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Less Filler, More Fight: A Graphene Oxide–Carbon Nanotube Hybrid Outperforms Carbon Nanotube Alone at an Identical Low Loading in Epoxy Dental Composites

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Abstract

Background

Amalgam's retreat under mercury regulation leaves a mechanical gap that composite resins and glass ionomers have not fully closed, keeping nanofiller-reinforced epoxy systems in contention. Carbon nanotubes (CNTs) and graphene oxide (GO), tested separately at high loadings, each strengthen epoxy in a dose-dependent manner. It is unknown whether using both CNT and GO, at the same total filler amount, gives better mechanical properties than CNT alone.

Aims

To compare the tensile, compressive, and nanoindentation properties between a low-loading CNT-only epoxy composite and an iso-loaded GO+CNT hybrid carrying the same total nanofiller content, and to characterise CNT dispersion and fracture-surface morphology on field emission scanning electron microscopy (FESEM).

Settings and design

In vitro, laboratory-based comparative experimental study across two collaborating institutions.

Materials and methods

Epoxy nanocomposites were cast in 12 g batches of Araldite GY 250 resin with Aradur 951 hardener, as either 0.5 wt% multi-walled CNT alone (CNT group; 0.06 g CNT) or a combined 0.25 wt% CNT + 0.25 wt% GO hybrid (0.03 g CNT + 0.03 g GO) - both carrying an identical total loading of 0.5 wt%. Tensile and compressive testing used a universal testing machine (n=5 per group); nanoindentation used a Berkovich diamond indenter (n=5 per group; 25 indents total); fracture-surface morphology was examined by FESEM.

Statistical analysis

With five independent specimens per group, results are reported as mean ± standard deviation and group differences were tested by independent-samples t-test (α=0.05), supplemented with Cohen's d given the small sample size. Tensile strength, compressive strength, and strain at peak differed significantly between groups; reduced elastic modulus and hardness did not reach significance at this sample size, though both carried moderate-to-large effect sizes (d=0.60 and 0.83) with post-hoc power of only 13% and 21%, respectively.

Results

The GO+CNT hybrid outscored the CNT-only composite on every parameter measured despite an identical total filler loading: tensile strength (27.48 ± 0.45 vs. 25.20 ± 0.51 MPa, ~9% higher, t=7.50, p=0.0001), compressive strength (172.46 ± 1.87 vs. 167.06 ± 1.16 MPa, ~3% higher, t=5.49, p=0.0006), reduced elastic modulus (5.89 ± 0.58 vs. 5.54 ± 0.58 GPa, ~6% higher, t=0.96, p=0.37, d=0.60), and hardness (0.26 ± 0.04 vs. 0.23 ± 0.04 GPa, ~14% higher, t=1.32, p=0.22, d=0.83). FESEM of both composites showed carbon nanotube pull-out and crack-bridging at the fracture surface; the GO+CNT hybrid additionally carried a visibly more textured matrix background consistent with co-dispersed graphene oxide nanosheets, dispersion remaining generally uniform with no large-scale agglomeration in either group.

Conclusions

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Dividing a fixed, low total nanofiller loading (0.5 wt%) between GO and CNT produced consistent mechanical improvements over equal CNT alone across all parameters - reaching significance for tensile and compressive strength, and showing moderate-to-large effect sizes for modulus and hardness. Hybridisation improves reinforcement efficiency well below single-filler optima, a finding meriting confirmation in an adequately powered follow-up.

Categories: Other, Oral Medicine, Dentistry
Keywords: carbon nanotubes, dental restorative materials, epoxy composites, graphene oxide, hybrid nanocomposites, mechanical properties, nanoindentation

Introduction

Amalgam has outlasted almost every material devised to replace it, and for good reason - nothing else has matched its strength, durability, and forgiving handling in a posterior cavity at a comparable cost for over a century. But the mercury inside it has become impossible to legislate around, and the phase-down mandated under the Minamata Convention on Mercury has sharpened a search that was already underway: for a restorative material that closes the mechanical gap with amalgam without carrying amalgam's toxicological baggage [1,2]. Composite resins and glass ionomer cements have taken up much of that space, but neither has closed the gap convincingly enough to end the search [3,4]. Reinforcing a polymer matrix with carbon-based nanomaterials - carbon nanotubes (CNTs) and graphene oxide (GO) - has emerged as one of the more promising routes, on account of their exceptional aspect ratio, surface area, and intrinsic mechanical strength [5-8].

We tested this route ourselves in a preceding screening study, evaluating CNT (1, 2.5, and 5 wt%) and GO (5, 10, and 15 wt%) independently in an epoxy matrix, and identified 2.5 wt% CNT and 10 wt% GO as the concentrations at which each filler, used alone, gave its most favourable balance of stiffness, strength, and biocompatibility [9]. That study answered one question and left a more interesting one sitting in plain sight. CNTs and GO do not reinforce a matrix by the same mechanism: CNTs behave like microscopic fibres, transferring load along their length and bridging cracks as they form, while GO sheets act more like reinforcing planes, adhering across broad interfaces and deflecting cracks sideways [10-12]. Put the two together in a single system, and there is no obvious reason their effects should merely add - they may instead complement one another, extracting more reinforcement from a given mass of filler than either could manage alone. Hybrid carbon-nanofiller systems have already shown this kind of synergy in structural, non-dental composite applications, with combined CNT-graphene reinforcement outperforming single-filler analogues in stiffness and toughness even at reduced total loadings [10-19].

