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Investigation of calcium-silicate content and conversion degree of resin-based pulp capping materials

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ABSTRACT

The present study aimed to measure the microstructure of the TheraCal LC (Bisco Inc., Schaumburg, IL) and Calciplus LC (Imicryl, Konya, Turkey) and to investigate the effect of excitation durations and different light sources on their 'degree of conversion' properties. Three excitation modes of the resinous pulp capping materials were tested at 5, 10, and 20-s: light-emitting diode, and near-infrared laser excitation at 532 and 785 nm wavelengths. Resinous materials and excitation durations factors were analyzed with the two-way ANOVA test and Tukey Post Hoc tests ($\alpha=0.05$). It is found that the near-infrared laser cannot polymerize the samples and TheraCal LC had greater mean values than Calciplus LC at 5, 10, and 20-s ($p < 0.001$, $p = 0.011$, and $p = 0.062$, respectively). Energy-dispersive X-ray spectroscopy and X-ray powder diffraction analysis were used to assess the calcium-silicate content. Calcium-silicate phases were observed in TheraCal LC but these phases did not exist in the Calciplus LC. TheraCal LC and Calciplus LC have similarities in their conversion degree properties with LED excitations at 20-s, however, the calcium-silicate content measured in the experiments is not in agreement with the data of the Calciplus LC declared by the manufacturer.

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KEYWORDS

Calcium-silicates; degree of conversion; near-infrared laser; polymerization; pulp capping material

1. Introduction

Biocompatible materials are frequently used in the management of deep caries of vital teeth [1]. For this purpose, hydraulic calcium silicate-based bioceramics are the first option in the exposed pulp [2]. Also, resin-based bioceramics could be preferred when the pulp is not exposed [1,2]. For resin-based biomaterials, calcium silicates are physically embedded into the methacrylate-based media without crosslinking [3]. The main strategy is aimed to enhance their stability and integration with restorative materials whereas, cause to command the curing time [4]. The first representative of the calcium-silicates-contained-resinous pulp capping material (RPC) is TheraCal LC

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(Bisco Inc., Schaumburg, IL). More specifically, the chemical composition of TheraCal LC consists of type III Portland Cement (calcium silicate source), radiopacifier agent, hydrophilic thickening agent, and bisphenol A-glycidyl methacrylate (BisGMA)-based resin media [5,6].

Recently, a urethane dimethacrylate (UDMA)-based RPC representative named ‘Calciplus LC’ has been introduced [7]. The product has affixed Conformité Européenne (CE) marking, however, very limited data is accessible on it. Notably, the literature-based content data seem not to overlap related to the manufacturer’s data. More specifically, its calcium silicate or calcium hydroxide content that appeared in the manufacture datasheet does not in agreement with a previous study (Please see the manufacturer data in the [Supplemental file S1](#)) [7]. In addition, there is no literature-based information about the Calciplus LC content and characterization research is not conducted for the RPC material.

Complete polymerization of a resin-based material is an ideal objective for the clinical success of restorations [8]. The degree of carbon double bond conversion or DC is related to the final mechanical properties of polymer-based materials where higher molecular weight provides higher stability and hardness [9,10]. The high DC corresponds to a resin-based material success since lower monomer–polymer conversion relates to low mechanical properties and increased risk of releasing toxic substances (residual monomers) from the material [11,12]. DC of the resin-based materials could be quite related to the adhesive type of failure between the dentin and bonding agent, cohesive type of failure within the dentin or resin composite, or some mixed type of failure modes [13]. Notably, increasing DC affects the hydrophobicity of the polymeric material which may reduce the wettability or adhesion ability of resinous material with the dentin. Also, higher DC will tend to higher contraction stresses [14]. Vice versa or relatively lower DC may increase the risk of incidence of the above-mentioned failure modes of a resinous material [15,16]. Hence, minimal polymerization shrinkage that is caused by cross-linking and polymerization should generally be an antagonistic goal to obtaining optimum DC [14].

Very recently, the polymerization degrees of TheraCal LC and the novel TheraCal PT (Bisco Inc., Schaumburg, IL) have been reported [17–19]. Yet, the polymerization degree has not been assessed specifically for the Calciplus LC as a counterpart that includes conflicting ingredients. The unique DC data is necessary to understand one of the significant physicochemical characteristics of RPCs. Despite some concerns available about the usage RPCs in direct pulp capping treatment [20–22], RPCs are frequently used under restorative materials and in indirect pulp capping treatments in day-to-day clinical practices [23]. For that reason, demonstrating the unknown physicochemical parameters and researching controversial issues for the RPCs are relevant.

A light excited from any source can transmit into the material or reflect from the surface. Also, the transmitted light can attenuate through the bulk of the material during the photoactivation of a material [24]. Hence, the visible light source at a ‘high energy level’ is used for the polymerization of light-curable dental materials in clinical practice. NIR spectral region lies approximately in the 700–1100 nm wavelength range [25]. The relatively high wavelength range of NIR spectra causes low

energy transfer to the living tissues [25]. Accordingly, NIR laser applications are increasing in healthcare due to minimizing the transferred energy on human tissues [26]. To eliminate the high energy transfer-related drawbacks of ionizing radiation, a diagnostic device in dentistry uses an NIR at 780–850 nm wavelength range to detect non-cavitated proximal caries [27]. Also, photoinitiation, radical polymerization, and proton generation have been shown with the NIR laser excitation [28,29]. Yet, the NIR laser excitation effect on the polymerization of resin-based materials has not been studied.

