

**NRICON 2016**

---

## **COMPRESSIVE, FLEXURAL AND TENSILE PROPERTIES OF GRAPHENE MODIFIED GROUTS FOR PIPELINE REHABILITATION**

Umi Soleha Salim<sup>1</sup>, Alireza Valipour<sup>1</sup>, Siti Nur Afifah Azraai<sup>1</sup>, Lim Kar Sing<sup>1,2</sup>, Libriati Zardasti<sup>1\*</sup>, N.M. Noor<sup>1</sup> & Nordin Yahaya<sup>1</sup>

<sup>1</sup> Faculty of Civil Engineering, Universiti Teknologi Malaysia, 81310, Johor, Malaysia

<sup>2</sup> Faculty of Civil Engineering and Earth Resources, Universiti Malaysia Pahang, Lebuhraya Tun Razak, Gambang, Kuantan, Pahang 26300, Malaysia

\*Corresponding Author: *libriati@utm.my*

---

**Abstract:** This experimental study consists of the characterization of the epoxy grout where graphene nanoplatelets were added to commercial neat epoxy grout at an amount of 0.2%, 0.5%, 0.8% and 1.0% to evaluate their behaviour regarding neat epoxy grout. In order to study the characteristics of each type of grout epoxy, three tests were conducted which are compressive test, tensile test, and flexural test. By comparing graphene-based and neat epoxy grout, there is no significant increase in strength due to the weak interface in the graphene nanoplatelets/epoxy composite. Moreover, the tensile strength of tensile and compressive strength of modified grout epoxy is much lower than neat epoxy grout. Nevertheless, graphene modified grout showed more consistent results according to a smaller standard deviation of strength. In addition, modified grout epoxy shows an improvement in the ductility of the grout hence reducing the brittle behavior of the grout. With this result, the performance of graphene-based grout is able to minimize the sudden rupture and this is important due to repair design of damaged pipeline is of deterministic nature.

**Keywords:** *Epoxy Grout, Graphene Nanoplatelets, Corrosion, Pipeline*

### **1.0 Introduction**

The oil and gas industry uses steel pipeline to transport the crude oil and natural gas from the oil platform to the processing plant. Pipelines system is one of the most important parts of energy transportation infrastructure to the country. The location of these pipelines is usually located underneath inside the sea and underground. These pipelines are coated with protective coating and cathodic protective system, supplement by periodic inspection equipped with sensor for inspection (Matheswaran and Ramsay, 2010). These pipelines are subjected to deterioration such as third party damage, natural forces, material and construction defects and corrosion (Noor *et al.*, 2010; Yahaya *et al.*, 2009; Shamsuddoha *et al.*, 2013). The general damage in oil and gas industry is

corrosion, which is caused by the instantaneous actions of some rhetorical behavior of the crude oil (Mariem-Benziane and Zahloul, 2013). According to Ashraf *et al.* (2016), the corrosion can be grouped into two categories; uniformed and localized corrosion. The corrosion estimation is required to determine the allowable corrosion for structural designs, inspection planning and maintenance scheduling (Wang *et al.*, 2011). Corrosion pipeline is a deleterious defect, the loss of metal in pipeline cause the pipeline to loss in strength hence the resistant become lower and it will burst at lower pressure. This event can be preventing by repair with composite wrap to reinforced the pipeline and avoid bursting. Composite wrap worked as an external reinforcement towards the pipeline that is applied at the defected area especially around corroded area of pipeline. Composite wrap is made up of two components, which is fiber-reinforced polymer (FRP) and resin epoxy. Fiber reinforced polymer has a high strength property which can withstand the internal pressure of a pipe. Epoxy resins are widely used as matrices of composites because it has unique characteristics such as high stiffness, high adhesion strength and low shrinkage in cure.

In repairing damaged pipelines, epoxy grouts are widely used as infill material to provide a smooth surface for the composite wrapping and provide a continuous support to minimize the outward distortion. Epoxy grouts play an important role of transferring the load from pipe to the composite to increase the load resistance of the structures. In order to predict the behavior of a repair system for an optimum design, the properties of the infill material play a significant parameter. Repair efficiency may be increase with the high performance infill material if it can be serve as second protection layer if failure of composite occurs. Numerous studies have been done in order to enhance the properties of infill material. The enhancement of mechanical properties can be done through the incorporation of filler such as carbon nanotubes, graphene nanoplatelets, nanofibers, nanosilica, nanoalumina and others. The recent research of inclusion of graphene nanoplatelets for use as nanofillers in epoxy resin is being studied but their effect on the mechanical properties is not very clear yet. Therefore, it is essential to characterize the mechanical properties of graphene/epoxy grout in determination of their efficiency as infill materials in the pipeline repair. Hence, this study has conducted to investigate the compressive, tensile and flexural properties of graphene/epoxy grout to be used as infill material.

