ORIGINAL ARTICLE

Effect of preheating of low shrinking resin composite on intrapulpal temperature and microtensile bond strength to dentin

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A B S T R A C T

The effect of preheating of the silorane-based resin composite on intrapulpal temperature (IPT) and dentin microtensile bond strength (μTBS) was evaluated. For the IPT, teeth ($n = 15$) were sectioned to obtain discs of 0.5 mm thickness (2 discs/tooth). The discs were divided into three groups ($n = 10$/group) according to the temperature of the Filtek LS™ silorane-based resin composite during its placement, either at room temperature ($23 \pm 1\,°C$) or preheated to 54 °C or 68 °C using a commercial Calset™ device. Discs were subjected to a simulated intrapulpal pressure (IPP) and placed inside a specially constructed incubator adjusted at 37 °C. IPT was measured before, during and after placement and curing of the resin composite using K-type thermocouple. For μTBS testing, flat occlusal middentin surfaces ($n = 24$) were obtained. P90 System Adhesive was applied according to manufacturer's instructions then Filtek LS was placed at the tested temperatures ($n = 6$). Restorative procedures were done while the specimens were connected to IPP simulation. IPP was maintained and the specimens were immersed in artificial saliva at 37 °C for 24 h before testing. Each specimen was sectioned into sticks (0.9 ± 0.01 mm^2). The sticks (24/group) were subjected to μTBS test and their modes of failure were determined using scanning electron microscope (SEM). For both preheated groups, IPT increased equally by 1.5–2 °C upon application of the composite. After light curing, IPT increased by 4–5 °C in all tested groups. Nevertheless, the IPT of the preheated groups required a longer time to return to the baseline temperature. One-way ANOVA revealed no significant difference between the μTBS values of all groups. SEM revealed predominately mixed mode of failure. Preheating of silorane-based resin composite increased the IPT but not to the critical level and had no effect on dentin μTBS.

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Introduction

A category of dental composite with a resin matrix, based on ring-opening monomers, has been introduced to the market. This hydrophobic composite drives from the combination of siloxane and oxirane, thus given the name silorane. The major advantage of this restorative material is its reduced volumetric shrinkage [1,2].

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Additionally, the technique of application is one of the ways to improve the success of the restorations. The high viscosity and stickiness of contemporary resin composites make the insertion, as well as adaptation, of the material to preparation walls difficult and unpredictable [3,4]. Preheating of resin based restorative materials (54 or 68 °C) prior to placement and contouring may facilitate ease of composite extrusion and enhance composite adaptation to preparation walls. Other potential benefits include increasing the degree of conversion and wear resistance [5,6].

Combination between the use of low shrinking resin composite and the modified technique of application that was achieved by preheating would be suggested to attain better adaptation [7] and bond strength. Nevertheless, preheating of resin composite was found to increase the intrapulpal temperature [6]. This may raise a concern about the adverse effects on the pulp beyond its physiological tolerable limit especially in deep cavities.

So, it would be of interest to study the effect of preheating of low shrinking resin composite on the dentin microtensile bond strength and the intrapulpal temperature changes. The null hypotheses were: (1) There is no difference in intrapulpal temperature whether silorane-based resin composite is preheated or not. (2) Dentin microtensile bond strength would not differ if silorane-based resin composite was applied at room temperature or after preheating.

Material and methods

A low shrinking silorane-based resin composite Filtek LS (Shade A_3, 3M ESPE, St Paul, MN, USA) and its corresponding adhesive system two-step self-etch adhesive system P90 System Adhesive (3M ESPE, St Paul, MN, USA) were used in this study. Table 1 shows the material brand names, compositions, manufacturers, and batch numbers.

A total of 39 sound upper human premolars; extracted from an age group of 18–20 years, were stored in phosphate buffer solution containing 0.2% sodium azide at 4 °C pending uses within 1 month [8].

