



## Evaluation Shear Bond Strength of Three Self-Adhesive Resin Cements Bond to Different Substrates

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### **Abstract**

**Aims:** This study aims to evaluate the shear bond strength of three types of dual-cure self-adhesive resin cement (SARCs) bonded to three different substrates with detection types of failure, EDX analysis for substrates, control substrate, and after three resin cement applications.

**Materials and methods:** Thirty specimens from enamel and the same number from dentin and zirconia are prepared. Zirconia discs prepared by CAD-CAM system. Dental specimens prepared from intact surface 3<sup>rd</sup> molar for enamel and premolar for dentin. After teeth sectioning, all specimens are embedded in auto-cure acrylic resin in a plastic tube. All specimens were polished with sandpaper and zirconia sandblasting, rubber mold was fixed on the surface of the specimen. Three types of dual-cures (SARCs) were applied on the surface of the specimens and after curing the specimens were kept in distilled water at 37 °C inside the incubator for 24 hours. Then specimens subjected to shear bond strength on a universal test machine after the failure occurs specimens examined under a stereomicroscope to detect the mode of failure. Three control specimens enamel, dentin, and zirconia without any treatment, and nine were from failure specimens send to EDX analysis. **Results:** The results of this study analyzed with SPSS 25 edition, results showed no significant difference in shear bond strength (SBS) between the three (SARCs) bonds to enamel substrate, while in dentin and zirconia, there was a significant difference in SBS between self-adhesive resin cement. Failure mode detected under the stereomicroscope examination shows no significant difference between the three substrates in failure mode when the bond to BisCem<sup>®</sup> and TheraCem<sup>®</sup>Ca, but RelyX<sup>™</sup>U200 showed a significant difference. EDX analysis shows the presence of Calcium, Phosphorus ions on the zirconia surface when TheraCem<sup>®</sup>Ca was applied while not present when using the other two types of cement. The amount of calcium and phosphorus ions different from one specimen to another related to the type of substrate and the cement when compare with the control group. **Conclusion:** TheraCem<sup>®</sup>Ca showed improvement in SBS to zirconia due to its composition

such as acidic monomer MDP and calcium and phosphorus ions. BisCem and RelyXU200 show better performance with tooth structure and comparable results with zirconia.

#### الخلاصة

**الأهداف:** تهدف الدراسة الى تقييم قوة رابطة القص لثلاثة انواع من الملاط الراتنجي ذاتي اللصق مزدوج التصلب لثلاثة انواع من الأسطح المتفاعلة المختلفة مع تحديد نوع الفشل المرافق و اجراء فحص مطيافية تشتت الطاقة بالأشعة السينية للتعرف على تأثير الملاط على الاسطح المختلفة. **المواد وطرائق العمل:** يتم اعداد ثلاثين عينة لكل من المينا والعاج ومادة الزركون حيث يتم اعداد عينات الزركون باستخدام تقنية حاسوبية للقص والنحت اما عينات الاسنان فتعد من اسنان سليمة خالية من اي عيوب او معالجة سابقة ويستخدم الطاحن الثالث لا اعداد عينات المينا بينما تستخدم الضواحك لا اعداد عينات العاج وبعد قطع العينات يتم تثبيتها بواسطة راتنج ذاتي التصلب في انبوب بلاستيكي وبعد ان يتم اجراء عملية صقل للأسطح المتفاعلة من مينا وعاج ومعالجة الزركون باستخدام الرمال المقذوفة نقوم بتثبيت قالب من المطاط على سطح العينة ثم يتم اضافة الملاط الراتنجي على الاسطح المتعددة وبعد تصلبها ضوئيا يتم حفظها في درجة حرارة سبعة وثلاثين مئوية داخل حاضنة لمدة اربعة وعشرين ساعة بعدها يتم فحص قوة رابطة القص وبعد حصول الفشل يتم فحصها بواسطة المجهر ثم ارسال عينات من الاسطح المتفاعلة غير المعالجة للمقارنة مع العينات المعالجة بالملاط الراتنجي والنتائج كانت كما يأتي لا يوجد فارق معنوي لانواع الملاط الثلاثة من ناحية التصاقها بمينا السن بينما يوجد فارق بينها عند التصاقها بمادتي العاج والزركون نتائج فحص الفشل اثبتت انه لا فارق معنوي بين الاسطح المتفاعلة عند التصاقها بنوعين (TheraCem Ca, BisCem) من الملاط الراتنجي بينما اظهر النوع الثالث (ReLyu200) فارقا معنويا مع كل من العاج والزركون فيما اظهر فحص مطيافية تشتت الطاقة تغيرا في الوزن النسبي لعنصري الكالسيوم والفسفور بعد معالجتها بالملاط الراتنجي تتغير طبقا لنوع الملاط كما اظهر تواجد كلا العنصرين على سطح الزركون والتي تخلو مادة الزركون منهما تعود لتركيبية مادة الملاط (TheraCem Ca) **الاستنتاجات:** اثبتت الدراسة ان الملاط الراتنجي المعزز بالكالسيوم (TheraCem Ca) اظهر زيادة في قوة رابطة القص للزركون بالمقارنة مع النوعين الاخرين بينما اظهر النوعان الاخران زيادة نسبية في قوة رابطة القص مع مينا السن والعاج.

