The effect of different light curing units on Vickers microhardness and degree of conversion of flowable resin composites

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This study aimed to assess the influence of different light curing units (LCUs) on the polymerization of various flowable resin composites. Three LCUs (Optilux 501, Elipar™ DeepCure-L LED and Bluephase®20i) and eight flowable resin composites: MI FIL Flow, Estelite Flow Quick, Estelite Universal Flow (medium), Estelite Universal Flow (super low), Beautifil Flow Plus, Clearfil Majesty ES Flow, Filtek Supreme Ultra flowable and TetricEvo Flow were tested. For Vickers microhardness (VHN) test and degree of conversion (DC), specimens were prepared and polymerized for 20 s. VHN test was performed at top surfaces (3 indentations) and DC for each specimen was measured using Fourier transform infrared (FT-IR) spectroscopy after 24 h dry storage in dark at 37°C. The data were analyzed with 2-way ANOVA and t-test with Bonferroni correction. DC and hardness values showed a relationship between materials and LCUs. The curing efficacy of LCU type may depend on the material composition.

Keywords: Light curing units, Flowable resin composites, Vickers microhardness, Degree of conversion, Polymerization efficacy

INTRODUCTION

Since flowable resin composite appeared during the 1990s, it was a valuable improvement for resin-based restorative dental materials⁴. Flowable resin composites represent low viscosity resin composites resulting of lower filler content 37–53% (volume) than conventional composites². These materials have lower viscosity and subsequently offer a higher flow to allow easier filling the cavity, better adaptation to cavity walls and greater elasticity compared with earlier available products⁹. The newly developed flowable resin composites are accepted higher wear resistance as compared to the conventional resin composites⁴ and enhanced mechanical properties leading to a wider range of applicability⁴,⁶. In an adhesive restorative procedure, adequate polymerization is essential for optimal physical, mechanical properties and clinical longevity of resin composites⁷.

The choice of light-curing units (LCUs) is essential for polymerizing these materials⁶. These light-activated materials can be cured with either quartz-tungsten-halogen (QTH) or light-emitting diode (LED) curing units. QTH units have been used for a long time, but they demonstrate a reduction in irradiance output over time caused by the degradation of the lamp and filter. LED lights exhibit less degradation, with a specific blue-emitting light that does not need a filter. However, single wavelength LED devices with a narrow wavelength spectrum is specific for the camphorquinone photoinitiator, whereas QTH and polywave LED curing units have a wide spectral range and output⁸.

Although significant enhancements in flowable composite materials have made, there is still the potential issue of inadequate degree of conversion (DC)⁵⁰ and poor mechanical properties due to inefficient polymerization which can cause reduced wear resistance, lack of color stability, monomer elution and early restoration failure⁴.⁵.

The DC of resin composite is the amount of monomer converted to polymer. For the determination of the DC of dental resin composites, Fourier transforms infrared spectroscopy (FT-IR) and microhardness (MH) are frequently used. Infrared spectroscopy is used to determine the DC by the proportion of the remaining concentration of aliphatic C=C double bonds in a cured composite sample relative to the total number of C=C bonds in the uncured material². The hardness of a composite is the important property that enables it to resist plastic deformation, penetration, indentation, and scratching. The microhardness of dental composite materials is typically used to predict their resistance to abrasion when used for restoration in load-bearing areas¹³.

There were a few studies to analyze the interaction...
between newly developed flowable resin composites and different LCUs, which may impact the clinical performance of composite restorations\textsuperscript{14-17}. This study aimed to evaluate the influence of different LCUs on the polymerization of various flowable resin composites. It was hypothesized that different LCUs have varying effects on the DC and microhardness of various flowable resin composites.

**MATERIALS AND METHODS**

Eight commercial flowable resin composites; MI FIL Flow (MIF; GC, Tokyo, Japan), Estelite Flow Quick (EFQ; Tokuyama Dental, Tokyo, Japan), Estelite Universal Flow (medium) (EUF-M; Tokuyama Dental), Estelite Universal Flow (super low) (EUF-S; Tokuyama Dental), Beautifil Flow Plus (BFP; Shofu, Kyoto, Japan), Clearfil Majesty ES Flow (CMF; Kuraray Noritake Dental, Tokyo, Japan), Filtek Supreme Ultra flowable (FSU; 3M ESPE, St. Paul, MN, USA), and Tetric Evo Flow (TEF; Ivoclar Vivadent, Schaan, Liechtenstein) were tested in this study. The detail information according to the manufacturers was shown in Table 1.

