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Rheological properties of resin composites according to variations in composition and temperature

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ABSTRACT

Objective. To study the effect of compositional variables and temperature (25 °C and 37 °C) on the rheological properties of contemporary resin composites.

Methods. Fourteen commercial resin composites with different resin matrices of Bis-GMA and TEGDMA, different filler loading (41–65, v/v) and particle sizes (ranging 5 nm to 20 μm) were studied using a parallel plate rheometer. Shear sweep measurements were made to determine each composite's viscosity over the angular frequency range of $\omega = 10^{-4}$ – 10^2 rad/s. Data were analyzed using one way ANOVA, Bonferroni post hoc and t-independent tests ($p < 0.05$).

Results. All composites exhibited viscosity reducing with shear rate (pseudoplasticity). Viscosity averages were calculated over the shear rate range. These viscosities increased as the percentage of filler loading (by volume) increased ranging between 0.05–349.33 kPa s at 25 °C and 0.03–132.00 kPa s at 37 °C. Filtek Supreme XTE exhibited the highest viscosity at both temperatures ($p < 0.05$). Filtek Bulk Fill on the other hand, showed the lowest viscosity at both temperatures ($p < 0.05$). Viscosity significantly decreased with the increase in the temperatures for all materials ($p < 0.05$). The reduction ranged from 40.8% (for Venus Diamond Flow) to 92.2% (for Spectrum TPH3).

Significances. There was a considerable variation in the resin composite viscosities measured at 25 °C as an average value for shear-rate sweep. The factor between the least and highest viscosities was about 7000.

The formulation differences that led to such a great variation were primarily: (i) volume fraction of fillers and (ii) particle size, the latter influencing total particle surface area.

Rheological properties such as viscosity are key parameters influencing perceived differences in handling behavior of resin composites.

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1. Introduction

Resin composites are used every day in almost every dental clinic to create permanent tooth-colored restorations. Favorable handling properties of such materials should include ease of placement, shaping within the restored site, adherence to tooth surface but not to an instrument, and maintaining shape or being resistance to slumping. Prior to light curing these characteristics are indispensable in restorative procedures. One of the main characteristics is the consistency, as it affects the application and manipulation of the material [1]. Composites are available with a wide range of consistencies and/or viscosities. The more fluid ones are termed flowable composites. Generally, composites are categorized as viscoelastic materials positioned intermediately between elastic materials, such as ceramics, and viscous materials, such as oils [2–5]. The effect of any external forces on any direct dental restorative material is governed by the absolute values of their elasticity and viscosity, and their viscosity to elasticity ratios [6]. Rheological properties of unset materials govern the way materials perform which in turn affects the handling properties of the material. A material that adheres to tooth structure, performs well within the hands of dental practitioners, and is able to reconstruct the lost original anatomy of the tooth plays a vital role in the final success of the restoration, by which the function and esthetics of the tooth is restored.

Resin composites are highly filled with inorganic particles in an organic matrix. They have both liquid-like and solid-like behaviors. Such mixtures usually show complex rheological properties such as shear-thinning (i.e. the viscosity decreases as the shear rate increases), thixotropy (i.e. the viscosity depends on the time) or yield stress (i.e. the material does not flow if the applied stress is insufficient) [7].

The viscosity of resin composites has an impact on the handling properties of the material. It affects the ease of manipulation, the ability of the composite and associated adhesives to adhere to the tooth structure [2,8], the operation time and the quality of the restoration [1,2,8–11]. Flowable composite are usually formed by retaining the same small particle sizes of the conventional hybrid composites, but at reduced filler contents, which allow reduced viscosity of the mixture [9]. They can be applied into a cavity using a loading tip and syringe and can be used in situations of difficult access or that require good penetration. Nevertheless, packable or condensable posterior resin composites are now available. Their viscosity is increased by increasing the filler content or by modifying the filler particle morphology and even by incorporating short glass fibers [12]. Such packable resin composites can be placed into a cavity and packed using amalgam instruments, and good interproximal contacts in a class II cavity can be achieved.