The present study is twofold. To eliminate the gaps in the literature, the aims of the present study were to measure the microstructure of the samples and to investigate the effect of excitation durations and different light sources on the ‘degree of conversion’ properties of RPC materials. The null hypothesis of this study is no difference between LED and NIR laser in the conversion degree of TheraCal LC and Calciplus LC.

2. Materials and methods

2.1. Materials and sample preparation

The information about the tested RPCs is given in Table 1. Each sample was prepared using a couple of potassium bromide (KBr) plates for the Fourier-transform infrared (FT-IR) spectroscopy experiments. The weight of each sample of FT-IR experiments is 1.2 mg. The samples were randomly divided into 10 subgroups according to the excitation modes ($n = 7$):

- Uncured sample.
- LED-light excitation for 5 s (LED-05).
- LED-light excitation for 10 s (LED-10).
- LED-light excitation for 20 s (LED-20).
- NIR excitation at 532 nm wavelength for 5 s (NIR_{532nm}-05).
- NIR excitation at 532 nm wavelength for 10 s (NIR_{532nm}-10).
- NIR excitation at 532 nm wavelength for 20 s (NIR_{532nm}-20).
- NIR excitation at 785 nm wavelength for 5 s (NIR_{785nm}-05).
- NIR excitation at 785 nm wavelength for 10 s (NIR_{785nm}-10).
- NIR excitation at 785 nm wavelength for 20 s (NIR_{785nm}-20).

The LED curing unit has a 385–515 nm wavelength range at 900 mW/cm² (D-Lux; DiaDent Group Int. Chungcheongbuk-do, Korea). The NIR laser has a 532–785 nm wavelength range (InVia instrument, Renishaw plc., Wotton-under-Edge, UK). The theoretical irradiances of NIR_{785nm} and NIR_{532nm} were at 100 and 1000 mW/cm², respectively. Each curing source was placed at a 1 mm distance from the sample surface and then activated per subgroup. Polymerized samples were immediately transferred to the FT-IR experiments. The DC was assessed with FT-IR spectroscopy and confirmed by Raman.

For the Raman, EDS, and XRD analysis, Teflon molds were used. A cylindrical Teflon mold (0.3 mm thickness, 7 mm inner diameter) was slightly overfilled with

Table 1. The information about the resin-based materials.

Material (manufacturers/form)	Tested material lot no.	Composition from literature	Safety data sheet summary ^a	Safety data sheet print-date	Curing time ^b (s)
TheraCal LC (Bisco Inc, Schaumburg, IL, USA/single flowable paste)	1900006974	<i>Uncured paste:</i> CaO, calcium silicate particles (type III Portland cement), Sr glass, fumed silica, barium sulfate, barium zirconate. <i>Resin matrix:</i> Bis-GMA. ^ψ <i>Uncured paste:</i> Active ingredients in MTA 44%, and barium sulfate 6%. <i>Resin matrix:</i> Aerosil [®] 8.0%, Biocompatible hydrophilic resin 42.5% (BisGMA 20%, biocompatible resin-FDA 77.25%, modifying agent 2.4%; initiating agent 0.32%, stabilizer for the initiating agent 0.032%). ^φ	30–50% Portland cement (CAS: 65997-15-1) 5–10% Bis-GMA (CAS: 1565-94-2) 1–5% barium zirconate (CAS: 12009-21-1)	19.01.2018	20 s ^Ω
Calciplus LC (Imicryl Inc. Konya, TURKEY/single flowable paste)	19109	<i>Resin Matrix:</i> UDMA, ultra-low shrinkage monomer, photo initiator, stabilizers. <i>Filler:</i> Ultra fine bioactive glass, fluoroaluminasilicate glass, antibacterial Nano composite filler. ^φ Calcium silicate and calcium hydroxide ^Ω	N/A	N/A	20 s ^Ω

BisGMA shows the bisphenol A-glycidyl methacrylate; UDMA shows the urethane dimethacrylate. CAS shows 'Chemical Abstracts Service Registry Number', N/A shows not applicable. ^ψThe information is derived from Gandolfi et al. [4].

^φThe information is derived from Korkut et al. [7].

^ΩThe information is derived from manufacturer data (please see Appendix A of Supplementary file S1).

^aMaterial description is presented as informed by the manufacturers; ^bmanufacturer recommendation for light curing.

the RPCs. To prevent oxygen inhibition, a Mylar strip was placed above the resin and flattened ($n = 3$).

2.2. FT-IR spectroscopy investigation

Spectral changes were detected by FT-IR spectroscopy. For this purpose, Spectrum two FT-IR Spectrometer with LiTaO₃ detector (Perkin Elmer Inc., Waltham, MA, USA) was used for recording spectral data with the potassium bromide (KBr) plate method. The spectral range of each record was 4000–400 cm⁻¹ with a spectral resolution of 4 cm⁻¹.

