Quasi-static compression characterization of binary nanoclay/graphene reinforced carbon/epoxy composites subjected to seawater conditioning

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Abstract
Fiber reinforced polymer composite (FRPC) materials are superior to other conventional materials because of their high strength to weight ratio, corrosion resistance, and moisture resistance. FRPC materials are preferred in many high-end applications such as marine, automobile, aerospace, and advanced sporting goods. The aim of this study was to investigate the in-plane quasi-static compressive and durability studies of nanophased FRPC materials. Composite samples were fabricated using unmodified epoxy and epoxy modified with montmorillonite nanoclay (MMT), graphene nanoplatelets (GNP), and a combination of the two as a binary reinforcement with carbon fibers. Quasi-static compression tests were conducted for mechanical property evaluation. Seawater conditioning was performed for a six-month period both at room and arctic cold temperatures. The results indicated that addition of GNP and MMT improved the compressive properties of carbon/epoxy composites compared to unmodified carbon samples. Specific compressive strength and modulus of GNP infused samples improved by 30 and 41% respectively; the samples showed a relatively higher strain to failure than the unmodified samples. Specific compressive strength and modulus increased by 32 and 47%, respectively, for carbon/epoxy samples with MMT reinforcement. Performance of hybrid carbon/glass/epoxy composites was lower compared to other FRPC materials considered in the study. The mode of failure of fractured samples investigated using scanning electron microscopy (SEM) showed a rough morphology after incorporation of nanoparticles into the polymer matrix. This is indicative of enhanced interfacial bonding between carbon/epoxy and the nanoparticles.

1. Introduction
FRPC materials are being considered for high-end applications because of higher strength to weight ratio, improved fatigue performance, higher corrosion resistance, and improved performance under dynamic loading. The reinforcing fiber and matrix are the major constituents in FRPC materials. Mechanical properties of FRPC materials mainly depend on the type of fiber, matrix, and the interface between them. Epoxy polymer resins are preferred as matrix material over other polymers in many high-end applications. A wide range of properties that include excellent resistance to chemical substances, low shrinkage during cure, and low release of volatile contents are the main reasons behind this [1].

Nanoparticles as secondary reinforcement tend to improve the mechanical and thermal properties of matrix material as well as improve the interfacial bond between fiber and matrix [2–6]. Various nanoparticles have been infused with matrix materials to improve the performance of FRPC materials. Han et al [3] found maximum enhancement of interlaminar shear strength (ILSS) at only 0.10 wt% of graphene oxide addition. Yasmin et al [7] also investigated effect of graphene platelets addition on the mechanical and thermal properties of graphite platelet/epoxy composites. 2.5 wt% graphite platelets reinforced composites showed the highest tensile strength compared with the neat and 5 wt% counterpart. However, the cost of graphene is higher than other...
nanomaterials that are being used commonly for FRPC materials. Less expensive organic materials like nanoclay is also popular as secondary reinforcement for FRPC materials [8–10]. Optimum enhancement in thermal and mechanical properties was achieved in case of 2 wt% of nanoclay addition [11].

To ensure the reliability of FRPC materials under various loading conditions, the mechanical responses of materials must be characterized accurately these conditions. Researchers have performed extensive studies on the characterization of FRPC materials under different compressive loading in order to understand the mechanical behavior of these materials [12–16]. However, there is a gap in the literature about MMT, GNP infused carbon/epoxy composites under quasi-static compressive loading.

Environmental factors such as seawater, low temperature, ultraviolet radiation condition can also affect the mechanical behavior of FRPC materials by developing complex state of stresses. Two factors are responsible for the change in properties at low temperatures: (1) increased stiffness of matrix and (2) residual stresses due to different CTE values of matrix and fiber [17]. The damage mechanism also changes when they are exposed to arctic cold temperatures. Elamin et al. [18] found that at low test temperatures, the specimens experienced more severe internal damages than at room temperature by fiber breakage, delamination, interface debonding, and foam core crushing and shearing. As a result, impact strength was decreased at low temperatures with increase in stiffness. Glass and carbon FRP composites also have shown that samples became more brittle when exposed to cold temperatures [19, 20].

There is no work yet reported on the thermal and mechanical characterization of binary nanoclay/graphene reinforced carbon/glass/epoxy hybrid composites subjected to seawater conditioning. Therefore, in this research, five different combinations of FRPC materials had been fabricated: (1) carbon/epoxy, (2) glass/carbon/epoxy, (3) carbon/nanoclay/epoxy, (4) carbon/graphene/epoxy, and (5) carbon/nanoclay/graphene/epoxy. Static compression tests were conducted using the MTS 810 material testing system. Environmental conditioning was performed at three different conditions: room temperature, seawater conditioning for 6 months, and combined seawater/cold temperature conditioning for 6 months period. SEM was used to investigate the damage mechanism of FRPC materials.

