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EFFECT OF DENTINE SURFACE TREATMENT ON BONDING OF BIOACTIVE RMGI-BASED RESTORATIVE MATERIAL TO DENTINE

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ABSTRACT

Objectives: To evaluate the effect of different dentine surface treatment methods on bonding of bioactive RMGI-based restorative material to dentine

Methods: A total number of 25 caries-free human permanent molars were used as the bonding substrate. The occlusal enamel was removed using a low-speed diamond saw (Isomet, Buehler, USA). The selected molars were divided into 5 groups (n=5) according to the dentine surface treatment method used; saline (control), 10% citric acid (10s),17% EDTA (10s), 37% Phosphoric acid (10s) or polyacrylic acid (10s). Then, restorative material was placed to cover the exposed dentine surface following the manufacturer instructions. The hybrid/hybrid-like layer was observed using environmental field emission scanning electron microscopy E-FESEM operated in secondary electron/ back -scattered detection modes. Furthermore, the tooth/restoration interface was subjected to an elemental analysis using Energy Dispersive X-ray (EDX) and EDX mapping.

Results: The micromorphological analysis of tooth/restoration interface showed that bioactive RMGI-based restorative material (Activa) exhibited good hybridization with dentine following surface treatment with 10% citric acid, as well as, saline. The outcome of elemental analysis showed that 10% citric acid did not adversely affect the calcium content of dentine in comparison with surface treatment using phosphoric acid.

Conclusions: Water plays a great role in bonding of bioactive restorative materials to dentine. These materials can directly bond to dentine after rinsing of the surface with saline. Although, bioactive RMGI restorative material manufacturer recommends conditioning the dentine with phosphoric acid prior to insertion of materials, it is highly advisable and more conservative to use 10% citric acid.

KEYWORDS: Bioactive restorations, RMGIC, EDX, Micromorphological Analysis Tooth/ restoration interface.

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INTRODUCTION

Biomineralization and preservation of natural tooth substrate become one of the essential goals for modern restorative dentistry. Thus, several bioactive restorative materials were introduced to the field of dentistry; mineral trioxide aggregate (MTA), Tri-calcium silicates, bioactive glass and new generations of glass ionomer cements (GIC). This category of materials can influence a response from living tissue or organisms such as inducing formation of hydroxyapatite.[1]

The bonding of bioactive materials to dentine facing many challenges including the presence of smear layers and excessive water content of dentine (particularly in caries-affected dentine areas).[2] Hence, the manufacturers of these recommend performing surface treatment prior to the application of the restorative materials using different conditioning agents (e.g. phosphoric or polyacrylic acids). Nevertheless, these dentine-conditioning methods might remove/modify the smear layer and enhance hybridization with dentine, this theoretical assumption is not widely acceptable among researchers and academician. Actually, most of these acids might disrupt the mineral content of dentine reducing binding sites and subsequently deteriorate bonding to dentine.[3 4]

Recently a new RMGI-restorative material (Activa, Pulpdent, USA) was introduced in the dental market. The manufacturer of this material claims that this restorative material shows bioactivity and excellent mechanical properties compared to silicate-based cements.[5] This material gains popularity in the United States after obtaining the FDA approval.[6] Initially the manufacturer recommends applying the material directly to dentine without any surface treatment, however, currently the manufacturing company instruct clinicians to use phosphoric acid for 10s prior to the application of restorative materials.[7]

These conflicting instructions encourages us to conduct the current study to evaluate the effect of dentine surface treatment on bonding of bioactive RMGI-based restorative material to dentine. This study was designed to test the null-hypothesis of that there is no significant difference in micromorphology and chemical structure of tooth/restoration interface following application of different dentine surface treatment methods.

MATERIALS AND METHODS

Commercially available calcium silicate and bioactive RMGI-based restorative materials were used in this study (Table 1).

