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Biomaterials Research Report

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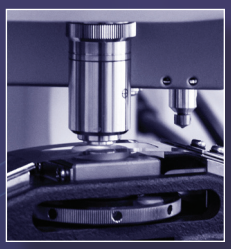

Number 129–November, 2019

Laboratory Evaluation of FIT SA

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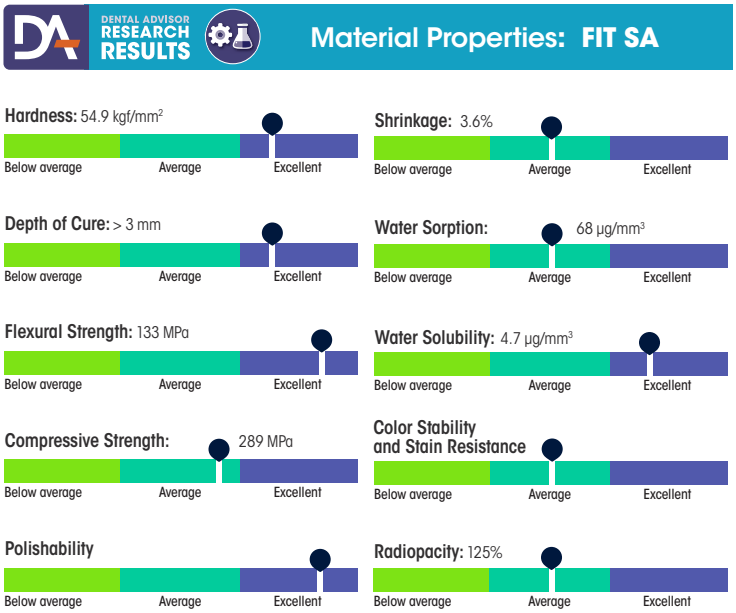
Introduction:

Shofu has developed a new self-adhesive flowable light cured composite including their patented S-PRG filler technology that releases six beneficial ions. We recently performed a battery of tests to see how it holds up compared to similar composites in its class to determine the important characteristics of strength, esthetics, stain resistance, shrinkage, sorption, and solubility. Overall, ***FIT SA*** performs above average to excellent in all categories and is indicated for Class I, III, V restorations, and as a base/liner.



Hardness:

Micro-Vickers Hardness, (n=5): 2 mm thick by 10 mm diameter discs were cured in a mold covered by a Mylar sheet before being stored for 24 h in 37°C water. They were tested using HMV-G21D Micro Hardness Tester (Shimadzu) using a 100 gf load and 10 second dwell time with 3 indentations per specimen. Indentation dimensions and hardness values were calculated utilizing the HMV Pattern Test software under 400X magnification.

RESULTS:

Hardness of dental composites is an important property that can correlate to compressive strength, wear resistance, and resistance to scratching. ***FIT SA*** has a hardness value that is above average for flowable composites which should provide additional resistance to compressive forces and scratch resistance.

HV/0.1 = 54.9 (1.0) kgf/mm²

Flowable composite range: [40-60]

Flexural Strength:

METHODS:

Flexural strength and modulus, (n=10): 2 mm x 2 mm x 25 mm bar specimens were tested after each set of specimens had been stored in distilled water for 24 hours at 37 ºC according to ISO 4049:2009. They were tested using an Instron 5866 universal test machine with a 1 mm/min crosshead speed.

RESULTS:

Flexural Strength: 133 (5) MPa

Flexural Modulus: 7.9 (0.2) GPa

The flexural strength is well above the ISO 4049 requirement of 80 MPa for a composite restorative indicated for occlusal surface restorations. ***FIT SA*** showed one of the highest flexural strength of flowable composites and a higher flexural strength than a number of universal packable composites.

Compressive Strength:

METHODS:

Compressive Strength, (n=10): 4 mm diameter x 8 mm cylinder specimens were made in a teflon split mold and stored in distilled water for 24 hours at 37 ºC. They were tested using an Instron 5866 universal test machine with a 1 mm/min crosshead speed.

RESULTS:

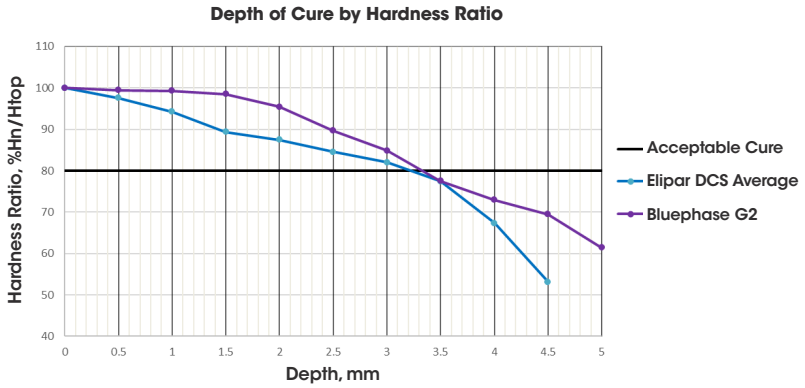
Compressive Strength: 289 (17) MPa

Flowable Composite Range: [240-340]

Compressive strength provides resistance to occlusal forces and this is especially important for replacing cusps and when opposing dentition is in direct contact. ***FIT SA*** showed an average compressive strength among flowable composites.

