VER
Vascular & Endovascular Review

Evaluating the Depth of Cure under Chemical and Dual Cure Modes in Novel Biosustainable Dual Cure Flowable Composites Reinforced with Rice Husk Nanohybrid Silica

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ABSTRACT

Dual-cure composites are designed to achieve increased depth of cure through the synergistic action of both chemical and light-activated initiator systems. Due to these enhanced curing properties, they are widely used as core buildup and luting agents. The present study aimed to evaluate and compare the depth of cure of newly formulated dual-cure flowable composites prepared with varying ratios of base and diluent monomers. The evaluation was conducted under two curing conditions: light curing (dual-curing mode) and without light curing (chemical curing mode). The newly formulated dual-cure flowable composites incorporated nanohybrid silica derived from rice husk as the filler material. The experimental groups consisted of two formulations with bisphenol A-glycidyl methacrylate (Bis-GMA) to triethylene glycol dimethacrylate (TEGDMA) ratios of 50:50 and 30:70, two groups with urethane dimethacrylate (UDMA) to TEGDMA ratios of 50:50 and 30:70, and one group combining Bis-GMA, UDMA, and TEGDMA in a 25:25:50 ratio. Paracore Slow was utilized as the control material. Depth of cure was assessed employing the ISO 4049 scraping method. Statistical analysis of the results was performed using one-way analysis of variance (ANOVA) followed by Tukey's post hoc test for multiple comparisons. All formulations containing urethane dimethacrylate (UDMA) exhibited higher mean depth of cure values. The group with a UDMA to TEGDMA ratio of 30:70 demonstrated the greatest depth of cure among all tested groups. Moreover, every group exceeded the minimum depth of cure threshold established by ISO standards. Notably, depth of cure measurements were consistently higher under chemical curing conditions across all groups. In conclusion, the enhanced depth of cure observed in the novel DCFCs supports their potential application as sustainable dental restorative materials, contingent upon further comprehensive evaluation.

KEYWORDS: Depth of cure, dual cure, core build up, nanohybrid silica fillers, rice husk, biosustainable.

How to Cite: Zartashia Arooj, Yanti Johari, Dasmawati Mohamad, Matheel AL-Rawas, Abdalbseet A Fatalla, Adam Husien (2025) Evaluating the Depth of Cure under Chemical and Dual Cure Modes in Novel Biosustainable Dual Cure Flowable Composites Reinforced with Rice Husk Nanohybrid Silica, Vascular and Endovascular Review, Vol.8, No.15s, 61-67

INTRODUCTION

Resin-based composites (RBCs) are commonly chosen for direct restorations because they offer favorable physical properties along with superior esthetic appeal (Alzahrani et al., 2023). The depth of cure refers to the thickness of a light-cured resin that can be effectively converted from monomer to polymer when exposed to a light source under defined conditions. Increment thickness of 2 mm is the gold standard for composite placement and curing (Anand Yokesh et al., 2017). Light-cured composite resins can be polymerized only up to a specific depth, determined by the extent to which visible light penetrates the composite material (Rezaei et al., 2020). The limited depth of cure in light-cured RBCs is mainly due to light attenuation from absorption by monomers and photoinitiators, along with scattering and refraction at filler-matrix interfaces, which together reduce effective polymerization depth (Alzahrani et al., 2023). Depth of cure can be improved by increasing material translucency, raising photoinitiator levels, or employing multiple photoinitiator systems. Enhanced translucency facilitates deeper light transmission, while optimized photoinitiator concentration and synergistic photoinitiator combinations boost radical generation, collectively enhancing polymerization efficiency at greater depths (Stansbury, 2000).

By integrating both photo and chemical curing initiators, dual-cure restorative materials are designed to address and mitigate the limitations imposed by restricted depth of cure. Many studies concluded the improved depth of cure of dual cure composites due to chemical curing initiator reaching upto 6mm (Alzahrani et al., 2023; Thomaidis et al., 2024; Vandewalker et al., n.d.).