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## INTRODUCTION

Proper selection of cement materials considers the main factor for successful indirect restoration because it affects greatly the long-term success of these restorations. The primary function of dental cement is to fill the spaces or gaps between the tooth structure and the indirect restorative material, which may lead to enhancing the resistance to restoration dislodgment during function<sup>(1)</sup>.

Self-adhesive resin cement is a hybrid material that combines the composition of the resin, self-etch adhesive, and glass-ionomer<sup>(2,3)</sup>. It was developed to overcome some limitations of conventional resin cement because of its simplified cementation protocol. There is no need for pretreatment on the prepared tooth structure, decrease technical complication, reduce postoperative sensitivity, increase the retention of fiberglass posts, decrease the risk of contamination, satisfactory adhesion to dentin/restorative materials, releasing calcium, and fluoride by partially dissolving glass particles<sup>(2,3)</sup>.

The mechanism of adhesion in SARCs is of self-etching characteristic in the early stage of the chemical reaction acidic functional group of phosphoric and carboxylic acid simultaneously condition and infiltrate the dental tissue enamel and dentin binding to calcium ion from hydroxyapatite come from demineralization of dentin and the intaglio surface of the restoration create adhesion between them<sup>(4)</sup>.

Zirconia is the material of choice in contemporary restorative dentistry because of its biocompatibility, and its superior strength, toughness, fatigue resistance, excellent wear properties<sup>(5)</sup>. The longevity of zirconia as an indirect restoration depends on the integrity of the cement margin<sup>(6)</sup>, because zirconia is innate material, traditional cement cannot be effectively used<sup>(7)</sup>. Glass-ionomer cement, modified glass-ionomer and resin cement consider as the primary choice for the bonding of zirconia.

Nowadays, the incorporation of bioactive fillers in a resin matrix of composite restoration and cement gets higher attention from researchers. Many studied materials showed a sustained release of supersaturated ions of calcium and phosphate<sup>(8,9)</sup>, and showed the remineralizing capacity to enamel and dentin lesion in vitro<sup>(10)</sup>, also these bioactive fillers can bond with living tissue chemically by forming a calcium phosphate layer at the tooth material interface that improved durability of the restoration and prevent bacterial invasion<sup>(11)</sup>.

Energy dispersive x-ray spectroscopy(EDX) is probably the most frequently used technique for dental biomaterial chemical characterization, as it is normally installed on an scan electron microscope (SEM), EDX providing qualitative elemental analysis of a sample in few minutes and it can be used to analyze abroad range of material and sample size. EDX does not provide quantitative analysis

it be used as semi-quantitative method if known standard similar to the sample is available<sup>(12)</sup>.

This study aims to evaluate the shear bond strength of three types of dual-cure self-adhesive resin cement, one of them calcium ions releases the two other non-calcium release bonded to three different substrates (enamel, dentin, and zirconia) with detection of the types of failure, EDX analysis for control substrate and after three resin cement application.

## MATERIALS AND METHODS

Thirty specimens from each type of different substrates enamel, dentin, and zirconia were prepared as discs of  $7.4 \pm 0.1$  mm diameter \*  $2.8 \pm 0.06$  mm height. The zirconia was prepared from (VITAYZ, high translucent, Germany). Using computer aid design- computer aid manufacturing (CAD-CAM) (Vinyl Smart Optic, Germany), and sintered in high-temperature furnace (VITA ZYICOMAT 6000MS). after that specimens embed in auto-cure acrylic resin inside plastic tube of 12.7 mm diameter \* 20 mm height and kept in 100% humidity until cement application.

Forty five intact surface molar and premolar with no caries no cracks or previous restorative treatment of enamel and dentin specimens were sectioned, after cleaning teeth kept in 9% NaCl with 0.1 percentage thymol for 2 weeks then in distilled water alone until the time of use. For enamel, specimens 15 surgically

extracted 3<sup>rd</sup> molar sectioned in buccolingual direction by using D&Z diamond wheel disc (Germany) using slow speed headpiece and copious water coolant. Then specimen embed in auto-cure acrylic resin inside a plastic tube with enamel exposed up word and by diamond, wheel disk used to prepare a straight surface enough to cover by 3.8 mm diameter mold for cement application.