Study design and specimen preparation

A total number of 50 caries-free human permanent molars were used as the bonding

TABLE (1) Materials used in the study

		Ingredients		Company
Dentine Conditioning Agent	**Citric Acid**	10% citric acid solution		Prepared in the lab
	Super Etch	37% Phosphoric acid gel		SDI, Australia
	Riva Conditioner	25-30 % Polyacrylic Acid		
	EDTA	17% Ethylene diamine tetra acetic acid solution		Prepared in the lab
Restorative Material	**ACTIVA**	Blend of diurethane and other methacrylates with modified polyacrylic acid	44.6%	Pulpdent , USA
		Silica, amorphous	6.7%	
		Sodium fluoride	0.75%	

substrate. The occlusal enamel was removed using a low-speed diamond saw (Isomet, Buehler, USA). The teeth were stored in 0.5% chloramine T solution at 4°C and used within six months following extraction. The selected molars were divided into 5 groups (n=5) according to the dentine surface treatment method used; saline (control), 10% citric acid (10s),17% EDTA (10s), 37% Phosphoric acid (10s) or polyacrylic acid (10s). Then, restorative materials was placed to cover the exposed dentine surface following the manufacturer instructions.

Elemental analysis of restoration/tooth interface using EDX mapping

The bonded specimens were vertically sectioned through the restoration center into two halves; one half was used for elemental analysis (EDX), while the remaining section was utilized in micromorphological evaluation of tooth/restoration interface. The cut surface was wet polished with (600-, 800-, 1200-, 2400- and 4000- grit) silicon carbide papers (Microcut™, Buehler,Lake Bluff, IL, USA) respectively, then followed by lapping with a polishing cloth using 6, 3, 1 μm diamond pastes (Diamat, Pace Technologies, Tuscon, AZ, USA). Specimens were ultrasonically cleaned in distilled water. Non-coated tooth sections were utilized in this test. The resin/dentine interface was subjected to EDX analysis with EDX software attached to a field emission scanning electron microscopy.

Micromorphological analysis of restoration/ tooth interface under SEM

The second restoration/tooth combination half was utilized in this test. Resin/dentine interface was subjected to an acid-base challenge using 10% orthophosphoric acid solution for 5s, followed by application of 5.5% sodium hypochlorite solution for 5 m. The observing surface was gold-sputter coated and observed under scanning electron microscopy operated in secondary electron/ back -scattered detection modes.

RESULTS

Elemental analysis of restoration/tooth interface

The micromorphological analysis of tooth/restoration interface showed that bioactive RMGI-based restorative material (Activa) exhibited good hybridization with dentine following surface treatment with 10% citric acid compared to saline group. The outcome of elemental analysis showed that 10% citric acid did not adversely affect the calcium content of dentine in comparison with surface treatment using phosphoric acid (Fig.1) (Tables 2 and 3). Moreover, EDX results revealed that application of phosphoric acid gel (etchant) showed a significant mineral loss in both superficial and sub-surface dentinal layers (Fig.2) (Tables 4 and 5)

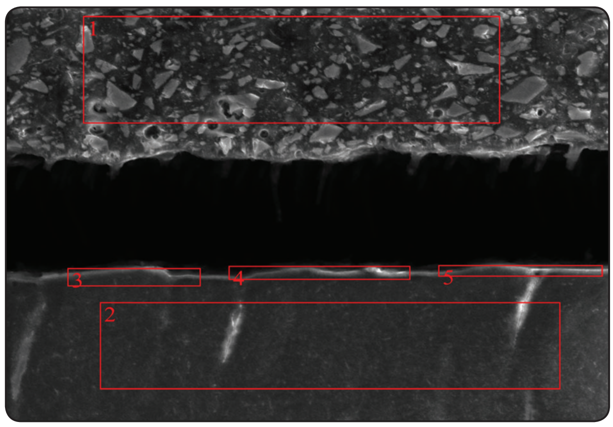


Fig. (1) EDX analysis of tooth/restoration interface of preconditioned surface with 10% citric acid.