## **2.0 Materials and Methods**

### *2.1 Materials*

In this study, three-part pourable epoxy grout was used which it is consists of modified epoxy resin, hardener and fine silica sand. The properties for the choose resin is 14 N/mm<sup>2</sup> in tensile strength, 100 N/mm<sup>2</sup> in compressive strength and 20 N/mm<sup>2</sup> in flexural strength with the manufacture recommendation of mixing ratio of 2:1:12 parts by weight.

This epoxy resin is a common used in industrial for grouting and filling. The graphene nanoplatelets were selected as a filler material to improve the strength and efficiency of infill material. Graphene nanoplatelets have strength 200 times than steel and its thickness about 0.68-3.41 nm averagely.

## 2.2 Sample Preparation

The graphene-based epoxy grouts were prepared as the suggested manufacture instruction. Firstly, weight the epoxy resin, hardener and silica according to manufacturer's recommendation. After undergo the dispersion method that used for preparation of graphene based epoxy, where specified weight percentage of graphene nanoplatelets (0.2%) in hardener and mix continuously using high speed electrical mixer for about 20 minute to get a homogenous suspension. Next, epoxy resin was added to the mixture and mixing process continues until a smooth consistency paste is obtained. When the graphene filler and resin was homogeneously mixed, silica sand was added and all parts were mixed until homogeneous grout was produced. After the three parts was completely mixed, the mixture was poured into designated molds and cured for 24 hours at room temperature. The process was repeated with 0.5%, 0.8% and 1.0% graphene nanoplatelets and neat epoxy resin. The process of mixing modified graphene based epoxy is shown in Figure 1.

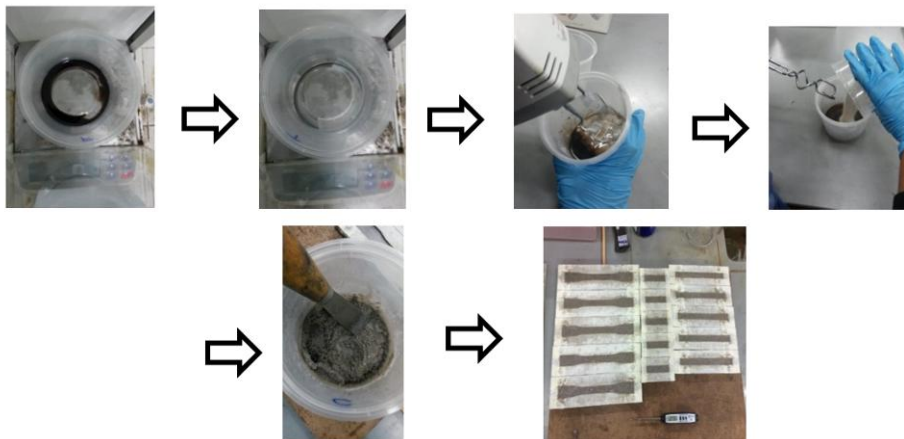


Figure 1: Process of mixing modified graphene based grout

### 2.3 Compressive Test

According to ASTM D695, this test required five (5) specimens with dimension 12.7 mm x 12.7 mm x 50.8 mm for each specimen. A 25 kN universal testing machine (INSTRON) was used to carried out this test. For each specimen, a load was imposed at a constant speed of 1.3 mm/min. The machine used for this test is shown in Figure 2. The calculation of compressive strength results by using equation (Eq. 1):

$$C.S = \frac{P}{b.d} \quad (1)$$

where:

C.S = compressive strength, N/mm<sup>2</sup>

P = maximum load, N

b = width of specimen, mm

d = thickness of specimen, mm



Figure 2: Compression test

### 2.4 Tensile Test

The tensile test was performed on the standard test accordance to ASTM D638 where five (5) specimens are needed with 13.0 x 3.2 mm dimensions. The specimen was undergoing tension where the specimen was pull at both ends with constant speed of 5 mm/min at room temperature. The results of tensile strength and modulus are obtained from the tensile curves. The material toughness can be determined from the area under the tensile curves. The machine used for carried test is shown in Figure 3.



