RESEARCH PAPER

Comparative Evaluation of the Mechanical Properties of Mineral Trioxide Aggregate, KedoBioD, and Novel Graphene Oxide–Copper Oxide Reinforced Calcium Silicate Cement - An In Vitro Study

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Abstract

Background: Calcium silicate–based cements such as MTA Angelus and KedoBioD are widely used in pediatric restorative dentistry because of their bioactivity and sealing ability. This study compared the compressive strength and mechanical behavior of MTA, KedoBioD, and a novel Graphene Oxide–Copper Oxide Reinforced Calcium Silicate Cement (GO–CuO-CSC).

Materials and Methods: Thirty cylindrical specimens were divided into three groups (n = 10): Group A – MTA, Group B – KedoBioD, and Group C – GO–CuO-CSC. Mechanical properties including maximum force, compressive stress, displacement at break, strain at break, and stress at break were evaluated using a Universal Testing Machine. Statistical analysis was performed using one-way ANOVA and Tukey's post hoc test.

Results: KedoBioD demonstrated the highest maximum force and compressive stress values, followed by GO–CuO-CSC, while MTA showed the lowest values. No statistically significant intergroup differences were observed for maximum force, compressive stress, or stress at break ($p > 0.05$). However, GO–CuO-CSC exhibited significantly higher displacement at break and strain at break values ($p < 0.05$), indicating enhanced flexibility and toughness.

Conclusion: GO–CuO-CSC demonstrated compressive properties comparable to MTA with superior deformation resistance and strain tolerance, suggesting its potential as a mechanically reliable and functionally advanced calcium silicate cement for pediatric restorative applications.

Keywords: Graphene oxide, calcium silicate cement, compressive strength, nanotechnology, pediatric restorative dentistry.

How to cite this article: 1Soundaria Srinivasan 2Maria Anthonet Sruthi. Comparative Evaluation of the Mechanical Properties of Mineral Trioxide Aggregate, KedoBioD, and Novel Graphene Oxide–Copper Oxide Reinforced Calcium Silicate Cement - An In Vitro Study. Int J Drug Deliv Technol. 2026;16(88s):1211-1219. DOI: 10.25258/ijddt.16.88s.113.

Introduction:

The evolution of biocompatible cements has transformed pediatric restorative dentistry. The ability of restorative materials to provide a biological seal, promote dentin regeneration, and withstand masticatory forces has remained central to the development of contemporary biomaterials. Mineral Trioxide Aggregate (MTA), introduced by Torabinejad in the 1990s, remains a benchmark material due to its biocompatibility, ability to release calcium ions, and superior sealing capability (1). It has been successfully employed in vital pulp therapy, apexification, root perforation repair, and retrograde fillings (2). Despite its benefits, MTA is not without drawbacks. Its extended setting time, susceptibility to washout in moist conditions, potential for tooth discoloration, and difficult handling have limited its acceptance in certain clinical scenarios (3).

To address these limitations, Biodentine was developed as a calcium silicate–based alternative. Marketed as "dentin in a capsule," Biodentine is composed of tricalcium and dicalcium silicates, calcium carbonate, and zirconium oxide, with a liquid phase consisting of calcium chloride and a hydrosoluble polymer in an aqueous solution (4). Its favorable properties, including improved handling, shorter setting time, and high compressive strength, have expanded its role in pediatric and restorative dentistry (5). Numerous investigations have shown Biodentine to effectively promote mineralization, induce odontoblastic differentiation, and provide long-term clinical success in vital pulp therapies (6). In pediatric patients, Biodentine is especially valuable due to its ability to maintain pulp vitality and facilitate minimally invasive management of deep caries (7). KedoBioD, a novel formulation recently introduced, represents an advancement in this family of biomaterials. It has been reported to possess superior compressive strength compared to conventional Biodentine and MTA, owing to modifications in its formulation and the inclusion of radio-opacifiers such as barium oxide (8).