Depth of Cure:

Composite material was injected into a Teflon split mold, 5 mm in diameter, and 12 mm in height, and covered by Mylar. The curing lights were placed 2 mm from the top surface and concentric with the mold. The total height was measured and halved per the ISO 4049 method. One half of the split mold was removed and half of the restorative removed by grinding with 320 grit SiC paper and finished though 800 (P2400) grit paper and polished. The exposed portion of the material had the Vickers Microhardness measured 3 times for each vertical dimension, every 0.5 mm from the top surface (closest to light exposure). A graph depicting the change in hardness was made with the level marked at which the hardness ratio, (Hbot/Htop)*100 =80% is deemed a clinically acceptable level of cure, and used to give a depth of cure in mm. The calculated depth of cure per curing light is reported by the hardness method and ISO 4049 method for two curing lights, ***Bluephase G2*** (Ivoclar Vivadent), and ***Elipar DeepCure-S*** (3M).



Curing Light	BluePhase G2 in low power mode	Elipar DeepCure-S
Irradiance, mW/cm²	650	1,470
Curing Time	20 Seconds	10 Seconds
ISO 4049 DoC	3.7	3.6
Hardness DoC	3.4	3.2

RESULTS:

FIT SA consistently showed a greater than 3 mm depth of cure, which provides a large safety margin for use in small fillings in one layer.

Radiopacity:

Radiopacity According to ISO 4049, n=3: Composite specimens 1 mm thick and 10 mm diameter had digital x-rays taken alongside an aluminum step wedge and evaluated in an image analysis software using the histogram function to determine grey levels and measure the radiopacity in units of mm composite/mm aluminum. 1 mm of aluminum is roughly equivalent in radiopacity to sound dentin.

Radiopacity: 1.25 (0.07) mm composite/mm aluminum
This is greater than the ISO 4049 requirement of 1.0 mm aluminum equivalent and will show greater radiopacity than dentin.

Shear Bond Strength:

Shear Bond Strength, n=10: Human, adult, extracted third molars, sterilized in a 1% chloramine T solution, were embedded in acrylic resin discs and ground through 600-grit SiC paper to form bonding substrates of superficial dentin or enamel. Specimens were then ultrasonically cleaned in deionized water for 5 minutes.

Composite was placed on top of the substrate utilizing the Ultradent Shear Test mold and jig to produce a 2.38 mm diameter shear test cylinder according to ISO 29022:2013. The cylinder was light cured in accordance with the manufacturer's instructions while in the mold. The specimens were then transferred to a 37 °C deionized water bath until testing. Testing was performed using an Instron 5866 at a crosshead speed of 1 mm/min and shear bond strength results are given with means and standard deviations.

Shofu Fit SA Viscosity	Substrate	Shear Bond Strength, MPa
F02	Dentin	10.9 (1.6)
	Enamel	25.0 (3.7)
F10	Dentin	10.5 (2.7)

This excellent self-etched enamel bond strength is similar to that achieved by most self-etching universal adhesives, while the dentin bond strength is above average compared to self-etching self-adhesive cements. This should provide adequate initial adhesion for most indications, though bond durability should be tested with thermocycling in the future.

Water Sorption:

Water Sorption and Solubility According to ISO 4049:2009, n=5: Disc shaped specimens with 15 mm diameter and 1 mm thickness were prepared in a Teflon mold and light cured with overlapping exposures with an *Elipar Deep-Cure S* curing light for 10 seconds. Specimens were dried in a desiccator at 37ºC to a constant mass (m1) and stored vertically in deionized water for 7 days, excess water was air dried and weighed (m2). Specimens were then reconditioned in a desiccator at 37 ºC to a constant mass and weighed to give a final dry mass(m3).

Composite	Wsp, µg/mm³	Wso, µg/mm³
Shofu Fit SA	67.8 (2.9)	4.7 (1.5)

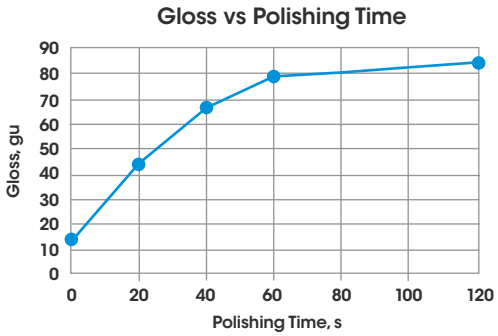
ISO 4049 gives maximum values of 40 µg/mm³ for water sorption and 7.5 ug/mm³ for water solubility. While ***FIT SA*** has a higher water sorption than this specification, this is likely necessary for the purpose of facilitating ion-exchange and is still lower than restorative materials such as glass ionomers or resin modified glass ionomers. All ***FIT SA*** specimens passed the water solubility test with only 4.7 µg/mm³ or 0.3 % of water solubility which indicates wash out shouldn't be a concern.

