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Thermal Effect Shock on the Enamel-composite Restoration Interface

Hazem Abouelleil[1*], Christophe Jeanin[1,2] and Brigitte Grosgogeat[1,2]

[1]Laboratoire des Multimatériaux et Interfaces, UMR CNRS 5615, Université Lyon1, Villeurbanne, France.
[2]UFR Odontologie, Université Lyon1, Service de Consultations et de Traitements Dentaires, Hospices Civils de Lyon, Lyon, France.

Authors' contributions

This work was carried out in collaboration between all authors. Authors CJ and HA designed the experiment, wrote the protocol and wrote the first draft of the manuscript. Authors HA and BG managed the analyses of the study and literature searches. All authors read and approved the final manuscript.

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ABSTRACT

Aim: To study the effect of thermal shock on the enamel – composite restoration interface as compared to standard thermal cycling protocol.
Methodology: Box shaped cavities were prepared in thirty mandibular third molars, the cavities were restored using two step etch and rinse adhesive: Adper™ Scotchbond™ 1 XT (3M™ ESPE™, St. Paul, USA), and nano-hybride resin composite Filtek™ Z250 (3M™ ESPE™, St. Paul, USA). Specimens were divided in 3 groups. The first group was thermal cycled for 600 cycles, the second group was submitted to 600 thermal shock cycles using Oral B waterjet device, and the third group was a control group. Teeth specimens were evaluated for dye leakage using 2% Basic Fuchsin dye for 24 hours, all bonded teeth were subsequently sectioned perpendicularly into

**Corresponding author: E-mail: hazemabouelleil@yahoo.com;*

0.9 ± 0.1 mm^2 sticks that were loaded on universal testing machine to obtain the ultimate tensile strength. Values were analyzed with one way ANOVA post hoc Tukey HSD (SPSS version 23) with 95% confidence interval.

Results: Both thermal shock and thermal cycling groups had significantly higher dye leakage values along the interface as compared to the control group. The microtensile bond strength values were significantly lower for the thermal shock group as compared to the control group, no significant difference was found between the thermal cycling and the control group.

Conclusion: Thermal shock was shown to induce more stress on the interface, which may lead to cracks and gap formation overtime.

Keywords: Thermal shock; thermal cycling; dental composite; restoration interface.

1. INTRODUCTION

In modern day practice, resin composites are the first choice for most dental practitioners, not only because of increased patient demand for more esthetic restorations, but also due to advances in material composition and polymerization techniques, leading to better mechanical and physical properties and increase in the life expectancy of the restoration [1,2].

Dental composite restorations are composed of inorganic filler dispersed in a resin based oligomer matrix; a coupling agent such as silane is used to bond these two components. Long term aging significantly reduces the mechanical properties, proposedly because of degradation of the polymer network and leaching of the unreacted polymers [3]. According to long term studies, secondary caries is considered to be the main cause of resin restoration replacement. The tooth material interface seem to be the most vulnerable area where the effect of moisture, polymerization contraction together with mechanical stresses and the stresses caused by fluctuating temperature and pH; lead to eventual failure of the adhesive interface and gap formation [3,4,5]. Another important point, is that even though the thermal diffusivity and thermal conductivity of composite restorations are close to that of the human teeth, the adhesive resin layer is markedly different due to its polymer composition and absence of filler content [6,7].

No single method exists for evaluating the effect of aging on the mechanical properties of resin composites and the tooth material interface. Aging in water is the conventional method used to simulate intraoral aging; basic standardized tests demands a minimum of 24 hours of water immersion before testing [8]. It was reported that water immersion can lead to significant reduction in the mechanical properties of resin composites (30- 55%). The varying results obtained with different materials are attributed to difference in polymer matrix composition or type of filler. It is believed that water sorption and swelling of the network lead to reduction in friction between the polymer networks and softening of the material [3,9,10].

On the other hand, the oral environment encompasses a wide range of temperatures (−5 to 76.3℃). The differences between the physicothermal properties of the tooth components and the restorative material used, lead to the development of thermal stresses with the maximum stress on the bonding interface. These together with other masticatory stresses can easily induce the failure of the bonding interface, and hence failure of the restoration [11].

The exchange of hot and cold food and drink usually results in an abrupt and sudden change in the oral temperature, meanwhile the temperature of the dental tissues, restorative materials and the bonded interface between them occurs at a finite rate according to their heat transmission properties [12,13].