Measurement of intrapulpal temperature

Preparation of specimens

Crown segments of fifteen sound human premolar teeth were cut horizontally using a slow-speed diamond saw sectioning machine (Buehler Isomet Low Speed Saw, Lake Bluff, IL, USA) under water coolant into discs of approximately 0.5 ± 0.05 mm thickness (Fig. 1A). From each crown segment two discs were obtained. A digital caliper (Mitutoyo digital caliper, Mitutoyo Corp., Kawasaki, Japan) was used to check the thickness of the discs. Dentin discs were divided into three groups ($n = 10$/group) according to the temperature of Filtek

Table 1 Materials-brand name, compositions, manufacturers and batch numbers.

Materials-brand name	Composition	Manufacturer	Batch no.
P90 System Adhesive Two-step self-etch adhesive system	*Primer:* Phosphorylated methacrylates, Vitrebond copolymer, Bis-GMA, water, ethanol, silane-treated silica fillers, initiators, Stabilisers. PH = 2.7 *Bond:* Hydrophobic dimethacrylate, phosphorylated methacrylate, TEGDMA, silane treated silica fillers, initiators, stabilisers	3M ESPE Dental product, St. Paul, MN, USA	N313983
Filtek LS Low Shrinking Posterior resin composite (Shade A_3)	Silorane resin, initiating system; camphorquinone, iodonium salt, electron donor. Quartz filler, yttrium fluoride, stabilisers, pigments	3M ESPE Dental product, St. Paul, MN, USA	N431331

Bis-GMA = Bis-phenol-glycidyl-methacrylate, TEGDMA = Triethylene glycol dimethacrylate.
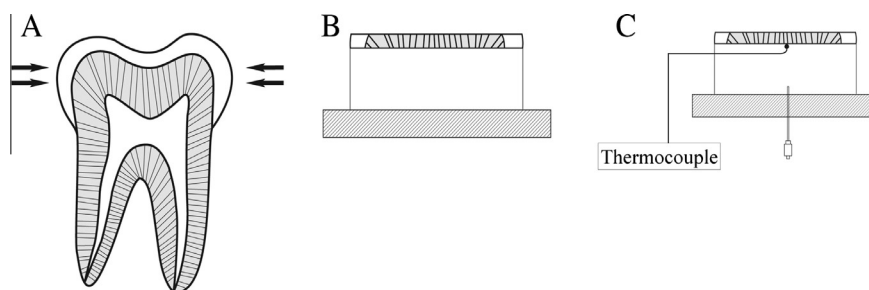

Fig. 1 Specimen preparation for intrapulpal temperature measurement; tooth sectioning to obtain dentin discs (A); dentin disc attached to the transparent tube and the Teflon plate (B); that was penetrated with a butterfly needle connected to the intrapulpal pressure assembly while the thermocouple was fixed (C).

LS resin composite during its application, either applied at room temperature or preheated to 54 °C or 68 °C.

Dentin discs were glued on top of transparent plastic tubes (2 mm diameter and 5 mm length) using cyanoacrylate adhesive (Rocket heavy, Dental Ventures of America, Inc., USA), then, centrally attached to a Teflon plate (15 mm diameter and 1 mm thickness) (Fig. 1B). A 19 gauge butterfly needle (Shanchuan Medical Instruments, Co., Ltd., Zibo, China) was inserted through the centre of the plate. A pin point hole was made 1 mm from the top of the transparent tube to allow a K-type thermocouple (Chromel Alumel, bead style) of a digital logger (Apollo DT301/DT302, Instrumart Green Mountain Dr S Burlington, VT, USA) to penetrate the tube. The thermocouple was positioned against the pulpal side of the dentin disc (Fig. 1C). The whole set-up was then connected to an intrapulpal pressure simulating assembly [9] and placed in a specially constructed incubator adjusted at 37 °C throughout the IPT measurements.

Resin composite application and intrapulpal temperature (IPT) measurement

Resin composite application was conducted under simulated intrapulpal pressure adjusted to 20 mmHg at the dentin surface as checked with the sphygmomanometer [9]. This pressure was held 15 min before and throughout the application of the resin composite restoration. Resin composite was applied in one increment of 1.5 mm thickness either without preheating or preheated to 54 °C or 68 °C using the preheating device (Calset™ device, AdDent Inc., Danbury, CT, USA). Resin composite increment was polymerised for 40 s using blue phase C5 light curing unit (Ivoclar Vivadent, Schaan, Liechtenstein), at intensity 550–590 mW/cm². The light curing tip was positioned 1 mm from the resin composite. Light intensity was checked using LED radiometer (Kerr dental specialties, West Collins Orange, USA) at the beginning and throughout the study [10].