The LCUs used in this study were a conventional QTH lamp with standard mode (Optilux 501, Kerr, Orange, CA, USA), a monowave LED LCU (Elipar\textsuperscript{TM} DeepCure-L, 3M ESPE) and a polywave LED LCU with high mode (Bluephase\textsuperscript{®}20i, Ivoclar Vivadent). The specifications of the three LCUs were shown in Table 2.

**Analysis of LCU**

The spectral radiant power output of each LCU was

| Code | Composite | Chemical composition | Filler % by weight (volume) | Manufacture | Lot No. |
|------|-----------|----------------------|-----------------------------|-------------|---------|
| MIF  | MI FIL Flow (Nanohybrid flowable composite) | UDMA, TEGDMA,Bis-MEPP, Silicon dioxide, Strontium glass, photoinitiator | 69% (50%) | GC, Tokyo, Japan | 1605261 |
| EFQ  | Estelite Flow Quick (Supra-nano filled flowable composite) | Bis-MEPP, TEGDMAUDMA, Silica-zirconia filler, Silica-titania filler, Camphorquinone, Radical amplifying agent | 71% (53%) | Tokuyama Dental, Tokyo, Japan | J062 |
| EUF-M| Estelite Universal Flow (Medium low flow) (Supra-nano filled flowable composite) | Bis-GMA, Bis-MEPP, TEGDMA, UDMA, Spherical silica-zirconia filler, Camphorquinone, Radical amplifying agent | 71% (57%) | Tokuyama Dental | 008 |
| EUF-S| Estelite Universal Flow (Super low flow) (Supra-nano filled flowable composite) | Bis-GMA, Bis-MEPP, TEGDMA, UDMA, Spherical silica-zirconia filler, Camphorquinone, Radical amplifying agent | 70% (56%) | Tokuyama Dental | 011047 |
| BFP  | Beautifil Flow Plus (F00) (Flowable hybrid composite) | Bis-GMA, TEGDMA, Fluoroboroaluminosilicate, S-PRG (surface pre-reacted glass), photoinitiator | 67.3% (47%) | Shofu, Kyoto, Japan | 051629 |
| CMF  | Clearfil Majesty ES Flow (Super low flow) (Nanohybrid flowable composite) | TEGDMA, Hydrophobic aromatic dimethacrylate, Silanated barium glass filler, Pre-polymerized organic filler | 78% (59%) | Kuraray Noritake Dental, Tokyo, Japan | 3E0023 |
| FSU  | Filtek Supreme Ultra Flow (Nanofilled flowable composite) | Bis-GMA, TEGDMA, Ytterbium trifluoride, Zirconia/silica Nanocluster, nanofiller | 65% (46%) | 3M ESPE, St. Paul, MN, USA | N749471 |
| TEF  | Tetric Evoflow (Nanohybrid flowable composite) | Bis-GMA, UDMA, DDDMA, Barium glass filler, Ytterbium trifluoride, Silicon dioxide, Mixed oxide and copolymer | 58% (31%) | Ivoclar Vivadent, Schaan, Liechtenstein | V45960 |

Bis-GMA, bisphenol A-glycidyl methacrylate; Bis-EMA, Bisphenol A polyethylene glycol diether dimethacrylate; Bis-MEPP, 2, 2’-bis (4-methacryloyloxy polyethoxyphenyl) propane; DDDMA, dekandioldimethacrylate; TEGDMA, Triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate
measured by using a spectroradiometer (USR-45, Ushio, Tokyo, Japan). The wavelength region of each unit was analyzed from 350 to 550 nm at a scanning rate of 1 nm. The irradiance (mW/cm²) of each unit was calculated by integrating output over the spectral irradiance per wavelength. The intensity of each LCU was periodically monitored using a dental radiometer Bluephase Meter II (Ivoclar Vivadent) before the commencement of experimental work.

**Vickers microhardness (VHN) testing**

The composites were inserted into a circular Teflon mold (8 mm diameter and 2 mm thick) and covered with polyester strips on both sides (KerrHawe Striproll, Bioggio, Switzerland). A glass microscope slide was placed on the top surface to extrude excess material and ensure the mold was filled. The composite surface was irradiated over the center of the sample in contact with the top surface through the glass slide and the polyester strip. Eight specimens of each composite were cured with each LCU for 20 s. Specimens were stored dry in the dark at 37°C for 24 h, then the VHN was measured (HM-108, Mitutoyo, Kanagawa, Japan) at the top surface of each specimen (three indentations for each specimen), using a 0.49 N load for 15 s.

