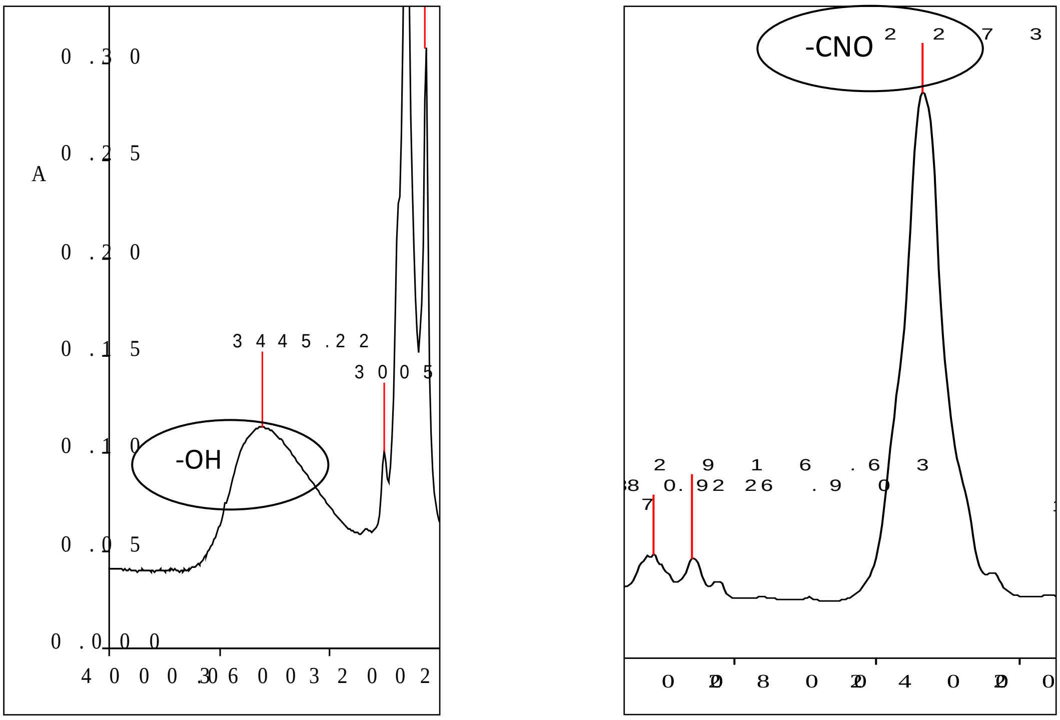
4.1 Identification of experimental urethane acrylate macromer FT-IR spectrum

The FT-IR spectra of alkyd polyol (AlkOA65N) and methylene diphenyl diisocyanate (MDI) showed peaks of hydroxyl group (-OH) at 3445 cm$^{-1.}$ It also showed a sharp peak of the isocyanate group (-CNO) at 2273 cm^{-1} (Figure 4.1). The FT-IR spectrum of experimental urethane acrylate macromer (UAM) did not show the presence of isocyanate groups at 2273 cm^{-1} and hydroxyl groups at 3445 cm^{-1}, while the peaks of urethane groups (-N-H) at 3365 cm^{-1} were observed. The UAM spectrum showed carbonyl groups (-C=O) absorption peaks at 1737cm^{-1}, bending and stretching vibration of the vinyl groups (-C=C-) peaks at 1608 cm^{-1} and 1636 cm^{-1} (Figure 4.2). All these chemical groups indicate the urethane forming reaction, and consequently the UAM was successfully synthesized.



(A) (B)

Figure 4.1 Spectra of starting materials
(A) Alkyd polyol showing peak of hydroxyl groups
(B) Methylene diphenyl diisocyanate showing peak of isocyanate groups

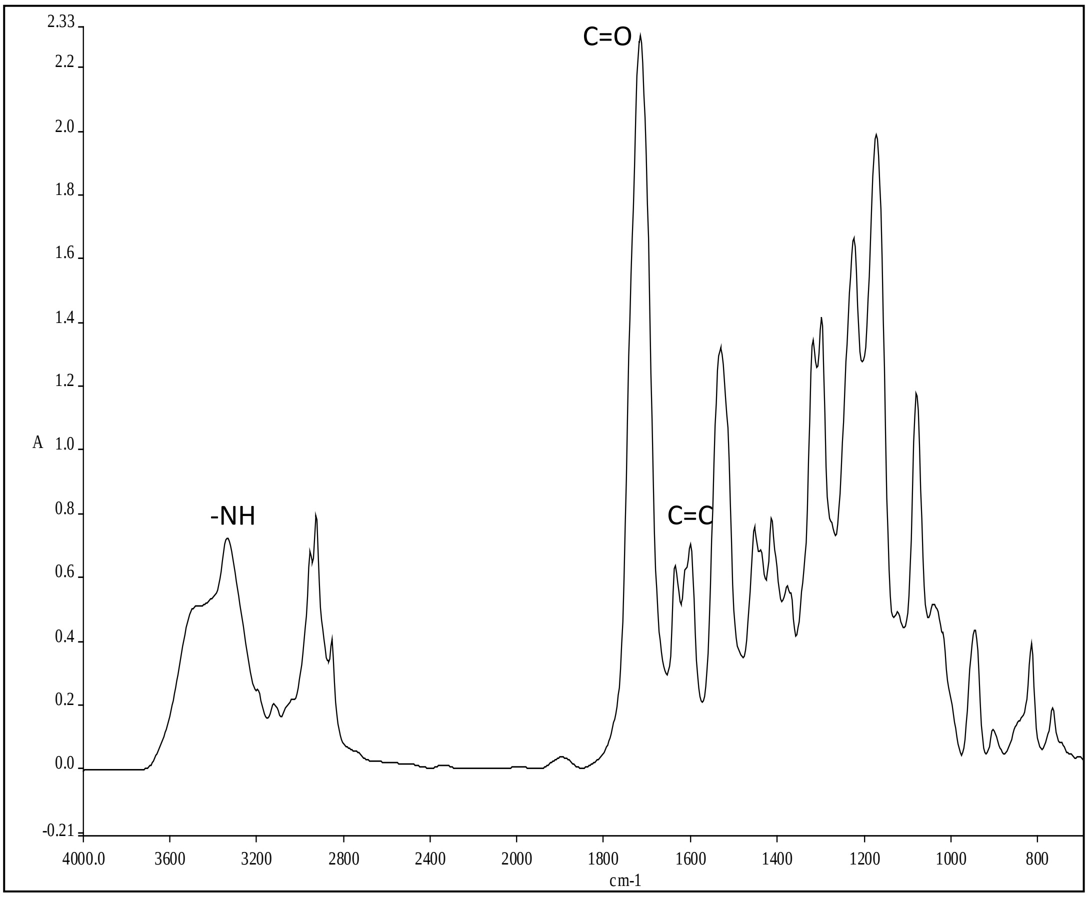


Figure 4.2 Spectrum of UAM illustrating the presence of –NH peak and the absence of -CNO (2273) and –OH (3445) peaks

4.2 Experimental resins

4.2.1 Viscosity

The mean viscosities (cp) of experimental resins determined at 25ºC are shown in Table 4.1. The viscosity mean of Bis-GMA was much higher compared to UAM.

Table 4.1 Viscosity (cp) of uncured experimental resins

Experimental resins	n	Mean (SD) (cp)
Bis-GMA	3	407,350 (51905)
UAM	3	4,453 (217)

4.2.2 Degree of conversion and cross-linking density of experimental resins

The mean percentage of degree of conversion (% DC) of experimental resins UAM and Bis-GMA is shown in Table 4.2. The mean cross-linking density (CLD) value of UAM was 0.42 (±0.07), however, the cross-linking density value of Bis-GMA could not be calculated, due to its degree of conversion being lower than 50%.

Table 4.2 Mean percentage degree of conversion of experimental resins

Experimental resin	n	Mean (SD) (%)
Bis-GMA	5	40.29 (3.25)
UAM	5	63.59 (3.06)

Assumption of normality was checked and results of the preliminary analysis using a histogram indicated that the data does not approximate the normal distribution curve and this was further confirmed by the Shapiro-Wilk normality test showed $p < .05$

(Appendix VIa). The paired comparison Mann-Whitney U test showed that the degree of conversion of UAM was significantly higher than that of Bis-GMA ($p < .05$) as is shown in Table 4.3.

Table 4.3 Comparison of means for degree of conversion between experimental resins

Experimental resin	**n**	**Mean Rank**	**Sum of Ranks**	**Wilcoxon W[a] (Z)**	**p value**
Bis-GMA	5	3.00	15.00	15	.009
UAM	5	8.00	40.00	(-2.611)	

a Mann-Whitney U test at p = .05

4.2.3 Volumetric polymerization shrinkage of experimental resins

Table 4.4 shows the mean percentage of volumetric polymerization shrinkage percentage (% VPS) for experimental resins UAM and Bis-GMA.

Table 4.4 Mean percentage of volumetric polymerization shrinkage of experimental resins

Experimental resins	**n**	**Mean (SD) (%)**
Bis-GMA	10	3.83 (0.20)
UAM	10	6.99 (0.26)

A histogram indicated that the data does not approximate the normal distribution curve and this was further supported by the Shapiro-Wilk normality test showed $p < .05$ (Appendix VIb). The non-parametric Mann-Whitney U test was conducted and it showed that the percentage volumetric polymerization shrinkage of UAM was significantly higher than that of Bis-GMA as is shown in Table 4.5.

Table 4.5 Comparison of mean for volumetric polymerization shrinkage between experimental resins

Experimental resins	n	Mean Rank	Sum of Ranks	Wilcoxon W[a] (Z)	p value
Bis-GMA	10	5.50	55.00	55	< .001
UAM	10	15.50	155.00	(-3.78)	

[a] Mann-Whitney U test at $p = .05$

4.2.4 Water sorption and solubility of experimental resins

Table 4.6 shows the means and standard deviations for water sorption and water solubility ($\mu g/mm^3$) for both the experimental resins, UAM and Bis-GMA.

Table 4.6 Means water sorption and solubility ($\mu g/mm^3$) of experimental resins

Experimental resins	n	Mean water sorption (SD)	Mean water solubility (SD)
Bis-GMA	10	32.36 (2.91)	4.79 (2.31)
UAM	10	52.45 (2.30)	12.28 (1.51)

A histogram indicated that the data of water sorption does not approximate the normal distribution curve and the Shapiro-Wilk normality test showed $p < .05$ (Appendix VIc), however, the data for water solubility does approximate the normal distribution curve as was illustrated by the histogram and the Shapiro-Wilk normality test showed $p > .05$ (Appendix VId). The skewness and kurtosis values of the water solubility data were had the acceptable values, which were -.108 and -1.474 respectively. The non-parametric Mann-Whitney U test was conducted for water sorption, and it was found that the water sorption of UAM was significantly higher than Bis-GMA ($p < .05$) as is shown in Table 4.7. an independent t-test was conducted for water solubility and it showed a similar pattern (Table 4.8).

