

# Physical Properties of a New Low Shrink Resin

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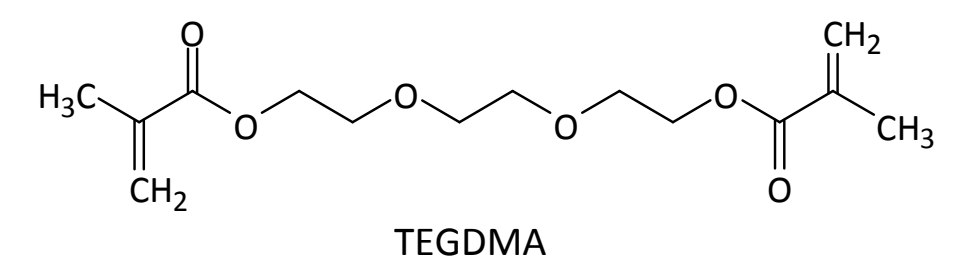
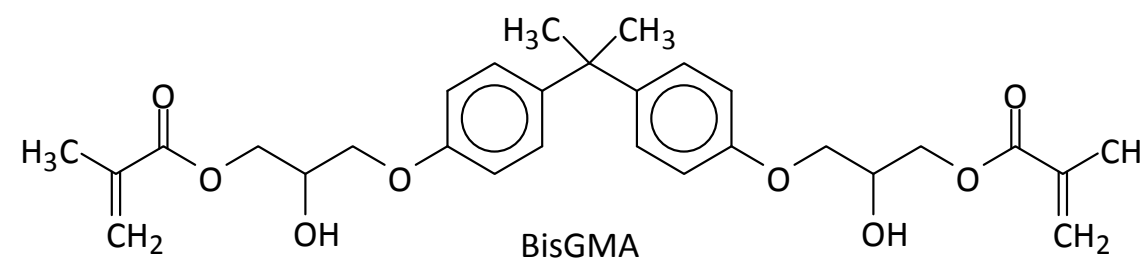
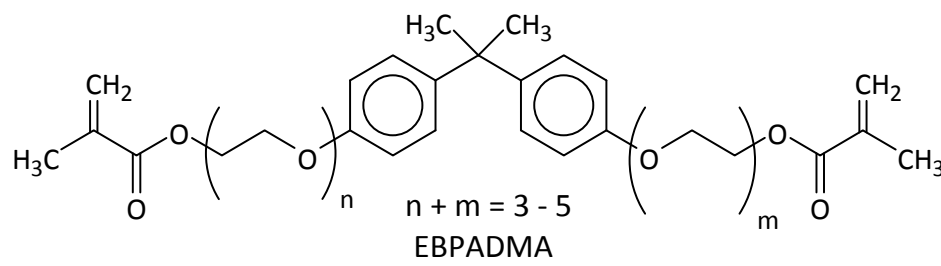
## INTRODUCTION:

The curing of dental composites induces polymerization shrinkage stresses, which lead to strain at the biological interface. The primary approach of dental researchers to overcome this inherent problem has been to optimize the various ingredients used to make these materials. This research focuses on improving the properties of dental composites via the introduction of a new monomer referred to as X-852-0000. The test methods used to evaluate X-852-0000 as a prospective dental monomer include degree of conversion, percent volumetric shrinkage, ultimate transverse and flexural strength.

The average degree of conversion for the homopolymer of X-852-0000 was 32% higher than a BisGMA/TEGDMA blend with an identical photoinitiator package and curing protocol. In percent volumetric shrinkage analysis, X-852-0000 averaged 1.68% while the BisGMA/TEGDMA averaged 5.32%. Ultimate transverse strength testing revealed that X-852-0000 is stronger and tougher than a BisGMA/EBPADMA/TEGDMA blend. Similar testing indicated that X-852-0000, when used as an additive in BisGMA/TEGDMA systems, also increased strength and toughness.

## MATERIALS:

The monomers used in this research include ethoxylated bisphenol A dimethacrylate (EBPADMA, Esstech Inc.), bisphenol A diglycidyl ether dimethacrylate (BisGMA, Esstech Inc.) and triethylene glycol dimethacrylate (TEGDMA, Esstech Inc.). Structures of each of these materials are shown in (Figure 1). Various formulations of these traditional dental materials were analyzed conjointly with X-852-0000 to serve as a basis for comparison.