2.2.1. Theory/calculation

In the FT-IR spectral interpretation, the chemical shifts of aliphatic and aromatic CC groups were observed at 1637 and 1608 cm⁻¹, respectively. The degree of conversion is calculated using the ratio of peak intensities of the aliphatic (C=C) and aromatic (C··C) CC groups. The following Equation (1) is employed for the calculation of conversion degree:

$$\text{Degree of conversion (\%)} = \left(1 - \frac{\left(\frac{\text{Abs(C=C)}}{\text{Abs(C}\cdots\text{C)}} \right)_{\text{polymerized}}}{\left(\frac{\text{Abs(C=C)}}{\text{Abs(C}\cdots\text{C)}} \right)_{\text{unpolymerized}}} \right) \times 100 \quad (1)$$

2.2.2. Data analysis

Statistical comparisons of DC were performed only for the LED groups. Resinous materials and excitation durations factors were analyzed with the two-way ANOVA test and Tukey Post Hoc tests (The Jamovi project (2021), Jamovi (Version 1.6) (Computer Software). Retrieved from <https://www.jamovi.org>) ($\alpha = 0.05$). Due to polymerization did not occur, statistical analysis of NIR laser data was not made.

2.3. Raman spectroscopy investigation

Renishaw Raman instrument (Renishaw plc., Wotton-under-Edge, UK) was used for the experiments. Following the placement of each sample, an infrared monomer spectrum was obtained using 32 scan resolution for samples. The CCD detector of the spectrometer has a 1024 × 256 pixel resolution. Each spectrum ranged from 400 to 4000 cm⁻¹ with a resolution of 4 cm⁻¹. The calibration of the wavenumber axis was conducted by a silicon calibration wafer. Spectra were recorded with a single scan, 10 s exposure for each sample. The spectral data were post-processed using the Wire software (Renishaw plc., Wotton-under-Edge, UK).

2.4. Microstructure analysis

Characterization of the chemical content was assessed together with EDS and XRD analysis. In EDS analysis, UltimExtreme instrument (Oxford Instruments, High

Wycombe, UK) coupled with a field emission scanning electron microscope (Hitachi Regulus 8,230; Hitachi High-Tech Co., Tokyo, Japan) was used. Teflon cylindrical molds were prepared for sample standardization for the EDS analysis. The dimension of each mold is 2 mm in diameter and 1 mm in thickness. A Mylar strip was placed on the bottom of the mold to allow specimen filling. Before the EDS examination, each mold filled with the sample was attached to metal stubs using carbon tape without coating application. The EDS data of the uncured sample was the reference. Accordingly, carbon, oxygen, fluorine, calcium, silicon, zirconium, barium, and ytterbium were assigned for analysis. Trace elements of specimens were not considered.

Due to the inhomogeneous nature of these RPC biomaterials, the data were collected from the central region of the image surface (area selection tool, at 1000 \times) at high-magnification mode. Each EDS data was obtained from a single reading for each sample ($n = 3$). In total, the average of the triplet EDS spectra of each LED subgroup was considered for the calculation of the elemental percentage. The accelerating voltage was 10.0 kV at high-vacuum pressure.

In XRD analysis, three specimens per group were calcined at 500 °C for 5 h ($n = 3$, per each RPC). The rest of the specimens were not calcined ($n = 3$, per each RPC). Empyrean diffractometer (Malvern Panalytical B. V., Malvern, UK) equipped with the PIXcel1D detector (Malvern Panalytical B. V., Malvern, UK) was used in the analysis. The diffractometer emits copper alpha radiation at 40 mA and 45 kV, and the detector rotates between 5° and 90° at 2θ . Each step corresponds to 0.016° with a 10 s scan rate. Each diffractogram was matched with the 'International Centre for Diffraction Database' (ICDD) using HighScorePlus v4.6a software (Malvern Panalytical B.V., Malvern, UK).

3. Results

3.1. Interpretation of spectral data

Vibrational modes of FT-IR spectra are listed in Table 2. In FT-IR spectra, the peaks observed at 1608 and 1637 cm^{-1} belong to CC groups for TheraCal and Calciplus. Regarding increased the duration of LED excitation, a significant decrease in their intensity was gradually observed in the vibration band belonging to the CC with no

Table 2. Vibrational modes of Fourier-transform infrared spectra of resin-based pulp capping materials.

Group	Characteristic vibrational modes	Observed wavenumbers ranges (cm^{-1})	Interpretation
TheraCal LC	CC	1608 and 1637ab	Strong
	CO	1718 ^a	Strong
	CH ₃	2873 ^a	Strong
	CH ₂	2949 ^a	Strong
Calciplus LC	CC	1608 and 1637 ^a	Weak
	CO	1720 ^a	Strong
	CH ₃	2957 ^a	Weak
	CH ₂	2974 ^a	Strong

^aShows 'the intensities of these bands changed with light emitted diode excitation with no chemical shift'; ^bshows 'the intensities of the aromatic and aliphatic CC vibrational modes slightly changed with the near-infrared laser excitation at 532 nm wavelength'.

