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MARGINAL ADAPTATION, COMPRESSIVE STRENGTH, WATER SORPTION, SOLUBILITY AND ION RELEASE OF A CLAIMED BIOACTIVE RESTORATIVE MATERIAL

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ABSTRACT

Objectives: This study was conducted to investigate marginal adaptation, compressive strength, water sorption, solubility, fluoride and calcium release of a claimed bioactive restorative material (ACTIVA BioACTIVE Restorative) compared to glass ionomer (Fuji IX) and resin composite (SphereTEC).

Materials and methods: ACTIVA was evaluated relative to Fuji IX and SphereTEC one. Marginal gap width was detected via scanning electron microscope before and after thermo-cycling. Compressive strength was tested using universal testing machine. For measuring water sorption and solubility, specimens were immersed in distilled water, subjected to drying cycles and weighed. Fluoride ion release was measured using ion-selective electrode while released calcium ions were detected using Atomic Absorption Spectrophotometer after 1, 14 and 28 days. Data analysis was performed using ANOVA, paired t-test and independent sample t-test.

Results: Marginal gap was reduced in both ACTIVA and Fuji IX after thermocycling while it increased in SphereTEC one. After thermo-cycling, the marginal gap was larger in dentin compared to enamel. SphereTEC one showed the highest compressive strength mean value. Fuji IX represented the highest water sorption values, followed by ACTIVA which also exhibited the highest solubility. Fuji IX showed higher fluoride release than ACTIVA whose calcium release did not reach 1ppm.

Conclusion: ACTIVA restoration can provide a potential marginal seal. Activa's compressive strength was limited compared to resin composite. ACTIVA's water sorption and solubility are within the acceptable range. However, its fluoride and calcium release was limited.

KEY WORDS: Bioactive restoration, Marginal adaptation, Compressive strength, Calcium release, Fluoride release.

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INTRODUCTION

Direct restorations are believed to be an elemental constituent of the field of restorative dentistry. One of the main determining aspects of the performance and sustainability of a dental restoration is an appropriate and a long-term sealing potential to tooth structure [1]. An area of clinical interest is the tooth- restoration interface, as inadequate sealing can result in marginal discoloration, secondary caries, and pulpitis. Accordingly, adequate sealing between the restoration and tooth structure is a necessity for ideal clinical performance [1].

Composite resin is one of the most frequently used restorative materials owing to their high esthetics, adequate mechanical properties, and command setting. In spite of the latest remarkable improvements in composite resins and adhesive systems technology, polymerization shrinkage which develops during composite curing remains a problem. Such shrinkage pulls the restorative material away from the cavity walls resulting in lack of marginal adaptation, microleakage and secondary caries development [2].

Glass ionomers, such as high viscosity glass ionomer cements (HVGICs), are known to chemically bond to the mineral content of teeth via ionic bonding to calcium ions and thus produce an adaptive seal [3]. As HVGICs release fluoride ions into the adjoining tooth structure, these materials are supposed to have the potential of slowing the development of carious lesions [3]. HVGICs are thus anticipated to be ideally suitable for the management of dental caries. Moreover, they may potentially simplify the tooth restorative strategy and allow the dentine-pulp complex to respond against the caries progression [4]. Despite the advantages of GIC, there are some drawbacks that compromise its use, such as low fracture strength, surface wear, and slow setting reaction that might delay or even jeopardize its final strength [5] .

Regardless the various advantages and latest modifications within restorative materials for dental applications, an urge for the generation of alternative, smart materials is perceived. A relatively recent development is ACTIVA BioACTIVE Restorative launched by Pulpdent Corporation, Watertown, MA in 2013 [6]. Such product was regarded as an equivalent to resin reinforced glass ionomer containing glass particles and polyacid constituents of glass ionomer, which undergoes acid-base setting reaction. They are also composed of a resin matrix, having both light and chemical polymerization ability. Such resin matrix is a patented bioactive shock-absorbing rubberized ionic-resin (Embrace resin) matrix that includes a small percentage of water with no Bisphenol A (BPA), Bisphenol A-glycidyl methacrylate (Bis-GMA) or BPA derivatives [6].

As declared by the manufacturer, ACTIVA triggers a natural reaction that stimulates apatite synthesis and remineralization process that binds the restoration and tooth simultaneously and seals margins as a protection from microleakage and secondary caries [7]. Regarding mechanical properties, compressive and diametral tensile strength of ACTIVA is claimed to be near to that of composites and significantly higher than glass ionomers.

According to PULPDENT, water absorption of ACTIVA is slightly more than resin composites and is considerably lower than glass ionomers. ACTIVA's solubility is supposed to be comparable with leading composites being much lower than glass ionomers. Its ionic resin matrix allows the diffusion of calcium, phosphate and fluoride ions. The patented resins and reactive glass fillers within ACTIVA restoration are formulated to provide bioactivity which requires water. It was also stated that ACTIVA releases fluoride similar or even more than glass ionomer restorations, delivering indelible benefits to improve oral health care [7].