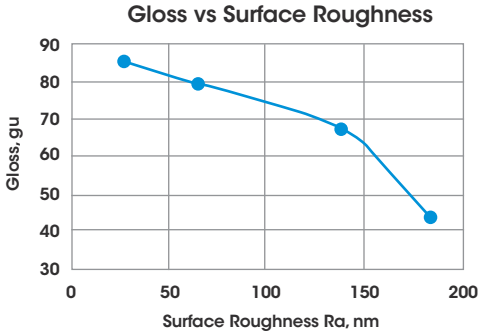
Polishability:

Polishing: Gloss and Surface Roughness, n=5: The composites (n=5 per polishing system) were cured in a mold (10 mm in diameter, 2-mm thick) with a Mylar strip and light cured for 20 seconds. The specimens were uniformly finished with 600 grit SiC paper and the gloss measured. The center 10 mm diameter portion was polished for 20, 40, 60 seconds with the Soflex Diamond Polishing System (3M) according to manufacturer's instructions and then the gloss measured over a 2 mm x 2 mm area using a small area glossmeter at 60° (Novo-Curve, Rhopoint Instruments), with 3 measurements taken every 120° of orientation per time point. Mean values and standard deviations of gloss was determined at each time point to generate a time dependent gloss curve. Surface roughness was measured using an atomic force microscope (Bruker Dimension Icon) over an 80 x 80 µm area. Average surface roughness (Ra) is calculated using Nanoscope Analysis software (Bruker).

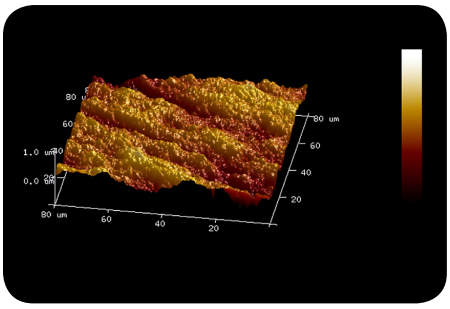
Condition	Gloss, gu	Ra, nm
Mylar	84.7 (1.0)	27.9 (4.0)
600-grit	12.8 (1.0)	NA
20-second polishing	43.7 (6.9)	185.0 (13.5)
40-second polishing	66.7 (5.6)	139.2 (36.2)
60-second polishing	78.7 (1.7)	66.9 (1.0)
120-second polishing	84.2 (0.7)	31.2 (3.7)

RESULTS: Overall, ***Fit SA*** shows excellent polishing characteristics rarely seen with flowable composites. Gloss and surface roughness similar to an ideal mylar surface was able to be achieved, and an average surface roughness below 0.2 µm needed to minimize bacterial adhesion was achieved in less than 20 seconds.

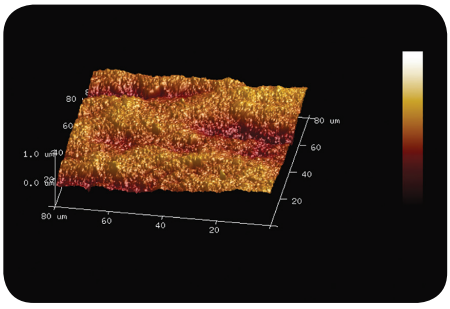




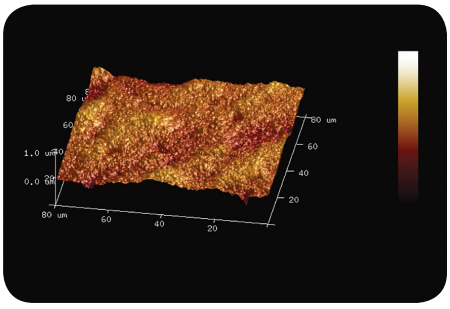
Representative 3D images of the surface roughness after each polishing time compared to mylar.



