



Evaluation of Adding Silane Monomer to Dental Composite Adhesive

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Abstract

Background: Silane monomers like 3-(Trimethoxysilyl) propyl methacrylate (3- TMSPMA) are commonly used in composites to promote bonding between a filler and the surrounding matrix in composite. **Aims:** This study aims to use a silane monomer in different concentration as a dilute to prepare photo-cured composite adhesive and evaluate the properties of the composite adhesive. Adding 0.1 wt.% graphene gives the best mechanical properties in a previous study; therefore, the same percentage was added in this study. **Materials and methods:** The silane monomer, 3-(Trimethoxysilyl) propyl methacrylate (3- TMSPMA), was added at different weight percentages to Bis-phenol A glycerolate dimethacrylate (Bis-GMA) -0.1 wt.% graphene, photocured, and nanoindentation mechanical properties determined. **Results:** of the tested concentrations, it was found that 40wt.% Bis-GMA GMA yielded the largest increase in hardness and indentation modulus while decreasing the viscosity. Tunable viscosity is a valuable characteristic of adhesive components designed to be applied in narrow spaces, such as dental cavities. While the mechanical properties which are represented in load-displacement curves indicate at the same concentration presented the lowest plastic deformation (high elastic deformation) with low viscosity.

Introduction:

Bis-phenol A glycerolate dimethacrylate (Bis-GMA) is a viscous crosslinker and a high molecular weight dimethacrylate monomer. However, the disadvantage of Bis-GMA is that it is challenging to manage due to its high viscosity (1). To mitigate the effect of the high molecular weight and viscosity, Bis-GMA is often combined with a low molecular weight monomer as a diluent to optimize the viscosity and mechanical properties (2). 3-TMSPMA has multiple functional groups and a low viscosity, which helps to serve as a suitable diluent with Bis-GMA. Since 3-TMSPMA contains silicon atoms and other organic compounds in the chemical structure, it is an active monomer capable of binding dissimilar organic and inorganic compounds and is dual reactive (3). The chemical structures of Bis-GMA, 3-TMSPMA, and CQ are shown in Figure (1) (a,b,c), respectively.

J. M. Antonucci et.al were used silane monomer to enhanced the bonding between the filler and matrix in the composite materials for filling the cavities in the process called silanization (4). Because of tri-alkoxy and silicon groups in the chemical structure, Silane monomer like 3-TMSPMA was used for bonding by cross-linked processes (5). Also, 3-TMSPMA was used to cover the surface of additives (silanization process) and then added to the matrix to improve the mechanical properties (6).

The efficiency of the photocuring process for the adhesive composite depends on the composition, including the type and composition of the monomer used(7–9). Furthermore, the composition of monomers (matrix) is important in affecting the properties of the adhesive, and the desired parameters for these properties depend on the application (10). The matrix can be a combination of two or more types of monomers with varying molecular weights (i.e., different viscosities), which can be an essential factor that impacts the efficiency of the adhesive (11).

In this study, graphene (G) was used as an additive to enhance the mechanical properties of this photo-cured composite

adhesive due to its superior properties even at low concentrations (12). A photo-cured composite adhesive composed of Bis-GMA, 3-TMSPMA, and graphene with enhanced mechanical properties was prepared. Several concentrations of Bis-GMA and 3-TMSPMA with 0.1 wt. % graphene was evaluated for changes in physical (viscosity) and mechanical properties (nanoindentation) of the photo-cured composite adhesive. Nanoindentation experiments were performed to determine the hardness and indentation modulus. Fourier Transform Infrared (FTIR) spectroscopy was used to determine the degree of conversion of double bonds into single bonds for uncured and cured materials (13,14). Rheometry was used to determine the viscosities of the composite adhesive.