Parallel to these developments, nanotechnology has introduced new possibilities in biomaterial design (9). Graphene oxide, a derivative of graphene, has been

extensively studied for its extraordinary tensile strength, antimicrobial properties, and ability to support cell adhesion and proliferation (10). Its incorporation into dental biomaterials has been proposed to enhance bioactivity(11). Copper nanoparticles, known for their antimicrobial and anti-inflammatory properties, have also been evaluated as additives to dental cements (12). Combining graphene oxide and copper oxide into a tricalcium silicate matrix has shown to provide synergistic benefits (13). Novel Graphene Oxide–Copper Oxide Reinforced Calcium Silicate Cement (GO–CuO-CSC) , a graphene-copper–infused tricalcium silicate cement, was synthesized to explore this potential. This study therefore aimed to compare its compressive strength against established materials MTA and KedoBioD.

Materials and Methods:

Study Design and Setting

This in vitro experimental study was conducted in the Department of Pediatric and Preventive Dentistry following institutional ethical approval (SRB/SDC/PEDO-2302/25/380). The study design involved the preparation of standardized cylindrical cement specimens, which were subjected to compressive strength testing under controlled laboratory conditions. All procedures were performed in accordance with established protocols to ensure reproducibility and reliability of the obtained results.

Preparation of Graphene oxide nanoparticles

GO–CuO-CSC was synthesized through a multistep process. Graphene oxide was prepared using a modified Hummers' method (**Figure 1C**) . Initially, 1 gram of graphite powder was carefully mixed with 25 mL of concentrated sulfuric acid while maintaining the mixture in an ice bath. To this, 0.5 g of sodium nitrate and 3 g of potassium permanganate were gradually added, ensuring the temperature remained below 5°C. The reaction mixture was then stirred at room temperature for two hours. Following this, 50 mL of distilled water was slowly added to the mixture, which was subsequently heated to approximately 98°C for 15 minutes. The reaction was terminated by adding 100 mL of distilled water and 5 mL of hydrogen peroxide , which caused the mixture to turn a bright yellow. The product was washed repeatedly with distilled water and centrifuged at 10,000 rpm until a neutral pH was obtained. The resulting GO nanoparticles were dried at 60°C and stored in airtight containers. For further use, 0.100 g of the GO powder was dispersed in 5 mL of ethanol.

Preparation of copper oxide nanoparticles

Copper oxide nanoparticles were synthesized through a green chemistry approach utilizing *Glycyrrhiza glabra* extract as a reducing agent, a technique selected for its eco-friendly nature and ability to yield stable nanoparticles(**Figure 1A, B**) .Dried root powder of *Glycyrrhiza glabra* extract was boiled in 100 mL of distilled water at 50°C for 20 minutes and filtered through muslin cloth to obtain a clear extract. Separately, a 30 mM copper sulfate ($CuSO_4$) solution was prepared by dissolving the salt in 50 mL of distilled water and then mixed in equal proportion (50 mL) with the plant extract. The mixture was stirred continuously at 450 rpm for 48 hours. The CuO nanoparticles were collected by centrifugation at 8000 rpm for 10 minutes, washed thoroughly, and stored under refrigeration for further use.

Materials Included and Preparation

The nanoparticle-modified cement was formulated by incorporating graphene oxide (GO) and copper oxide (CuO) nanoparticles into a tricalcium silicate base powder. Both nanoparticles were added at a concentration of 1% by weight each, ensuring uniform distribution within the cement matrix to enhance its mechanical and functional properties. The powders were thoroughly blended using a mortar and pestle to achieve a homogenous mixture and prevent nanoparticle agglomeration.

For the liquid phase, a 30% calcium chloride ($CaCl_2$) solution was prepared using analytical-grade calcium chloride dissolved in distilled water under controlled conditions. The solution was mixed until complete dissolution was achieved, resulting in a clear, homogeneous liquid. Calcium chloride was selected as an accelerator to reduce the setting time and improve the early mechanical strength of the cement.

The prepared nanoparticle-incorporated powder and calcium chloride solution were subsequently used for cement manipulation according to standardized mixing protocols

The materials used in this study were categorized into three groups based on their composition and application:

- Group A: Mineral Trioxide Aggregate (MTA)
- Group B: KedoBioD
- Group C: GO–CuO reinforced calcium silicate cement

The respective materials and their components are illustrated in Figure 2 (A–C).

Group A – Mineral Trioxide Aggregate (MTA)

Mineral Trioxide Aggregate (MTA Angelus, Londrina, Brazil) was prepared according to the manufacturer's instructions. The powder was mixed with sterile distilled water in a 3:1 powder-to-liquid ratio on a glass slab using a metal spatula. Mixing was performed for approximately 30 seconds to obtain a sandy, putty-like consistency. The material was used immediately after mixing to avoid premature setting.

