

RESEARCH PAPER

Influence of Filler Loading and Dispersion on the Mechanical Properties of Epoxy/Graphene Nanocomposites

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ABSTRACT

This study looked at how both the amount of graphene added and how well it's mixed into epoxy affect its mechanical properties. Basically, the goal was to figure out how these two factors interact and find out where you get the best mix of strength, stiffness, and toughness. So, they made samples with five different graphene levels, from 0 up to 1 wt%, and used two very different mixing methods: one that was pretty basic (just mechanical stirring, which doesn't mix things very well) and one that was way more advanced (ultrasound and surface treatment for better dispersion). To check what was happening inside, they used X-ray diffraction, electron microscopy, and Raman spectroscopy. For mechanical testing, they put the samples through tensile, three-point bending, and Charpy impact tests. Here's what happened. When graphene wasn't dispersed well and you added more than 0.1 wt%, big clumps formed around 15 μm in diameter. The tensile strength kept dropping, going down to 47 MPa with 1 wt% graphene, which is actually worse than pure epoxy. But when the graphene was dispersed properly, with particle sizes under 3 μm and spread out evenly, things really improved at 0.5 wt%. Tensile strength shot up by 30% to 84 MPa, fracture toughness climbed 77% to 1.08 MPa/m², and impact energy increased 75% to 21.7 kJ/m². Past this peak, even with good dispersion, both elongation and toughness went down so the material started behaving more brittle because of the graphene. Bottom line here is that Mixing quality matters way more than just piling in more graphene. If the graphene isn't well dispersed, adding extra actually makes things worse. You need good dispersion to really tap into what graphene can do. Just increasing its percentage isn't the main thing people should focus on here.

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INTRODUCTION

Epoxy resins have always been essential in advanced industries. You'll find them everywhere as mentioned by Mirzapour et al., from wind turbine blades to aerospace parts thanks to their excellent adhesion, chemical resistance, and dimensional stability [1]. But there's a catch as once cured, they turn brittle and stiff. That's a big problem in applications where you need flexibility or the ability to absorb impacts. Because of this, researchers like Pour & Ghaemy started looking at reinforcing epoxy with nanoparticles to toughen up the resin and boost strength, while still holding onto the qualities people love about epoxies [2]. Out of all the nanofillers, graphene stole the spotlight. This two-dimensional sheet of carbon is incredibly strong, its Young's modulus is close to a terapascal [3-6]. Even a tiny amount can seriously boost the mechanical properties of epoxy. But Jiang et al. putting this into practice which isn't simple. To really get those benefits, graphene must be evenly spread out, single layers, no clumps, and tightly bonded with the epoxy which is tough [7].

When you look at the experimental results like Rafiee et al., they don't quite live up to the theoretical models. There's a gap between what scientists expect and what they actually get [8]. This comes down to two things: how much graphene you add, and how well you mix it in. It's tempting to just add more graphene, but that backfires fast. Extra graphene starts sticking together van der Waals forces take over and forms big clumps [9-12]. Not only does that mess up stress transfer, but as noted by Maldonado et al., those clumps also act as weak points where cracks can easily start [13].

Going over the research, most studies focus on either tweaking surface treatments or dialing in a single parameter [14-18]. Very few actually look at both the amount added and how it's dispersed, even though those factors interact. You see, some reports show that after a certain point, increasing the graphene content actually drops tensile strength despite good mixing. Meanwhile, other studies like Xue et al., using different mixing methods with the same loading show improved strength [19]. The inconsistency tells us something's missing: we need a more systematic approach to understand how each factor shapes the composite's behavior [20-22].

This becomes even more important if you want to make these materials at a bigger scale. Based

on Zhang et al., it's not enough to find a recipe that works once in a lab [23]. For real-world production, we need to balance strength, stiffness, and toughness not just chase the highest value in a single property [24-27]. None of this optimization happens without untangling how dispersion quality and graphene content work together, and as stated by Wan et al., understanding which failure mechanisms take over at which point [28]. That knowledge is still lacking, and it's holding back smarter, more predictable design of these composites [29-32].