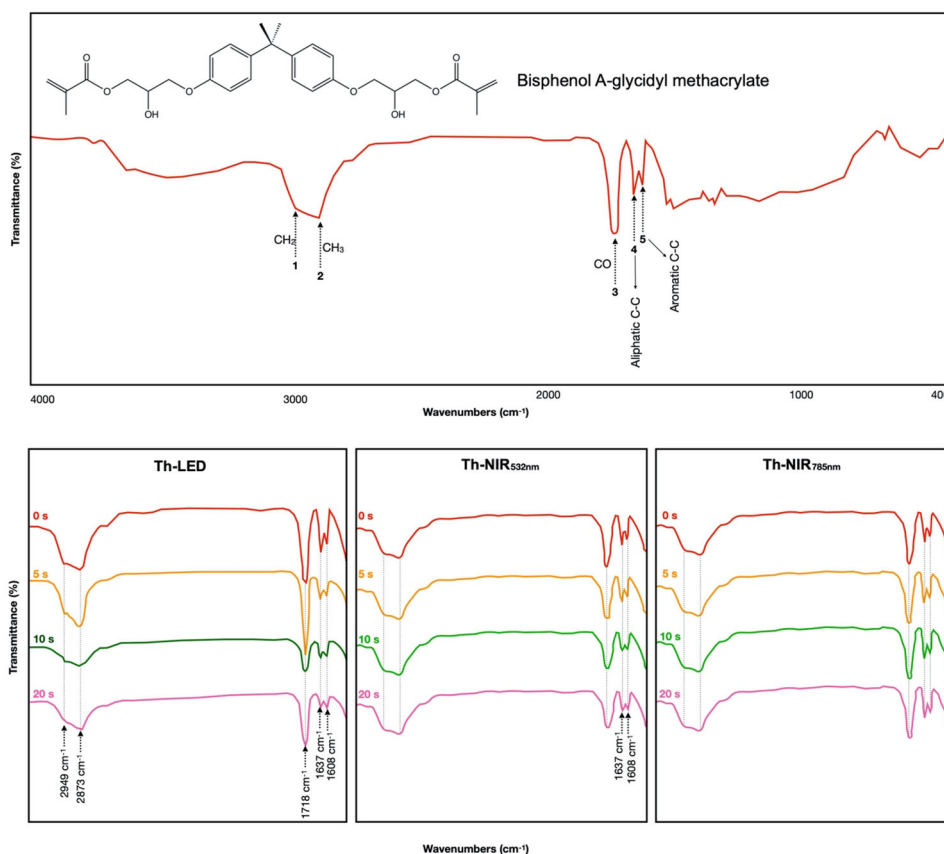


Figure 1. Representative FT-IR spectra of TheraCal. Th shows TheraCal LC. LED shows light-emitting diode excitation. NIR_{532nm} shows near-infrared laser excitation at 532 nm wavelength, NIR_{785nm} shows near-infrared laser excitation at 785 nm wavelength.

chemical shift for both RPC-LED groups. In TheraCal-LED groups, CO, CH₃, and CH₂ vibrational modes were seen at 1718, 2873, and 2949 cm⁻¹, respectively. The intensities of these bands changed with LED excitation with no chemical shift. The intensities of the aromatic and aliphatic CC vibrational modes slightly changed with the TheraCal-NIR_{532nm} excitation with no chemical shift (Figure 1). There was no significant chemical shift or intensity transition observed in the vibration motions belonging to the identified groups with NIR_{785nm} excitation. In Calciplus-LED groups, CO, CH₃, and CH₂ vibrational modes were seen at 1720, 2957, and 2974 cm⁻¹, respectively. The intensities of these bands changed with LED excitation with no chemical shift. There was no significant chemical shift or intensity transition observed in the vibration motions belonging to the identified groups with NIR excitation (Figure 2).

In Raman spectral interpretations, vibrational modes of aliphatic and aromatic CC groups were seen at 1637 and 1608 cm⁻¹, respectively. These findings were in agreement with the FT-IR results (Figure 3).