Hence, the aim of this study was to investigate, in vitro, the marginal adaptation (seal), compressive strength, water sorption, solubility, fluoride and calcium release of the claimed bioactive restorative

material (ACTIVA BioACTIVE Restorative) in comparison to glass ionomer (GC Fuji IX GP FAST) and resin composite (Ceram.x SphereTEC™ one). The null hypothesis adopted was that there is no difference in marginal adaptation, compressive strength, water sorption, solubility, fluoride release and calcium release between ACTIVA, GC Fuji IX GP FAST and Ceram.x SphereTEC™ one.

MATERIALS AND METHODS

Materials

Materials used in this study, their descriptions, compositions, manufacturers and lot numbers are shown in table 1

Methods

Marginal gap width

TABLE (1) Materials used in this study, their descriptions,compositions, manufacturers and lot numbers

Product	Description	Composition	Manufacturer	Lot. number
ACTIVA BioACTIVE Restorative	Bioglass-reinforced resin modified glass ionomer	• Bioactive ionic resin matrix • Shock-absorbing rubberized resin component • Reactive ionomer glass fillers.	Pulpdent Corporation, Watertown, MA, USA.	181015
Ceram.x® SphereTEC™ one	Nano-hybrid-composite with pre-polymerized fillers.	**-Resin matrix**: is based on a modified version of the polysiloxane comprising matrix from the original Ceram•X® combined with poly-urethane-methacrylate as well as bis-EMA and TEGDMA. **-Fillers:** a blend of spherical, prepolymerized SphereTEC™ fillers, non-agglomerated barium glass and ytterbium fluoride. Depending on the shade, the filler load ranges from 77-79 weight-% total	Dentsply,Sirona, York, Pennsylvania, USA.	1809000824
GC Fuji IX GP FAST	-Posterior glass ionomer restorative cement in capsules	**Powder**: fluoroaluminosilicate glass, microencapsulated potassium persulfate, ascorbic acid and pigments Liquid: polyacrylic acid, distilled water, polybasic carboxylic acid	GC corporation, Tokyo,Japam	1810241
Adper Single Bond 2	Bonding agent	Ethanol; water; Bis-GMA; UDMA; silanized silica particles;HEMA; 1-glycerol; 3-dimethacrylate; copolymer of acrylic acid and itaconic acid.	3M ESPE; St Paul, MN, USA	NA22903
SwissTEC SL Etchant gel	Acid etch gel	35% phosphoric acid	Coltene Whaledent Private Limited, Navi Mumbai, Maharashtra, India	J20030

Sample size

Sample size for marginal gap width testing was calculated using G*Power version 3.1.9.2 for sample size analysis at α=0.05 and 80% power and effect size equal to 1.74 which yields a sample size of 3 samples per group. Six samples per group were performed to gain extra power.

Specimens' preparation

Eighteen bovine incisors were stored in 0.5% chloramine solution for not more than three months after extraction. Teeth were arbitrary allocated to one of three experimental groups according to the type of restoration used either ACTIVA or Fuji IX glass ionomer or Ceram.xSphereTEC resin composite.

Cylindrical diamond burs (ref#2094, KG Sorensen, Barueri, SP, Brazil) were used to prepare class V cavities (4 mm width×2mm length×2 mm depth) with enamel margins and axial walls in dentin [8]. The internal walls of each cavity were perpendicular to the top and bottom surfaces. Cavity dimensions were additionally checked using a periodontal probe.

For composite restorations, cavities were acid etched for 15 s in dentin and 30 s in enamel, washed and dried with a damp cotton pellet. The adhesive system Adper Single Bond 2 was applied as per manufacturer's instructions to all cavity walls to be restored with composite resin. The adhesive layer was thinned with a low-pressure air stream and light-cured for 10 s at 1200 mW/cm^2 using light emitting diode curing unit (Elipar S10 free light, 3M ESPE, USA). Sphere TEC one resin composite was placed in the cavity as one increment of 2 mm depth, covered by Mylar strip and light cured for 20 s according to manufacturer's instructions.

Concerning ACTIVA, cavities were previously etched for 10 s (enamel and dentine) as recommended by the manufacturer. ACTIVA restorative material was extruded in the cavity, covered by Mylar strip and light cured for 20 s according to manufacturer's instructions. As for Fuji IX, dentine was conditioned with 10% polyacrylic acid gel for 20 s, rinsed with water, then, washed off gently with distilled water and dried by air syringe. [9] Mixed glass ionomer capsule was removed from the amalgamator and loaded into the GC Capsule Applier, mixture was directly extruded in the prepared cavity, covered by Mylar strip and allowed to set.

After 24 h of storage in distilled water at 37°C in an incubator, each tooth was sectioned longitudinally through the center of the restoration using a precision diamond saw (IsoMet 4000; Buehler, USA) under water coolant, resulting into two halves with exposed adhesive interface.

Testing procedures

The tooth/restoration interface of one half of each tooth was examined directly with scanning electron microscope, while the other half was subjected to 10,000 thermal cycles between 5°C and 55°C with 15s dwell time and 10s transfer time (SD Mechatronik Gmbh Feldkirchen-Westerham, Germany) before examination.

An environmental scanning electron microscope (ESEM) (TESCAN VEGA3, Czech Republic) was used to detect marginal gaps at 350X, 500X and up to 2000X magnifications. Adhesive interfaces were divided into seven regions as shown in Figure 1. Marginal gaps at these regions were measured using ESEM image scale bar. Width of marginal gaps was recorded in micrometres.