That's what this research is all about. Instead of picking apart each factor alone, the goal here is to create a clearer picture of how filler amount and dispersion quality interact. The key questions as if the dispersion isn't great, can you fix it by adjusting how much graphene you add? Or, can better mixing make it possible to use more graphene without sacrificing performance? Getting real answers will help move the industry past trial and error, leading to more targeted, reliable engineering of epoxy-graphene nanocomposites. It's a necessary step if we want to make smarter, stronger materials and actually bring them out of the lab and into the world.

MATERIALS AND METHODS

Raw Materials and Preparation of Nanofiller

For this study, we used an epoxy resin based on diglycidyl ether of bisphenol A, with a set epoxide equivalent mass and cured it with a cycloaliphatic amine hardener. Both came from reliable commercial sources. Our nanofiller was graphene sheets, averaging 5 μm in lateral size and less than 10 nm thick, made by electrochemical exfoliation. To get two different levels of dispersion quality, we split the graphene into two batches: one stayed raw with no treatment, and the other got a mild amine group functionalization to help it mix better with the epoxy. All solvents, like acetone and ethanol, were lab grade and dried to remove moisture before use. We loaded the nanofiller at 0, 0.1, 0.3, 0.5, and 1% by weight, so we could see how things change across the point where aggregation tends to kick in.

Nanocomposite manufacturing process and control of mixing variables

To actually mix the nanoparticles, we took two very different approaches. One gave us poor dispersion: we threw raw graphene into the resin

and mixed it hard 2,000 rpm for half an hour on a mechanical stirrer. For good dispersion, we first put the amine-functionalized graphene in acetone and hit it with a 200-watt ultrasonic bath for an hour to break up the layers. After that, we mixed it into the resin, then used a vacuum oven at 60°C to remove acetone and any trapped air bubbles. The curing agent went in last, measured precisely and mixed gently to avoid introducing any new stresses or clumping. Every sample was poured into silicone molds and cured through two temperature steps: first at room temperature, then at 120°C for three hours.

Microstructural characterization and mechanical performance evaluation

We checked how well the nanoparticles got dispersed using field emission scanning electron microscopy on fracture surfaces, and high-resolution TEM for super-thin sections sliced by ultramicrotomy. X-ray diffraction tracked how the graphene sheets separated in the matrix. For mechanical properties, we ran tensile tests (ASTM D638) on dumbbell-shaped samples at 1 mm/min to get stress-strain curves, telling us the ultimate

tensile strength, Young's modulus, and break elongation. We also measured Mode I fracture toughness with three-point bending (ASTM D5045) on single-edge-notched samples to see how sensitive the material is to cracks. And after testing, the fracture surfaces were imaged under electron microscopes at different magnifications to tie back how the particles spread out, how cracks traveled, and what kind of fracture mechanics were in play.

RESULTS AND DISCUSSION

To really understand how epoxy/graphene nanocomposites perform, you have to look at two things on their own: how much graphene you put in, and how you mix it into the epoxy. In this study, they kicked things off by taking a close look at the nanofiller itself. That way, they had a solid baseline for figuring out what's going on with the mechanical properties later.

Table 1 shows the graphene had a large surface area compared to its volume, plus pretty solid structural quality. A low ID/IG value means there weren't many defects in the lattice, and a higher C/O ratio tells us the surface was hydrophobic.

Table 1. Physicochemical characteristics of the graphene nanoplatelets.

Parameter	Value
Lateral size (μm)	4.2 ± 1.8
Thickness (nm)	7.5 ± 2.4
BET surface area (m ² /g)	385
ID/IG ratio (Raman)	0.18
C/O atomic ratio (XPS)	23.4
d-spacing (002) (nm)	0.341

Table 2. XRD-derived interlayer spacing and full width.

Graphene Loading (wt%)	Poor Dispersion – d002 (nm)	Poor Dispersion – FWHM (°)	Optimal Dispersion – d002 (nm)	Optimal Dispersion – FWHM (°)
0.1	0.344	0.82	0.338	1.45
0.3	0.345	0.79	0.339	1.52
0.5	0.345	0.74	0.340	1.61
1.0	0.346	0.71	0.341	1.58

Table 3. Mean agglomerate size and area fraction of agglomerates.