Materials and Methods

1.Materials

A high molecular weight monomer Bis-GMA (519.59 g/mol Mw) was blended with 0.1 wt.% of graphene. 3-TMSPMA ($\geq 98\%$ pure, 248.35 g/mol Mw) was used as a dilute. Camphorquinone or (CQ, $\geq 96.5\%$ pure) was picked as a photoinitiator with (166.22 g/mol Mw.), and solvent was used like ethanol ($\geq 99.5\%$ pure, 46.07 g/mol Mw.). All chemicals were ordered from Sigma Aldrich, (St. Louis, MO USA). Graphene (G) bi-layers were purchased from Asbury Graphite (Asbury, NJ, USA) and used as an additive without further purification, as shown in Figure (1). Figure (2) was illustrating the chemical structures of the functional monomers, which are including Bis-GMA, 3-TMSPMA, and CQ.

2.Sample preparation of photo-cured composite adhesive

The photo-cured composite adhesive of Bis-GMA: 3-TMSPMA: 0.1 wt. % G were prepared by mixing the high viscosity (Bis-GMA), a low molecular weight diluent (3-TMSPMA), with 1 wt. % CQ, 8.9 wt. % ethanol, and 0.1 wt.% Graphene,

as further described in **Error! Reference source not found.**). Note the sample 50 wt. % Bis-GMA, 40 wt. % 3-TMSPMA, and 0 wt. % graphene serves as the control, labeled as “C” in the figures. A Mettler Toledo weight scale was used to weigh each component with an accuracy of ± 0.002 g. Graphene concentrations were prepared by mixing 3-TMSPMA, CQ, ethanol, and G using a mixing process, or shearing technique, for 1 minute to obtain proper dispersion (15), as shown in Figure (3). Bis-GMA was then added to the blends and then stirred using magnetic stirrer (1 hr. at 1000 rpm) then placed overnight in a dark room on a shaking table to homogenize at room temperature in amber glass (16). The total number of samples per each concentration are six ($n=6$), which include three samples ($n=3$) for liquid phase (before cured) and three samples ($n=3$) for solid phase (after cured) (17,18).

Liquid samples were placed in a cylindrical silicone mold with dimensions (5 mm diameter and 2 mm depth). The liquid was cured with a blue light source with average wavelength from 420-480 nm. This wavelength was used to activate the CQ photoinitiator (19). The light-tip was fixed at (1 cm) above the sample surface to ensure coverage of the silicone mold. After this, the cured specimens were stored in the dark at room temperature for one week before further testing.

Characterization

1.Nanoindentation

A Hysitron TriboIndenter TM (USA) was used to measure the cured specimens, hardness and indentation modulus (mechanical properties). A three-sided pyramidal shaped Berkovich diamond tip was used with a (20 nm) nominal average tip radius of curvature. The mechanical properties were calculated from the load-unload curve for each nanoindentation using standard analysis (20). Standard materials were used for calibration like Fused quartz which is used to calibrating the tip-area function, and a soft aluminum sample was used for tip-optic calibration (21). Three specimens ($n=3$) per each

blend were tested using load control nanoindentation at a load of 25,000 μ N in a 5x5-grid pattern (25 nanoindents) per each sample. A 20 μ m spacing between each nanoindent was used to avoid the influence of any residual stress from the last nanoindent as illustrated in Figure (4). The loading cyclic for each nanoindent included (1,500 second load, 10 second hold, and 1,500 second unload). The values of hardness and indentation modulus were obtained via the Hysitron software. The standard deviation and statistical average for the mechanical properties were calculated for each group.

2.Fourier transform infrared spectroscopy (FTIR)

The various compositions of Bis-GMA: 3-TMSPMA: 0.1 wt. % G were characterized using a Perkin Elmer FTIR. The parameters like wavenumber 4000-400 cm^{-1} , resolution 4 cm^{-1} and accumulations over 32 scans were used for each sample.

The spectra analysis was performed on each composition in both its liquid state and solid state (cured). The DC % for the carbon-carbon double bonds ($\text{C}=\text{C}$) was measured using the area under the aliphatic peak $\text{C}=\text{C}$ ($A_{\text{C}=\text{C}}$) at a wavelength of $\sim 1637 \text{ cm}^{-1}$ and comparing it with the area under the internal standard peak $\text{C}=\text{O}$ ($A_{\text{C}=\text{O}}$) at a wavelength of $\sim 1710 \text{ cm}^{-1}$. This was performed for both the solid (cured) and liquid phase (uncured) samples, and the DC % was calculated using the equation below (22). OriginProTM 2018b program was used to calculate the area of the peaks for the relevant bonds.

