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Synthesis and characterizations of new dental composites using calciumfluoro aluminosilicate glass

Volume 2 Issue 3 - 2018

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Received: February 09, 2018 | **Published:** May 09, 2018

Abstract

New dental composites prepared from Bis GMA/TEGDMA with different ratios of Calcium fluoroaluminosilicate glass used as filler. The particle size of Calcium fluoroaluminosilicate glass is (1¬10)µm. The visible light of 480 nm and duration time 40s was used in polymerization method. The physical and mechanical properties of the dental material were studied. These properties included, shrinkage; depth of cure, thermal expansion coefficient, Degree of conversion and diametral tensile strength (DTS). The static flexural strength and flexural modulus were also measured using a three-point bending set up according to the ISO-4049 specification. The volumetric shrinkage values was found between 2.6% to 2.9%,The degree of conversion was found to be 79.57% to 68.27%, the depth of cure is 2.6to3.3mm, and thermal expansion coefficient between 45.5 and 54.6×10^{-6} ºC. For the mechanical properties, the highest flexural strength 90.35 MPa, flexural modulus (11.29 GPa), and DTS (54.27 MPa). All tested composites complied with the requirement of ISO 4904: 2008 for all tested samples and coplied with ANSI/ADA specification No. 27 for cure resins.

Keywords: dental composites, calcium fluoroaluminosilicate glass, mechanical properties, restorations, methacrylate chemistry, glass draw, camphorquinone

Abbreviations: DMAEMA, dimethylaminoethylmethacrylate; DTS, diametral tensile strength; FS, flexural strength; DOC, depth of cure

Introduction

In Dentistry, posterior class I or II restorations require composites that show high mechanical properties whereas anterior restorations need composites that have superior aesthetics.[1] Composite material consist a resin matrix, filler and matrix-filler coupling. The organic matrix based on methacrylate chemistry, especially cross-linking dimethacrylates.[2] Much effort has been devoted to investigations of the relationship between filler particles in composite resins and their properties. Dental properties for composite significantly associated with values of mechanical properties, biocompatibility, color stability and would therefore be expected to be associated with clinical success of the restoration, influenced by numerous factors, including the a type and size of fillers,[3] RBC shade,[4,5] photo initiator type and concentration,[6] refractive index mismatch, light irradiation source and irradiation duration[7,8] intensity of light and for a given irradiance period.[9] Because of major influence of the fillers on the physical properties, the classification of dental filling composites is based on the type and the particle size of fillers.[10] The filler content seems to be the most important factor in the determination of the properties of composites.[11] Calcium fluoroaluminosilicate glass draw the attention of researchers due to its comparable properties such as rigidity which is near to the bone rigidity and its ability to release the fluoride ions which will sterilize the teeth on using this type of fillers in teeth filling.[12–15] The physical and mechanical properties of dentalcomposites have improved significantly. However, dentalcomposites possess two undesired properties, polymerization shrinkage and stress, which contributes to clinical problems in the integrity of the restoration margin.[16] Flexural strength, is the material property that implies the quantity of flaws within the material that may have the potential to cause catastrophic failure during loading. Complex geometric forms of dental composite restoratives can lead to the development of the tensile forces during mastication. The purpose of this study is to evaluate the effects of Calcium fluoroaluminosilicate glass as filler level on selected properties of ten experimentally prepared composite resin series, each of which filled with different filler compositions. The properties examined were depth of cure, strength shrinkage, and degree of conversion, flexural strength, and DTS.

Material and methods

Preparation of composites

Ten experimental light-activated composites were tested, containing the resins Bis–GMA (Sigma Aldrich (UK))/TEGDMA (Sigma Aldrich (UK)) in a w/w ratio of 70/30 as the base resins. Camphorquinone (CQ, Aldrich (UK)) and dimethylaminoethylmethacrylate (DMAEMA, Wako Pure Chemical Industries) were added to resin as catalysts. Calcium fluoroaluminosilicate glass was synthesized and sintered in our laboratory[15] (Average size of particles: 1.2µm), treated with silane-coupling agent with loaded ratio of 76 wt% to resin. The compositions of the studied dental composites are shown in Table 1.