Classification of composites has often been based on filler particle size: macrofill, minifill, microfill, and hybrid composites [13]. Nowadays, they can also be classified as flowable, medium viscosity and packable [9,11] based on viscosity or fluidity [11,14,15].

Several methodologies can be applied to measure the visco-elasticity of dental materials, such as: creep, stress relaxation, steady shear and dynamic measurements. In

particular, dynamic oscillatory shear tests are commonly used to study the linear viscoelastic properties of a polymer [4,11]. Ferracane et al. [8] used a rotational cone and plate viscometer to measure the viscosity of chemically cured resin composites. Opdam et al. [1] used a press method to compare the consistencies of different resin composites. Lee et al. [11] used a dynamic oscillatory rheometer and a vertical oscillation rheometer [16] to evaluate the viscoelastic properties of resin composites. Mesquita et al. studied the viscoelastic properties of resin composites after polymerization by dynamic mechanical analysis (DMA) [17].

The aim of the present study was to evaluate the influence of resin mixture, filler content and temperature on the viscosity of recent resin composites, using a dynamic oscillatory shear test. The null hypotheses were that there are no significant effects of resin mixture, filler content and temperature on the viscosity of resin composites.

2. Materials and methods

Fourteen resin composites were studied. Their filler amount incorporated ranged between 41% and 65% by vol. The brand names and manufacturers are listed in (Table 1) together with further information about the filler sizes and matrix monomers.

A rheometer (Bohlin Instruments Ltd., UK, Fig. 1) was used to measure the dynamic viscoelastic behavior of the resin composite. A shear sweep was used to measure the viscosities. The geometry used in this test was a parallel plate design. Composite paste was placed on the lower plate of the rheometer, after which the upper aluminum plate with a diameter of 20 mm was moved downward to adjust the gap to 250 μm . This was the smallest gap that could be used with all of the materials. Any excess composite and air bubbles present around the circumference of the plate were removed, prior to initiating active measurements.

After the residual normal stress was relieved, a frequency sweep was completed over the angular frequency range of $\omega = 10^{-4}$ – 10^2 rad/s [18]. The resulted shear-rate range experienced by each material was dependent upon its viscosity. The specimens were placed under increasing torque frequencies (synonymous with increasing shear rates) while the resulting shear stress developed in the resin was measured. The shear stress to shear strain values obtained enabled the calculation of the materials instantaneous viscosity (Eq. (1)).

$$\dot{\eta} = \frac{\tau}{\gamma} \quad (1)$$

where $\dot{\eta}$ is the viscosity, τ is the shear stress, and γ is the shear strain rate (i.e. velocity gradient).

Since the viscosity varied with shear-rate, viscosities over the measured shear-rate range were averaged and tabulated for each material and temperature. Three measurement runs were made for each material both at 25 °C and at 37 °C.

Data were analyzed using statistical software (SPSS ver.20, IBM Corp, Armonk, NY, USA) at ($p < 0.05$) significance level. One-way ANOVA and Bonferroni post hoc test were used to compare the viscosity between resin composites at each temperature (25 °C and 37 °C). Also, t-test for independent data

Table 1 – Resin composites studied, order with increasing filler fraction, by volume.