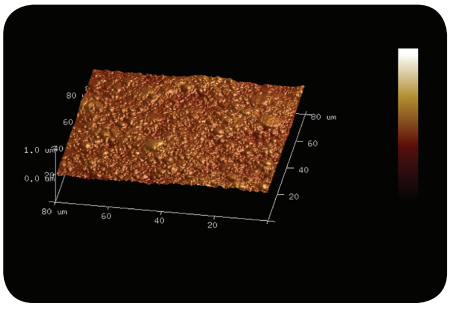
FIT SA 20s



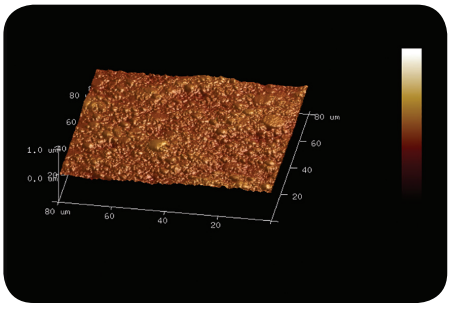
FIT SA 40s



FIT SA 60s



FIT SA 120s

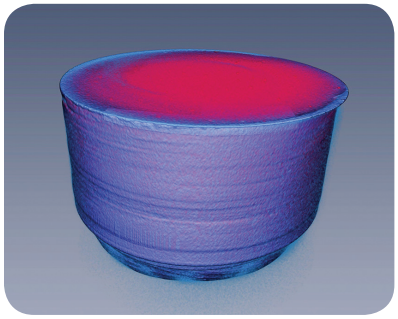


FIT SA Mylar

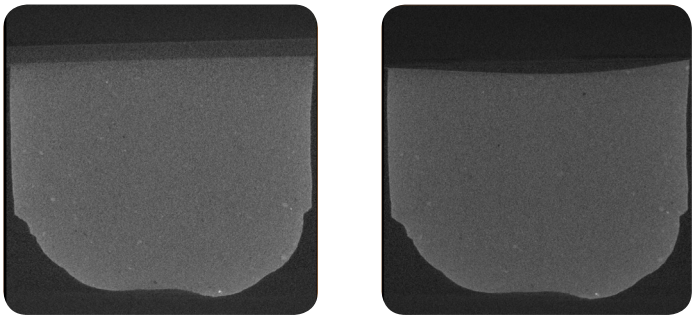
Volumetric polymerization shrinkage:

MicroCT Evaluation of Polymerization Shrinkage, n=3: Plastic molds with 4 mm diameter, 2.5 mm deep cavities were filled with composite and covered with a plastic microscope slide. They were scanned unpolymerized with a Zeiss Versa 520 3D X-Ray microscope with 800 projections, 120 kV 10W x-ray source and 4X objective giving a 4.3 µm voxel resolution and 42 minute average scan times. Composite was light cured for 20 seconds with an *Elipar Deep-Cure S* curing light and rescanned. Total volume of composite before and after polymerization was measured using Avizo software (Thermo Fisher Scientific). The volumes of one specimen before and after curing were overlaid to show the location of greatest shrinking below (shown in red).

RESULTS: Volumetric polymerization shrinkage percentage: 3.60 (0.10) %



3D rendering of composite before and after polymerization. Volumetric Shrinkage shown in red.



XZ ortho slices of composite before and after curing.

FIT SA had an average value for volumetric shrinkage for flowable composites which range from roughly 3-5% . The bulk of the shrinkage was from the top surface, with no apparent shrinkage occurring at the critical margins, or cavity bottom.

Color Stability and Staining:

Color Stability and Staining, n=5: 10 mm x 1 mm thick specimens were finished through 800 grit SiC paper and polished and submitted to The University of Texas Houston Center for Biomaterials and Biomimetics for testing. Specimens were subjected to accelerated aging with 450 kJ/m^2 in an Atlas weathering chamber and immersion in coffee for 3 days, with L*a*b* measurements made before and after aging or staining using an X-Rite Color-Eye 7600 spectrophotometer. ΔE values were calculated and means with standard deviations are reported below.

		L*	a*	b*	dL	da	db	dE	TP
Coffee Staining	Before	75.2 (0.7)	1.0 (0.2)	12.5 (0.1)	-2.3 (0.3)	0.6 (0.1)	3.2 (0.4)	4.0 (0.4)	17.5 (0.5)
	After	72.9 (0.8)	1.6 (0.2)	15.7 (0.5)					16.9 (0.9)
Accelerated Aging	Before	75.6 (0.6)	0.9 (0.2)	12.5 (0.3)	-0.8 (0.8)	-2.1 (0.5)	7.0 (1.3)	7.4 (1.4)	17.4 (0.6)
	After	74.9 (0.7)	-1.2 (0.6)	19.5 (1.6)					18.8 (0.7)

RESULTS: There was a larger increase in yellow color (positive change in b* parameter) in the accelerated aging test than coffee staining. There was a larger decrease in L* (darkening) with coffee staining. Coffee staining showed about a 2-shade difference on the Vita shade guide, while accelerated aging showed 3-4 shade changes. Some of this color change can be improved with additional polishing, especially with coffee staining. Overall, ***FIT SA*** showed comparable stain resistance to other composites on the market.