Silica is one of the most common fillers to enhance the properties of resin based composites and is produced both naturally and synthetically. Silica derived locally from rice husk has shown significant potential as a filler material in the formulation of resin composites overall with particularly promising applications in flowable composite systems (Azlisham et al., 2025, 2024, 2023; Johari et al., 2018; Lin et al., 2021; Noushad et al., 2016; Yusoff et al., 2019; Zulkifli et al., 2013). The properties of composite systems are significantly influenced by multiple factors such as filler weight fraction, morphology, particle size, alignment, and distribution within the resin matrix. Achieving optimal physical, mechanical, and biological outcomes relies heavily on the formation of robust interfacial bonds between the fillers and the resin matrix (Aminoroaya et al., 2021).

The three predominant monomers utilized in dental resin composites are Bis-GMA, UDMA, and TEGDMA. Bis-GMA and UDMA serve as primary base monomers, while TEGDMA functions as a diluent to optimize viscosity for effective filler dispersion. Compared to Bis-GMA, UDMA presents several benefits including greater molecular flexibility, reduced viscosity , and a refractive index more closely matched to silica (Aminoroaya et al., 2021; Szczesio-Wlodarczyk et al., 2021).

Currently, no studies have reported the fabrication of dual-cure composites incorporating biosustainable nanohybrid silica derived from rice husk. Therefore, this study aims to evaluate the depth of cure of these novel dual-cure flowable composites (DCFCs) under both chemical and dual-curing conditions. The development of biosustainable DCFCs with optimal cure depth is crucial to ensure their effectiveness as restorative materials and to improve their long-term clinical durability and performance.The null hypothesis of our study was 1) no significant difference exists in depth of cure among DCFCs with different monomer ratios 2) no significant difference exists in depth of cure under dual and chemical curing.

MATERIALS AND METHODS

Sample size calculation: The sample size for each group (n = 5) was determined using Power and Precision software (version 3.0), based on a 95% confidence level, 80% statistical power, and an anticipated 10% dropout rate. The study involved the development of biosustainable DCFCs incorporating silica derived from rice husk as the inorganic filler. A total of five experimental groups were formulated, as outlined in Table 1.

Bisphenol A-glycidyl methacrylate (Bis-GMA) and urethane dimethacrylate (UDMA) were employed as the primary base monomers, while triethylene glycol dimethacrylate (TEGDMA) was used as the reactive diluent. The groups were categorized based on variations in the base monomer-to-diluent ratio, while maintaining a constant monomer-to-filler ratio across all formulations. This variation in monomer composition was designed to investigate the influence of resin matrix composition on the depth of cure of the dual-cure composites.Group distribution novel DCFCs final composition of dual cure composites with varying ratios of groups are depicted in Figure 1.

Table 1: Composition of novel DCFCs and commercial product

Experimental DCFCs	Monomers		Filler	
	Type	Loading (wt%)	Type	Loading (wt%)
AU5T5	UDMA: TEGDMA	50:50	Nanohybrid silica	50
BU30T7	UDMA: TEGDMA	30:70		
CB2U2T5	BisGMA:UDMA:TEGDMA	25:25:50		
DB3T7	BisGMA:TEGDMA	30:70		
EB50T50	BisGMA:TEGDMA	50:50		
FPS (Paracore slow)	Bis-GMA, UDMA, TEGDMA	NA	Barium glass, amorphous silica	74

UDMA: urethane dimethacrylate TEGDMA: Triethylene glycol dimethacrylate BisGMA: bisphenol A-glycidyl methacrylate NA: not applicable

Fabrication of novel dual cure flowable composites

The filler-to-monomer ratio of the novel DCFCs was fixed at 50:50, as this proportion provided optimal flow characteristics comparable to commercial products. These composites were formulated using locally sourced nanohybrid silica (Patent ID: MY-187327-A). Bisphenol A-glycidyl methacrylate (Bis-GMA; Sigma Aldrich) and urethane dimethacrylate (UDMA; Sigma Aldrich) were employed as the primary base monomers, while triethylene glycol dimethacrylate (TEGDMA; Sigma Aldrich) functioned as the diluent monomer. Experimental compositions included two Bis-GMA to TEGDMA ratios of 50:50 and 30:70, two UDMA to TEGDMA ratios of 50:50 and 30:70, and a ternary blend of Bis-GMA, UDMA, and TEGDMA at 25:25:50. Paracore Slow served as the control material.