Intrapulpal temperature was measured during the application of the resin composite (T_1). Another thermocouple connected to the logger was used to measure the room temperature which was adjusted to be 23 ± 1 °C outside the incubator (T_2). The readings (thermal per time interval) were digitally displayed on the screen. The IPT records were started at baseline and continued during application and curing of resin composite until returning to the baseline temperature. Ten intrapulpal temperature curves were obtained for each group. An average curve was created for each group and was descriptively analysed.

Microtensile bond strength measurements

Twenty four premolars were used in this test. Occlusal enamel of each tooth was trimmed perpendicular to its long axis, exposing the dentin using a slow-speed diamond sectioning machine (Buehler Isomet Low Speed Saw, Lake Bluff, IL, USA) under water coolant. Additional cut was made parallel to the occlusal surface, 2 mm below the cementoenamel junction to expose the pulp chamber (Fig. 2A). Remnants of pulp tissue in the pulp chamber were removed using a discoid excavator (Carl Martin GmbH, Solingen, Germany) without touching the walls of the pulp chamber [11]. Dentin surfaces were then wet polished with 600-grit SiC paper to create a standard surface roughness and smear layer. The specimens (n = 24) were connected to the intrapulpal pressure assembly during bonding and 24 h storage following the same procedures described by Mobarak [9] (Fig. 2B–E).

Prepared specimens were divided into three groups, (n = 8), according to the same Filtek LS resin composite temperatures chosen for IPT measurements (Fig. 2E). In all groups, P90 System Adhesive was applied to all prepared dentin specimens according to manufacturer's instructions. Primer and adhesive bottles were shacked well before their uses; the
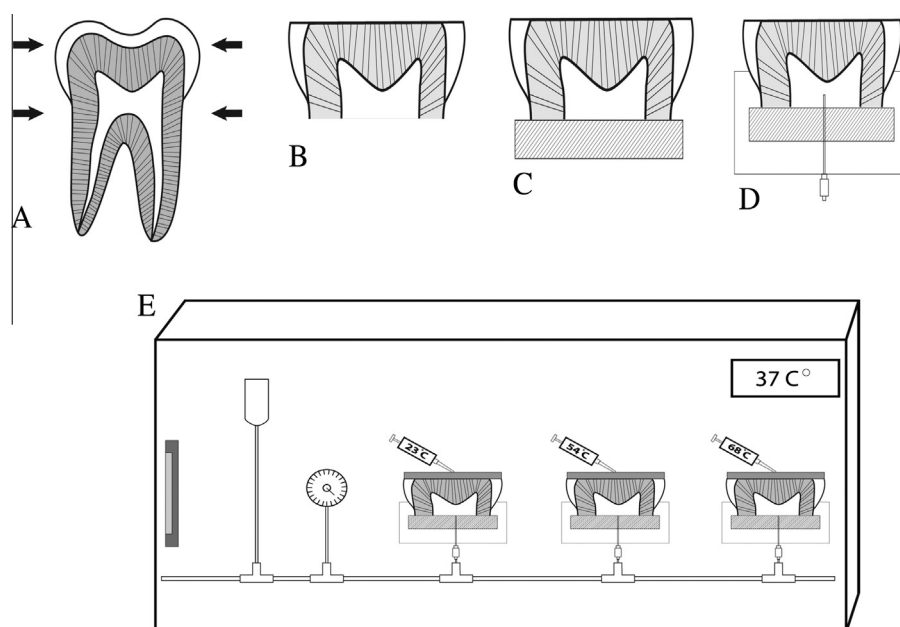

Fig. 2 Specimen preparation for microtensile bond strength; The coronal and cervical cuts (A); The pulp chamber was cleaned (B); The coronal section was fixed to the Teflon plate (C); that was penetrated with the butterfly needle (D); to be connected to the intrapulpal pressure assembly while contained in a specially constructed incubator (E).