Dentin specimens were prepared from 30 intact premolar teeth extracted for an orthodontic reason. Teeth sectioned perpendicular to the long axis of the tooth below the dentin-enamel junction with copious water coolant. After embedding all specimens were kept in distilled water at 4 °C till cement application. Before cement application polishing enamel and dentin with (600) grit aluminum oxide sandpaper under running water for 30 seconds. For zirconia, after zirconia polishing with 400, 600, 800, and 1200 girt (Orientcraft, China) sandpaper. then exposed to sandblasting for 10 seconds, with 50µm aluminum oxide under 2.5 bar pressure 10 mm distance from the nozzle of the device<sup>(13)</sup>.

Three main groups of 30 specimens of each substrate, and each group subdivided into 10 specimens for each cements type, 90 types of cement cylinder created by rubber mold of 3.8 mm diameter \* 3.14 mm height after tight fixation to the specimens by using P.V.C. tape.

**Table (1):** Composition of the Resin Cement Used in the Study.

Material \trade name	Main composition
Dual-cured, self-adhesive resin cement\ BisCem <sup>®</sup>	Bis (Hydroxyethyl methacrylate) phosphate, Tetra-ethylene glycol di- methacrylate, glass particles, amorphous silica
Dual-cured, self-adhesive resin cement Rely X <sup>TM</sup> U200 Automixd	Base: methacrylate monomer containing phosphoric acid gp., initiator, methacrylate monomers, stabilizers, rheological additives. Catalyst: methacrylate monomers, alkaline fillers, silanated fillers, initiator compounds, stabilizers, pigments, rheological additives, zirconia, silica fillers
Dual-cured, self-adhesive resin cement \ TheraCem <sup>®</sup> Ca	Base: calcium base filler, glass fillers, dimethacrylate, initiator, amorphous silica. Catalyst: glass fillers, MDP methacryloyloxydecyl-dihydrogen-phosphate, rheologic modifier.

Three types of resin cement mix according to manufacturer instruction. Cement cured by (Super-LED light cure unit, China) with 1200 mW \ cm<sup>2</sup> light intensity at distance 1±.2mm for (20-30 second) for BisCem & TheraCem Ca and (20 seconds) for RelyXU200. The curing was done after compression the material with microscope glass slid. Careful removal of the rubber mold after 6 minutes to avoid premature failure. Then store in distilled water for 24 hours at 37°C inside the incubator. Each specimen was subjected to shear loading installed on the universal test machine (Gertner Total test solution, China), along with the bonding interface, at a crosshead speed of 1mm\1min until bond failure, force in newton converted to shear bond strength in MPa according to the equation  $SBS = Peak$

load in failure area (Newton)\ Bonding area (mm)<sup>2</sup>.

All the specimens examined under stereomicroscope attached to the digital camera (Optika microscopes, Italy) under magnification power 1.5 X, failure either cohesive in cement material more than 40% of the cement covered bonded area or adhesive at the interface less than 10% of the bonded area, or mix type less than 40% cover bonded area <sup>(14)</sup>.

Twelve EDX specimens were divided into (4) groups each group consists of (3) substrate, control group include enamel, dentin and zirconia without any treatment, BisCem<sup>®</sup>, TheraCem<sup>®</sup>Ca, and RelyX<sup>TM</sup>U200 resin cement cylinder bonded to enamel, dentin, and zirconia, after SBS failure, send to EDX analysis.

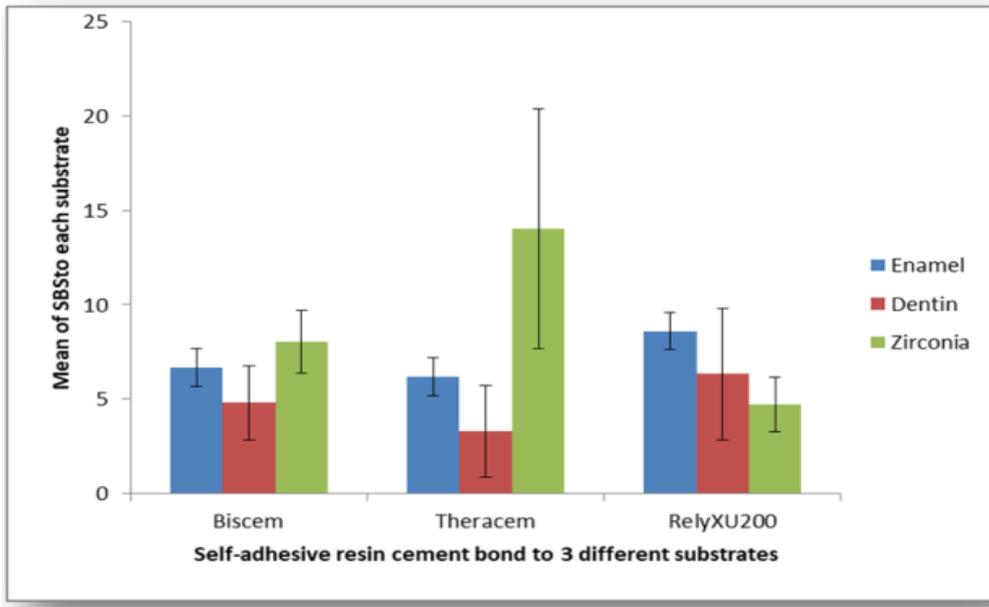
The statistical analysis of SBS, detection mode of failure, obtained in this study was done using the IBM SPSS statistics version 25 for the windows computer program, the data were analyzed using one way ANOVA at a level of confidence of 95% and (Post hoc) Duncans multiple comparison tests utilized to compare between cement groups. Nonparametric Kruskal Willis unrelated sample use for failure mode detection concerning each cements\ substrates.