The novel DCFCs was prepared as two distinct components: a light-activated resin and a chemically initiated resin. Both photoinitiators and chemical initiators were incorporated to enhance curing depth. The light-activated component contained camphorquinone (CQ), dimethylaminoethyl methacrylate (DMAEMA), and a tertiary amine catalyst to facilitate photo-polymerization. The chemically cured component was formulated with benzoyl peroxide as the initiator to promote chemical polymerization. Nanohybrid silica filler was incrementally added and homogeneously dispersed through thorough mixing. Final composite specimens were produced by manually blending equal volumes of the light-curing and chemically curing components prior to characterization. Figure 2 shows light and chemical curing components of novel DCFCs.

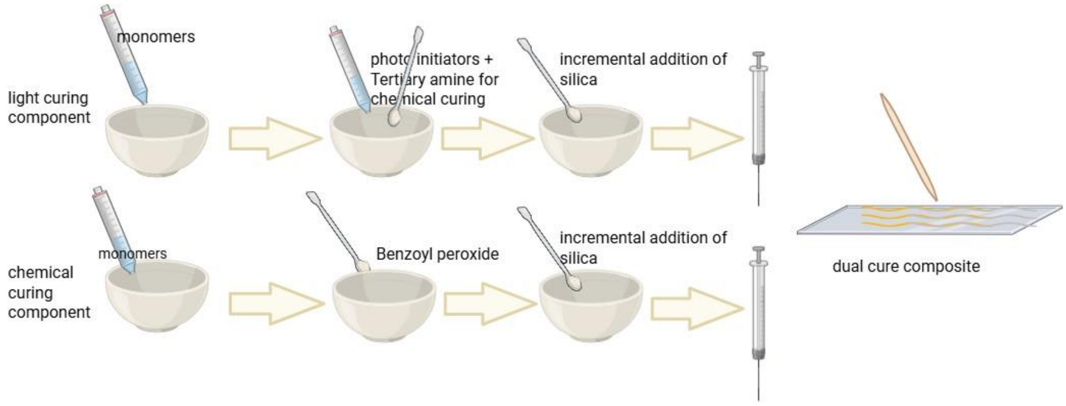

Fig 1: Fabrication of experimental dual cure flowable composites

Fig 2: Light curing (yellow) and chemical curing(white) component of DCFCs

For sample preparation equal quantities of both components were measured using analytical balance (Joanlab, China) and were mixed on glass slab. Manufacturer details of the materials are given in Table.

Measuring the depth of cure:
The depth of cure for the novel DCFCs was evaluated utilizing the scraping technique as outlined in ISO 4049 (*Dentistry : polymer-based filling, restorative and luting materials*, 2000). A customized silicone mold, measuring 14 mm in length and 4 mm in diameter, was fabricated for specimen preparation. Equal masses of the two composite components were precisely weighed using an analytical balance (Joanlab, China) and manually blended on a glass slab. The homogenized mixture was then introduced into the mold, with the top surface sealed by a transparent mylar strip (Kem Dent). Specimens were categorized into two subgroups to assess curing under chemical self-curing and light-activated conditions.

One subgroup underwent self-curing by being left undisturbed for eight minutes. Subsequently, samples were extracted from the mold, and any unpolymerized material was removed via scraping with a wooden spatula. The length of the polymerized specimen was accurately measured with a digital caliper (Mitutoyo, Japan). The final reported depth of cure was calculated by halving the measured specimen height, in accordance with the procedures defined by the ISO 4049 standard.

Specimens in the light curing groups were subjected to irradiation using a LED light-curing unit (Blue Dent LED Smart, Bulgaria) operating at an intensity of 1300 mW/cm² for a duration of 20 seconds. Immediately following light exposure, the samples were removed from the mould, and any uncured material was carefully scraped away using a wooden spatula. The length of the polymerized specimen was then measured precisely with a digital caliper (Mitutoyo, Japan). Consistent with ISO 4049 guidelines, the final depth of cure was determined by halving the measured specimen height.