Preparation of the tested samples

Dental properties were determined according to the specification standard for composite (ISO 4049:2000).[17] pecimen where put inside mold fortest, then, they were irradiated for 40s. The light-curing unit (SDS Kerr Optilux 501 Halogen Bulb, USA) had an exit-window diameter of 8 mm while the light intensity used was 500 mW/cm^2 with the curing tip placed 1 mm from the glass plate.

Shrinkage strain and rate of shrinkage

The bonded disk technique[18] was used to measure the shrinkage of the samples during light curing. The samples were cured for 40 sec. The shrinkage was measured continuously and total shrinkage strain of the samples was measured500 s after starting the light radiation, at which time the contraction had plateaued-out.

Degree of conversion

Degree of conversion of the composites was measured using FTIR spectroscopy (Brouker). The samples were placed between two polyethylene films, pressed to form a very thin film (0.10 mm) and the absorbance peaks of the uncured samples were obtained. The samples were then light cured for 40sec and the peaks were collected for the cured samples. For calculating Degree of conversion (DC %), the aliphatic C=C (peak at 1638 cm^{-1}). And that for the aromatic C-C (peak at 1608 cm^{-1}) were compared in each spectrum before and after the polymerization reaction. The ratio between the two peaks was then calculated as previously described,[19] the degree of conversion was then calculated as follows:

$$DC\% = \left(1 - \frac{\left(1636/1608\,cm^{-1}\right) peak\ area\ after\ curing}{\left(1636/1608cm^{-1}\right) peak\ area\ befor\ curing}\right) \times 100$$

Depth of cure

Three specimens from each group were condensed into a stainless steel mold; each specimen was of 4 mm in diameter and 10 mm in depth. The ISO defines depthofcure as 50 percent of the length of the composite specimen after the uncured material is removed spatula with a plastic.[17]

Thermal expansion coefficient

Thermal expansion coefficient of cured composites was measured using a Thermo Mechanical Analyzer in the temperature range from 20 to 70ºC. Specimen disks approximately 7 mm in diameter and 3-5 mm in thickness were fabricated in an stainless-steel mold, The specimens were then light cured for 40sec, after cured the Specimen were immersed in distilled water at 37ºC for 24hrs.The linear coefficient of thermal expansion (a) was calculated according to the well-known formula:

$$\alpha = \frac{\Delta L}{L_0 \Delta T}$$

Where L_0 is the original height of the specimen and ($\Delta L/\Delta T$) is the slope of the thermal expansion curve measured as the tangent to the curve at a given temperature, or determined as the straight line between two temperatures.

Mechanical properties

Measurement of flexuralstrengthandflexural modulus

Flexural strength of the composites was conducted according to the 3-point bending method suggested in ISO 4049. The bar specimens (2mm×2 mm×25 mm) were prepared in stainless-steel rectangular mold utilizing the light curing unit. An overlapping regime was applied to irradiate the whole specimens on both sides (40s for each irradiation). Having stored in deionized water at37 ºC for one day, the three-point bending test was performed on the specimen s using universal testing machine at a cross-head speed of 1 mm/min. The flexural strength (FS) in MPa was calculated as:

$$FS = \frac{3pL}{2bd^2}$$

Where p stands for load at fracture (N), L is the span length (20 mm), and b and d are, respectively, the width and thickness of the specimens in mm. The elastic modulus was determined from the slope of the initial linear part of stress–strain also curve. The flexural modulus of the unfilled matrix resin was also measured in the same conditions.

Measurement of diametral tensile strength (DTS)

Diametral tensile strength (DTS) test was performed adopting the procedure of ANSI/ADA specification No.27forcure resins. The composite pastes were inserted into a cylindrical stainless-steel mold with the internal diameter of and height of 3 mm and cured for 40 s from both sides using the light-curing unit. The specimens were removed from old and stored in deionized water for one day at 37ºC prior to the test. A universal testing machine (SMT-20, Santam, Iran) was utilized for the test at across-headspeedofmm/min. The DTS (MPa) was then calculated according to the following equation:

$$DTS = \frac{2p}{\pi DL}$$

Where p is the load at fracture (N), D (mm) and L (mm) are diameterand height of specimens, respectively.

