Mechanical and optical properties of a novel bis-acryl ‘semi-permanent’ restorative material

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Fracture toughness

The fracture toughness of Filtek Supreme Ultra (FSU) (3M ESPE), LuxaCrown (DMG) and Vita Enamic were measured using the single edge V-notched beam (SEVNB) under 3-point bending test. The preparation and testing parameters followed ASTMD5045-14; ISO/NP 13586.

A custom mold was created (21.0 ± 0.1mm in length, 4.0 ± 0.1mm in depth and 3.0 ± 0.1mm in thickness) from polyvinylsiloxane impression material for sample preparation. Materials were carefully injected into the mold and covered by a transparent ethylene film and glass slide. Slight pressure (5-10N) was applied to the center of the glass slide to evenly distribute the material and extrude excess. FSU samples were photo-polymerized according to the manufacturer's recommended time of exposure using a visible light curing unit (Elipar DeepCure-S, 3M ESPE) with mean light irradiance of 1200 mW/cm^2. The irradiance of the light curing unit was tested every 24 hours using the MARC Light Collector (BlueLight Analytics) to ensure the consistency of curing conditions. LuxaCrown samples were allowed to self-polymerize according to the manufacturer's recommended time. Each sample was inspected for defects prior to polishing. If defects were significant, they were discarded. Remaining samples were polished under water using 600-grit silicon-carbide abrasive paper (CarbiMet, Buehler, Lake Bluff, Illinois) to remove excess material. A digital micrometer with an accuracy of 0.01mm (QuantuMike Micrometer, Mitutoyo Corporation, Japan) was used to monitor the dimensions during polishing. The final width (b) and thickness (w) of each sample was recorded before storing at 37°C in distilled water for 24 hours to allow for full maturation.

Vita Enamic CAD/CAM blocks were sectioned using an IsoMet-1000 sectioning saw. A 15 HC diamond coated blade (Buehler) was used under water at 150 rpm according to manufacturer's recommendations. The Enamic block was fixed to a flat vice and secured by melted wax during sectioning. The achieve the final sample dimensions, three cuts were made (21.0 ± 0.1mm in length,

4.0 ± 0.1mm in depth and 3.0 ± 0.1mm in thickness). Due to the accuracy of the sectioning machine, no further processing of Enamic was necessary before testing.

After a 24-hour maturation, each sample was remounted in the IsoMet 1000 to create a 0.50mm deep notch at the center using a 150 µm thick diamond coated blade. The notch was then coated with diamond polishing paste (3.5 um, Kent Supplies, USA) and a razor blade was used to form the notch into a V-shape with a final depth of 0.80mm-1.20mm. A consistent horizontal motion and force (5-10 N) with the razor blade ensured a uniform notch formation. Each side of the notch was measured using a light microscope with a > 50x magnification and averaged for a final notch depth.

A Universal Instron machine with an attached 3-point bending fixture was used to determine the fracture toughness of the samples. Samples were placed evenly on the fixture and loaded until failure with a crosshead speed of 0.5 mm/min. The peak fracture load was recorded to three significant figures and the fracture toughness [K_{IC} (MPa * $m^{1/2}$)] was determined according to the formula:

$$\mathbf{K_{IC} = P / bh^{1/2} * L / h * 3a^{1/2} / 2 (1-a)^{3/2} * Y}$$

Y=1.9887-(1.326*a)-((3.49-(0.68*a)+(1.35*(a^2)))*(a)*(1-a))/((1+a)^2)