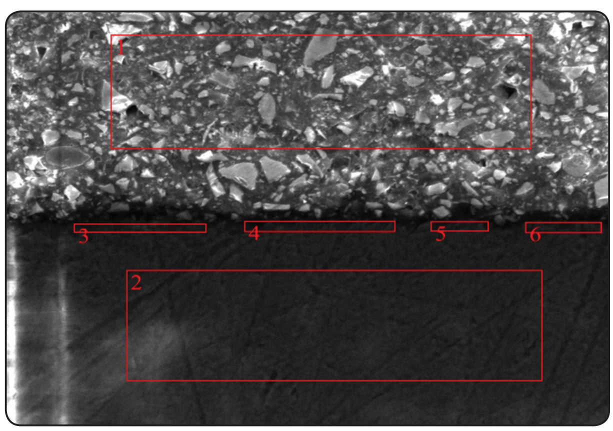


Fig. (2) EDX analysis of tooth/restoration interface of preconditioned surface with phosphoric acid gel.

TABLE (2) Shows mineral content of Box No.2 (deep dentine) in figure 1

Elt.	Line	Intensity (c/s)	Atomic %	Atomic Ratio	Conc.	Units	Error 2-sig	MDL 3-sig	
F	Ka	4.31	3.930	1.0000	2.250	wt.%	0.242	0.669	
Na	Ka	13.02	5.037	1.2815	3.489	wt.%	0.217	0.594	
Mg	Ka	21.21	6.798	1.7296	4.980	wt.%	0.243	0.594	
Al	Ka	13.86	3.974	1.0112	3.231	wt.%	0.194	0.472	
Si	Ka	12.85	3.421	0.8705	2.895	wt.%	0.182	0.419	
P	Ka	123.46	33.287	8.4694	31.067	wt.%	0.614	1.680	
S	Ka	5.99	1.926	0.4899	1.860	wt.%	0.171	0.475	
K	Ka	4.69	1.516	0.3858	1.787	wt.%	0.183	0.498	
Ca	Ka	109.82	40.110	10.2055	48.441	wt.%	0.967	2.826	
			100.000		100.000	Wt.%			Total

TABLE (3) Shows average mineral content of boxes No.3, 4 &5 (Superficial dentine) in figure 1. The outcome of EDX analysis revealed that application of 10% citric acid have a minimal effect on the mineral content in both superficial and sub-surface dentinal layers

Elt.	Line	Intensity (c/s)	Atomic %	Atomic Ratio	Conc.	Units	Error 2-sig	MDL 3-sig	
F	Ka	3.52	11.169	1.0000	6.624	wt.%	0.790	1.355	
Na	Ka	3.33	4.766	0.4267	3.420	wt.%	0.419	1.151	
Mg	Ka	4.23	4.983	0.4461	3.781	wt.%	0.412	1.012	
Al	Ka	3.74	3.878	0.3472	3.266	wt.%	0.377	0.962	
Si	Ka	8.22	7.938	0.7107	6.959	wt.%	0.540	1.448	
P	Ka	27.27	27.476	2.4600	26.567	wt.%	1.117	3.103	
S	Ka	1.25	1.451	0.1299	1.452	wt.%	0.283	0.969	
K	Ka	1.09	1.275	0.1141	1.556	wt.%	0.329	0.956	
Ca	Ka	28.11	37.065	3.3185	46.374	wt.%	1.836	5.334	
			100.000		100.000	Wt.%			Total

