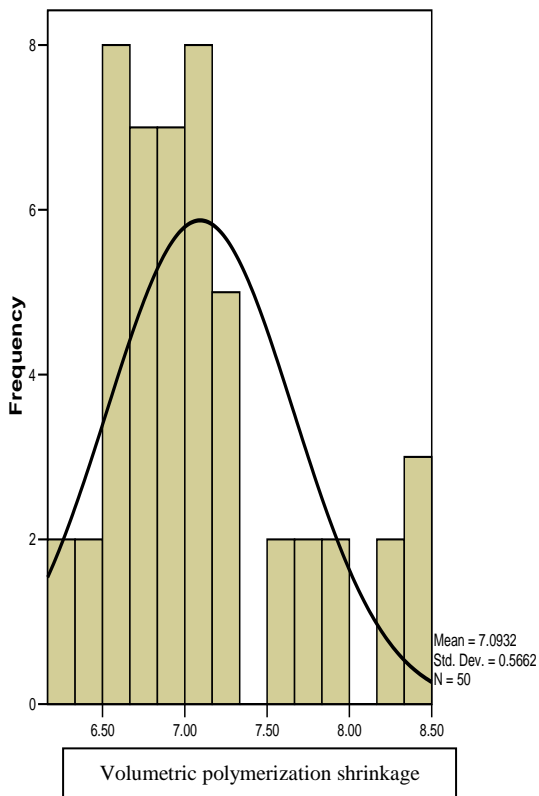
Shapiro-Wilk		
Statistic	df	Sig.
.898	25	.017

**c) Cross-linking density**



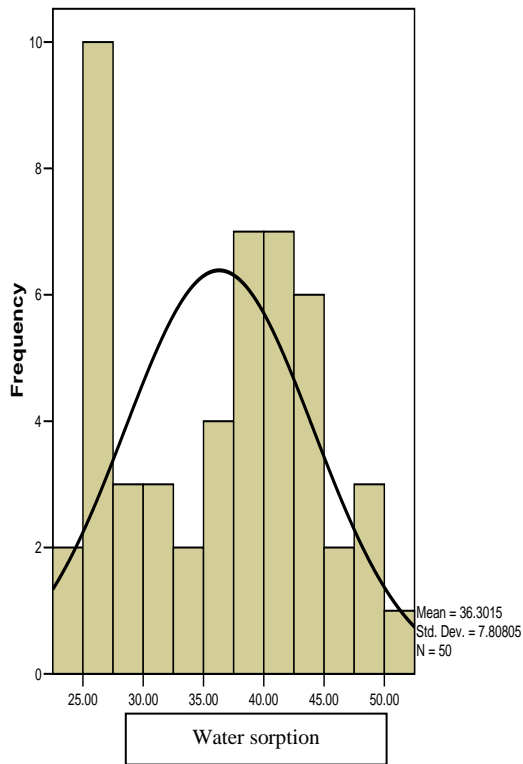
Shapiro-Wilk		
Statistic	df	Sig.
.906	25	.025

**d) Volumetric polymerization shrinkage**



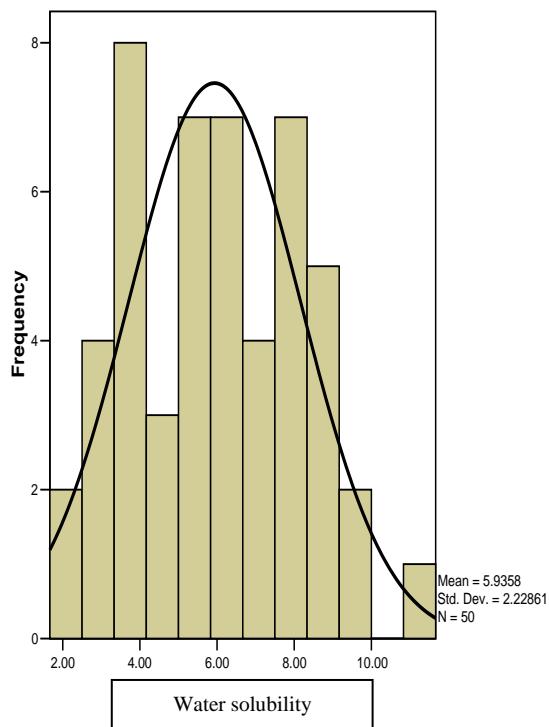
Shapiro-Wilk		
Statistic	df	Sig.
.903	50	< .001