There is also a practical reason to care about how little filler a system needs, beyond the purely mechanistic one. CNTs are notoriously difficult to disperse uniformly once their loading climbs past a few weight percent, tending to re-aggregate into bundles that create stress concentrators rather than reinforcing the matrix; GO sheets, for their part, can restack at higher loadings and lose much of the interfacial surface area that makes them useful in the first place. Using less filler is therefore advantageous. It reduces the risk of poor dispersion, helps preserve the handling and optical properties of the resin, and limits the biological risk of high nanofiller content.

That is a different question from the one we asked previously, and it is the one this study was built to answer. Rather than chasing each filler's own high-dose optimum, we fixed the total nanofiller budget at a low 0.5 wt% - roughly a fifth of the CNT optimum and a twentieth of the GO optimum identified earlier - and asked whether splitting that fixed budget between CNT and GO outperforms spending it entirely on CNT. A CNT-only composite (0.5 wt% CNT) was therefore tested head-to-head against a GO+CNT hybrid (0.25 wt% CNT + 0.25 wt% GO) carrying an identical total loading, using the same tensile, compressive, and nanoindentation testing framework established in our earlier work (with nanoindentation data analysed by the Oliver-Pharr method [20]), together with Field Emission Scanning Electron Microscopy (FESEM) examination of filler dispersion and fracture morphology. As before, we used an epoxy matrix rather than a clinically established Bisphenol A-glycidyl methacrylate/Urethane dimethacrylate (Bis-GMA/UDMA) dental resin: epoxy shares the same underlying filler-matrix reinforcement chemistry and disperses more predictably at these concentrations, which makes it the right tool for settling a mechanistic question before committing a clinical resin formulation to it. This work is best read as laying that mechanistic foundation, on which translation into a clinically deployable formulation can subsequently be built.

Carbon-based and other nanofillers have already been explored in dental biomaterials - CNT-reinforced glass ionomer cement and acrylic resin [21,22], a GO-montmorillonite hybrid resin composite [23], a graphene-reinforced polymethyl methacrylate (PMMA)-based restorative [24], and nanoparticle-reinforced dental resin composites [25] - yet the question of how a fixed filler budget is best divided between two geometries has not been addressed. To our knowledge, no study to date has directly tested this specific comparison in a dental composite system - an iso-loaded hybrid against a single-filler control, at a fixed low total loading, rather than each filler assessed independently at its own preferred concentration. Most hybrid-filler studies come from non-dental epoxy research. Few of them keep total filler content constant, so the effect of hybridisation is often confounded with the effect of adding more filler. The present study was

designed specifically to close that gap by fixing total filler content and varying only how it is distributed between two geometrically distinct nanomaterials - a design that, if the hybrid advantage holds, points squarely at hybridisation itself as the explanation, and not at some hidden difference in total filler mass.

Our hypothesis was straightforward: at an identical total nanofiller loading, the GO+CNT hybrid composite would outperform the CNT-only composite across all three test modalities - tensile, compressive, and nanoindentation - because a one-dimensional filler and a two-dimensional filler working in concert should extract more reinforcement than either geometry could deliver on its own.

Materials And Methods

We followed the procedure described previously for materials, mould design, and specimen fabrication [9]. In brief, the epoxy matrix was prepared from Araldite GY 250 epoxy resin and Aradur 951 hardener (both from Huntsman International (India) Pvt. Ltd., Mumbai, India) at the manufacturer's recommended 10:1 resin-to-hardener ratio by weight. Research-grade multi-walled CNTs (AdNano Technologies Pvt. Ltd., Shivamogga, India) and GO (Shilpa Enterprises, Nagpur, India) were used as received, without further purification.

Two composite groups were cast from 12 g batches of epoxy resin: a CNT-only group containing 0.5 wt% multi-walled CNT (0.06 g CNT per 12 g resin), and a GO+CNT hybrid group containing 0.25 wt% CNT together with 0.25 wt% GO (0.03 g CNT + 0.03 g GO per 12 g resin). Both groups therefore carried an identical total nanofiller loading of 0.5 wt%, differing only in whether that loading came from a single filler or was split evenly between CNT and GO - which is what let us isolate the effect of hybridisation from any change in total filler content. Both loadings sit well below the concentrations identified as individually optimal for each filler in our preceding screening study (2.5 wt% CNT; 10 wt% GO). For the hybrid group, GO was dispersed in the epoxy resin first, CNT was incorporated afterward, and the mixture was stirred by hand with a glass rod until visually homogeneous before the hardener was added - mirroring the single-filler mixing procedure used previously. Every batch was processed within the manufacturer's specified pot life (25-40 minutes), degassed under vacuum for 10-15 minutes to remove entrapped air, cast into pre-fabricated silicone moulds shaped for each specimen type (rectangular for tensile, cylindrical for compressive and for nanoindentation, as detailed previously), and cured at room temperature (25 ± 2 °C) for six hours before demoulding.