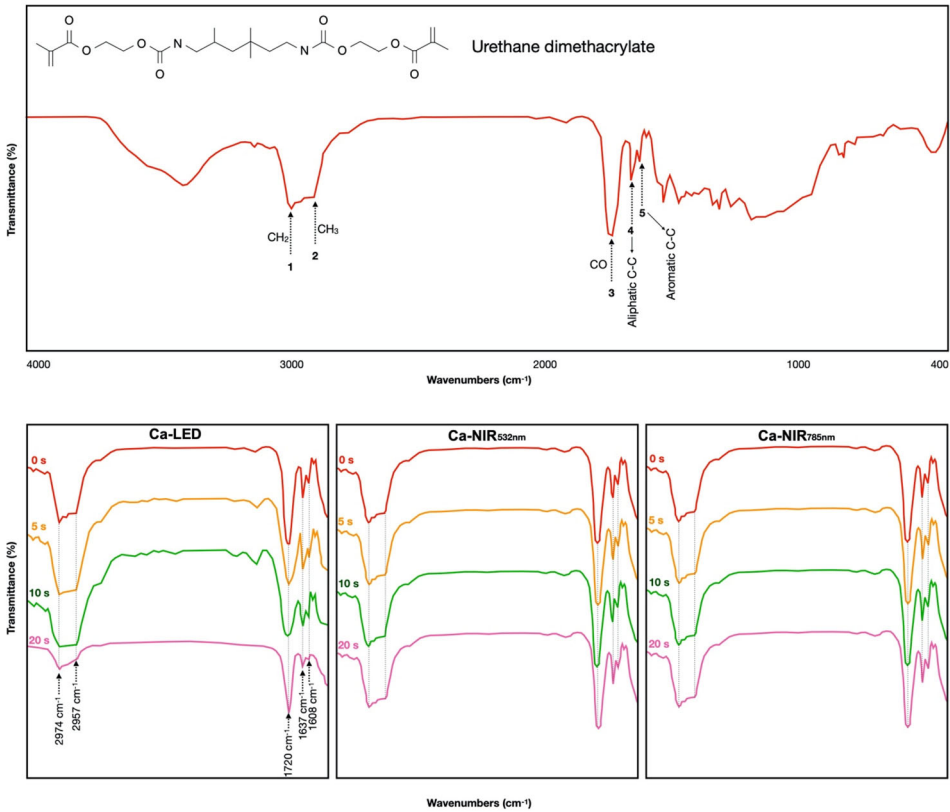


Figure 2. Representative FT-IR spectra of Calcipus. Ca shows Calcipus LC. LED shows light-emitting diode excitation. NIR_{532nm} shows near-infrared laser excitation at 532 nm wavelength, NIR_{785nm} shows near-infrared laser excitation at 785 nm wavelength.

3.2. Degree of conversion findings

The research data that support the findings of this study are provided by Dikmen et al. [30]. The presentation of the statistical analysis of the DC% is summarized in Figure 4. Significant differences were revealed among the time subgroups curing times for both RPC biomaterials ($p < 0.001$). These differences were gradually decreased with increased curing time. TheraCal had the highest DC% means at 5, 10, and 20 s ($p < 0.001$, $p = 0.011$, and $p = 0.062$, respectively). According to the manufacturers' data, 20 s light curing is recommended in both RPC biomaterials. The DC% means of TheraCal-LED-20 and Calcipus-PC-LED-20 were 78.5% and 75.0%, respectively ($p = 0.062$).

DC was calculated between 11% and 16% for TheraCal-NIR_{532nm} samples at 20 s excitation, however, the authors considered that the low conversion degree had no clinical significance, therefore, statistical analysis was not made for NIR laser samples.

3.3. Calcium-silicate content

A noteworthy observation was the inconsistent EDS data between TheraCal and Calcipus regarding their calcium and silicon contents. In the descriptive EDS data,