### e) Water sorption



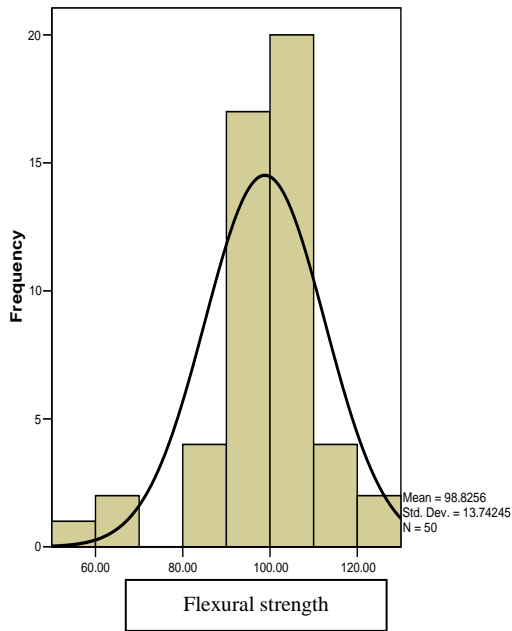
Shapiro-Wilk		
Statistic	df	Sig.
.942	50	.016

### f) Water solubility



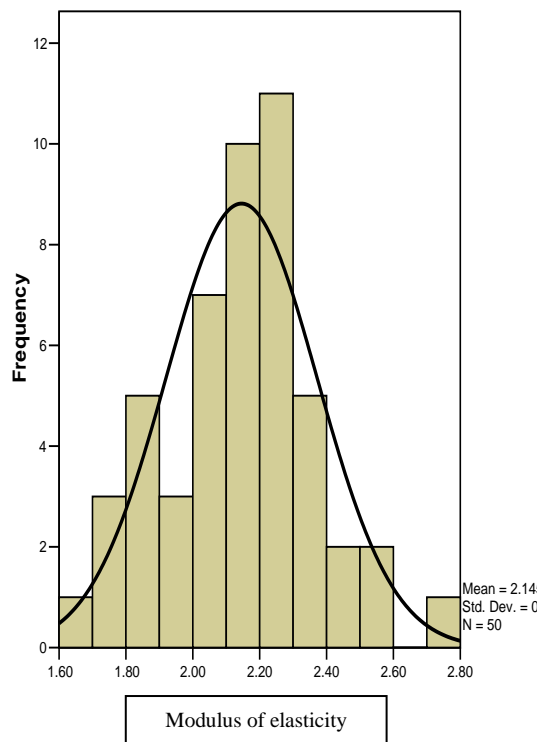
Shapiro-Wilk		
Statistic	df	Sig.
.974	50	.339

### g) Flexural strength



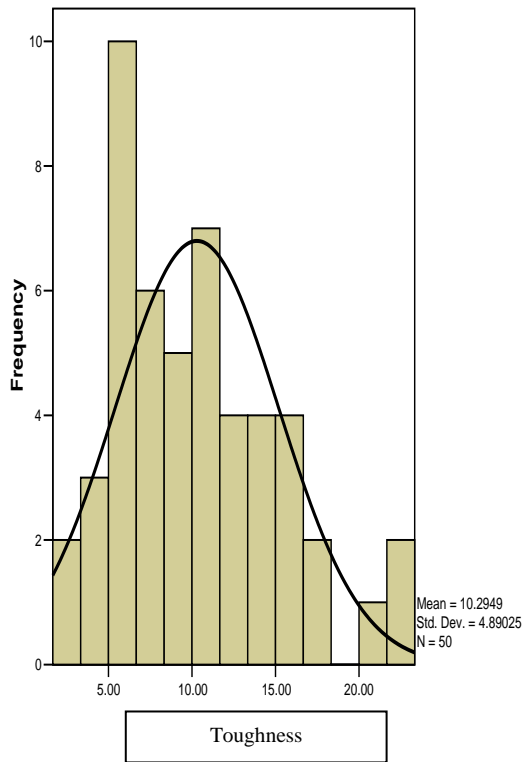
Shapiro-Wilk		
Statistic	df	Sig.
.926	50	.004