Code	Material	Manufacturer	Lot. no.	Composite description	Resin matrix	Filler content	
						Size	Load (vol%)
VDF	Venus Diamond Flow	Heraeus Kulzer GmbH, Hanau, Germany	010105	Flowable nano-hybrid	Multifunctional methacrylates (UDMA, EBADMA)	Ba-Al-F silicate glass, YbF ₃ and SiO ₂ (0.02–5 μm)	41
FBF	Filtek Bulk Fill	3M ESPE, ESPE, St. Paul, MN, USA	N390563	Bulk-Fill	Bis-GMA, bis-EMA, UDMA	A combination of ytterbium trifluoride filler (sizes range from 0.1 to 5.0 μm) and zirconia/silica (size range of 0.01 to 3.5 μm)	42.5
GAUF	G-aenial Universal Flo	GC Corporation Tokyo, Japan	1204112	Flowable nano-hybrid	UDMA bis-MePP TEGDMA	Silicon dioxide (16 nm), strontium glass (200 nm)	50
TEC	Tetric EvoCeram	Ivoclar Vivadent AG, Schaan, Liechtenstein	P35262/P18603	Nano-hybrid composite	Bis-GMA, UDMA, DMDMA	Barium glass, ytterbium trifluoride, mixed oxide and prepolymer. Mean particle size 550 nm	54
SP TPH3	Spectrum TPH3	Dentsply Detrey GmbH, Konstanz, Germany	1107001855	Sub-micron hybrid composite	Bis-GMA, bis-EMA, TEGDMA	Barium aluminio borosilicate (<1 μm). Barium fluoro aluminio borosilicate (<1 μm) Highly dispersed silicon dioxide (10–20 nm)	57
eXP	ever XPosterior	GC Corporation Tokyo, Japan	1212261	Reinforced base	Bis-GMA, TEGDMA, PMMA	Hybrid filler fractions & E-glass fibers	57
IPS ED	IPS Empress Direct	Ivoclar Vivadent AG, Schaan, Liechtenstein	P43019	Nano-hybrid composite	Dimethacrylates (20–21.5 wt%)	40–3000 nm with a mean particle size of 550 nm	58
VP	Venus Pearl	Heraeus Kulzer GmbH, Hanau, Germany	VP101210	Nano composite	TCD-DI-HEA and UDMA	Barium aluminum fluoride glass, pre-polymerized filler, highly discrete nanoparticles (5 nm to 5 μm)	59
EXHD	Esthet. XHD	Dentsply Detrey GmbH, Konstanz, Germany	1105311	Nano-hybrid composite	Bis-GMA, a bis-EMA, and TEGDMA	Barium fluoroborosilicate glass (<1 μm) and nano filler silica (0.04 μm)	60

– Table 1 (Continued)

Code	Material	Manufacturer	Lot. no.	Composite description	Resin matrix	Filler content	
						Size	Load (vol%)
GAA	G-aenial Anterior	GC Corporation Tokyo, Japan	1204232	Micro-filled hybrid composite	UDMA and dimethacry- late. bis-GMA free	Pre-polymerized fillers (16–17 μ). Silica, strontium and lanthanide fluoride. Silica and fluoroaluminosilicate >100 nm, fumed silica <100 nm	63
FS XTE	Filtek Supreme XTE	3M ESPE, ESPE, St. Paul, MN, USA	N284875	Nano-hybrid composite	Bis-GMA, UDMA, TEGDMA, PEGDMA and bis-EMA	20 nm silica filler, 4–11 nm zirconia filler, and aggregated zirconia/silica cluster filler (comprised of 20 nm silica and 4–11 nm zirconia particles)	63.3
VD	Venus Diamond	Heraeus Kulzer GmbH, Hanau, Germany	010046	Nano composite	TCD-DI-HEA and UDMA	Barium aluminum fluoride glass – highly discrete nanoparticles (5 nm to 20 μ m)	64
GDP	Gradia Direct Posterior	GC Corporation Tokyo, Japan	1203121	Micro-filled hybrid composite	UDMA and dimethacry- late co-monomers	Methacrylates of average particle size: 0.85 μ m, fluoro-alumino-silicate glass average particle size: 0.85 μ m	65
GAP	G-aenial Posterior	GC Corporation Tokyo, Japan	1204251	Micro-filled hybrid composite	UDMA and dimethacry- late. bis-GMA free	Pre-polymerized fillers (16–17 μ). Silica, strontium and lanthanide fluoride. Silica and fluoroaluminosilicate >100 nm, fumed silica <100 nm	65