Result and discussion

Shrinkage strain

Three specimens were Figure 1 prepared for each composite materials tested. Mean values and standard deviation of the shrinkage of the materials tested are shown in Figure3.1 the values varied from 2.65% for M10 to 2.97 for M2. We determine shrinkage until equilibrium about 400sec.The time of maximum shrinkage rate of polymerization for all samples at~3s the results show that the shrinkage strain of composites are about a half that of the methacrylate based composite. Compared to the results of shrinkage strain obtained, which range between 2.65% for M10 and 2.97± 0.06 for M2 (Maximum value), with some recent research 2.64±0.11%, Compo glass F 2.96±0.02%, Fuji II LC 3.25±0.17% and Filter P60 1.99±0.06%[20] (Figure 2).

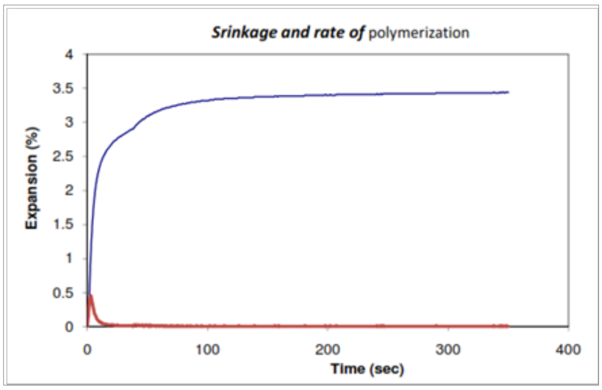

Figure 1 shrinkage curve and rate of polymerization for M2 composite test.

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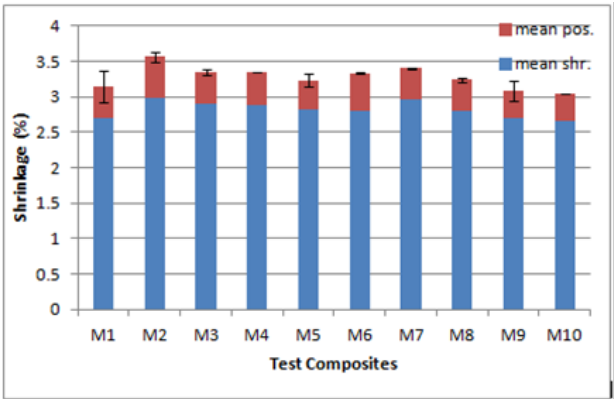

Figure 2 The mean shrinkage and post shrinkage of composite materials tested.

Depth of cure (DOC)

Four specimens of each tested composites group were done. The length of the cured sample is measured to the nearest (0.0l mm) using a micrometer. Regarding the results of the present study, all samples at 40sec curing time passed the requirements of ISO 4049:2000, as all records of DOC are more than 2.5 mm requested; which is in fact half of the actual length measured by the micrometer this is explained according to Fan 2009.The result of DOC reported in Figure 3; DOC between 2.6 to 3.4 mm this low DOC (< 3 mm) in some tested composites, Fillers may have contributed to the darker shade of the composite. Fillers may have contributed to the darker shade of the composite. This is a possible explanation for the low depth of cure values. Another possible explanation is the agglomeration of the filler particles that could have impaired light transmission into the matrix and resulted in shallow curing depths

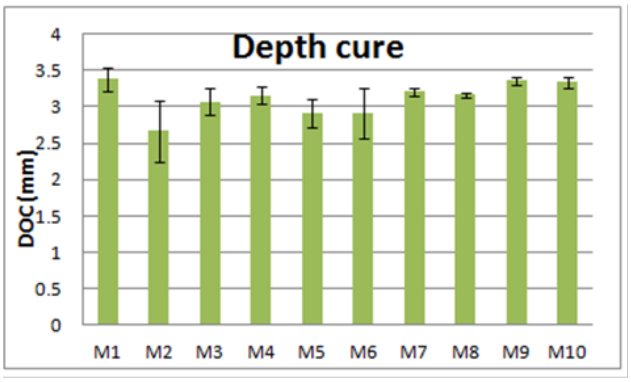

Figure 3 Depth of cure of tested samples.

Degree of conversion (DC)

Figure 4 shown the FTIR spectrum for M1 test composites and varies between uncured and cured for rang 1580cm^{-1} to 1660cm^{-1}. The degree of conversion depends on the wavelength of the light, exposure time and composition of light-activated resin-based material.