### h) Modulus of elasticity



Shapiro-Wilk		
Statistic	df	Sig.
.983	50	.668

### i) Toughness



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



## Appendix IX

### Homogeneity of variances test

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

<b>Levene Statistic</b>	<b>df1</b>	<b>df2</b>	<b>Sig.</b>
2.294	4	10	.131

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

<b>Levene statistic</b>	<b>df1</b>	<b>df2</b>	<b>Sig.</b>
.962	4	45	.438

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

<b>Levene statistic</b>	<b>df1</b>	<b>df2</b>	<b>Sig.</b>
4.075	4	45	.007

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

<b>Levene statistic</b>	<b>df1</b>	<b>df2</b>	<b>Sig.</b>
2.994	4	45	.028

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

<b>Levene statistic</b>	<b>df1</b>	<b>df2</b>	<b>Sig.</b>
1.024	4	45	.405

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

	<b>Levene Statistic</b>	<b>df1</b>	<b>df2</b>	<b>Sig.</b>
Flexural strength	1.861	4	45	.172
Toughness	2.191	4	45	.085

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

<b>Levene statistic</b>	<b>df1</b>	<b>df2</b>	<b>Sig.</b>
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:

$$\alpha_{FWE} \leq 1 - (1 - \alpha_{EC})^c$$

where by

$\alpha_{FWE}$  = familywise error rate

$\alpha_{EC}$  = alpha rate for an individual test (.05)

$c$  = exponent (where C is the total number of pairwise comparisons)

In this study, the calculation is shown as:

$$\alpha_{FWE} \leq 1 - (1 - .05)^{10}$$

$$\alpha_{FWE} \leq 1 - (.599)$$

$$\alpha_{FWE} \leq .40$$

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:

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

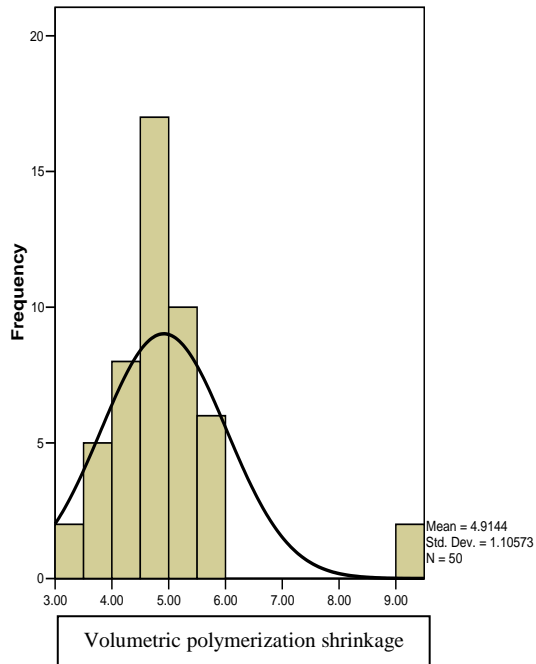
where  $\alpha_B$  is the new alpha based on the Bonferroni test that should be used to evaluate each comparison or significance test,  $\alpha_{FWE}$  is the familywise error rate as computed in the first formula, and  $c$  is the number of comparisons (statistical tests).

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

## Appendix XI

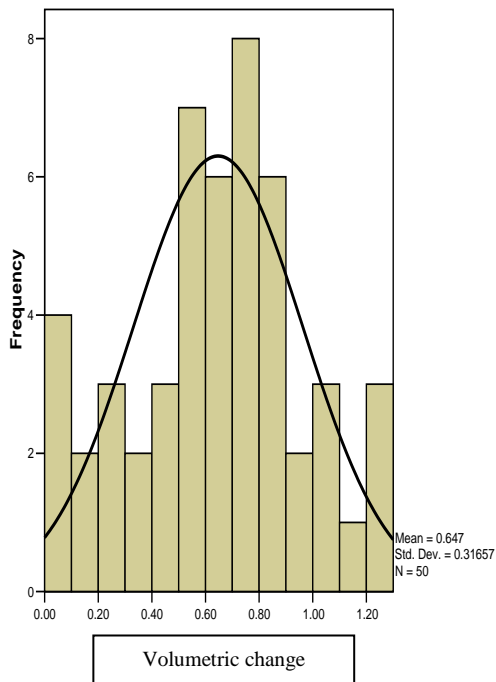
Histogram with normal distributed curve and Shapiro-Wilk test of flowable composites