**FT-IR spectroscopy**

Eight specimens (2 mm diameter and 2 mm thick) were prepared from each resin composite using a silicone mold. The composite surface was covered with a Mylar strip and glass slide, then the light-curing device tip was placed in contact with the glass slide. Each specimen was polymerized for 20 s and stored dry in the dark at 37°C for 24 h. The infrared spectrum of an uncured specimen prepared from each resin composite using a silicone mold (three indentations for each specimen), using a 0.49 N load for 15 s.

C=C before and after curing, DC of resin composite was calculated by the following equation:

\[ \text{DC}(\%) = 100 \times \left[1 - \left( \frac{R_{\text{cured}}}{R_{\text{uncured}}} \right) \right] \]

where R=peak at 1,634 cm⁻¹/peak at 1,608 cm⁻¹

**Statistical analysis**

VHN and DC data were analyzed using two-way ANOVA, followed by t-test with Bonferroni correction for pairwise comparison. Pearson correlation coefficient used to check the correlation between hardness and DC for each material. For correlation within each light-curing unit, Kendall tau correlation was used as the data showed non-parametric distribution. All statistical calculations were performed using the Statistical Package for the Social Sciences for Windows (SPSS 23, SPSS, Chicago, IL, USA) with α=0.05.

### RESULTS

According to the spectroradiometer measurement, Fig. 1 shows the wavelength range of the Optilux 501 (QTH) has 390–510 nm, Elipar™ DeepCure-L (monowave) has 410–490 nm and Bluephase®20i (polywave) has 390–490 nm respectively. Figures 2 and 3 reveal the means and standard deviations of the VHN and the DC for the eight flowable composites after curing with the LCUs.

Two-way ANOVA performed on hardness and DC showed that these were influenced by both the light source (p≤0.001) and composite (p≤0.001). Hardness and DC were also affected by the interaction between light sources and composite (p≤0.001).

Among the eight materials, the highest VHN was obtained from EFQ regardless of the light source (p≤0.001). The lowest VHN was recorded for TEF (p≤0.001). Also, the results showed that when obtaining the VHN for each material, the monowave LCU had significantly higher VHN than QTH and polywave for EFQ, EUF-S, and FSU (p<0.05) while monowave had significantly lower VHN than QTH and polywave for CMF and TEF (p≤0.001). The polywave LCU showed significantly higher than the monowave and QTH LCU for CMF and TEF (p>0.05).

The highest DC was obtained from BFP (p<0.05) and the lowest from EQF (p<0.05) regardless of LCU used. The results showed there were no statistically significant differences among LCUs for material MIF, EUF-M, EUF-S, and FSU (p>0.05) except for CMF and TEF. For CMF and TEF, the monowave LCU produced significantly lower DC than the QTH and polywave.

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**Table 2 Characteristics of light curing units used**

| Light Curing Units     | Optilux 501   | Elipar™ DeepCure-L | Bluephase®20i |
|------------------------|---------------|--------------------|---------------|
| Type                   | Halogen       | Monowave LED       | Polywave LED  |
| Light intensity (mW/cm²)| 600           | 1,200              | 1,200         |
| Wavelength (nm)        | 390–520       | 430–490            | 390–510       |
| Curing time (s)        | 20            | 20                 | 20            |

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Fig. 1  Spectral output of LCUs.

![Spectral output of LCUs](image)

Horizontal bars show no significant difference between LCUs ($p > 0.05$) and the same lower-case letters indicate no statistical difference among materials ($p > 0.05$).

Fig. 2  VHN of the materials tested.

![VHN of the materials tested](image)

Horizontal bars show no significant difference between LCUs ($p > 0.05$) and the same lower-case letters indicate no statistical difference among materials ($p > 0.05$).

Fig. 3  DC of the materials tested.

![DC of the materials tested](image)

Horizontal bars show no significant difference between LCUs ($p > 0.05$) and the same lower-case letters indicate no statistical difference among materials ($p > 0.05$).
For the pooled data, the Pearson correlation coefficient \((r)\) showed the negative correlation between hardness and DC for each LCU as follow; Halogen; \(r=-0.415, p \leq 0.001\), Monowave; \(r=-0.221, p=0.01\) and Polywave; \(r=-0.336, p \leq 0.001\) (Fig. 4). Kendall tau correlation coefficient \((r_\tau)\) showed the significant correlation between hardness and DC resulted for CMF \((r_\tau=0.924, p \leq 0.001)\), EFQ \((r_\tau=0.44, p=0.032)\), and TEF \((r_\tau=0.890, p \leq 0.001)\) and other composites showed an insignificant correlation between hardness and DC (Fig. 5).