α= average V-notch depth of group

P= fracture load

b= width of Sample

w= thickness of Sample

L = distance between support beams

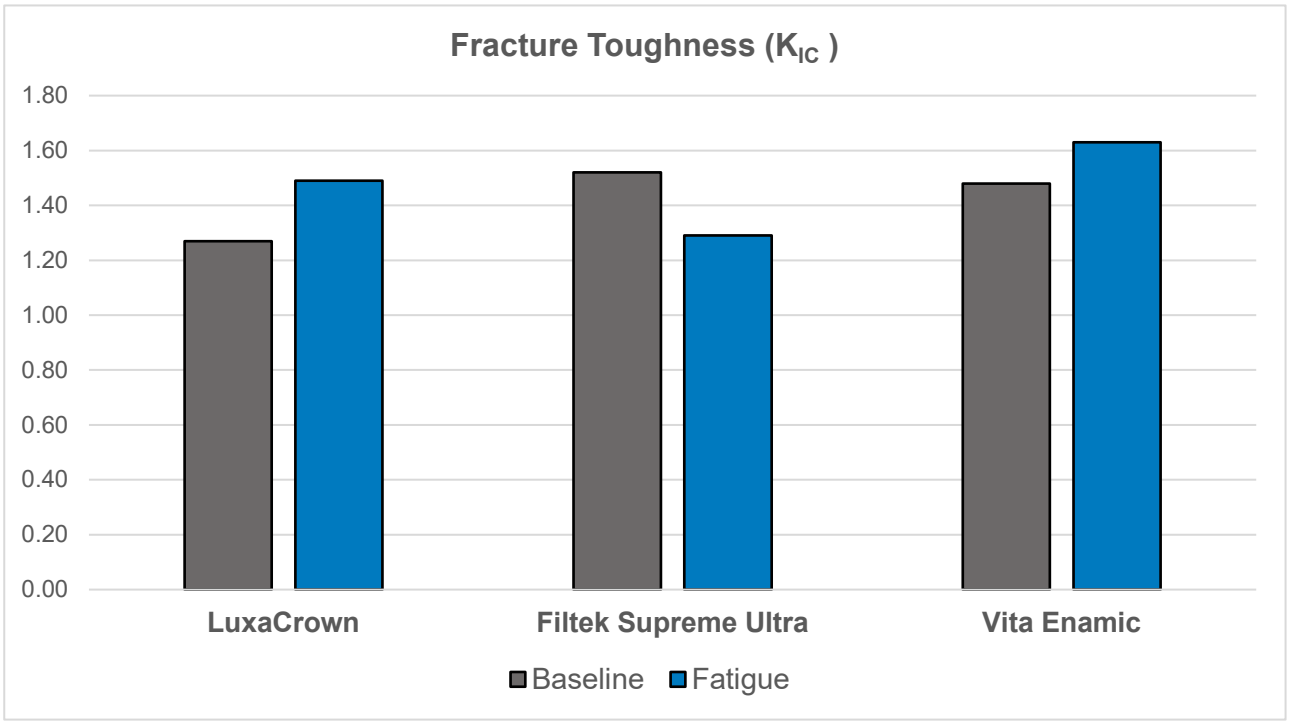
Fatiguing and artificial aging

Samples for fracture toughness were prepared for artificial aging. Each group was mounted in a custom fabricated stainless-steel holder with acrylic resin. Acrylic resin was then allowed to fully set for the manufacturers recommended time prior to loading. Stainless-steel holders were

then mounted in the chewing simulator (CS-4.8, SD Mechatronik). The force was calibrated using a force meter (KM-3, SD Mechatronik) with a weight of 4kg mounted to an antagonist bar (1kg). The machine was set to 100 cycles to obtain an average z-axis force. The testing parameters were set for 1,200,000 mechanical cycles (1.2Hz) at 50N with simultaneous thermocycling in deionized water (5 and 55°C) for a 30s dwell time. A break detection system (PM-3, SD Mechatronik) was installed in each chamber to monitor premature fracture. Surviving samples were tested for fracture toughness under 3-point bending with a Universal Instron machine.

Results:

	Filtek Supreme Ultra		**LuxaCrown**		**Enamic**	
	Mean	**SD**	**Mean**	**SD**	**Mean**	**SD**
Fracture Toughness	1.52	0.14	1.27	0.13	1.48	0.05
Fracture Toughness (Fatigue)	1.29	0.07	1.49	0.08	1.63	0.09



Flexural strength

The flexural strength of Filtek Supreme Ultra (FSU), LuxaCrown and Vita Enamic was done under a 3-point bending test. The testing parameters and preparation followed ISO standard 4049. A custom mold (2.0 ± 0.1mm in thickness, 2.0 ± 0.1mm in width and 21.0 ± 0.1mm in length) was fabricated from PVS for sample preparation. Materials were carefully injected into the mold and covered by a transparent ethylene film and glass slide. Slight pressure (5-10N) was applied to the center of the glass slide to evenly distribute the material and extrude excess. The polymerization of FSU composite resin and LuxaCrown was performed as previously mentioned according to manufacturer's instructions. Samples were carefully removed from the mold and inspected for defects. If any defects were present, the samples were discarded from testing.

Removal of excess material was done under running water using 600-grit silicon-carbide abrasive paper. A digital micrometer was used to monitor the dimensions during polishing. The final width (b) and thickness (w) of each sample was recorded before storing at 37°C in distilled water for 24 hours to allow for full maturation.

Vita Enamic CAD/CAM blocks were sectioned using an IsoMet-1000 sectioning saw. A 15 HC diamond coated blade (Buehler) was wafered under water at 150 rpm according to manufacturer's recommendations. Precise cuts were made (25.0 ± 0.1mm in length, 2.0 ± 0.1mm in depth and 2.0 ± 0.1mm in thickness).