## RESULTS

Results of shear bond strength of the three self-adhesive resin cement BisCem<sup>®</sup>, TheraCem<sup>®</sup> Ca, RelyX<sup>™</sup>U200 bonded to three substrate enamel, dentin, and zirconia include Mean and Stander deviation. The mean value of SBS of three SARCes showed comparable results with enamel substrate, while a decrease for all cement with dentin but with zirconia shows wide variation between TheraCem Ca and RelyXU200 Table (1), Fig.(1).

**Table (2):** Results of SBS of (3) SARCes Bond to Three Substrate Mean and Standard Deviation

Substrate	Type of resin cements	Mean in MPa	Std Deviation
Enamel	<u>BisCem</u>	6.6577	2.82786
	<u>TheraCem Ca</u>	6.1975	3.38634
	RelyXU200	8.6014	2.88546
Dentin	<u>BisCem</u>	4.7969	1.95837
	<u>TheraCem Ca</u>	3.2912	2.42001
	RelyXU200	6.3271	3.49022
Zirconia	<u>BisCem</u>	8.0453	1.65853
	<u>TheraCem Ca</u>	14.0435	6.34330
	RelyXU200	4.7194	1.46046



**Figure (1):** Diagram Showed Results analysis of SBS of Three SARC's Bonded to Three Substrates

One-Way ANOVA test for three substrates (enamel, dentin, and zirconia) showed that there was no significant

difference in SBS between three self-adhesive resin cement bonded to enamel ( $p=0.192$ ), Table(3).

**Table (3):** One –Way ANOVA Result of SBS of Three SARC's Bond to Enamel

	Sum of squares	DF*	Mean square	F	Sig
<b>Between groups</b>	32.561	2	16.281	1.758	0.192
<b>Within groups</b>	250.110	27	9.263		
<b>Total</b>	282.671	29			

In case of dentin One-Way ANOVA test showed there are significant differences

between the three resin cement in SBS ( $p\leq 0.05$ ), Table(4).

**Table(4);** One-Way ANOVA Results of SBS of Three SARC's Bond to Dentin

	Sum of squares	DF	Mean square	F	Sig
<b>Between groups</b>	46.083	2	23.042	3.160	0.05
<b>Within groups</b>	196.865	27	7.291		
<b>Total</b>	242.948	29			

Duncan's comparison test showed no significant difference between BisCem was compared to TheraCem Ca, also between BisCem was compared to RelyXU200

cement but there was a significant difference between TheraCem Ca with RelyXU200. RelyXU200 showed better performance with dentin, Table(5).

**Table (5):** Duncan's comparison test of Three SARC's Cement Bond to Dentin

Duncan	Cement groups	Subset alpha=0.05	
		1	2
	<u>TheraCem<sup>®</sup>Ca</u>	3.2912	
	BisCem <sup>®</sup>	4.7975	4.7975
	Rely X <sup>TM</sup> U200		6.3271
	Sig.	.223	.216

In the case of zirconia, the One Way ANOVA test showed significant

differences between three resin cement ( $p \leq 0.05$ ), Table(6).

**Table(6):** One-Way ANOVA result of SBS of Three SARC's Bond to Zirconia

	Sum of squares	DF	Mean square	F	Sig
<b>Between groups</b>	446.600	2	223.300	14.847	0.000
<b>Within groups</b>	406.090	27	15.040		
<b>Total</b>	852.690	29			

Duncan's comparison test showed a difference in the case of TheraCem Ca when compared with BisCem and RelyXU200. TheraCem Ca showed higher

bond strength to zirconia followed by BisCem and the lowest is RelyXU200, Table(7)

