DENTAL MATERIALS 35 (2019) 1594–1602

Available online at www.sciencedirect.com

ScienceDirect

journal homepage: www.intl.elsevierhealth.com/journals/dema

Pre-heating effects on extrusion force, stickiness and packability of resin-based composite

Jiawei Yang[a], *Nikolaos Silikas*[a,*], *David C. Watts*[a,b,*]

[a] *Dentistry, School of Medical Sciences, University of Manchester, Manchester, UK*
[b] *Photon Science Institute, University of Manchester, Manchester, UK*

ARTICLE INFO

Keywords:
Resin composite
Extrusion force
Handling properties
Stickiness
Preheating
Degree of conversion

ABSTRACT

Objectives. To measure temperature effects on stickiness and packability of representative resin-based composites and the effect of pre-heating time on pre-cure properties of *Viscalor*, including extrusion force.

Methods. Five resin-based composites (RBC) and an additional RBC, *Viscalor,* used with a Caps Warmer (VOCO, Germany) were studied. The extrusion force (N) and extruded mass (g) were measured from *Viscalor* compules heated in T3 mode for 30 s (T3-30 s) and 3 min (T3-3 min). For stickiness and packability measurements, RBCs were packed into a brass cylindrical cavity controlled at 22 and 37 °C. A flat-ended probe was lowered into the RBC pastes at constant speed. Stickiness: F_{max} (N) and W_s (N mm), and packability: F_p (N), were measured. *Viscalor* was LED photo-cured at 1200 mW/cm^2 for 40 s. The degrees of conversion at 5 min and 24 h post cure (DC_{5min} and DC_{24h}) of *Viscalor* (no heat, T3-30 s and T3-3 min) were measured by ATR-FTIR. Data were analysed by one-way ANOVA, independent *t*-test and Tukey *post-hoc* tests ($p < 0.05$).

Results. The maximum temperature of the Caps Warmer, in T3 mode, reached 68 °C in 20 min. *Viscalor* temperatures of 34.5 °C and 60.6 °C were recorded after 30 s and 3 min pre-heating, respectively. Pre-heating significantly reduced extrusion force and increased extruded mass, especially after 3 min. RBCs varied in F_{max}, W_s and F_p ($p < 0.05$). Temperature also affected F_{max} ($p = 0.000$), W_s ($p = 0.002$) and F_p ($p = 0.000$). Pre-heating *Viscalor* for either 30 s or 3 min did not increase the post-cure DC at either 5 min or 24 h, relative to no pre-heating ($p > 0.05$).

Significance. The composites varied to an extent in stickiness and packability but the overall magnitudes remained within a clinically acceptable range. Pre-heating was beneficial in placement of *Viscalor* and caused no adverse effects through premature polymerization.

1. Introduction

Resin-based composites (RBCs) are designed and manipulated with suitable esthetic and physico-chemical properties to match the tooth structure. They can be fabricated in a range of consistencies and are therefore widely used as direct restorative materials in dentistry [1–3]. The maximum obtained properties and longevity of composites are dependent on the clinician's skill level and operating conditions [4,5]. Thus, 'technique sensitivity' should be reduced for good marginal integrity and successful restoration [6]. To avoid the formation

---

* *Corresponding authors at*: University of Manchester, School of Medical Sciences, Coupland 3 Building, Oxford Road, Manchester, M13 9PL, UK.

E-mail addresses: nick.silikas@manchester.ac.uk (N. Silikas), david.watts@manchester.ac.uk (D.C. Watts).

https://doi.org/10.1016/j.dental.2019.08.101

Table 1 – Manufacturer information of investigated composites.

	Code	Manufacturer	Resin system	Filler vol.%	Filler wt.%
Admira Fusion	AF	VOCO, Cuxhaven Germany	ORMOCER®	–	84
Filtek Supreme Ultra	FSU	3M ESPE, St. Paul, USA	bis-GMA, UDMA, TEGDMA, bis-EMA	63.3	78.5
TPH LV	TPH	Dentsply, Germany	Urethane modified bis-GMA, TEGDMA, polymerizable dimethacrylate	54.6	75.5
Tetric EvoCeram	TEC	Ivoclar Vivadent, USA	bis-GMA, urethane dimethacrylate, bis-EMA	54	75
Harmonize	HZ	Kerr, USA	bis-GMA, bis-EMA, TEGDMA	64.5	81
Viscalor	VC	VOCO, Cuxhaven Germany	bis-GMA, aliphatic dimethacrylate	–	83

of voids and gaps, both insertion technique and adaptation of composites need improvement [4]. The successful clinical handling and placement mainly depends on suitable pre-cure properties of composites that are determined by material composition and viscosity [7].

Pre-cure handling properties, such as flowability, stickiness, ease of placement and adaptation to cavity walls affect product selection for clinical restoration [8,9]. Stickiness - the adhesion force between two contacted surfaces - has been studied previously [8,10–12]. In a related field, the Avery Adhesive Test (AAT) with a spherical probe was used to study pressure-sensitive adhesives (PSA) [13]. In 2003, a method was reported using a flat-ended stainless-steel probe [8,10]. Clinically, the relationship between stickiness to tooth cavity and stickiness to instruments should be well balanced [8,10,14]. High stickiness to instruments may result in difficult placement and more porosities/gaps may occur during restoration [8,10]. RBCs with adequate consistency and packability are important for adapting to the tooth cavity and optimizing approximal contact areas [10,15]. Tyas et al. designed a method to assess consistency of unset composites [10,15]. They placed materials in an 8 mm × 8 mm cylindrical mould and pressed with a flat-ended glass rod demonstrating the high consistency of RBCs with increased filler content [9,15].

RBCs exhibit both viscous and elastic properties against the applied force and the rheological nature of pre-cure RBCs affects their handling properties [8,10]. Viscosity directly relates to material's handling properties, operating time and quality of restoration [10,14,16,17]. Viscosity decreases with temperature according to the Arrhenius Equation:

$$\eta = Ae^{E_a/RT}$$

where η, A, E_a, R and T represent viscosity, pre-exponential factor, activation energy for flow, universal gas constant and absolute temperature, respectively [16]. Viscosity also tends to increase exponentially with filler content [8,17].