$$\text{DC\%} = \left(1 - \frac{(A_{\text{C}=\text{C}}/A_{\text{C}=\text{O}})_{\text{solid}}}{(A_{\text{C}=\text{C}}/A_{\text{C}=\text{O}})_{\text{liquid}}} \right) \times 100$$

3.Viscosity

The viscosity was evaluated of the liquid phase of Bis-GMA :3-TMSPMA: 0.1 wt.% G blends with a Malvern Kinexus Rotational Rheometer (Malvern, Worcestershire, WR14, UK). A parallel plate (20 mm diameter) with a gap of 0.5 mm was setup with a shear rate ($d\gamma/dt$)

ranging from 2.155, 4.642, and 10 (s-1) with 3 points per decade (23). The average of three samples was calculated for each blend were tested at room temperature. The data was presented on a (log-log base 10 scale).

4. Statistical Analysis

Outliers were defined as values outside two standard deviations from the mean. For nanoindentation, being outside two standard deviations of the respective means for either Hardness or for Indentation Modulus would classify a datapoint as an outlier for both parameters. All the results are expressed in terms of (mean \pm standard deviation). The results were analyzed with one-way ANOVA and then by Tukey HSD post hoc test at the significance level of $p < 0.05$ where p value is the probability value. Tabel (2) and (3) shown the mean \pm standard deviation for hardness and indentation modulus. Tabel (4) shown the mean \pm standard deviation for the FTIR test, while Table (5) shown the mean \pm standard deviation for the viscosity test. The standard error was presented as a bar in the graphs.

Results and Discussion

This study has its limitations. First, only a small sample size was used. Multiple data points were taken for each concentration group, though each group only had $n=3$ samples. Second, though previous research has been performed with silane monomers, little has been done with this composition of the additives and monomers. This contributed to novelty and innovation but was a limitation as it means this study points toward the value of its future works. The future directions and implications are discussed within.

1. Nanoindentation

Figure () shows the mechanical properties of Bis-GMA: 3-TMSPMA: 0.1 wt. % G adhesive blends. The hardness value of 50 wt.% Bis-GMA in 3-TMSPMA without any additives is 0.16 ± 0.02 GPa. The hardness values of Bis-GMA in 3-TMSPMA with 0.1 wt.% G range from

0.15 ± 0.02 GPa to 0.19 ± 0.05 GPa, as shown in Figure (a). The graphene groups were found to be statistically significant from one another except for 50 wt. % Bis-GMA without graphene to 60 wt. % Bis-GMA with 0.1% graphene and 50 wt. % Bis-GMA with 0.1% graphene to 40 wt. % Bis-GMA with 0.1% graphene. Graphene gives higher hardness because graphene has high mechanical properties (24). For the Graphene groups, all groups were except for 50:50 with 0% graphene to 60:40 with 0.1% graphene and 50:50 with 0.1% graphene to 40:60 with 0.1% graphene.

The indentation modulus of 50 wt.% Bis-GMA in 3-TMSPMA without any additives is 1.5 ± 0.3 Gpa. The indentation modulus of Bis-GMA in 3-TMSPMA with 0.1 wt. % G range from 1.8 ± 0.4 Gpa to 2.6 ± 0.2 Gpa as shown in Figure (b). For the Graphene groups, all groups were found to be statistically significant from one another except for 70:30 with 0.1% graphene to 40:60 with 0.1% graphene. Figure (c) shows each sample's load-displacement (unload) curves. The elastic deformation decreases with an increase in the amount of 3-Bis-GMA with 0.1 wt. % G. in TMSPMA because of increased in viscosity and intermolecular interactions, the same result was found in previous study (25).

2. FTIR

The DC and FTIR spectra for Bis-GMA: 3-TMSPMA: 0.1 wt. % G are shown in Figure (). The DC of Bis-GMA in 3-TMSPMA without any additives is 17.38 ± 4.15 as shown in Figure (a). The DC of Bis-GMA in 3-TMSPMA with 0.1 wt.% G range from 7.52 ± 9.98 to 14.49 ± 0.75 . For the graphene groups, there was no significance between the groups. The addition of 0.1 wt. % G to Bis-GMA: 3-TMSPMA blends seemed to decrease the DC%, though not significantly, as shown in Figure (a). Specifically, there was no significance between any of the groups for the DC% of C=C to C=O ($p > 0.05$).