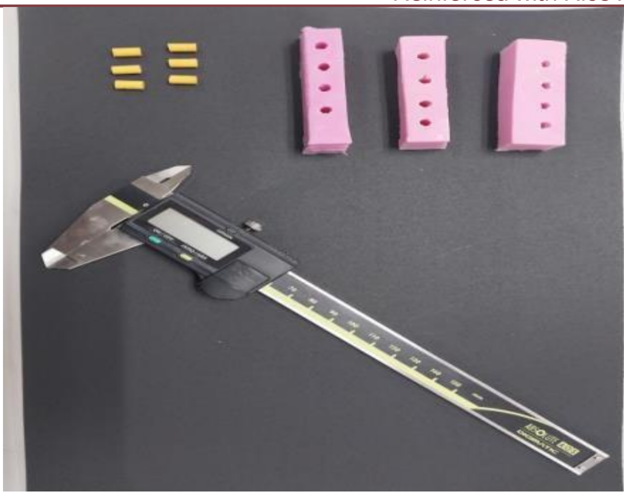

Figure 3: Silicon mould, Vernier Calliper **Figure 4: Measuring the sample with Vernier Calliper**

RESULTS

The data presented in the table illustrates the depth of cure for the novel dual-cure flowable composites (DCFCs) when cured in dual mode. The commercial reference material exhibited the greatest mean curing depth of 6.05 mm. Experimental formulations BU3T7 and CB2U2T5 achieved curing depths statistically comparable to the commercial product, with mean values of 5.91 mm and 5.83 mm, respectively ($p > 0.05$). In contrast, AU5T5, DB3T7, and EB5T5 demonstrated a sequential decrease in depth of cure, presenting mean values of 5.47 mm, 5.09 mm, and 4.97 mm, respectively. Among these, EB5T5 showed the most limited curing potential, with a depth of cure significantly lower than that of FPS and all other tested materials ($p < 0.05$).

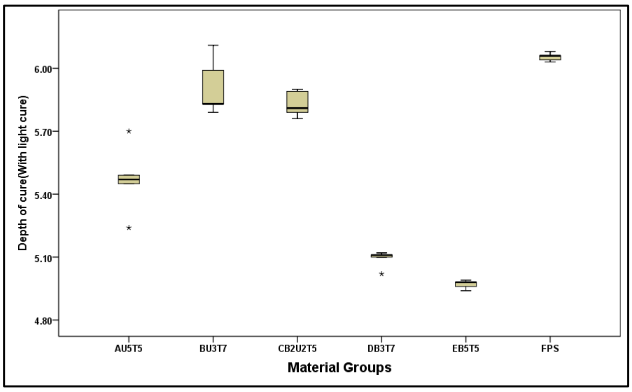

Figure 5: Box and Whisker plots for depth of cure of study groups (dual curing mode)

The values for the depth of cure of novel DCFCs in chemical curing mode are shown in table 2. The maximum depth of cure in chemical curing mode was 6.24 mm for BU3T7, which is similar to the 6.29 mm measured for the commercial group, FPS. The values of depth of cure of CB2U2T5 and DB3T7 follow the values of BU3T7 and are lower when compared to commercial FPS but both have statistically significant values. Lowest values of depth of cure was observed in EB5T5 that is 5.43mm. Figure 6 represents of the depth of cure using box and whisker plots.

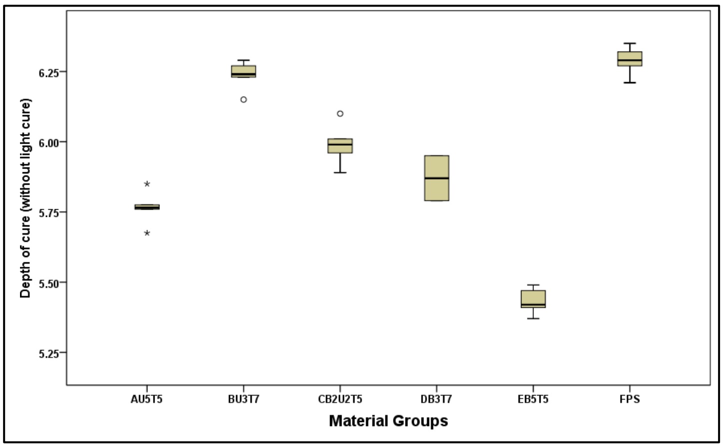

Figure 6: Box and whisker plot of depth of cure of study groups in chemical curing mode