Several methods exist for the characterization of the thermal properties of the tooth components, yet significant differences have been obtained with the reported results, this could be due to the heterogeneity of the samples, or more over to the heterogeneity of the dental structure itself. The heat transfer mechanism inside a restored tooth is very difficult to measure, mostly because of the complex tooth geometry, and the varying thermophysical properties of the different constituents. The thermal performance of restored teeth was reported to differ significantly from intact ones, due to differences in thermal properties of tooth components and the restorative material [14].

Thermal cycling is considered as the most effective method for simulating the aging process

in the oral cavity. Conversely several numerical methods such as the finite element method (FEM) and the finite difference method (FDM) models were developed to analyze the temperature transfer across enamel, dentin and various restorative materials, but significant discrepancy still exists between experimental measuring and mathematical modelling [9,14].

It was found that regardless of the protocol, significant decrease in bond strength occurs after thermal cycling [13,15]. Accelerating the rate of thermal change across the tooth surface, would presumably lead to more stress build up. The difference in heat transfer rate and the thermal coefficient of each of the constituents of the tooth material interface would play the main role in stress build up, while the resistance to bond failure would depend mainly on the mechanical properties of the interface components, most notably the elastic modulus [14].

In this work an experimental setting was done that would convey sudden thermal change to the tooth surface, as compared to conventional thermal cycling, and compared to a control group. The null hypothesis would be that no difference exists in the integrity of the interface between the two groups as compared to the control group.

2. MATERIALS AND METHODS

2.1 Specimen Preparation

Following removal from patients (ages 17-27) (following informed verbal consent and in compliance with French legislation, the local ethical committee and the World Medical Association Declaration of Helsinki 2008), thirty caries-free freshly extracted third molars, were kept in a 0.5%-chloramine solution at 4℃ temperature for five days then in distilled water, until further processing (ISO/TS 11405 norm). After cleaning and removal of superficial debris from the surface using a scalpel blade, occlusal surface was flattened and standardized box shaped cavities (4 × 3 × 3 mm) were prepared using diamond and tungsten carbide burs in a high-speed handpiece under copious water spray. The depth of cavities was standardized by marking the burs at 3 mm length prior to use, and the measure was controlled using a periodontal probe. Burs were replaced after ten cavities and no bevels were added at any margin of the preparation. Cavity floors were inspected for absence of pulp exposure. The teeth were kept wet until the adhesive treatment procedure started. The adhesive used were two step etch and rinse: Adper™ Scotchbond™ 1 XT (3M™ ESPE™, St. Paul, USA), and the resin composite used was the nano-hybride Filtek™ Z250 (3M™ ESPE™, St. Paul, USA). Materials were manipulated according to the instructions by the manufacturer: enamel and dentin were etched and then rinsed with water for 10 seconds. Excess water was blotted using a mini-sponge. Immediately after blotting, 2 consecutive coats of adhesive were applied to etched enamel and dentin for 15 seconds with gentle agitation, gentle air blast was applied for five seconds to evaporate solvents. The adhesive was then light cured for 10 seconds. The composite was inserted incrementally in 3 layers cured for 20 seconds each. Restored teeth were inserted in cold cure resin, except for the crown portion to enable handling for the dye leakage, and micro tensile bond strength measurement. Specimens were then divided in 3 groups (10 each) and stored in distilled water for 24 hours at 37℃.

The first group was thermal cycled for 600 cycles (30 seconds dwelling time and 3 seconds interval) (5℃ -55℃), The second group was submitted to 600 thermal shock cycles (10 seconds hot and 5 seconds cold with no interval) (5℃ - 55℃) using Oral B waterjet device regulated electronically with a special electronic board to obtain the required duration and number of cycles (Fig. 1). The third group was a control group that was kept in distilled water inside an incubator at 37℃.

2.2 Dye Leakage

Teeth specimens were covered with two layers of nail polish except for the composite restorations and 1mm around the cavity margins, they were then dipped in a 2% Basic Fuchsin dye for 24 hours, the dye film on teeth surface was then polished off with a polishing disc, and each tooth was then sectioned 2 vertical sections through the center of the restoration using diamond-disc-operating saw at slow speed and under constant irrigation (Isomed, Buehler Ltd, Lake Bluff, IL, USA).

The sectioned teeth were then assessed using a stereomicroscope (Olympus CKX41, Olympus-Europe, Hamburg, Germany) and image analysis software program to measure the length of dye penetration along the interface.