A Universal Instron machine with an attached 3-point bending fixture was used to determine the fracture toughness of the samples. Samples were placed evenly on the fixture and loaded until failure with a crosshead speed of 0.5 mm/min. The peak fracture load was recorded to three significant figures and the flexural strength was determined according to the formula:

$\alpha = 3FL / 2wt$

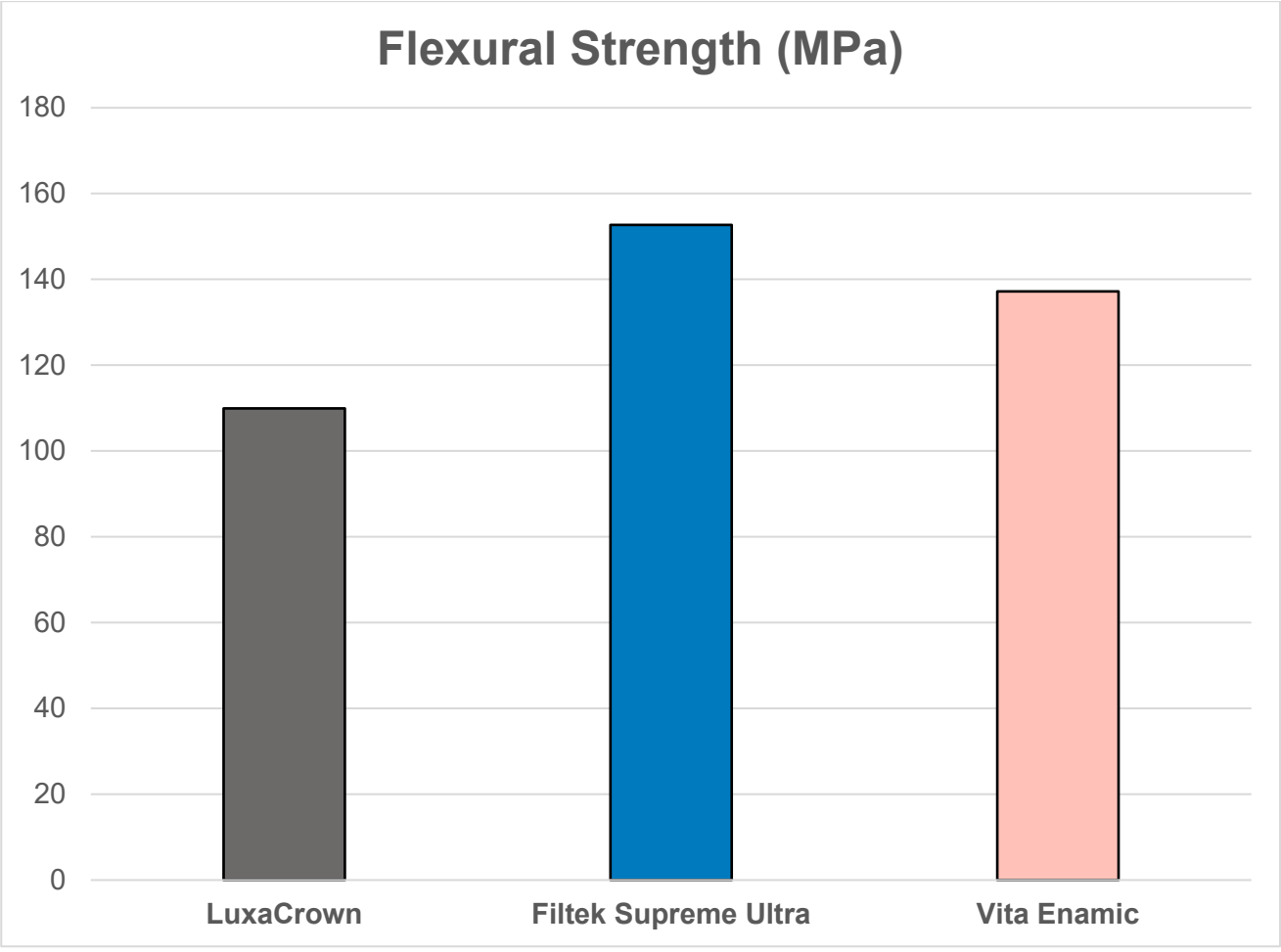
F= maximum force applied

L= distance between support beams

w= width of specimen

t= thickness of specimen

	Filtek Supreme Ultra		LuxaCrown		Enamic	
	Mean	**SD**	**Mean**	**SD**	**Mean**	**SD**
Flexural Strength (MPa)	152.65	9.87	109.93	4.87	137.16	6.41



Compression Strength

The compressive strength of Filtek Supreme Ultra (FSU) (3M ESPE), LuxaCrown (DMG) and Enamic (VITA) was tested according to ISO standard 9917.