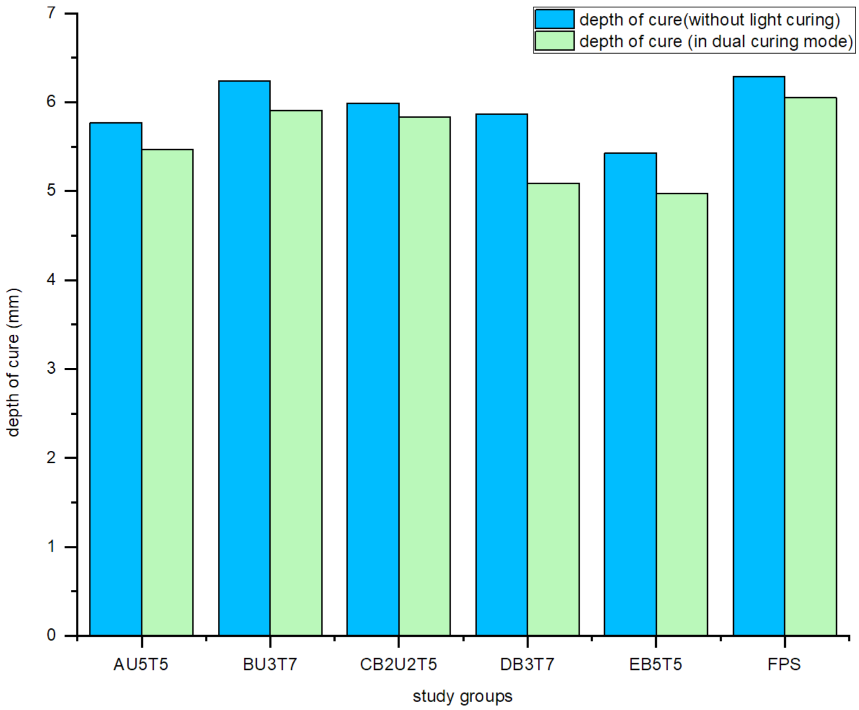

Figure 7: Comparison of depth of cure in chemical curing and in dual curing mode

Material	**Mean Depth of Cure in chemical curing mode(mm)**	**Mean Depth of Cure in Dual curing mode (mm)**
AU5T5	5.77±0.062[c]	5.47±0.163[a]
BU3T7	6.24±0.054[a]	5.91±0.136[ab]
CB2U2T5	5.99±0.076 [a, b]	5.83±0.062[ab]
DB3T7	5.87±0.080[b]	5.09±0.041[b]
EB5T5	5.43±0.048[d]	4.97±0.020[c]
FPS	6.29±0.053a	6.05±0.019[a]

Table 2 : Mean values ± standard deviations in parentheses. The same superscript letters in a column denote no statistical significance according to (p>0.05)

DISCUSSION

The present investigation incorporates an auxiliary photoinitiator within the formulation, thereby synergistically combining the benefits of both light-activated and chemically initiated polymerization mechanisms. This approach significantly improves the overall polymerization depth of the composite material. The performance of the dual photoinitiator system was systematically evaluated using the standardized ISO depth of cure method (*Dentistry : polymer-based filling, restorative and luting materials*, 2000). Each newly formulated dual cure flowable composite group was divided into two groups. One group was allowed to set through chemical curing mechanism and other was exposed to light curing unit and hence polymerized through dual curing mechanism. The dual-cure system leverages rapid surface polymerization triggered by light, while the embedded chemical initiator sustains ongoing polymerization in subsurface layers. Consistent with prior research findings, this integrated curing mechanism yields a more homogeneous and comprehensive polymer network throughout the composite structure (Wang and Wang, 2020).

In summary, all experimental groups, under both self-curing and photocuring (dual-cure) conditions, demonstrated substantial depths of cure ranging between 4.97 and 6.24 mm. These results are consistent with prior investigations reporting similar polymerization depths for bulk-fill resin-based composites. The findings confirm the effectiveness of the dual-curing system in facilitating adequate polymerization and achieving clinically relevant curing depths (Vandewalker et al., n.d.; Windle et al., 2022). Dual-cure composites can reach significant curing depths of 6 mm or greater, surpassing the constraints of light cure technologies on their own (Wang and Wang, 2020). The analysis indicated that the reference material FPS attained the greatest depth of cure, closely followed by the BU3T7 formulation, while the remaining experimental groups showed comparatively lower values. The majority of the tested materials attained clinically meaningful polymerization depths, corroborating findings reported in prior literature.