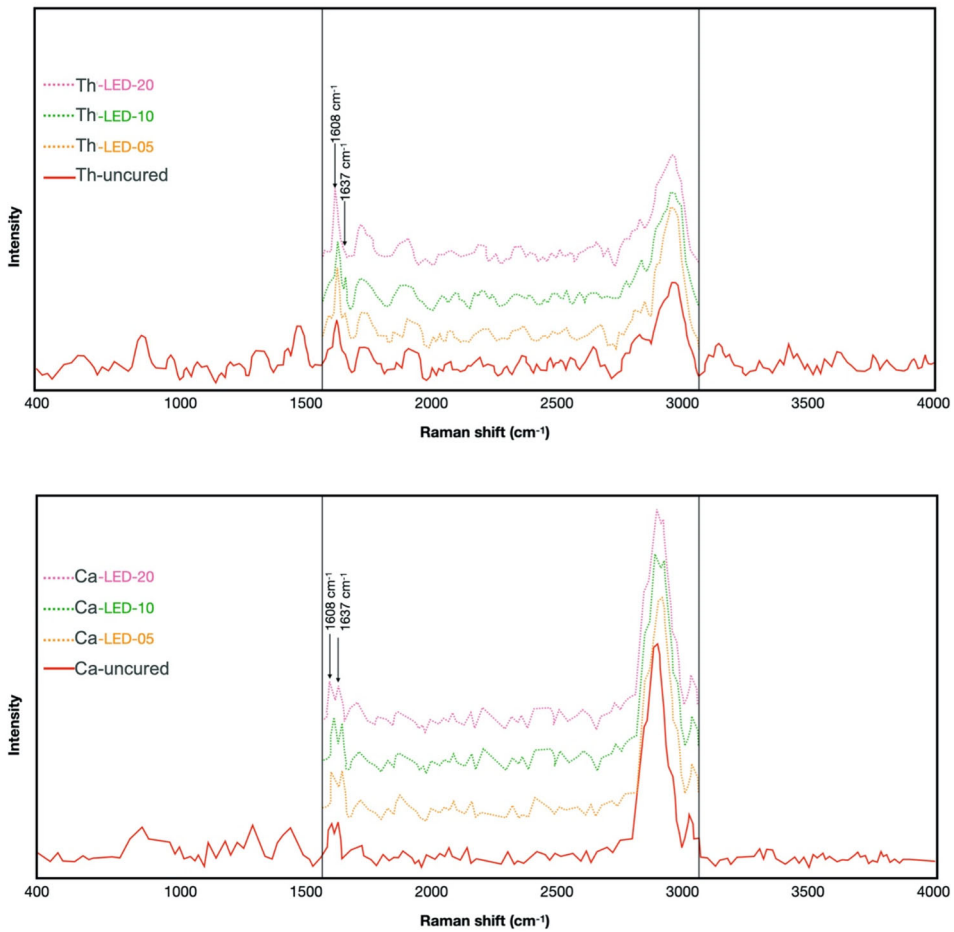


Figure 3. Representative Raman spectra of TheraCal-LED and Calciplus-LED. Th shows TheraCal, Ca shows Calciplus. LED shows light-emitting diode excitation at 5 s (LED-05), 10 s (LED-10), and 20 s (LED-20).

the calcium content was not detected in the Calciplus whereas, the silicon content of Calciplus was relatively lower than TheraCal. EDS revealed that the percentage of calcium and silicon content increased with the prolonged photo-activation in TheraCal (Figure 5).

In XRD analysis, halo peaks were revealed between 5 and 15° in the uncalcined specimens due to the existence of the organic phases. Tricalcium silicate ($76.7 \pm 0.4\%$; ICDD: 00-055-0740; monoclinic), dicalcium silicate ($6.8 \pm 0.3\%$; ICDD: 98-024-5079; monoclinic), and barium-zirconium-oxide phase ($16.6 \pm 0.7\%$; ICDD: 04-006-0973; cubic) were detected in TheraCal. Whereas, ytterbium fluoride ($77.4 \pm 1.1\%$; ICDD: 04-010-148; orthorhombic), calcium fluoride ($11.7 \pm 0.5\%$; ICDD: 01-088-2301; cubic), silicon dioxide ($9.6 \pm 0.5\%$; ICDD: 01-076-0912; tetragonal) and zirconium oxide ($1.3\% \pm 0.1$; ICDD: 04-016-2969; monoclinic) were detected in Calciplus (Figure 6).

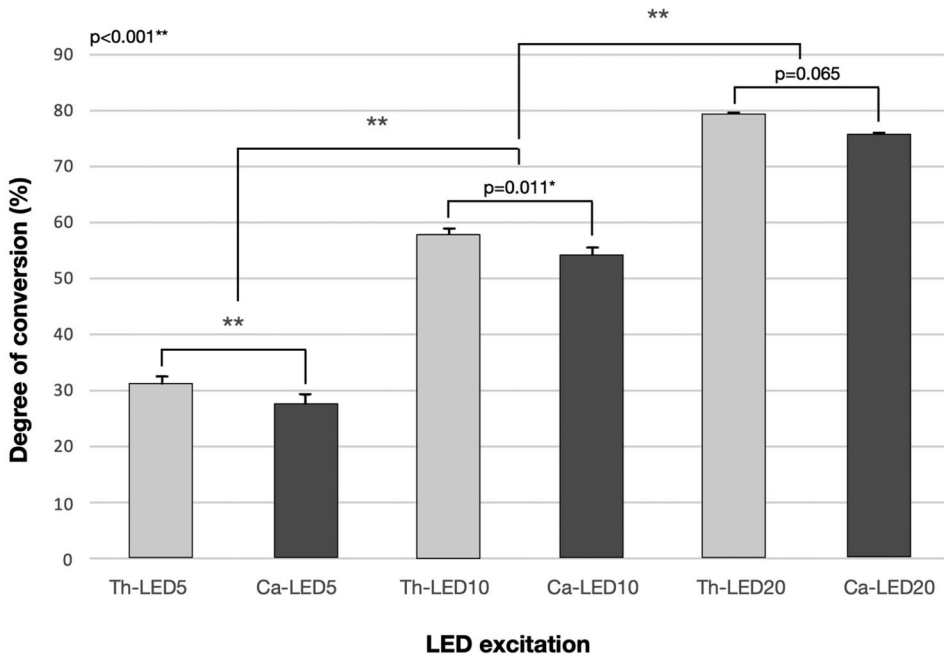


Figure 4. The bar graph representing mean and standard deviation percentages of degree of conversion of TheraCal and Calciplus. Th shows TheraCal, Ca shows Calciplus. LED shows light-emitting diode excitation at 5 s (LED-05), 10 s (LED-10), and 20 s (LED-20).

4. Discussion

The increasing conversion degree of each sample with increasing excitation duration could be explained by the positive relationship between the amount of transferred energy increased over time, as expected. Despite the manufacturers having recommended a 20-s curing duration, the results indicated that the polymerization did not complete. Therefore, the null hypothesis was rejected. Previous studies demonstrated that the unpolymerized monomers or resin parts could be toxic to pulp cells, while polymerized resins presented no toxic effects [11,12]. In addition, RPC materials have been shown to be more cytotoxic to murine odontoblasts cells and human dental pulp stem cells than resin-free calcium silicates (e.g. powder form of calcium silicates) [31,32]. Giraud et al. [21,22] have discussed the drawbacks of the RPC with the chemical content of the materials. Accordingly, it has been stated that the resinous structure freezing the calcium-silicate content in the resin matrix led to insufficient interaction with the pulp–dentin complex, and therefore, RPCs should not be placed in direct contact with the pulp tissue [21,22]. The presence of residual uncured or unconverted monomer demonstrated in the present study was in agreement with the cytotoxicity of RPC materials shown in the previous studies [21,22,31,32].