Group B – KedoBioD

KedoBioD , was used as supplied by the manufacturer. The material consists of a powder and proprietary liquid component, which were dispensed in premeasured proportions. Mixing was carried out on a sterile mixing pad using spatula for approximately 30–40 seconds until a smooth, homogeneous consistency was achieved. The prepared material was immediately transferred into the molds for specimen fabrication.

Group C – Novel GO–CuO-CSC

The experimental GO–CuO-CSC consisted of a powder component containing calcium silicate, graphene oxide, and copper oxide nanoparticles, along with a liquid component. The powder and liquid were combined on a sterile glass slab using a stainless steel spatula in a proportion adjusted to achieve a clinically acceptable, workable consistency. Mixing was carried out for 30–45 seconds until a uniform, cohesive paste was obtained. The prepared material was immediately transferred into molds for specimen fabrication."

Compressive strength testing

For all groups, cylindrical samples measuring 4 mm in diameter and 6 mm in height were fabricated using stainless steel molds. The mixed cements were packed into the molds with care to minimize porosity and covered with glass slides at both ends to produce flat surfaces. The molds were stored at 37 °C and 100% relative humidity for 24 hours to allow complete setting and tested (Figure 3B).

Compressive strength testing was carried out using a Universal Testing Machine (Instron, UK) (Figure 3A**)**. Each sample was placed vertically along the loading axis, and force was applied at a crosshead speed of 1 mm/min until fracture. The peak force was recorded in Newtons, and compressive stress was calculated in megapascals. Additional parameters, including displacement at break, strain at break, and stress at break, were also recorded to provide a comprehensive assessment of mechanical behavior.

Statistical Analysis

Statistical analysis was conducted using IBM SPSS v22 (Armonk, NY). Normality was confirmed using the Shapiro–Wilk test. One-way ANOVA was performed to identify intergroup differences, and Tukey's post hoc test was applied for pairwise comparisons. A p-value <0.05 was considered statistically significant .

Results

The compressive strength and overall mechanical behavior of MTA Angelus, KedoBioD, and the novel Graphene Oxide–Copper Oxide Reinforced Calcium Silicate Cement (GO–CuO-CSC) were evaluated using universal testing analysis. The assessed parameters included maximum force, compressive stress, displacement at break, strain at break, and stress at break. The mean values and statistical comparisons are summarized in Tables 1 and 2.

KedoBioD demonstrated the highest maximum force (336.42 ± 24.18 N) among all experimental groups, followed by the novel GO–CuO-CSC (318.56 ± 22.64 N), whereas MTA exhibited the lowest maximum force values (294.18 ± 21.42 N). Similarly, compressive stress values were highest in the KedoBioD group (48.76 ± 3.82 MPa), followed by GO–CuO-CSC (46.92 ± 3.48 MPa), while MTA showed the lowest compressive stress (41.52 ± 3.14 MPa). Stress at break values also followed a similar trend, with KedoBioD demonstrating the highest values (44.12 ± 3.14 MPa), followed by GO–CuO-CSC (42.68 ± 2.96 MPa) and MTA (38.41 ± 2.62 MPa). However, statistical analysis revealed no significant intergroup differences for maximum force ($p = 0.058$), compressive stress ($p = 0.067$), or stress at break ($p = 0.079$), indicating that the novel GO–CuO-CSC maintained compressive properties comparable to conventional calcium silicate cements.

In contrast, significant differences were observed in parameters associated with deformation behavior and strain tolerance. The novel GO–CuO-CSC exhibited the highest displacement at break value (1.046 ± 0.05 mm), indicating a greater capacity for deformation before fracture. MTA demonstrated intermediate displacement values (0.918 ± 0.08 mm), while KedoBioD exhibited the lowest displacement at break (0.782 ± 0.06 mm). Similarly, strain at break was highest in the GO–CuO-CSC group (17.84 ± 1.22%), followed by MTA (14.83 ± 1.44%), whereas KedoBioD showed the lowest strain at break values (12.96 ± 1.08%). One-way ANOVA demonstrated statistically significant intergroup differences for displacement at break ($p = 0.018$) and strain at break ($p = 0.011$).