TABLE (4) Shows mineral content of Box No.2 (deep dentine) in figure 2

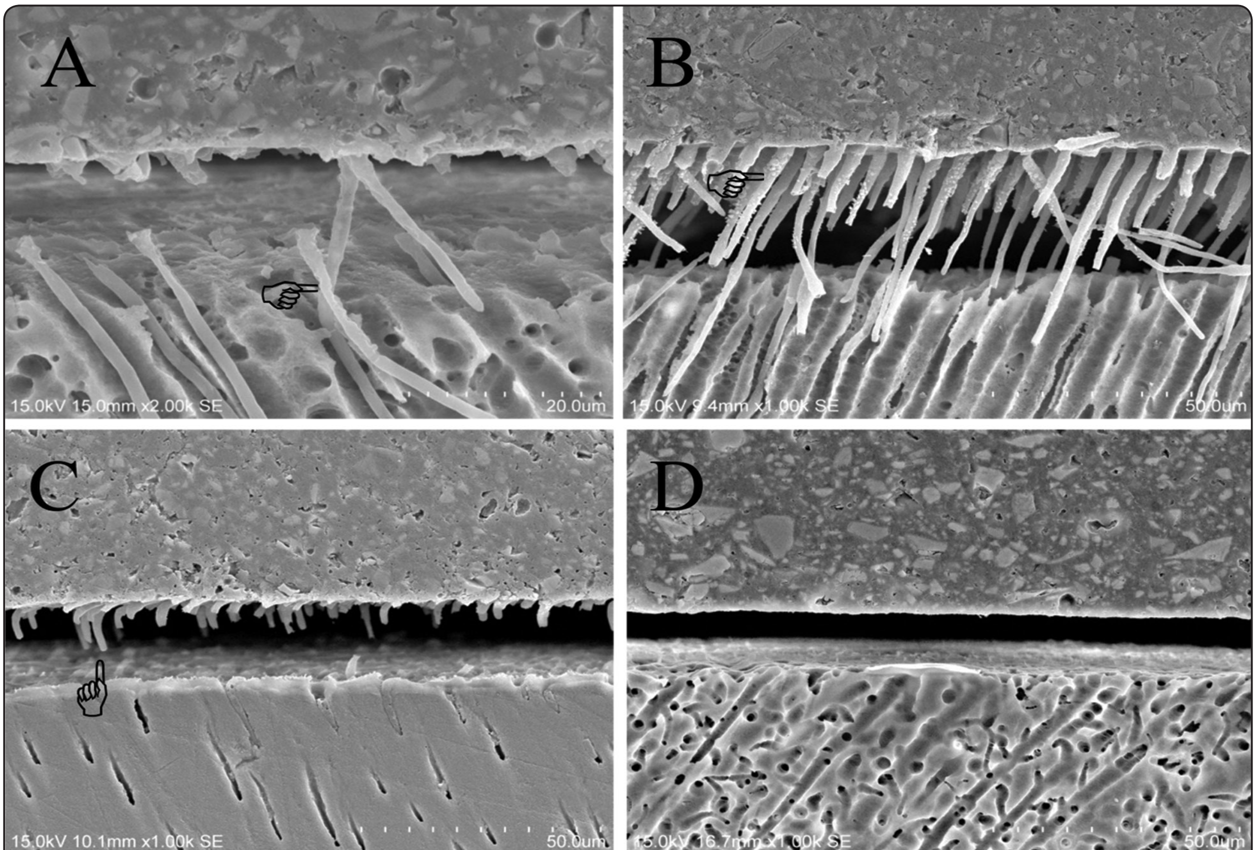
Elt.	Line	Intensity (c/s)	Atomic %	Atomic Ratio	Conc.	Units	Error 2-sig	MDL 3-sig	
F	Ka	7.41	5.660	1.0000	3.527	wt.%	0.290	0.748	
Na	Ka	9.84	3.453	0.6100	2.603	wt.%	0.186	0.461	
Mg	Ka	9.96	2.951	0.5214	2.353	wt.%	0.168	0.418	
Al	Ka	40.55	10.751	1.8994	9.513	wt.%	0.335	0.847	
Si	Ka	131.02	35.787	6.3225	32.963	wt.%	0.640	1.723	
P	Ka	44.87	15.467	2.7326	15.711	wt.%	0.516	1.390	
S	Ka	7.37	2.610	0.4611	2.745	wt.%	0.220	0.591	
K	Ka	6.03	2.128	0.3760	2.729	wt.%	0.246	0.587	
Ca	Ka	54.88	21.192	3.7440	27.855	wt.%	0.788	2.246	
			100.000		100.000	Wt.%			Total