primer was applied to the entire surface and gently rubbed for 15 s. The primer was spread to an even film by using gentle stream of air and was cured for 10 s. Thereafter, bond was applied to the entire surface, spread gently using air stream and finally cured for 10 s. Resin composite buildup of 3 mm height was made in two increments (1.5 mm thickness/increment). Each resin composite increment was polymerised 40 s. Light intensity of the curing unit was checked using an LED radiometer (Kerr dental specialties, West Collins orange, USA) at the beginning throughout the study period. Bonded specimens were stored in artificial saliva [12] at 37 °C and kept under simulated intrapulpal pressure. Each tooth bonded specimens was longitudinally sectioned into multiple sticks of approximately $0.9 \pm 0.01 \, mm^2$ for the microtensile bond strength (μTBS) test. From each tooth, the central sticks of similar cross-sectional area and remaining dentin thickness were tested ($n = 24$/group). A digital caliper was used to check the cross-sectional area and length of the sticks. Each stick was fixed to the jig [13] with a cyanoacrylate adhesive (Rocket Heavy, City, CA, USA) and stressed in tension using a universal testing machine (Lloyd Instruments Ltd., Ametek Company, West Sussex, UK) at a cross-head speed of 0.5 mm/min until failure. The tensile force at failure was recorded and converted to tensile stress in MPa units using computer software (Nexygen-MT Lloyd Instruments). Sticks that failed before testing were counted as 0 MPa [14,15]. Cohesively failed specimens in the resin composite or the dentin were discarded and not included in the calculations [16].

Mode of failure analysis

Both fractured sections of each stick (dentin side and resin composite side) were mounted on an aluminium stub, gold sputter coated and observed with a scanning electron microscope (SEM) (Scanning electron microscope 515; Philips, Eindhoven, Netherlands) at different magnifications. Failure mode was allocated into Type 1; Adhesive failure at dentin side; Type 2: Cohesive failure in the adhesive layer; Type 3: Mixed failure (adhesive failure at dentin side and cohesive failure in the adhesive layer); Type 4: Mixed failure (cohesive failure in the adhesive layer and cohesive failure in resin composite); Type 5: Mixed failure (adhesive failure at dentin side, cohesive failure in the adhesive layer and cohesive failure in resin composite). The frequency of each mode of failure was expressed as percentage value for each group [17]. Representative photomicrographs were obtained.

Statistical analysis

The mean values of the recoded ten graphs for each temperature group (room temperature, 54 °C and 68 °C) were calculated. The mean value results for each group were presented in a common graph. A one-way analysis of variance (ANOVA) was used to test significant difference among the bond strength values of the different resin composite temperature groups. Bonferroni test for pairwise comparison was used if indicated. The significance level was set at $p \leqslant 0.05$. Data were analysed using the SPSS program for windows (Statistical package for Social Sciences, release 15 for MS Windows, 2006, SPSS Inc., Chicago, IL, USA). Regarding the mode of failure

analysis, the frequency of each mode of failure was calculated for each group.

Results

Intrapulpal temperature measurements

Time–temperature profiles of the Filtek LS resin composite are presented in Figs. 3–5. For all groups, intrapulpal baseline temperature was found to be 33 ± 0.5 °C. Upon application of Filtek LS silorane-based resin composite (point A), at room temperature, IPT remained unchanged. Whereas, in preheated groups, IPT increased by 1.5–2 °C and held at 34.8 ± 0.5 °C till light curing started (point B). During which, IPT increased gradually in all groups reaching a peak value of 38 ± 0.5 °C. After the curing time (40 s), at point C, IPT decreased gradually to its baseline temperature again (point D). The time interval between points C and D was ≈15 s for room temperature group and ≈40 s for preheated groups.

Microtensile bond strength measurements

Table 2 shows means, standard deviation of microtensile bond strength values as well as test of significance of the tested groups. No significant differences were observed among the dentin bond strength values when the resin composite was applied at different temperatures ($p = 1$).