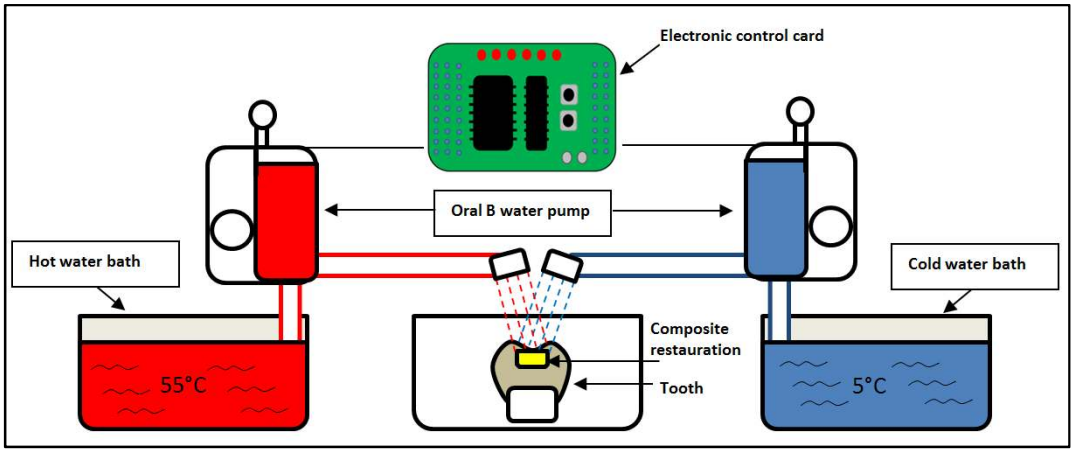

Fig. 1. The experimental setup for the thermal shock experiment

Dye penetration at the restoration tooth interface was scored for the enamel margins

Score 0: No leakage visible at the tooth restoration interface.

Score 1: Penetration of dye along the cavity wall but less than one-half of the length.

Score 2: Penetration of dye along the cavity wall but short of the axial wall.

Score 3: Penetration of dye to and along the axial wall.

The worst score from the all sections of each specimen was recorded.

The microleakage data were analyzed using Kruskal-Wallis statistical tests at a significance level of 5%.

2.3 Microtensile Bond Strength Testing (μTBS)

All bonded teeth were subsequently sectioned perpendicularly and through to the bonded interface into 0.9 ± 0.1 mm^2 sticks using diamond disk wafering blades 15HC (Buelher, D¨usseldorf, Germany) at slow speed and under constant irrigation (IsoMet® Low Speed Saw, Buehler, Lake Bluff, IL, USA). Two stick samples were retrieved from each tooth. The bonded surface area was calculated before each test by measuring the width with digital caliper.

Each specimen was attached to an aluminum device constituted of two symmetric parts, having a central notch (2 mm of depth and width) in order to allow auto alignment. Device surfaces were cleaned with alcohol. Tensile load was applied using a universal testing machine (DY34, Adamel Lhomargy SARL, Roissy-en-Brie, France), at a crosshead speed of .5 mm/min, to obtain the ultimate tensile strength, using a load cell of 1 KN.

Bond strengths of sticks from the same tooth were averaged and the mean taken as one statistical unit. Sticks that failed prematurely were included in the data and given the value of 2 MPa.

The obtained values were analyzed with one way ANOVA post hoc Tukey HSD (SPSS version 23) with 95% Confidence Interval (P=0.05).

3. RESULTS

The results obtained for the dye penetration test are shown in Table 1.

The control group was significantly different from the thermal shock and the thermal cycling groups, while both groups were not statistically different.

Results for the bond strength are shown in Table 2.

Fracture mode was determined at × 50 magnifications with a stereoscopic microscope (Wild Heerbrugg TYP 376788, Wild Heerbrugg, Switzerland) and recorded as cohesive failure and adhesive failure; results are shown in Table 3, the samples that failed prematurely were considered among the adhesive failure group. Fig. 2 shows examples of the adhesive and cohesive failure samples.

Table 1. Dye penetration percentage for each group

Margins for the study groups	0	1	2	3	Mean
Thermal shock	-	5	3	2	1.7
Thermal cycling	-	5	4	1	1.6
Control	4	6	-	-	0.6

A total of 9 samples failed prematurely for the thermal shock group, 3 for the thermal cycling group, while no premature failure was found for the control group.