Table 4.7 Comparison of water sorption between experimental resins

Experimental resins	**n**	**Mean Rank**	**Sum of Ranks**	**Wilcoxon W[a] (Z)**	**p value**
Bis-GMA	10	5.50	55.00	55	< .001
UAM	10	15.50	155.00	(-3.78)	

a Mann-Whitney U test at $p = .05$

Table 4.8 Comparison of water solubility between experimental resins

Variable	**Bis-GMA (n=10) Mean (SD)**	**UAM (n=10) Mean (SD)**	**Mean diff. (95%CI)**	***t*-stat[a] (df)**	**p value**
Water solubility	5.55 (2.01)	12.27 (1.51)	-6.72 (-8.40,5.05)	-8.45 (18)	< .001

a Independent t-test at $p < .05$ with equal variance assumed (Levene's test $p = 0.459$)

4.2.5 Flexural strength, modulus of elasticity and toughness of experimental resins

The means and standard deviations for flexural strength (MPa), the modulus of elasticity (GPa) and toughness (kJ/m^2) of UAM and Bis-GMA are presented in Table 4.9.

Table 4.9 Means flexural strength, modulus of elasticity and toughness of experimental resins

Experimental resins	**n**	**Mean flexural strength (SD) (MPa)**	**Mean modulus of elasticity (SD) (GPa)**	**Mean toughness (SD) (kJ/m^2)**
Bis-GMA	10	50.97 (19.06)	1.61 (0.64)	2.64 (1.56)
UAM	10	68.63 (5.81)	1.53 (0.13)	13.39 (5.45)

A histogram showed that the data does not approximate the normal distribution and the Shapiro-Wilk normality test showed that the p value was less than .05, for all parameters, flexural strength, modulus of elasticity and toughness (Appendix VIe, f and g). A Mann-Whitney U test was conducted and it showed that the flexural strength of UAM is significantly higher than that of Bis-GMA as is shown in Table 4.10, however, the modulus of elasticity of the later is higher than the former which $p < .05$ (Table 4.11).

The stress-strain graph of UAM showed a larger area under the stress/strain deflection curve than that of Bis-GMA, as is illustrated in Appendix VII, which indicated that the toughness of UAM is higher than Bis-GMA. This was further confirmed by the Mann-Whitney test which showed that the toughness was significantly higher ($p < .05$) compared to Bis-GMA as is shown in Table 4.12.

Table 4.10 Comparison of flexural strength between experimental resins

Experimental resins	**n**	**Mean Rank**	**Sum of Ranks**	**Wilcoxon W^a (Z)**	**p value**
Bis-GMA	10	7.90	79.00	79	.049
UAM	10	13.10	131.00	(-1.96)	

a Mann-Whitney U test at p = .05

Table 4.11 Comparison of modulus of elasticity between experimental resins

Experimental resins	**n**	**Mean Rank**	**Sum of Ranks**	**Wilcoxon W^a (Z)**	**p value**
Bis-GMA	10	13.50	135.00	75	.023
UAM	10	7.50	75.00	(-2.26)	

a Mann-Whitney U test at p = .05

Table 4.12 Comparison of toughness between experimental resins

Experimental resins	**n**	**Mean Rank**	**Sum of Ranks**	**Wilcoxon W^a (Z)**	**p value**
Bis-GMA	10	5.50	55.00	55	< .001
UAM	10	15.50	155.00	(-3.78)	

a Mann-Whitney U test at p = .05

4.3 Experimental resin systems

4.3.1 Viscosity

Figure 4.3 shows the mean viscosity and standard deviation for all experimental uncured resin systems, which ranged from 1915 to 3381 cp. The utilization of the diluents caused further decrease in the viscosity of resin systems, for examples TEGDMA lowered the viscosity of Bis-GMA from 407,350 cp to 2787 cp in the BT resin system. Bis-EMA also lowered UAM viscosity from 4,453 cp to 2576 cp and 2048 cp and this was seen in the U/E (3/1) and U/E (1/1) resin systems respectively.

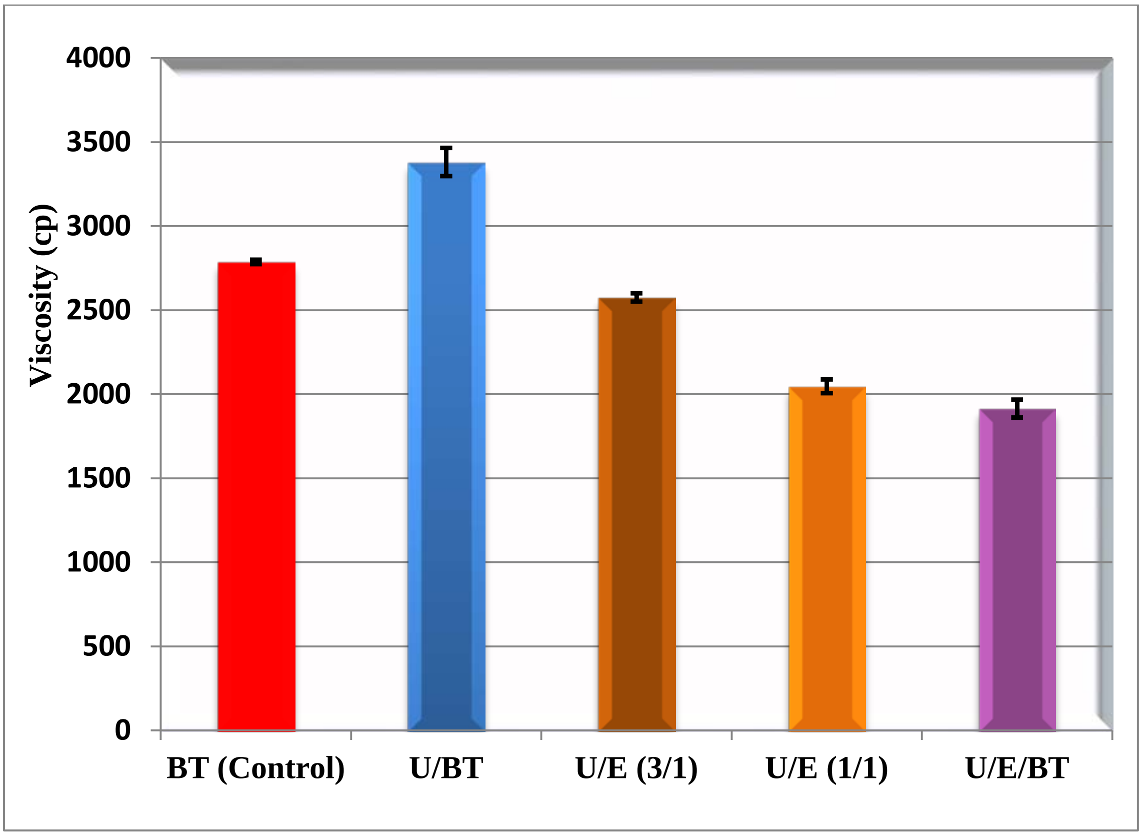


Figure 4.3 Mean viscosity of experimental resin systems

BT (Control): blending of Bis-GMA and TEGDMA monomers.
U/BT: blending of UAM, Bis-GMA and TEGDMA monomers.
U/E(3/1): blending of UAM and Bis-EMA monomers, mass ratio 3/1.
U/E(1/1): blending of UAM and Bis-EMA monomers, mass ratio 1/1.
U/E/BT: blending of UAM, Bis-EMA, Bis-GMA and TEGDMA monomers.

The histogram showed that the data does approximate the normal distribution curve and this was supported by the Shapiro-Wilk test which showed $p > .05$ (Appendix VIIIa). Furthermore, the skewness and kurtosis values were, 0.369 and -1.09 respectively.

Table 4.13 shows a summary of SPSS outputs of a One-way ANOVA test, which revealed the existence of significant difference between the experimental resin systems. The subsequent post hoc Dunnett t (2-sided) test was conducted at $p = .05$, where the common Bis-GMA/TEGDMA (BT) formulation was used as a control (Cont) group which was compared against UAM-based resin systems (Table 4.14). The viscosity of BT was significantly lower than that of U/BT, however it was significantly higher than that of U/E(3/1), U/E(1/1), and U/E/BT uncured resin systems.

Table 4.13 Effect of various resin systems on viscosity

Variable	**n**	**Mean (SD) (cp)**	**Confidence interval for mean**		**F-stat[a] (df)**	**p value**
			Lower bound	**Upper bound**		
BT (Control)	3	2787 (13)	2754.07	2820.05	414.183 (4,10)	< .001
U/BT	3	3381 (84)	3171.16	3592.16		
U/E(3/1)	3	2576 (25)	2512.77	2640.83		
U/E(1/1)	3	2048 (41)	1947.01	2150.80		
U/E/BT	3	1915 (54)	1779.60	2051.28		

a One-way ANOVA test at .05

Table 4.14 Multiple comparisons of mean viscosity of UAM-based resin systems against BT resin system

Experimental resin system code	BT (Control)	Confidence interval for mean	
		Lower bound	Upper bound
U/BT	< .001	475.68	713.51
U/E(3/1)	.002	-329.17	-91.34
U/E(1/1)	< .001	-857.07	-619.24
U/E/BT	< .001	-990.53	-752.70

Dunnett t (2-sided), p = .05

For assumption of homogeneity of variance, Levene's test indicated that the homogeneity of variance can be assumed (Appendix IXa). The Tukey HSD post hoc test was conducted to find out the significant difference amongst the UAM-based resin systems as is shown in Table 4.15. The viscosity of U/BT was significantly higher than others, and U/E(1/1) and U/E/BT showed significantly lower viscosity than U/E(3/1). However, no significant difference in viscosity was observed between the U/E (1/1) and U/E/BT uncured resin systems.

Table 4.15 Multiple comparisons of the mean viscosity among UAM-based resin systems

Experimental resin systems code	U/E(3/1)	U/E(1/1)	U/E/BT
U/BT	<.001	<.001	<.001
U/E(3/1)	-	<.001	<.001
U/E(1/1)	-	-	.054

Tukey HSD test, p = .05

4.3.2 Degree of conversion and cross-linking density of experimental resin systems

For the experimental resin systems, both means DC (%) and CLD and their standard deviations are shown in Figure 4.4.