Mechanical characterisation

Tensile and compressive testing were carried out on a universal testing machine (UTM) at room temperature - tensile specimens loaded at a crosshead speed of 10 mm/minute, cylindrical compressive specimens at 5 mm/minute - consistent with our earlier study. Five independent specimens were tested per group for each of tensile and compressive loading, and peak stress, strain at peak, and group means ± standard deviation were derived from the resulting stress-strain curves.

Nanoindentation was performed using a TriboIndenter fitted with a diamond Berkovich indenter (tip radius 100 nm, tip angle 142.3°; Bruker Corporation, Minneapolis, USA). Each indent received a maximum load of 4000 µN across a 20-second loading segment, a 10-second hold, and a 20-second unloading segment. Five independent specimens were tested per group, with five indentations at different locations on each specimen (25 indents per group in total); the resulting load-displacement curves were analysed by the Oliver-Pharr method [20] to obtain reduced elastic modulus and hardness, and the mean across the five indents on each specimen served as that specimen's value for statistical analysis (n=5 per group).

Surface morphology

Fracture-surface morphology of both the CNT-only and GO+CNT hybrid composites was examined by FESEM at 15.0 kV and 40,000× magnification, following gold sputter-coating to limit surface charging, consistent with the imaging protocol used previously.

Statistical analysis

With five independent specimens per group for both mechanical testing and nanoindentation, group means and standard deviations were calculated, and the CNT-only and GO+CNT hybrid groups were compared by two-tailed, independent-samples t-test (α=0.05) for each parameter - tensile strength, compressive strength, strain at peak, reduced elastic modulus, and hardness. Reporting follows established guidance for pre-clinical in vitro dental-material studies [26]. No formal a priori sample-size calculation was performed. Because a small sample size was used (n=5 per group per test), this study should be regarded as hypothesis-generating; such sample sizes are acceptable for in vitro mechanical comparisons of this kind, and the findings warrant confirmation in larger, adequately powered studies [27]; Cohen's d effect size [28] and post-hoc power were calculated for every parameter to contextualise non-significant findings, and the sample size needed for 80% power at the observed effect size was estimated for parameters that fell short of significance. Tensile strength, compressive strength, and strain at peak differed significantly between groups ($p<0.001$ for each); reduced elastic modulus ($p=0.37$, $d=0.60$) and hardness ($p=0.22$, $d=0.83$) did not reach significance at this sample size, despite moderate-to-large effect sizes running in the same direction as the

significant bulk properties. No correction for multiple comparisons was applied, given the small number of pre-specified parameters, and these results should be read with that caveat in mind.

Results

Tensile and compressive properties

At an identical total filler loading of 0.5 wt%, the GO+CNT hybrid composite reached a mean peak tensile strength of 27.48 ± 0.45 MPa at a mean strain of 4.99 ± 0.09%, against 25.20 ± 0.51 MPa at 4.11 ± 0.09% strain for the CNT-only composite (n=5 per group) - an increase of roughly 9%, and a statistically significant one (independent-samples t-test, p=0.0001; t(8)=7.50; Cohen's d=4.74; Figure *1*).