Several factors can influence in the degree of conversion (DC), such as light intensity, light-tip size, wavelength,

amount of photoinitiator amount, inorganic filler (size and shape), and the types and composition of monomers in the matrix (26). The type and the composition of monomers can affect the degree of conversion, as the increased addition of high viscous monomer to low viscous monomer tends to decrease the degree of conversion (27,28). In this study however, there was no statistically significant difference with the addition of the graphene nor for the increased concentration of the Bis-GMA in the groups. Figure (6,b) shows the uncured and cured spectra of 50wt.% Bis-GMA: 3-TMSPMA: 0.1, which is used to calculate the area under each peak and applied to the equation. The spectra show how the area under the double bond peak decreases after being due to the conversion of double bonds to single bonds (29).

3. Viscosity

The viscosity of Bis-GMA in (3-TMSPMA with 0.1 wt.% G) is shown in Figure (). The viscosity was decreased with increased the weight percentage of silane monomer, which is low amount of double bonds in chemical structure. This drop was related to decrease the amount of Bis-GMA (high molecular weight) in the composite adhesive, so decrease the amount of double bonds.

As to be expected, the viscosity decreases slightly with increasing shearing rate, as shown in Figure (a) (30). The viscosity-shear rate relationship depends on the concentration of the high and low molecular weight monomers. For the lowest concentration of high molecular weight monomer, 40 wt. % Bis-GMA in (3-TMSPMA with 0.1 wt.% G), the decrease in viscosity with shear rate is the most significant, as compared with 50, 60, and 70 wt. % Bis-GMA in (3-TMSPMA with 0.1 wt.% G). As expected, the viscosity increases with increasing Bis-GMA concentration, since Bis-GMA is a high molecular weight monomer, as shown in Figure (b). Comparing the control of 50 wt.% Bis-GMA mixed with 40 wt. % 3-TMSPMA and the same monomer concentration but with graphene, 50 wt. % Bis-GMA with (3-

TMSPMA with 0.1 wt.% G), it is evident that the graphene also increases the viscosity of the blend. For Graphene blends, all groups were significantly different with the trend being a higher concentration of Bis-GMA to 3-TMSPMA resulting in a higher viscosity ($p < 0.01$).

Conclusion

Adding 3-TMSPMA to Bis-GMA decreased the viscosity of high molecular weight monomer (Bis-GMA), which is difficult to use in narrow application like filling the cavities. In dental application, it is necessary to control on the viscosity of the composite adhesive, to allow the adhesive layer to penetrate into the layers of the tooth (enamel and dentin). For this reason, silane monomer was added to Bis-GMA to physical property of the composite adhesive.

Adding 3-TMSPMA to Bis-GMA optimized the blends' hardness and indentation modulus (mechanical properties) as evaluated with nanoindentation. The addition of 40 wt.% Bis-GMA to 3-TMSPMA with 0.1 wt. % G increased in the hardness and indentation modulus but decreased the viscosity, which is important especially in narrow application like filling cavity. For the degree of conversion, the addition of 40 wt.% Bis-GMA to 3-TMSPMA with 0.1 wt. % G increased in the degree of conversion. In contract, adding 3-TMSPMA did not impact the degree of conversion (DC%) evaluated with FTIR. In future work, this composite adhesive should be applied to the surface of cow teeth and test the efficiency of this adhesive with the tooth layer (enamel and dentin).