2. Materials and methods

2.1. Materials

The polymer used for this study was a diglycidyl ether of bisphenol A (DGEBA) based epoxy resin system, SC-15 supplied by Applied Poleramic Inc. The polymer is a two-part room temperature cure toughened epoxy resin system with low viscosity. The mixing ratio of part A to part B was 10:3 by mass, as specified by the manufacturer for optimum properties. Eight-inch harness satin weave (8HS) carbon fiber mats with density of 0.3 kg m$^{-2}$, 3k tow size, and thickness of 0.46 mm, supplied by US Composites Inc., were used as the reinforcement to fabricate FRPC materials used in the study. The surface of the fibers had been treated with silane for better interfacial adhesion with the matrix. Montmorillonite nanoclay (MMT) sold under the trade name Nanomer I.30E was supplied by Sigma Aldrich, USA. The surface of the MMT had been modified and contained 25–30 wt% octadecylamine. The modification is intended to transform the nanoclay from hydrophilic platelets to hydrophobic so that they can be easily dispersed into resin system. Functionalized graphene nanoplatelets (GNP) were supplied by ‘Cheap Tubes’ Inc., (Vermont, USA). Average thickness of GNP is less than 5 nm and specific surface area about 700 m$^2$ g$^{-1}$. The GNP was covalently functionalized by amine (-NH2) in order to achieve desired degree of dispersion into epoxy polymer matrix. The concentration of functional groups was less than 7%. Industrial seawater used for seawater conditioning was purchased from Doctors Fosters and Smith. The seawater had a pH of 8.5 and specific gravity of 1.027.

2.2. Dispersion of nanoparticles

Proper dispersion of nanoparticles is very important for the improvements in the properties of FRPC materials. Different techniques were used to disperse the nanoparticles in epoxy matrix (figure 1). For nanoclay, 8 grams of MMT and 400 grams of SC-15 resin (2 weight% MMT in epoxy resin), were measured and dried in an oven for 2 h at 100 °C. Specifically, the nanoparticles mixed with 301.54 grams of part A was magnetically stirred at 350 rpm and 40 °C for 3 h. Then, 90.46 grams of part B was added to the mixture that was further mechanically stirred at 800 rpm for 10 min. For graphene, 0.4 grams of GNP, was weighed, added with 307.38 grams of part A (0.7 weight% GNP in epoxy resin), and the mixture was sonicated for 1 h at 40 °C to facilitate the dispersion and minimize the agglomeration. Three roll shear mixture at 3 different gaps was applied. Then the mixture was stirred magnetically stirring at 350 rpm at 40 °C for 3 h before adding 92.22 grams of part B. For the binary MMT/GNP nanoparticles, the dispersion technique was as for GNP for proper comparison.
Figure 1. Dispersion of MMT and GNP with SC-15 epoxy matrix.

Figure 2. Wabash compression molding equipment.

Figure 3. Quasi-static compression testing setup.
2.3. Manufacturing of composite samples

Fabrication of FRPC samples was done using the 8-inch carbon fiber mat as reinforcement and SC 15 reinforced with 0.1 wt% GNP, 2 wt% MMT and a combination of 0.1 wt% GNP and 2 wt% MMT as the matrix. Layers of carbon fiber mats were wet with the hardener mixed resin mixture to fabricate composite laminates using a combination of hand layup and compression mold methods. Ten layers of woven carbon fabrics were manually impregnated individually with a brush and a roller and then stacked together ensuring proper alignment of fiber tows. For the hybrid laminates, four carbon and four glass fabrics with a GGCCCCGG ordering were used. The stack was then wrapped with a porous Teflon sheet, a bleeder cloth, and a non-porous Teflon sheet and placed on the platen of Wabash hot compression molding equipment (figure 2). The temperature was kept at 60 °C for

### Table 1. Contents of the seawater used for conditioning.

| Characteristic                        | Measurement |
|---------------------------------------|-------------|
| pH                                    | 8.3         |
| Specific Gravity                      | 1.027       |
| Appearance                            | Clear       |
| Malodor                               | None        |
| Live Bacteria per Gallon of Saltwater | > 11,000,000|

