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EFFECT OF PREHEATING CYCLES ON MICROSHEAR BOND STRENGTH OF NANOHYBRID RESIN COMPOSITE LUTED TO CAD/CAM CERAMIC

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ABSTRACT

Objective: This study aimed to investigate the microshear bond strength of adhesive resin cement to Leucite-reinforced CAD/CAM ceramic and compare it with that of prewarmed nanohybrid resin composite.

Materials and methods: Sixty Empress CAD ceramic plates were prepared. Each ceramic plate received five Tygon tube micro-cylinders filled with bonding agents creating 5 resinous micro-cylinders on each ceramic plate. In the first group (GpA), they were filled with resin cement, nanohybrid resin composite as a bonding agent was applied in the second group (GpB), after warming at 50°C, in the third group (GpC), two prewarming cycles were performed. Light-curing for 20seconds was applied. Micro-shear bond strength testing (µSBS) was performed using universal testing machine with a crosshead speed of 1 mm/min until failure occurred. The µSBS was calculated in MPa by dividing the load (Newton) over the respective surface area (mm 2). Data were tabulated and analyzed and showed normal distribution when checked using Kolmogorov–Smirnov test. One-way ANOVA used to compare between the tested groups for µSBS data followed by Tukey HSD post-hoc test for pairwise comparisons. Significant level was set at 5% (α=0.05).

Results: One- way ANOVA showed a significant difference between tested groups ($p<0.001$). Where heated composite (two prewarming cycle) showed the highest mSBS compared to other groups ($p<0.05$). Nevertheless, heated composite (one prewarming cycle) showed an improved µSBS compared to resin cement group, but the increase was insignificant ($p=0.081$).

Conclusion: Pre-heated nanohybrid resin composites seem to be a potential alternative to resin cement to lute ceramic restorations.

KEYWORDS: Nanohybrid composite, resin cement, preheating, prewarming, leucite Ceramic.

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INTRODUCTION

The growing demand for esthetics in dentistry led to the development of wide variety of materials that can be used for direct and indirect esthetic restorations.

Ceramics have excellent properties; they are chemically stable, biocompatible, has low thermal conductivity, high compressive strength, as well as high translucency and fluorescence, and coefficient of thermal expansion similar to that of tooth structure. Moreover in order to improve their mechanical properties, the development of new materials with higher mechanical strength, such as those reinforced by leucite, or lithium disilicate, or those infiltrated by glass, or the injected or machined ones occurred.[1]

The clinical success of indirect ceramic restorations depends on many factors; proper diagnosis, correct design, operator knowledge, and the composition of the ceramic, in addition to a very important factor which is the cementation (material and technique) that affect the bond strength of the ceramic restoration. [2]

For retention adhesive cementation is necessary for most dental ceramics, especially leucite reinforced ceramics. Self-adhesive resin cements is a simple, less sensitive material that overcame some limitations of conventional cements. [3]

The use of light-curing composites is recommended with indirect ceramics since they allow prolonged working time, excess removal after insertion of indirect ceramics, and their color is stable.[4]

Nanohybrid composites have been produced by adding Nano sized particle (5-100μm) in the microhybrid resin composites in order to improve the mechanical properties. Many latest findings stated that refrigerating resin composites as was previously recommended, is one of the worst things dentists were asked to do. [5]

On the contrary, preheating of resin material prior to placing has recently gained popularity because it reduces the viscosity, improves marginal adaptation, enhances handling, increases polymerization and degree of conversion, and improves mechanical and physical properties of the material. [6]

Since little information was available regarding the bond behavior of tooth, different adhesive cements-ceramic restoration complex, the aim of this *in vitro* study was designed to investigate the microshear bond strength of adhesive resin cement to Leucite-reinforced ceramic and compare it with that of prewarmed nanohybrid resin composite. The null hypothesis was that the microshear bond strength of nanohybrid resin composite will not improve by warming.

MATERIALS AND METHODS

I) Materials

Brand name, material description, composition, and manufacturer are listed in table (1).