The findings related to the depth of cure (DoC), illustrated in Table 2 indicate notable variations between dual-cure flowable composites (DCFCs) that use urethane dimethacrylate (UDMA) as the base monomer and those that incorporate bisphenol A-glycidyl methacrylate (Bis-GMA) as the base monomer. The observed differences are consistently apparent in both chemical curing and dual curing modes, highlighting the impact of monomer composition on the effectiveness of polymerization.

The enhanced depth of cure noted in UDMA-based DCFCs can be largely ascribed to the greater translucency of these composites. The improved translucency results from the precise matching of the refractive indices between the UDMA resin matrix and the nanohybrid silica fillers integrated into the composite. Both UDMA and silica demonstrate high and closely aligned optical densities, enhancing optical compatibility within the composite matrix. The alignment of the refractive index reduces light scattering at the interfaces between the filler and matrix, which enhances the effective transmission of curing light across the material (Azlisham et al., 2023; Fujita et al., 2005). As a result, this optical synergy facilitates polymerization at increased depths, leading to enhanced curing performance in UDMA-based composites when compared to Bis-GMA alternatives. The results align with previous research that emphasizes the essential importance of filler-matrix optical compatibility in improving depth of cure. For example, Azlisham et al. (2023) showed that adding silica obtained from rice husk to flowable composites greatly enhanced the depth of cure. The enhancement observed can be linked to the exceptional optical characteristics and advantageous light scattering behaviour provided by the rice husk silica, which improved light penetration and polymerisation efficiency (Azlisham et al., 2023). The findings of the present study support these observations, highlighting the importance of optimising the refractive index match between resin matrices and filler particles to enhance polymerisation depth in dual-cure flowable composites.

This indicates that photoactivation could potentially disrupt the chemical curing process in these materials (Taubçck et al., n.d.). This phenomenon can be elucidated through the interplay between the degree of conversion and the mobility of monomers throughout the polymerisation process. At the initial stages of polymerisation, light activation triggers the swift creation of oligomers, leading to an increase in the material's viscosity and a decrease in monomer mobility (Sideridou et al., 2002). The decrease in mobility leads to a reduced frequency of collisions between unreacted active groups, which in turn impedes the advancement of the slower chemical self-curing reactions, ultimately resulting in a diminished overall degree of cure (Asmussen and Peutzfeldt, 2003).

It is also observed in present study that mean values of depth of cure for chemical curing mode were greater. The possible explanation for this is that in absence of light exposure the self cure mechanism proceed without any hinderance because of absence of formation of oligomers which form on exposure to light and cause reduced mobility of monomers. This is due to the reduced formation of oligomers at early stages, which enables monomers to maintain mobility and engage in more thorough reactions. On exposure to light, the photoactivation results in early network formation and heightened viscosity. This phenomenon limits the chemical curing reaction and diminishes polymerization efficiency. This is in agreement with previous studies (Taubçck et al., n.d.).

CONCLUSION

The study demonstrated that dual-cure flowable composites with varied base-to-diluent monomer ratios and rice husk-derived nanohybrid silica fillers achieve adequate depth of cure beyond ISO 4049 requirements. Formulations containing UDMA showed superior polymerization depths, with the 30:70 UDMA to TEGDMA ratio yielding the highest results. Additionally, chemical curing alone provided greater depth of cure compared to light-assisted curing. These findings suggest that the novel dual-cure composites possess promising curing efficiency for dental restorative applications, warranting further investigation into their long-term performance and clinical viability. These results highlight the potential of the novel composites as sustainable dental restorative materials, subject to further comprehensive evaluation.

DECLARATIONS

Funding This study was funded by the Research University Grant from Universiti Sains Malaysia RUI (1001/PPSG/8012380).
Data availability The data will be available from the corresponding author upon request.
Declarations
Competing interests:
The authors declare no competing interests.
Ethical approval
This research does not contain any studies with human participants performed by any of the authors or any studies with animals performed by any of the authors.

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