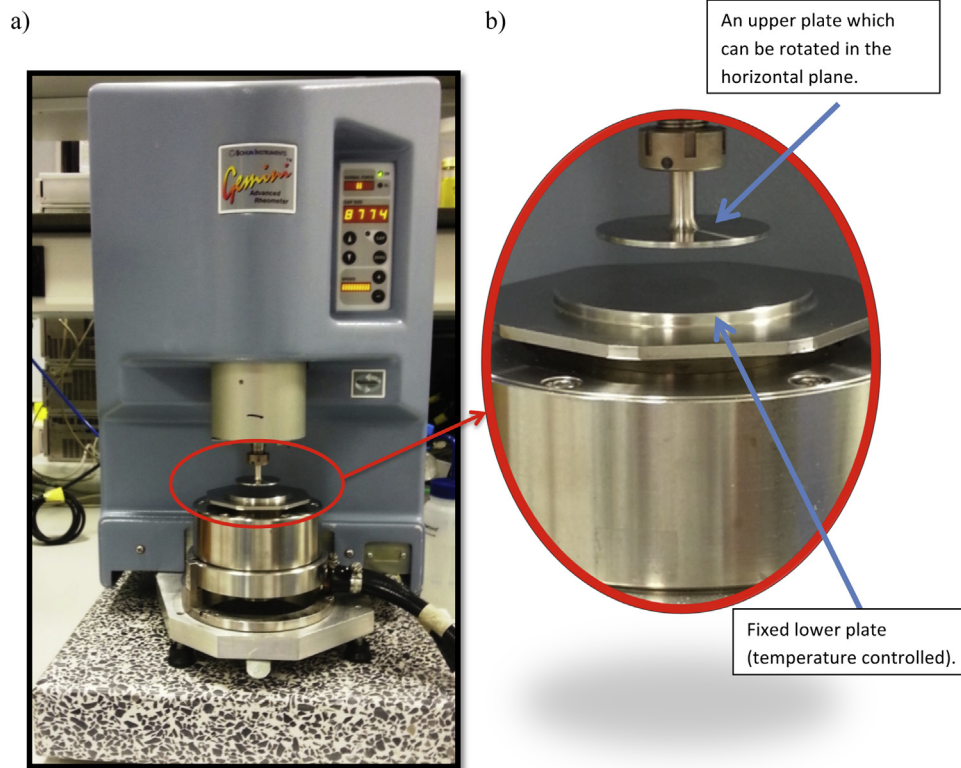


Fig. 1 – Bohlin rheometer (a) and the plate assembly (b).

was used to compare the viscosity at both temperatures (25 °C and 37 °C) within each resin composite.

Reduction in viscosity was calculated according to (Eq. (2))

$$\left\{ \frac{(V_0 - V_1)}{V_0} \right\} \times 100 \quad (2)$$

where V_0 is the viscosity at 25 °C, and V_1 is the viscosity at 37 °C.

3. Results

All composites exhibited pseudoplasticity “shear thinning”. Fig. 2 is a representative double-logarithmic plot of the shear-rate dependence of the resin-composites, showing the lowest Filtek Bulk Fill (FBF) and the highest Filtek Supreme XTE (FS XTE) viscosities amongst the materials examined. Both show pseudoplastic behavior, but not in accordance with a simple power-law model, for which these plots would be linear. The low viscosity FBF resulted in measurement over a high range of shear-rate. The high viscosity FS XTE experienced a lower range of shear-rate displacement. The viscosity increased as the percentage of filler loading (by volume) increased (Fig. 3).