Bis-GMA has higher viscosity than other dimethacrylates, resulting in low degree of conversion (DC) and requiring diluent monomers to facilitate filler particle incorporation [2,18–22]. High viscosity of highly-filled RBCs may cause insufficient adaptation to the cavity preparation, poor marginal integrity, and final restoration failure [23].

There are several possible strategies to achieve good cavity adaptation via reduced viscosity. Ideally, materials should flow into every corner of the cavity but not flow after removing the applied force [8,10]. High viscous RBC pastes are hard to extrude from the syringe or compule, which may lead to macroscopic voids/porosities during manipulation [8,16,23] and this was a major reason for developing flowable composites [24–26].

The SonicFill system (Kerr, USA), contains a highly-filled resin matrix including special viscosity modifiers that respond to ultrasonic energy (UE) and reduce the viscosity by 87%. Once UE is stopped, the viscosity returns to high levels, suitable for carving and contouring [27].

Several studies have evaluated pre-heating RBCs before placement [25,28,29]. Pre-heating makes highly-packed RBCs more fluid and easier to manipulate, without compromising their superior mechanical properties [30]. But after pre-heating the elevated temperature may cause thermal damage to the pulp [31]. The pulp has a normal temperature of 34–35 °C, and with temperature increases ranging from 5.5 to 16 °C, the possibility of pulp necrosis may increase from 15% to 100% [32].

Existing pre-heating devices, such as Calset heater (AdDent Inc., Danbury, CT, USA) and ENA heat (Micerium, Avegno, Italy), have operating temperature ranges of 37–68 °C [7,26,32–34].

A new pre-heating RBC, *Viscalor,* has been designed for use with a *Caps Warmer* device (VOCO, Germany). This has three working modes (T1, T2 and T3) to cover the temperature range 37–68 °C. The objectives of this study were to measure pre-cure properties including stickiness and packability of representative RBCs at different temperatures, and determine the effect of pre-heating time on pre-cure properties of *Viscalor*, including extrusion forces. The Null Hypotheses were: (1) composites did not vary in stickiness and packability at different temperatures, and (2) pre-heating period had no effect on *Viscalor*'s post-cure DC% measured at either 5 min or 24 h.

2. Materials and methods

Five commercial RBCs and an additional RBC: *Viscalor* used with a Caps Warmer (VOCO, Germany), were studied. The manufacturers' information is shown in Table 1.

A type-K thermocouple was inserted into the Caps Warmer (Fig. S1) to characterize its temperature profile in T3 mode. When it reached its maximum temperature, *Viscalor* compules were put into the Caps Warmer for 30 s and 3 min pre-heating times. Temperature was measured via a type-K thermocouple inside the compule. After pre-heating, the compule was removed from the Caps Warmer.

The extrusion force (N) of *Viscalor* from both full and half-full compules was measured using a modified com-

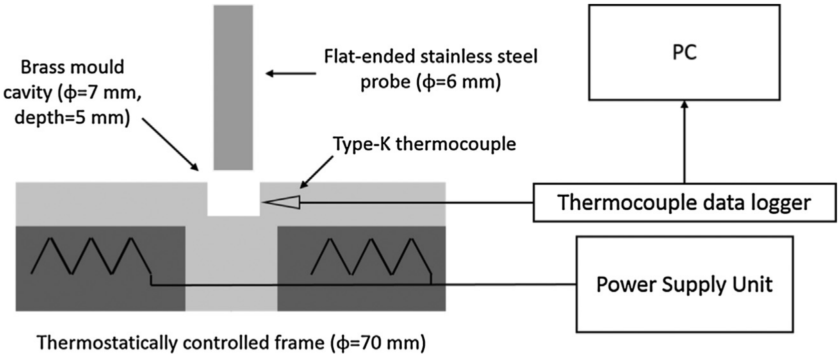

Fig. 1 – Mold setup with temperature regulation.

pule dispenser and a universal testing machine (Zwick/ Roell Z020, Leominster, UK) (Fig. S2). *Viscalor* was pre-heated before measurement using the Caps Warmer in T3 mode for 30 s (T3-30 s) and 3 min (T3-3 min). Compressive force was applied at 1 mm/s until either an upper force limit of 150 N or the maximum extrusion distance of 10 mm was reached (n = 3). The mass of extruded composite (g) was also measured.

A Texture Analyzer (Fig. S3) (TA.XT2i, Stable Micro Systems, Godalming, UK) was used to measure stickiness: via maximum separation force (F_{max}, N) and work of probe-separation (W_s, N mm), and packability: maximum packing force (F_p, N). Force was applied to a flat-ended cylindrical stainless-steel probe (φ = 6 mm). A thermostatically controlled mold at 22 °C and 37 °C with a cylindrical cavity (ϕ = 7 mm, depth = 5 mm) was fixed to a stand (Fig. 1). Composite paste was carefully packed into the cavity (n = 5).

For stickiness measurement, during the ‘bonding’ phase, the probe was lowered into the surface of unset composite with a pre-test speed of 0.50 mm/s. When a ‘trigger’ force of 0.05 N was registered, data acquisition commenced at rate of 400 p/s until a compressive force of 1 N was recorded, and held constant for 1 s. In the subsequent ‘debonding’ phase, the probe was raised vertically at 2 mm/s. Since the unset composite paste adhered to the probe, it elongated and exerted a tensile force as the probe ascended. With further elongation, tensile stress increased until it reached the interfacial strength and the composite paste separated from the probe.

Packability measurement used a similar experimental setup. Before measurement, the probe position was set 10 mm above the cavity. The probe was lowered into the surface of unset composite at 0.50 mm/s. When a ‘trigger’ force of 0.05 N was registered, data acquisition commenced until the probe penetrated 2 mm. Then the probe ascended vertically at 2 mm/s.

Fourier Transform Infrared (FTIR) Spectroscopy with an attenuated total reflectance (ATR) device (ALPHA II FTIR Spectrometer, Bruker Optik GmbH) was used to measure the DC% of *Viscalor* syringe/compule (no heat, T3-30 s and T3-3 min) at 5 min and 24 h post-cure. A background reading was collected between 400 to 4000 cm^{-1} using 32 scans at a resolution of 4 cm^{-1}. Composite paste was placed in an acetal mold (4 mm diameter x 2 mm thickness) directly on top of the ATR crystal. A Mylar strip and a glass slide were pressed onto the mold to remove air bubbles and excess paste. The spectrum of uncured *Viscalor* was collected. Photo-cure was achieved using an Elipar S10 LED unit (3 M ESPE, USA) of mean irradiance 1200 mW/cm^2 for 40 s at zero distance from the top surface. Then the spectrophotometer’s screw was applied to fix the cured specimen tightly on the reading crystal. The spectrum of the 5 min post-cured composite was collected. Then the spectra were acquired continually in real time for 24 h to obtain DC% at 24 h post-cure.