### Table 2. Obtained strength values from quasi-static compression test for dry samples.

| FRPC samples | Strength (MPa) | Density (g/c.c.) | Specific Strength (MPa/g/c.c.) | Comparison w.r.t Baseline (%) |
|--------------|----------------|------------------|--------------------------------|-----------------------------|
| Neat C       | 171.36 ± 24    | 1.47             | 116.6 ± 16                     | —                           |
| MMT/C        | 232.0 ± 35     | 1.50             | 154.7 ± 23                     | 32.7                        |
| GNP/C        | 220.5 ± 11     | 1.45             | 152.1 ± 8                      | 30.5                        |
| MMT + GNP/C  | 227.1 ± 32     | 1.48             | 153.4 ± 21                     | 31.6                        |
| Hybrid C + G | 124.82 ± 33    | 1.71             | 73.0 ± 19                      | −37.4                       |

### Table 3. Obtained modulus values from quasi-static compression test for dry samples.

| FRPC samples | Modulus (GPa) | Density (g/c.c.) | Specific Modulus (GPa/g/c.c.) | Comparison w.r.t Baseline (%) |
|--------------|---------------|------------------|-------------------------------|-----------------------------|
| Neat C       | 62.21 ± 15    | 1.47             | 42.3 ± 10                     | 47.0                        |
| MMT/C        | 93.3 ± 12     | 1.5              | 62.2 ± 8                      | 41.8                        |
| GNP/C        | 87.02 ± 6     | 1.45             | 60.0 ± 4                      | 37.3                        |
| MMT + GNP/C  | 85.98 ± 15    | 1.48             | 58.1 ± 10                     | −27.2                       |
| Hybrid C + G | 52.68 ± 6     | 1.71             | 30.8 ± 3                      | −27.2                       |
4 h at a pressure of 1 ton as specified by the supplier to attain optimum properties. Composite panels were then post cured in an oven set at 100 °C and maintained for 2 h to obtain completely cured FRPC materials. The thickness of laminates obtained was approximately 3 ± 0.5 mm. Composite test coupons were machined from each panel for various tests according to their respective ASTM standards.

2.4. Quasi-static compression testing
Quasi-static compression tests were conducted using the MTS machine as shown in the figure 3. at a speed of 1.27 mm min⁻¹. The effect of nanomaterial addition on the compressive properties of composite materials were determined. The sample size of 12.7 × 12.7 mm was used for all the tests. In-plane loading was applied to the samples. The width and thickness of the samples were measured and recorded. The test specimen was placed between the surfaces of the compression tool and the crosshead of the testing machine was adjusted. The compression test was started at the specific crosshead speed. For each sample, the compressive strength, compressive yield strength, offset yield strength, modulus of elasticity, mean values, and standard deviations were calculated. The stress-strain curve was obtained from the raw data to understand the compressive response of the materials.
2.5. Scanning electron microscopy (SEM):
Fracture morphology and failure mechanism of the damaged FRPC samples were studied using field emission scanning electron microscope (FE-SEM) (JOEL JSM -7200F). Surfaces of failed samples were initially sputtered with thin layer of gold-palladium under vacuum prior to SEM imaging. Several SEM micrographs for analysis were obtained from each sample at various magnifications.

2.6. Environmental conditioning
Samples were conditioned to determine the seawater effects. The contents of the seawater used in this study is shown in the table 1.

Initial weights of selected sample were measured and recorded as baseline, and after 6 months of complete immersion in seawater both at room temperature and at a cold temperature (−20 °C), in order to compare weight changes as function of exposure time. A refrigerator was used for the cold temperature conditioning. Mechanical and thermal properties of the conditioned FRPC materials were characterized. Figure 4 illustrates a typical setup for conditioning the samples.
The water absorption of the conditioned samples was calculated using the following equation:

\[
\% \text{ water absorption} = \left( \frac{W_f - W_i}{W_i} \right) \times 100\
\]

(1)

where, \( W_i \) and \( W_f \) are the initial and final weight of the films before and after conditioning.
3. Results and discussions

3.1. Quasi-static compression test

3.1.1. Dry samples

Quasi-static compressive properties of the composites were collected from the experiments. For the baseline neat carbon/epoxy samples, the average specific compressive strength was found to be 116.6 MPa g$^{-1}$/c.c. and the modulus was found to be 42.3 GPa g$^{-1}$/c.c. When combined with glass fiber, the specific compressive strength and specific modulus were found to be 73.0 MPa g$^{-1}$/c.c. and 30.8 GPa g$^{-1}$/c.c., respectively. FRPC samples modified with graphene nanoplatelets (GNP) seemed to improve the specific compressive strength by 30.5% and modulus by 41.8% compared to the neat carbon samples. Whereas, binary montmorillonite nanoclay (MMT) and GNP modified FRPC samples showed a 31.6% and 37.3% increase in specific strength and modulus, respectively, compared to the neat carbon samples. Among the five types of FRPC materials, MMT/carbon samples showed the best compressive properties for in-plane quasi-static compression tests. An increase of 32.7% was observed in case of specific strength. Specific modulus was improved by 47%. Tables 2 and 3 represent the obtained values from the quasi-static compression tests.