TABLE (1): List of Materials

Brand name	Material description	Composition	Manufacturer
IPS Empress CAD	Leucite-reinforced high-performance blocks for the CAD/CAM technology	Contains up to 45% by volume tetragonal leucite for fabricating all-ceramic sintered restorations.	Ivoclar Vivadent AG, Liechtenstein
Variolink Esthetic LC	Light curing luting composite	*The monomer matrix is: -urethane dimethacrylate. -methacrylate monomers. *The inorganic fillers are: -ytterbium trifluoride, spheroid mixed oxide. *Initiators. *stabilizers *pigments The particle size is 0.04-0.2 µm. The mean particle size is 0.1 µm. The total volume of inorganic fillers is approx. 38%v.	Ivoclar Vivadent AG, Liechtenstein
Tetric EvoCeram Composite	Universal Nano-hybrid composite	*The monomer matrix is : -Bis GMA, -TEGDMA *The inorganic fillers are: -Barium glass, silica dioxide, ytterbium trifluoride, bariumalumino, fluorosilicate glass. The particle size is: ranging in size from 40 to 3,000 nm Average particle size of 550 nm (0.7 µm) The total volume of inorganic fillers is 53-55% by volume (75-76% by weight).	Ivoclar Vivadent AG, Liechtenstein
Porcelain Etchant	9.5% Acid Gel	Buffered Hydrofluoric (HF) Acid	Bisco Products Inc., USA
Porcelain Primer	Pre-Hydrolyzed Silane Primer; enhances the bond between resin cements and ceramics, porcelain and pre-cured composites.	Mmethacryloxy propyl trimethoxy	Bisco Products Inc., USA

II) Methods

Empress CAD plates were fabricated.

Specimens Preparation

Empress CAD blocks were used to prepare sixty ceramic plates. Preparation was done by cutting the blocks using ISOMET 4000 (BUEHLER, LAKEBLUFF, USA) (figure 1) with blade speed of 2150 rpm and continous water irrigation.

Each ceramic plate obtained had the following dimensions [14mm x 12mm x 2mm thickness] (figure 2). Ceramic plates were divided into three groups twenty plates each (n=20), according to the type of bonding system used; in the first group (GpA) self adhesive light cured resin cement was used, in the second group (GpB) heated resin composite (one pre-warming cycle) was used, while in the third group (GpC) heated, cooled and reheated resin composite (two pre-warming cycles) was used.

Fig. (1) Empress CAD block cutting.

Fig. (2): Empress CAD plates, 2mm thickness

Surface Treatment

Each ceramic plate was embedded in a wax block prior to surface treatment, in order to facilitate handling and fixation during micro-shear testing. Plates surfaces were treated with 9.5 % porcelain acid etch [7] for 60 seconds then washed for 180 seconds with air/water spray and finally dried with hot air blower in order to insure complete removal of the acid.

After the micromechanical treatment (etching) of the ceramic plates, a micro-brush was used to add a single layer of silane coupling agent [8] that was allowed to dry for 60 seconds, then further dryness was done for 10 seconds using oil/water free compressed air.

Application of bonding system

Each ceramic plate received five Tygon tube micro-cylinders; 1mm in diameter and 1mm height. Tubes were positioned over each plate surface and filled with bonding agent according to groups, creating 5 resinous micro-cylinders on each ceramic plate.

Tygon tubes of the first group (GpA) were filled with resin cement which was injected into the tubes lumens using an intra-radicular tip with the auto-mixing tip.

In order to use nanohybrid resin composite as a bonding agent in the second group (GpB), composite was warmed and softened first at 50°C,[9], using a Dental Resin Composite Heater /Warmer device (3H Magic Box Dental Resin Composite Softener Heater Warmer 40°C-45°C-50°C, Italy Design/33500020/composite softner) (figure 3), The composite needed a maximum of 10 minutes to reach 50°C, then the warmed light cured resin composite was injected in the tygon tube micro-cylinders lumens, (one prewarming cycle) was performed.

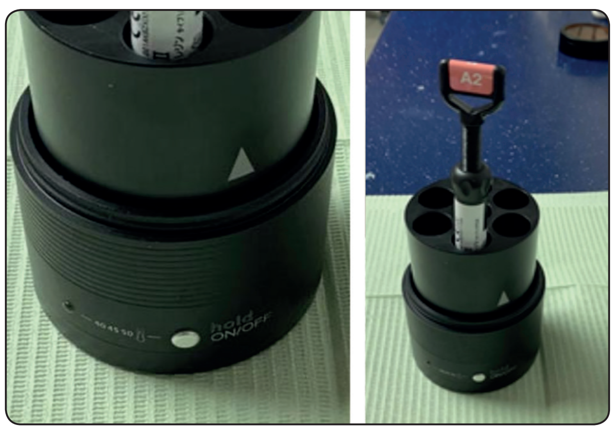

Fig. (3): Dental Resin Composite Heater / Warmer

While in the third group (GpC), two prewarming cycles were performed which means that the resin composite was warmed up to 50°C, using the same device and procedure done with (GpB), bench cooled to room temperature and rewarmed to 50°C, then the two-cycle warmed resin composite was injected in the tygon tube micro-cylinders lumens.