Compressive strength

Sample size

Sample size for compressive strength testing was calculated using G*Power version 3.1.9.2 for sample size analysis at α=0.05 and 80% power and effect size equal to 1.871 which yields a sample size of 3 samples per group. Five samples per group were performed to gain extra power.

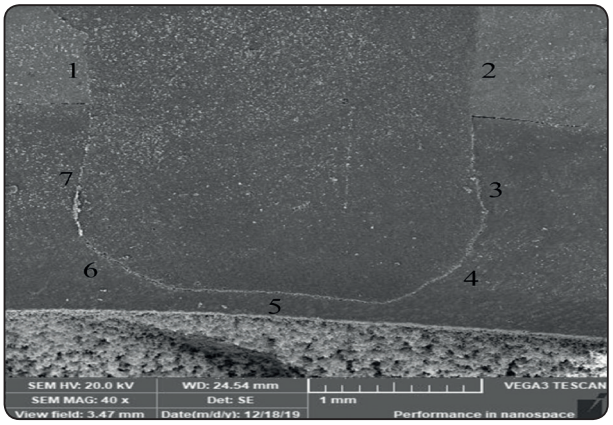

Fig. (1) Regions where marginal gap readings were recorded, two readings in enamel (1&2) and five readings in dentin (3, 4, 5, 6&7)

Specimens' preparation

Cylindrical specimens of SphereTEC resin composite, ACTIVA and Fuji IX were prepared in a teflon split mold with dimensions of 4 mm diameter and 6 mm length. Specimens were constructed by a single operator following manufacturer's instructions. ACTIVA and SphereTEC specimens were light-cured using light emitting diode curing unit (Elipar S10 free light, 3m Espe, USA) for 40 s on both top and bottom surfaces. The specimens were removed from the Teflon mold and further light-cured for 40s on each side of the cylindrical specimen [10]. As for Fuji IX glass ionomer, mixture within the capsule was extruded into the mould and packed in excess. The packed mold was placed in a screw clamp and stored for 1 hr in an incubator at 37°C; specimens were then removed from the mold and immersed in distilled water at 37°C for 24 h [11].

Testing procedure

Compressive strength test was performed using a universal testing machine (Instron 3365, High Wycombe,UK) with a cross head speed of 1mm/min. The compressive strength (MPa) was calculated using the following equation:

$\sigma = F/A$

Where, σ =compressive strength, F= maximum load and A= cross sectional area of the specimen

Water sorption and solubility

Sample size

Sample size for water sorption and solubility testing was calculated using G*Power version 3.1.9.2 for sample size analysis at α=0.05 and 80% power and effect size equal to 2.24 which yields a sample size of 3 samples per group. Five samples per group were performed to gain extra power.

Specimens preparation

Disc-shaped specimens (15mm diameter, 1mm thickness) were prepared at room temperature (23±1 ˚C) according to ISO 4049:2009 [12] and manufacturers' instructions. ACTIVA and SphereTEC specimens were light cured by light emitting diode curing unit (Elipar S10 free light, 3m Espe, USA) using a 10mm diameter curing tip with light intensity 1200 mW/cm^2. The light curing was performed initially on the middle of the specimen, and then continued in five overlapping sections from both sides for 20s each time. Regarding Fuji IX, specimens were allowed to set for 15min at 37±1° C then immersed in distilled water at 37°C for 24 h [13].

Testing procedure:

The specimens were carefully removed from the mold then transferred to a desiccator at 37° C ± 2°C for 22h. The specimens were then transferred into a second desiccator at room temperature and maintained for 2h. Specimens were weighed by a digital scale (Sartorius, Cubis®, Germany). This cycle was repeated every 24 h until a constant mass was reached; this was recorded as m1. An average value for both thickness and diameter was recorded to calculate the volume of each specimen (V)

Each specimen was immersed separately in 10 ml distilled water at 37°C and the readings were retaken after 7 days (m2). After this weighing,

the specimens were returned to the desiccator at 37°C and the drying cycle was repeated again. When a constant mass was reached, it was recorded as m3. Water sorption (Wsp) and solubility (Wsl) were calculated as follows:

Wsp= m2-m1/V

Wsl=m1-m3/V

Flouride and calcium ion release

Sample size

Fluoride and calcium release were measured for Fuji IX glass ionomer and ACTIVA only. Sample size was calculated using G*Power version 3.1.9.2 for sample size analysis at α=0.05 and 80% power and effect size equal to 1.1647 which yields a sample size of 4 samples per group. Five samples per group were performed to gain extra power.

Specimens' preparation

A split Teflon mold (6 mm diameter and 4 mm thickness) was used for the preparation of specimens. Each mold was placed on the top of a microscope glass slide and a Mylar strip. The mold was then packed with two increments of 2 mm depth using ACTIVA. Each increment of the inserted material was photo-polymerized for 20s according to manufacturer's recommendations. The top side of the mold was covered by a Mylar strip and another glass slide [14]. As for Fuji IX the material was bulk packed into the teflon mold in the same manner as ACTIVA. The Teflon mold with the glass slides were clamped with equal pressure on the specimen using a screw clamp. The whole assembly was transferred to the incubator at 37 ° C for 1h then conditioned in distilled water for 24 h [15].