Graphene Loading (wt%)	Poor Dispersion – Mean Agglomerate Size (μm)	Poor Dispersion – Agglomerate Area Fraction (%)	Optimal Dispersion – Mean Agglomerate Size (μm)	Optimal Dispersion – Agglomerate Area Fraction (%)
0.1	1.9	0.7	Not detected	<0.1
0.3	4.5	2.3	0.6	0.3
0.5	8.2	4.8	1.1	0.6
1.0	14.6	9.5	2.7	1.4

That hydrophobicity made the graphene group together more in the polar epoxy resin. Once the composites were made, the team checked how the graphene's crystal structure changed inside the matrix using XRD.

As the loading went up in the poor dispersion series, the interlayer spacing changed hardly, and the peak stayed pretty narrow (See Table 2). That basically shows the graphene kept its stacked structure. But in the optimal dispersion series, the peak got noticeably broader, and the d-spacing dropped a bit clear signs that polymer chains slipped between the layers, breaking them apart more effectively. You can actually see this under SEM, which made it possible to measure just how these stacks were holding up.

When dispersion was poor, agglomerates grew

to about 15 μm as the loading hit 1% by weight, taking up over 9% of the cross-section.

But in the optimally dispersed samples, even at the highest loading, the particles usually stayed under 3 μm .

The main way mechanical performance shows up is in the tensile properties, especially strength and modulus, which are the first things we look at.

In the poor dispersion group, tensile strength drops as the content goes past 0.1 wt%, sinking to 47 MPa at 1 wt%—actually lower than what you'd get from pure epoxy. In contrast, with optimal dispersion, strength climbs as the content increases, hitting 84 MPa at 0.5 wt%. Modulus goes up in both groups, but the increase is much steeper with better dispersion. As for elongation, it drops quickly in poor dispersion, but with

Table 4. Tensile strength of neat epoxy and nanocomposites.

Sample	Tensile Strength (MPa)
Neat Epoxy	64.8 ± 2.1
0.1 wt% – Poor Dispersion	67.2 ± 2.5
0.3 wt% – Poor Dispersion	63.5 ± 2.9
0.5 wt% – Poor Dispersion	57.4 ± 3.4
1.0 wt% – Poor Dispersion	46.9 ± 3.8
0.1 wt% – Optimal Dispersion	71.6 ± 2.0
0.3 wt% – Optimal Dispersion	78.3 ± 2.2
0.5 wt% – Optimal Dispersion	84.1 ± 2.5
1.0 wt% – Optimal Dispersion	80.7 ± 2.6

Table 5. Tensile modulus of neat epoxy and nanocomposites.

Sample	Tensile Modulus (GPa)
Neat Epoxy	3.02 ± 0.09
0.1 wt% – Poor Dispersion	3.18 ± 0.10
0.3 wt% – Poor Dispersion	3.35 ± 0.11
0.5 wt% – Poor Dispersion	3.52 ± 0.14
1.0 wt% – Poor Dispersion	3.71 ± 0.16
0.1 wt% – Optimal Dispersion	3.22 ± 0.09
0.3 wt% – Optimal Dispersion	3.49 ± 0.10
0.5 wt% – Optimal Dispersion	3.81 ± 0.11
1.0 wt% – Optimal Dispersion	3.97 ± 0.13

Table 6. Elongation at break of the prepared composites.

Sample	Elongation at Break (%)
Neat Epoxy	5.3 ± 0.4
0.1 wt% – Poor Dispersion	4.8 ± 0.4
0.3 wt% – Poor Dispersion	3.5 ± 0.5
0.5 wt% – Poor Dispersion	2.2 ± 0.4
1.0 wt% – Poor Dispersion	1.4 ± 0.3
0.1 wt% – Optimal Dispersion	5.4 ± 0.3
0.3 wt% – Optimal Dispersion	5.6 ± 0.4
0.5 wt% – Optimal Dispersion	5.8 ± 0.4
1.0 wt% – Optimal Dispersion	4.9 ± 0.4

optimal dispersion, it rises slightly (but notably) up to 0.5%. Fig. 1 shows both strength and modulus together, so it's easier to spot the trends.