A custom mold (6.0 ± 0.1 mm in diameter and 4.0 ± 0.1 mm in height) was fabricated from PVS for sample preparation. Materials were carefully injected into the mold and covered by a transparent ethylene film and glass slide. Slight pressure (5-10N) was applied to the center of the glass slide to evenly distribute the material and extrude excess. FSU composite resin and LuxaCrown were polymerized as previously mentioned. Samples were carefully removed from the mold and inspected for defects. If any defects were present, the samples were discarded from testing. Vita Enamic CAD/CAM blocks were used to mill cylindrical samples.

Polymerized samples (FSU and LuxaCrown) were carefully polished to the desired dimension using 600-grit silicon-carbide abrasive paper. A digital micrometer was used to confirm diameter and thickness of each sample. The diameter was measured twice, each at 90 degrees from the previous and averaged for a final diameter. Samples were stored in deionized water at 37°C for 24 hours prior to testing.

An Instron Universal machine was used to calculate the compression of each group. Calibration of the Instron was done prior to testing according to manufacturer's instructions. Cylindrical shaped samples were placed flat at the center of the compression plate and loaded until fracture with a crosshead speed of 0.5mm/min. The peak load was recorded and the DTS was determined according to the formula:

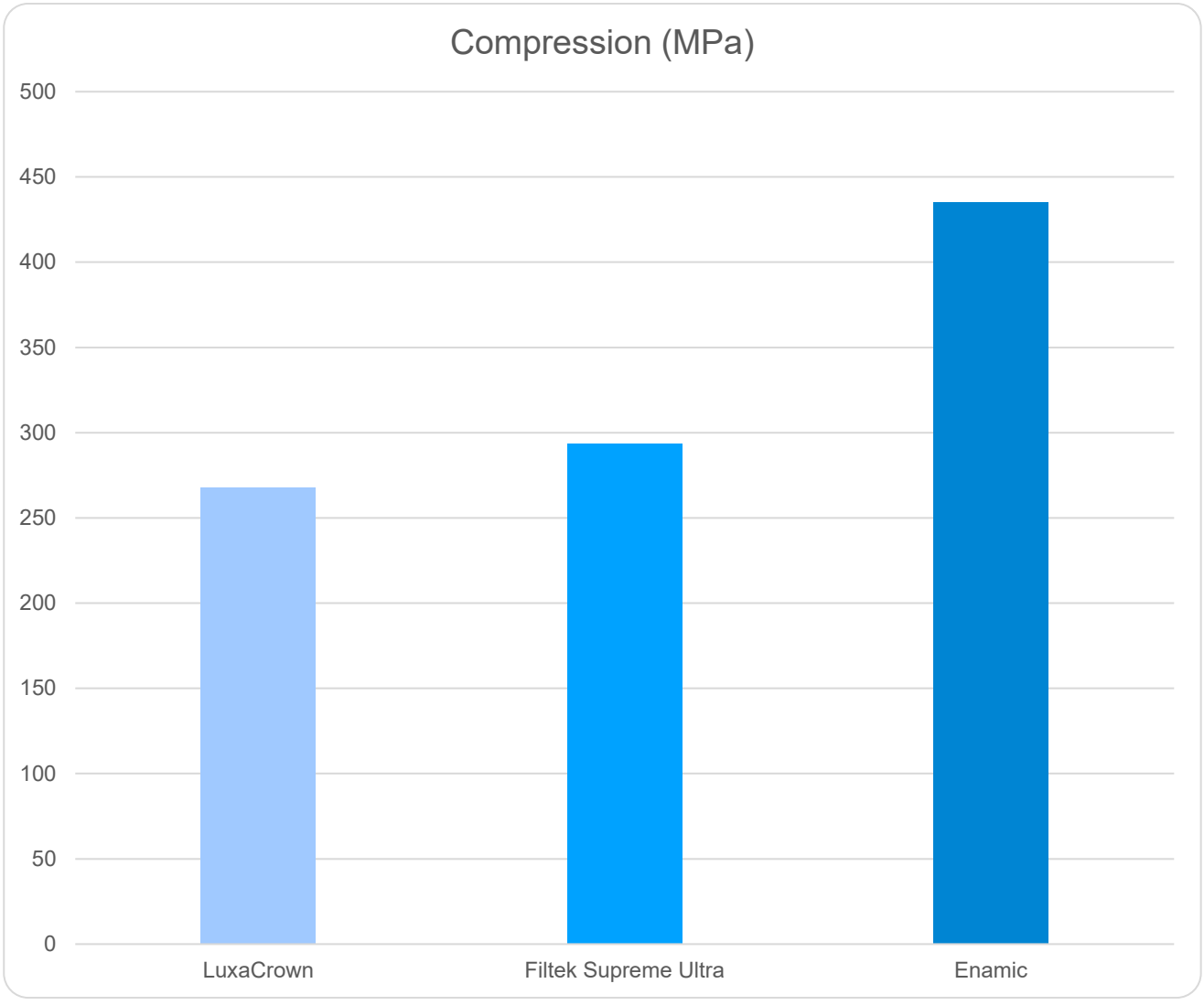
$DTS = 2F/\pi dt$

F= maximum force applied

d= diameter of Sample

t= thickness of Sample

	Filtek Supreme Ultra		LuxaCrown		Enamic	
	Mean	**SD**	**Mean**	**SD**	**Mean**	**SD**
Compression (MPa)	293.55	35.30	267.95	22.27	435.06	15.60