Testing procedure

Each specimen was immersed in 5 ml deionized water in sealed 50 mL sterile CELLSTAR® polypropylene tubes *(Greiner Bio One International, GmbH, Germany)* and stored in an incubator at 37°C till the time of testing [15]. The amount of fluoride and calcium release for ACTIVA and Fuji IX was measured at different time intervals as follows: 1 day, 14 days and 28 days. At the time of testing, each plastic bottle was thoroughly shaken, then specimens were removed, washed with deionized water, dried and then stored again in 5 ml of fresh deionized water and incubated. Total leached fluoride was determined using a fluoride ion-selective electrode (ISE) [Orion EA 940, Thermo Electron Corporation, Houston, Texas, USA] [15]. On the other hand, released calcium ions were detected using SavantAA Atomic Absorption Spectrophotometer from GBC Scientific Equipment, Melbourne, Australia.

Statistical analysis

Statistical analysis was computed using SPSS (statistical package for social sciences, IBM SPSS Statistics for mac, version 24 software, Armonk, NY: IBM Corp,USA).

Data were presented as means and standard deviations. Data were checked for normality using Kolmogorov – Smirnov test and Shapiro- test and were found to be normally distributed. Statistical analysis was carried out using factorial analysis of variance (ANOVA) to explore the effect of different restorative materials and thermocycling on marginal gap width for whole tooth structure, enamel and dentin. Following significant interactions, One-way between-groups analysis of variance (ANOVA) was conducted to further explore the effect of different materials on marginal gap width. This was followed by Post-hoc comparisons using the Tukey's test. Paired t-test was conducted to explore the effect of thermocycling (before thermocycling, after thermocycling) and type of tooth structure (enamel, dentin) on marginal gap.

One-way between-groups analysis of variance (ANOVA) was conducted to explore the effect of different materials on compressive strength, water sorption and solubity.

Independent sample t-test was conducted to explore the effect of material on fluoride release

and calcium release. Repeated measure analysis of variance (ANOVA) was conducted to explore the effect of different time points on fluoride release and calcium release.

RESULTS

Marginal gap width

1 Marginal gap width along the entire tooth/restoration interface (both enamel and dentin) before and after thermocycling

Table 2 shows that whether before or after thermocycling, Fuji IX showed the significantly largest marginal gap width followed by Sphere TEC one. ACTIVA showed the significantly smallest marginal gap width. After thermocycling, the marginal gap decreased for Fuji IX and ACTIVA and increased regarding Sphere TEC one.

2. Marginal gap width at enamel/restoration interface versus dentin/restoration interface, before and after thermocycling

Results of marginal gap width at either enamel or dentine/restoration interface are represented in table 3 and figure 2, followed the same trend as that present along the entire tooth/restoration interface described above in table 2.

On comparing marginal gap width at enamel versus dentine/restoration interface, ACTIVA showed no significant difference in marginal gap width between enamel and dentin before thermo-cycling. After thermo-cycling, the marginal gap was larger in dentin compared to enamel. SphereTEC one and Fuji IX experienced significantly larger marginal gap width in dentin compared to enamel both before and after thermo-cycling

3. SEM of marginal gap width at enamel and dentin /restorations interfaces

Figures 3II &3III show the reduction in marginal gap width at ACTIVA /dentin interface after thermocycling compared to that before thermo-cycling (Fig.3 I). Figure 3V shows an increase of marginal gap width at Sphere TEC one /dentin interface after thermocycling compared to that before thermo-cycling (Fig.3 IV). Marginal gap width at Fuji IX/dentin interface experienced significant reduction after thermo-cycling as shown in figure 3 VII compared to figure 3 VI

Compressive strength

SphereTEC one showed the significantly highest compressive strength, followed by ACTIVA. Fuji IX showed the lowest compressive strength as shown in table 4.

TABLE (2) Mean values and standard deviations of marginal gaps' width (μm) of different restorative materials before and after thermocycling

	Thermocycling				P-value
	Before Thermocycling		After Thermocycling		
	Means	Standard Deviation	Mean	Standard Deviation	
Activa Bioactive Restorative	5.63^{cA}	0.09	2.70^{cB}	.24	0.0001
Ceram X SpherTEC one	18.67^{bB}	1.55	24.83^{bA}	2.07	0.0001
Fuji IX	42.87^{aA}	2.81	39.16^{aB}	2.10	0.018
P-value	0.001		0.0001		

Different small letters indicates significant difference within the same column. Different Capital letters indicates significant difference within the same row for every material type.