Failure mode analysis

Type 3 [mixed failure (adhesive failure at the dentin side/cohesive failure in the adhesive layer)] followed by Type 5 [mixed failure (adhesive failure at the dentin side, cohesive failure in the adhesive layer and cohesive failure in resin composite)] was the mostly allocated modes of failure in the room temperature group and 54 °C preheated group. While for the 6 °C preheated group, Type 4 [mixed failure (cohesive failure in the adhesive layer/cohesive failure in resin composite)]
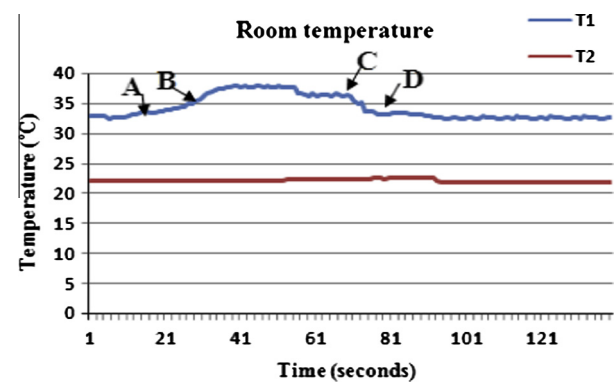
Fig. 3 Representative time–temperature profile of Filtek LS resin composite applied at room temperature; application of Filtek LS silorane-based resin composite (point A); start of light curing (point B); end of light curing (point C); regain to baseline temperature (point D); (T1) recorded intrapulpal temperature and (T2); room temperature.

Fig. 4 Representative time–temperature profile of Filtek LS resin composite preheated to 54 °C; application of Filtek LS silorane-based resin composite (point A); start of light curing (point B); end of light curing (point C); regain to baseline temperature (point D); (T1) recorded intrapulpal temperature and (T2); room temperature.
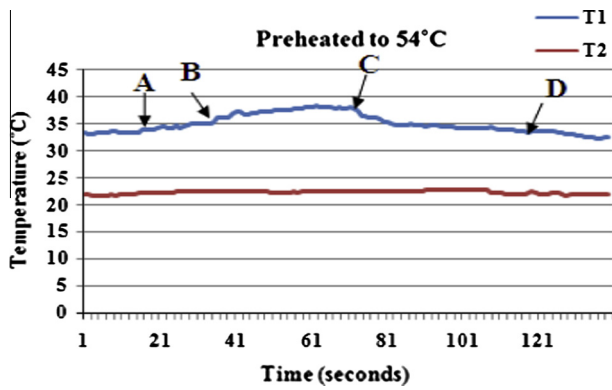

Fig. 5 Representative time–temperature profile of Filtek LS resin composite preheated to 68 °C; application of Filtek LS silorane-based resin composite (point A); start of light curing (point B); end of light curing (point C); regain to baseline temperature (point D) ; (T1) recorded intrapulpal temperature and (T2); room temperature.
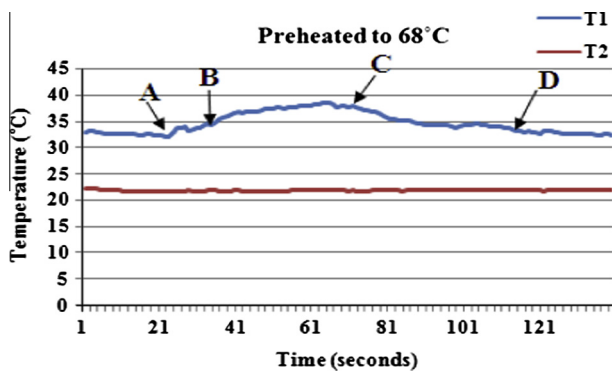
followed by Type 3 [mixed failure (adhesive failure at the dentin side/cohesive failure in the adhesive layer)] was the predominant modes of failure. Fig. 6 represents the percentage modes of failure in the tested groups, whereas the representative SEM photomicrographs of recorded modes of failure of the tested groups were shown in Fig. 7.

Discussion

The results of the present study reject of the first null hypothesis where there was a difference in the IPT among the tested

groups and accept the second null hypothesis as preheating had no effect on dentin microtensile bond strength.

Various factors, including light curing unit type, power density, exposure duration, the distance between tooth and/or composite surface and light guide tip end, composite shade, and thickness of both composite material and remaining dentin [18–21] might influence the extent of temperature rise during photopolymerisation. Filtek LS manufacturer has recommended that light curing procedure should exceed 20 s to activate the initiator. It also specified that curing time should be 40 s with either LED (intensity 500–1000 mW/cm^2) or Halogen (intensity 500–1400 mW/cm^2).