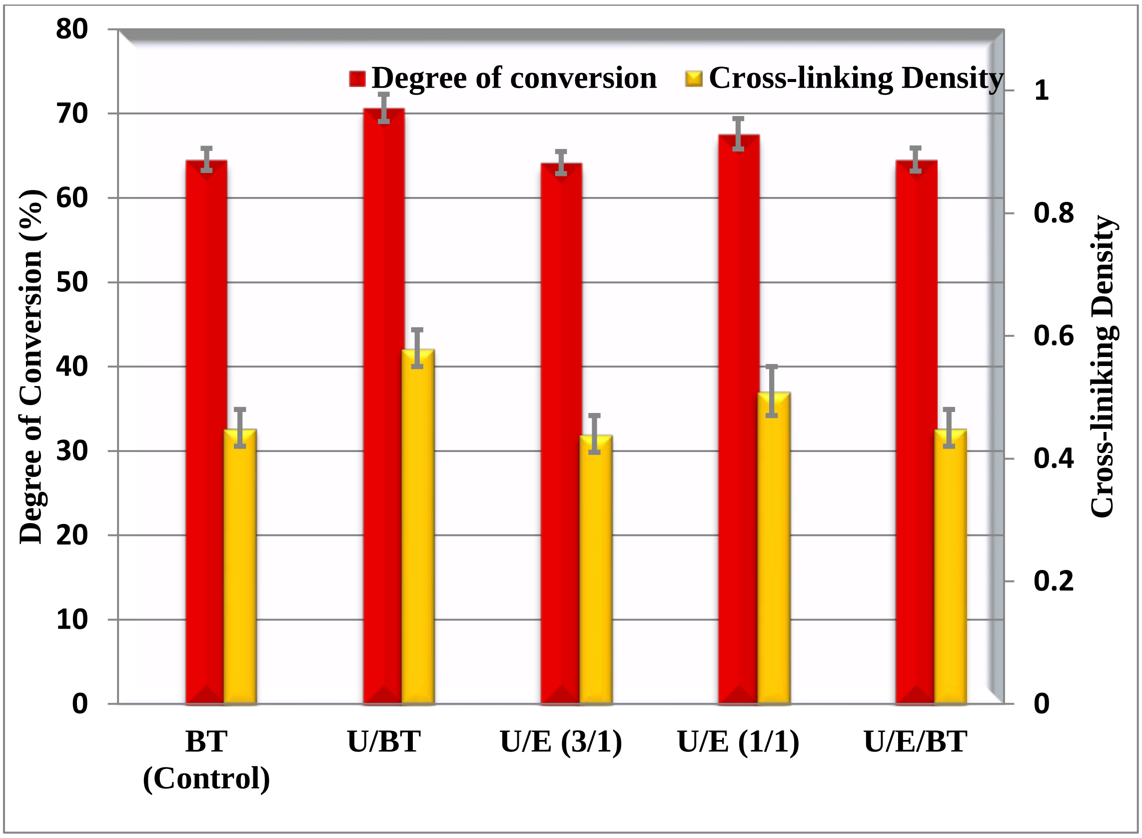


Figure 4.4 Mean degree of conversion and cross-linking density of experimental resin systems

The histogram shows that the data does not approximate the normal distribution curve and the Shapiro-Wilk normality test showed p <.05 for both DC and CLD (Appendix VIIIb and c). The Kruskal-Wallis test indicated a significant difference for DC and CLD between the experimental resin systems, as is shown in Table 4.16. The post hoc paired comparison Mann-Whitney U test was conducted to ascertain any significant difference between the experimental resin systems at a new alpha p = .04, as is shown in Table 4.17. A new alpha was calculated to control the Type I error as multiple paired comparisons had to be conducted. The detailed calculation is described in Appendix X. The DC and CLD of U/BT and U/E(1/1) resin systems were significantly higher compared to BT (control) and U/E/BT experimental resin systems. In addition, significantly higher DC and CLD was also observed for U/E(1/1) compared to U/E(3/1).

Table 4.16 Effect of various experimental resin systems on degree of conversion and cross-linking density

Variable	**n**	**Mean rank**	**Chi-square[a] (df)**	**p value**
BT (Control)	5	9.00	16.076 (4)	.003
U/BT	5	22.40		
U/E(3/1)	5	7.00		
U/E(1/1)	5	17.40		
U/E/BT	5	9.20		

a Kruskal Wallis test at p = .05

Table 4.17 Multiple comparisons of the mean degree of conversion and cross-linking density among all experimental resin systems

Experimental resin system code	**U/BT**	**U/E(3/1)**	**U/E(1/1)**	**U/E/BT**
BT (Control)	.009	.465	.028	.917
U/BT	-	.009	.047	.009
U/E(3/1)	-	-	.028	.465
U/E(1/1)	-	-	-	.028

Mann-Whitney U test, p = .04

4.3.3 Volumetric polymerization shrinkage of experimental resin systems

Figure 4.5 shows the means volumetric polymerization shrinkage percentage VPS (%) and standard deviations of experimental resin systems.

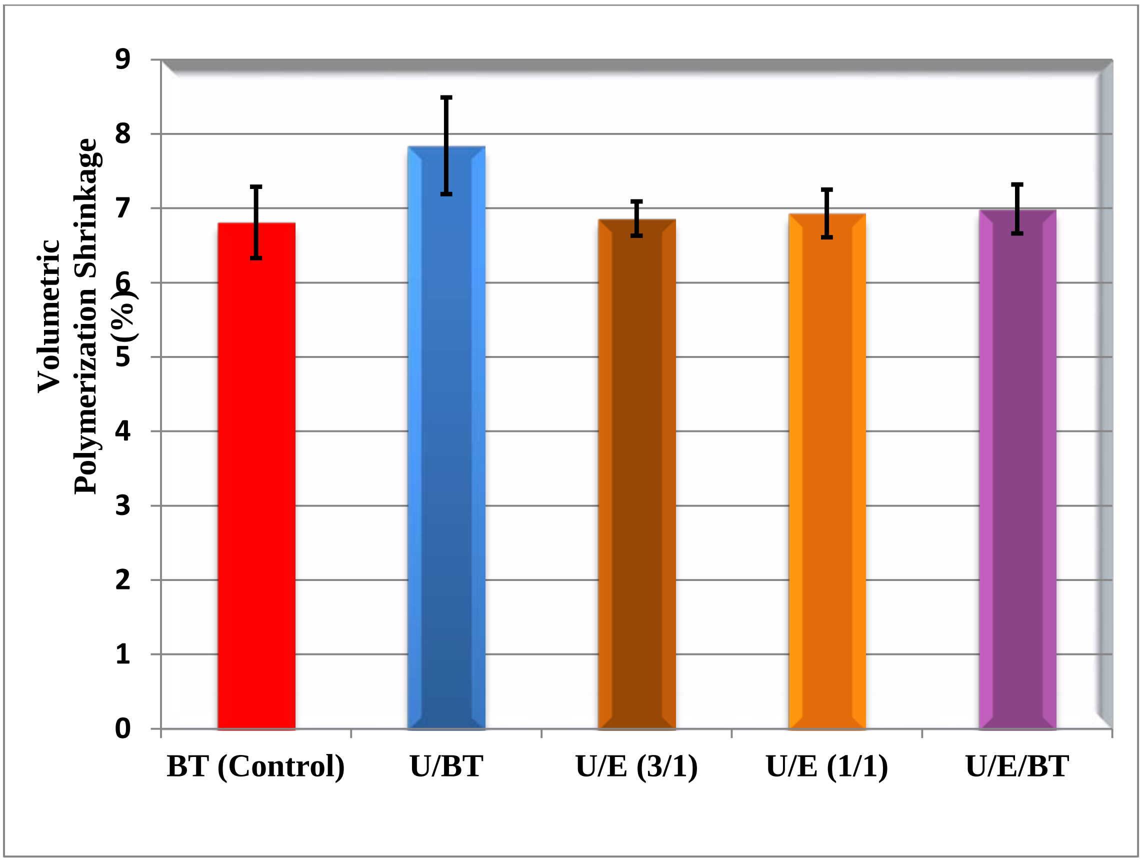


Figure 4.5 Mean volumetric polymerization shrinkage of experimental resin systems

The data showed a deviation from the normal distribution curve. When plotted, the histogram and the Shapiro-Wilk normality test showed $p < .05$ (Appendix VIIId). The Kruskal-Wallis test indicated that there is a significant difference in VPS between the experimental resin systems, as is shown in Table 4.18. A further post hoc paired comparison, Mann-Whitney U test was conducted to ascertain significant difference between the experimental resin systems at a new alpha at $p = .04$, as is shown in Table 4.19. The U/BT experimental resin system exhibited a highly significant VPS compared to other experimental resin systems.

Table 4.18 Effect of various experimental resin systems on volumetric polymerization shrinkage

Variable	n	Mean rank	Chi-square[a] (df)	p value
BT (Control)	10	17.90	14.622 (4)	.006
U/BT	10	40.50		
U/E(3/1)	10	20.90		
U/E(1/1)	10	22.90		
U/E/BT	10	25.30		

a Kruskal Wallis test at p = .05

Table 4.19 Multiple comparisons of the mean volumetric polymerization shrinkage among all experimental resin systems

Experimental resin system code	U/BT	U/E(3/1)	U/E(1/1)	U/E/BT
BT (Control)	.004	.545	.326	.199
U/BT	-	.002	.005	.008
U/E(3/1)	-	-	.650	.545
U/E(1/1)	-	-	-	.545

Mann-Whitney U test, p = .04

4.3.4 Water sorption and solubility of experimental resin systems

Figure 4.6 shows the means water sorption and water solubility (µg/mm^3) and the standard deviations of experimental resin systems.

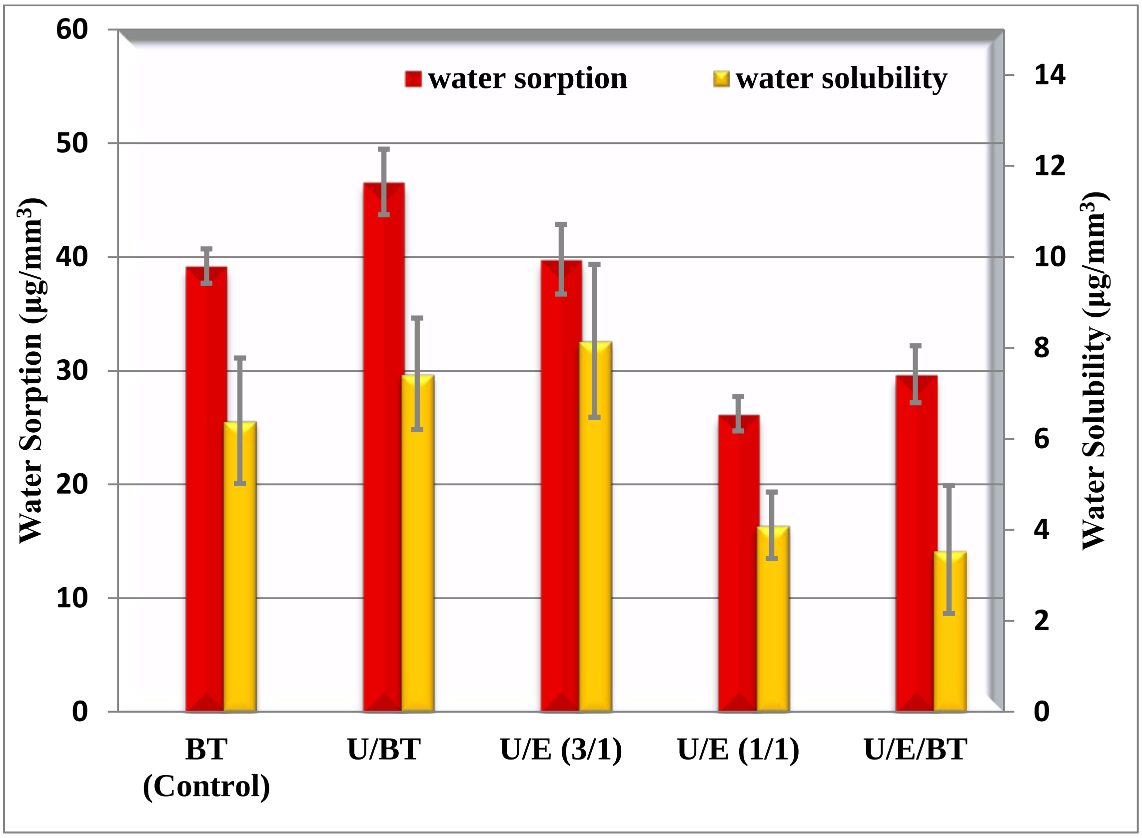


Figure 4.6 Mean water sorption and water solubility of experimental resin systems

The histogram indicates that the data of water sorption does not approximate the normal distribution and the Shapiro-Wilk normality test showed p < .05 (Appendix VIIIe). However, the data for water solubility did approximate the normal distribution curve and the Shapiro-Wilk normality test showed p > .05 (Appendix VIIIe and f). The skewness and kurtosis values of water solubility were considered to be acceptable, -.124 and -.825 respectively.