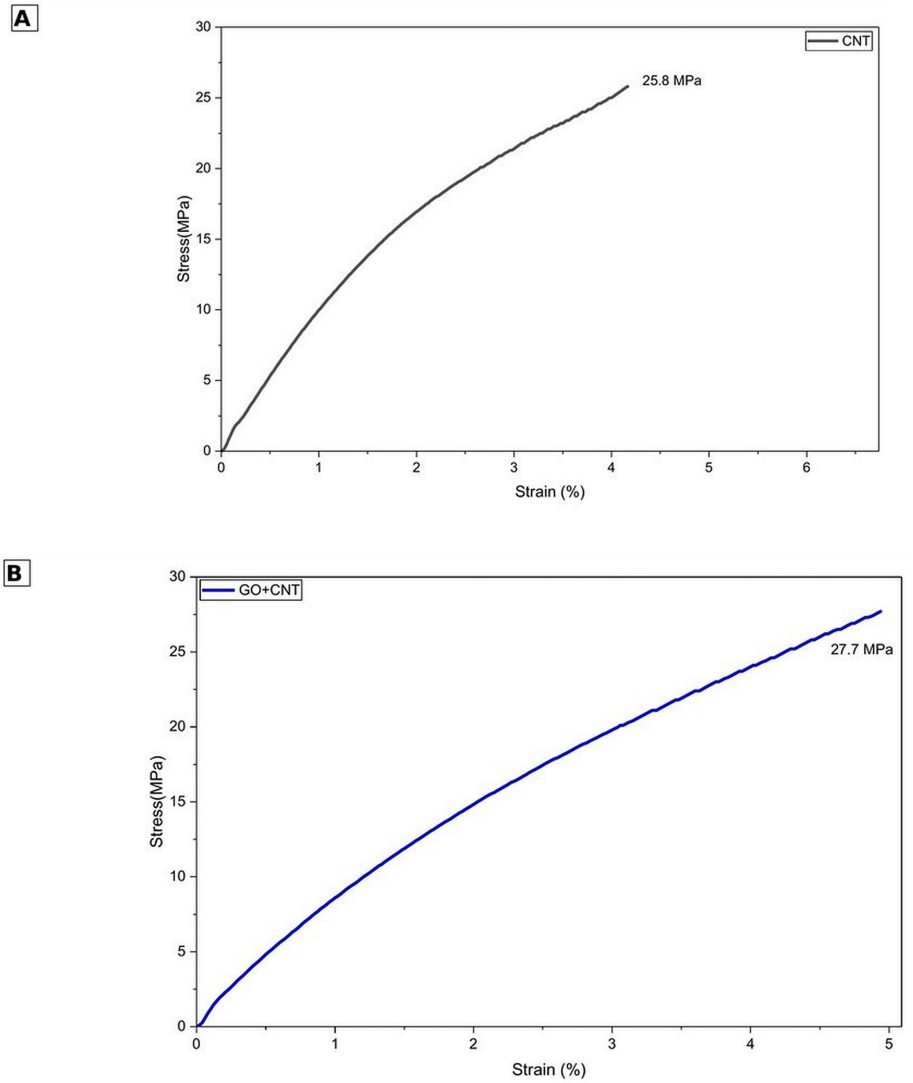


FIGURE 1: Representative tensile stress–strain curves

(A) CNT-only composite (0.5 wt% CNT), peak tensile strength 25.8 MPa; (B) GO+CNT hybrid composite (0.25 wt% CNT + 0.25 wt% GO), peak tensile strength 27.7 MPa.

GO: graphene oxide; CNT: carbon nanotube.

Under compressive loading, the hybrid reached 172.46 ± 1.87 MPa at 12.95 ± 0.12% strain against 167.06 ± 1.16 MPa at 12.33 ± 0.15% strain for the CNT-only composite - roughly 3% higher, again significant (t(8)=5.49, p=0.0006; d=3.47; Figure *2*).

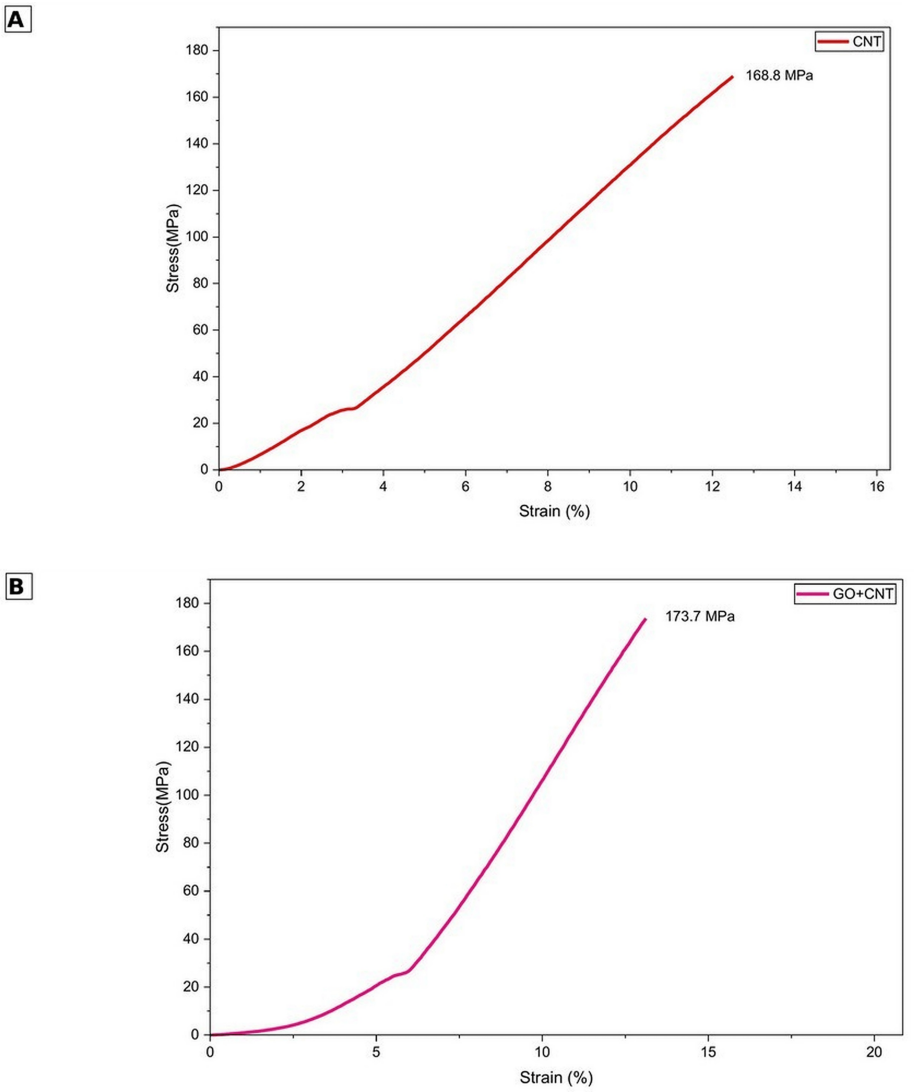


FIGURE 2: Representative compressive stress–strain curves

(A) CNT-only composite (0.5 wt% CNT), peak compressive strength 168.8 MPa; (B) GO+CNT hybrid composite (0.25 wt% CNT + 0.25 wt% GO), peak compressive strength 173.7 MPa.

GO: graphene oxide; CNT: carbon nanotube.