**Table (7):** Duncan's comparison test of Three SARC's Bond to Zirconia

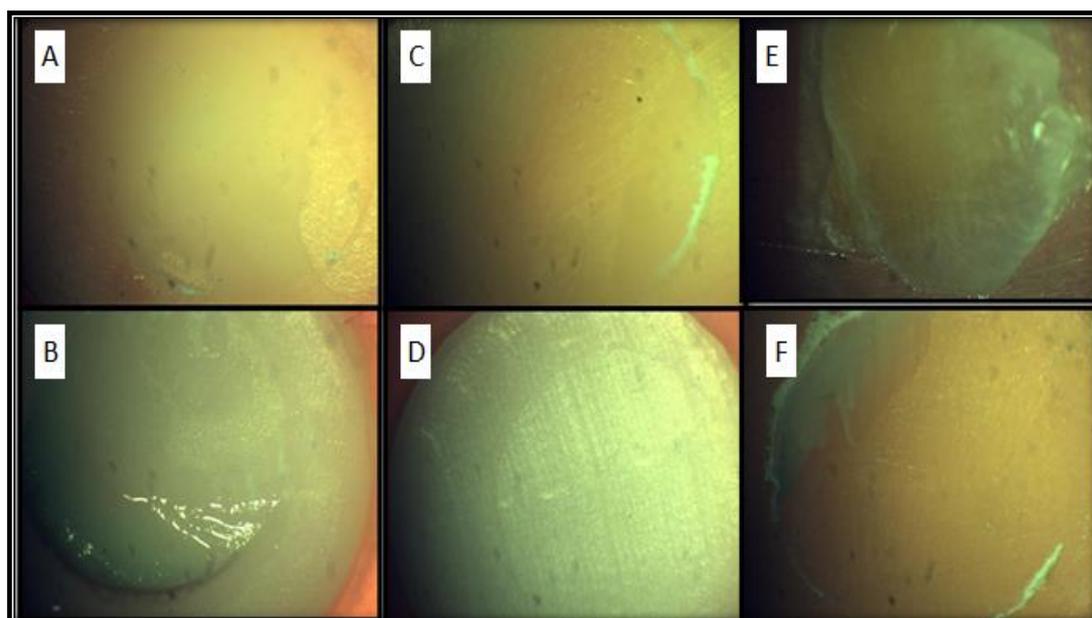
Duncan	Cement groups	Subset alpha=0.05	
		1	2
	Rely X™U200	4.7194	
	BisCem®	8.0453	
	TheraCem®Ca		14.0435
	Sig.	0.066	1.000

Results of the mode failure percentage of three SARC's bonded to three different substrates shown in Table (8), Fig(2) BisCem® bond to enamel showed high adhesive failure mode but with dentin mixed failure is higher, zirconia showed adhesive failure mode higher. TheraCem®Ca bonded to enamel showed the three types of failure modes, adhesive followed by mixed and last one is cohesive. With dentin TheraCem Ca showed mixed type

followed by adhesive. Thera Cem®Ca when bonded to zirconia, showed a higher percentage of mixed failure. RelyX™U200 showed a high percentage of mixed failure with enamel, but with dentin showed the three-failure mode with a higher percent of mixed failure followed by cohesive failure and the lowest was adhesive one, with zirconia showed a high percentage of adhesive failure and lower mixed failure with no cohesive failure.

**Table (8):** Showed Mode of Failure Percentage of Three SARC's Bond to Three Substrates

Substrates	Cements types	Adhesive	Cohesive	Mixed
Enamel	BisCem	60%	0	40%
	TheraCem Ca	50%	10%	40%
	RelyXU200	30%	0	70%
Dentin	BisCem	40%	0	60%
	TheraCem Ca	40%	0	60%
	RelyXU200	10%	40%	50%
Zirconia	BisCem	60%	0	40%
	TheraCem Ca	20%	10%	70%
	RelyXU200	80%	0	20%



**Figure (2):** Types of Failure Under Stereomicroscope with Magnification 1.5X, (A&B) Showed Cohesive Failure, (C&D) Showed Adhesive Failure, (E &F) Showed Mixed Failure.

For non-parametric data collected from stereomicroscope after give code to each type of failure, Kruskal Willis test of unrelated samples results showed that the three SARC's bonded to three different substrates have no significant difference in failure mode. When BisCem bonded to the three substrates (enamel, dentin, and

zirconia)  $P=0.596$ . TheraCem Ca bonded to enamel, dentin and zirconia showed no significant difference occur in failure mode  $P=0.380$ . RelyXU200 showed a significant difference when it bonded with dentin cohesive failure mode occur in high level and the case of adhesive failure mode with zirconia  $P=0.026$ , Table(9).

