Thermal Behavior of Hybrid Composite, Giomer, Ormocer and Silorane: A Comparative Study Using Two Light Curing Intensities

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***Abstract* – Purpose of the study was to evaluate the effect of type of composite resin (CR) and the intensity of curing device on thermal behavior. A microhybrid composite (C), a giomer (G), an ormocer (O) and silorane (S) were chosen. Specimens were polymerized with a higher (H) and a lower (L) energy LED curing device. Coefficient of thermal expansion (CTE) measurements were performed with a dilatometer at temperature range 20-60ºC with temperature raise 2ºC/min. CTE values in lower temperatures (21ºC-37ºC) (CTEa) were compared to CTE values in higher temperatures (37ºC-55ºC) (CTEb). CTEa for all resin composites ranged from 5.43 – 38.67 (x 10^{-6}/ºC) and CTEb from 22.20 – 48.87 (x 10^{-6}/ºC), thus depending on CR type, curing intensity and temperature range. Type of CR and light intensity of curing device have an effect on thermal behavior. The higher the temperature, the higher the CTE for all materials. It is advisable to polymerize C with lower intensity curing unit and S and O with higher intensity. G exhibits the highest CTE values with both curing devices, while O exhibits intermediate results with both light intensities in all temperatures. Higher intensity LED resulted in inhomogenous thermal behavior of the tested materials, especially of S.**

***Index Terms* – Coefficient of Thermal Expansion, Composite Resin, Silorane, Ormocer, Giomer**

I. INTRODUCTION

An ultimate goal of adhesive dentistry and composite resin restorations is to have a material that is dimensionally and chemically stable, with the ability to seal tightly the margins of the restoration. To date, this is reflected in the variety and continuous evolution of composite resin types, regarding altered monomer chemistry or filler technology. Besides conventional hybrid composites, giomers, ormocers and siloranes are also available, the latter as an effort to reduce polymerization shrinkage.

Wide thermal fluctuations in the oral environment are of dietary origin. During the consumption of hot food and drink, maximum tooth surface temperature is approximately 47°C. Temperature ranges between 0-67°C have also been reported, but they are considered unrealistic [1] - [3]. Such fluctuations imply corresponding volumetric increases and decreases, undermining mechanical properties [4]. The marginal gap created due to polymerization shrinkage and poor adhesion to the dentinal walls, may increase due to thermally induced loads continuously introduced to restored teeth, resulting in marginal degradation and

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microleakage [5] – [10]. The amount of deformation due to thermal loading is given by the coefficient of thermal expansion (CTE). The CTE is an inherent characteristic of each material at a specific temperature. When dealing with thermally induced volumetric changes, comparison of CTE values of the restorative material and the tooth substance is more important than the CTE value of the material itself [11]. When two materials expand or contract at a similar rate, gap formation at the interface is almost a nonissue, thus microleakage is negligible [5], [8]. The CTE of enamel is approximately 17x10-6/°C and 11x10-6/°C for dentine [6]. It was found since 1920s that dental materials expand about 7 times more than dental tissues [12]. Recent studies demonstrate that for composite resins, the CTE range is 20 - 80x10-6/°C [9], [13], [16], and is affected by type of organic copolymers [14] and by filler load [15], [16] but there is lack of comparative information among different types of composite resins available nowadays, giomers, ormocers and silorane.

In order to obtain optimum properties, sufficient light in the proper wavelength must reach all areas of a light-activated restoration to polymerize it totally and ensure its longevity [17]. Light intensity of the curing device ranges from $400mW/cm^2$, being the minimum intensity for proper 60 ‘‘ curing in 2 mm depth [18], to $>4000mW/cm^2$ up to date. Not receiving the appropriate amount of energy may lead to reduced degree of conversion, increased cytotoxicity, reduced hardness, increased wear and marginal breakdown [19]. On the other hand high intensity curing light can lead to extensive shrinkage stress, correlated with a higher degree of conversion [20]. Polymerization with plasma arc units results in higher CTE values [21], however LED curing units are more widely used. The effect of curing light intensity of LED curing devices on thermal behavior, has not yet been investigated.

This study evaluated the CTE of 4 types of CR, using 2 different LED curing devices with a lower and a higher light intensity. Null hypothesis was that there is no difference in CTE values exhibited by different types of CR, when polymerized with a lower or higher intensity curing device.