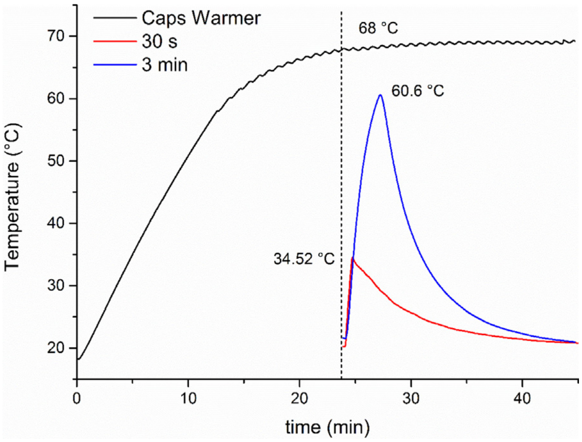

Fig. 2 – Representative temperature/time profiles of Caps Warmer (T3 mode) and *Viscalor* following pre-heating for different time periods.

The spectral region between 1600–1700 cm^{-1} was selected to identify the heights of the aliphatic C = C absorbance peak at 1637 cm^{-1} and the aromatic C = C absorbance peak at 1608 cm^{-1}. The DC% was calculated as:

$$DC\% = 1 - \frac{(H_{1637\,cm^{-1}}/H_{1608\,cm^{-1}})_{cured}}{(H_{1637\,cm^{-1}}/H_{1608\,cm^{-1}})_{uncured}} \times 100\%$$

where $H_{1637\,cm^{-1}}$ is the height of aliphatic C = C peak, $H_{1608\,cm^{-1}}$ is the height of aromatic C = C peak, respectively.

Data were entered into statistical software (SPSS, SPSS Inc., Illinois, USA) and analysed using one-way ANOVA test, independent *t*-test and Tukey post-hoc tests ($p < 0.05$). Homogeneity of variables was calculated using the Kruskal-Wallis Test ($p < 0.05$).

Table 2 – Extrusion force (N) and the mass of extruded composite (g) of new/half-used *Viscalor* compule (no heat, T3-30 s and T3-3 min).

Materials	Force (N)		Mass (g)	
	New	Half-used	New	Half-used
Viscalor (no heat)	153.62$^{a\ A}$ (1.56)	152.40$^{a\ A}$ (2.38)	0.0055$^{a\ B}$ (0.00)	0.0134$^{a\ C}$ (0.00)
Viscalor (T3-30 s)	145.45$^{a\ A}$ (8.15)	150.59$^{a\ A}$ (0.36)	0.1028$^{b\ B}$ (0.04)	0.2261$^{b\ C}$ (0.01)
Viscalor (T3-3 min)	66.49$^{b\ A}$ (14.16)	51.29$^{b\ A}$ (11.93)	0.1756$^{b\ B}$ (0.04)	0.2834$^{c\ C}$ (0.02)

For each material, same lower case superscript letters indicate homogeneous subsets among the materials. For each measurement (F, m), same capital superscript letters indicate homogeneous subsets among different conditions (new, half-used).

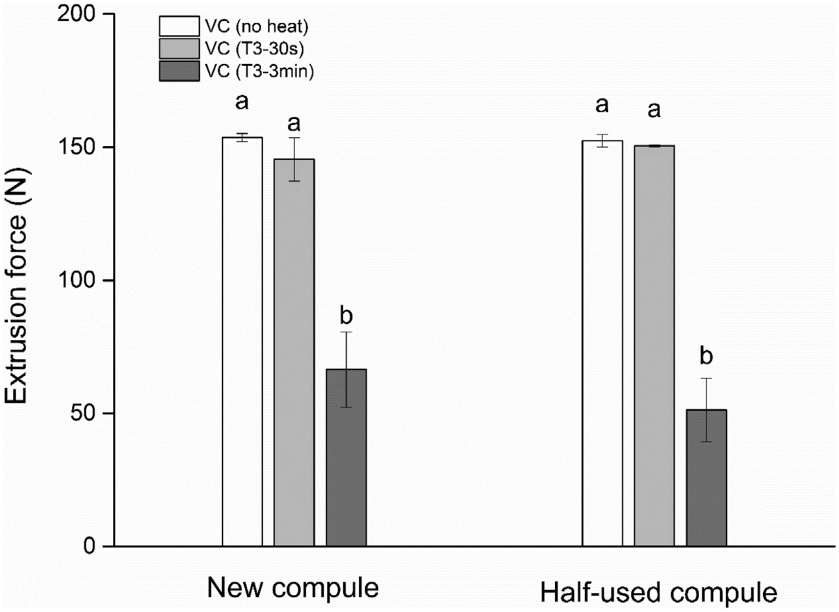

Fig. 3 – Extrusion force (N) of new/half-used *Viscalor* compule (no heat, T3-30 s and T3-3 min).

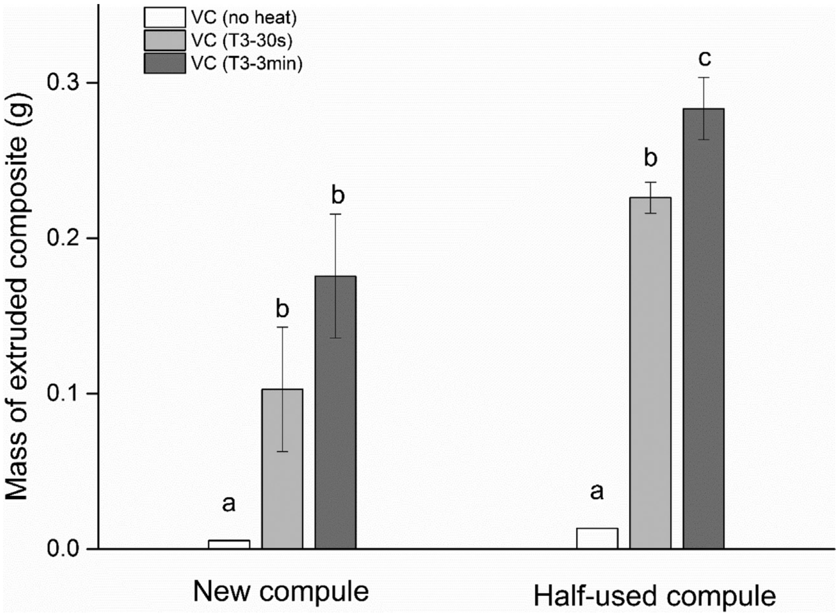

Fig. 4 – Mass of extruded composite (g) of new/half-used *Viscalor* compule (no heat, T3-30 s and T3-3 min).