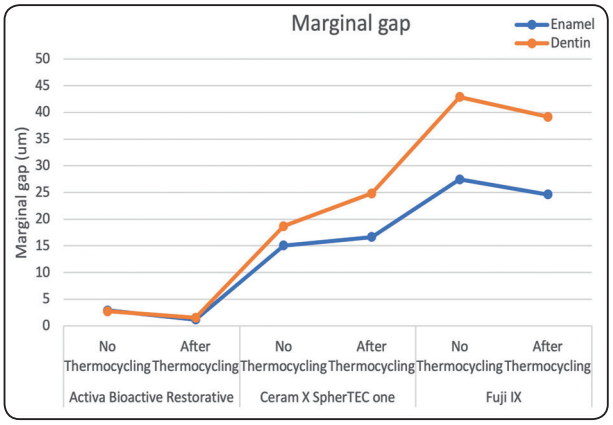

Fig. (2) Marginal gap width for different restorative materials used at enamel and dentin interface

Water Sorption and Solubility

Table 5 showed that Fuji IX represented the significantly highest water sorption mean values, followed by ACTIVA. SphereTEC One showed the least water sorption mean values. Regarding solubility, ACTIVA represented the significantly highest solubility mean values, followed by SphereTEC One. Fuji IX showed the lowest solubility mean values.

Fluoride release

Table 6 shows that Fuji XI exhibited significantly higher fluoride release compared to ACTIVA in all time periods (1 day, 14 days , 28 days). The highest fluoride release for ACTIVA was at day 1 and day 14 with no significant difference between them, which significantly decreased at day 28. Fuji IX showed the highest fluoride release at day 1, followed by day 14. The lowest fluoride release was recorded at day 28.

Calcium release

Table 7 represents mean values of calcium release of ACTIVA and Fuji IX. ACTIVA showed significantly higher calcium release compared to Fuji IX in all time intervals (1 day, 14 days, 28 days).The highest calcium release for ACTIVA was at day 1. The lowest calcium release was at day 14 and day 28 without significant difference.

Fuji IX showed the significantly lowest calcium release at day 1 and day 14 with no significant difference between them. The highest calcium release was recorded at day 28.

TABLE (3) Mean values and standard deviations of marginal gaps' width (μm) of different restorative materials, before and after thermo-cycling, at enamel versus dentine/restoration interface:

		Thermocycling				P-value
		Before Thermocycling		After Thermocycling		
		Mean	Standard Deviation	Mean	Standard Deviation	
Activa Bioactive Restorative	Enamel	2.90^{aA}	0.19	1.17^{bB}	0.11	0.0001
	Dentin	2.72^{aA}	0.10	1.53^{aB}	0.13	0.0001
	P-value	0.077		0.0001		
Ceram X SpherTEC one	Enamel	15.04^{bB}	0.98	16.66^{bA}	1.34	0.0001
	Dentin	18.67^{aB}	1.55	24.83^{aA}	2.07	0.0001
	P-value	0.0001		0.0001		
Fuji IX	Enamel	27.46^{bA}	1.50	24.64^{bA}	1.63	0.258
	Dentin	42.87^{aA}	2.81	39.16^{aB}	2.10	0.001
	P-value	0.0001		0.0001		

Different small letters indicates significant difference within the same column. Different capital letters indicates significant difference within the same row for every material type.

TABLE (4) Mean values and standard deviations of compressive strength (*MPa*) of Activa SphereTEC one and Fuji IX

Compressive strength					
Material	Mean	Std. Deviation	95% Confidence Interval for Mean		P-value
			Lower Bound	Upper Bound	
Activa Bioactive Restorative	196.005[b]	24.64281	178.3766	213.6334	0.0001
Ceram X SphereTEC one	226.718[a]	24.90994	208.8985	244.5375	
Fuji IX	117.264[c]	19.83322	103.0762	131.4518	

Different small letters indicates significant difference within the same column

TABLE (5) Mean values and standard deviations of water sorption and solubility ($\mu g/mm^3$) of Activa , SphereTEC one and Fuji IX

	Material	Mean	Std. Deviation	95% Confidence Interval for Mean		P-value
				Lower Bound	Upper Bound	
Water sorption	Activa Bioactive Restorative	34.4820[b]	1.50294	32.6159	36.3481	0.0001
	Ceram X SphereTEC one	10.9060[c]	.39979	10.4096	11.4024	
	Fuji IX	129.3440[a]	4.27785	124.032	134.655	
Solubility	Activa Bioactive Restorative	3.4060[a]	.29754	3.0366	3.7754	0.0001
	Ceram X SpherTEC one	2.7220[b]	.48023	2.1257	3.3183	
	Fuji IX	-16.9340[c]	.41119	-17.4446	-16.4234	

Different small letters indicates significant difference within the same column

TABLE (6) Mean values and standard deviations of fluoride release (ppm) of Activa Bioactive restorative and Fuji IX

Time	Material				P-value
	Activa Bioactive Restorative		Fuji IX		
	Mean	Std. Deviation	Mean	Std. Deviation	
1 day	3.3140[aB]	0.22930	8.0620[aA]	0.25519	0.0001
14 days	3.2200[aB]	0.28080	6.6340[bA]	0.17184	0.0001
28 days	2.5800[bB]	0.07141	4.2420[cA]	0.19162	0.0001
P-value	0.007		0.0001		

Different small letters indicates significant difference within the same column. Different capital letters indicates significant difference within the same row.

TABLE (7) Mean values and standard deviations of calcium release (ppm) of Activa Bioactive restorative and Fuji IX.