Figure 3: Tensile test

### 2.5 Flexural Test

This test was done according ASTM D790 guidance, where five (5) specimens required with dimensions of 127 mm x 12.7 mm x 3.2 mm were tested under two-point load using flexural testing machine as shown in Figure 4. The test was conducted at room temperature with a constant cross speed of 1.365 mm/min. From the test, bending strength ( $\sigma_f$ ) and modulus ( $E_B$ ) will be gain according to equation (Eq. 2) and (Eq. 3):

$$\sigma_f = \frac{3.P.L}{2.b.d^2} \quad (2)$$

$$E_B = \frac{m.L^3}{4.b.d^3} \quad (3)$$

where:

$\sigma_f$  = flexural strength, N/mm<sup>2</sup>

P = load at a given point on the load-deflection curve, N

L = support span, mm

b = width of beam tested, mm

d = depth of beam tested, mm

m = the slope of the tangent



Figure 4: Flexural test

### 3.0 Results and Discussion

#### 3.1 Mechanical Properties of Neat Epoxy Grout

Neat epoxy grout is the commercial infill that usually used in the composite wrap for pipeline repair. The mechanical properties that were investigated are compressive, flexural and tensile strength. Table 1 tabulated the summary of mechanical properties of neat epoxy grout. Figure 5 shows the neat epoxy grout has high compressive strength capability with the maximum load of 78.6 MPa and has higher strain capability under compressive load. From Figure 6, it can be seen that neat epoxy grout has high tensile strength of 19.86 MPa but it shows a sudden failure in tensile after reach the ultimate tensile strength. This shows that the neat epoxy grout does not have high resistance in tensile compare to compressive and it is brittle in tensile load. Figure 7 shows the flexural load of the neat epoxy grout and based on the graph, neat epoxy grout able to resist higher loading and more ductile under flexural load compare to compressive load.

Table 1: Summary of Neat Epoxy Properties

<i>Grout</i>	<i>Compressive Strength (MPa)</i>	<i>Tensile Strength (MPa)</i>	<i>Flexural Strength (MPa)</i>
<i>Neat epoxy</i>	78.619 ± 21.25	19.862 ± 9.23	32.885 ± 6.50

#### 3.2 Mechanical Properties of Modified Graphene Based Epoxy Grout

Four specimen of modified graphene based epoxy were studied under flexural, compressive and tensile test with different percentage of graphene. Table 2 shows the summary of the modified graphene grout based properties for compressive strength,

tensile strength and flexural strength. Under compressive loading, Figure 5 shows that 0.8% of graphene modified epoxy grout able to withstand high compressive load and high compressive modulus compare to 1% of graphene modified epoxy grout that have low capability in compressive strength and compressive modulus. Figure 6 shows the result of tensile test showing the 0.5% of graphene based epoxy grout have the highest tensile modulus but it has the lowest tensile strength. However, 0.8% graphene based epoxy have highest tensile strength and lowest tensile modulus which is vice versa of the 0.5% of graphene based epoxy. Furthermore, flexural test shows all the four specimen of modified based graphene epoxy behave almost the same as each specimen. The specimen does not have a distinguish difference in term of flexural strength or flexural modulus.

Table 2: Summary of the Modified Graphene Based Epoxy Properties

<i>Grout</i>	<i>Compressive Strength (MPa)</i>	<i>Tensile Strength (MPa)</i>	<i>Flexural Strength (MPa)</i>
<i>0.2% modified graphene based epoxy</i>	$56.090 \pm 11.34$	$9.628 \pm 4.64$	$35.905 \pm 1.42$
<i>0.5% modified graphene based epoxy</i>	$53.154 \pm 13.56$	$7.111 \pm 6.23$	$32.946 \pm 6.01$
<i>0.8% modified graphene based epoxy</i>	$72.744 \pm 14.69$	$11.937 \pm 6.47$	$34.849 \pm 7.38$
<i>1.0% modified graphene based epoxy</i>	$18.151 \pm 5.91$	$9.735 \pm 7.26$	$68.852 \pm 12.24$