Tukey's post hoc analysis further demonstrated that differences in maximum force, compressive stress, and stress at break among the experimental groups were not statistically significant ($p > 0.05$). However, statistically significant differences were observed for displacement at break and strain at break between all pairwise group comparisons ($p < 0.05$). The comparison between MTA and GO–CuO-CSC demonstrated significantly higher displacement and strain values for the novel material, suggesting enhanced flexibility and improved resistance to brittle fracture. Similarly, comparison between KedoBioD and GO–CuO-CSC revealed significantly greater strain tolerance and deformation capacity in the novel nanocomposite cement.

Discussion

The present investigation evaluated the mechanical characteristics of the novel GO–CuO-CSC in comparison with Mineral Trioxide Aggregate (MTA) and KedoBioD. The findings demonstrate that incorporation of graphene oxide (GO) and copper

oxide (CuO) into a calcium silicate cement matrix results in a material that preserves baseline mechanical strength while significantly improving deformation characteristics.

The novel GO–CuO-CSC exhibited compressive properties comparable to MTA, with no statistically significant difference across parameters such as maximum force and compressive stress. This is clinically relevant, as MTA continues to be regarded as the gold standard pulp capping material due to its bioactivity and sealing ability. The mechanical limitations of MTA, particularly its relatively low early strength and brittle nature, remain a concern in load-bearing situations (14) . The present results indicate that modification with GO and CuO does not compromise these baseline properties, thereby maintaining equivalence with established materials. Graphene oxide's ability to accelerate early hydration reactions ensures the formation of a dense microstructure even at early stages of setting, a factor that could contribute to improved early strength and dimensional stability (15). Copper nanoparticles, on the other hand, not only provide secondary mechanical reinforcement but also introduce antimicrobial and anti-inflammatory capabilities (16). Copper ions may act as additional nucleation sites, stimulating early C–S–H formation (17). From a biological perspective, copper is known to disrupt microbial membranes and generate oxidative stress, contributing to antimicrobial defense (18). Copper also contributes to antimicrobial and anti-inflammatory properties(19). While these antimicrobial aspects were not measured in the present compressive strength study, they highlight a potential advantage of GO–CuO-CSC over MTA in clinical practice, where microbial control is paramount for pulp preservation.

The most significant enhancement observed in the novel material was in displacement at break and strain at break, which were higher than both MTA and KedoBioD. These parameters reflect the material's ability to undergo deformation before fracture, indicating improved toughness and reduced brittleness. This behavior can be attributed to the reinforcing effect of graphene oxide. GO has been widely reported in biomedical literature to improve the mechanical performance of composite materials through crack-bridging mechanisms and enhanced interfacial bonding, leading to increased resistance to fracture propagation (20,21).

When compared with KedoBioD, the novel GO–CuO-CSC demonstrated slightly lower compressive strength but superior flexibility and strain tolerance. KedoBioD showed the highest compressive stress and maximum force values(8). This microstructural refinement allows for increased nucleation of hydration products and formation of a more continuous calcium silicate hydrate (C–S–H) network. Such a dense C–S–H structure not only resists microcrack initiation but also impedes crack propagation under functional loads(22). In primary molars, where functional loading is often eccentric and less controlled, restorative cements must resist fracture despite reduced dentinal support (23). KedoBioD's reduced displacement and strain at break indicate increased stiffness, which reduces the risk of restoration collapse or microleakage over time (24). In contrast, the novel GO–CuO-CSC exhibited a more ductile behavior, suggesting an improved ability to absorb and dissipate stresses. This is particularly important in restorative dentistry, where materials are subjected to complex mechanical forces, including compression, tension, and shear. A material with higher strain tolerance can better accommodate these forces, reducing the likelihood of marginal breakdown and internal crack propagation (25). The Improved stress at break of the novel material compared to MTA, indicating enhanced resistance to fracture under applied stress. This suggests that the synergistic interaction between GO nanosheets and the calcium silicate matrix may enhance structural cohesion. Graphene-based nanomaterials have been shown to improve load transfer efficiency within composite systems, thereby increasing their mechanical resilience (20). The novel GO–CuO-CSC achieves an optimal balance, maintaining sufficient strength while significantly improving deformation capacity.