### a) Volumetric polymerization shrinkage



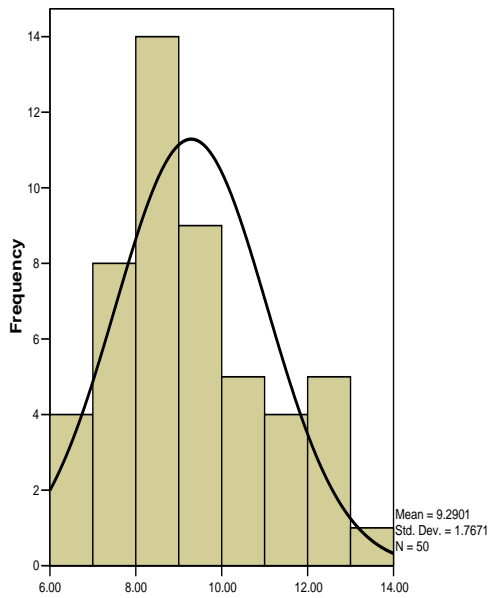
Shapiro-Wilk		
Statistic	df	Sig.
.771	50	< .001

### b) Volumetric change



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

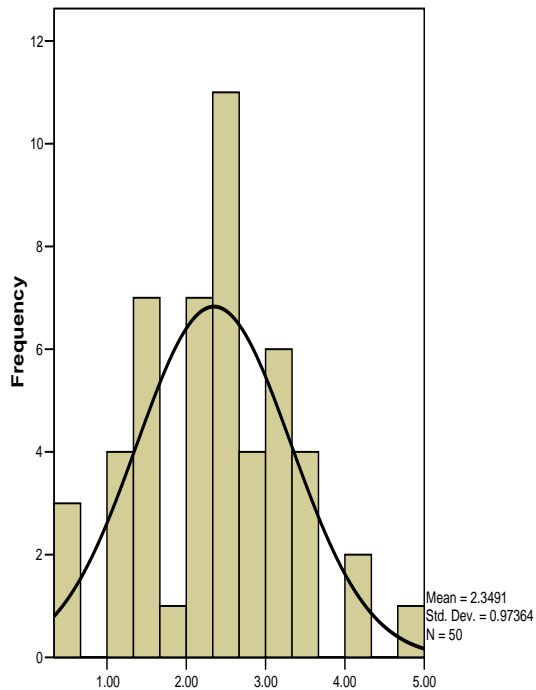
### c) Water sorption



Water sorption

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

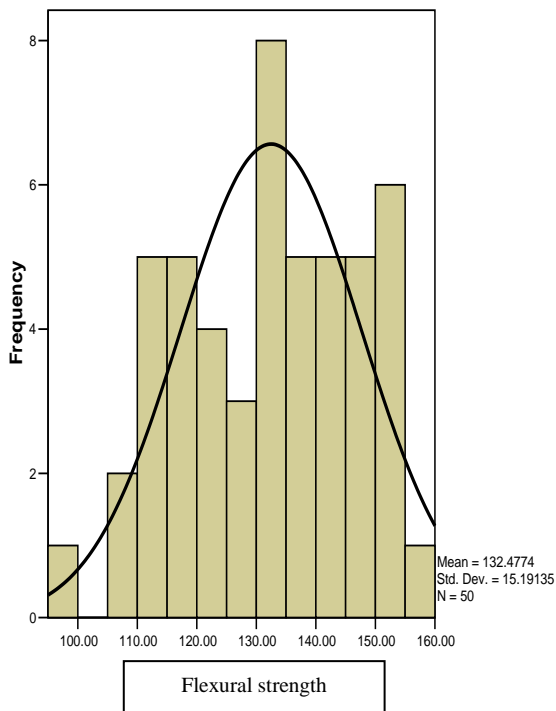
### d) Water solubility



Water solubility

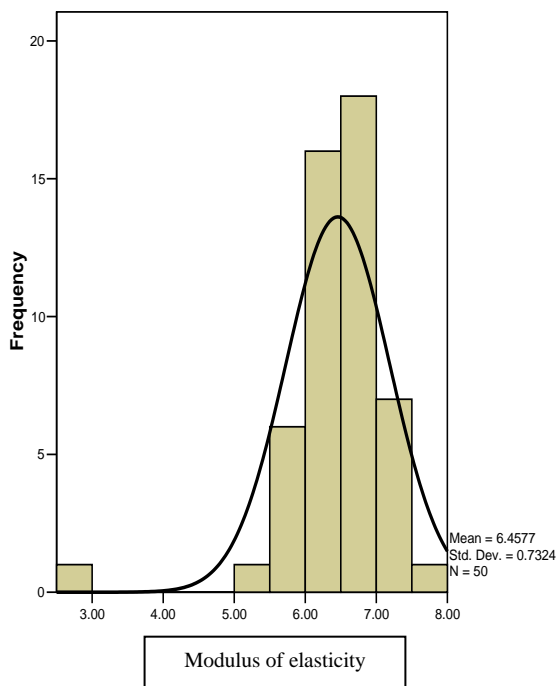
Shapiro-Wilk		
Statistic	df	Sig.
.982	50	.658

**e) Flexural strength**



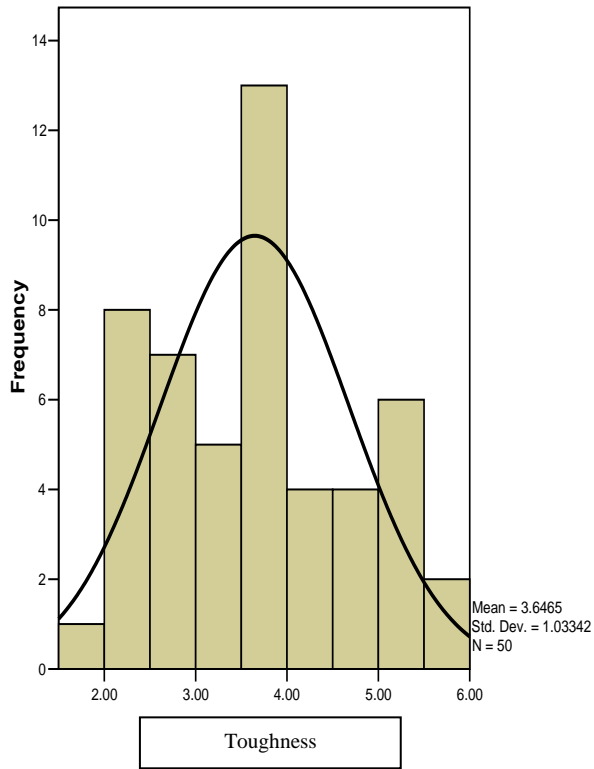
Shapiro-Wilk		
Statistic	df	Sig.