### 3.3 Relationship of Strength and Ductility Between Neat Epoxy Grout and Graphene Based Epoxy Grout

Under the compressive load, the neat epoxy grout has the highest advantage as it exceeded the strength and ductility under compression. Due to this result, the graphene based epoxy grout does not contribute in improving the epoxy under compression. In comparison to that, tensile test epoxy based graphene grout shows a significant difference, where the neat epoxy grout shows that it is brittle under tensile as the epoxy based graphene managed to increase the ductility of the epoxy grout. A study from Yasmin *et al.* (2003), explained that the reduction in tensile strength of graphene based epoxy is due to the efficiency of dispersion method used in graphene to produce one layer of graphene. Last but not least under flexural load, the differences of strength and ductility can be seen to be small and the behavior of neat epoxy and graphene based epoxy is to be almost the same. In general, graphene does not help in enhancing the compression and tensile strength of epoxy grout rather than that graphene decrease the strength of epoxy grout in term of tension and compression. This behavior is highly

probably due to the dispersion method that was used to disperse the graphene (Tjong, 2011).

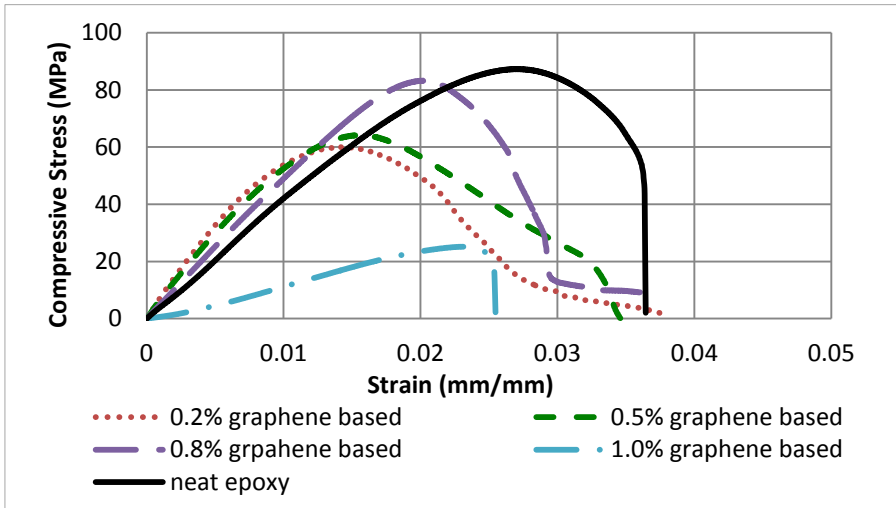


Figure 5: Relationship of stress-strain for compressive strength

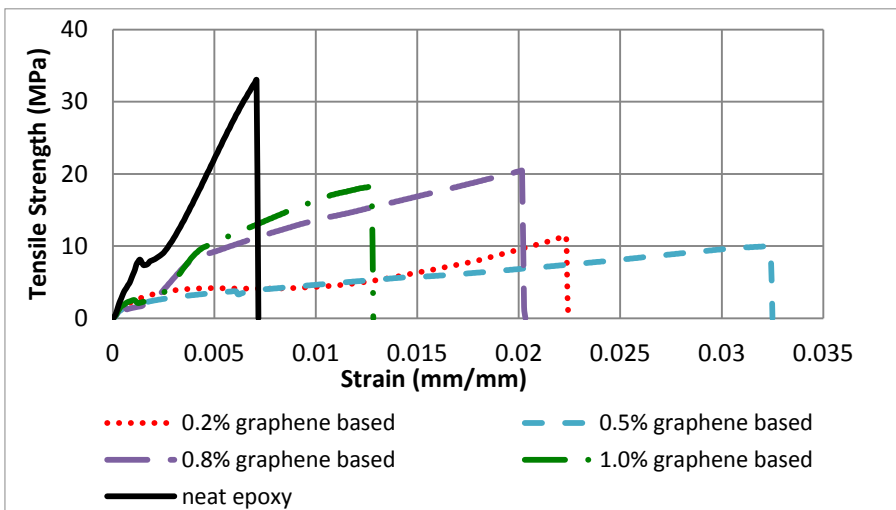


Figure 6: Typical stress-strain behaviors of tensile specimens



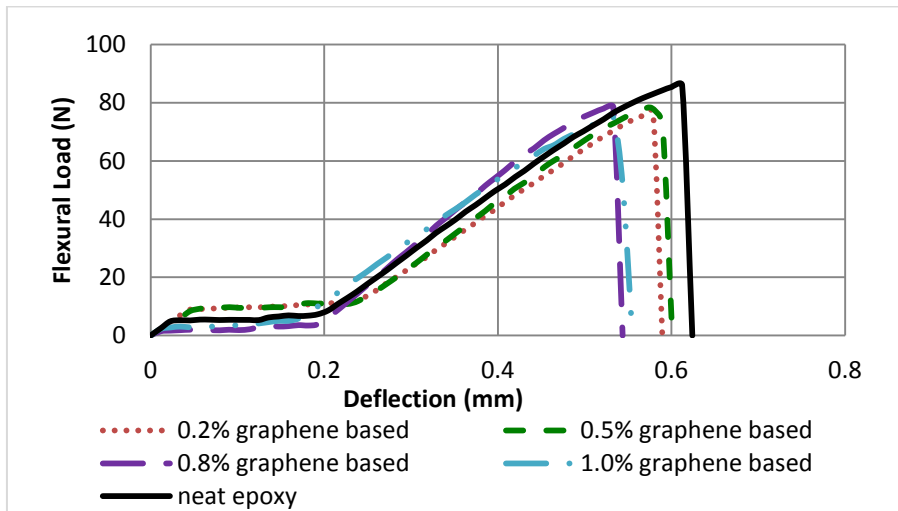


Figure 7: Load-deflection for flexural test

#### 4.0 Conclusions

The mechanical properties of neat epoxy grout and graphene modified epoxy grout were investigated based on experiments work. Based on this study, the following conclusions can be drawn:

1. Neat epoxy has weak properties in terms of tensile test, the strength is high but it shows brittle performance. Ductility and stiffness in flexure is good. Neat epoxy shows similar behavior with concrete in compressive test, which is high in strength and strain capacity.