The compressive strength of pulp capping agents is a vital property because these materials must withstand occlusal loading once restored. Insufficient compressive resistance predisposes the cement to fracture or microcrack formation, which in turn leads to microleakage, secondary bacterial invasion, and eventual treatment failure (26). The superior performance of KedoBioD in this study supports its clinical adoption, particularly in pediatric contexts where thin enamel and dentin layers place greater demand on the restorative material (27). In contrast, GO–CuO-CSC , by demonstrating parity with MTA, positions itself as a viable candidate for clinical use. Thus, the comparable strength to MTA is significant because it validates novel GO–CuO-CSC's basic mechanical viability while opening avenues for further biological exploration

While compressive strength provides important baseline validation, the clinical success of pulp capping agents depends equally on biological outcomes. Materials must be biocompatible, promote reparative dentinogenesis, and prevent bacterial infiltration (28). Graphene oxide, by virtue of its ability to support stem cell adhesion and proliferation, may encourage pulp healing and dentin bridge formation (29). Likewise, copper ions are known to

stimulate angiogenesis and tissue remodeling, processes essential for successful pulp regeneration (30). The combination of these properties positions GO–CuO-CSC as more than a mechanically competent material it represents a multifunctional bioactive cement. Its potential advantages include reduced microbial colonization, accelerated pulp healing, and improved resistance to reinfection compared to conventional cements. However, such claims require validation through cytocompatibility assays, calcium ion release studies, antimicrobial testing, and in vivo trials. For instance, Roy et al. demonstrated that Biodentine outperformed MTA in reducing apical microleakage in simulated young permanent teeth, underlining the importance of microstructural sealing ability in determining long-term clinical outcomes (31). Similarly, Acharya et al. compared Biodentine, NeoPutty MTA, and calcium hydroxide in indirect pulp therapy of deciduous teeth and confirmed that calcium silicate–based materials exhibited superior outcomes over traditional formulations (32). A calcium strontium silicate–infused alginate–ECM scaffolds for periodontal regeneration reported excellent cell viability, hemocompatibility, and osteogenic stimulation, further validating the broader role of silicate-based biomaterials in regenerative dentistry (33). Graphene oxide enhances early strength development by acting as a nucleation site for C–S–H formation and improving microstructural densification. This is in agreement with the findings of Govindarajan D et al., who highlighted the superior mechanical strength and bioactivity of graphene-based materials (34). Additionally, graphene oxide contributes to improved fracture resistance through nanoscale reinforcement and crack-bridging mechanisms. Similar improvements in calcium silicate systems have been reported with bioactive modifications, as demonstrated by Patel T et al., supporting the role of advanced additives in enhancing both mechanical and regenerative properties (35).

Incorporating these contemporary findings into the interpretation of GO–CuO-CSC suggests that pediatric dentistry is moving toward multifunctional bioactive cements where antimicrobial, regenerative, and mechanical properties converge

Limitations

The in vitro design of this study constitutes a primary limitation. Laboratory testing of compressive strength under static loading conditions does not replicate the complex intraoral environment, where factors such as saliva, thermal fluctuations, enzymatic activity, and cyclic masticatory stresses influence long-term material behavior.Additionally, advanced imaging (SEM, TEM) and spectroscopic techniques (FTIR, XRD) are needed to characterize hydration products and elucidate the role of graphene and copper at the molecular level. Long-term in vivo studies in animal models followed by randomized clinical trials will be essential before GO–CuO-CSC can be positioned as a clinically viable alternative.

Future scope

The findings of this study align with the growing trend toward multifunctional restorative biomaterials that integrate mechanical durability with biological functionality. KedoBioD currently sets the benchmark for mechanical strength, ensuring predictable resistance under occlusal loading. However, novel GO–CuO-CSC , by achieving mechanical equivalence to MTA and offering potential antimicrobial and regenerative benefits, represents the future trajectory of material development.In pediatric pulp therapy, where long-term preservation of vitality is critical, a material like GO–CuO-CSC could provide the dual benefit of mechanical stability and biological protection. If future studies confirm its regenerative and antimicrobial capabilities, novel GO–CuO-CSC may redefine pulp capping strategies by addressing both the mechanical and biological shortcomings of current materials. Its development underscores the translational potential of nanotechnology in dentistry, reflecting a broader paradigm shift toward bioactive and multifunctional restorative cements. This study reinforces KedoBioD as a mechanically superior material while also validating GO–CuO-CSC as a novel nanotechnology-driven cement with compressive strength equivalent to MTA. Continued exploration of its biological properties will determine whether GO–CuO-CSC can translate from laboratory innovation to a clinically transformative biomaterial in pediatric restorative dentistry.