Regarding IPT results, the recorded values did not exceed the previously reported critical physiological limit [7], therefore, the data were described without statistical analysis. As in such cases, even if the statistical analysis showed significance it would not be of clinical impact. In the current study, the recorded baseline IPT (33 ± 0.5 °C) was consistent with other studies [22,23]. In the present study, the temperature rise values over that of the physiological baseline were noted when composite was applied on the dentin disc. Temperature changes were also recorded when composite application was completed, during light curing and after its completion [24]. It was found that both preheated resin composite groups recorded higher IPTs than group applied at room temperature. The elevation was 1.5–2 °C which is in line with Daronch et al. [24]. In their study, they referred it to the dentin behaviour as a thermal barrier against harmful stimuli providing protection to the pulp [24]. In that study the dentin thickness was 1 mm and not 0.5 mm as the present study in which the worst case scenario was represented. A previous study demonstrated that the tooth acts as a heat sink, which aids in rapidly decreasing the warmed composite temperature [25]. In addition, the fluid flow applied with the intrapulpal pressure simulation prior to and during restoration could have been taken away a part of the heat by convection and dissipation. Other researchers showed that the temperature indicated by the device was not the actual temperature acquired by the composite [6], denoting that the heat was not fully delivered. Also there was a rapid loss of composite temperature upon compule removal from the heating unit till its application on the tooth [6]. A pilot study was conducted to measure the temperature rise of the resin composite during its preheating cycles. We found that the desired preheating temperatures were not reached so that when the device denoted 54 °C and 68 °C, the resin composite temperatures inside the compule were 52 °C and 64 °C, respectively.

After application of light curing, IPT markedly increased by ≈5 °C above the baseline, in all groups regardless to the pre-delivery composite temperature. The same increase was reported when light curing unit with an intensity of 800 mW/cm^2 was used [26] although they were not concerned with the additional effect of resin composite preheating. In another study, the temperature of 1.25 mm-thick composite discs, that

Table 2 Dentin microtensile bond strength values (MPa) of the Filtek LS applied at the three tested temperatures.

	Control group 23 °C	Preheating to 54 °C	Preheating to 68 °C	p-value[*]
Mean (SD)	28.79 (7.2) [Ptf/tnt = 0/24]	27.66 (6.5) [Ptf/tnt = 0/24]	27.07 (6.3) [Ptf/tnt = 1/24]	0.92

[*] One way ANOVA; $p < 0.05$, [ptf/tnt = pre-test failure/total number of tested sticks].
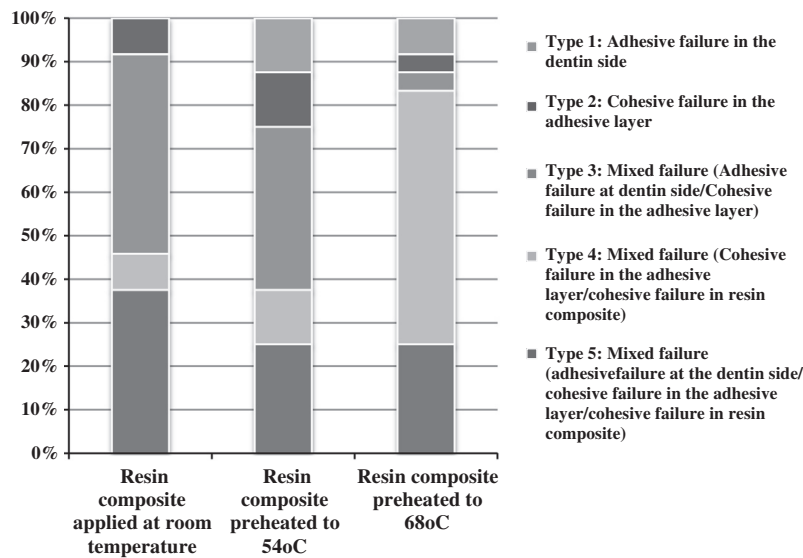
Fig. 6   Percentages of the recorded modes of failure of tested groups.
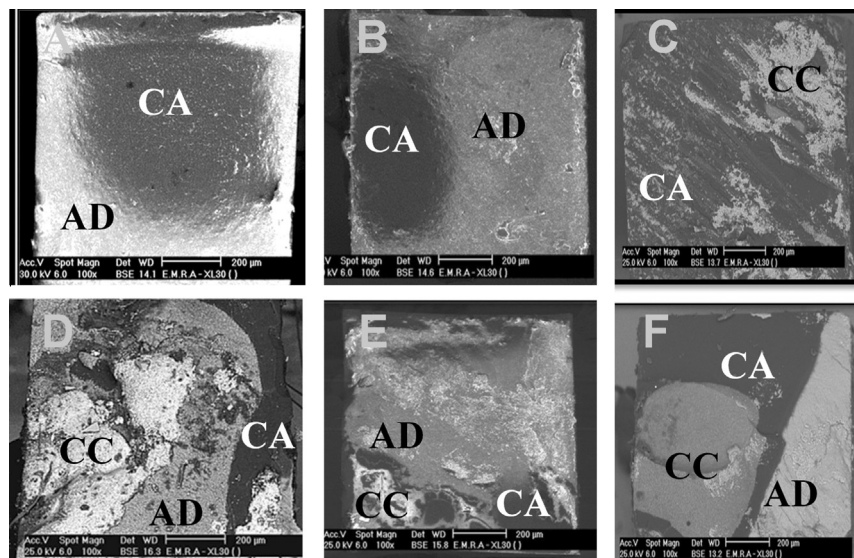