Thermal expansion coefficient (CTE)

In Table 1 the results of the determination of the coefficient of thermal expansion are displayed. The Figure 5 shows the lowest coefficients for M10, which exhibit almost equal values (45×10^{-6} °C^{-1}). Which are more heavily filled resins. A strong inverse correlation has been reported between filler content and the coefficient of thermal expansion.[21,22] Clinically, it is desirable for the thermal dimensional changes of restorative materials to approximate those of tooth structure to control marginal leakage and to maintain enamel bonding. The true clinical picture is quite complicated because thermal equilibrium seldom obtained in the oral environment.

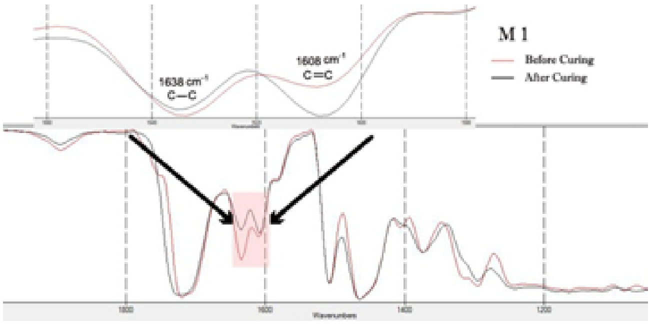

Figure 4 FTIR spectrums for M1 test composites.

Table: 1 Composition (W %) of Calcium-Fluoroaluminosilicate Glass

Glass	SiO_2	Al_2O_3	CaF_2	Al_2PO_4	AlF_3	NaF
M1	22	18		22	15	23
M2	22	19	10	39	13	7
M3	29	16.6	34.2	9.9	5.3	5
M4	35	25	20	8	6	6
M5	39.52	23.6	13.65	3.62	9.7	9.91
M6	24.3	27.5	14.0	19.1	15.1	
M7	33.9	17.5	8	15	10	15.6
M8	56.5	33.5				10
M9	48.9	29.1	15			7
M10	36.3	22	12	9	14.3	6.4

Flexural strength (FS)

Flexural strength (FS) Figure 6 is measures of the material strength under different force condition. The stronger material has the higher value of compressive strength and flexural strength. International Organization for Standardization (ISO) specification 4049 for polymer based restoratives classifies dental polymer based restorative materials into two different groups all materials investigated showed higher mean flexural strength values than ones recommended by the ISO suggesting that these materials can be used as direct restorative materials[16] observed a mean FS value for composite, which in turn were higher than the mean FS value reported by (92 MPa).[23] Herculite XRV Ultra exhibit FS mean value (83.37 MPa) less than the mean values reported by Tran, 2010 (137 MPa).[24] Also Tetric Evo Cerm exhibit mean value (70.21 MPa) which was less than literature means (98.61MPa)[25,26] Flexural modulus describes stiffness, a measure of the resistance to deformation under load of the material, with a high number indicating greater stiffness. When compared to the module of human enamel and dentin, which are about 84 GPa and 14 GPa, respectively, resin composites had much lower values Figure 7.

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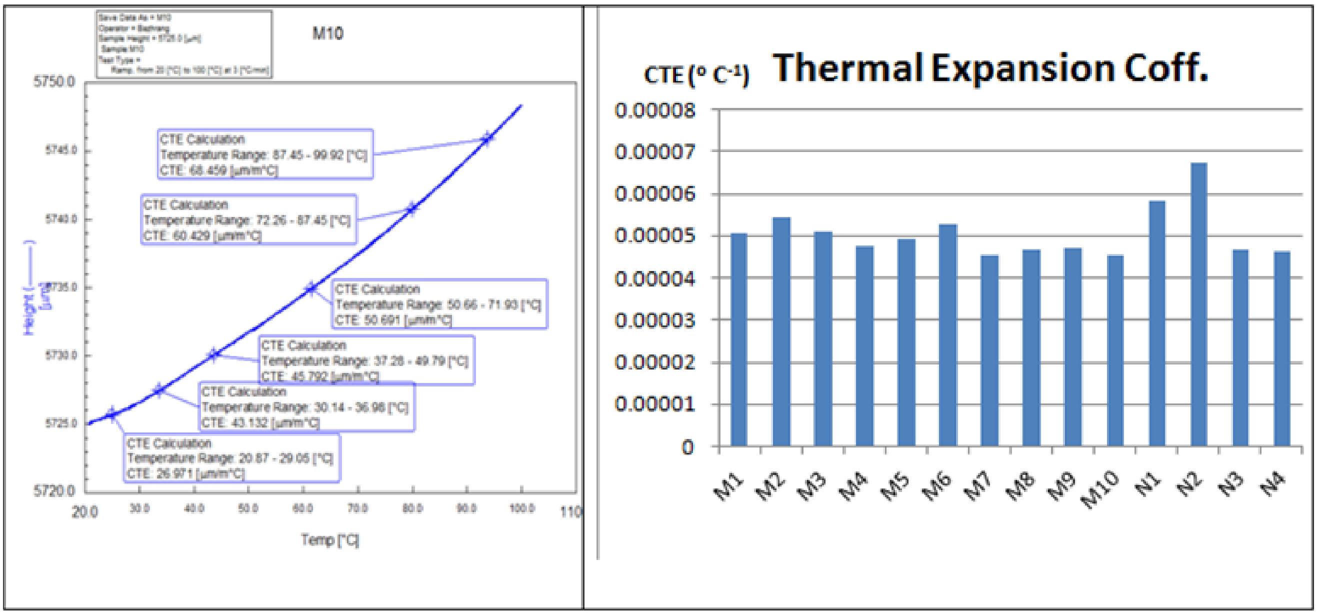