You can see the difference between the two dispersion paths in Fig. 1. With just 0.5% by weight loading, the optimally dispersed sample is 46% stronger than the one with poor dispersion.

The modulus goes up in both cases, but the optimally dispersed sample at 0.5% loading

actually hits 3.8 GPa. The weak dispersion doesn't get there at 1%. When it comes to fracture toughness and impact energy, these factors really highlight how much the quality of the dispersion matters.

As loading goes up, the fracture toughness drops when dispersion is poor. That pretty much means those agglomerates start turning into places where cracks begin. But when the dispersion's

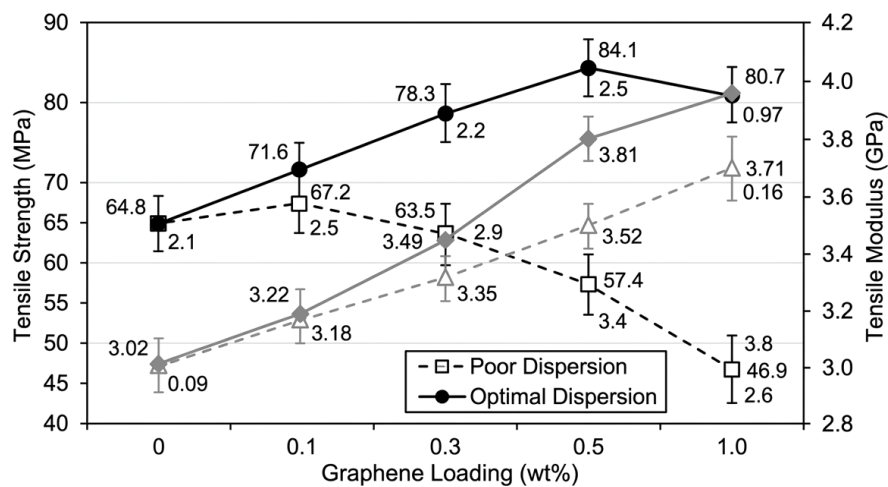


Fig. 1. Tensile strength and tensile modulus versus graphene loading.

Table 7. Mode I fracture toughness (K_{IC}) of the epoxy/graphene systems.

Sample	K_{IC} (MPa·m ^{1/2})
Neat Epoxy	0.61 ± 0.04
0.1 wt% – Poor Dispersion	0.64 ± 0.05
0.3 wt% – Poor Dispersion	0.62 ± 0.05
0.5 wt% – Poor Dispersion	0.55 ± 0.06
1.0 wt% – Poor Dispersion	0.48 ± 0.06
0.1 wt% – Optimal Dispersion	0.73 ± 0.04
0.3 wt% – Optimal Dispersion	0.89 ± 0.05
0.5 wt% – Optimal Dispersion	1.08 ± 0.05
1.0 wt% – Optimal Dispersion	1.01 ± 0.06

Table 8. Unnotched Charpy impact strength of the neat epoxy.

Sample	Impact Strength (kJ/m ²)
Neat Epoxy	12.4 ± 1.1
0.1 wt% – Poor Dispersion	12.9 ± 1.3
0.3 wt% – Poor Dispersion	11.7 ± 1.4
0.5 wt% – Poor Dispersion	10.2 ± 1.5
1.0 wt% – Poor Dispersion	8.6 ± 1.6
0.1 wt% – Optimal Dispersion	15.1 ± 1.2
0.3 wt% – Optimal Dispersion	18.3 ± 1.4
0.5 wt% – Optimal Dispersion	21.7 ± 1.5
1.0 wt% – Optimal Dispersion	19.5 ± 1.6

optimized and you've got 0.5% graphene mixed in, K_{IC} jumps to 1.08 MPa/m² a solid 77% boost compared to plain epoxy. You see the same thing happening with impact energy, too. If you want to see how toughness and ductility stack up against each other, just check out the graph in Fig. 2.