The water sorption, Kruskal-Wallis test indicated a significant difference in water sorption between the experimental resin systems, as is shown in Table 4.20, and the post hoc paired comparison Mann-Whitney U test was conducted at p = .04 as shown in Table 4.21. The U/BT showed higher water sorption than other experimental resin systems, however, the water sorption of U/E(1/1) was significantly lower compared to all other experimental resin systems.

Table 4.20 Effect of various experimental resin systems on water sorption

Variable	**n**	**Mean rank**	**Chi-square**[a] **(df)**	**p value**
BT (Control)	10	29.55	43.41 (4)	< .001
U/BT	10	45.20		
U/E(3/1)	10	31.75		
U/E(1/1)	10	6.70		
U/E/BT	10	14.30		

a Kruskal Wallis test at p = .05

Table 4.21 Multiple comparisons of the mean water sorption among all experimental resin systems

Experimental resin system code	**U/BT**	**U/E(3/1)**	**U/E(1/1)**	**U/E/BT**
BT (Control)	<.001	.473	<.001	<.001
U/BT	-	<.001	<.001	<.001
U/E(3/1)	-	-	<.001	<.001
U/E(1/1)	-	-	-	.004

Mann-Whitney U test, p = .04

For the water solubility of experimental resin systems, Table 4.22 shows a summary of SPSS outputs of One-way ANOVA showed $p < .05$. The post hoc Dunnett t (2-sided) test was carried out at $p = .05$ for a multiple comparison using the BT experimental resin system as a control (Table 4.23). The water solubility of U/E(1/1) and U/E/BT was significantly lower than BT, however U/E(3/1) showed significantly higher water solubility than BT.

Table 4.22 Effect of various experimental resin systems on water solubility

Variable	**n**	**Mean (SD) ($\mu g/mm^3$)**	**Confidence interval for mean**		**F-stat[a] (df)**	**p value**
			Lower bound	**Upper bound**		
BT (Control)	10	6.40 (1.38)	5.41	7.40	23.20 (4,45)	< .001
U/BT	10	7.43 (1.23)	6.55	8.32		
U/E(3/1)	10	8.16 (1.68)	6.95	9.36		
U/E(1/1)	10	4.1 (0.73)	3.57	4.62		
U/E/BT	10	3.57 (1.41)	2.55	4.58		

a One-way ANOVA test at = .05

Table 4.23 Multiple comparisons of mean water solubility of UAM-based resin systems against BT resin system

Experimental resin system code	**BT (Control)**	**Confidence interval for mean**	
		Lower bound	**Upper bound**
U/BT	.260	-.47	2.53
U/E(3/1)	.018	.24	3.25
U/E(1/1)	.001	-3.81	-.80
U/E/BT	< .001	-4.34	-1.33

Dunnett t (2-sided), $p = .05$

For assumption of homogeneity of variance, Levene's test indicated that the homogeneity of variance can be assumed (Appendix IXb). The Tukey HSD post hoc test was conducted to test out the significant difference between the UAM-based experimental resin systems as is shown in Table 4.24. The water solubility of experimental resin systems U/E(1/1) and U/E/BT was significantly lower than U/BT and U/E(3/1) experimental resin systems.

Table 4.24 Multiple comparisons of the mean water solubility among UAM-based resin systems

Experimental resin system code	**U/E(3/1)**	**U/E(1/1)**	**U/E/BT**
U/BT	.742	<.001	<.001
U/E(3/1)	-	<.001	<.001
U/E(1/1)	-	-	.898

Tukey HSD test, p = .05

4.3.5 Flexural strength, modulus of elasticity and toughness of experimental resin systems

Figure 4.7 shows the means and standard deviations for flexural strength (MPa) and modulus of elasticity (GPa) of the experimental resin system. The mean toughness (kJ/m^2) is shown in Figure 4.8.

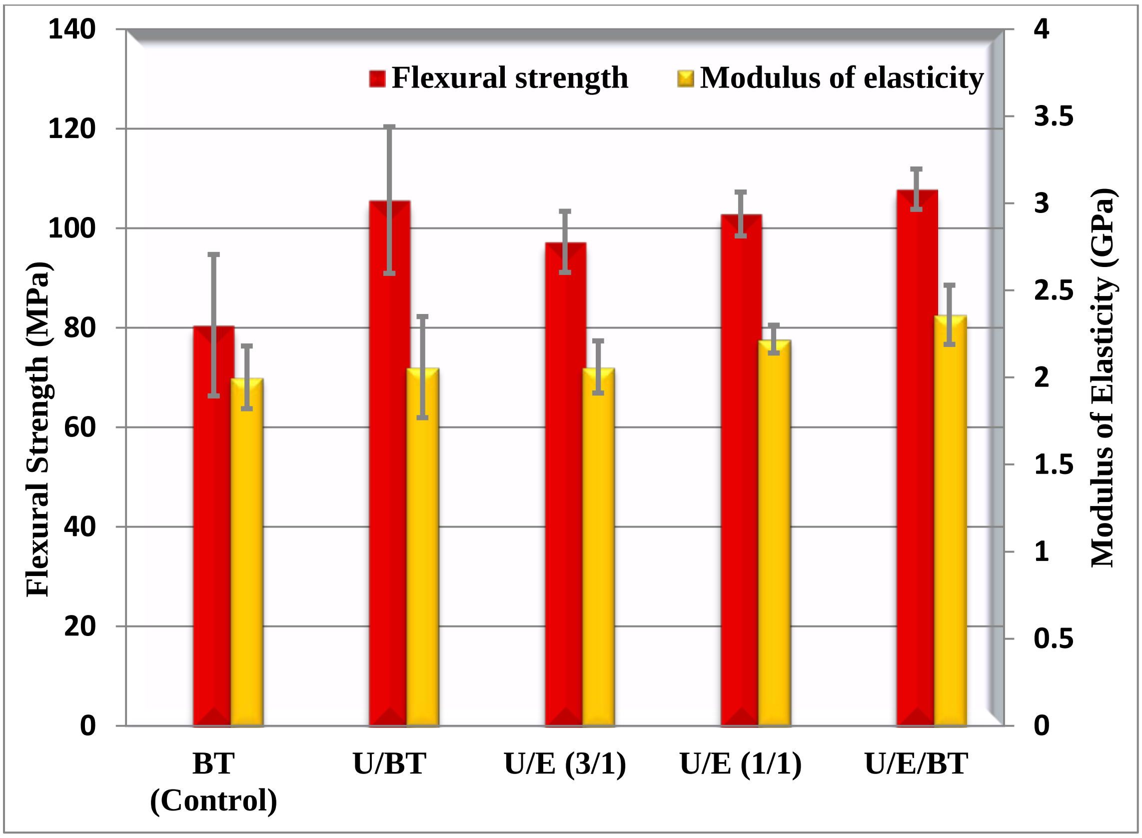


Figure 4.7 Mean flexural strength and modulus of elasticity of experimental resin systems

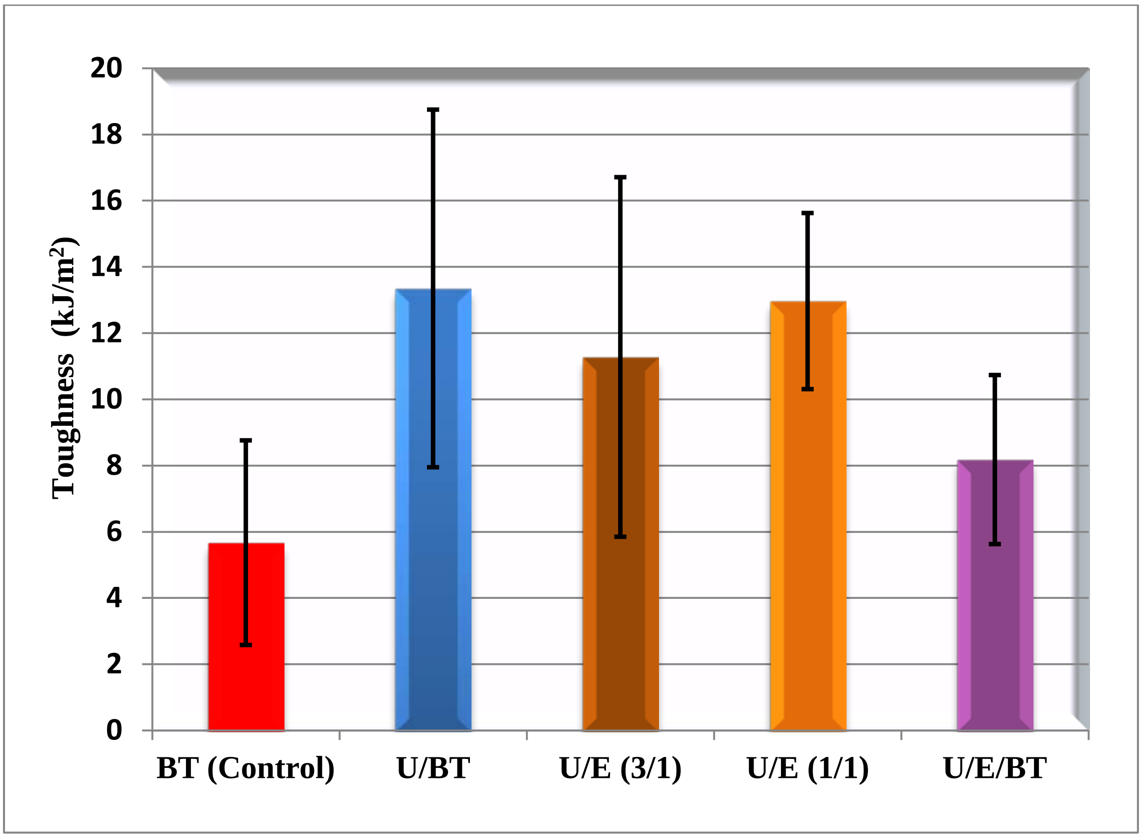


Figure 4.8 Mean toughness of the experimental resin systems

The histogram indicates that the data for flexural strength and toughness does not approximate the normal distribution curve and the Shapiro-Wilk normality test showed $p < .05$ (Appendix VIIIg and i). However, the data of modulus of elasticity showed the approximate normal distribution curve and this was supported by the skewness and kurtosis values of .087 and .293 respectively, while the Shapiro-Wilk test showed $p > .05$ (Appendix VIIIh).