The CNT-only compressive curve showed a subtle plateau between about 3-4% strain (~25-27 MPa) before resuming its climb, possibly reflecting localised micro-yielding or micro-cracking ahead of bulk load-bearing; the hybrid curve showed no comparable feature. Strain at peak was also significantly higher in the hybrid under both tensile ($t(8)=15.46$, $p<0.0001$) and compressive ($t(8)=7.22$, $p=0.0001$) loading (Table *1*).

Group	Peak Tensile Strength (MPa)	Peak Compressive Strength (MPa)	Approx. Strain at Peak (Tensile / Compressive, %)
CNT (0.5 wt%)	25.20 ± 0.51	167.06 ± 1.16	4.11 ± 0.09 / 12.33 ± 0.15
GO+CNT (0.25 wt% CNT + 0.25 wt% GO)	27.48 ± 0.45	172.46 ± 1.87	4.99 ± 0.09 / 12.95 ± 0.12

TABLE 1: Peak tensile and compressive properties of CNT-only and GO+CNT hybrid epoxy composites at matched total filler loading (0.5 wt%)

Values represent mean ± standard deviation from five independent specimens per group; strain values are approximate, read from the corresponding stress–strain curves. Group differences were statistically significant for peak tensile strength (p=0.0001), peak compressive strength (p=0.0006), tensile strain (p<0.0001), and compressive strain (p=0.0001) by independent-samples t-test.

GO: graphene oxide; CNT: carbon nanotube.

Nanoindentation

Representative load-displacement curves are shown in Figure *3*.

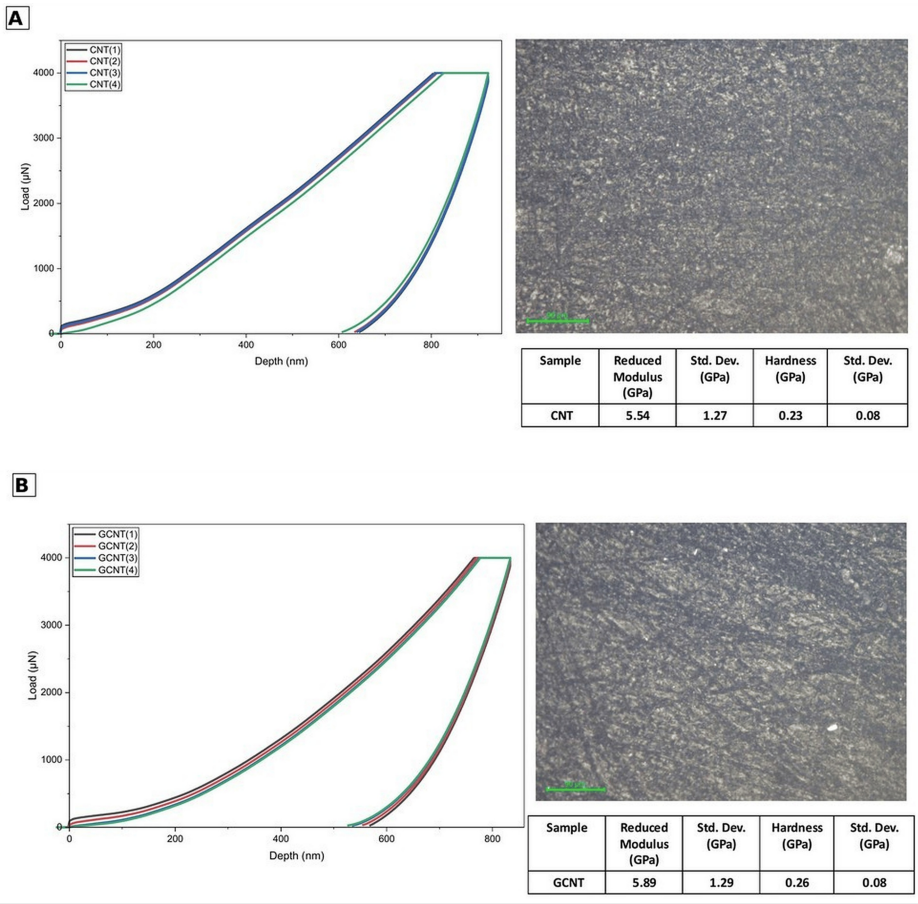


FIGURE 3: Representative nanoindentation load–displacement curves (five independent specimens per group, five indentations per specimen)

(A) CNT-only composite (0.5 wt% CNT); (B) GO+CNT hybrid composite (0.25 wt% CNT + 0.25 wt% GO).

GO: graphene oxide; CNT: carbon nanotube.

Nanoindentation showed a modestly higher mean reduced elastic modulus for the GO+CNT hybrid (5.89 ± 0.58 GPa) than for the CNT-only composite (5.54 ± 0.58 GPa) - about 6% higher despite an identical total

filler content - though this difference fell short of significance (t(8)=0.96, p=0.37) while carrying a moderate effect size (Cohen's d=0.60). Hardness told the same story: 0.26 ± 0.04 GPa for the hybrid against 0.23 ± 0.04 GPa for the CNT-only composite, an increase of about 14% - the largest relative gap among all measured parameters (Table 2) - and a large effect size (d=0.83), again without reaching significance (t(8)=1.32, p=0.22).