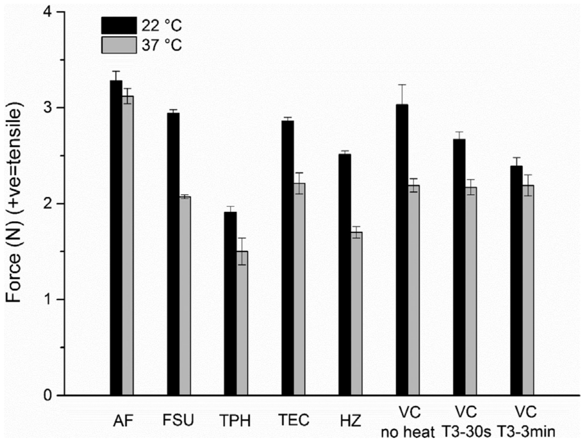

Fig. 5 – Maximum separation force (F_{max}) of investigated composites at 22 and 37 °C.

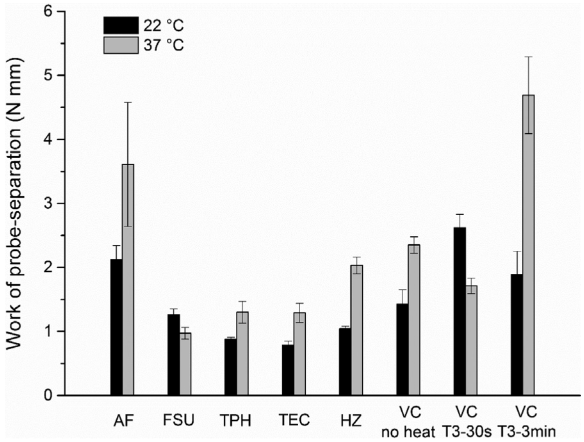

Fig. 6 – Work of probe-separation (W_s) of investigated composites at 22 and 37 °C.

3. Results

Fig. 2 shows representative temperature/time profiles of the Caps Warmer in T3 mode and the *Viscalor* temperatures following different pre-heating times. The Caps Warmer in T3 mode reached 68 °C after ca. 20 min. Composite temperature increases of 14.3 °C and 39.1 °C were recorded after 30 s and 3 min pre-heating, respectively. After removed from the Caps Warmer, composite temperature gradually returned to ambient temperature.

The extrusion force (N) and extruded mass (g) of full or half-full *Viscalor* compules (for no heat, T3-30 s and T3-3 min) are shown in Table 2 and Figs. 3 and 4. The extrusion force varied with pre-heating conditions, with 3 min heating giving the lowest extrusion force ($p = 0.000$). Partial usage of the compule had no significant influence on the measured extrusion force ($p = 0.866$). *Viscalor* compules with no heating yielded the lowest mass of extruded composite ($p = 0.000$). Half-used *Viscalor* compules (no heat, T3-30 s and T3-3 min) showed slightly higher extruded mass, in a fixed period, than full compules ($p < 0.05$).

Table. 3 and Figs. 5–7 show stickiness (F_{max} and W_s) and packability (F_p) data for different composites at 22 and 37 °C. F_{max}, W_s and F_p ranged from 1.50 to 3.28 N, from 0.79 to 4.69 N mm and from 10.79 to 41.56 N, respectively. Different RBCs varied in F_{max} ($p = 0.000$), W_s ($p = 0.000$) and F_p ($p = 0.032$). Temperature also had a significant effect on F_{max} ($p = 0.000$), W_s ($p = 0.002$) and F_p ($p = 0.000$), for which temperature rise reduced F_{max} and F_p, but increased W_s.

Fig. 8 represents real-time DC% vs. time during 24 h post-polymerization of *Viscalor* syringe/compule (no heat, T3-30 s and T3-3 min). Real-time DC% curves of *Viscalor* syringe and compule develop over 24 h with a similar trend. Table 4 and Fig. 9 report the DC% at 5 min and 24 h post-cure (DC_{5min} and DC_{24h}) of *Viscalor* syringe/compule (no heat, T3-30 s and T3-3 min). After 24 h, DC% increased to approximately 60%. There were no significant differences in DC% results between syringe and compule ($p > 0.05$). Pre-cure heating of *Viscalor* syringe/compule for either 30 s or 3 min in a 68 °C Caps

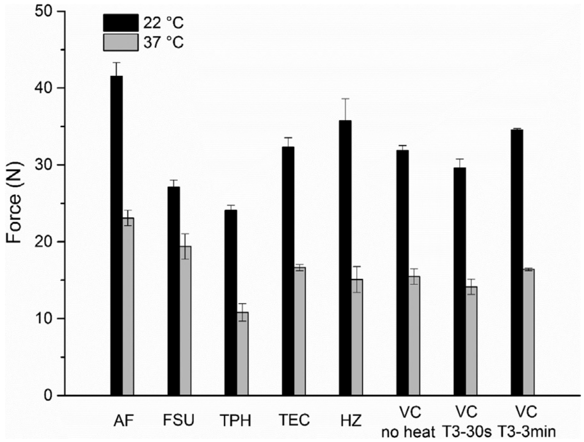

Fig. 7 – Maximum packing force (F_p) of investigated composites at 22 and 37 °C.