The Kruskal-Wallis test was conducted for flexural strength and toughness, which indicated a significant difference among the experimental resin systems, as is shown in Table 4.25 and Table 4.26 respectively. The post hoc paired comparison Mann-Whitney U test at $p = .04$ are tabulated in Table 4.27 and Table 4.28 for flexural strength and toughness respectively. Each UAM-based experimental resin system (U/BT, U/E, and U/E/BT) showed a significantly higher flexural strength and toughness than BT (control) resin system. Although U/BT exhibited the highest flexural strength amongst all UAM-based resin systems, the differences were not significant. The same trend was also observed for the toughness values except when U/E/BT and U/E(1/1) was compared, where the toughness of U/E(1/1) was significantly higher.

Table 4.25 Effect of various experimental resin systems on flexural strength

Variable	**n**	**Mean rank**	**Chi-square[a] (df)**	**p value**
BT (Control)	10	6.80	27.896 (4)	< .001
U/BT	10	29.40		
U/E(3/1)	10	21.60		
U/E(1/1)	10	30.50		
U/E/BT	10	39.20		

a Kruskal Wallis test at $p = .05$

Table 4.26 Effect of various experimental resin systems on toughness

Variable	n	Mean rank	Chi-square[a] (df)	p value
BT (Control)	10	10.20	21.2 (4)	< .001
U/BT	10	33.90		
U/E(3/1)	10	27.30		
U/E(1/1)	10	36.10		
U/E/BT	10	20.00		

a Kruskal Wallis test at p = .05

Table 4.27 Multiple comparisons of the mean flexural strength among all experimental resin systems

Experimental resin system code	U/BT	U/E(3/1)	U/E(1/1)	U/E/BT
BT (Control)	<.001	.002	<.001	<.001
U/BT	-	.496	.650	.406
U/E(3/1)	-	-	.049	.001
U/E(1/1)	-	-	-	.016

Mann-Whitney U test, p = .04

Table 4.28 Multiple comparisons of the mean toughness among all experimental resin systems

Experimental resin system code	U/BT	U/E(3/1)	U/E(1/1)	U/E/BT
BT (Control)	.002	.010	.001	.010
U/BT	-	.29	.940	.023
U/E(3/1)	-	-	.174	.226
U/E(1/1)	-	-	-	.001

Mann-Whitney U test, p = .04

For the modulus of elasticity of experimental resin systems, Table 4.29 shows a summary of SPSS outputs of One-way ANOVA showed $p < .05$. Furthermore, post hoc Dunnett t (2-sided) test for multiple comparisons against control resin system (BT) was carried out as is shown in Table 4.30. There is no significant difference between the BT and UAM-based resin systems except the U/E/BT resin system showed higher modulus of elasticity than BT.

Table 4.29 Effect of various experimental resin systems on modulus of elasticity

Variable	n	Mean (SD) (GPa)	Confidence interval for mean		F-stat[a] (df)	p value
			Lower bound	Upper bound		
BT (Control)	10	2.00 (0.18)	1.87	2.14	5.593 (4,45)	.001
U/BT	10	2.06 (0.29)	1.85	2.27		
U/E(3/1)	10	2.06 (0.15)	1.95	2.18		
U/E(1/1)	10	2.22 (0.08)	2.16	2.28		
U/E/BT	10	2.36 (0.17)	2.23	2.48		

a One-way ANOVA test at p = .05

Table 4.30 Multiple comparisons of mean modulus of elasticity of UAM-based resin systems against BT resin system

Experimental resin system code	BT (Control)	Confidence interval for mean	
		Lower bound	Upper bound
U/BT	.914	-.16	.27
U/E(3/1)	.890	-.15	.27
U/E(1/1)	.052	-.00	.43
U/E/BT	.001	.13	.56

Dunnett t (2-sided), p = .05

For assumption of homogeneity of variance, Levene's test indicated that the homogencity of variance cannot be assumed (Appendix IXc). The Dunnett T3 post hoc test was conducted to identify the significant difference between the experimental UAM-based resin systems as is shown in Table 4.31. The only significant difference was between U/E/BT and U/E(3/1), where U/E/BT exhibited significantly higher modulus of elasticity.

Table 4.31 Multiple comparison of the mean modulus of elasticity among UAM-based resin systems

Experimental resin system code	U/E(3/1)	U/E(1/1)	U/E/BT
U/BT	.100	.657	.137
U/E(3/1)	-	.130	.011
U/E(1/1)	-	-	.329

Dunnett T3, p = .05

4.4 Flowable composite

4.4.1 Volumetric polymerization shrinkage of flowable composite

Figure 4.9 shows the mean percentage of volumetric polymerization shrinkage (% VPS) and standard deviation for both experimental flowable composites and commercial Esthet.X flow.

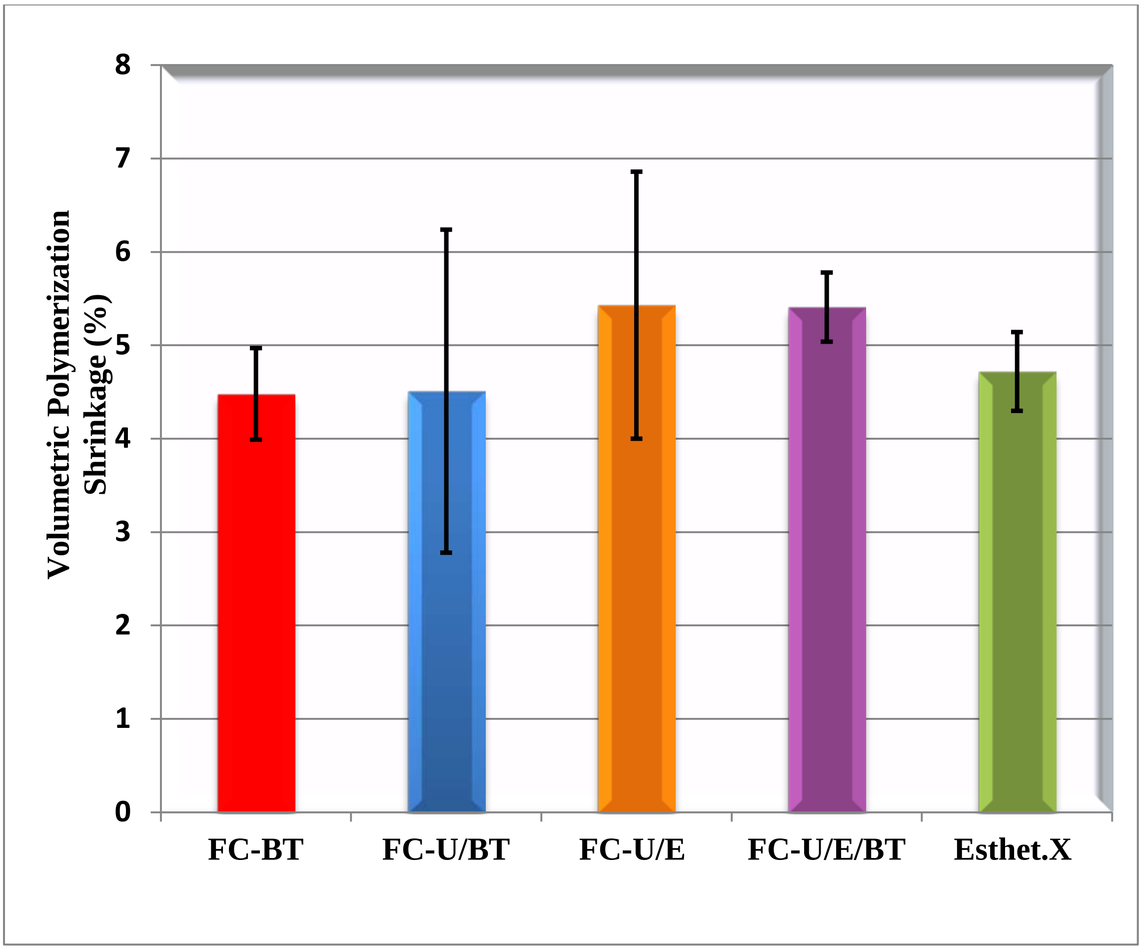


Figure 4.9 Mean volumetric polymerization shrinkage of flowable composites

The histogram shows that the data does not approximate the normal distribution curve and the Shapiro-Wilk normality test showed p < .05 (Appendix XIa). The Kruskal-Wallis test indicated that there was a significant difference in volumetric polymerization shrinkage among the flowable composites, as is shown in Table 4.32. A further post hoc paired comparison Mann-Whitney U test was conducted at a new alpha which p = .04 as is shown in Table 4.33. The volumetric polymerization shrinkage of FC-U/E and FC-U/E/BT was significantly higher compared to the experimental control (Exp-Cont) group FC-BT. Only FC-U/E/BT showed significantly higher volumetric polymerization shrinkage than commercial control (Com-Cont) group Esthet.X flow. However, the volumetric polymerization shrinkage of FC-U/BT was significantly lower than FC-BT (Exp-Cont) and Esthet.X flow (Com-cont).

Table 4.32 Effect of various flowable composites on volumetric polymerization shrinkage

Variable	**n**	**Mean rank**	**Chi-square[a] (df)**	**p value**
FC-BT	10	19.20	21.236 (4)	< .001
FC-U/BT	10	13.70		
FC-U/E	10	32.40		
FC-U/E/BT	10	40.10		
Esthet.X flow	10	22.10		

a Kruskal Wallis test at p = .05

Table 4.33 Multiple comparisons of the mean volumetric polymerization shrinkage among all flowable composites

Flowable composite code	**FC-U/BT**	**FC-U/E**	**FC-U/E/BT**	**Esthet.X flow (Com-Cont)**
FC-BT (Exp-Cont)	.131	.023	.001	.597
FC-U/BT	-	.013	.005	.034
FC-U/E	-	-	.131	.049
FC-U/E/BT	-	-	-	.001

Mann-Whitney U test, p = .04

4.4.2 Volumetric change of flowable composites

Figure 4.10 shows the mean percentage of volumetric change and standard deviation for flowable composites.