Time	Material				P-value
	Activa Bioactive Restorative		Fuji IX		
	Mean	Standard Deviation	Mean	Standard Deviation	
1 Day	0.96[aA]	0.12	0.06[bB]	0.02	0.0001
14 days	0.48[bA]	0.08	0.06[bB]	0.01	0.0001
28 days	0.36[bA]	0.06	0.12[aB]	0.02	0.001
P-value	0.001		0.005		

Different small letters indicates significant difference within the same column. Different capital letters indicates significant difference within the same row.

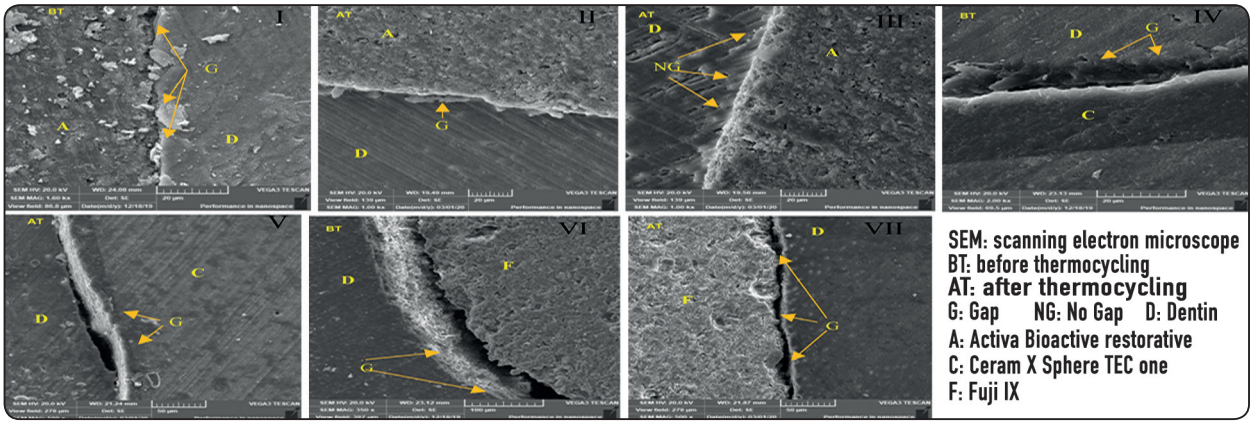

Fig. (3) SEM images of marginal gap width at dentin /restorations interfaces. Figure 3 I shows ACTIVA /dentin interface before thermocycling. Figures 3 II &3 III show ACTIVA /dentin interface after thermocycling. Figure 3 IV shows Sphere TEC one /dentin interface before thermocycling .Figure 3V shows Ceram X Sphere TEC one /dentin interface after thermocycling. Figure 3VI shows Fuji IX/dentin interface before thermocycling. Figure 3VII shows Fuji IX/dentin interface after thermocycling. Arrows denote dentin/restorations interfaces

DISCUSSION

Bioactive materials are continuously emerging in the market adding advantageous properties over that available in present restorative materials. This in-vitro study addressed properties related to the clinical use and performance of ACTIVA, a restorative material with declared bioactivity.

ACTIVA is considered a hydrophilic resin modified GIC supplemented with bioglass and strengthened with a patented rubberized polymer resin. [16] Accordingly, two restorative materials were selected for this study to be compared with ACTIVA, a nanohybrid resin based composite (SphereTEC) in addition to a high viscosity glass ionomer (Fuji IX). SphereTEC is known for its spherical fillers that are claimed to provide incomparable adaptation to the tooth cavity walls [17]. Fuji IX is a high strength glass ionomer that bonds chemically to enamel and dentine, releases fluoride and has a coefficient of thermal expansion close to the tooth structure [3].Hence, both materials tend to have properties comparable to those of ACTIVA as per manufacturer's claims.

ACTIVA was evaluated in a 1-year clinical follow-up of posterior restorations in adults. The main causes for failure mentioned in this randomized study were a high rate of post-operative sensitivity, secondary caries and loss of restorations [18]. It was hence essential to study this material's potential to achieve good cavity seal, which is of significance for the material's performance.

Class V preparations were selected to study the marginal adaptation. Class V restorations are convenient for in vitro/in vivo comparison due to the simple accessibility of the restoration margins for examination and evaluation. Furthermore, the placement of Class V restorations presents less variableness regarding the cavity size, application of the adhesive system, packing technique, curing method [19]. Class V cavities also represent a challenge for restorative strategies due to its high C-factor design and are used in studies assessing the potential of an adhesive restoration [20].

ACTIVA was placed in the cavity without bonding agent to test the self-adhesion capability stated by the manufacturer, who guarantees a strong resin-hydroxyapatite complex and a positive seal against microleakage [7] (PULPDENT™ Corporation). Furthermore, any adhesive applied on the cavity walls may be an impediment for the claimed bioactivity of the material [16].

Tooth sectioning was performed to examine the entire tooth/restoration interface in either enamel

or dentine. Thermo-cycling was used as a method of aging to mimic oral conditions due to the intake of food and fluids at different temperatures which may risk the long term performance of restorative materials. The number of cycles used in this study was 10,000 cycles, which is comparable to one-year service in previous studies [21].