After filling all tygon tubes, with either resin cement or resin composite (Gps B&C), each was light-cured for 20 seconds using Woodpecker (*Zhengzhou Linker Trading, HE NAN, CHINA*) LED light curing unit of 1100mW/cm^2, as recommended by the manufacturer.

Micro-shear bond strength test

Micro-shear bond strength testing (µSBS) was performed using universal testing machine (*Intsron 3345, BOSTON, USA*). Wax blocks were fixed in the lower jig of the universal testing machine and a wire (0.2 mm diameter) was fixed to the upper jig and attached to the resinous micro cylinder as shown in. Micro-shear test was applied with a crosshead speed of 1 mm/min until failure occured. The µSBS was calculated in MPa by dividing the load (Newton) over the respective surface area (mm^2).

Statistical analysis

Data showed normal distribution when checked using Kolmogorov–Smirnov test. One-way ANOVA used to compare between the tested groups for µSBS data followed by Tukey HSD post-hoc test for pairwise comparisons. Significant level was set at 5% (α=0.05). Statistical analysis was performed with statistical package for social sciences (IBM SPSS, version 23, Armonk, USA).

RESULTS

One- way ANOVA showed a significant difference between tested groups ($p<0.001$). Where heated composite (two prewarming cycle) showed the highest mSBS compared to other groups ($p<0.05$). Nevertheless, heated composite (one prewarming cycle) showed an improved µSBS compared to resin cement group, but the increase was insignificant ($p=0.081$). (Table 2) (Figure 4).

TABLE (2): Mean and Standard deviation of µSBS for different tested groups.

	Microshear (MPa)		*p*-value
	Mean	**SD**	
Resin cement	29.59^b	3.45	<0.001
Heated composite (one pre-warming cycle)	32.09^b	3.1	
Heated composite (two pre-warming cycle)	35.52^a	2.72	

Means with different letter within each column indicates a significant difference at p<0.05

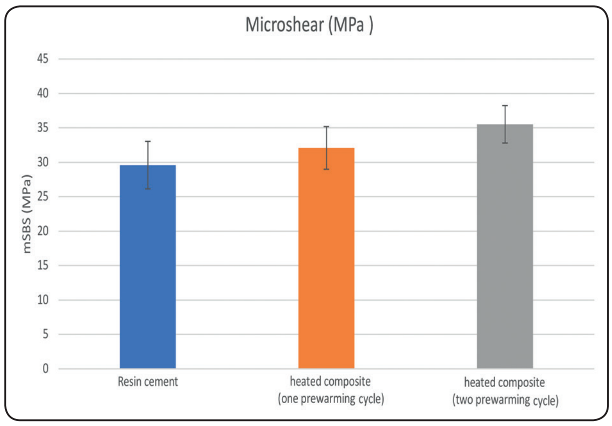

Fig. (4): Bar chart showing the µSBS.

DISCUSSION

Ceramics are prone to fracture under chewing loads, they should be adhesively bonded to the underlying tooth to dissipate the occlusal forces, gain support, and form an integrated structure. Since the prognosis of ceramic restorations and their clinical success are influenced by bonding effectiveness[10], it is important to identify and evaluate the resin cement bond strength; at the cement/tooth and the cement/ceramic restoration interface.

The selection of resin cement material depends on the type of material, its clinical behavior, cement film thickness, fit of the ceramic restoration, and cement bonding (adhesion of resin cement to ceramic restorations has two aspects; adhesion of resin cement to enamel and dentin, and adhesion of resin cement to etched and silanized ceramic surface), so if little or no retention is available in the preparation design, the bonding has to be effective. Therefore, the durability of adhesive resin cement to ceramic material will basically depend on the bond strength of the luting cement both to the tooth and the ceramic.[11]

The resin cement material bonding capacity to the ceramic substrate and the bonding failure types and rates vary due to variations in chemical composition, wetting capacity, viscosity, and mechanical properties of each resin cement.[12] Therefore, it is recommended in many researches to cautiously evaluate the adhesive performance of resin cement materials.