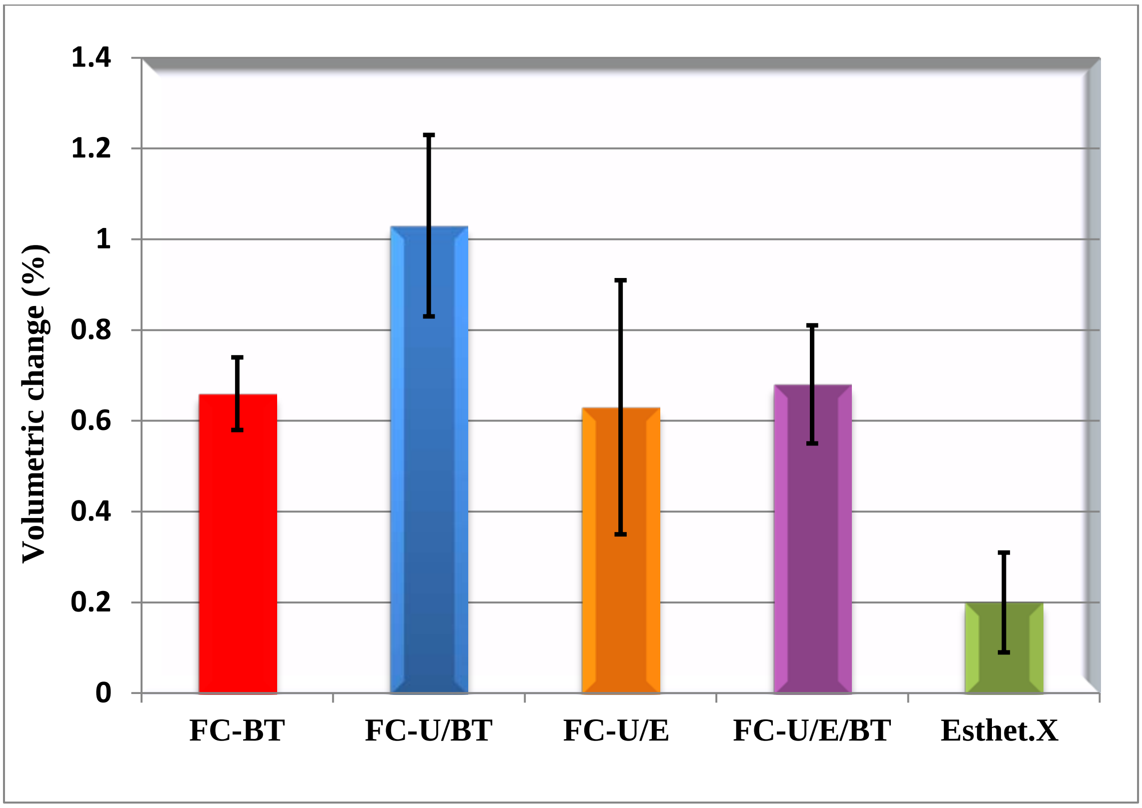


Figure 4.10 Mean volumetric change of flowable composites

The histogram indicates that the data of volumetric change approximates the normal distribution curve with acceptable skewness and kurtosis values of -.118 and -.271 respectively and the Shapiro-Wilk normality test showed p > .05 (Appendix XIb). One-way ANOVA showed a significant difference amongst the flowable composites as is shown in Table 4.34. The post hoc Dunnett t (2-sided) test was conducted twice; one FC-BT was selected as experimental control (Table 4.35) and the other Esthet.X flow was selected as commercial control (Table 4.36). The volumetric change of FC-U/BT was significantly higher than both FC-BT (Exp-Cont) and Esthet.X flow (Com-Cont).

Esthet.X flow showed a significantly lower volumetric change than other experimental flowable composites.

Table 4.34 Effect of various flowable composites on percentage of volumetric change

Variable	n	Mean (SD) (%)	Confidence interval for mean		F-stat[a] (df)	p value
			Lower bound	Upper bound		
FC-BT (Exp-Cont)	10	0.66 (0.08)	.60	.72	26.96 (4,45)	< .001
FC-U/BT	10	1.03 (0.2)	.89	1.18		
FC-U/E	10	0.63 (0.28)	.43	.84		
FC-U/E/BT	10	0.68 (0.13)	.59	.77		
Esthet.X flow (Com-Cont)	10	0.2 (0.11)	.12	.29		

a One-way ANOVA test at p = .05

Table 4.35 Multiple comparisons of mean volumetric change of flowable composite against FC-BT (experimental control)

Flowable composite code	FC-BT (Exp-Cont)	Confidence interval for mean	
		Lower bound	Upper bound
FC-U/BT	< .001	.16	.57
FC-U/E	.986	-.23	.17
FC-U/E/BT	.998	-.18	.22
Esthet.X flow	< .001	-.66	-.25

Dunnett t (2-sided), p = .05

Table 4.36 Multiple comparisons of mean volumetric change of experimental flowable composite against Esthet.X flow (commercial control)

Experimental flowable composite code	Esthet.X flow (Com-Cont)	Confidence interval for mean	
		Lower bound	**Upper bound**
FC-BT	< .001	.25	.66
FC-U/BT	< .001	.62	1.03
FC-U/E	< .001	.22	.63
FC-U/E/BT	< .001	.27	.68

Dunnett t (2-sided), p = .05

For the assumption of homogeneity of variance, Levene's test indicated that the homogeneity of variance cannot be assumed (Appendix IXd). The Dunnett T3 post hoc test was chosen to identify the difference between flowable composites based on experimental UAM for volumetric change at p = .05 as shown in Table 4.37. FC-U/E showed a lower volumetric change when compared to FC-U/BT.

Table 4.37 Multiple comparisons of the volumetric change among UAM-based flowable composites

Experimental flowable composites code	FC-U/E	FC-U/E/BT
FC-U/BT	.022	.003
FC-U/E	-	1.00

Dunnett T3 test, p = .05

4.4.3 Water sorption and solubility of flowable composites

Figure 4.11 shows the means of water sorption and water solubility (µg/mm^3) and standard deviations of flowable composites.

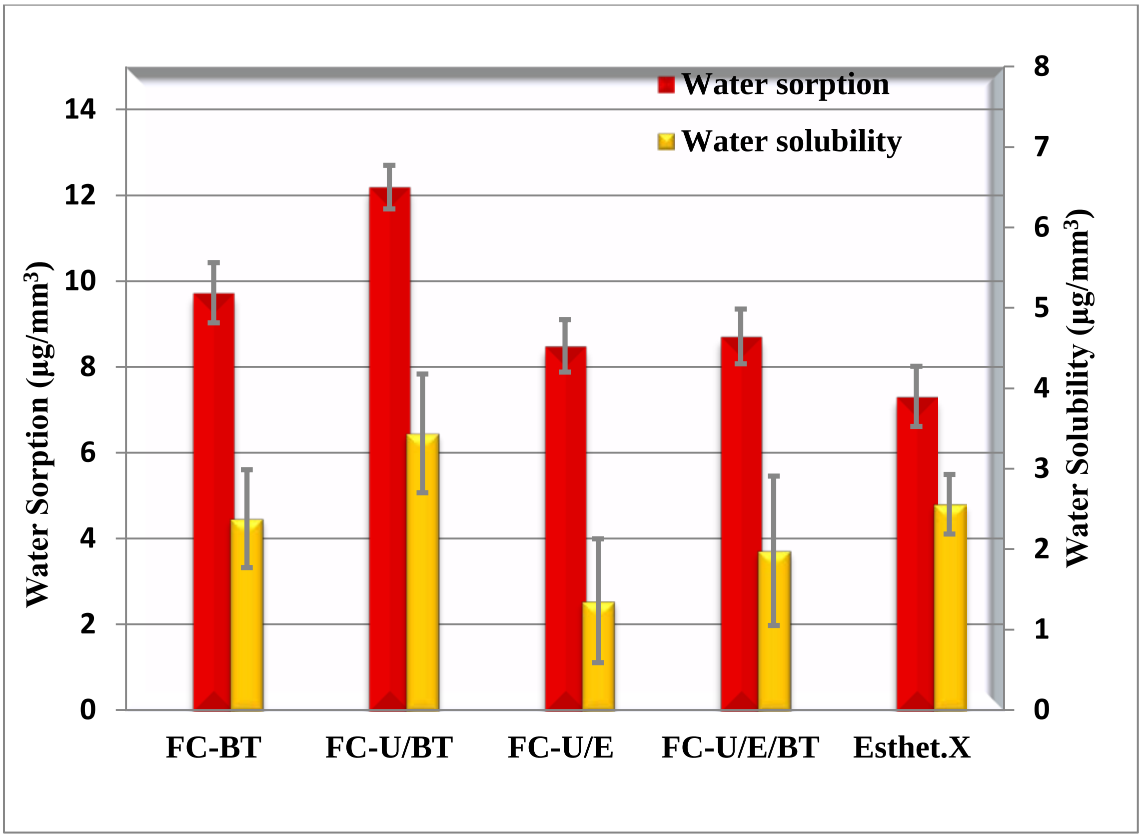


Figure 4.11 Mean water sorption and water solubility of flowable composites

The histogram indicates that the data for water sorption does not approximate the normal distribution curve and the Shapiro-Wilk normality test showed $p < .05$ (Appendix XIc). However, data for water solubility showed approximation to the normal distribution curve with skewness and kurtosis values of .130 and .065 respectively, and the Shapiro-Wilk normality test showed $p > .05$ (Appendix XId).

The Kruskal-Wallis test was conducted for water sorption and it indicated that there was a significant difference among the flowable composites, as is shown in Table 4.38. A further post hoc paired comparison Mann-Whitney U test was conducted at p = .04 as is shown in Table 4.39. The water sorption of FC-U/E and FC(U/E/BT) was significantly lower compared to the FC-BT (Exp-Cont), however, Esthet.X flow (Com-Cont) showed lower water sorption compared to all groups.

Table 4.38 Effect of various flowable composite on water sorption

Variable	**n**	**Mean rank**	**Chi-square[a] (df)**	**p value**
FC-BT	10	32.95	39.27 (4)	< .001
FC-U/BT	10	45.50		
FC-U/E	10	19.30		
FC-U/E/BT	10	22.45		
Esthet.X flow	10	7.30		

a Kruskal Wallis test at p = .05

Table 4.39 Multiple comparisons of the mean water sorption among all flowable composites

Flowable composite code	**FC-U/BT**	**FC-U/E**	**FC-U/E/BT**	**Esthet.X flow (Com-Cont)**
FC-BT (Exp-Cont)	<.001	.002	.008	<.001
FC-U/BT	-	<.001	<.001	<.001
FC-U/E	-	-	.384	.003
FC-U/E/BT	-	-	-	.001

Mann-Whitney U test, p = .04

For the water solubility of flowable composites, Table 4.40 shows a summary of SPSS outputs of the One-way ANOVA test, which p < .05. Subsequently the post hoc Dunnett t (2-sided) test was carried out twice; one for experimental control FC-BT (Table 4.41) and the other for commercial control Esthet.X flow (Table 4.42). Water solubility of FC-U/BT was significantly higher compared to the FC-BT (Exp-Cont), while FC-U/E exhibited significantly lower solubility. A similar pattern was observed for the water solubility of Esthet.X flow (Com-Cont).