Table 2. Microtensile bond test results (MPa), similar letters denote statistically homogenous values

	Shock	Cycling	Control
Mean	6,04^{A}	9,11^{AB}	10,38^{B}
Std, deviation	(5,03)	(4,44)	(4,98)

Table 3. Number of adhesive and cohesive failures in each of the three groups

	Adhesive	Cohesive
Thermal shock	16 (80%)	4 (20%)
Thermal cycling	14 (70%)	6 (30%)
Control	5 (25%)	15 (75%)

4. DISCUSSION

Thermal cycling has long been used as the standard method for aging to predict the clinical reliability of various restoration types. Though the entire of the oral environment seem too complicated to be reproduced, it has long been perceived that thermal and mechanical stresses play an important role in the deterioration of the physical and mechanical properties of the restorations. Mostly resin composite restorations and the adhesive interface are the most vulnerable to the oral environment, in comparison with metallic or ceramic restorations [9,16].

In this study amplifying the effect of thermal transition between hot and cold was used to investigate the effect of thermal change on the enamel resin restoration interface. In this work the temperatures used as reference in literature between 5 and 55℃ were used. Some studies have been reported using more elevated temperature whether in actual experimental setup or in simulation computer models, the justification for such exaggerated values was found by the authors to be of little scientific evidence [13,14].

During the act of eating and drinking hot and cold food or drinks, the temperature transfer to the tooth surface occurs abruptly. The thermal conductivity and more significantly the thermal diffusivity of the material control the thermal energy transfer inside the material contained by the fluctuating temperatures inside the oral cavity [10,17].

In heat transfer analysis, thermal diffusivity $\boldsymbol{\alpha}$ is divided by density and specific heat capacity.

$$\boldsymbol{\alpha} = \boldsymbol{k}/(\boldsymbol{\rho c_p})$$

- $\boldsymbol{k}$ is thermal conductivity (W/(m·K))
- $\boldsymbol{\rho}$ is density (kg/m³)
- $\boldsymbol{c_p}$ is specific heat capacity (J/(cal/ g K))

According to Fourier's law of heat conduction the heat flux per unit area $\boldsymbol{q}$ (W/m^2) is given in terms of the temperature $\boldsymbol{T}$ by

$$\boldsymbol{q} = -\boldsymbol{k}\,\Delta\boldsymbol{T}$$

The surface heat transfer coefficient is responsible for the rate at which the temperatures exchange between the tooth surface and the hot or cold food or drink. Its value depends on the nature of the conductive and convective heat transfer processes in the layer of liquid adjacent to the surface of the tooth [11,12]. Understandably this reveals the importance of the existing difference in thermal properties between the three components; dental enamel, restorative filled resin composite and unfilled resin adhesive [18,19].

The differences in physical properties and composition between the components of the interface (Enamel, adhesive resin and composite resin restoration) accordingly would mean that the thermal energy flow inside of each of these components occur at a different rate. Moreover the corresponding amount of thermal energy needed to change the temperature or to affect thermal contraction or expansion is also different [12].

According to Table 4, the thermal diffusivity of enamel is more than double that of the filler free

resin adhesive layer [18,20], meaning that the temperature would travel two times faster into the enamel to the depth X, when at the same time the adjacent resin adhesive and resin composite restorative haven't reached the same temperature. The difference in temperature and coefficient of thermal expansion; would lead to increased stresses between the two (enamel and filler free resin adhesive layer). These thermally induced elastic stresses would lead to the appearance of micro cracks over time.

The magnitude of the stress resulting from a temperature change from T_0 to T_f could be calculated using the equation:

$$\sigma = E_{\alpha l}(T_0 - T_f) = E_{\alpha l}\Delta T$$

Where E is the modulus of elasticity and α_l is the linear coefficient of thermal expansion.

The increase in the rate of change with the thermal shock protocol would also mean less time for the resin to gain or lose thermal energy at depth due to its inferior thermal diffusivity values, accordingly subjecting the interface to more internal stresses [21]. The thermal shock resistance parameter *TSR* takes in account the material elasticity able to absorb such stresses [22], and is given by:

$$TSR \cong \frac{\sigma k}{E_{\alpha l}}$$

The enamel adhesive interface is composed of resin tags mechanically interlocked inside the enamel; constraining its expansion/contraction with thermal changes. Furthermore unfilled resin (adhesive) has relatively higher thermal expansion coefficient compared to that of enamel and even that of the filled composite resin [19].

It was suggested by many authors that that temperature fluctuations during meals are frequent and variable and that alterations in oral temperature occur rapidly while the return to baseline temperature occurs more slowly [10,13]. More over the effect of thermal shock has been examined in a number of studies perhaps to reveal its overlooked impact on the adhesive interface, as shown by the previous equations [10,12,22].