II. MATERIALS AND METHODS

A hybrid composite (C), a giomer (G), an ormocer (O) and silorane (S) were tested (Table 1). Eight cubic specimen 4mm thick were made per product in A2 shade, by placing the material into an open-ended stainless-steel mold. Mold was bulk filled and CR was polymerized over a thin cellulose strip, with a higher (n=4) and a lower light intensity curing device (n=4). The tip of the curing device was placed in contact with the cellulose strip, during the curing process. Higher light intensity LED (H) was Flash max (CMS Dental, Copenhagen, Denmark) (3’’ curing time, >4000mW/cm2) and lower light intensity LED (L) was Radii plus (SDI, Bayswater, Australia) (20’’ curing time, 1500mW/cm2). Light intensity of both curing devices was checked with a radiometer, and appeared to be withing the limits of the manufacturer’s statement. Groups were marked as: CH, GH, OH, SH for higher intensity LED and CL, GL, OL, SL for lower intensity LED. Samples were stored in distilled water 37oC in a dark container for 24h before CTE testing. Dilatometer DIL 402C (NETZSCH) was used to determine the values of CTE of composite resin materials at temperature range 20 – 60oC. Temperature range was divided into lower temperatures (21-37oC) and higher temperatures (37-55oC), regarding 37oC (body temperature) as division point. Thus thermal behavior was evaluated at two temperature groups. Extreme low and extreme high temperatures were excluded in order to attain unbiased results, by eliminating possible material shock. The probe of the dilatometer was calibrated with standard Al2O3 cylinders in the beginning. Each specimen was held horizontally in a chamber and volumetric changes were detected by a displacement transducer. Temperature raised at a slow rate of 2oC/min in order to obtain uniform distribution. Only one measurement was per specimen, in order to reduce additional polymerization shrinkae cause by the elevated temperatures. CTE was calculated using internal software and diagrams were extracted which reproduce each material’s thermal behavior. CTE was internally calculated using the following formula:

$A=\Delta L/L0 \text{ x } \Delta T$,

where ΔL represents the probe displacement when temperature change is ΔT and L0 corresponds to initial specimen length. $\Delta L/L0$ represents linear shrinkage.

Table 1. Material Composition

Type of material	Brandname	Manufacturer	Composition	Filler load (w/w)
Hybrid Composite (C)	Filtek Z250	3M-ESPE, St. Paul, MN USA.	Bis-GMA, UDMA and Bis-EMA, zirconia/silica fillers with average size 600 nm.	82%
Giomer (G)	Beautifil II	Shofu Dental GmbH, Rattingen, Germany	Bis-GMA, TEGDMA, Aluminofluoro-borosilicate glass as S-PRG fillers 10-20 nm, Al_2O_3, DL-Camphorquinone.	83.3%
Ormocer (O)	Admira	Voco GmbH, Cuxhaven, Germany	Ormocer, BIS-GMA, UDMA with fillers 700 nm.	77%
Silorane (S)	Filtek Silorane	3M-ESPE	Epoxycyclohexylcyclopolymethylsiloxane, BIS-3,4-Epoxycyclohexylethyl-phenyl-methylsilane, Quartz/Yttrium trifluoride fillers 5 nm-4000 nm, mixture of various by-products,	76%

Table 2. CTE Results ($x10^{-6}$)

	Lower Temperature		**Higher Temperature**	
Group	Mean	Standard Deviation	Mean	Standard Deviation
CL	5.434	3.962	22.20	9.937
CH	12.04	15.49	30.69	13.12
GL	15.49	9.453	47.06	6.159
GH	38.67	23.44	48.87	14.13
OL	14.89	3.58	35.82	3.217
OH	8.760	5.075	23.77	5.564
SL	16.47	12.44	47.54	9.128
SH	5.753	2.640	41.74	28.76

Thickness is measured for every specimen at 4mm and as a result volumetric changes were calculated by changes in specimen length.

Statistical analysis was performed using Tukey’s test (SPSS 20.0). Level of significance was set at 5%.

III. RESULTS

Mean values and standard deviations are demonstrated in Table 2. Ascending CTE values for lower temperature range are: CL < SH < OH < CH < OL < GL < SL < GH and for higher temperature range are: CL < OH < CH < OL < SH < GL < SL < GH. CTE values are increased with elevated temperature for all materials. All types of CR expanded with increasing temperature. Fig. 1 – 4 show the relationship between the displacement of the probe (volumetric change in specimen) and increase in temperature during heating. The lower the CTE of the specimen, the smaller the displacement of the probe. Regarding CTE, CL is generally lower than CH. S and G exhibited the highest CTE values when cured with either of the devices. However, SH is lower than SL for both temperature groups. O behaves well with both light intensities for both temperature groups. Differences between all the comparisons are not statistically important ($p>0.05$).