Figure 11. Rate of water absorption for 6-month seawater conditioned FRPC materials.

Table 5. Obtained modulus values from quasi-static compression test for 6-month seawater conditioned samples.

| FRPC samples | Modulus (GPa) | Density (g/c.c.) | Specific Modulus (GPa/g/c.c.) | Comparison w.r.t Baseline (%) |
|--------------|---------------|------------------|-----------------------------|-----------------------------|
| Neat C       | 71.5 ± 7      | 1.59             | 45.1 ± 4                    | —                           |
| MMT/C        | 89.5 ± 12     | 1.7              | 52.7 ± 7                    | 16.9                        |
| GNP/C        | 90.7 ± 14     | 1.6              | 52.2 ± 8                    | 15.7                        |
| MMT + GNP/C  | 97.1 ± 10     | 1.68             | 57.8 ± 5                    | 28                          |
| Hybrid C + G | 54.5 ± 21     | 1.8              | 30.7 ± 13                   | −31.9                       |

Table 6. Comparison of water uptake of different FRPCs after seawater conditioning for 6-month period.

| FRPC samples | Unconditioned Weight (mg) | Weight After Conditioning SW-6 (mg) | % Change |
|--------------|---------------------------|------------------------------------|----------|
| Neat C       | 873.08                    | 877.67                             | 0.53     |
| MMT/C        | 1060.57                   | 1064.19                            | 0.34     |
| GNP/C        | 883.36                    | 886.51                             | 0.35     |
| MMT + GNP/C  | 783.59                    | 786.12                             | 0.33     |
| Hybrid C + G | 806.95                    | 810.219                            | 0.4      |

3.1.1. Quasi-static compression test

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From the graphical representation (figures 5, 6, 7), it was also clear that MMT/carbon samples showed increased performance compared to the other types of samples. Both the specific strength and modulus were...
improved with MMT modification. Hybrid carbon/glass composites showed lower properties than the baseline unmodified carbon samples.

3.1.2. Seawater conditioning
Nanoparticles seem to play important role in the case of seawater conditioned samples. MMT/carbon samples showed increments of 49.7% and 16.9% in specific strength and modulus, respectively, compared to the neat carbon samples. The GNP/carbon and binary MMT/GNP/carbon also showed a significant improvement in specific strength and modulus compared to the unmodified carbon samples (figures 8, 9, 10). Nanoparticles improve the interfacial bonding between fiber and matrix. Therefore, adding nanoparticles improved the compressive properties of FRPC materials. Tables 4 and 5 represent the obtained values from the quasi-static compression tests of FRPC materials subjected to seawater conditioning.

The rate of water absorption was also lower in case of nano-phased FRPC materials (figure 11). Incorporation of MMT, GNP, and binary MMT/GNP showed less water absorption compared to the unmodified samples. MMT/carbon showed 35.85% less water absorption compared to the unmodified samples. Similarly, GNP/carbon and binary MMT/GNP/carbon showed 33.97% and 37.74% less water absorption, respectively, compared to unmodified counterpart. The reason behind this might be that the
entrance of water is hampered by nanoparticles. Table 6 represents the data results obtained from moisture absorption studies.

3.1.3. Combined seawater and cold temperature conditioning
Combined seawater and cold temperature conditioning for 6-month period affected the compressive properties of FRPC materials. Addition of nanoparticle improved the compressive strength and modulus compared to the unmodified neat carbon samples (figures 12, 13). However, the nano-phased samples showed sudden failure after reaching the ultimate stress level. The cold temperature and seawater conditioning adversely affected the FRPC materials by building complex state of stresses. Therefore, the samples showed sudden fracture even though the compressive properties showed improvement (figure 14). Tables 7 and 8 represent the obtained values from the quasi-static compression tests of FRPC materials subjected to combined seawater and cold-temperature conditioning.

The rate of water absorption was also lower in case of nano-phased FRPC materials compared to the unmodified samples as shown in figure 15. The unmodified carbon samples showed water absorption of 0.21% after 6 months combined conditioning. Whereas, MMT/carbon, GNP/carbon, and binary MMT/GNP/carbon showed water absorption.
absorption of 0.14, 0.17, and 0.08%, respectively. Table 9 represents the data results obtained from moisture absorption studies.