Volumetric Wear Loss

The volumetric wear loss of the materials was determined using a chewing simulator (CS 4.8, SD Mechatronik) and a wear measurement system (PM-3, SD Mechatronik). Eight samples were prepared for each group (N=24) using custom made (inner Ø 10 mm depth 3 mm) stainless steel holders. FSU and LuxaCrown were injected into the holders and each polymerized according to the manufacturer's instructions as previously described. Enamic samples (n=8) were prepared from CAD/CAM blocks using an IsoMet 1000 sectioning machine. Blocks were mounted to the vise arm and sectioned in 2mm discs with a diamond coated blade (Buehler). Enamic discs were then mounted in custom made (inner Ø 18 mm depth 3 mm) stainless steel holders using acrylic resin (VariDur 200, Buehler).

All samples were polished in a series to establish a flat finished surface. Excess material was removed using 600-grit silicon carbide abrasive and then finished with 1200-grit silicon carbide paper (MicroCut, Buehler, Lake Bluff, Illinois). Each group was mounted in the chewing simulator and the wear measurement system was calibrated to establish a zero-point for each chamber. Samples were submitted to a wear test, measuring the progression of wear after 5000, 10000, 20000, 40000, 60000, 80000, 100000, and 120000 cycles.

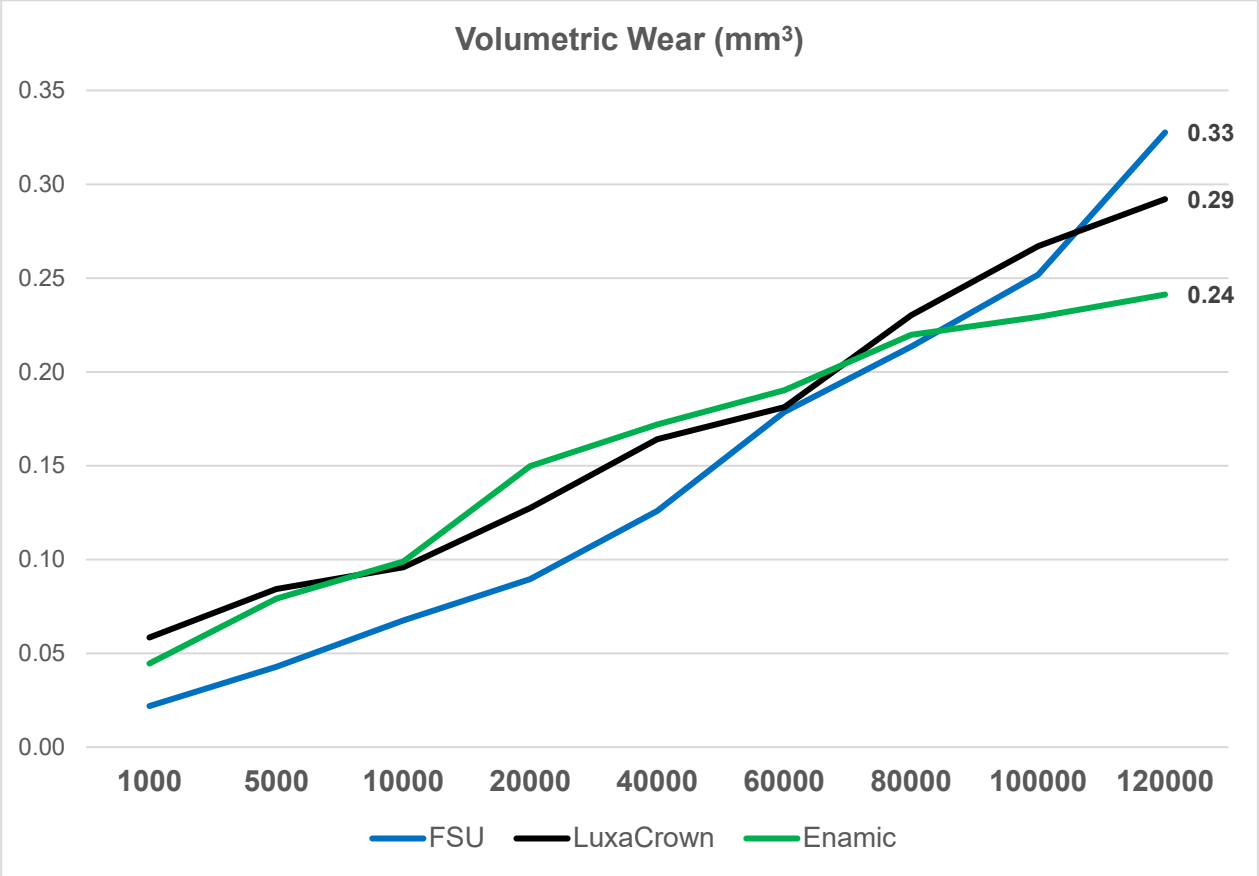
The following parameters were set in the chewing simulator for cyclic fatigue and thermocycling:

Load	5 Kg
Upstroke	2 mm
Downstroke	1 mm
Horizontal movement	0.7 mm
Upward speed	60 mm/s
Downward speed	60 mm/s
Horizontal speed	40 mm/s
Frequency	1HZ
Thermocycling	5°C-55°C 30s holding time, Transfer time 15s total cycle 90s
Direction	Back and Forth.
Antagonist	Steatite

After each cycle interval was complete (refer above), impressions of the wear were taken using a combination of light body impression material (Honigum Pro Light, DMG) and putty material (Virtual Putty Fast Set, Ivoclar Vivadent). Putty was hand mixed with a 1:1 ratio (base: catalyst) and

quickly placed inside of cylindrical tray before setting. Light body was then inserted into the wear mark and the tray was carefully placed over the sample. After the light body set, the tray was removed and the impression was inspected. If any defects were present, the impression was retaken. Impressions were scanned using a 3D laser scanner (LAS-20, SD Mechatronik) with a 0.2mm resolution. The digital scan was then uploaded to Geomagic 64 software (3D Systems) to calculate the volumetric wear loss. Using the digital points uploaded from the scan, a 3D model of the wear mark was created. The volume of the wear was calculated at each measurement interval to create a trend per material.

	Filtek Supreme Ultra		**LuxaCrown**		**Enamic**	
	Mean	**SD**	**Mean**	**SD**	**Mean**	**SD**
Wear (mm^3)	0.33	0.05	0.29	0.05	0.24	0.05



Optical Properties

The color stability, translucency and light blockage of three materials (Filtek Supreme Ultra (FSU), LuxaCrown (DMG), and VITA Enamic) were tested after 1 day, 7 days and 14 days. A custom mold (1.2mm depth by 10mm diameter) was used to create 20 samples for each material (N=60). The samples were divided into four groups (n=5); coffee (55°C), grape juice (5°C), distilled water (25°C) and thermocycling (5-55°C with a 30-second dwell time).

FSU and LuxaCrown were carefully injected into the mold to prevent trapping air. A clean transparent ethylene film and a glass slide was place over the mold to confine the material and slight pressure (5-10N) was applied to the glass slide to extrude excess material. Light-curable materials (FSU) were carefully photo-polymerized according to the manufacturer's instruction using a 1200 mW/cm^2 visible light curing unit (Elipar Deepcure-S, 3M ESPE). Self-curable (LuxaCrown) was allowed to set for manufacturer's recommended time. The specimens were carefully removed from the mold and inspected for defects. If no defects were present, samples were polished with 600-grit silicon-carbide abrasive paper to remove excess material. To finish the surface, 1200-grit silicon carbide paper was used for 60 seconds per side. The thickness of each specimen was confirmed using a digital caliper (World Precision Instruments, Sarasota, FL). Prior to baseline measurement, samples were stored in distilled water at 37°C for 24 hours to allow for full maturation.