**DISCUSSION**

This study used VHN to evaluate the curing performance of various LCU units on resin composites with different compositions. Two millimeters thick specimen and A2 shade were chosen to ensure a uniform polymerization and to limit the impacts of shade on the light polymerization.
In this study, significant differences were noted in the VHN and DC mean values among tested flowable resin composites, three nanohybrid, three supra-nanofilled, one nanofilled and one hybrid flowable resin composites with a different composition. Adding nanosized fillers to flowable composites increases the viscosity because of filler surface area and also matrix-filler interaction or between filler particles; also, it improves the mechanical and flowable characteristics. Marovic et al., analyzed the hardness of nanofilled composites and proposed that these fillers can achieve a higher hardness as well as good polishability. Additionally, nanofillers can accomplish more close contact with the matrix resin than microfillers. According to the outcomes regardless of LCUs, EFQ indicated a significantly higher hardness compared with all other materials evaluated because it contains supra-nano spherical fillers resulting in superior matrix filler interaction. Generally, the higher the filler content of a resin composite, the higher the surface microhardness. TEF showed significantly lower hardness value than other materials because of its lower filler content. Although CMF showed highest filler content nanohybrid composite among material used in this study, it could not achieve higher hardness value than EFQ because it contained prepolymerized resin fillers, also included in EUF-M, EUF-S and FSU. Filler content did not seem, by all accounts, to be the main affecting parameter. Most manufacturers added prepolymerized filled resin particles to increase filler loading. Prepolymerized fillers never achieve as high hardness value as the composite without prepolymerized particles. It was additionally suggested that different factors, for example, monomer type and ratio, filler shape and size, the degree of polymer cross-linking, and photoinitiators appear to affect surface hardness. This could explain the different hardness values assessed among the materials used in this study.

The degree of polymerization of dental resin composites is fundamentally affected by the nature and the quantity of individual monomers in their composition. The primary monomer used in commercial dental composites is bisphenol A-glycidyl methacrylate (Bis-GMA), which is likewise present in all materials tried in this investigation except for MIF, EFQ, and CMF. Bis-GMA has a high viscosity that can produce a more rapid hardening and production of stronger and stiffer resins. On the other hand, the low mobility of Bis-GMA enable it to accomplish a high DC. Consequently, Bis-GMA must be mixed with diluent monomers of low viscosity, such as triethylene glycol dimethacrylate (TEGDMA), urethane dimethacrylate (UDMA) or bisphenol A polyethylene glycol diether dimethacrylate (Bis-EMA) so as to achieve acceptable levels of polymerization. It was reported that the ultimate DC of different monomer systems increases in the following order: Bis-GMA<Bis-EMA<UDMA<TEGDMA. Despite LCUs, this study evaluated that the highest DC was obtained for BFP while the lowest DC was for EFQ. When Bis-GMA is mixed with the low viscosity monomer, TEGDMA, a synergistic effect on the rate of polymerization and DC has been observed. This could explain the significantly higher DC of BFP than that of other materials since it might rely upon the diluted ratio of low viscosity monomer. Moreover, the inclusion of different parameters, such as the types of monomer and their proportions, and filler morphology and distribution may vary greatly among materials. The absence of data provided by manufacturers concerning the accurate proportions of the various monomers makes clarification of the present outcomes difficult.

It is generally accepted that the greater DC is associated with the higher hardness. The previous studies have proven a positive correlation between the hardness and DC within the same material. Regarding the LCUs, this study showed there was a significantly strong correlation between the DC and the hardness, distinctly seen in CMF and TEF respectively. On the contrary, other studies have found no correlation between DC and hardness for several materials. In the present study, there was a negative correlation between the DC and hardness comparing all materials with each LCU, which concurred with other investigation. Ferracane and other studies revealed that the hardness value cannot be used to compare the DC among various materials because the results greatly depend on the composition of the material. Additionally, it ought to be emphasized that filler characteristics may impact on the hardness, and monomer type and proportions may affect the DC while comparing different materials.