Table 4.40 Effect of various flowable composites on water solubility

Variable	**n**	**Mean (SD) ($\mu g/mm^3$)**	**Confidence interval for mean**		**F-stat[a] (df)**	**p value**
			Lower bound	**Upper bound**		
FC-BT (Exp-Cont)	10	2.38 (0.61)	1.94	2.82	11.576 (4,45)	< .001
FC-U/BT	10	3.44 (0.74)	2.90	3.98		
FC-U/E	10	1.36 (0.77)	0.81	1.91		
FC-U/E/BT	10	1.98 (0.93)	1.31	2.64		
Esthet.X flow (Com-Cont)	10	2.56 (0.37)	2.29	2.83		

a One-way ANOVA test at p = .05

Table 4.41 Multiple comparisons of mean water solubility of flowable composite against FC-BT (experimental control)

Flowable composite code	**FC-BT (Exp-Cont)**	**Confidence interval for mean**	
		Lower bound	**Upper bound**
FC-U/BT	.007	.24	1.86
FC-U/E	.009	-1.83	-.21
FC-U/E/BT	.520	-1.21	.39
Esthet.X flow	.944	-.62	.98

Dunnett t (2-sided), p = .05

Table 4.42 Multiple comparisons of mean water solubility of experimental flowable composite against Esthet.X flow (commercial control)

Experimental flowable composite code	Esthet.X flow (Com-Cont)	Confidence interval for mean	
		Lower bound	Upper bound
FC-BT	.944	-.98	.62
FC-U/BT	.030	.06	1.68
FC-U/E	.002	-2.01	-.39
FC-U/E/BT	.212	-1.39	.21

Dunnett t (2-sided), p = .05

Levene's test indicated that the homogencity of variance can be assumed (Appendix IXe). The Tukey HSD post hoc test was conducted to test out the significant difference between the experimental UAM-based flowable composites as is shown in Table 4.43. The water solubility of FC-U/BT was significantly higher compared to the other experimental flowable composites, which were based on UAM.

Table 4.43 Multiple comparisons of the mean water solubility among UAM-based flowable composites

Experimental flowable composites code	FC-U/E	FC-U/E/BT
FC-U/BT	<.001	<.001
FC-U/E	-	.316

Tukey HSD test, p = .05

4.4.4 Flexural strength, modulus of elasticity and toughness of flowable composites

Figure 4.12 shows the means flexural strength (MPa) and modulus of elasticity (GPa) and standard deviations of flowable composites. The mean toughness (kJ/m^2) is shown in Figure 4.13.

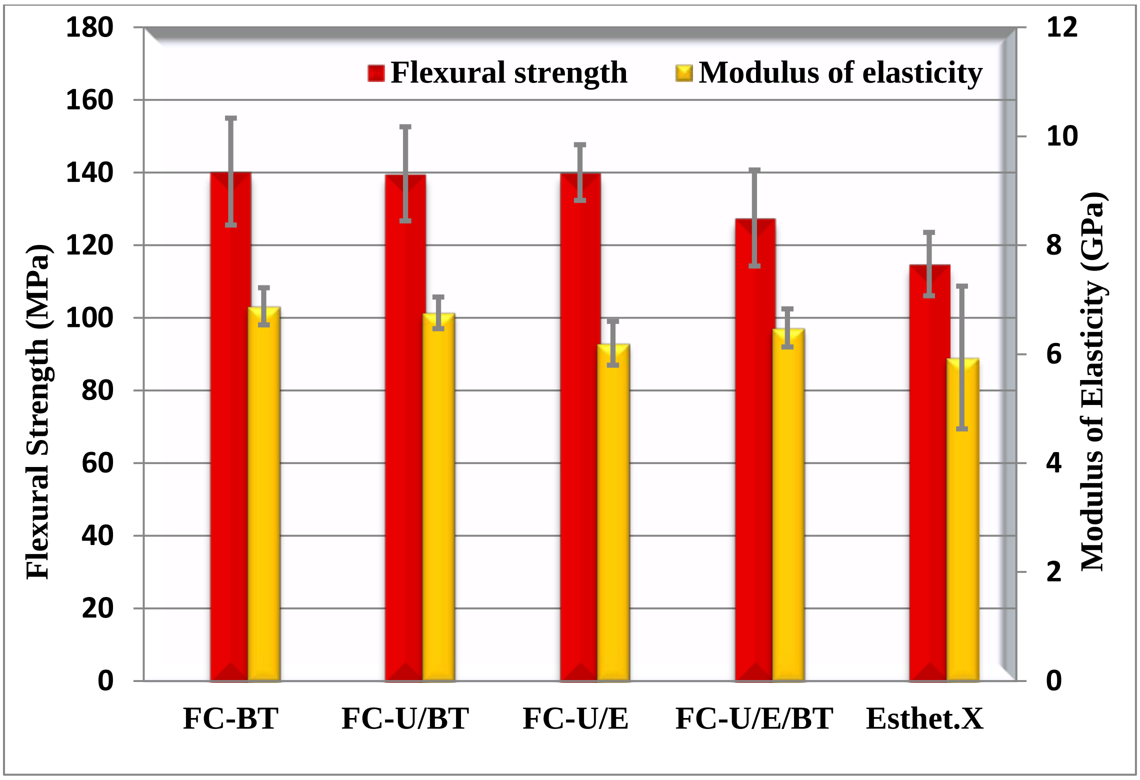


Figure 4.12 Mean flexural strength and modulus of elasticity of flowable composites

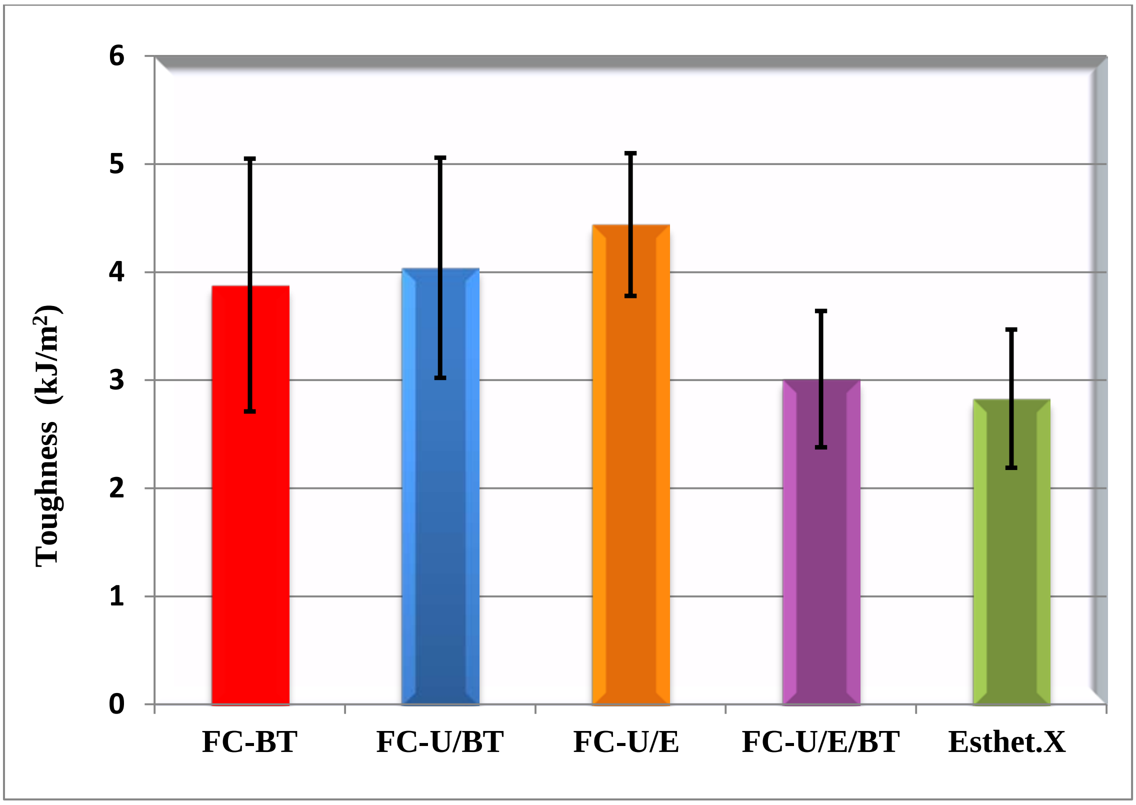


Figure 4.13 Mean toughness of flowable composites

The histogram indicates that the data of flexural strength and toughness does approximate the normal distribution curve and the Shapiro-Wilk normality test shows p > .05 (Appendix XIe and g). The values of skewness and kurtosis for flexural strength were -.216 and -.874 respectively. For toughness they were .286 and -.893 respectively and this is considered acceptable. However, the modulus of elasticity data does not approximate to the normal distribution curve from the histogram as with a normal curve and this was further supported by the Shapiro-Wilk normality test, which p < .05 (Appendix XIf).

One-way ANOVA showed that there was a significant difference in flexural strength (Table 4.44) and toughness (4.45) among the flowable composites.

Table 4.44 Effect of various flowable composites on flexural strength

Variable	**n**	**Mean (SD) (MPa)**	**Confidence interval for mean**		**F-stat[a] (df)**	**p value**
			Lower bound	**Upper bound**		
FC-BT (Exp-Cont)	10	140.31 (14.71)	129.79	150.84	10.52 (4,45)	< .001
FC-U/BT	10	139.70 (12.95)	130.44	148.97		
FC-U/E	10	140.02 (7.67)	134.53	145.51		
FC-U/E/BT	10	127.51 (13.18)	118.08	136.94		
Esthet.X flow (Com-Cont)	10	114.82 (8.7)	108.59	121.04		

a One-way ANOVA test at p = .05

Table 4.45 Effect of various flowable composites on toughness

Variable	n	Mean (SD) (kJ/m^2)	Confidence interval for mean		F-stat[a] (df)	p value
			Lower bound	Upper bound		
FC-BT (Exp-Cont)	10	3.88 (1.17)	3.04	4.73	6.416 (4,45)	< .001
FC-U/BT	10	4.04 (1.02)	3.30	4.77		
FC-U/E	10	4.44 (0.66)	3.96	4.92		
FC-U/E/BT	10	3.01 (0.63)	2.56	3.46		
Esthet.X flow (Com-Cont)	10	2.83 (0.64)	2.37	3.29		

a One-way ANOVA test at p = .05

For post hoc Dunnett t (2-sided) multiple comparisons, Table 4.46 illustrates that there is no significant difference between UAM-based flowable composites and FC-BT (Exp-Cont), however, Table 4.47 shows that the flexural strength of FC-BT and UAM-based flowable composite, except FC-U/E/BT, were significantly higher than Esthet.X flow (Com-Cont). The same pattern was observed for the toughness, Table 4.48 showed no significant difference between the UAM-based flowable composite and FC-BT (Exp-Cont). However, UAM-based flowable composites, except for FC-U/E/BT, and FC-BT showed higher toughness than Esthet.X flow (Com-Cont) as is shown in Table 4.49.