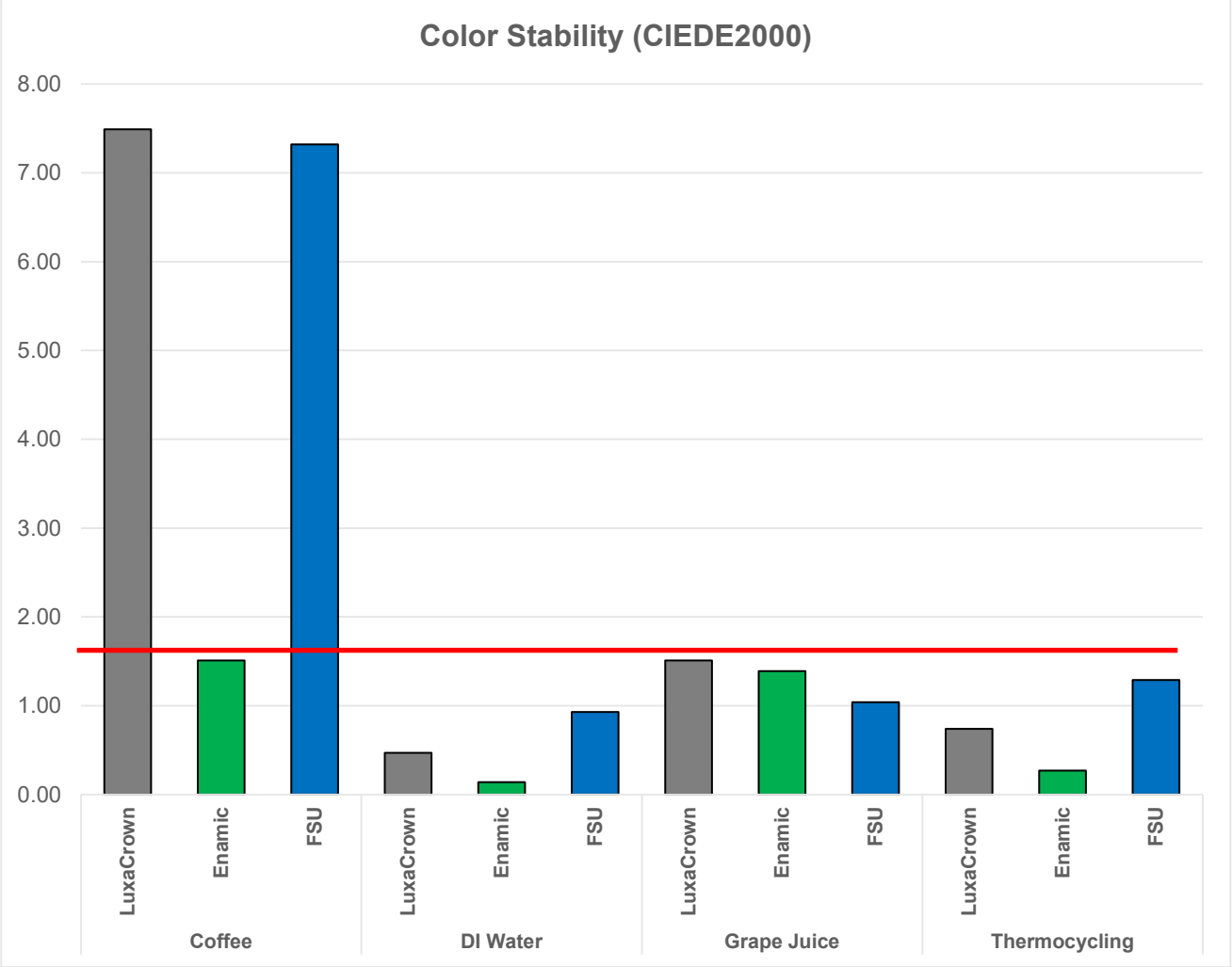
A spectrophotometer (CI7600, X-rite) was used to measure color coordinates (L*a*b*, C, H) values, Translucency Parameter, and % light blockage after 1 day, 7 days and 14 days. Each measurement was compared to the baseline measurements to calculate the change of each optical property.

Calibration of the spectrophotometer was performed prior to measurements. A spectral reflectance mode was used for measuring the values. A 6mm aperture plate was loaded to the reflectance chamber and calibration proceeded with a white tile and black trap under CIE D65 illuminant according to manufacturer's instructions. Each sample was removed from storage media

and ultrasonically cleaned for 5 minutes in distilled water. The L*a*b, C, H values were then measured for each sample under a white and black backing. A small amount of saturated sucrose solution between the sample and the backing assisted with holding the sample in place (via capillary action) and increasing optical continuity for a more accurate measurement. Measurements were performed twice and averaged. The color stability and translucency were calculated using the CIEDE2000 formula. Light blockage was calculated after 1 day, 7 days and 14 days following the reflectance measurements. The spectrophotometer was calibrated for total transmission with a 6mm aperture plate and sample holder according to manufacturer's instructions. Samples were ultrasonically cleaned for 5 minutes in distilled water to remove remaining sucrose. Measurements were performed twice within the transmission compartment and the total light blockage (TM203%) was recorded. Change in color values was compared to the acceptability threshold of 1.8 as reported by Paravina et al.