It is noteworthy that teeth sectioning was performed before rather than after thermo-cycling, to examine one half directly and subject the other half of the same tooth to thermo-cycling. This exposed the dentin/restoration interface to aging conditions, which is not the case in clinical situations. However this was the only method available to accurately determine effects of thermo-cycling on marginal gaps, by using two similar halves of the same tooth hence eliminating manipulative discrepancies that might occur between one restored tooth and another.

This study describes a technique to quantify the marginal gap at tooth structure/restorations interface, where gap distance was assessed via corresponding width. On assessing the overall gap distance (in both enamel and dentine), whether before or after thermocycling, Fuji IX showed the largest marginal gap followed by Sphere TEC. ACTIVA showed the smallest marginal gap.

As for Activa, such small gap distance before thermo-cycling might be due to the high flow of such restoration when extruded in the tooth cavity .Such high flow of Activa was witnessed during manipulation and confirmed by its low filler loading (56 wt %) compared to Sphere TEC (77-79wt%)[17,22]. Their results seem to be in line with the nearly 99% intact margins described by Hughes et al.[23], after being estimated approximately 1 h after placement of ACTIVA. Another study also recorded a more encouraging picture for marginal adaptation of ACTIVA immediately after placement[16].

Although the high flow and bonding ability of Adper single bond 2 adhesive placed prior to Sphere TEC one might be a reason for good marginal adaptation, the polymerization shrinkage stresses of such composite might have attributed to a gap distance greater than that of ACTIVA. The magnitude of polymerization shrinkage stress is relatively dependent on the material's stiffness and its capability to flow. Higher filler loading of materials may result in a greater degree of stiffness, which in turn causes higher shrinkage stresses [24]. If stresses bypass the bond strength between the dental substrate and the restoration, a contraction gap develop, adversely affecting the restoration's longevity [25]. Higher stiffness of Sphere TEC one compared to ACTIVA due to higher filler loading might have lead to higher polymerization shrinkage stresses and larger marginal gaps.

Regarding Fuji XI glass ionomer, it showed the largest gap distance which seems contradicting with glass ionomers properties as they connect directly with the enamel and the dentin by achieving a chemical bond with hard tooth tissue. It may be due to its high viscosity thus could not adapt adequately to cavity wall microstructures. Microleakage of Fuji IX was evaluated in certain studies as a method of assessing marginal adaptation, it was found to exceed resin modified glass ionomer and composite[26].

Another reason for such larger gap in Fuji XI glass ionomer, is the dryness of specimens that was performed prior to SEM examination which might have lead to volumetric shrinkage of glass ionomer due to water content, such effect was not obvious in ACTIVA and Sphere TEC one due to their resinous nature and lower or no water content.

After thermo-cycling, the marginal gap decreased for Fuji IX (Fig 3VI, 3VII) and ACTIVA(Fig 3I,3II,3III) and increased for Sphere TEC one. Such reduced marginal gap after thermocycling in ACTIVA might be due to its higher water sorption (34.48 $\mu g/mm^3$) compared to solubility (3.4 $\mu g/mm^3$) which is nearly ten times higher , a volumetric expansion is expected and thus a reduced marginal gap. Bioactivity of ACTIVA in terms of ionic exchange between saliva and restorative materials

cannot be overlooked. It is claimed that recharge of ions such as calcium, phosphate, and fluoride take part in apatite synthesis at the restoration–tooth interface, which moreover binds the restoration to the tooth structure[16] .Calcium and fluoride release measured for ACTIVA in this study showed minimum values to participate by any means in reduction of marginal gap.

Fuji IX also showed a reduction in the marginal gap due to its high water sorption values (129.3 μg/mm^3) compared to negative values of solubility (-16.9 μg/mm^3). Such results indicate that these materials have undergone water sorption to an extent that could have masked the real solubility values [27].

Concerning Sphere TEC one resin composite, a significant increase in marginal gap occurred (Fig 3IV, 3V). The adhesive used in this study might be a reason for such increase. A previous study reported that ethanol/water-based adhesive systems (Adper Single Bond 2) showed higher water sorption and solubility values when compared to the water-based adhesives [28].Some studies indicated that the hydrophilic portion of most adhesive systems (*i.e*. 2-hydroxyethyl methacrylate, HEMA) could drastically decrease the evaporation of residual water[29]. Adper Single Bond 2 adhesive system used in the present study comprises a certain proportion of HEMA in their composition. Solvent retention within the adhesive may be a cause for adhesive degradation thus developing marginal gaps[28].

Concerning marginal gap width at enamel versus dentin/restoration interface, SphereTEC one and Fuji IX showed larger marginal gap width in dentin compared to enamel, before and after thermo-cycling. This might be due to the challenging conditions of bonding to dentin as its hydrophilic nature and presence of higher organic component compared to enamel. ACTIVA displayed the same results after thermo-cycling, however before thermocycling no difference was detected in marginal gap width between enamel and dentin. The high flow of ACTIVA might be the reason for such finding.

Regarding compressive strength, SphereTEC one resin composite resin showed the highest compressive strength. Mechanical properties of resin composites are influenced by monomer composition in addition to filler loading. BisEMA (ethoxylated version of bisphenol A diglycidyl methacrylate) is one of the monomer constituents of SphereTEC one resin matrix. BisEMA with its reduced viscosity and high molecular weight, besides poly-urethane-methacrylate enhanced the mechanical properties of such resin composite [30]. High filler loading of SphereTEC one (77-79 wt %) may have also participated in its strengthening.