Viscosity significantly decreased with the increase in temperatures for all materials ($p < 0.05$). The viscosity ranged between 0.05–349.33 kPas at 25 °C and 0.03–132.00 kPas at 37 °C. FS XTE exhibited the highest viscosity among the materials at both temperatures ($p < 0.05$). FBF on the other hand,

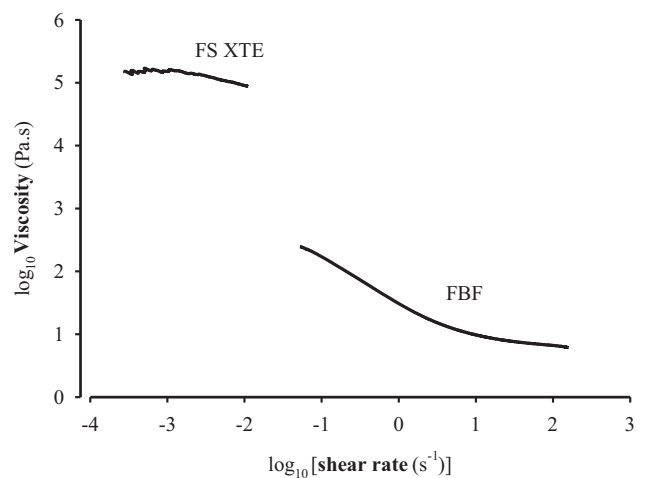


Fig. 2 – Double-logarithmic plot of viscosity versus shear-rate for Filtek Bulk Fill (FBF) and Filtek Supreme XTE (FS XTE) composites. These materials represent the lowest (FBF) and the highest (FS XTE) viscosities amongst the materials examined. Both show pseudoplastic behavior, but not in accordance with a simple power-law model, for which these plots would be linear. The low viscosity FBF resulted in measurement over a high range of shear-rate. The high viscosity FS XTE experienced a lower range of shear-rate displacement.

Table 2 – Viscosity (kPa s) of resin composites measured at 25 °C and 37 °C. The viscosity of each resin composite decreased significantly ($p < 0.05$) on increasing the temperature from 25 °C and 37 °C.

Resin composites	Viscosity (V_0) at 25 °C (n=3)		Viscosity (V_1) at 37 °C (n=3)		Viscosity change $\{(V_0 - V_1)/(V_0)\} \times 100$	
	Mean	SD	Mean	SD	(%)	p values
VDF	0.12	0.02	0.07	0.02	40.8	0.019
FBF	0.05	0.00	0.03	0.01	48.7	0.003
GAUF	1.59	0.06	0.50	0.03	68.4	0.000
TEC	44.92	2.03	8.82	1.65	80.3	0.000
SP TPH3	83.33	15.04	6.48	0.93	92.2	0.012
eXP	101.83	14.89	16.44	1.17	83.8	0.001
IPS ED	21.82	1.67	9.35	0.58	57.1	0.000
VP	104.20	0.72	13.14	2.87	87.3	0.000
EX HD	281.00	23.30	41.72	2.57	85.1	0.003
GAA	8.79	0.92	1.91	0.04	78.2	0.006
FS XTE	349.33	13.80	132.00	16.09	62.2	0.000
VD	84.71	1.46	28.43	0.60	66.4	0.000
GDP	12.06	0.89	2.44	0.13	79.7	0.000
GAP	286.33	29.26	57.93	1.82	79.7	0.005

showed the lowest viscosity at both temperatures ($p < 0.05$) (Fig. 4 and Table 2).

The reduction in viscosity ranged from 40.8% (for Venus Diamond Flow) to 92.2% (for Spectrum TPH3) (Fig. 5).

4. Discussion

Dental composites are composed of resin matrices; mainly bis-GMA diluted with TEGDMA and inorganic fillers (i.e. zirconia, silica fillers) of different loading (i.e. by volume or weight). The presence of fillers enhances the resistance of the composite to abrasion, improves its mechanical strength and resistance to indentation, and reduces its shrinkage during polymerization. The resin matrix composition and ratio [19,20], the filler content shape, size, loading and its silane surface treatment [21], interlocking between filler particles, and interfacial interaction between filler particles and the matrix resin greatly affect the handling properties of the composite including viscosity. Mechanical properties of cured composites have been extensively studied over many years according to variations

in their formulation [22–24] but there have been fewer studies on rheology of uncured paste formulations.