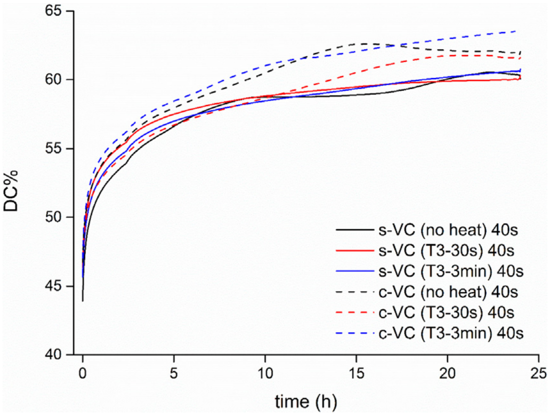

Fig. 8 – Real-time DC% vs. time during 24 h post-polymerization for *Viscalor* syringe/compule (no heat, T3-30 s, T3-3 min).

Table 3 – Stickiness parameters: F_{max} (N) and W_s (N mm), and packability, F_p (N) at 22 and 37 °C.

Materials	F_{max} (N)		W_s (N mm)		F_p (N)	
	22 °C	37 °C	22 °C	37 °C	22 °C	37 °C
Admira Fusion	$3.28^{f\ A}$ (0.10)	$3.12^{d\ A}$ (0.08)	$2.12^{d\ A}$ (0.22)	$3.61^{d\ A}$ (0.97)	$41.56^{f\ C}$ (1.77)	$23.09^{e\ B}$ (1.00)
Filtek Supreme Ultra	$2.94^{e\ B}$ (0.04)	$2.07^{c\ A,B}$ (0.02)	$1.26^{b,c\ A}$ (0.09)	$0.97^{a\ A}$ (0.09)	$27.11^{b\ D}$ (0.91)	$19.40^{d\ C}$ (1.66)
TPH LV	$1.91^{a\ A}$ (0.06)	$1.50^{a\ A}$ (0.14)	$0.88^{a,b\ A}$ (0.03)	$1.30^{a,b\ A}$ (0.17)	$24.10^{a\ C}$ (0.62)	$10.79^{a\ B}$ (1.14)
Tetric EvoCeram	$2.86^{d,e\ C}$ (0.04)	$2.21^{c\ B,C}$ (0.11)	$0.79^{a\ A}$ (0.06)	$1.29^{a,b\ A,B}$ (0.15)	$32.30^{c,d\ E}$ (1.24)	$16.64^{c\ D}$ (0.40)
Harmonize	$2.51^{b,c\ A}$ (0.04)	$1.70^{b\ A}$ (0.06)	$1.04^{a,b,c\ A}$ (0.04)	$2.03^{c\ A}$ (0.13)	$35.75^{e\ C}$ (2.86)	$15.08^{b,c\ B}$ (1.68)
Viscalor (no heat)	$3.03^{e\ B}$ (0.21)	$2.19^{c\ A,B}$ (0.07)	$1.42^{c\ A}$ (0.23)	$2.35^{c\ A,B}$ (0.13)	$31.88^{c,d\ D}$ (0.66)	$15.46^{b,c\ C}$ (1.01)
Viscalor (T3-30 s)	$2.67^{c,d\ A}$ (0.08)	$2.17^{c\ A}$ (0.08)	$2.62^{e\ A}$ (0.21)	$1.71^{b,c\ A}$ (0.12)	$29.58^{b,c\ C}$ (1.18)	$14.13^{b\ B}$ (0.99)
Viscalor (T3-3 min)	$2.39^{b\ A}$ (0.09)	$2.19^{c\ A}$ (0.11)	$1.89^{d\ A}$ (0.36)	$4.69^{e\ B}$ (0.60)	$34.55^{d,e\ D}$ (0.17)	$16.39^{b,c\ C}$ (0.17)

For each temperature, same lower case superscript letters indicate homogeneous subsets among the materials. For each material, same capital superscript letters indicate homogeneous subsets among different conditions.

Table 4 – Degree of conversion of *Viscalor* (no heat, T3-30 s and T3-3 min) at 5 min and 24 h post cure (DC_{5min} and DC_{24h}).

Materials	Syringe		Compule	
	DC_{5min}	DC_{24h}	DC_{5min}	DC_{24h}
Viscalor (no heat)	40.78 $\%^{a\ A}$ (0.01)	58.04 $\%^{a\ B}$ (0.03)	41.99 $\%^{a\ A}$ (0.01)	60.17 $\%^{a\ B}$ (0.03)
Viscalor (T3-30 s)	42.77 $\%^{a\ A}$ (0.01)	58.49 $\%^{a\ B}$ (0.01)	42.76 $\%^{a\ A}$ (0.01)	58.88 $\%^{a\ B}$ (0.01)
Viscalor (T3-3 min)	40.71 $\%^{a\ A}$ (0.01)	58.30 $\%^{a\ B}$ (0.01)	41.65 $\%^{a\ A}$ (0.00)	60.60 $\%^{a\ C}$ (0.01)

For each DC, same lower case superscript letters indicate homogeneous subsets among the materials. For each material, same capital superscript letters indicate homogeneous subsets among different conditions.

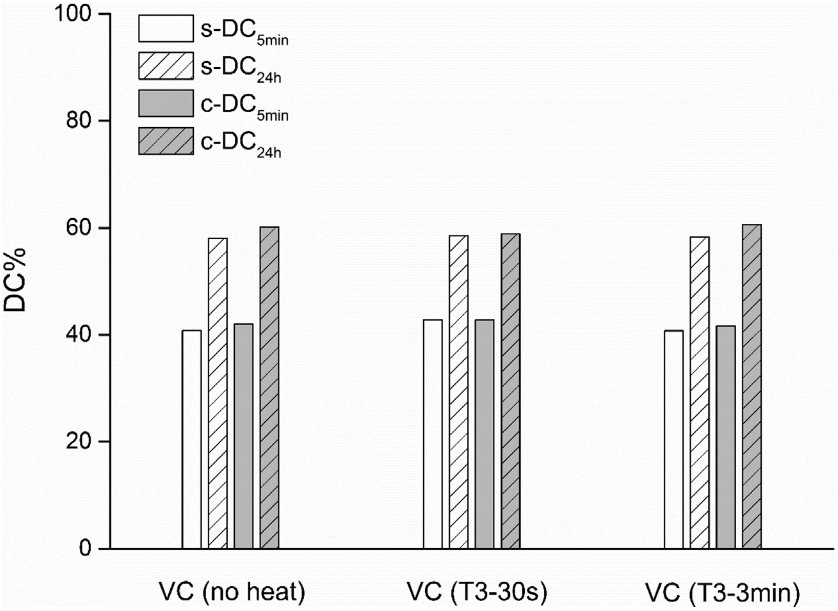

Fig. 9 – DC% results of *Viscalor* syringe/compule (no heat, T3-30 s and T3-3 min) at 5 min and 24 h post cure (DC_{5min} and DC_{24h}).