**Table (9):** kruskal willis Test Results of the Three SARC's Bonded to Three Substrates

Cement \substrate	Mean rank	Kruskal willis test	Asym sig
BisCem\ Enamel	14.50	1,036	0.596
BisCem\Dentin	17.50		
BisCem\Zirconia	14.50		
TheraCemCa\Enamel	13.05	1.935	0.380
TheraCem Ca\Dentin	15.60		
TheraCem Ca\zirconia	17.85		
RelyXU200\Enamel	18.40	7.290	0.026
RelyXU200\Dentin	18.20		
RelyXU200\Zirconia	9.90		

Results of EDX analysis comparison between the control group and specimens after cement application. Showed that the calcium and phosphorus ions reduced from dental surface enamel and dentin. Zirconia specimens did not contain calcium and

phosphorus ion except in the case of the TheraCem Ca both ions present on the zirconia surface strontium ion in form of (SrF<sub>2</sub>) standard label present in both TheraCem Ca and BisCem. As shown in Table (10,11,12)

**Table (10):** Showed the EDX Analysis for Enamel Substrate after Cement Application Compared With Control Enamel Group

Element	Enamel control	BisCem bind to Enamel wt%	TheraCem Ca bind to Enamel wt%	RelyXU200 bind to Enamel wt%
CK	20.91	19.32	29.42	30.90
OK	57.60	62.57	61.66	58.68
Na K	0.85	0.96	0.76	0.70
Mg K	0.15	0.22	-	0.11
Al K	0.14	-	-	-
PK	8.27	7.84	4.83	4.89
Ca K	12.07	8.96	3.33	4.61
Cl K	-	0.12	-	-
NK	-	-	-	-
Si K	-	-	-	0.12
Sr K	-	-	-	-
SK	-	-	-	-

**Table (11):** Showed EDX Analysis For Dentin Substrate After Cement s Application Compared With Control Dentin Group

Element	Dentin control	<u>BisCem</u> bind to Dentin wt%	<u>TheraCem Ca</u> Bind to Dentinwt%	<u>RelyXU200</u> bind toDentin wt%
C K	21.23	41.79	51.96	20.38
O K	57.70	46.50	38.22	57.98
Na K	0.78	0.24	0.26	0.80
Mg K	0.19	0.17	0.12	-
Al K	-	0.62	-	-
P K	8.29	0.96	1.97	8.61
Ca K	11.81	0.71	2.4	12.09
Cl K	-	-	-	0.12
N K	-	7.19	0.75	-
Si K	-	1.35	0.97	-
Sr K	-	0.38	0.38	-
Br	-	-	0.85	-
S K	-	0.08	0.18	-

**Table (12):** Showed EDX Analysis of Zirconia Substrate after Application of Resin Cement Compared With Zirconia Control Group

Element	Zirconia control wt%	<u>BisCem</u> bind to zirconia wt%	<u>TheraCem Ca</u> bind to Zirconia wt%	<u>RelyXU200</u> bind to zirconia wt%
C K	25.27	36.77	57.82	-
O K	32.81	29.90	39.08	42.87
Na K	-	-	-	-
Mg K	-	-	-	-
Al K	0.42	1.22	-	3.25
P K	-	-	0.12	-
Ca K	-	-	0.03	-
Cl K	-	-	-	-
N K	-	-	-	-
Si K	-	0.31	1.22	-
Sr K	-	-	0.29	-
Zr K	21.72	18.24	-	50.23
B K	17.92	11.97	-	-
Os K	1.29	0.98	-	-
Hf K	0.56	0.62	-	1.09
Y K	-	-	-	2.56
F K	-	-	0.71	-
Br K	-	-	0.69	-
S K	-	-	0.03	-
Yb	-	-	0.01	-

## DISCUSSION

Nowadays, there is a high demand to develop indirect restorative material, which stimulates the natural appearance of dental tissues and is associated with excellent mechanical properties, cementation consider a very important step for the success of such restoration<sup>(15)</sup>. The purpose of this study is to evaluate and compare SBS of three different SARC's BisCem<sup>®</sup>, TheraCem<sup>®</sup>Ca and RelyX<sup>TM</sup>U200 bonded to three different substrates enamel, dentin, and zirconia. Study failure mode under a stereomicroscope with EDX analysis to the surface of the substrate.

As known, self-adhesive resin cement did not require substrate surface conditioning or priming procedure before cement application, the only pre-application procedures are polishing for dental specimens and polishing with sandblasting for zirconia.

The study results of shear bond strength for three SARC's bonded to enamel showed no statistical performance difference between them. Although RelyXU200 gives higher bond strength. While with dentin, all three SARC's showed a decrease in bond strength except RelyXU200, which showed relatively higher bond strength when compared with TheraCem Ca, which showed the lower bond strength. TheraCem Ca showed higher bond strength with zirconia when compared with BisCem and RelyXU200, while RelyXU200 showed lower bond strength.