**3.2. Comparison in terms of conditioning**

Table 10 and 11 represents the variation of compressive properties of different combinations of FRPC materials at dry, seawater, and combined seawater and cold temperature conditions. FRPC materials undergo
microstructural changes in presence of seawater and cold temperature. Therefore, FRPC materials show a different behavior when subjected to seawater conditioning. For seawater conditioning at room temperature for a six-month period, the compressive strength decreased with respect to dry samples.

Seawater and cold temperature might affected the interfacial bonding between fibers and matrix. Therefore, modulus, strength, and water absorption values varied with different materials and conditioning parameters. The seawater and cold temperature response of MMT infused samples showed better properties compared to GNP and MMT/GNP samples because of enhanced cohesiveness of the epoxy with MMT.

3.3. SEM analysis

The SEM images showed clearly the improvement of FRPC materials by adding nanoparticles. In case of unmodified carbon sample, the fiber surface seems to have smoother surface compared to the modifications. However, rougher surfaces were observed in case of nanoparticle modification, indicating good interfacial bonding between the fiber and matrix (figure 16). The binary MMT/GNP seems to show lower strength compared to the MMT modification due to intercalation with binary nanoparticles.

In case of hybrid carbon/glass samples, lack of interaction and intercalation decreased the properties of hybrid samples. Rougher surfaces were observed in SEM, but more voids and intercalation deteriorate the interfacial bonding between fiber and matrix. Figure 17 shows the effect of seawater conditioning in the fractured samples. In case of dry samples, the interfacial bonding between fiber and matrix was stronger. However, due to detrimental effect of seawater conditioning, the bonding between fiber and matrix was weakened.

Figure 17 shows the effect of seawater conditioning in the fractured samples. In case of dry samples, the interfacial bonding between fiber and matrix was stronger. However, due to seawater conditioning, fiber and matrix bonding weakened.

Combined seawater and cold temperature conditioning also deteriorated the properties of FRPC materials (figure 18). FRPC materials show more severe damage with mostly delamination in case of combined seawater and cold temperature conditioning. However, dry samples showed fracture by mostly fiber breakage and delamination.

![Figure 16. SEM images of (a) neat carbon, (b) MMT/carbon, (c) GNP/carbon, (d) MMT/GNP/carbon.](image-url)
Seawater and cold temperature make the bonding between fiber and matrix weaker. Therefore, FRPC materials show more severe internal damage after 6 months conditioning in seawater and cold temperature.

4. Conclusion

Modification of epoxy matrix with montmorillonite nanoclay (MMT) and graphene nanoplatelets (GNP) was found to improve the compressive and dynamic mechanical properties of fiber reinforced composite polymer (FRPC) materials. However, the addition of glass fibers with carbon fibers seems to deteriorate the compressive properties.

- The MMT/carbon samples seem to provide the best performance in terms of the compressive strength and modulus among all other FRPC samples. Rough fiber and matrix fracture surfaces indicate the interfacial bonding between the fiber and matrix was improved with the MMT modification.
- The GNP modified FRPC samples improved the compressive strength and modulus, but that is less than the improvement with MMT modification. The dispersion process of GNP into the matrix might be modified to improve the interfacial bonding. In this research, only 0.1% GNP was used; this might also be changed to improve the compressive properties.
- Binary MMT/GNP increased the compressive strength better than that with GNP modification but less than that with MMT modification. However, in case of modulus, the improvement was similar to that for the modification with MMT.
• Hybrid carbon/glass composites exhibited lower compressive strength and modulus with respect to other combinations FRPC materials. The reason behind this might be the poor interaction and bonding between the interphases.

• MMT, GNP, and MMT/GNP incorporation decreased the percent water absorption after seawater conditioning at room and cold temperature for a 6-month period. Addition of nanoparticle reduces the tendency of water absorption. Nanoparticles might improve the properties by making matrix more coherent, reducing the heterogeneity. Therefore, bonding between the fiber and matrix was enhanced.

• Seawater conditioning affects the interfacial bonding between the fiber and matrix. Therefore, after a 6-month seawater conditioning at room temperature, addition of nanoparticles did not improve the properties in comparison with unconditioned samples.

• Combined seawater and cold temperature conditioning develop a complex state of stresses among the FRPC materials. Although the strength and modulus increased after nanoparticle reinforcement, samples showed brittle fracture with severe damage.

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