Warmer did not increase the post-cure DC% at either 5 min or 24 h, compared to data for no pre-heating ($p > 0.05$).

4. Discussion

Dental RBCs are designed to exhibit good mechanical properties and esthetics after restoration, but their pre-cure properties, including stickiness and packability, mainly affect the clinical handling and placement [35]. These handling properties depend upon the inherent material characteristics and rheological nature of composites [14]. Hence, this study investigated extrusion force, stickiness and packability at different temperatures and evaluated post-cure DC% at 5 min and 24 h for *Viscalor* after different pre-heating times. Thus, the first null hypothesis was rejected and the second null hypothesis was accepted. Regardless of pre-heating time, no significant change in DC% of *Viscalor* was measured ($p > 0.05$).

The thermal properties and heating rates of both Caps Warmer and pre-heated *Viscalor* were previously unreported.

Thus, temperature profiles of Caps Warmer in the T3 mode and *Viscalor* following different pre-heating periods were first characterized. Results demonstrated the efficacy of the Caps Warmer since it reached the stated preset temperature of 68 °C after about 20 min. When heating was stopped, a slight temperature rise of 2.09 °C and 0.35 °C was found, respectively. During pre-heating, thermal energy diffused gradually through the container (compule or syringe) into the composite.

With temperature rise the viscosity of *Viscalor* reduced, but its flowability was still somewhat less than certain flowable RBCs at room temperature [35]. After 3 min pre-heating, *Viscalor* had a lower internal temperature than the maximum temperature of the Caps Warmer in T3 mode (68 °C). This corresponds to previous studies where pre-heated composites were cooler than the pre-set temperature of heating devices. Thus reduced pulp temperature changes may ensue [32]. Reduced composite temperature rises also relate to filler properties since inert inorganic particles only absorb small amounts of thermal energy during heating [35,36]. The high filler content of *Viscalor* implies a low proportion of resin matrix and consequently a low temperature rise [36]. Different filler contents result in different temperature/time profiles. The temperature of *Viscalor* (T3-3 min) decreased to 37 °C within 3 min after removed from the Caps Warmer. To ensure minimal temperature drop and optimal performance, clinicians should work rapidly during manipulation of pre-heated composites.

To quantify the effect of pre-heating on *Viscalor*'s flowability, the extrusion force (N) and extruded mass (g) were measured for both full and half-used *Viscalor* compules. Results showed the beneficial effects of a longer pre-heating period, in which extrusion force reduced and extruded mass increased. This confirmed that 3 min pre-heating did increase the flowability of *Viscalor* leading to easy extrusion and a sufficient mass of extruded composite.

Stickiness measurements were based on previous studies on the effects of temperature and composite composition [8–10,14]. Generally, there are two types of force/displacement plots (Type I and Type II), in which Type I is more commonly observed (Fig. S4). A Type I plot has a single peak, whereas a Type II plot has a primary peak followed by a secondary peak [14]. The peak height (F_{max}) is the maximum tensile force during 'debonding'. The work of probe-separation (W_s) is the integrated area under the curve [10].

The force/displacement profiles observed were combined responses of RBC paste viscoelasticity and interfacial behaviour between the probe and paste [8,13]. F_{max} mainly depends on the wettability of the paste, its resistance against the debonding force, and the roughness of both probe and paste [8,13,14]. Other factors, such as temperature and viscoelastic properties of the paste also affect F_{max} [14]. W_s depended on the shear characteristics of the pastes, which relates to their molecular entanglements [8,13,14]. In the present stickiness results, Admira Fusion exhibited the highest F_{max} and W_s at both 22 and 37 °C. According to previous studies, high filler loading tends to produce low stickiness [8,17] as it hinders composite flowability and creates high viscosity [37]. Admira Fusion, *Viscalor* and Harmonize have high filler content (84 wt.%, 83 wt.% and 81 wt.%, respectively) and their F_{max} varied with filler content. However, they did not exhibit particularly low stickiness, as expected. This may be due to their matrix compositions. Admira Fusion is a ceramic-based RBC, in which ORMOCERs function as the matrix system [38–40]. Nanoparticles and glass ceramic particles are firmly embedded in the ORMOCER matrix [41]. The ART (Adaptive Response Technology) filler system in Harmonize acts as a rheological modifier.

Although containing relatively high filler loading, Filtek Supreme Ultra (78.5 wt.%) showed higher F_{max} and W_s compared to TPH LV (75.5 wt.%) and Tetric EvoCeram (75 wt.%). This may be due to both TEGDMA and bisphenol-A epoxylated dimethacrylate (bis-EMA) within its matrix system [16,17]. Previous studies have noted that the presence/absence of hydrogen bonding significantly affects viscosity. Bis-EMA, lacks two hydroxyl groups (−OH) in its chemical structure, compared to bis-GMA, which reduces viscosity [42]. But, with a low-viscous matrix system, TPH LV and Tetric EvoCeram still showed low F_{max} and W_s possibly related to their filler characteristics. Many previous studies established that all compositional variables affect RBC rheological and handling properties: resin matrix, filler particle content, shape, size and distribution, silane surface treatment, interlocking between particles and other interfacial interactions between resin matrix and filler [30,43]. Generally, increasing filler loading and using smaller, irregular-shaped particles increases viscosity [30,44]. Filler particle sizes of TPH LV (1.35 μm) [45] and Tetric EvoCeram (40 nm–3 μm) [46] are lower than those in Filtek Supreme Ultra (0.6–10 μm) [47]. For a similar filler loading, more particles means higher surface area, more matrix/particle interactions and thus higher viscosity [17]. TPH LV and Tetric EvoCeram had low stickiness. Different filler morphologies - following the sequence: round, grains, plates and rods - reduce viscosity of RBCs [17]. Silane surface treatment may slightly lubricate irregular particles and reduce viscosity [17]. However, in this study, the lack of filler morphology information limits the discussion.