Bonding of SARC's relies on chemical reactions rather than micromechanical interlocking.<sup>(16)</sup> During SARC's application on tooth structure the acidic functional monomer of the cement demineralized the hard dental tissue chelate the calcium ion of the hydroxyapatite, creating a chemical bond<sup>(17)</sup>, and despite the low initial acidic pH only superficial layer of dental tissue demineralized.

Due to the phosphoric group action on dental tissue, there was a release of alkaline fillers with dissolved ions of hydroxyapatite led to a gradual increase in pH level to reach pH7 and that finally neutralized the remaining acidic group the structure become neutral and hydrophobic<sup>(2)</sup>.

SARC's can partially dissolve the smear layer while keeping the smear plugs within the opened dentinal tubules<sup>(18,19)</sup>, increase smear layer thickness negatively affected the bond strength also ion from the dissolved smear layer could rapidly neutralize functional acidic monomer which led to decrease its effect on tooth structure and decrease bond strength. The low bond strength of SARC's recorded with dentin is related to their low ability to demineralizes and infiltrate the dentin<sup>(20)</sup> and viscosity which interfere with the formation of a true hybrid layer<sup>(21)</sup> which consider the main retentive aid in dentin.

A study conducted by Chen *et al.*, 2010 used BisCem and RelyXUnicem with different types of surface treatment found that application of both types of cement to

dentin without surface treatment other than polishing with 600-grite sandpaper showed lower bond strength. Scan electron microscope images of Chen study to the interface area proved that its smooth no resin tags extended from the surface of the resin cement or interlocking structure formed. This means that micromechanical retention was negligible for the bonding of SARC since no micromechanical interlocking contributed to the bond strength of the specimen to non-condition group chemical bonding and hybrid layer may possible maintained the bond.

Lozada & Morales, 2017 compare bond strength of four SARC with conventional control group bond dentin and ceramics and found that there were significantly lower bond strength values of SARC than conventional multistep system<sup>(20)</sup>. This may be related to the quality of the dentin interface is closely related to the extent of monomer infiltration into the demineralized dentinal collagen<sup>(24,25)</sup>. The lower bond strength may be partly or completely related to SARC's ability to interact chemically with hydroxyapatite in dentin, rather than to micromechanical adhesion since they only act superficially with dentin<sup>(26, 27, 28)</sup>

A study conducted by Han showed that 48 hours after polymerization only RelyXU100 presented a neutral value of 7 whereas pH for MaxCem was 3.6 a low pH value lasting for some time may have a deleterious impact on bonding strength to dentin<sup>(29,30)</sup>.

Elkamhawy *et al.*, 2016 studied SBS of ceramic laminate veneers to enamel and enamel and dentin complex bonded with different adhesive luting systems and concluded that enamel dentin complex showed lower SBS than enamel group which means that the type of adhesion surface had the highest effect on SBS value. These results agree with our study and with other similar studies such (Abo Hamar *et al*, Chiba *et al*, Ozturk *et al* and Biar *et al*<sup>(31-34)</sup>.

TheraCem<sup>®</sup> Ca showed higher bond strength with zirconia than BisCem<sup>®</sup> and RelyX<sup>™</sup>U200 and this may be due to the type of functional monomer which differ according to cement type besides surface treatment type<sup>(35,36,37)</sup>

TheraCem<sup>®</sup> Ca contains 10-methacryloyloxydecyl di-hydrogen phosphate (10-MDP) an ester functional acidic monomer that forms low soluble calcium salts and interact chemically with calcium ion in collagen fibrils in dentin<sup>(38-41)</sup> and induce high bond strength with dental ceramic.

MDP bond had a great affinity to the oxide layer on zirconia surface beside other factors such as fillers particle size and viscosity all affect the bond with zirconia. Bond strength is influenced by resin wettability on the zirconia surface, which reduces the contact angle between zirconia and adhesive resin cement and creates intimate contact between them.<sup>(35)</sup>

Initially after mixing the cement become highly acidic and hydrophilic for bonding

and better adaptation to the surface<sup>(42)</sup> and with the progression of the chemical reaction between acidic monomer with apatite and ion release from filler particle ( $\text{Ca}^{+2}$  & Ph) neutralized the remaining acid group the structure become neutral and hydrophobic.

Mechanical properties of resin cement and adhesive interface durability are considered to be proportional to the rate of inorganic filler contained in all resin cement which makes the material more resistant to aging in an acidic environment. TheraCem Ca induce a strong neutralization reaction due to its mineral contained resulting in low hygroscopic expansion stresses this mainly of high benefit in the case when there is no dental substrate only restorative material to help in the neutralization process which relies exclusively on the intrinsic self-neutralization reaction of the cement<sup>(43,44)</sup>.