Conclusion:

The novel graphene oxide–copper oxide reinforced calcium silicate cement GO–CuO-CSC demonstrated compressive strength comparable to Mineral Trioxide Aggregate, while exhibiting superior deformation characteristics indicative of enhanced toughness and reduced brittleness. Although KedoBioD showed the highest compressive strength, the ability of GO–CuO-CSC to maintain mechanical equivalence with MTA, combined with its improved strain tolerance, highlights its potential as a mechanically reliable and functionally advanced biomaterial.

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Figures and Tables

Table 1. One-way ANOVA comparison of mechanical properties among MTA, KedoBioD, and novel GO–CuO-CSC

Parameter	**MTA (Mean ± SD)**	**KedoBioD (Mean ± SD)**	**Novel GO–CuO-CSC (Mean ± SD)**	**p-value**
Maximum Force (N)	294.18 ± 21.42	336.42 ± 24.18	318.56 ± 22.64	0.058
Compressive Stress (MPa)	41.52 ± 3.14	48.76 ± 3.82	46.92 ± 3.48	0.067
Displacement at Break (mm)	0.918 ± 0.08	0.782 ± 0.06	1.046 ± 0.05	0.018*
Strain at Break (%)	14.83 ± 1.44	12.96 ± 1.08	17.84 ± 1.22	0.011*
Stress at Break (MPa)	38.41 ± 2.62	44.12 ± 3.14	42.68 ± 2.96	0.079

*Statistically significant difference observed ($p < 0.05$).

Table 2. Tukey's post hoc comparison of mechanical parameters between groups

Comparison	**Maximum Force (N)**	**Compressive Stress (MPa)**	**Displacement at Break (mm)**	**Strain at Break (%)**	**Stress at Break (MPa)**
MTA vs KedoBioD	0.052	0.061	0.042*	0.039*	0.074
MTA vs Novel GO–CuO-CSC	0.081	0.087	0.031*	0.028*	0.091
KedoBioD vs Novel GO–CuO-CSC	0.096	0.102	0.018*	0.021*	0.109

*Statistically significant difference observed ($p < 0.05$).

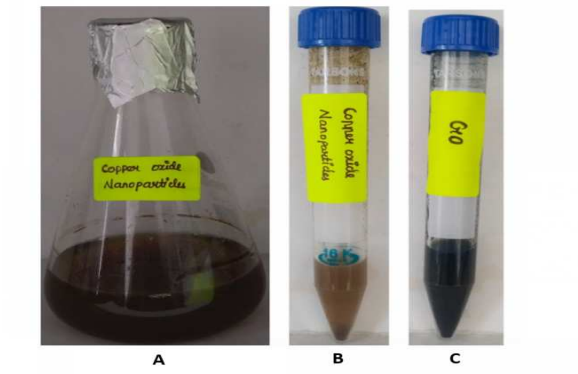


Figure 1: Preparation of nanoparticles—(A) Copper oxide (CuO) nanoparticle reaction mixture prior to centrifugation, (B) pelletized CuO nanoparticles obtained after centrifugation, and (C) graphene oxide (GO) nanoparticle dispersion prepared using the modified Hummers' method.



Figure 2: Materials used in the study— (A)Mineral Trioxide Aggregate, (B) KedoBioD and (C) Novel GO–CuO-CSC showing powder and liquid components.

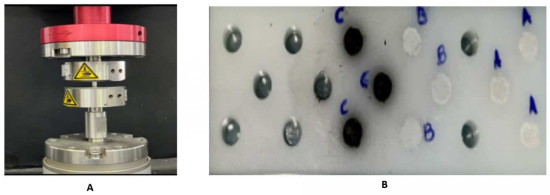


Figure 3: (A) Universal Testing Machine used for compressive strength testing of cylindrical specimens; (B) Prepared cylindrical samples of Groups (A)Mineral Trioxide Aggregate, (B) KedoBioD and (C) Novel GO–CuO-CSC following removal from molds.