The final physical properties of a resin-based material may influence clinical success [33]. Accordingly, an inferior monomer–polymer conversion can correlate to decreased mechanical stability and increased toxicity, ultimately leading to postoperative failures in the short term [34–36]. The presented incomplete polymerization findings at 20 s were in agreement with the reported DC values of flowable dental

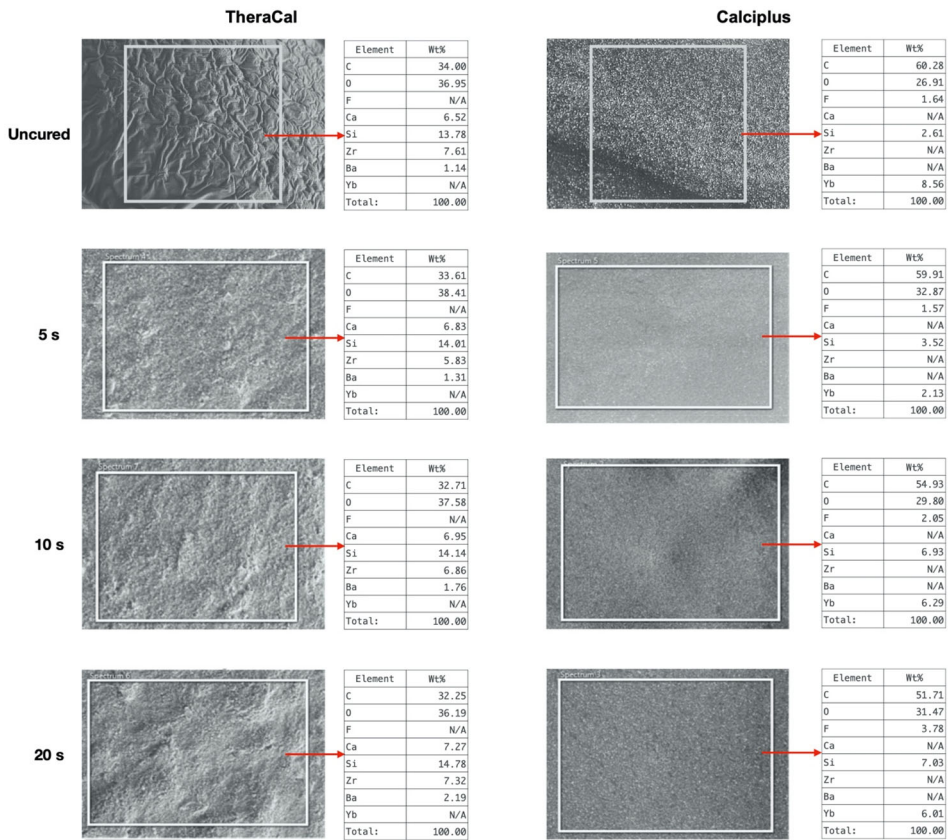


Figure 5. Mean EDS data of TheraCal and Calciplus.

composites (57.9–68.9%) [37]. The differences between our findings and the previous studies could be attributed to the light unit source or resin-based material differences.

The XRD findings confirmed the EDS findings corresponding to each RPC. The presence of calcium-silicate content and phase composition of TheraCal were in agreement with the previous reports [20,38]. However, in the findings, the manufacturer’s declared calcium silicate or calcium hydroxide content was not detected in the analyses of Calciplus. Because of these findings, the authors figured that it is a matter of debate whether the group representative is an RPC material or not. These content results, which contradict information from manufacturer-based data, indicate that ‘Calciplus LC’ is not calcium hydroxide or calcium-silicate-based pulp capping material. The mechanism of action of calcium silicates is similar to that of calcium hydroxides due to calcium hydroxide is a by-product of the hydration reaction of calcium silicates with water [39]. Calcium silicates and calcium hydroxide produce dentin bridge formations by modulating the inflammatory response of pulp tissue, due to their ability to stimulate bone morphogenetic protein production and therefore reparative dentinogenesis [40,41]. Consequently, it has been proven that calcium silicate- or calcium hydroxide-based reactions, which has been demonstrated their positive effects on the pulp–dentin complex, will not be presented by the Calciplus LC. Besides, the detected inorganic compositional differences might have contributed to