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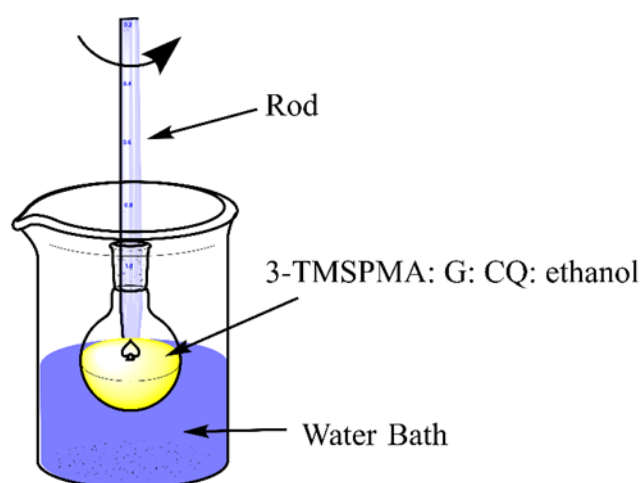


Figure (3): Mixing process or shearing technique to mix graphene into photo-cured adhesive systems.

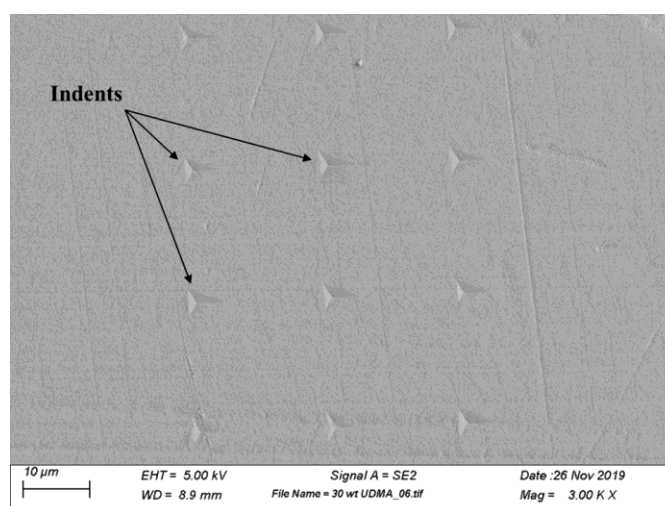
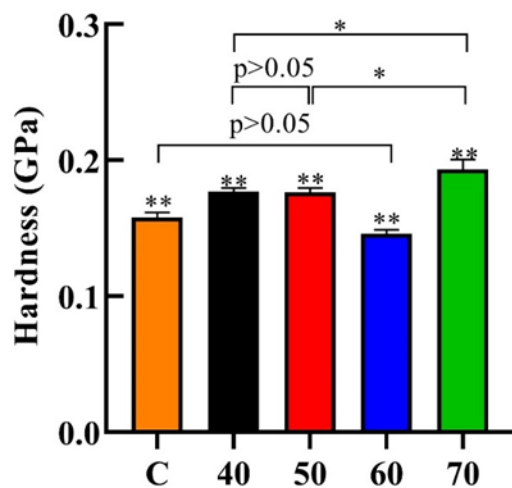
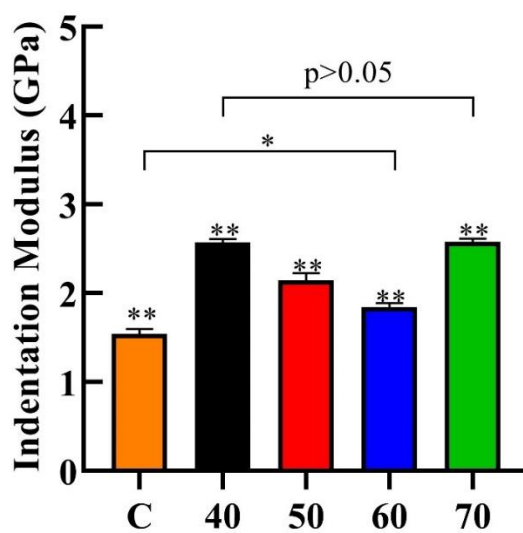