.969	50	.220

**f) Modulus of elasticity**



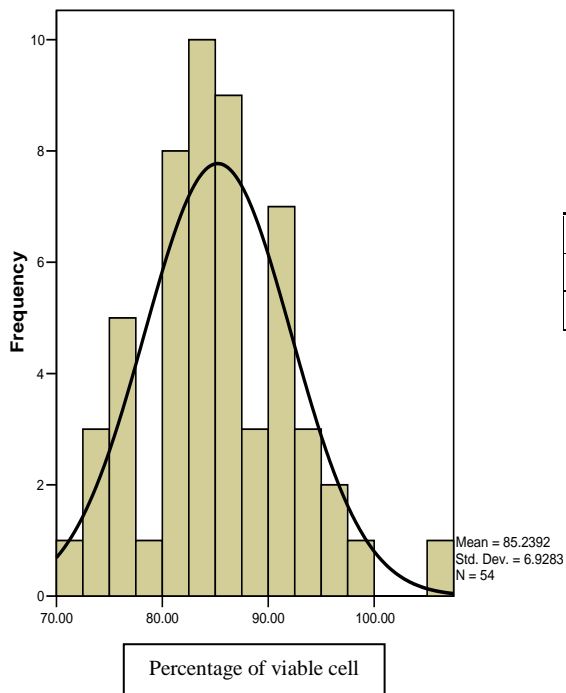
Shapiro-Wilk		
Statistic	df	Sig.
.736	50	.000

### 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

<b>Resin</b>	Bis-GMA/TEGDMA
<b>Type, Size and % filler</b>	Barium fluoroboroaluminosilicate glass and silica nanofiller. The particles size range is 0.01 to 5 µm and average approximately 0.9 µm. The filler load approximately 61 % by weight
<b>Flexural strength</b>	112 MPa
<b>Modulus of elasticity</b>	5.622
<b>Fluoride release</b>	From ~ 50 µg/gm in the first week to 200 µg/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.