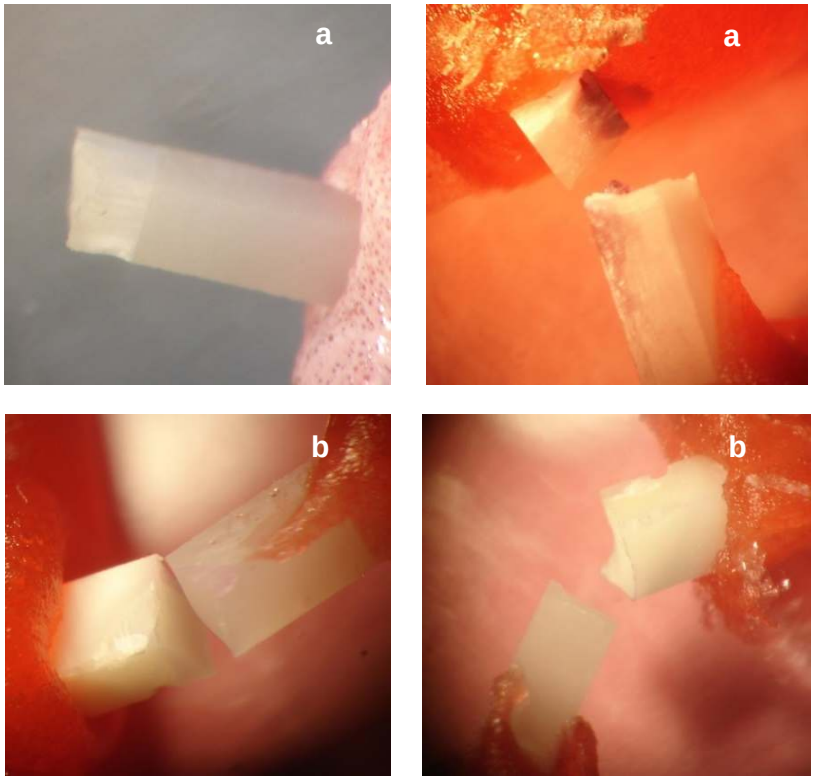

Fig. 2. Different types of failures; adhesive (a) or cohesive (b) as revealed under stereo microscopy

Table 4. Thermal and physical properties of dental tissues and the resin restoration adhesive interface

	Thermal conductivity *k* W /mK	Thermal diffusivity α m^2/s	Density g cm^{-3}	Specific heat cal/ g K	Coefficient of thermal expansion 10^{-6}/℃	Young's modulus GPa
Enamel	0.93	$4.69 \cdot 10^{-7}$	2.97	0.18	16.9	84.1
Resin composite (inorganic filler) Filtek™ Z250	1.1	$6.15 \cdot 10^{-7}$	2.4	0.19	33	16.6
Resin adhesive (no inorganic filler) Adper™ Scotchbond™	1.4	$1.9 \cdot 10^{-7}$	1.1	0.27	62	1.1

Numerical simulations through mathematical modeling have tried to reproduce the complicated oral environment with its complex dental geometry, material properties and *in vivo* biological functions. Yet in spite of the significant amount of research done; discrepancy between the results obtained with these models and experimental measurements show that some of the factors were not considered during the development of these models. The magnitude of the actual stress build up across the interface is frequently underestimated [14,23].

In the present work it was shown that thermal stresses on the enamel adhesive interface had a significant effect on the integrity of the marginal seal of the restoration, after taking in consideration the specific composition and thermal properties of the resin composite restoration and the resin adhesive used [24,25]. The effect was intentionally exaggerated through using a cavity configuration with an elevated C factor. It has been shown with previous studies that the cavity configuration can increase the amount of stress on the marginal adhesive interface [26].

In this work the results obtained for the dye penetration test, show clear tendency to gap formation and dye penetration for the thermal shock samples, and indicating that abrupt changes in temperature could have a more deteriorating effect on the interface, seemingly because of the increased stresses generated due wise [27].

The dye leakage method was criticized by several authors, for its inability to quantify the marginal leakage phenomenon. On the other hand the method was considered by other study groups as an overall evaluation of the interfacial integrity after aging [27].

The results obtained for the micro tensile bonding test showed significant difference in microtensile values between the control group and the thermal shock group, but not with the thermal cycling group, thus demonstrating effect of thermal shock on creating stresses and weakening the bonded adhesive interface. The microtensile bond strength is a widely accepted method for evaluating the bond strength across the interface [28]. In his review Heintze found that microtensile bond testing is more accurate in comparison to other methods used to evaluate the interface strength and the stresses that affect it [27].