Nanohybrid resin composite is viscous but it has been used in stress bearing areas and showed long-term success. There is a new originating interest in making this highly filled resin composite less viscous by preheating without influencing its properties. On the contrary preheating highly filled resin makes it easier to be extruded from compules or syringes, enhances its adaptation to cavity walls and margins, decreases voids formation, increases monomer conversion and thereby improves physical and mechanical properties of the final restoration.[13]

Many factors play an important role in the longevity of ceramic restorations, such as tooth preparation design, functional load, and the strength of the adhesive bond, that's why preheated nanohybrid resin composite has been recommended by some researchers for the adhesive luting of ceramic restorations. There is a bifunctional role of bonding etched porcelain with resin composite; the porcelain-resin-tooth complex retains and reinforces the brittle restoration. Low filler content resins as luting cements, have high polymerisation shrinkage and thermal expansion coefficient greater than of enamel and dentine, this will result in microleakage and crack formation within the ceramic restoration. Using nanohybrid rather than resin luting cement could be beneficial in reducing shrinkage stresses due to significantly lower polymerisation shrinkage and coefficient of thermal expansion.[(14)]

Microshear bond strength was introduced in the literature as a mechanical test that solves problems related to microtensile test, it is easier, cheaper and it prevents tension spread in the bond interface that occurs with microtensile test over large areas, also more than one several specimen can be obtained from a single sample without the need to cut it.[(15)]

The present study investigated the micro-shear bond strength of nanohybrid resin composite subjected to one or two prewarming cycles and compared it to micro-shear bond strength to of resin cement, both materials were bonded to ceramic plates in order to test their strength for adhesive purposes.

The results of the present study revealed that heated composite (one prewarming cycle) showed an improved μSBS compared to resin cement group, but the increase was insignificant. Where heated composite (two prewarming cycles; warming, cooling and rewarming) showed the highest μSBS compared to the two other groups.

Daronch et al [(16)] stated that pre-heated resin composite has the same degree of conversion or even higher than that of resin composite cured at room temperature even if the light-curing time was reduced as much as 75%. On the other hand it was observed by **Walter et al** [(17)] that precooling of resin composite decreases the polymerization shrinkage, therefore, manufactures always recommend keeping the resin composite syringes refrigerated.

So, both the effects of prewarming and precooling of resin composite on bond strength to ceramics should be studied.

By investigating the effect of prewarming and precooling on the nanohybrid resin composite, it was found that warming resin composite prior to placement increased the monomer conversion and the polymerization; free radicals and propagating polymer chains became more mobile as a result of decreased resin material viscosity resulting in more complete polymerization reaction (more double-bond formation), greater cross-linking and improved bond strength, all the previous findings were in agreement with **Choudhary et al** [18].

Again **Daronch et al**[16] stated that it is possible to find an increase in immediate degree of conversion of conventional composite by pre-heating and before curing, because when the composite is pre-heated, great shock of molecules occur due to greater molecular agitation, accelerating the polymerization reaction.

Moreover the effect of preheating temperatures on the monomer-to-polymer conversion was significantly affected by preheating the specimens. By improving the monomer conversion, the glass transition temperature increases, inducing a greater amount of conversion at higher polymerization temperatures. In Dimethacrylates small increase in temperature results in a large increase in the polymerization rate, and by improving the degree of conversion, greater cross-linking and better mechanical properties are expected.[19]

Another study revealed the flexural strengths of resin composite were not significantly different at temperatures of 4˚C and 25˚C, and that justified that resin composite syringe should not be refrigerated[20]

A study conducted by Froes-Salgado et al. [21] revealed different findings than the present study and stated that pre-heating nanofilled composite up to 68 degrees ºC prior to light curing did not affect the mechanical properties or the monomer conversion, it only enhanced its adaptation to cavity walls.

In the present study it was observed that the prewarming of nanohybrid resin composite reduced its film thickness to be similar to the film thickness of resin cement, and it is well known that film thickness is an important factor when luting indirect restorations because thick film thickness leads to marginal misfit, allows great amount of resin material to be polymerized with great volumetric shrinkage and so high susceptibility of failure.[7]

In accordance to the observations of this study, **Sampaio et al.**[22] found that the thinner film thickness was formed by the pre-heated conventional composite, probably that occurred because temperature increased agitation of molecules and allowed mass plasticization of unpolymerized material. Moreover preheating provides a lute with a smaller coefficient of thermal expansion, with less polymerization shrinkage and with greater wear resistance at restoration margins compared to conventional resin luting cement.

