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Evaluation of Micro-Tensile Bond Strength of A New Resin Composite Restorative Material at Two Different Dentin Depth After Aging (An In-Vitro Study)

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KEYWORDS

Tensile, Bond, Cyclic, Loading, Dentin

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ABSTRACT

Aim: Evaluation of micro-tensile bond strength of new resin composite restorative material to dentin at two different levels of dentin and after aging. **Subjects and Methods**: Twenty healthy human molar teeth were selected for the study. Flat preparation was done below the DEJ by 0.5mm. (superficial dentin)), and below the DEJ by 1.5 mm. (deep dentin), all teeth restored with (Estelite sigma quick) resin composite. The teeth were divided into four equal groups: group ST1(prepared in superficial dentin level (0.5mm below DEJ) and subjected to cyclic loading fatigue represented 24 hours), group ST2 (prepared in superficial dentin level (0.5mm below DEJ) and subjected to cyclic loading fatigue represented 6 months), group DT1 (prepared in deep dentin level (1.5mm below DEJ) and subjected to cyclic loading fatigue represented 24 hours), and group DT2 (prepared in deep dentin level (1.5mm below DEJ) and subjected to cyclic loading fatigue represented 6 months). Teeth were then sectioned in a mesiodistal and bucco-lingual to obtain beams. Micro-tensile bond strength was measured using universal testing machine. Statistical analysis was performed using Graph pad Instat (Graph pad,Inc); Software for windows. **Results:** There was a statistically significant difference between the four tested groups, The highest mean value of (MPa) recorded in groups prepared in the superficial dentin level (ST1) group followed by (ST2) group, The data showed there was a statistically significant difference between groups subjected to different cyclic loading time. **Conclusion**: Estelite sigma quick resin composite has good bond strength especially at superficial dentin depth.

INTRODUCTION

In recent years, study in this branch of restorative dentistry has been encouraged by the growing popularity of tooth-colored restorations. Because they are favoured by both patients and dentists, resin composites are widely employed in tooth restoration. They can be utilised in conservative cavity preparation, have good physical qualities, and have a colour that is close to that of a natural tooth, among other advantages.[1]

However, this process always needs an intermediate bonding agent that enters the dentin and/or enamel and largely creates what is known as micromechanical bonding. In order to establish micro-retentive porosities where resin monomers can enter and polymerize, the dental substrate must first have its minerals removed by acid etching.[2]

Dentin is made up of collagen matrix and apatite crystallites. Dentinal tubules extend from the pulp to the enamel and are bordered by hypermineralized, collagen-deficient peritubular dentin. Intertubular dentin is made up of mineralized collagen fibers and is found between dentinal tubules. Dentin mineral density, tube diameter, and collagen composition vary with depth. Dentinal tubules grow numerically and diametrically with depth, but intertubular dentin shrinks from superficial to deep dentin. As a result, the intertubular matrix in the superficial dentin is denser, forming the hybrid layer.[3] Because dentinal tubules account for the majority of dentin's water content, the intrinsic wetness of dentin increases with depth. [4] Because dentin substructures vary in depth, the effectiveness of adhesive systems differs, even when the same adhesive system is used.[5]

There are numerous testing modalities for measuring the bond strength of dental adhesives and restorative composites to the tooth substrate; one of the established testing procedures, more often used in recent years, is the micro-tensile bond strength test (MTBS). In comparison to standard testing methods, this test takes a relatively modest adhesive surface area into account and can distinguish well between various adhesives with good bonding performance.[6]

Chewing simulator is a device that try to recreate the oral environment in order to test dental materials under settings as similar to in-vivo as possible. It has a specimen positioning device that simulates human jaws and able to generate movements that resemble human mastication. it is advantageous to evaluate the effects of cyclic loading on bond strength.[7]

Therefore, this study was conducted to evaluate the micro-tensile bond strength of new resin composite restorative material to dentin at two different levels of dentin and after aging. The null hypothesis there is no difference in microtensile bond strength between superficial and deep dentin and there is no effect of cyclic loading on the microtensile bond strength of the new resin composite restorative material.

MATERIALS AND METHODS

Ethical regulation

This study was approved by the Ethics Committee of Faculty of Dentistry, Minia University, Egypt (Meeting no. 93 & Decision no. 696).