This study investigated the effects of different composite formulations at both room temperature (25 °C) and body temperature (37 °C) – that may be greater than some oral temperatures. The viscosities measured was statistically significantly influenced by both formulation and temperature. Thus our null hypothesis was rejected.

Filtek Bulk Fill and Venus Diamond Flow together showed the lowest viscosities (0.05 and 0.12 kPa s, respectively) among all composites. This can be directly attributed to their low filler loadings that were, respectively, 42.5% and 41% by volume. However, the smaller differences between their viscosities can be due to chemical differences between their resin matrix mixtures. Filtek Supreme XTE and Esthet-X HD had the highest viscosities (above 281 kPa s) although their filler loadings (63.3% and 60%, respectively) were less than Venus Diamond (64%). This could be due to differences in resin matrix as Filtek Supreme XTE and Esthet-X HD have bis-GMA, UDMA, TEGDMA and bis-EMA while Venus Diamond has

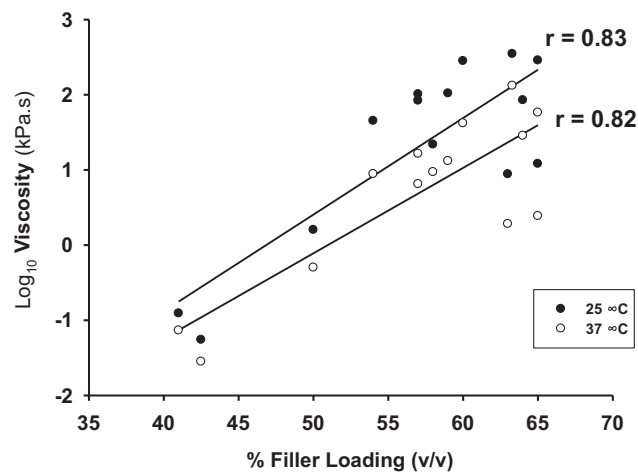


Fig. 3 – Correlation with filler loading (% v/v) of the log viscosity (kPa s) at 25 °C (solid circles) and 37 °C (open circles).

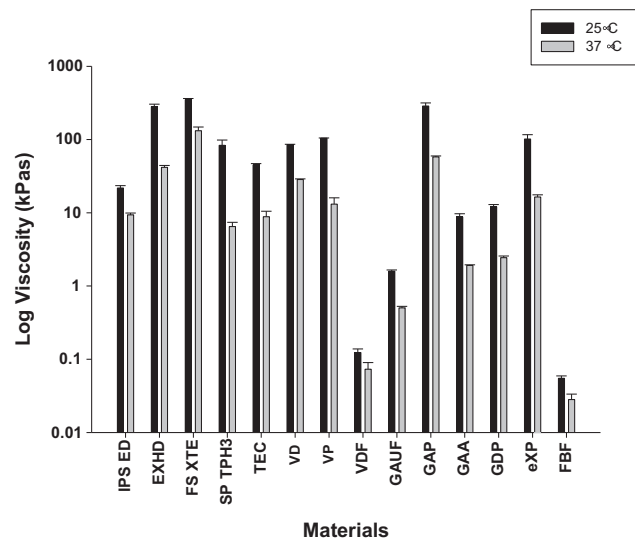


Fig. 4 – Viscosity significantly decreased with the increase of temperature for all composites.