Figure (4): SEM of nano- indents



(a) wt. % Bis-GMA in 3-TMSPMA with 0.1 wt. % G



(b) wt. % Bis-GMA in 3-TMSPMA with 0.1 wt. % G

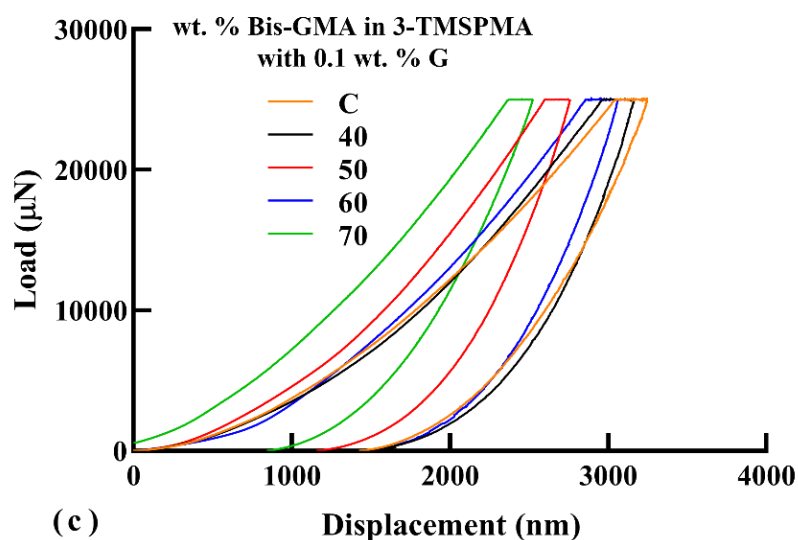
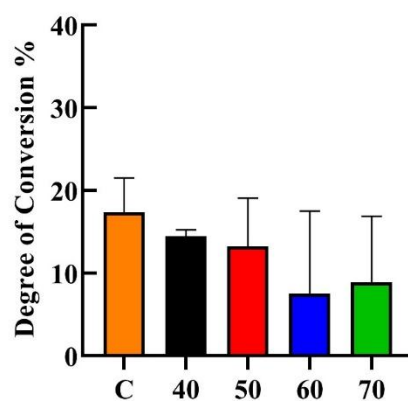


Figure (5): Nanoindentation results, (a) Hardness as a function of Bis-GMA wt. % in (3-TMSPMA with 0.1 wt % G), (b) Indentation Modulus as a function of Bis-GMA wt. % in (3-TMSPMA with 0.1 wt % G) (c) load-displacement curves for each sample (* indicates a significant difference with $p < 0.05$ and ** indicating $p < 0.01$).



(a) wt. % Bis-GMA in 3-TMSPMA with 0.1 wt. % G

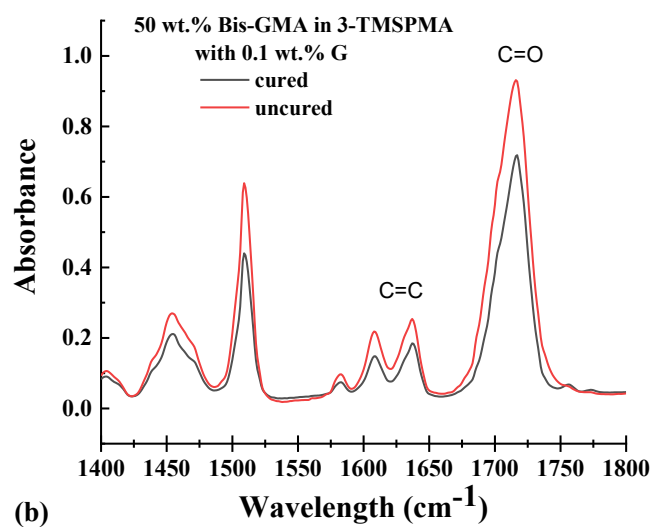
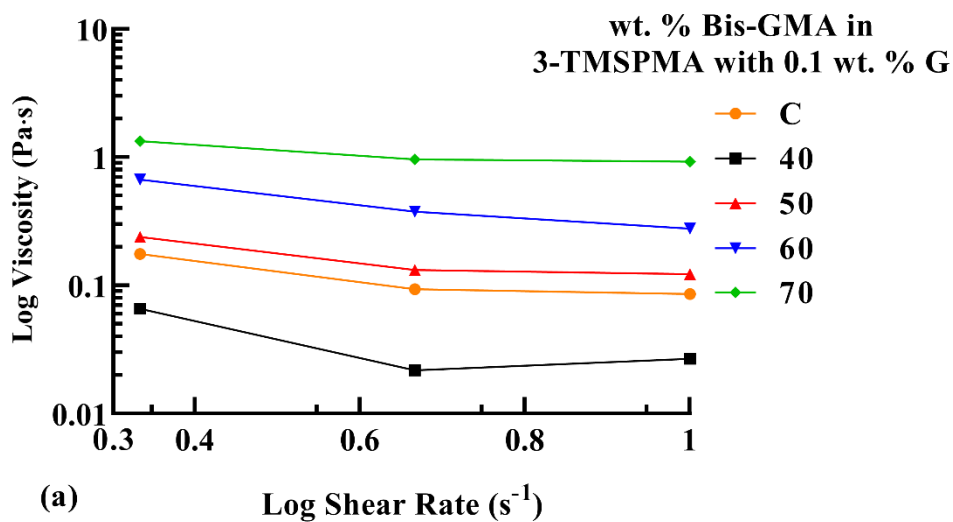
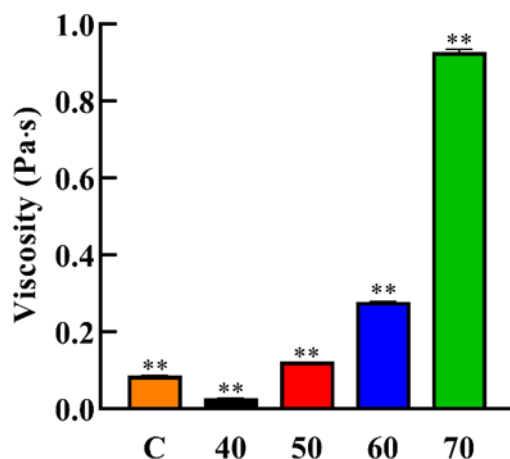