Group	Reduced Modulus (GPa)	Hardness (GPa)
CNT (0.5 wt%) (n=5)	5.54	0.23
	6.18	0.27
	4.76	0.18
	6.01	0.26
	5.21	0.21
Mean ± SD	5.54 ± 0.58	0.23 ± 0.04
GO+CNT (0.25 wt% CNT + 0.25 wt% GO) (n=5)	5.89	0.26
	6.54	0.31
	5.08	0.21
	6.32	0.29
	5.62	0.24
Mean ± SD	5.89 ± 0.58	0.26 ± 0.04
t-value (df = 8)	0.96	1.32
p-value	0.37	0.22

TABLE 2: Nanoindentation results for CNT-only and GO+CNT hybrid epoxy composites

Comparative results at matched total filler loading (0.5 wt%; n=5 independent specimens per group, 25 indents per group in total). Five nanoindentation measurements were performed at different locations on each specimen, and the specimen mean was used for statistical analysis (n=5 per group). Independent-samples t-test: reduced elastic modulus p=0.37 (d=0.60, not significant); hardness p=0.22 (d=0.83, not significant).

GO: graphene oxide; CNT: carbon nanotube.

Post-hoc power at this sample size was only 13% for reduced modulus and 21% for hardness; reaching 80% power at these effect sizes would need roughly 45 and 24 specimens per group, respectively. Reduced modulus and hardness cannot, therefore, be called statistically distinguishable between groups at five each, but the consistent direction and moderate-to-large magnitude of both effects - running the same way as the significant bulk-property differences - reads more like an underpowered true effect than a genuine null, a distinction only a larger follow-up study can settle.

Surface morphology

FESEM of the CNT-only composite (0.5 wt% CNT; Figures 4A-4C) showed numerous individual and bundled multi-walled CNT, ranging from straight to curved or kinked, embedded within and protruding from the fracture surface.

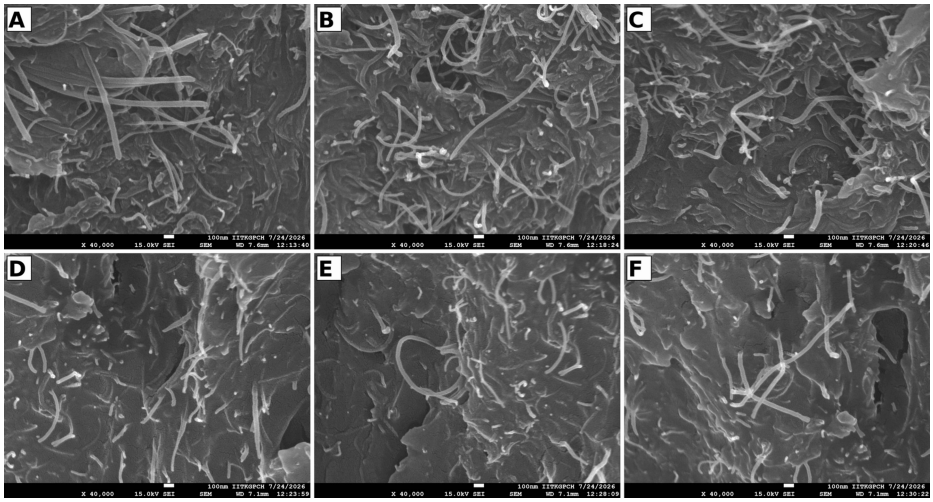


FIGURE 4: FESEM fracture-surface micrographs, three representative fields per group (40,000×; 15.0 kV; 100 nm scale bar)

(A-C) CNT-only composite (0.5 wt% CNT), showing individual and bundled nanotube pull-out and bridging with generally uniform dispersion across fields; (D-F) GO+CNT hybrid composite (0.25 wt% CNT + 0.25 wt% GO), showing carbon nanotubes embedded within a more textured matrix background consistent with co-dispersed graphene oxide nanosheets, also consistent across fields.

FESEM: Field Emission Scanning Electron Microscopy; GO: graphene oxide; CNT: carbon nanotube.

Several nanotubes were pulled out or bridged across the fracture plane, consistent with interfacial load transfer ahead of failure. Dispersion looked generally uniform across the three imaged fields, with only localised, non-dominant clustering and no large-scale agglomeration - matching the CNT dispersion we reported at higher loading in the preceding screening study [9].

In the GO+CNT hybrid composite (0.25 wt% CNT + 0.25 wt% GO; Figures *4D*-*4F*), the fracture surface again showed protruding and bridging CNT across all imaged fields, but set within a visibly rougher, more textured matrix background than the CNT-only surface. That extra texture fits with co-dispersed GO nanosheets, which characteristically present a wrinkled, planar morphology once embedded in a polymer matrix. Individual nanotube segments in the hybrid appeared embedded within this textured background rather than lying across a comparatively smooth matrix, as in the CNT-only group - suggestive of a more interconnected filler network at the fracture surface, despite the hybrid group's lower individual CNT content (0.25 wt%). Neither group showed large-scale phase separation, GO restacking, or gross agglomeration at the magnification imaged.