The problem associated with SBS test design was limited contact area, which made pressure application step to increase adaptation difficult, furthermore, the pH of SARC is another factor in their performance, which must provide the acidity required for demineralizing the substrate avoiding excessive hydrophilicity<sup>(42)</sup>. Light cured the cement associated with an increase in its viscosity which reduces penetration ability of the cement<sup>(45)</sup>, and the design of shear bond test imposes onus prepare cylindrical shape of the cement bond to the substrate, curing of such thick

specimens lead to high polymerization shrinkage away from the bonded surface<sup>(46)</sup>.

The failure mode-detection percent results for three substrates with each type of SARCs showed that each type of cement gives a different failure mode but mainly of adhesive and mixed type. However, cohesive failure occurs in a low percentage with TheraCem<sup>®</sup> Ca in the case of enamel and zirconia, but RelyX<sup>™</sup>U200 showed a relatively higher percentage with dentin. According to Barbosa *et al.*, 2012 cohesive failure type after 24 hours of cementation occur when resin cement was partially retained on substrate surface such as zirconia in the study, also due to chemical affinity between acidic monomer of self-adhesive cement and substrate such in RelyX<sup>™</sup>U200 and dentin in our study, but zirconia, in contrast, showed failure mode tended to be adhesive or mixed.

According to Pisani-Proenca *et al.*, 2011 neutralization due to the water released from the chemical reaction between resin cement and dental enamel further increases the pH of the material. high viscosity which compromises infiltration of resin particles, leading to short resin tags formation and deficient chemo-mechanical interaction of SARCs on dental enamel results in a less durable adhesive interface increasing the probability of adhesive failure<sup>(30)</sup>.

Malysa *et al.*, 2020 analyzed failure-mode of self-adhesive resin cement with CAD-CAM restoration from one side and dentin from the other and showed that all ceramics material used in the study bonded

to dentin with SARC, adhesive failure at the dentin \cement interface is the most prominent because dentin \cement interface was weaker than resin cement\ceramic interface, due to limited ability of the SARC to demineralized dentin<sup>(23,50,51,15,52)</sup>.

Statistical analysis for the three substrate bonds to each type of resin cement as following BisCem<sup>®</sup> and TheraCem<sup>®</sup>Ca showed no significant difference in a failure mode with the enamel, dentin, and zirconia but RelyX<sup>TM</sup>U200 showed significant difference when bonded to dentin, cohesive failure was relatively high also adhesive failure with zirconia was high. That means RelyX<sup>TM</sup>U200 showed favorable bond performance with dental tissue enamel and dentin but less performance with zirconia. TheraCem<sup>®</sup>Ca showed the best results with zirconia but low bond to dental tissues, BisCem<sup>®</sup> showed acceptable bond strength with three substrates.

When compared dental and zirconia control group with dental and zirconia specimens EDX analysis the results showed a decrease in calcium and phosphorus weight percent (wt %) after 24 of the resin cement application on tooth structure which is related to the type and pH of the acidic functional monomer of each resin cement. BisCem<sup>®</sup> showed a high reduction of calcium and phosphorus wt% followed by Theracem<sup>®</sup>Ca and lesser calcium and phosphorus reduction with the RelyX<sup>TM</sup>U200. while zirconia does not contain calcium and phosphorus ions in its

composition but the presence of calcium and phosphorus on its surface after TheraCem<sup>®</sup>Ca application due to cement main component. Also, the presence of other elements such as strontium atoms in form of strontium fluoride with BisCem<sup>®</sup> and TheraCem<sup>®</sup>Ca. Calcium and phosphorus ion neutralized resin cement as mentioned before also these ions can be incorporated into demineralized dental tissue or precipitated within the defect at the interface between the tooth and restoration and contribute to its sealing<sup>(53)</sup>.

Hassan *et al.*, 2012 explained that the Sr<sup>+2</sup> was included in the glass content of glass-ionomer and resin cement which use to give radiopacity but some studies document that the combined effect of F<sup>-</sup> & Sr<sup>+2</sup> in bioactive material give synergistic effect on the acidic medium lead to a decline in dental caries because the creation of few structural changes enhance remineralization process and make hydroxyapatite more resistance to acidic dissolution. F<sup>-</sup> & Sr<sup>+2</sup> might have substituted calcium and hydroxide in apatite crystal and incorporation into the crystal lattice of the precipitate on dentin surface, as result fluoride ion increase to reach 100 ppm lead to CaF<sub>2</sub> deposition<sup>(55-57)</sup>.

## CONCLUSION

The variation in performance of SARC is related to differences in components such as filler type and content & type of functional acidic monomer. Thera Cem<sup>®</sup>Ca show improvement in SBS to zirconia

because its contain calcium, phosphorus beside other elements in its composition and MDP functional acidic monomer, also the different substrates affect the SBS of SARC's BisCem and RelyX™U200 show better performance than TheraCem®Ca bond with dental tissues but lower SBS results with zirconia.

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