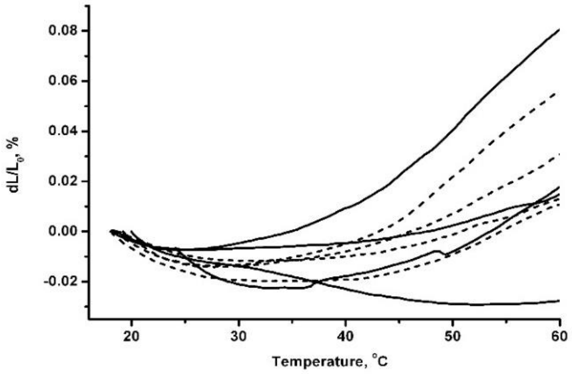

Fig. 1. Thermal expansion of group C. Continuous line corresponds to higher energy light intensity curing unit, while dotted line corresponds to lower intensity.

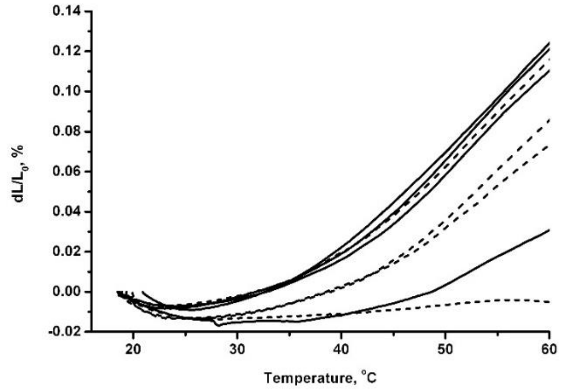

Fig. 2. Thermal expansion of group G. Continuous line corresponds to higher energy light intensity curing unit, while dotted line corresponds to lower intensity.

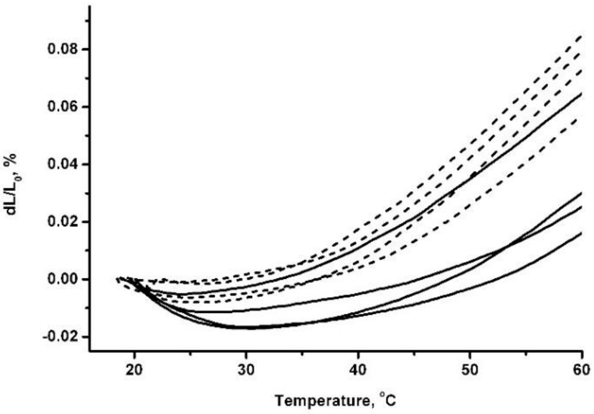

Fig. 3. Thermal expansion of group O. Continuous line corresponds to higher energy light intensity curing unit, while dotted line corresponds to lower intensity.

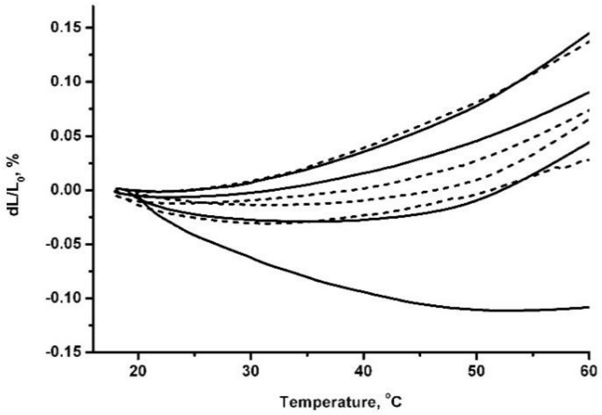

Fig. 4. Thermal expansion of group S. Continuous line corresponds to higher energy light intensity curing unit, while dotted line corresponds to lower intensity.

IV. DISCUSSION

The null hypothesis of this study was accepted. Differences in CTE values exhibited among different CR types, when polymerized with a lower or a higher intensity curing device, were not significant.

CTE measurement conditions in the present study were challenging for the materials tested. Data were collected during temperature increase [9], [14], [22], as it is thought to cause larger CTE variations because of material shock, compared to temperature decrease [23], when thermally induced volumetric changes are more even and milder. CTE changes with temperature are non-linear (Fig. 1-4), whatever the tested CR material is, and this is confirmed by Powers *et al.* 1979 [8], and Sidhu *et al.* 2004. [24] However, on the other hand, Versluis *et al.*, 1996 [16], demonstrate results of good linearity. The mathematical sign of the CTE values has no correlation with CTE value increase or decrease.

Increases in the CTE value demonstrate that the material expands (when CTE > 0) or contracts (when CTE < 0) at a higher rate, and vice versa. Published literature presents results as mean CTE values but fluctuations are considered the most influential factor in microleakage combined with the lack of adhesion [5]. Our results, as seen in the diagrams, show that CTE is defined at a specific temperature. However in order to comply with the literature, we divided the temperature range 20-60°C into two parts: 21-37°C for the lower temperatures and 37-55°C for the higher temperatures, as used by Sidhu *et al*. 2004. Differences were observed in the CTE values, depending on the temperature range examined, as shown in previously published studies [14], [25], [26].