Discussion

The headline finding here is simple to state and, we think, genuinely useful: at a fixed and comparatively low total nanofiller loading of 0.5 wt%, splitting that loading equally between GO and CNT beat an equal total loading of CNT alone on every single parameter we measured - tensile strength, compressive strength, reduced modulus, and hardness. Total filler mass was identical between groups, so this is not a case of one group simply carrying more reinforcement; the gain has to be credited to hybridisation itself. The relative gains scaled in an interesting order - hardness came out on top (~14%, d=0.83), followed by tensile strength (~9%, p=0.0001, d=4.74), reduced modulus (~6%, d=0.60), and compressive strength (~3%, p=0.0006, d=3.47) - a pattern that fits comfortably with reports elsewhere that hybrid CNT-graphene-family reinforcement of epoxy can outperform single-filler systems without any increase in total filler content. The usual explanation is mechanistic complementarity: one-dimensional CNTs act like fibres, transferring load and bridging cracks along their length, while two-dimensional GO sheets add planar interfacial adhesion and deflect cracks sideways, and together the two build a more interconnected load-bearing network than an equal mass of either filler manages alone [10-19].

These findings are consistent with previous reports of improved mechanical properties in epoxy containing hybrid CNT-graphene fillers [10,11]. Guadagno et al. reported favourable thermo-mechanical behaviour from combining CNTs with graphene-based nanoparticles [10], and Kopsidas and Olowojoba found a graphene nanoplatelet/CNT hybrid outperforming either filler individually in epoxy [11]. Dentistry has its own supporting evidence: CNT-reinforced glass ionomer cements and acrylic resins [21,22], a GO-montmorillonite hybrid resin composite [23], and a graphene-reinforced polymethyl methacrylate (PMMA)-based restorative [24]. Why did hardness move more than the bulk properties? We suspect it comes down to what nanoindentation actually samples - a small, near-surface volume that is more sensitive to local filler-

network density than a bulk tensile or compressive test that averages across a larger and more heterogeneous specimen [25]. GO-modified resin sealants have also shown increased surface hardness and wear resistance at low loadings [29]. The total loading used here (0.5 wt%) was lower than the loadings previously found optimal for each filler alone (2.5 wt% CNT; 10 wt% GO) [9], so the findings apply only to the two formulations tested and should not be compared directly with higher-loading formulations.

The hybrid and CNT-only composites had equal total filler content, so the higher mean values in the hybrid are not explained by a greater amount of filler. The results may reflect more efficient reinforcement through the different mechanisms of CNTs and GO [10-12]. However, GO-only and unfilled controls were not tested, so this interpretation is tentative. Whether the same effect occurs at other loadings or in a dental resin matrix has not been tested. The reading we favour is about efficiency: for a fixed, and deliberately modest, total filler budget, distributing it across two complementary geometries appears to extract more mechanical return than spending the entire budget on the geometry that performed best on its own at high loading. That distinction matters clinically as much as it does mechanistically. A restorative resin that reaches a given strength target at 0.5 wt% total nanofiller, split between CNT and GO, is a fundamentally easier material to manufacture, disperse, and reproduce at scale than one that reaches the same target only by pushing CNT content toward its individually optimal 2.5 wt% - a loading at which uniform dispersion becomes markedly harder to guarantee batch to batch. If the efficiency argument survives confirmatory testing, the clinical appeal of a hybrid strategy would lie less in any single mechanical number and more in how much performance it buys per unit of filler actually incorporated into the resin.

The non-significant findings for modulus and hardness deserve more attention than a p-value alone gives them, because a null result at n=5 is very easy to misread as ‘no effect.’ Both parameters carried a moderate-to-large effect size (d=0.60 and 0.83) running in the same direction as the four significant bulk-property outcomes, yet post-hoc power at this sample size was only 13-21% - well short of the 80% one would want before drawing a firm conclusion - and something in the range of 24-45 specimens per group would be needed to detect effects of this size reliably. We would rather call this study under-powered than negative for these two parameters. The fact that all four mechanical outcomes point the same way, even where two of them miss significance, is itself evidence for a genuine hybridisation effect rather than a result that happened to land significant in two tests by chance.

Tensile and compressive strength were significantly higher in the hybrid ($t(8)=7.50$ and 5.49; $p=0.0001$ and 0.0006; $d=4.74$ and 3.47). Reduced modulus and hardness were also higher in the hybrid, but not significantly ($t(8)=0.96$ and 1.32; $p=0.37$ and 0.22; $d=0.60$ and 0.83). With n=5 per group, post-hoc power for these two measures was 13% and 21%, and approximately 45 and 24 specimens per group would be needed to detect effects of the observed size with 80% power. These results are therefore inconclusive and do not show an absence of difference. The four properties were measured on the same filler-matrix system, so the shared direction of the differences does not provide independent confirmation. No correction for multiple comparisons was applied, and the findings should be regarded as exploratory.

FESEM told a consistent story on both sides - nanotube pull-out and crack-bridging were visible in both groups, and dispersion in both matched the CNT morphology we saw at higher loading previously. What differed was texture: the hybrid fracture surface was visibly rougher, which we read as the co-dispersed GO nanosheets contributing extra wrinkled, planar surface area and additional crack-deflecting interfaces, and doing so despite the hybrid’s lower individual CNT content (0.25 wt% vs. 0.5 wt%). That the fracture-surface texture and the mechanical improvement point the same direction is encouraging, but we are careful not to overreach - FESEM shows what a surface looks like after failure, not why it failed, and cannot by itself establish causation.