TABLE (5) Shows average mineral content of boxes No.3, 4, 5 & 6 (Superficial dentine) in figure 2. The outcome of EDX analysis revealed that application of phosphoric acid gel (etchant) showed a significant mineral loss in both superficial and sub-surface dentinal layers

Elt.	Line	Intensity (c/s)	Atomic %	Atomic Ratio	Conc.	Units	Error 2-sig	MDL 3-sig	
F	Ka	11.66	7.529	1.0000	4.924	wt.%	0.323	0.642	
Na	Ka	14.35	4.549	0.6041	3.600	wt.%	0.213	0.529	
Mg	Ka	8.80	2.429	0.3226	2.033	wt.%	0.154	0.394	
Al	Ka	39.18	9.724	1.2914	9.032	wt.%	0.323	0.819	
Si	Ka	185.09	48.001	6.3752	46.411	wt.%	0.758	2.050	
P	Ka	29.47	10.667	1.4167	11.374	wt.%	0.461	1.237	
S	Ka	8.95	3.189	0.4235	3.520	wt.%	0.256	0.698	
K	Ka	7.52	2.657	0.3528	3.576	wt.%	0.291	0.705	
Ca	Ka	29.53	11.255	1.4948	15.530	wt.%	0.591	1.592	
			100.000		100.000	Wt.%			Total

Micromorphological analysis of restoration/ tooth interface under SEM

The micromorphological analysis of tooth/ restoration interface showed that bioactive RMGI (Activa) exhibited good hybridization with dentine following surface treatment with 10% citric acid as well as, saline(Fig.3-A and 3-C). Thick funnel-shape resin tags were observed in phosphoric acid group with noticeable widening of dentinal tubule diameter (Fig.3-B). Conversely, thin resin tags were distinct in 10% citric acid group with normal diameter of dentinal tubules (Fig.3-A). In saline group, several resin tags were observed and just confined within the smear layer zone (Fig.3-C). Typical RMGIC bud-shape acid-resistant hybrid like layer was observed in polyacrylic acid group (Fig.3-D). Few tiny resin tags were noticed in EDTA group (Fig.3-E).



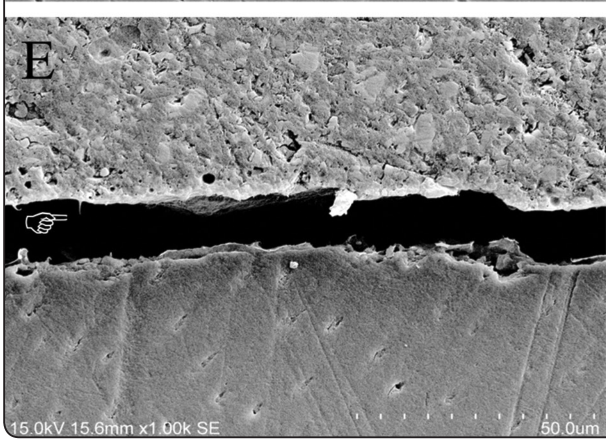


Fig. (3) SEM micrograph showing resin infiltration into dentine (Activa group) following dentine surface treatment with A:Citric acid, B:Phosphoric acid, C: Saline, D: Polyacrylic Acid , E: EDTA . It is obvious that this surface treatment method does not disturb the width of DT. Finger pointer show the resin tags