The information of the materials used for teeth restorations with different methods presented in Table 1.

Table (1) *The specification, composition, manufacturer, and lot number of the materials used in this study:*

Materials	Specifications	Composition	Manufacturer	Batch number
Estelite sigma quick resin composite	Visible light-cured resin composite	Supra-Nano Filled Universal Composite Filler system: Silica zirconia filler (82% by weight/ 71% by volume) Matrix system: Bis-GMAand TEGDMA.	Tokuyama Dental Corporation, Tokyo, Japan	291E79
Palfique Universal bond	One-step universal-bond self-cured	Contains phosphoric acid monomer, Bis-GMA, TEGDMA, HEMA, MTU-6, silane coupling agent, Peroxide, Borat catalyst, Acetone, Isopropanol and Purified water		099E30

Teeth selection:

Twenty healthy human molar teeth that had just undergone extraction from a patient (20 to 30 years old) were chosen for this study. The teeth were collected from the outpatient clinic of Minia University Dental Hospital which were extracted for periodontal reasons. A 7X lens was used to magnify the teeth to rule out teeth-containing fractures. All the chosen teeth were manually scaled, polished with non-eugenol polishing paste at low speed, and kept in a solution of 0.1% thymol at 4°C (Formula e Acao, Sao Paulo, Brazil) to be tested one month following extraction.[8]

Teeth mounting and preparation of occlusal surface:

Using hollow metallic cylindrical templates, the teeth's roots were inserted in acrylic resin blocks at the CEJ (cemento- enamel junction). To stabilize the mold during specimen preparation, an internal split Teflon component (20mm diameter x 20mm height) was ringed by an external copper ring (30mm diameter x 25mm height). Each tooth was then prepared to have two different occlusal levels according to the tested dentin levels as superficial (0.5mm below DEJ) and deep dentin (1.5mm below DEJ). so that both levels were subjected to the same test variables.

Each tooth's proximal surfaces were initially prepared flat with a cylindrical flat-ended diamond stone (ISO# 111/014, Mani Inc.). Utsunomiya, Japan) mounted on a high-speed hand piece (Sirona Inc. Germany) with copious air-water spray. This procedure was then repeated on the mesial and distal surfaces of each tooth. To standardize the superficial dentin level, a line was meticulously drawn half a millimeter below DEJ on both the mesial and distal sides of the tooth with a stable extra-fine black marker and a ruler. The two proximal threads were then carefully joined to form a circle around the tooth. Using the same diamond stone, the occlusal side of each tooth was ground flat to expose the designated superficial dentin level (0.5mm below DEJ) that is parallel to the occlusal and perpendicular to the long axis of the tooth.[9]

An extra-fine black marker was then used to split the occlusal surface into two equal mesial and distal portions. Three mark points were put 1 mm below the level of the prepared smooth dentin surface on the tooth's distal surface and the distal half of both buccal and lingual surfaces. To construct half a circle, a line was drawn connecting the marks on the three surfaces. After that, the distal half of each tooth was trimmed to the line with a cylindrical diamond stone installed in a high-speed handpiece with air-water spray to generate a deep dentin level (1.5mm below DEJ) in the distal half.[10] After that, the exposed dentin surface was polished with 600-grit silicon carbide paper to create a homogeneous standardized smear layer (fig. 1A).

Grouping of the specimens

The teeth specimens were divided into four groups of 10 specimens each: group ST1(prepared in superficial dentin level (0.5mm below DEJ) and subjected to cyclic loading fatigue represented 24 hours), group ST2 (prepared in superficial dentin level (0.5mm below DEJ) and subjected to cyclic loading fatigue represented 6 months), group DT1 (prepared in deep dentin level (1.5mm below DEJ) and subjected to cyclic loading fatigue represented 24 hours), and group DT2 (prepared in deep dentin level (1.5mm below DEJ) and subjected to cyclic loading fatigue represented 6 months).