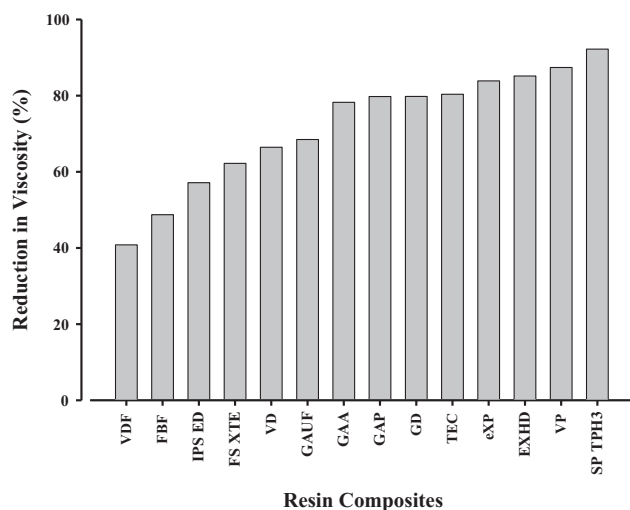


Fig. 5 – Reduction in viscosity (%) as a consequence of temperature increase for the resin composites.

TCD-DI-HEA and UDMA only. bis-EMA co-monomer has a low viscosity (≈ 0.7 Pa s) with high molecular weight (540 g/mol), compared to bis-GMA (viscosity = 600–1000 Pa s and molecular weight = 512 g/mol). This viscosity difference is attributed to the absence of the hydroxyl groups in bis-EMA. Since bis-EMA presents high molecular weight, its use as a diluent is interesting when compared to low-molecular weight monomers such as TEGDMA because TEGDMA increases polymerization shrinkage [25]. As expected, the resin mixtures containing high concentrations of bis-GMA had greater viscosity. This is related to the reduced chain flexibility and consequent reduced molecular mobility of bis-GMA, which reduces the degree of conversion of the material [26].

The flow of unset flowable resin composites is affected by the incorporation of inorganic fillers of different sizes, shapes and loadings [10,14,16,27]. Furthermore, the interlocking between filler particles and interfacial interactions between filler particles and the resin matrix play an important role in their viscoelastic properties [12]. Generally the viscosity of composites increases with increased filler content that depends to a great extent on the hydrodynamic force acting on the surface of particles or aggregates of particles, but which is generally unrelated to the particle density.

As temperature increases, the viscosity of the composites decreases. This is understood in terms of the Arrhenius equation: $\dot{\eta} = A e^{E/RT}$ ($\dot{\eta}$: viscosity; A: proportional coefficient; E: activation energy for flow; R: gas coefficient; T: temperature) which shows the relationship between the temperature and fluid viscosity. Composite pastes with high filler loadings are normally hard and stiff at room temperature when they are squeezed out of a compule or syringe, but they become more readily adaptable in the oral cavity and can be molded into a prepared tooth cavity [10].

A Bohlin Rheometer was used to measure the dynamic viscoelastic property of the composite resins. Various methodologies have been cited in the literature. Jacobsen et al. [2] and Ferracane et al. [8] used a rotational rheometer to study chemically cured dental composites and indicated that the

composites are pseudoplastic and their fluidity is strongly dependent on the resin phase. Opdam et al. [1] compared the consistency of several resin composites by a press method. Lee et al. [11] characterized the viscoelastic properties of resin composites using a dynamic oscillatory rheometer and, more recently, using a vertical oscillation rheometer [16]. Lee et al. studied the rheological properties of resin composites with variations in monomer and filler composition using a dynamic oscillatory rheometer and reported on significant differences in viscoelasticity between flowable, universal hybrid and packable composites. The viscosity of composites varies significantly between brands though they were nominally of the same class [10]. In all composites of that study, pseudoplastic behavior was exhibited as the oscillation frequency was increased.

This study showed the influence of the compositional variables of resin composites on the resin composites viscoelasticity and their rheological characteristics. Other clinical handling characteristics of these materials: stickiness, consistency, packing stress and slumping are currently being investigated.

5. Conclusions

- There was a considerable variation in the resin composite viscosities measured at 25 °C as an average value for shear-rate sweep. The factor between the least and highest viscosities was about 7000.
- The formulation differences that led to such a great variation were primarily: (i) volume fraction of fillers and (ii) particle size, the latter influencing total particle surface area.
- Temperature increases from 25 °C to 37 °C significantly reduced viscosities for all composites examined by between 40% and 90%.

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