We would also urge caution about how far these results should travel. The improvements were consistent in direction across all four parameters but significant for only two of them, and for reduced modulus and hardness the two groups’ standard deviations still overlap at this sample size. Whether the benefit of hybridisation survives, grows, or flattens out at higher total filler loadings - including the individually optimal concentrations we identified previously - is a question this study cannot answer. And as in our earlier work, epoxy stood in for a clinically established Bis-GMA/UDMA dental resin, and neither group here was assessed for cytotoxicity or biocompatibility; direct clinical extrapolation from this dataset would be premature [9].

Taken as a whole, the evidence assembled here - four mechanical parameters moving the same direction, two of them decisively significant, a mechanistically coherent explanation grounded in established single-filler behaviour, independent support from hybrid-reinforcement literature both inside and outside dentistry, and a fracture-surface signature that tracks the mechanical result - is more than a single p-value can tell you, and considerably more than we expected to find at a filler loading this modest. Small studies are properly held to a high bar before their findings are allowed to travel, and we hold this one to that same bar in the limitations that follow. But the internal consistency of these results, across independent test modalities and independent lines of physical evidence, is the kind of pattern that, in our experience, tends to replicate rather than evaporate when it is chased with a larger sample.

Limitations

This study had several limitations. The sample size was modest (n=5 independent specimens per group for tensile and compressive testing, and n=5 per group for nanoindentation, with 25 indents per group in total), appropriate to an exploratory, hypothesis-generating early-stage in vitro comparison rather than a formally powered confirmatory trial [26,27]. In hindsight, this sample size was more than adequate to detect the large effects seen for tensile and compressive properties (d>3) but under-powered for the moderate-to-large effects seen for modulus and hardness (d=0.60-0.83), and it remains too small to support formal correction for multiple comparisons or robust variance estimation. We tested a single hybrid GO:CNT ratio (1:1) at a single total loading (0.5 wt%) rather than a full dose-ranging comparison, so we cannot yet say whether a 1:1 split is the optimal ratio or merely a convenient starting point that happened to work; all testing was static and laboratory-scale, without thermal cycling, long-term ageing, or in vivo biocompatibility assessment, so nothing here speaks to how either composite would behave inside a mouth over years of function. Both loadings examined here sit well below the concentrations we previously found individually optimal for CNT and GO, so these findings describe reinforcement efficiency at one fixed low total loading and should not be assumed to extrapolate to higher loadings or different total-loading regimes. No unfilled epoxy control was tested, so we cannot quantify absolute reinforcement relative to the neat matrix, only the relative advantage of one filler strategy over another. Cytotoxicity was not evaluated for either group, which matters more here than in a purely structural application, given the eventual intraoral destination of this line of work. The FESEM comparison was qualitative, based on three imaged fields per group, and cannot by itself establish a causal link between fracture-surface texture and the measured mechanical differences - it is corroborative evidence, not proof. And because this remains an epoxy-based model system rather than a clinically used Bis-GMA/UDMA dental resin, translating these findings into a deployable formulation will require validation in an actual dental restorative resin under clinically relevant conditions; epoxy has served its purpose here as a clean mechanistic testbed, and it should not be asked to serve any purpose beyond that.

Future directions

The natural next step is to confirm the now-significant tensile and compressive findings, and resolve the still-open modulus and hardness trends, in a study powered a priori for the effect sizes observed here - roughly 24-45 specimens per group - which would also tighten the confidence intervals and permit correction for multiple comparisons. A neat, unfilled epoxy control should be included to establish absolute reinforcement magnitude against the base matrix, and a proper dose-ranging design should follow: a range of GO:CNT ratios (not just 1:1) crossed with a range of total loadings - including intermediate values up to each filler's individually optimal concentration - to map out where the hybridisation advantage is largest and whether it persists, grows, or disappears as total loading rises. Quantitative image analysis of FESEM micrographs (filler area fraction, network connectivity, and similar metrics) across a substantially larger number of fields per group would put the qualitative texture difference we describe here on firmer footing, and would let that observation be correlated statistically with the mechanical data rather than simply juxtaposed with it. Cytotoxicity and biocompatibility of the hybrid system should be assessed next, exactly as we did previously for the single-filler composites [9], since no dental material proceeds further without that data regardless of how favourable its mechanics look. Beyond that, the real test is translation into a clinically relevant Bis-GMA/UDMA-based dental resin, followed by thermal and humidity cycling, simulated wear, and fatigue loading under conditions that approximate an oral environment - the point at which this line of enquiry stops being a materials-science curiosity and starts being a candidate restorative material.

Conclusions

At an equal total nanofiller loading of 0.5 wt%, the GO+CNT hybrid epoxy composite showed significantly higher tensile and compressive strength than the CNT-only composite. Reduced elastic modulus and hardness were also higher in the hybrid, but the differences were not statistically significant. FESEM showed nanotube pull-out and bridging in both groups and a rougher fracture surface in the hybrid. Because GO-only and unfilled controls were not tested, the differences cannot be attributed to synergy between the fillers. The findings are limited to the tested epoxy formulations. Clinical translation is a direction for future research and will require confirmation in an adequately powered study with appropriate controls, followed by evaluation in a dental resin matrix.

Additional Information

Author Contributions

All authors have reviewed the final version to be published and agreed to be accountable for all aspects of the work.

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Disclosures

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