Table 4.46 Multiple comparisons of mean flexural strength of flowable composite against FC-BT (experimental control)

Flowable composite code	**FC-BT (Exp-Cont)**	**Confidence interval for mean**	
		Lower bound	**Upper bound**
FC-U/BT	1.000	-13.93	12.71
FC-U/E	1.000	-13.62	13.03
FC-U/E/BT	.063	-26.13	00.52
Esthet.X flow	< .001	-38.82	-12.17

Dunnett t (2-sided), p = .05

Table 4.47 Multiple comparisons of mean flexural strength of experimental flowable composite against Esthet.X flow (commercial control)

Experimental flowable composite code	**Esthet.X flow (Com-Cont)**	**Confidence interval for mean**	
		Lower bound	**Upper bound**
FC-BT	< .001	12.17	38.82
FC-U/BT	< .001	11.56	38.21
FC-U/E	< .001	11.87	38.53
FC-U/E/BT	.066	-.63	26.02

Dunnett t (2-sided), p = .05

Table 4.48 Multiple comparisons of mean toughness of flowable composite against FC-BT (experimental control)

Flowable composite code	**FC-BT (Exp-Cont)**	**Confidence interval for mean**	
		Lower bound	**Upper bound**
FC-U/BT	.985	-.82	1.12
FC-U/E	.413	-.41	1.52
FC-U/E/BT	.091	-1.84	.10
Esthet.X flow	.031	-2.02	-.07

Dunnett t (2-sided), p = .05

Table 4.49 Multiple comparisons of mean toughness of experimental flowable composite against Esthet.X flow (commercial control)

Experimental flowable composite code	Esthet.X flow (Com-Cont)	Confidence interval for mean	
		Lower bound	Upper bound
FC-BT	.031	.07	2.02
FC-U/BT	.011	.22	2.17
FC-U/E	.001	.63	2.58
FC-U/E/BT	.971	-.79	1.15

Dunnett t (2-sided), p = .05

Levene's test indicated that the homogeneity of variance can be assumed for both flexural strength and toughness (Appendix IXf). The Tukey HSD post hoc test was conducted at p = .04 to compare flexural strength and toughness as is shown in Table 4.50 and Table 4.51 respectively, among the UAM-based flowable composites. There is no significant difference of flexural strength and toughness among the experimental UAM-based flowable composites, except that FC-U/E which showed higher toughness than FC-U/E/BT.

Table 4.50 Multiple comparisons of the flexural strength among UAM-based flowable composites

Experimental flowable composites code	FC-U/E	FC-U/E/BT
FC-U/BT	1.00	.159
FC-U/E	-	.141

Tukey HSD test, p = .05

Table 4.51 Multiple comparisons of the toughness among UAM-based flowable composites

Flowable composites code	**FC-U/E**	**FC-U/E/BT**
FC-U/BT	.831	.077
FC-U/E	-	.005

Tukey HSD test, p = .05

For modulus of elasticity, Kruskal-Wallis test indicated a significant difference among all flowable composites, as is shown in Table 4.52. The post hoc paired comparison Mann-Whitney U test was conducted at p = .04 as is shown in Table 4.53. The experimental FC-U/E only showed lower modulus of elasticity than FC-BT (Exp-Cont). Esthet.X flow exhibited the lowest modulus of elasticity compared to all experimental flowable composites; however this observation was only significant for FC-BT and FC-U/BT.

Table 4.52 Effect of various flowable composites on modulus of elasticity

Variable	**n**	**Mean rank**	**Chi-square^a (df)**	**p value**
FC-BT (Exp-Cont)	10	37.40	17.729 (4)	< .001
FC-U/BT	10	33.50		
FC-U/E	10	15.60		
FC-U/E/BT	10	23.90		
Esthet.X flow (Com-Cont)	10	17.10		

a Kruskal Wallis test at p = .05

Table 4.53 Multiple comparisons of the mean modulus of elasticity among all flowable composites

Flowable composites code	**FC-U/BT**	**FC-U/E**	**FC-U/E/BT**	**Esthet.X flow (Com-Cont)**
FC-BT (Exp-Cont)	.226	.002	.041	.007
FC-U/BT	-	.007	.049	.010
FC-U/E	-	-	.112	.880
FC-U/E/BT	-	-	-	.226

Mann-Whitney U test, p = .04

4.4.5 Cytotoxicity of the flowable composite

Figure 4.14 shows the mean percentage of viable cell and standard deviation for flowable composites and solvent. The mean percentage of viable cells of experimental flowable composites and commercial flowable composite (Esthet.X flow) showed a narrow range between 82% and 85%.

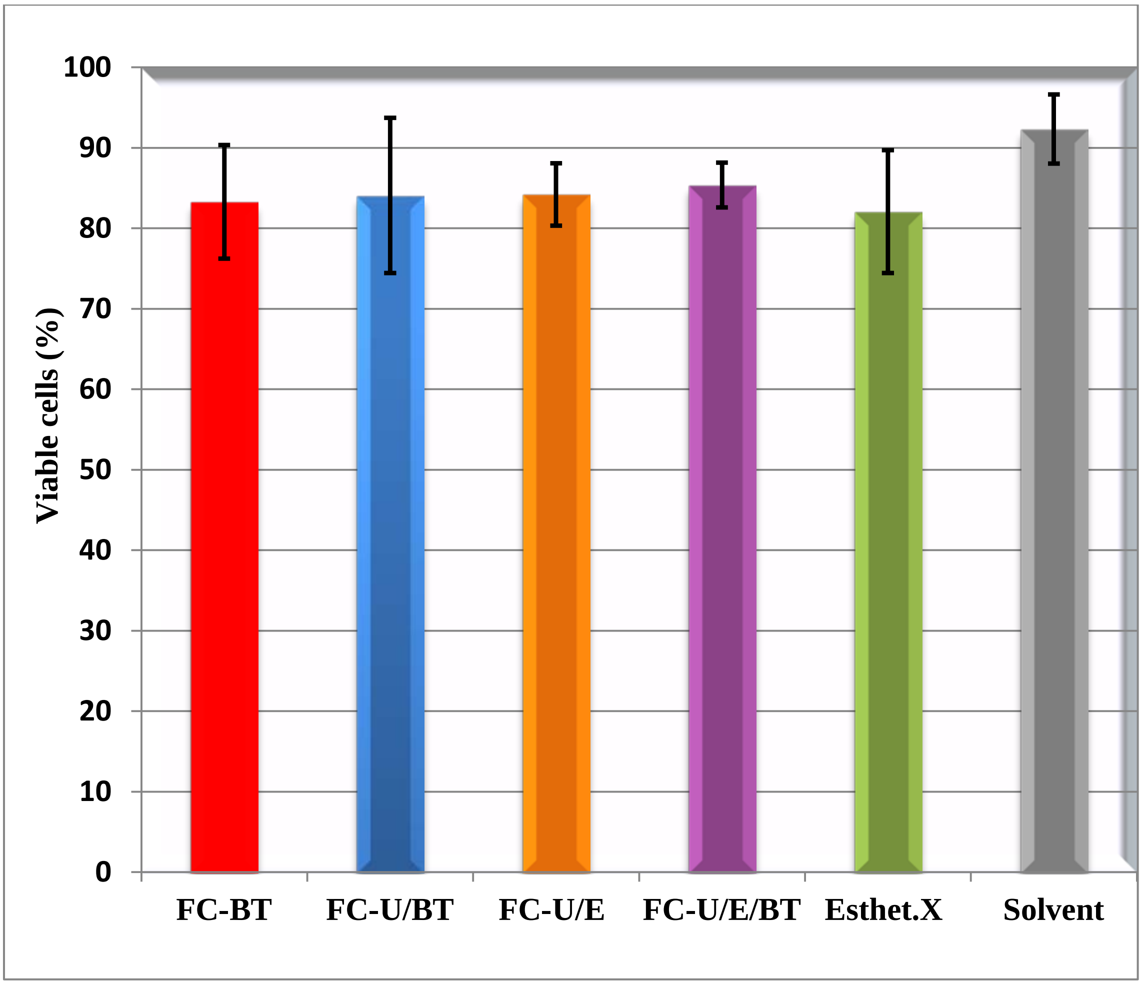


Figure 4.14 Mean viable cells of flowable composites

The histogram indicates that the data for percentage of viable cells does approximate the normal distribution curve and the Shapiro-Wilk normality test showed that p > .05 (Appendix XIh). Skewness and kurtosis values, were .403 and .557 respectively, and this is considered as acceptable. One-way ANOVA was carried out as is shown in Table 4.54. The post hoc Dunnett t (2-sided) test showed that there was no significant difference comparing the experimental UAM-based flowable composites against FC-BT (Exp-Cont) as is shown in Table 4.55. No significant difference was observed when UAM-based flowable composites were compared against Esthet.X flow (Com-Cont) as is shown in Table 4.56. The percentage of viable cell of solvent showed significant higher than both Esthet.X flow (Com-Cont) and FC-BT (Exp-Cont).

Table 4.54 Effect of various flowable composites on percentage of viable cells

Variable	n	Mean (SD) (%)	Confidence interval for mean		F-stat^a (df)	p value
			Lower bound	Upper bound		
FC-BT (Exp-Cont)	9	83.30 (7.06)	77.88	88.73	2.97 (5,48)	.020
FC-U/BT	9	84.08 (9.64)	76.66	91.49		
FC-U/E	9	84.21 (3.89)	81.22	87.20		
FC-U/E/BT	9	85.39 (2.78)	83.24	87.53		
Esthet.X flow (Com-Cont)	9	82.07 (7.64)	76.19	87.95		
Solvent	*9*	*92.35 (4.30)*	*89.04*	*95.66*		

a One-way ANOVA test at p = .05

Table 4.55 Multiple comparisons of mean viable cells of flowable composite against FC-BT (experimental control)

Flowable composite code	FC-BT (Exp-Cont)	Confidence interval for mean	
		Lower bound	Upper bound
FC-U/BT	.999	-7.02	8.57
FC-U/E	.998	-6.89	8.70
FC-U/E/BT	.934	-5.71	9.88
Esthet.X flow	.993	-9.03	6.56
Solvent	.018	1.24	16.84

Dunnett t (2-sided), p = .05

Table 4.56 Multiple comparisons of mean viable cells of experimental flowable composite against Esthet.X flow (commercial control)

Experimental flowable composite code	Esthet.X flow (Com-Cont)	Confidence interval for mean	
		Lower bound	Upper bound
FC-BT	.993	-6.56	9.03
FC-U/BT	.943	-5.79	9.80
FC-U/E	.927	-5.66	9.93
FC-U/E/BT	.703	-4.48	11.11
Solvent	.006	2.47	18.08

Dunnett t (2-sided), p = .05

Levene's test indicated that the homogeneity of variance can be assumed (Appendix IXg). The Tukey HSD post hoc test was chosen to identify the difference between flowable composites based on experimental UAM for viable cell at p = .05 as is shown in Table 4.57. There is no significant difference in viable cells among the experimental UAM-based flowable composites. There was also no significant difference between experimental UAM-based flowable composites and the solvent group.

Table 4.57 Multiple comparisons of the viable cells among UAM-based flowable composites and solvent

Experimental flowable composites code	FC-U/E	FC-U/E/BT	Solvent
FC-U/BT	1.000	.998	.082
FC-U/E	-	.999	.091
FC-U/E/BT			.205

Tukey HSD test, p = .05