A variety of CR has recently become available. G have the advantage of fluoride release, while O and S have been developed in order to reduce polymerization shrinkage. The differences between C, G, O and S are basically due to their monomers, chemical reactions and fillers. Differences in thermal behavior are either attributed to the organic matrix or to the inorganic fillers [14]. The tested materials contain some or all Bis-GMA, TEGDMA and UDMA in various concentrations (Table 1), however it has been previously demostrated that these monomers have a similar CTE [21]. Differences exhibited by S, could be attributed to the siloxane monomers, which make the material unique. It has also been noted that S, besides lower polymerization shrinkage, has an overall mixed mechanical performance [27]. Intermediate CTE values are exhibited by O, also containing a unique organic matrix ingredient. It can be assumed therefore, that type of organic matrix and chemical reactions may influence the thermal behavior of the restorative material at some extent. An intrisic polymer material characteristic is the temperature at which th polymer goes from the glassy to the rubbery state (T_g). CTE values are connected with T_g and it has been previously described [14] that this transition is evident in CTE curves (Fig. 1 - 4). Although Bis-GMA, TEGDMA and UDMA have similar CTE, as mentioned above, their copolymers mixed with fillers show a decrease in CTE [14]. Heat-generation pattern varies according to the type of CR [28] and although there is no such study published, heat absorption and distribution may be connected with CTE values. A linear correlation between the CTE and the filler concentration is noted [11], which is also exhibited for G in our study, having the highest filler load and highest CTE values (Table 1 and 2). Since this correlation does not apply for the other three tested materials it seems that different basic chemistry is more crucial in determing materials' behavior. Besides filler load, type and size of the fillers may also contribute to thermal behavior [15], [29]. All tested materials have different types of fillers, according to the manufacturers' data. Filler size however, does not seem as important as polymerization light intensity, as CTE values in C and O, having similar-sized fillers, are influence more by light intensity.

Two LED curing devices with different light intensities have been compared in this study. Their common feature is that they emit in continuous mode, near 460 – 470 nm, which is the absorption peak of camphoroquinone and is contained in CR as a photoinitiatior. Higher light intensity unit emitted almost three times stronger than lower light intensity unit, according to measurements with a radiometer. It has been previously demonstrated that higher light intensity of the curing device lead to lower CTE values in resinous materials [22], but this does not comply with all of the tested materials in this study. Only polymerization of S and O with higher light intensity unit, resulted in lower CTE values (Table 2). This is also evident in Fig. 3 and Fig. 4 since, by comparing the placement of the continuous line in regard to the dotted line. It has been reported, that under-polymerized specimens become softer and can expand more easily than those which are fully polymerized [21], resulting in higher CTE, values as exhibited in S and O. However, sufficient polymerization of CR is a combination of proper light intensity and duration, since emission spectrum is nearly the same for all devices, corresponding to absorption peak of camphoroquinone [18]. Judging only by light intensity, a safe conclusion about polymerization extent cannot be made. However, in the present study, regarding polymerization duration, manufacturer's instructions were followed for each curing device. It can be assumed, that polymerization of S and O with lower intensity curing unit and polymerization of C and G with higher intensity device, may result in insufficient polymerization, since those combinations exhibited higher CTE values. On the contrary, it should be noted, that C and G, may have exhibited lower CTE values when polymerized with lower intensity, because of the effect of temperature [30], on polymerization continuation and thus on additional polymerization shrinkage, since C and G are the two materials of the present study which have not been designed to reduce polymerization shrinkage. It is demonstrated that polymerization shrinkage of unfilled Bis-GMA/TEGDMA resins, accounts for minimal volumetric changes, compared to those expected from temperature raise during polymerization (up to 49^{o}C) [23]. However, C and G are highly filled (>80%) (Table 1) and thermal behavior differs significantly [14].

In conclusion, thermal behavior of each CR type depends on chemical composition and light intensity used for the polymerization. All CR types expand with temperature rise and CTE values are higher in elevated temperatures. The transition to higher temperatures, even if thermal equilibrium cannot be attained in a short time in the oral cavity, induces material fatigue [9] and this is why restorative materials are expected to have less predictable behavior at higher temperatures with respect to CTE. However great the challenge, oral conditions are much more complex and results of this study should be interpreted within certain limitations in mind. Higher intraoral temperatures last for a short time and are usually confined to the surface, depending on the thermal diffusivity of the tooth and on the action of the pulp as a heat sink with circulating blood supply [14]. Glass transition temperature of resinous materials [9] and moisture levels may also affect CTE measurements [25], [26]. In addition, tooth substance structures are not homogenous. An in vitro study showed that human dentine expands slightly on heating and contracts at higher temperatures [31].

Within the limitations of this in vitro study, it is suggested that S and O should be polymerized with higher intensity units, while for C and G, lower intensity curind devices should be chosen.

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