Restorative procedures:

The prepared tooth surface is rinsed with water and dried gently using air tip. The bonding procedure was done in accordance to the manufacturer's instructions. The bond (Tokuyama palfique universal bond , Tokuyama Dental, Tokyo Japan) was applied in a single coat and agitation is done for 20 seconds on the dentin surface using micro brush (Dental Bond Brush, Unipack, China), followed by gentle air drying for 20 sec. A nano-hybrid resin composite (Tokuyama Estelite sigma quick, Tokuyama Dental, Tokyo Japan) was used for buildup. Tofflemire matrix system (DDP, stainless steel, 2014, Pakistan) was used to encircle the tooth. The composite restoration was packed first in the deep dentin using gold plated applicator (AMERICAN EAGLE composite SET, USA), till reach the superficial level then light cured for 20 sec using light curing unit 1,470 mW/cm2 intensity (3M, Elipar, Deep Cure-S LED Curing Light USA). Another 2mm of resin composite was packed then light cured for 20 sec. After complete build up the tofflemire matrix band was removed and composite was cured for 40 seconds.

Cyclic Loading fatigue:

Mechanical loading with thermocycling that represent two different aging periods was performed using a programmable logic controlled equipment; the four stations multimodal ROBOTA chewing simulator (ACTA Fatigue tester, Amsterdam, Netherlands) integrated with thermo-cyclic protocol operated on servo-motor (Model Ach-09075dc-T, Ad-Tech Technology, Berlin, Germany). The specimens were embedded in Teflon housing in the lower sample holder. A weight of 5 kg, which is comparable to 49 N of chewing force, was exerted. The test was adjusted firstly to ST1 and DT1 specimens for 417 cycles to clinically simulate the 24 hours chewing condition. Chewing simulation was applied with the following parameters: 3 mm of rising / vertical movement, 1 mm of horizontal movement, 90 mm/s of rising / forward speed, 40 mm/s of descending / backward speed, and 1.6 Hz of cycle frequency.

The work was then repeated to subject ST2 and DT2 specimens to simulate 6 months of intraoral aging, the specimens were subjected to 75,000 cycles with 600 thermal cycles (5˚/55˚C, dwell time 25 seconds).[11]

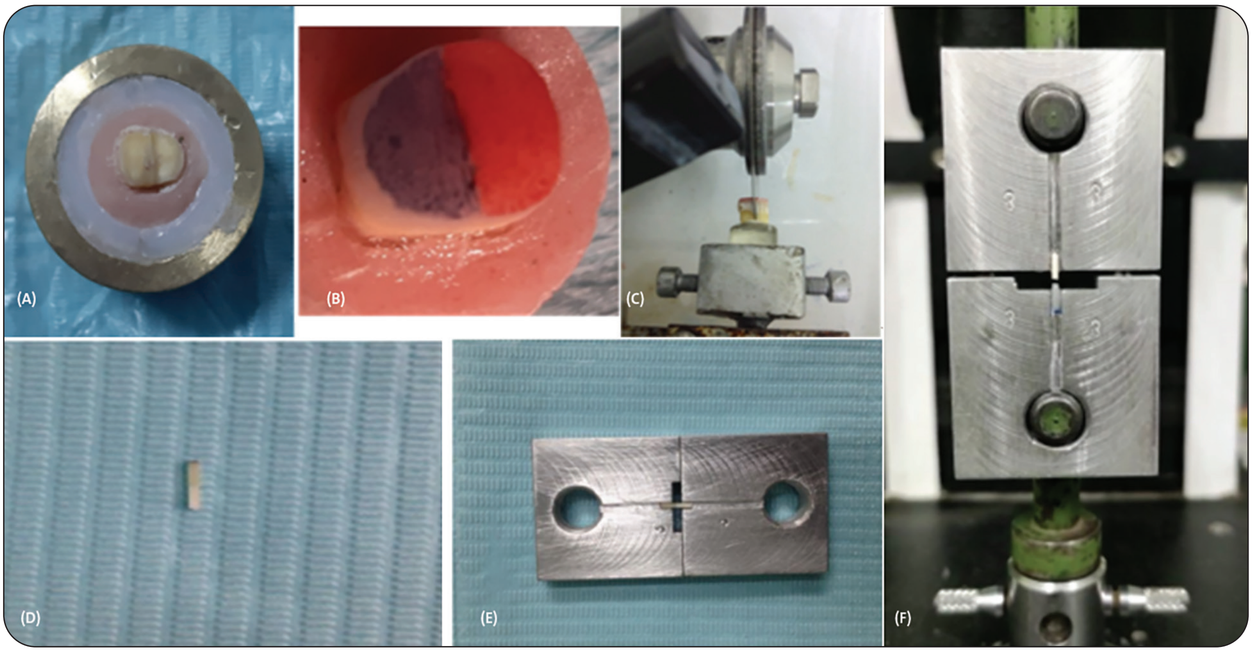

Fig. (1) (A) Occlusal dentin preparation with two levels superficial and deep. (B) Tooth color coded with black color for deep dentin and red color for superficial dentin. (c) Linear precision saw used for sectioning. (D) Beam after being separated. (E) Beam mounted in Geraldeli’s jig. (F) Beam after tensile load application.