Apart from the characteristics of filler and matrix, the light source has been shown to influence the degree of polymerization of resin composites. The spectral emission profiles of QTH, monowave and polywave LED LCUs are distinctly different. For instance, QTH lights deliver a broad spectrum of blue light ranging from 390 to 510 nm. The monowave LED-LCUs have a single peak of 470 nm that is perfect for the absorption spectrum of camphorquinone (CQ), which is the common photoinitiator of resin composites. The polywave LED LCU emits light with at least two different wavelength ranges. They produce both a shorter violet wavelength and a longer blue wavelength. Violet light is used to activate other photoinitiators such as PPD (1-phenyl-1, 2-propanedione) and Lucirin TPO (monoaclyphosphine oxide) or Iciverin, that are sensitive to light within the range of 350–420 nm wavelength.

The wavelength emitted by LCU should match the absorption spectrum or absorption peak of the photoinitiator in that composite. The polymerization of light-cured resin relies on the quantity of light as well as on its quality such as wavelength and output. In this study, the spectral radiant power of each LCU is shown in Fig. 1. The Optilux 501 produced a light output over a range of wavelengths between 390 and 520 nm, while the DeepCure-L showed a single wavelength peak from 430–490 nm, and the Bluephase20i had two-wavelength peak emissions at 390 and 510 nm that corresponded to the LED chips used in this unit. In our study, the result showed there were no significant differences for VHN and DC among LCUs in same materials, despite the
different radiant exposure (12 J/cm² for QTH and 24 J/cm² for 2 LED LCUs, respectively). This likely occurred because sufficient radiant exposure was delivered within the absorbed wavelength range from LCUs to cure resin composite.

The Vickers Hardness recorded for EFQ, EUF-S, and FSU after the monowave LED LCU curing were significantly higher than those for the QTH and polywave LCUs with these two LCUs not being significantly different from each other. This was because the narrow range of light wavelength emitted by the monowave makes more effective than polywave and QTH in EFQ, EUF-S, and FSU, which contained mainly CQ photoinitiator. Additionally, it might be expected that monowave emitted blue light only and more light energy was delivered in the 460 nm region to the CQ from the monowave LED LCU. However, in CMF and TEF, the monowave LED LCU was significantly lower VHN and DC than polywave and QTH. It has been mentioned that the effectiveness of the type of the LCUs on the polymerization may depend on the photoinitiator type. Therefore, the higher energy output of LED units would not improve the polymerization of resin composite, if the emitted light is not absorbed by the photoinitiator. In these materials, the polywave LCU have preferable curing effectiveness over the QTH and monowave. Price et al., demonstrated that such broad-spectrum LED units could polymerize 2-mm thick specimen of some resins to a more extent degree than the monowave LED; despite the fact that the two units were delivered similar irradiance value. Previous studies have additionally reported improved properties when resin composite with these alternative photoinitiators is photoactivated with broad-spectrum lights. Therefore, these polywave LED LCUs are used to initiate a wider range of photoinitiators. Concerning impact of LCU type used in this study, the polywave LCU would be advised superior performance than QTH LCU. However, the different positions of each of the light emitters along the same LCU tip could influence the homogeneity of the light yield through the light guide tip.

Our study revealed that the monowave LED LCU could not polymerize for some materials because the light output did not coordinate the spectral sensitivity of the photoinitiators present in these resins. However, the concentration of CQ and the presence of any other photoinitiators in the composition are unknown. Additionally, the photoinitiator composition of RBCs should well describe in the manufacturer-supplied material information sheets, so clinicians can best match the output of light with the spectral needs of a given restorative material.

The present investigation had a few limitations. The type and amount of photoinitiators incorporated in flowable resin composites used in this study could not identify. The manufacturers’ information did not specify the kind of photoinitiator used in the materials. Subsequently, further studies are necessary to decide the performance of different LCUs and other mechanical properties of flowable resin composites.

CONCLUSION

Within the scope of this study, the following conclusions were drawn: The adequacy of polymerization was demonstrated to be dependent not just on the type of LCU in addition on the composition of resin composite. Regardless of the energy delivered by LCUs, there was no significant differences among LCUs in same material when the wavelength of these lights coincides within the absorption spectrum of photoinitiators. The monowave LED LCU has poor performance on hardness and DC in CMF and TEF, regardless of whether light-cured composite receives sufficient energy but not a proper wavelength. The polywave LED unit can cure all flowable resin composite, despite the type of photoinitiator contained in their compositions.

CONFLICTS OF INTEREST

The authors declared any conflict of interest related to this study.

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