Fig. 2 shows that fracture toughness and elongation increase together up to the 0.5 wt% threshold in the optimum direction. But once the loading hits 1%, they start to split. At this higher loading, toughness stays relatively stable, but elongation drops, so too many particles hurt

elongation, even though toughness holds up.

But once the loading hits 1%, they start to split. At this higher loading, toughness stays relatively stable, but elongation drops, so too many particles hurt elongation, even though toughness holds up.

When samples aren't well dispersed, T_g drops. That suggests free volume forms around the agglomerates, messing up the sintering network. But when graphene's dispersed the right way, T_g goes up. That means the chains near the big graphene surface don't move as easily as before. The storage modulus backs this up, showing the

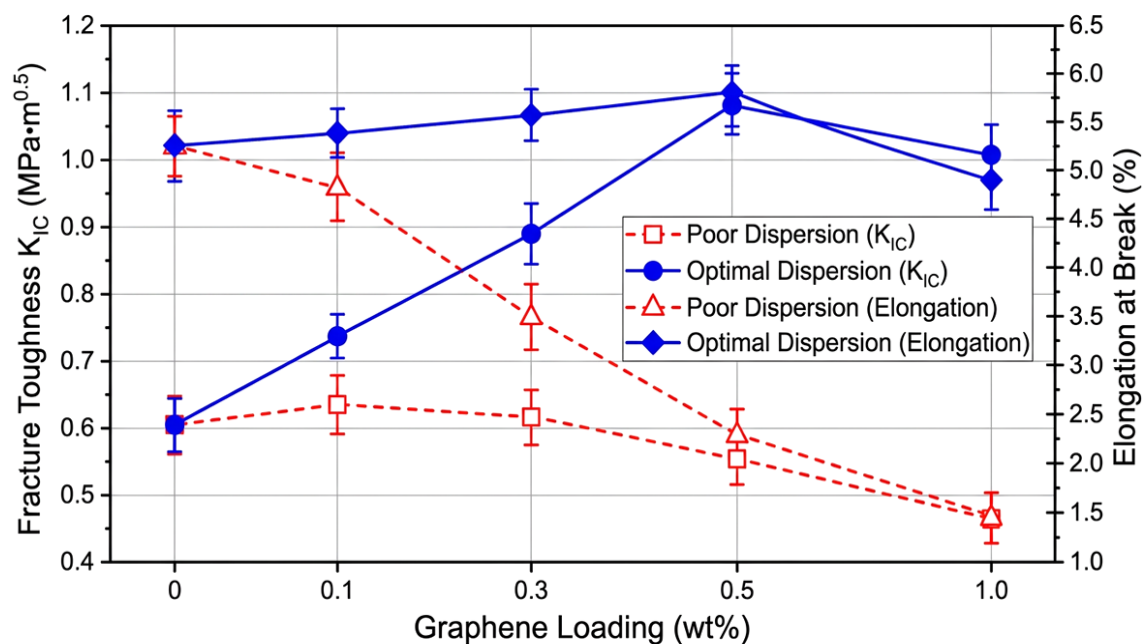


Fig. 2. Fracture toughness and elongation at break.

Table 9. Glass transition temperature (T_g).

Sample	T_g (°C)
Neat Epoxy	131.2 ± 1.0
0.5 wt% – Poor Dispersion	128.5 ± 1.2
1.0 wt% – Poor Dispersion	126.4 ± 1.5
0.5 wt% – Optimal Dispersion	134.7 ± 0.9
1.0 wt% – Optimal Dispersion	135.9 ± 1.1

Table 10. Storage modulus at 30°C.

Sample	Storage Modulus at 30°C (GPa)
Neat Epoxy	2.85 ± 0.07
0.5 wt% – Poor Dispersion	3.23 ± 0.09
1.0 wt% – Poor Dispersion	3.46 ± 0.12
0.5 wt% – Optimal Dispersion	3.64 ± 0.08
1.0 wt% – Optimal Dispersion	3.89 ± 0.10

Table 11. Crosslink density (v_e).