In another study done by **Rickman et al.** [23] hybrid composite did not prevent proper seating of the veneers. The use of preheated hybrid resin composite improved the handling characteristics of the material by decreasing its viscosity which also aided in achievement of excellent restoration margins. It is known that incomplete seating of a veneer would increase the thickness of resin in the lute space, which could exacerbate the effects of polymerization shrinkage and thermal expansion.

In order to make sure that temperature rise of resin composite will not have any harmful effect on the pulp, a previous study confirmed that when composite was preheated (54°C - 60°C) and placed on 1 mm remaining dentin thickness, the temperature rise inside pulp was 0.8°C while the rise due to light curing was 5°C.[24] So it is safe to warm composite resin within the biologically compatible temperatures and benefit of many improved properties.[25]

Other than warming, resin composite physical and mechanical properties (strength, elastic modulus, and fracture toughness) are also influenced by filler content, filler particles size and distribution, filler volume fraction and filler loading. Resin composites with the highest filler content by volume, exhibited the highest mechanical properties[26][27], this was obvious in the present study when resin composite revealed higher bond strength values compared to adhesive resin cement.

In a study by **Tomaselli** et al. [7] they found that filler content and pre-heating influenced the bond strength of conventional composite to ceramic, they stated that it seemed evident that there are advantages of using the thermoplastic technique, then they concluded that the pre-heated conventional composites (65 wt% filler), is a potential alternative to lute ceramic veneers over flowable composites (50 wt% filler).

The third group (GpC) in this study was performed in order to investigate the effect of repeated preheating and cooling of resin composite on its bond strength to IPS Empress CAD ceramic. Upon testing resin composite heating (warming), time to reach the desired temperature, the temperature reached, temperature change (temperature loss upon composite transfer from the heating unit and injection into the preparation), cycling time (cooling and repeated heating), all should be assessed.

It was stated before that it is possible to store resin composite syringes or compules in the warming device at desired temperature during all working hours. However, this extended heating storage at elevated temperatures has some drawbacks; low molecular weight components of the photoinitiator system could be volatilised, subsequently compromising light polymerisation. It was also reported that after eight hours of storage (prolonged) at 54.5°C, hybrid composite showed reduced degree of conversion compared to samples that had been stored at room temperature, it was also reported that after only four hours of storage (short-term) at the same temperature no adverse effect occurred. So, it is important to limit storage times to four hours and to recap the to be re-used compules.[28] In this short term storage spontaneous polymerization does not occur until temperature reaches (140°C to 200°C), evaporation and photoinitiator degradation does not occur until 90°C. [23]

Shortly after resin composite material extruded out of the compule or syringe that was removed from the heating device, large amount of pre-heating temperature is lost. [29] Daronch et al. [30] found that for microhybrid composite, 50% of the temperature gained was lost after two minutes and 90% was lost after five minutes, but degree of conversion is still increased compared to non-preheated resin with this degree of temperature loss (cooling), also during clinical application we place resin composite in small increments that allows quick placement and limited cooling.

On the other hand high degree of conversion is related to increased polymerization shrinkage, and so the delay between preheated resin composite application and its polymerization revealed some disadvantages as microleakage, due to shrinkage as the material cools and not immediately. Therefore, it might be of value to reduce light polymerization time for preheated composite in order to overcome its increased resin conversion[29] . But, as we knew the gained temperature is not surely maintained for a sufficient time (extrusion, placement and light curing), so this point needs further investigations.

After viewing all the previous findings, two prewarming cycles representing resin composite heating immediately prior to each restoration placement were performed, rather than prolonged warming hours, this showed significant improvement in microshear bond strength compared to the two other groups; by gaining more advantages of very short-term preheating together with avoiding the drawbacks of prolonged heating through the working hours.

Any mechanical tooth preparation has a risk of pulpal damage, no one of the previously discussed articles mentioned any consequence of using preheated resin composite on pulp vitality, but we still cannot surely verify the dramatic improvement of physical and mechanical properties of preheating highly filled resin restorations. The sure thing is enhanced handling and improved adaptability of these composites, so, using nanohybrid composites for porcelain luting purposes, makes composite resin heating devices a certain useful instrument to be added to our dental armamentarium[23].

CONCLUSION

Within the limitations of this study, it is possible to conclude that pre-heated nanohybrid resin composite seems to be a potential alternative to resin cement for luting ceramic restorations. Further investigations regarding the number of prewarming cycles and the prewarming temperatures should be conducted.

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