Color Stability (CIEDE2000): after 14 days

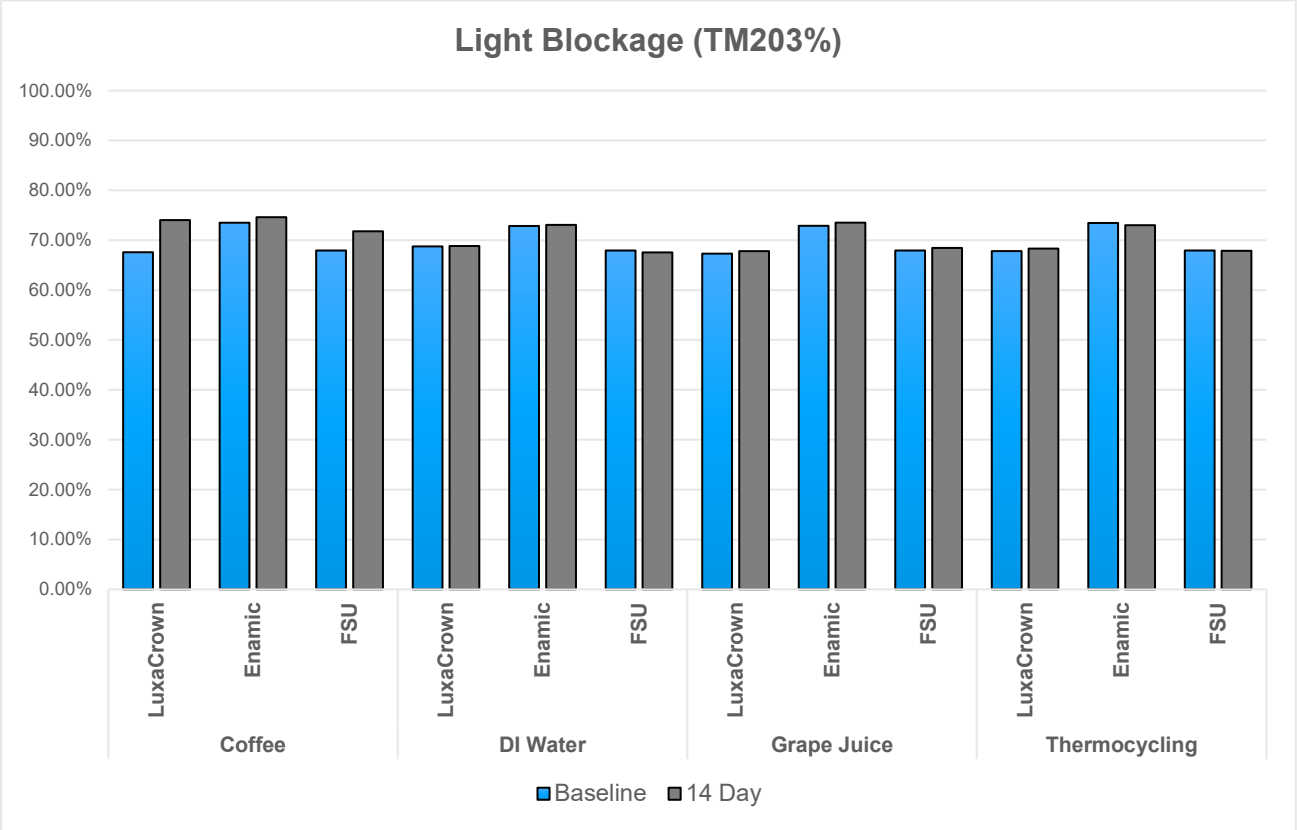
	Filtek Supreme Ultra		LuxaCrown		Enamic	
	Mean	SD	Mean	SD	Mean	SD
Coffee	7.32	0.38	7.49	0.32	1.51	0.21
Grape Juice	1.04	0.32	1.51	0.13	1.39	0.14
Distilled Water	0.93	0.10	0.47	0.13	0.14	0.05
Thermocycling	1.29	0.19	0.74	0.19	0.27	0.12



- Red line indicated the 1.8 acceptability threshold.

Light Blockage (TM203%): 14 days

	Filtek Supreme Ultra		LuxaCrown		Enamic	
	Mean	**SD**	**Mean**	**SD**	**Mean**	**SD**
Coffee	71.82	0.37	74.06	1.00	74.60	0.65
Grape Juice	68.43	1.50	67.84	0.58	73.50	0.40
Distilled Water	67.53	0.70	68.81	1.20	73.05	0.58
Thermocycling	67.86	0.93	68.33	0.90	73.02	0.21



Translucency Parameter (TP_{00}): after 14 days

	Filtek Supreme Ultra		LuxaCrown		Enamic	
	Mean	**SD**	**Mean**	**SD**	**Mean**	**SD**
Coffee	17.34	0.42	12.74	1.23	12.88	0.42
Grape Juice	20.68	1.07	18.52	0.46	12.87	0.62
Distilled Water	21.65	0.42	18.02	1.18	13.55	0.57
Thermocycling	21.44	0.81	18.24	0.97	13.73	0.14