Sample	$v_e \times 10^{-3}$ (mol/cm ³)
Neat Epoxy	2.45 ± 0.12
0.5 wt% – Poor Dispersion	2.31 ± 0.15
1.0 wt% – Poor Dispersion	2.18 ± 0.17
0.5 wt% – Optimal Dispersion	2.52 ± 0.11
1.0 wt% – Optimal Dispersion	2.58 ± 0.13

Table 12. Raman ID/IG and I2D/IG ratios of graphene.

Sample	ID/IG	I2D/IG
0.5 wt% – Poor Dispersion	0.21	0.38
1.0 wt% – Poor Dispersion	0.22	0.35
0.5 wt% – Optimal Dispersion	0.24	0.49
1.0 wt% – Optimal Dispersion	0.25	0.52

Table 13. Relative enhancement of key mechanical properties.

Property	Enhancement (%)
Tensile Strength	+29.8
Tensile Modulus	+26.2
Elongation at Break	+9.4
Fracture Toughness (K_{Ic})	+77.0
Impact Strength	+75.0
Storage Modulus (30°C)	+27.7

same pattern.

When samples aren't well dispersed, T_g drops. That suggests free volume forms around the agglomerates, messing up the sintering network. But when graphene's dispersed the right way, T_g goes up. That means the chains near the big graphene surface don't move as easily as before. The storage modulus backs this up, showing the same pattern.

The ID/IG ratio goes up a bit in the optimized samples compared to the starting powder (which sits at 0.18). That means ultrasonic delamination added a few edge defects. Even so, the I2D/IG ratio is higher, showing off thinner and more evenly spread graphene layers in the mix. That actually helps with stress transfer. In the end, the team compared improvements against pure epoxy, just to see the full impact in the best-case scenario.

Table 13 shows that fracture toughness and impact energy improved the most. That makes sense, since the main toughening comes from crack deflection and graphene layers bridging the gaps. But elongation didn't go up nearly as much. Even when the graphene's spread out perfectly, it's just too rigid as so the matrix can't really stretch

any farther.

CONCLUSION

This study set out to look at two things. How much graphene goes into the epoxy resin and how well the graphene sheets spread out in it. Turns out, both the amount and the quality of dispersion matter a lot. They're related, but each has its own impact. Overlooking either one can mess up your understanding of what the material is truly capable of. If you get the right amount of graphene mixed well, the improvement is serious tensile strength jumps about 30%, and fracture toughness goes up 77% compared to plain epoxy. But if you use the same amount and mess up the mixing, things actually get worse than the unfilled epoxy. Just packing in more graphene isn't automatically better. It's how you spread it around that counts. On a microscopic level, shrinking the size of the clustered graphene bits helps. When the particles stay mostly below 3 micrometers, stress transfer gets way more efficient. That's when toughening mechanisms like cracks veering off course and layers pulling out of the matrix kick in, making the fracture surfaces rougher and

showing clear evidence of the impact. When the graphene clusters are bigger, they act like little stress magnets, breaking the bond between the filler and resin too early and killing the composite's strength before the epoxy's even halfway done.

Tests using DMA backed this up good dispersion makes the polymer network stronger and more stable, upping the crosslink density and locking down chain movement. Agglomerates, on the other hand, mess up the structure by creating extra space and interrupting the curing process. Looking at practical applications, this research pinpoints 0.5 wt% grapheme with excellent dispersion as the sweet spot. That's where you get the best mix of strength, stiffness, toughness, and ductility. Go higher (even with good dispersion), and the filler starts taking over, making things brittle and cutting down on toughness. That tells industries hoping for lightweight, strong, and absorbent parts that piling in more graphene isn't the answer. You can waste money and actually end up with something weaker than the plain material if you miss this point. For moving forward, there's still more to figure out. The study recommends digging deeper into how different types of grapheme size, thickness, surface defects work together with dispersion quality for specific goals. Another gap: how these nanocomposites hold up over time, especially under repeated stress and rough environments. Better online monitoring during curing could clear up how the network forms when graphene's around. And finally, multiscale modeling, teamed up with experimental results like these, could guide future composite designs with more precision letting us leave trial-and-error behind.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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