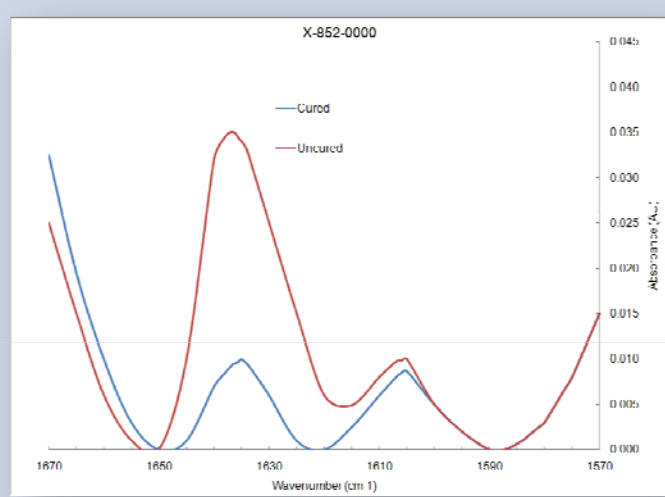


## METHODS:

### Degree of Conversion by FTIR-ATR:

Camphorquinone (CQ, 0.3 wt%, Esstech Inc.) and ethyl 4-dimethylaminobenzoate (EDAB, 0.8 wt%, Aldrich) were used as the visible light (470 nm) photoinitiator system without purification.

After the spectrum of the uncured resin was obtained, the same specimen was photo-cured using a conventional dental light-curing unit (Optilux 501, Demetron Research, Danbury, CT) by applying a 20-second exposure at a tip-to-resin distance of 2 mm. Five minutes after deactivation of light exposure, another infrared spectrum was obtained of the polymerized material. The resin was not removed from the ATR element during the process. The area ratios were then converted into degree of conversion data using methods common in the dental literature. [1, 2]



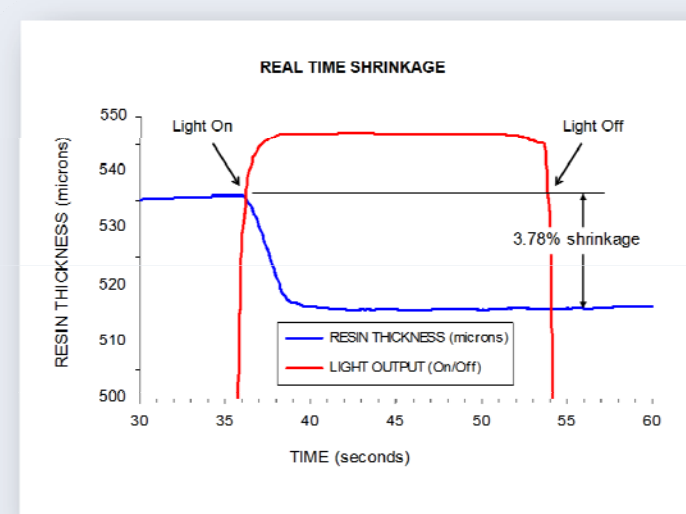
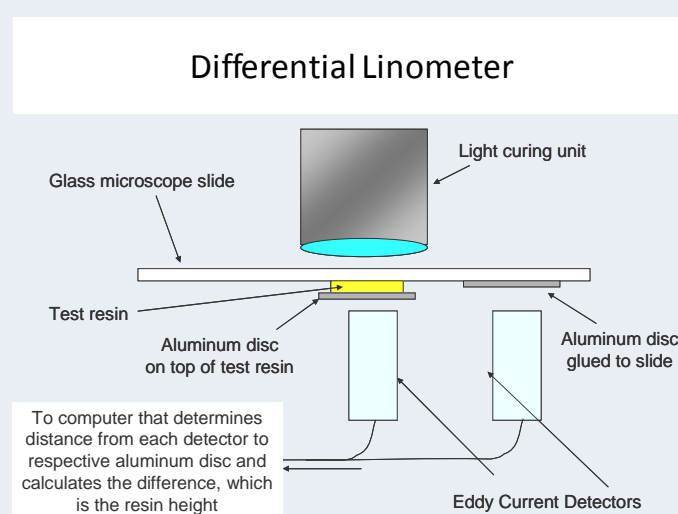
## RESULTS:

### Degree of Conversion (%)

BisGMA / TEGDMA n = 5		X-852-0000 n = 40	
Mean	SD	Mean	SD
53.64	0.91	85.31	5.04

### Volumetric Polymerization Shrinkage by Differential Linometer:

Volumetric polymerization shrinkage data was obtained using a differential linometer modeled after the device designed by de Gee et al. [3] Samples were photoinitiated and cured with the same system and protocol detailed above. The differential linometer system uses a photo-detector to monitor when the light curing unit is activated and deactivated. The eddy current data and the photodetector data were simultaneously recorded for each run. The samples used in the volumetric polymerization shrinkage analyses are identical to those described previously in the degree of conversion analyses. The light unit used for photo-curing in the degree of conversion testing was also used for the volumetric percentage shrinkage evaluation and was activated for the same exposure duration of 20 seconds. The height of the specimen disc (in microns) as well as the indicator of light curing activity is simultaneously displayed. The shrinkage value was determined 5 minutes after light deactivation.



### Volumetric Polymerization Shrinkage (%)

BisGMA / TEGDMA n = 4		X-852-0000 n = 40	
Mean	SD	Mean	SD
5.32	0.73	1.68	0.25

### Ultimate Flexural Strength Testing of X-852-0000 in BisGMA and TEGDMA blends:

To evaluate its use as an additive, X-852-0000 was added in 10 wt% and 25 wt% to a 70:30 wt% blend of BisGMA and TEGDMA. Irgacure 500 (3.0 wt%, Ciba) was used as a UV light photoinitiator system for this round of testing. Blend compositions of the control BisGMA/TEGDMA resin along with added X-852-0000 are summarized below. Test specimens were cured in syringes with an average diameter of 4.6 mm. Five test specimens were made from each blend. The specimens were tested in transverse flexural mode on a Shimadzu Autograph AGS-J universal tester with a 5 kN load cell. The span of the universal tester was set at 30 mm with a crosshead speed of 100 mm/min.

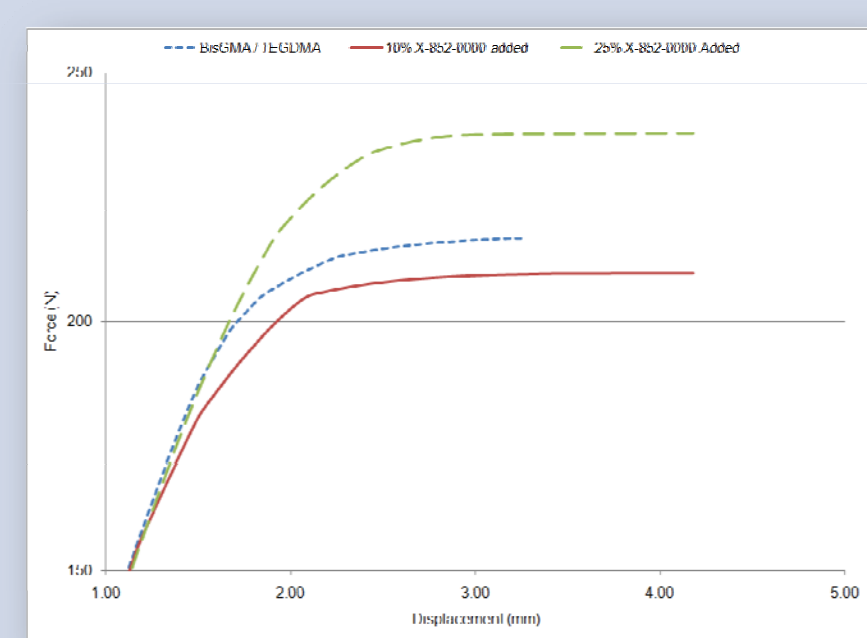
### Sample Compositions

	BisGMA / TEGDMA	10 wt% X-852-0000	25 wt% X-852-0000
X-852-0000 (g)	0	10	25
BisGMA (g)	67.9	60.9	50.4
TEGDMA (g)	29.1	26.1	21.6
Irgacure 500 (g)	3	3	3