Fig. 7   Representative scanning electron micrographs (SEM) for the most frequently detected failure modes of fractured specimens of Filtek LS applied at room temperature group (A and D); Filtek LS preheated to 54 °C group (B and E) and Filtek LS preheated to 68 °C group (C and F). AD = Adhesive failure at dentin side; CA = Cohesive failure in the adhesive layer; CC = Cohesive failure in resin composite.

were preheated to 54.5 °C, was increased by 4.3–7.5 °C during photopolymerisation [27]. It should be taken into consideration that using light curing units of higher intensity than that used in the current study, could further increase the IPT, which poses a greater concern.

Zach and Cohen [7] set a threshold temperature for irreversible pulpal damage when external heat was applied to a sound tooth as 5.5 °C increase in the IPT induced necrosis in 15% of the tested pulps. Other researchers found that the pulp was less susceptible to thermal injury than previously thought where none of the tested teeth, up to 91 days, became symptomatic or revealed histological evidence of pulpitis [22]. However, the results of the current study confirm that

the biggest risk to pulp health occurs during photopolymerisation. A new finding in the present study was that the intrapulpal temperature took longer time (40 s) to return to its baseline temperature after light curing termination in the preheated groups in comparison with the room temperature group (20 s). This could raise a concern about heat retention in this type of resin composite, calling for further chemical and thermal analyses.

After 24 h storage under intrapulpal pressure simulation and artificial saliva immersion at 37 °C, microtensile bond strength results of the three tested groups showed no significant differences. In the present study efforts were done to simulate the clinical situation as much as possible where

the intrapulpal pressure was applied, the specimens were immersed in artificial saliva and the whole setup was inserted in a specially constructed incubator to establish the physiologic temperature. The minimum increase in the IPT upon application of the preheated resin composite may explain the obtained comparable microtensile bond strength findings. The mechanism of interaction of P90 System Adhesive with dentin, similar to other self-etch adhesives of the same category [28], was limited to a few hundreds of nanometres, in which it produced intense intertubular microporosity and preserved the smear plug [29–32].

Results of the current study did not reveal negative effects from preheating of silorane-based resin composite. Nevertheless, these results should not be generalised. Preheating should be used with knowledge of its limitations thus to prevent any adverse biological and mechanical drawbacks in the restorative system.

Conclusions

Preheating of silorane-based resin composite increased the IPT but not to the critical level and had no effect on dentin μTBS.

Conflict of interest

The authors have declared no conflict of interest.

Compliance with Ethics Requirements

This article does not contain any studies with human or animal subjects. However for the used teeth, the procedures of obtaining the teeth were following the local ethical committee of the Faculty of Oral and Dental Medicine, Cairo University, Egypt.

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