Sectioning of the specimens:

Each tooth was colored coded with black color for composite bonded to deep dentin and red color for composite bonded to superficial dentin (fig. 1B). Restored teeth were serially sectioned in buccal-lingual and mesial distal direction by a diamond disc (fig. 1C) (Buehler Diamond Wafering Blade 11-4245, Buehler Ltd., Lake Bluff, IL, USA) in a cutting machine (Isomet 4000, Buehler Ltd., Lake Bluff, IL, USA) at 200 RPM, under copious amount of water to obtain beam-shaped specimens, with a cross section area of 1.0±0.1mm^2**.** (fig 1D)

Micro-tesile bond strength evaluation:

The universal testing machine (Instron model 3345 ; Instron, Norwood, MA, USA) was used to evaluate the microtensile bond strength of each beam.Half of the beams were evaluated after being subjected to 24 hour cycles, while the other half was examined after being subjected to 6 month cycles. Beams were mounted on the universal testing machine using Geraldeli's jig (fig. 1E). Each beam was positioned in the jigan's central groove and cemented in place with cyanoacrylatebased glue (Zapit, DVA Inc, USA). The jig was in turn mounted into the universal testing machine with a load cell of 50 N. Tensile load was applied, at a cross-head speed of 0.5 mm/min, until bonding failure of the specimen occurred (fig. 1F). Bond strength was estimated in Mega Pascal using a special designed software (Bluehill Lite soft ware, Instron, MA, USA).

Statistical analysis

Two–way ANOVA (analysis of variance) was performed to detect the effect of each variable. Student t-test was used for pair-wise comparison between the mean when ANOVA value were significant. The significant level was set at ≤0.005. Statistical analysis was performed using Graph pad Instat (Graph pad,Inc); Software for windows.

RESULTS

Table (2) *Mean and standard deviation (SD) of micro-tensile bond strength (MPa) for the different groups:*

Microtensile bond strength (MPa)	Dentin Depth	Cyclic loading		P -Value
		24 hours (T1)	6 months (T2)	
		Mean± SD	Mean± SD	
	Superficial (S)	24.96±0.64^{Aa}	20.63±1.24^{Ab}	<0.05*
	Deep (D)	19.11±2.6^{4B}a	16.32±1.6^{4B}b	<0.05*

Significant difference is shown by means with different capital letters in the same column; significant difference is indicated by means with different small letters in the same row.

** significant (p<0.05) Ns; non-significant (p>0.05)*

Table (2) showed that there was a statistically significant difference between groups prepared in superficial and deep dentin, where. The highest mean value of (MPa) recorded in groups prepared in the superficial dentin level (ST1) group followed by (ST2) group, while the lowest mean value measured in the group prepared in deep dentin level (DT2). The data showed there was a statistically significant difference between groups subjected to different cyclic loading time, where the highest mean value found in groups subjected cyclic loading (24 hours) (ST1) and (DT1) groups.

DISCUSSION

The µTBS test was chosen, because it allowed for the collection of multiple specimens from a single tooth, and because it was widely believed to be the most accurate method for determining the true interfacial binding strength between an adhesive substance and the target substrate. Additionally, compared to conventional procedures, which used a bonded area of 7–12 mm.[12], small bonded surface areas of about 1 mm^2 may offer stronger bond strengths in addition to better management of regional variances (superficial versus deep dentin).[13]

Estelite sigma quick resin composite, with filler sizes ranging from 100 to 1000 nm and an average of 200 nm, was chosen for the study because it comprises homogenous silica-zirconia supra-nano monodispersing spherical fillers. They have a 71% filler weight. The rapid amplified photo polymerization initiator (RAP technology) is used in Estelite Sigma Quick Composite. By using this technology, camphorquinone molecules can be recovered, and each one of them has the potential to produce several free radicals, which reduces the volume ratio of camphorquinone compared to other standard catalysts and speeds up polymerization.[13]

A new type of single-step self-etch adhesive, categorized as "universal" or "multi-mode" has been recently introduced for patient care. These adhesive systems are recommended by dental manufacturers for use both with and without acid pretreatment of enamel surfaces.