DISCUSSION

Nowadays, bioactive RMGI restorative martials are widely used in daily clinical work. This material gains popularity after obtaining the FDA approval in the United States. This study was conducted to solve the confliction about classification of this material and reduce the conflicting pre-application surface-treatment manufacturer instructions. Although, the manufacturer claims that Activa is a bioactive resin-composite restorative material, the official FDA documents reveal that this material is belong to RMGICs. The outcome of the micromorphological analysis of tooth/restoration interface supports the official classification of this material, particularly when dentine was pre-conditioned with polyacrylic acid for 10 s. The typical acid-base resistant hybrid-like layer with noticeable budding hybridization confirms the GIC nature of this restorative material. Furthermore, the formation of resin tags with 37% phosphoric acid and 10%citric acid is attributed to resin content of the material (Table 1).[4] Therefore, Activa can be classified as a RMGIC with slightly high resinous ingredients in comparison with standard RMGICs.

Energy dispersive X-ray elemental analysis was selected in this study due to the simplicity and accuracy of the testing method. [8] In a previous study by Hamama et al,[9] it was reported that this testing method shows 93% accuracy in flat polished dentine specimens. Also, it is a user-friendly testing method which can provide detailed information about the chemical structure of tested surfaces. Furthermore, the use EDX software 'area selection' tool is considered as an accurate method in detection of minor variations in mineral content among intimately close zones (e.g. superficial and sub- surface dentine layers). In order to obtain accurate results in EDX analysis was conducted on non-coated specimens.

In the current study different dentine conditioners were used to find an appropriate dentine surface treatment method suitable for this relatively newly introduced bioactive restorative material. The rationale of using 37% acid etching gel was based on the manufacturer recommendations of this material.[7] Polyacrylic acid was used in this study to evaluate the GIC nature of this restorative material.[10] The 10% citric acid was used due to its relatively higher pH (mild acidity) compared to phosphoric acid which consequently can preserve the mineral content of the tooth substrate.[11-13] Ethylene diamine tetra acetic acid solution (pH ~8) was used to evaluate the effect of application of alkaline solutions on bonding of Activa to dentine.[11]

It is well-known that the presence of water plays great role in the bioactivity of the material. The water helps in crystallization.[14] Also, water acts as hosting medium which allows exchange of ions between the restorative material and tooth substrate. Accordingly, this can explain the favorable interaction of Activa with underlying smear layer after rinsing the surface with saline. The EDX outcome revealed that dentine-surface treatment with 10% citric acid did not deteriorate the mineral content of tooth substrate, especially calcium and phosphorus ions. Also, it preserves the inter-tubular dentine (avoid over etching) which can help in achieving maximum conservation of tooth substrate. The non-favorable hybridization of bioactive RMGI with dentine following using EDTA is attributed to the interference of acid-base reaction of Activa GIC components. This also can verify the RMGIC nature of the material. It is feasible to state that each bioactive restorative material has its own unique surface treatment protocol , and there is no standard method can be used for all material. Sometime a great variation can be noticed between manufacturer's recommendations and the most appropriate surface treatment protocol for the material.

Finally, in light of the current study results, the null-hypothesis of that there is no significant difference in micromorphology and chemical structure of tooth/restoration interface following application of different dentine surface treatment methods was rejected.

CONCLUSIONS

Surface treatment with 10% citric acid prior to application of bioactive RMGI restorative material (Activa) has no adverse effect on the mineral content of dentine and does not result in unduly widening of dentinal tubule diameter. Water plays a great role in bonding of bioactive restorative materials to dentine. These materials can directly bond to dentine after rinsing of the surface with saline. Although, bioactive RMGI restorative material (Activa) manufacturer recommends conditioning the dentine with phosphoric acid prior to insertion of materials, it is highly advisable and more conservative to use 10% citric acid

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SCINTIFIC PRESENTAION

A part of this study was presented as a poster in the 97th General Session of IADR held at Vancouver, British Columbia, Canada (June 2019). https://iadr.abstractarchives.com/abstract/19iags-3180831/effect-of-dentine-surface-treatment-on-bonding-of-bioactive-restorations-to-dentine

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