For packability measurements, compressive force (N) was plotted against probe displacement (mm) (Fig. S5). F_p reduced with decreased filler loading. Admira Fusion and TPH LV had the highest and lowest F_p values at both 22 and 37 °C, respectively.

In addition to paste composition, temperature also affected stickiness and packability: reducing F_{max} and F_p, but increasing W_s. Temperature increases the mobility of matrix monomers. Low viscous RBCs are more fluid so temperature further reduces F_p. Composite pastes bond more easily to the probe, increasing F_{max} and W_s [8,29]. However, some studies found that F_{max} and W_s may be lower at high temperature [9,11,14,17]. Since segmental movement is greater at high temperature, matrix monomers are insufficiently resistant to slippage of internal components. This factor tends to reduce F_{max} and W_s [9,11,14,17].

Viscalor (no heat) showed generally comparable F_{max}, W_s and F_p to the other investigated RBC pastes. Different pre-heating times had significant influence on F_{max}, W_s and F_p at either 22 or 37 °C ($p < 0.005$), except for F_{max} at 37 °C ($p = 0.884$). Composite temperature can reduce rapidly to the ambient physiological level after removed from a pre-heating device [7,29,32]. Thus, pre-heated *Viscalor* (T3-30 s and T3-

3 min) inserted into the brass cavity showed similar F_{max} results to *Viscalor* (no heat) at 37 °C due to the similar composite temperature. However, *Viscalor*'s W_s changed significantly with different pre-heating times. So evidently W_s was more sensitive than F_{max} to changes in elongation and arguably more appropriate to describe stickiness [14].

Moderate temperature rise after pre-heating generates greater mobility of monomer free radicals - *as and when they are generated by photo-initiation*. The temperature rise delays auto-deceleration during polymerization and leads to the increased DC% [7,26,29,32,48,49]. Higher monomer conversion has been observed after pre-heating composites at 54 °C, however, high polymerization shrinkage also occur with high DC% [7,26,50]. But, after 30 s pre-heating, F_p decreased slightly at either 22 or 37 °C.

To further identify the effect of pre-heating time on pre-cure stickiness and packability of *Viscalor*, DC% was measured. After 24 h at 37 °C, DC% increased [48,49]. The use of *Viscalor* syringe or compule had no significant influence on DC_{5min} and DC_{24h}. Real-time DC% curves of both *Viscalor* syringe and compule specimens increased similarly. Different pre-heating time had no significant effect on *Viscalor* syringe/compule DC% either measured after 5 min or 24 h. Generally, temperature rise has a positive effect on DC%, since temperature rise aids polymer chain propagation.

Three minutes pre-heating did not affect the DC% of *Viscalor* syringe/compule specimens. This suggests that no premature monomer curing occurred.

5. Conclusions

Within the limitations of this study, the following conclusions are drawn:

1) The Caps Warmer exhibited good efficacy as a pre-heating device: pre-heated *Viscalor* showed greatly reduced extrusion force and increased flowability, especially after the longer pre-heating time (3 min).
2) The RBC pastes varied to a statistically significant but limited extent in stickiness and packability. But, their overall magnitudes remained within what may be considered a clinically acceptable range.
3) Pre-heating had no adverse effects on *Viscalor* through any thermal activation causing premature polymerization.

Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.dental.2019.08.101.

REFERENCES

[1] Sakaguchi RL, Powers JM. Craig's restorative dental Materials-E-Book. Elsevier Health Sciences; 2012.
[2] Anusavice KJ, Shen C, Rawls HR. Phillips' science of dental materials. Elsevier Health Sciences; 2013.
[3] Antoniac IV. Handbook of bioceramics and biocomposites. Berlin Heidelberg, New York: Springer; 2016.
[4] Nedeljkovic I, Teughels W, De Munck J, Van Meerbeek B, et al. Is secondary caries with composites a material-based problem? Dent Mater 2015;31(11):e247–77.
[5] Demarco FF, Corrêa MB, Cenci MS, Moraes RR, Opdam NJM. Longevity of posterior composite restorations: not only a matter of materials. Dent Mater 2012;28(1):87–101.
[6] Ferracane J, Hilton T, Stansbury J, Watts D, Silikas N, Ilie N, et al. Academy of Dental Materials guidance—resin composites: part II—technique sensitivity (handling, polymerization, dimensional changes). Dent Mater 2017;33(11):1171–91.
[7] Lohbauer U, Zinelis S, Rahiotis C, Petschelt A, Eliades G. The effect of resin composite pre-heating on monomer conversion and polymerization shrinkage. Dent Mater 2009;25(4):514–9.
[8] Kaleem M, Satterthwaite JD, Watts DC. A method for assessing force/work parameters for stickiness of unset resin-composites. Dent Mater 2011;27(8):805–10.
[9] Kaleem M, Watts DC. Stiffness of uncured resin-composites assessed via cavity-packing forces. Dent Mater 2016;32(9):e199–203.
[10] Al-Sharaa KA, Watts DC. Stickiness prior to setting of some light cured resin-composites. Dent Mater 2003;19(3):182–7.
[11] Ertl K, Graf A, Watts D, Schedle A. Stickiness of dental resin composite materials to steel, dentin and bonded dentin. Dent Mater 2010;26(1):59–66.
[12] Hoseney RC, Smewing JO. Instrumental measurement of stickiness of doughs and other foods. J Texture Stud 2007;30(2):123–36.
[13] Chuang H, Chiu C, Paniagua R. Avery adhesive test yields more performance data than traditional probe. Adhes Age 1997;40(10):18–23.
[14] Kaleem M, Satterthwaite JD, Watts DC. Effect of filler particle size and morphology on force/work parameters for stickiness of unset resin-composites. Dent Mater 2009;25(12):1585–92.
[15] Tyas MJ, Jones DW, Rizkalla AS. The evaluation of resin composite consistency. Dent Mater 1998;14(6): 424–8.
[16] Silikas N, Watts D. Rheology of urethane dimethacrylate and diluent formulations. Dent Mater 1999;15(4):257–61.
[17] Lee J-H, Um C-M, Lee I-b. Rheological properties of resin composites according to variations in monomer and filler composition. Dent Mater 2006;22(6):515–26.
[18] Papakonstantinou AE, Eliades T, Cellesi F, Watts DC, Silikas N. Evaluation of UDMA's potential as a substitute for Bis-GMA in orthodontic adhesives. Dent Mater 2013;29(8):898–905.
[19] Gajewski VE, Pfeifer CS, Fróes-Salgado NR, Boaro LC, Braga RR. Monomers used in resin composites: degree of conversion, mechanical properties and water sorption/solubility. Braz Dent J 2012;23(5):508–14.
[20] Miletic V. Development of dental composites. In: Dental composite materials for direct restorations. Springer; 2018. p. 3–9.
[21] Peutzfeldt A. Resin composites in dentistry: the monomer systems. Eur J Oral Sci 1997;105(2):97–116.
[22] Gonçalves F, Azevedo CL, Ferracane JL, Braga RR. BisGMA/TEGDMA ratio and filler content effects on shrinkage stress. Dent Mater 2011;27(6):520–6.
[23] Lucey S, Lynch CD, Ray NJ, Burke FM, Hannigan A. Effect of pre-heating on the viscosity and microhardness of a resin composite. J Oral Rehabil 2010;37(4):278–82.
[24] Lee I-B, Son H-H, Um C-M. Rheologic properties of flowable, conventional hybrid, and condensable composite resins. Dent Mater 2003;19(4):298–307.