Palfique universal adhesive has a new three-dimensional self-reinforcing (3D-SR) monomers that have more number of groups interacting with calcium groups and enhanced 3D cross-linking, and MTU-6 components present in Bond A and also y-MPTES present in Bond B, which are unique in Palfique Universal Bond.[14] Additionally, there is no curing step that avoids the possibility of losing its effectiveness makes this system to have superior results when compared with others.

With a more significant impact associated with bulk filling and deep narrow cavities, flat dentin preparation was chosen because cavity dentin design was proven to alter μTBS most likely due to changes in the configuration ("C") component. In Class I and Class V cavities, "cavity" design bond strength values were much lower than "flat dentin" values. Since a greater C-factor increases the strains caused by polymerization contraction across the tooth-composite interface, and the bond in the restored cavity becomes weaker. [15]

The results of the current study showed that palfique universal bond had higher micro-tensile bond strength on superficial dentin level than deep level at after cyclic loading 24hrs and 6months aging. Depth-associated variations in dentin components include tubule density, diameter and the mineral contents of the matrix influence mechanical properties, such as elastic modulus, which reduces the position moves from the DEJ to the pulp, the reduced mechanical properties of deep dentin induced a lower resistance to fracture, which resulted in lower bond strength and increased internal fracture formation when compared with superficial dentin.[16]

These results corroborated those of Yang B et al. (2006)[17], who discovered that superficial dentin had a significantly higher μTBS than deep and cervical dentin due to the possibility of greater micromechanical adhesion to collagen fibrils in the hybrid layer because superficial dentin has a larger intertubular dentin area rich in collagen fibrils. Additionally in agreement with Pegado RE et al., 2010[18], who discovered that for all adhesive systems tested, the bond strength obtained with superficial dentin was significantly higher than that of deep dentin due to the low contents of intertubular dentin and collagen fibrils as well as the high water content.

This was also explained by Kumari R et al.,2015[19] to either variations in chemical composition or variations in regional wetness (dentin permeability). The organic solvent of some bonding systems may be diluted by this water, leading monomers to exit the soluble phase and form resin globules in the solution. The hydrophilicity of the adhesive system to superficial dentin and highly acidic monomers that are able to form strong ionic interactions with dentin's calcium content are the reasons Cevik P et al., 2020[20] found greater μTBS to shallow dentin surface than to deep dentin surface. Deep dentine's decreased binding strength can be explained by the fact that the mineral content declines as dentin depth increases.

However, these results were at variance with those of Kwong S. et al., 2002[21], who discovered that a self-etch adhesive system had a better binding strength with deep dentin. This may be because self-etch adhesives are beneficial at bonding since they can simultaneously demineralize and infiltrate the dentine surface to the same depth, which should presumably prevent the adhesive from partially penetrating the exposed collagen network.

The space occupied by resin tags, the region of intertubular dentin where the resin is infiltrated, and the area of surface adhesion all affect the bond strength of dentin-bonding agents at any depth. Due to an increase in the total surface area accessible for the formation of hybridised tubule walls and intertubular dentin, deep dentin was able to produce stronger bond strengths.[22] Additionally, Zhang L et al. (2014)[23] discovered no distinction in the bonding strength between superficial and deep dentin. The primary cause of the variation across research may be the different type and chemical makeup of the investigated adhesives. According to reports, the adhesives' hydrophobicity, wetting capacity, and solvent type may all have an impact on how well they adhere to various materials.

Many problematic variables exist in the oral environment surrounding dental resto-rations, including humidity, acidic or basic pH, cyclic loading, and temperature.[24,25] Clinically, mechanical failure of dental restorations develops after many years of service due to cyclic interactions between maxillary and mandibular teeth, limiting the restorations' survival probability and lifetime.[26] The current study used a ROBOTA chewing simulator integrated with a thermo-cyclic protocol driven on a servo-motor to simulate load cycling and temperature fluctuations in the mouth.