Figure 5 CTE of composite materials tested.

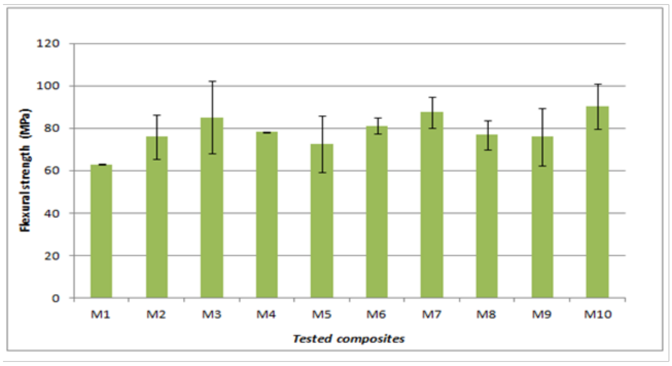

Figure 6 Flexural strength of tested samples.

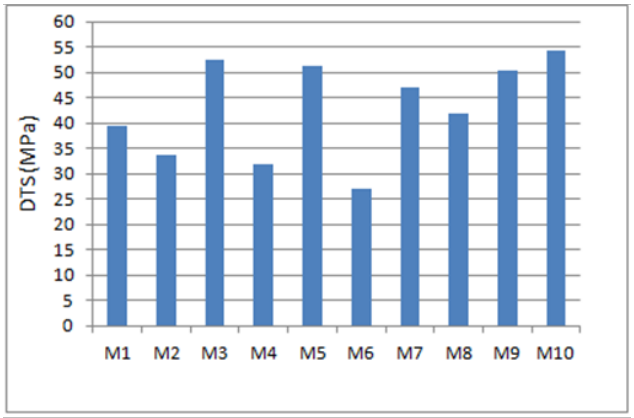

Figure 8 Diametric tensile strength of Tested Samples.

Diametric tensile strength (DTS)

Mean values and standard deviation of the DTS of composites tested are shown in Figure 8. The values varied from 27.2 MPa for M6 to 54.27 MPa for M10.

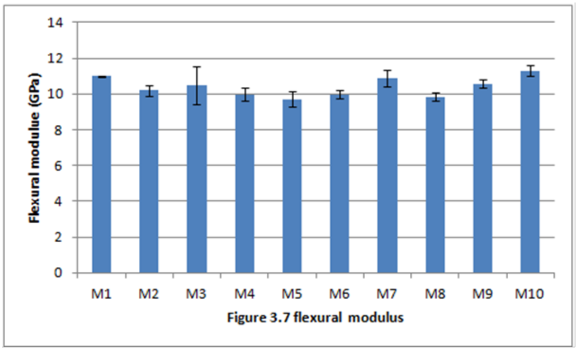

Figure 7 Flexural modulus describes stiffness.

Conclusion

We conclude from the above studied that all physical measurements were within ADA specification and all prepared composites for teeth filling were passed the requirements that to be used for dental purposes

Acknowledgements

The authors would like to thank the Iraqi Ministry of Higher Education and Research for offering Rafid the scholarship to do this work.

Conflict of interest

The authors declare that they have no conflict of interest.

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