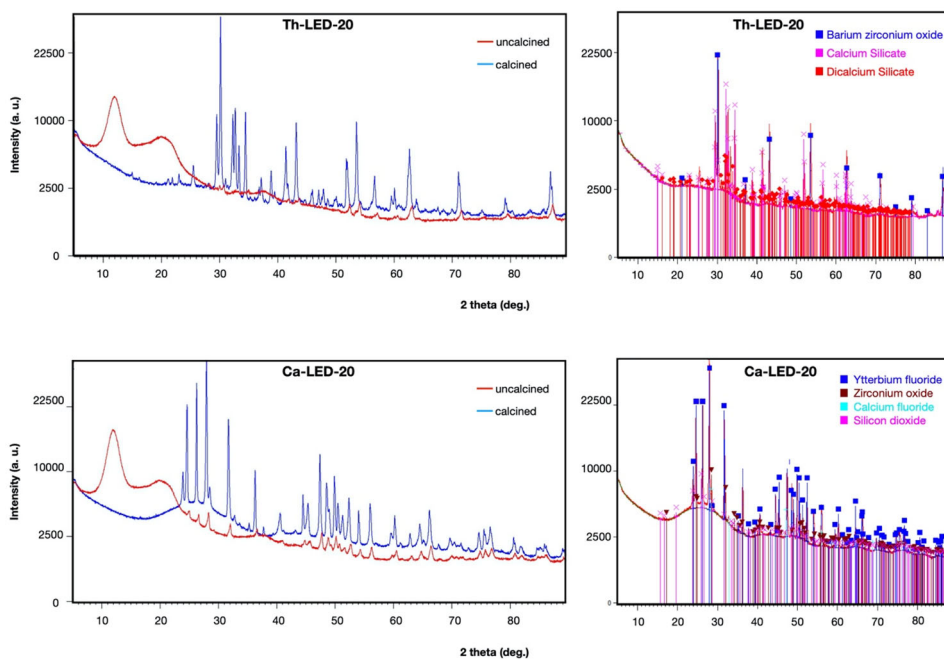


Figure 6. Representative diffractogram of resin-based pulp capping materials. Th shows TheraCal, Ca shows Calciplus. LED shows light-emitting diode excitation at 20 s (LED-20).

the variety in DC findings between the materials. DC of a resin-based material is influenced by not only the resin matrix type but also the type, shape, and size of the inorganic filler [42].

The NIR laser excitation was also studied on TheraCal and Calciplus LC. However, polymerization did not occur in any samples after NIR_{785nm} excitations. The intensities of the aromatic and aliphatic CC vibrational modes slightly changed with NIR_{532nm} laser excitation in TheraCal. The NIR laser at 532 nm wavelength has a higher amount of energy compared to the 785 nm wavelength. The observed findings in the NIR laser at the 532 nm wavelength may explain by the transferred high amount of energy to the BisGMA media.

To assess the DC of resinous materials, various thickness designs could be used in the sample preparation. In agreement with a previous study [43], to achieve thinner samples, KBr plates were used under pressure in the present study. The literature-based depth of cure value of TheraCal LC is 1.7 mm [4]. The range of distances between the curing light and the specimens was reported to be set at 1 mm [19], 2 mm [17], and 3 mm [18] in the previous studies. FT-IR experiments were conducted with coupled micro-attenuated total reflectance crystal (ATR) technique in the previous studies [17–19]. Furthermore, the presented theoretical calculations of DC% have variety in the previous studies [17,18]. Hence, FT-IR experimental methodology, theoretical calculation, curing unit type, or curing distance could affect the variety of the DC% results of TheraCal LC. It is challenging to directly correlate *in vitro* conditions to clinical situations. Especially, the simulation of environmental conditions cannot be possible such as various cavity depths. In the study, all curing modes were

performed at a 1 mm distance from the sample surface. Thus, this could be considered a limitation of this *in vitro* study. A light source transfers a relatively low amount of energy to TheraCal and Calciplus LC in deep cavities compared to a 1 mm distance. This could be contributing to a decrease in the DC value of TheraCal and Calciplus LC.

Light-curable RPCs have the significant benefits of ease of placement, command curing, excellent physical strength, less solubility, and reduced heavy metal release [23]. The hydrophilic polymer matrix of the light-curable RPCs allows the release of calcium and hydroxide ions [23]. Despite RPCs being promising materials for pulp capping treatments, further studies need to understand their actual characterization and functions. Further studies are needed to investigate changes in the mechanical properties of the TheraCal correlating with the DC. Also, further studies are needed to obtain preclinical and clinical data on the Calciplus LC.

Within the limitations of this current study, it was concluded that biocompatible NIR laser excitation at 532 nor 785 nm cannot be utilized for the polymerization of resin-based materials. Despite the resin-based pulp capping materials present good conversion degree properties after LED excitations at 20-s, uncured monomers, which may contribute to their toxicity, can exist. The calcium-silicate content measured in the experiments is not in agreement with the data of the Calciplus LC declared by the manufacturer.

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Authors' contributions

Gokhan Dikmen: Writing – review & editing, Methodology, Investigation, Formal analysis, Supervision, Data curation. Okan Uslu: Writing – review & editing, Formal analysis, Methodology, Investigation, Conceptualization. Ekim Onur ORHAN: Writing – review & editing, Writing – original draft, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization.

Disclosure statement

The authors report there are no competing interests to declare. Also, the authors have stated explicitly that there are no conflicts of interest or financial interest in connection with resin-based pulp capping material manufacturing or dental material-related products.

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Data availability statement

The data that support the findings of this study are openly available in Mendeley data v1, 2022 (<https://doi.org/10.17632/kg7vhhx8n9.1>) [30].

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