An important point to be taken in consideration is the premature failure of the samples; the scientific community pointed the importance to integrate those into the results, while the absolute value was not a matter of agreement [29,30]. The value used in this study to represent premature failure have been 2 MPa, which represents half the minimal bond strength value obtained during testing and in order not to use markedly low values. It should be taken in consideration that 9 out of 20 samples failed prematurely for the thermal shock group, while only 3 for the thermal cycling group, and none for the control group samples. On the other hand the type of fracture whether cohesive or adhesive (Fig. 2) and the number of premature failures as represented in Table 2; show clearly the effect of thermal stresses on the adhesive interface.

The samples studied under scanning electron microscopy as shown in Fig. 3, provided proof that the initiation of failure was mainly between the enamel and adhesive resin part of the interface.

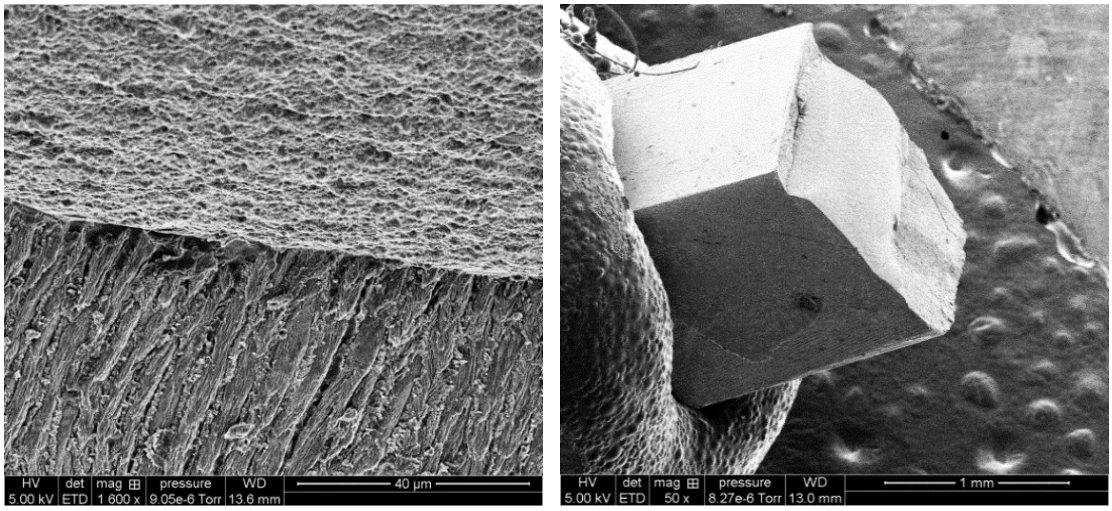

Fig. 3. Scanning electron microscopy images showing initiation of failure between enamel and the adhesive layer

The samples were subjected to 600 thermal cycles corresponding to one month of function in the oral cavity, and which is considered by the norm ISO to be appropriate for simulating the aging of biomaterials *in vivo* [8,13]. The experimental setup has not taken into account the exact time interval between the thermal shock and the thermal cycling (5 and 10 seconds for the thermal shock with no interval between the hot and cold, and 30 /30 seconds for thermal cycling with 3 seconds for the samples to pass between the hot and cold water baths). The slow transition and adaptation that is compensated in the thermal cycling method was meant to be eliminated in thermal shock setup, moreover the continuous waterjet projected on the tooth surface would allow closer contact and more efficient temperature transfer [31].

Adhesion to dentin is more vulnerable to stress and failure. In the present study enamel was chosen as a substrate for testing the interface, mainly because in the clinical situation, enamel anatomically covers the dentin, and the enamel resin interface is the part more exposed in the oral cavity and more subject to temperature changes, physical and mechanical stresses, while most of these are much attenuated by the time they reach dentin [10].

The major shortcomings of the present study are the limited number of samples, together with the limited number of thermal cycles used during the test. In this study only one type of composite resin and adhesive were used, a large variety of adhesives and composite resins should be used to cover the varied compositions and thermophysical properties of existing materials. Future work should evaluate the temperatures, time durations and experimental setup used in the current study.

5. CONCLUSION

Within the limitations of this work, it was shown that thermal shock induces more stress on the enamel-composite restoration interface, which may lead to cracks and gap formation, possibly leading to eventual failure of the restoration overtime.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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