The results of the current study showed that Estelite sigma quick resin composite had higher microtensile bond strength values at 24hrs compared to 6months cyclic loading either in the specimens prepared in superficial or deep dentin level. The water of thermocycling is responsible for bond deterioration, it causes repeated thermal expansion and shrinkage of the materials used, which causes fatigue in the interphase and, therefore, reduction in the bond strength.[27] Nikaido et al., 2002[28] also reported that the fatigue stress accelerated the degradation or alteration of substrates peripheral to the hybrid layer. Hybrid layer formation is an important factor to create a strong bond between the resin and dentin. The loading stress is concentrated mostly in the interface between adhesive and top of the hybrid layer.

The current study's findings were in line with those of Castro et al., 2004[29], who discovered that thermo-mechanical loading decreased the microtensile bond strength of nanohybrids. They explained this by stating that thermal cycling promoted stress on the interface, and that when mechanical cycling was applied, the effect of loading was accelerated by thermo-cycling, resulting in lower bond strength.

According to the results of this study, the null hypothesis was rejected because there was a difference in microtensile bond strength between superficial and deep dentin and there was an effect of cyclic loading on the microtensile bond strength of the new resin composite restorative material.

CONCLUSION

Within the limitation of this study, it can be concluded that:

1. Estelite sigma quick resin composite has good bond strength especially at superficial dentin depth.
2. Cavity depth has an apparent influence on bond strength of Estelite sigma quick resin composite.
3. Load and thermal cycling deteriorate the bond strength of resin composite to dentin.

RECOMMENDATIONS

1. Estelite sigma quick resin composite could be used in small and medium sized cavities.
2. It is advisable to use chewing simulator as a research tool for stimulation of the environmental condition.

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قوة رابطة الشد الدقيقة للمواد الترميمية الجديدة المركبة من الراتينج عند مستويين من عاج الأسنان وبعد الشيخوخة (دراسة في المختبر)

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الملخص :

الهدف: تقييم قوة الرابطة الشد الدقيقة للمواد التصالحية الجديدة المركبة من الراتينج للعاج عند مستويين مختلفين من العاج وبعد التقادم.

المواد والطرق: تم اختيار عشرين ضرسًا بشريًا سليمًا للدراسة. تم التحضير المسطح أسفل DEJ بمقدار 0.5 مم. (العاج السطحي)، وتحت DEJ بمقدار 1.5 ملم. (العاج العميق). ترميم جميع الأسنان بمركب الراتنج (ESTELITE SIGMA QUICK). تم تقسيم الأسنان إلى أربع مجموعات متساوية: مجموعة ST1 (محضرة في مستوى عاج سطحي (0.5 مم أقل من DEJ) وتعرضت لإجهاد تحميل دوري يمثل 24 ساعة). المجموعة ST2 (محضرة في مستوى عاج سطحي (0.5 مم أقل من DEJ) وتعرضت. إلى إجهاد التحميل الدوري يمثل 6 أشهر) . مجموعة DT1 (محضرة في مستوى عاج عميق (1.5 مم أقل من DEJ) وتعرض لإرهاق تحميل دوري يمثل 24 ساعة) . ومجموعة DT2 (محضرة في مستوى عاج عميق (1.5 مم أقل من DEJ) وتعرض لإجهاد التحميل الدوري لمدة 6 أشهر). ثم تم قطع الأسنان في الوسط القاصي واللغة الشدقية للحصول على الحزم. تم قياس قوة رابطة الشد الصغير باستخدام آلة اختبار عالمية. تم إجراء التحليل الإحصائي باستخدام لوحة الرسم البياني INSTAT (لوحة الرسم البياني. وشركة)؛ برنامج ويندوز.

النتائج: كان هناك فرق ذو دلالة إحصائية بين المجموعات الأربع المختبرة. حيث سجلت أعلى قيمة متوسطة لـ (MPA) في المجموعات المحضرة في مجموعة مستوى العاج السطحي (ST1) تليها مجموعة (ST2). وأظهرت البيانات وجود دلالة إحصائية. الفرق بين المجموعات المعرضة لوقت تحميل دوري مختلف.

الاستنتاج: مركب ESTELITE SIGMA الراتينج لديه قوة ارتباط جيدة خاصة عند عمق العاج السطحي.