Figure (6): FTIR results, (a) the degree of conversion % vs. wt. % Bis-GMA: 3-TMSPMA: 0.1 wt. % G; (b) FTIR spectra of uncured and cured samples of the blends.





(b) wt. % Bis-GMA in 3-TMSPMA with 0.1 wt. % G

Figure (7): (a) viscosity of Bis-GMA: 3-TMSPMA with 0.1 wt. % G blends as a function of shear rate; (b) viscosity vs. wt. % Bis-GMA in 3-TMSPMA with 0.1 wt. % G at shear rate 10 /s. Statistical significance of differences is given by ** indicating $p < 0.01$.

Table (1): Photo-cured composite adhesive system composition (wt.%)

Bis-GMA (wt. %)	3-TMSPMA (wt. %)	G (wt. %)	CQ (wt. %)	Ethanol (wt. %)
50	40	0	1	9
40	50	0.1	1	8.9
50	40	0.1	1	8.9
60	30	0.1	1	8.9
70	20	0.1	1	8.9

Table (2): mean \pm standard deviation values for hardness test

Hardness			
Group 1			
Samples Name	Mean	Stdve	SEM
50:50, 0% Graphene	0.180941803	0.052707572	0.006255238
70:30, 0.1% Graphene	0.125199841	0.0390154	0.004696902
60:40, 0.1% Graphene	0.161960972	0.044848569	0.005285455
50:50, 0.1% Graphene	0.174942536	0.037165383	0.004474186
40:60, 0.1% Graphene	0.139187276	0.031754077	0.004169515
Group 2			
50:50, 0% Graphene	0.157916231	0.022486859	0.003600779
70:30, 0.1% Graphene	0.192645805	0.04535313	0.007082969
60:40, 0.1% Graphene	0.146631929	0.02459376	0.002939517
50:50, 0.1% Graphene	0.176429554	0.028086664	0.003265008
40:60, 0.1% Graphene	0.176843014	0.023587072	0.002760658
Group 3			
50:50, 0% Graphene	0.180941803	0.052707572	0.006255238
70:30, 0.1% Graphene	0.126413776	0.037557217	0.004588344
60:40, 0.1% Graphene	0.137817931	0.018817347	0.002217646
50:50, 0.1% Graphene	0.117483833	0.013460019	0.001831677
40:60, 0.1% Graphene	0.080310587	0.007734731	0.001140424