### Ultimate Flexural Strength Testing

	BisGMA / TEGDMA		10 wt% X-852-0000		25 wt% X-852-0000	
	Mean	SD	Mean	SD	Mean	SD
Elastic Modulus (GPa)	3.18	0.086	3.16	0.088	3.15	0.082
Maximum Force (N)	216.7	23.99	209.8	37.36	238	8.38
Energy to Break (J)	0.299	0.161	0.329	0.026	0.481	0.195



### Ultimate Transverse Strength Testing of X-852-0000:

The procedure developed by Lindsog, et al [4] was used to measure the ultimate transverse strength (UTS) of the sample materials. In this testing, a BisGMA/EBPADMA/TEGDMA was blended in a weight percentage of 50:25:25 respectively to represent variations in commercial dental composite formulations. The BisGMA/EBPADMA/TEGDMA was photo-initiated with CQ (1 wt%, Esstech) and dimethylamino ethylmethacrylate (2 wt%, 98% purity, Aldrich). The X-852-0000 was photo-initiated with 0.3 wt% CQ and 0.8 wt% EDAB. A diamond saw was used to cut specimens from the cured materials with approximate dimensions of 20 mm X 3.5 mm X 2.4 mm. Each specimen was individually measured by micrometer and inspected for imperfections before use. Test specimens with obvious flaws were rejected. The test specimens were loaded to failure on an Instron 1125 with MTS controller upgrade variable-strain mechanical tester with a 5 kN load cell, 15 mm span and a crosshead speed of 1 mm/min. Twenty-four specimens of X-852-0000 were prepared for ultimate transverse flexural strength testing.

### Ultimate Transverse Strength Testing

	BisGMA / EBPADMA / TEGDMA n = 3		X-852-0000 n = 24	
	Mean	SD	Mean	SD
Width (mm)	2.61	0.06	3.23	0.34
Thickness (mm)	2.42	0.08	2.32	0.07
Ultimate Transverse Strength (MPa)	61.9	3.7	103.9	14.7
Elastic Modulus (GPa)	1.04	0.009	2.22	0.36
Energy to Break (J)	0.039	0.01	0.096	0.027

## DISCUSSION:

The homopolymer of X-852-0000 had an average degree of conversion of 85%. The average degree of conversion for a BisGMA/TEGDMA blend with identical photoinitiator package and curing protocols was 53%. In percent volumetric shrinkage analysis using the same resins, X-852-0000 averaged 1.68% while the BisGMA/TEGDMA averaged 5.32% volumetric shrinkage. The mechanical properties of X-852-0000 surpassed that of a BisGMA/EBPADMA/TEGDMA blend in the aspects of ultimate transverse strength, elastic modulus and energy to break. Ultimate flexural strength testing revealed the effects of using X-852-0000 as an additive in a BisGMA/TEGDMA blend. The elastic modulus of X-852-0000 as an additive in BisGMA/TEGDMA showed no significant change in comparison to the control BisGMA/TEGDMA blends. This suggests similar wear performance under stress. The maximum force to break the samples was significantly different for 25 wt% X-852-0000, suggesting that X-852-0000 can handle stress over time better than traditional materials.

## CONCLUSION:

- The degree of conversion of X-852-0000 was greater than the conversion of the BisGMA/TEGDMA control with identical photoinitiator packages and curing protocols.
- Along with a high degree of conversion, X-852-0000 exhibited less percentage volumetric shrinkage than the control.
- Best ultimate flexural strength results were achieved when X-852-0000 was formulated into the blend at 25% by weight. This formulation resulted in a 61% increase in the average energy to break, while retaining equivalent wear properties.
- In comparison to a BisGMA/EBPADMA/TEGDMA blend, X-852-0000 demonstrated higher strength, stiffness and toughness.
- The unique combination of increased conversion, low polymerization shrinkage and improved flexural strength makes X-852-0000 an ideal component for dental restorative materials.

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