Active Bioactive restorative showed higher compressive strength compared to Fuji IX glass ionomer. The shock-absorbing resin component of ACTIVA as mentioned by the manufacturer might be the reason for its higher compressive strength. This finding is in line with previous studies [6, 31] which found that mechanical properties of ACTIVA were significantly higher than certain types of resin modified glass ionomers.

Restorative materials are directly exposed to the oral environment; the current study thus assessed their water sorption and solubility. Water sorption is also necessary for bioactive materials, which demand water to allow ionic exchange. SphereTEC one showed the lowest water sorption in the current study which may be explained by the existence of stable polymeric structure in its matrix compared to ACTIVA and Fuji IX. Highest water sorption values were experienced in Fuji IX since it is a hydrophilic material and is sensitive to moisture. Water plays an essential role on the physical-mechanical properties of GICs as they set by an acid-base reaction [32].

Water sorption values of ACTIVA was higher than that of SphereTEC one, which might be due to the bioactive ionic resin matrix which shows a degree of hydrophilicity as claimed by the manufacturer. However, the water sorption values of ACTIVA

complies the ISO 4049:2009 requirements in which water sorption of resin-based materials should not exceed 40μg/mm^3 [12].

As for solubility results, SphereTEC one and ACTIVA showed solubility results related to that of water sorption, higher water sorption results of ACTIVA were accompanied with higher solubility values. ACTIVA solubility results were within the accepted range of ISO 4049:2009 requirements in which solubility of resin-based materials were not to exceed 7.5 μg/ mm^3 [12]. Accordingly, SphereTEC one showed lower solubility results compared to ACTIVA. Many researchers [33, 34] have ascertained that materials with low sorption exhibit low solubility.

However, Fuji IX represented negative solubility values despite its highest water sorption which suggests that these materials encountered water sorption to an extent that might have masked its actual solubility. It can be explained by the high hydrophilicity of GIC-based materials [27].

Sustained fluoride release prevents recurrent caries due to lower solubility and lower crystal energy of fluorapatite compared to hydroxyapatite [35]. It was thus recommended to assess the fluoride release of ACTIVA compared to glass ionomer. Fuji IX exhibited higher fluoride release compared to ACTIVA in all time periods (1 day, 14 days, 28 days). Such finding was in agreement with previous studies which reported lower fluoride releasing profile of ACTIVA compared to glass ionomer [36, 37]. Garoushi et al., reported a tendency for ACTIVA to release most fluoride ions in the first 24 h, followed by a decrease in fluoride release reaching a continuous plateau, which is in agreement with our results [37]. Fuji IX showed the same trend of fluoride release.

Calcium release via a restorative material is strongly recommended, as providing long-term remineralization and caries inhibition might aid in providing better marginal seal [38]. Calcium release was examined for ACTIVA as the manufacturer claims such property and for Fuji X due to the presence of calcium in its glass particles. Calcium ions measurement was performed via immersing specimens in distilled water rather than subjecting the restorations to low cariogenic pH challenge, as our study aimed at simulating a non-carious clinical condition to check the ability of the investigated restorative materials to provide adequate seal which might help in preventing recurrent caries and to also simulate the same testing conditions of marginal gap evaluation.

Although ACTIVA calcium release was significantly higher than that of Fuji IX, its maximum levels did not reach 1 ppm. A previous study tested the calcium release of ACTIVA base compared to other tricalcium silicate cements, researchers concluded that calcium ion release was totally absent in the resin-based glass ionomer cement (ACTIVA) [38].

Based on the aforementioned results concerning marginal gap width, compressive strength, water sorption and solubility, fluoride and calcium release, the null hypothesis was rejected.

Despite the importance of laboratory studies to answer some questions in the short term, the actual performance of restorations can only be evaluated via long-term clinical trials. However such laboratory studies allow comparisons between various materials under accurate conditions without the variabilities present in the oral environment. Another limitation of this study was teeth sectioning prior to thermo-cycling, thus subjecting the tooth/ restoration interface to extreme aging conditions, where all interfaces were subjected to moisture and thermal fluctuations unlike the clinical context. However such strategy was adopted to accurately assess the effect of aging on marginal adaptation, where specimens (whether aged or not) of the same restorative material were obtained from the same tooth.

Clinical trials are recommended to compare clinical performance of ACTIVA with the other restorative materials

CONCLUSIONS

Within the limitations of this study it could be concluded that, ACTIVA restoration can provide a potential marginal seal even when subjected to aging conditions. However, ACTIVA restoration failed to fulfill the manufacturer's claims regarding having a compressive strength similar to that of resin composites. ACTIVA was found to undergo water sorption and solubility within the acceptable range for resin-based restorations. ACTIVA's fluoride release was incomparably lower than that of conventional glass ionomer; its calcium ion release is also considered insufficient to induce a hermetically sealed tooth/restoration interface.

Compliance with ethical standards

Conflict of interest: The authors declare that they have no conflict of interest.

Ethical approval: This article does not contain any studies with human participants or animals performed by any of the authors.

Informed consent: For this type of study, formal consent is not required

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