Table (3): mean \pm standard deviation values for indentation modulus test

Indentation Modulus			
Group 1			
Samples Name	Mean	Stdve	SEM
50:50, 0% Graphene	2.89366012	0.739401346	0.085378713
70:30, 0.1% Graphene	2.495426687	0.462735996	0.0565322
60:40, 0.1% Graphene	2.333087394	0.594877036	0.070598916
50:50, 0.1% Graphene	2.864986493	0.760156685	0.090214001
40:60, 0.1% Graphene	2.447026644	0.474058754	0.061717193
Group 2			
50:50, 0% Graphene	1.579356721	0.33304981	0.05078962
70:30, 0.1% Graphene	2.5777166	0.228216558	0.036084206
60:40, 0.1% Graphene	1.842162771	0.388597505	0.046446286
50:50, 0.1% Graphene	2.12839008	0.73311681	0.084653038
40:60, 0.1% Graphene	2.568920635	0.311773266	0.036242906
Group 3			
50:50, 0% Graphene	2.89366012	0.739401346	0.085378713
70:30, 0.1% Graphene	2.672081971	0.382625465	0.046062693
60:40, 0.1% Graphene	2.646547746	0.162493734	0.019284458
50:50, 0.1% Graphene	2.515022782	0.131744676	0.017764448
40:60, 0.1% Graphene	1.49817317	0.112602235	0.016424724

Table (4): mean \pm standard deviation values for FTIR test

FTIR			
Group 1			
Samples Name	Mean	Stdve	SEM
50:50, 0% Graphene	35.91577458	18.08074846	10.43892499
70:30, 0.1% Graphene	8.997852613	1.729713355	0.998650471
60:40, 0.1% Graphene	14.85676908	8.935796107	5.159084288
50:50, 0.1% Graphene	24.46492119	1.897956038	1.09578543
40:60, 0.1% Graphene	28.05851739	4.707285091	2.717752314
Group 2			
50:50, 0% Graphene	17.37901771	7.19147111	4.151997781
70:30, 0.1% Graphene	8.884274114	13.86945538	8.007533796
60:40, 0.1% Graphene	11.7388518	16.50314524	9.528095349
50:50, 0.1% Graphene	13.23389729	10.1231849	5.844623529
40:60, 0.1% Graphene	14.49317609	1.293359167	0.746721263
Group 3			
50:50, 0% Graphene	35.91577458	18.08074846	10.43892499
70:30, 0.1% Graphene	12.70463777	9.502450829	5.486242544
60:40, 0.1% Graphene	22.03180517	1.058596242	0.611180825
50:50, 0.1% Graphene	24.3178173	3.314292574	1.91350771
40:60, 0.1% Graphene	47.31629268	8.672662036	5.007163761

Table (5): mean \pm standard deviation values for viscosity test

Viscosity			
Group 1			
Samples Name	Mean	Stdve	SEM
50:50, 0% Graphene	34.4622403	8.67968249	0.121516041
70:30, 0.1% Graphene	121.9054595	21.33116551	0.298608249
60:40, 0.1% Graphene	74.31114465	16.37686164	0.22927698
50:50, 0.1% Graphene	47.74042649	1.284303062	0.018390399
40:60, 0.1% Graphene	25.78197334	1.27977986	0.018188162
Group 2			
50:50, 0% Graphene	85.85057232	11.09600435	0.155344682
70:30, 0.1% Graphene	926.8326797	142.0454064	8.120195409
60:40, 0.1% Graphene	277.7993266	37.56162337	1.25836173
50:50, 0.1% Graphene	122.8555619	22.34500775	0.31459363
40:60, 0.1% Graphene	26.84478695	15.03212569	0.210450601
Group 3			
50:50, 0% Graphene	34.4622403	8.67968249	0.121516041
70:30, 0.1% Graphene	151.0428624	11.38265409	0.218052106
60:40, 0.1% Graphene	25.49128518	23.83683143	0.333716975
50:50, 0.1% Graphene	18.21744257	13.44253098	0.188196186
40:60, 0.1% Graphene	8.512447146	0.977382341	0.013855567

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