2. Higher percentage of graphene has much better strength and ductility. 0.5% modified graphene based epoxy is found to have well performance in tensile test compare to other graphene-modified epoxy.
3. Although neat epoxy is high in strength but modified graphene based is found more ductile than neat epoxy under tensile loading.

#### 5.0 Acknowledgements

The work was financially supported by Universiti Teknologi Malaysia (Grant No. 06H50 and 02G48), and the Ministry of Education Malaysia, MOE (Grant No. FRGS 4F530).

## References

- Ashraf, T. M., Abo-Dief, H. M., and Alzahrani, E. (2016). *Inhibition of Pipelines Corrosion Using Natural Extracts*. World Academy of Science, Engineering and Technology International Journal of Chemical, Molecular, Nuclear, Materials and Metallurgical Engineering Vol:9.
- Matheswaran and Ramsay (2010). *Inhibition of the Corrosion of Mild Steel and Aluminum in Acidic Media by Some Purines*. World Wide Science.
- Meriem-benziane, M. and Zahloul, H. (2013). *Effect of Corrosion on Hydrocarbon Pipelines*. World Academy of Science, Engineering and Technology, 7(3), 252–254.
- Noor N.M.; Yahaya, N.; Ozman, N.A.N. & Othman S.R. (2010). The forecasting residual life of corroding pipeline based on semi-probabilistic method. *UNIMAS e-Journal of Civil Engineering*. 1(2), 1-6.
- Shamsuddoha, M., Islam, M. M., Aravinhan, T., Manalo, A., and Lau, K. T. (2013). *Characterization of mechanical and thermal properties of epoxy grouts for composite repair of steel pipelines*. Material and Design, 52, 315-327.
- Tjong, S. C. (2011). *Polymer nanocomposite bipolar plates reinforced with carbon nanotubes and graphite nanosheets*. Energy Environment. Sci. 4, 605–626.
- Wang, Y., Zhao, Y., Liu, W., and Zheng, H. (2011). *Improvement in Mechanical Properties of Modified Graphene / Epoxy Nanocomposites*. ICCM18, 862, 1–5.
- Yasmin, A., Abot, J. L., and Daniel, I. M. (2003). *Characterization of structure and mechanical behavior of clay/epoxy nanocomposites*. In: Proceedings of the 14th international conference on composite materials. San Diego, CA.
- Yahaya, N.; Norhazilan N.M.; Din M.M. & Nor S.H.M. (2009). Prediction of CO<sub>2</sub> corrosion growth in submarine pipelines. *Malaysian Journal of Civil Engineering*. 21(1), 69-81.