[25] Blalock JS, Holmes RG, Rueggeberg FA. Effect of temperature on unpolymerized composite resin film thickness. J Prosthet Dent 2006;96(6):424–32.

[26] Deb S, Di Silvio L, Mackler HE, Millar BJ. Pre-warming of dental composites. Dent Mater 2011;27(4):e51–9.

[27] SonicFill Portfolio of Scientific Research. Available from: ftp://ftp.endoco.com/links/KerrSonicFillResearch.pdf.

[28] Wagner W, Aksu M, Neme A, Linger J, Pink F, Walker S. Effect of pre-heating resin composite on restoration microleakage. Oper Dent 2008;33(1):72–8.

[29] Lempel E, Őri Z, Szalma J, Lovász BV, Kiss A, Tóth Á, et al. Effect of exposure time and pre-heating on the conversion degree of conventional, bulk-fill, fiber reinforced and polyacid-modified resin composites. Dent Mater 2019;35(2):217–28.

[30] Metalwala Z, Khoshroo K, Rasoulianboroujeni M, Tahriri M, Johnson A, Baeten J, et al. Rheological properties of contemporary nanohybrid dental resin composites: The influence of preheating. Polym Test 2018;72:157–63.

[31] Da Costa JB, Hilton TJ, Swift JEJ. Preheating composites. J Esthet Restor Dent 2011;23(4):269–75.

[32] Daronch M, Rueggeberg FA, Hall G, De Goes MF. Effect of composite temperature on in vitro intrapulpal temperature rise. Dent Mater 2007;23(10):1283–8.

[33] Tauböck TT, Tarle Z, Marovic D, Attin T. Pre-heating of high-viscosity bulk-fill resin composites: effects on shrinkage force and monomer conversion. J Dent 2015;43(11):1358–64.

[34] Nada K, El-Mowafy O. Effect of precuring warming on mechanical properties of restorative composites. Int J Dent 2011;2011:5.

[35] Daronch M, Rueggeberg FA, Moss L, De Goes MF. Clinically relevant issues related to preheating composites. J Esthet Restor Dent 2006;18(6):340–50.

[36] Al-Qudah AA, Mitchell CA, Biagioni PA, Hussey DL. Thermographic investigation of contemporary resin-containing dental materials. J Dent 2005;33(7):593–602.

[37] Bayne SC, Thompson JY, Swift EJJ, Stamatiades P, Wilkerson M. A characterization of first-generation flowable composites. J Am Dent Assoc 1998;129(5):567–77.

[38] Judeinstein P, Sanchez C. Hybrid organic–inorganic materials: a land of multidisciplinarity. J Mater Chem 1996;6(4):511–25.

[39] Moszner N, Salz U. New developments of polymeric dental composites. Prog Polym Sci 2001;26(4):535–76.

[40] Cavalcante LM, Schneider LFJ, Silikas N, Watts DC. Surface integrity of solvent-challenged ormocer-matrix composite. Dent Mater 2011;27(2):173–9.

[41] Scientific Compendium: Admira Fusion. Available from: http://212.227.148.121/e-paper/scientific_compendium/admira-fusion_en/.

[42] Kalachandra S, Sankarapandian M, Shobha HK, Taylor DF, McGrath JE. Influence of hydrogen bonding on properties of BIS-GMA analogues. J Mater Sci Mater Med 1997;8(5):283–6.

[43] Al-Ahdal K, Silikas N, Watts DC. Rheological properties of resin composites according to variations in composition and temperature. Dent Mater 2014;30(5):517–24.

[44] Habib E, Wang R, Zhu XX. Correlation of resin viscosity and monomer conversion to filler particle size in dental composites. Dent Mater 2018;34(10):1501–8.

[45] TPH Spectra Universal Composite Restorative Science EN. Available from: https://www.dentsplysirona.com/content/dam/dentsply/pim/manufacturer/Restorative/Direct_Restoration/Composites__Flowables/Universal_Composites/TPH_Spectra_Universal_Composite_Restorative/TPH-Spectra-xnzlfpn-en-us-1402.

[46] Tetric EvoCeram Instructions for Use. Available from: http://downloads.ivoclarvivadent.com/zoolu-website/media/document/827/Tetric+EvoCeram.

[47] Filtek Supreme Ultra Technical Product Profile. Available from: http://multimedia.3m.com/mws/media/629066O/filtektm-supreme-ultra-universal-restorative.pdf.

[48] Al-Ahdal K, Ilie N, Silikas N, Watts DC. Polymerization kinetics and impact of post polymerization on the degree of conversion of bulk-fill resin-composite at clinically relevant depth. Dent Mater 2015;31(10):1207–13.

[49] Daronch M, Rueggeberg FA, De Goes MF, Giudici R. Polymerization kinetics of pre-heated composite. J Dent Res 2006;85(1):38–43.

[50] Trujillo M, Newman SM, Stansbury JW. Use of near-IR to monitor the influence of external heating on dental composite photopolymerization. Dent Mater 2004;20(8):766–77.