[Research Paper]

Blister Test to Evaluate the Multiwall Carbon Nanotubes (MWCNT) - Woven Carbon Fiber Reinforced Epoxy used for Repairing Pipelines

Abstract

Purpose:

Pipelines are subject to pits, holes, and cracks after staying in service for a while, especially in harsh environments. To repair the pipelines, composite materials are used, due to their low cost, high corrosion resistance, and easy handling.

Design/methodology/approach

This work investigates the reliability of the blister test for evaluating the bonding strength of multiwall carbon nanotube (MWCNT) on woven carbon reinforced epoxy. Flexural, hardness, and Izod impact tests were used to evaluate MWCNT effect on the epoxy by adding different amounts, 0.2, 0.4, 0.6, 0.8, and 1 wt. %, of MWCNT, to be compared with pure epoxy.

Findings

The results showed that 0.8 wt.% gives the highest strength. The experimental results of 0.8 wt.% MWCNT reinforced carbon composite was compared with the finite element model under blister test, and the results showed high similarities.

Originality

Evaluation of the reliability and the advantages of MWCNT considering the high aspect ratio and high tensile strength which is more than 15 times compared to steel, MWCNT enhances the strength, stiffness and toughness of epoxy used as a matrix in repairing pipelines which leads to an increase in the resistance of composite materials against oil internal pressure, before delamination.

Keywords: MWCNT, carbon reinforced epoxy, bister test, finite element analysis, pipeline, composite repair, woven reinforcement, nano reinforcement.

1. Introduction

Pipelines are subject to pits, holes, and cracks after staying in service for a while, especially in harsh environments. To repair the pipelines, composite materials are used, due to their low cost, high corrosion resistance, and easy handling (Budhe *et al.*, 2017), (Nariman, 2015), in addition, to retrieving the bending and tension stiffness of the pipelines resulting from high internal pressure (De Barros *et al.*, 2019). Aramid, glass, and carbon-reinforced polymer composites are the main materials used for pipeline repairs (Mahdi and Eltai, 2018).

Composite materials used to repair pipelines commonly uses Epoxy as a matrix. Epoxy is a thermoset polymer, that turns amorphous and crosslinked upon curing, having outstanding properties, such as comparatively high strength, stiffness, and hardness (Wetzl *et al.*, 2006). However, some curing treatments lead to the form of epoxy with low fracture toughness, and low resistance to crack initiation and propagation (Garg and May, 1988). For this reason, adding fillers has been used to improve the fracture toughness of the matrix (Li *et al.*, 2015). Carbon NanoTubes (CNT), which were found around thirty years ago, has a hexagonal arrangement made from carbon atoms bonded by covalent bonds forming a single atomic layer sheet wrapped up on itself forming a tube. These nanotubes are either made of a single-layer SWCNT (single-walled carbon nanotube) or multilayer MWCNT (multi-walled carbon nanotube). CNTs are incorporated with other materials to enhance their properties like strength, durability, and improving crack propagation resistance when added to the resin. Moreover, improving the delamination resistance in glass fiber reinforced composites (Alberto *et al.*, 2018).

Dannenberg (Danberg, 1961) was the first who proposed the blister test to evaluate the adherence of polymeric coatings to metals. In a typical blister inspection, the tested sample should be made up of a coating stuck over a metallic base with a round puncture placed at the center. A fluid is browbeating across the puncture as opposed to the bottom of the coating, resulting in debonding in the coating from the base and creating a blister (Taheri *et al.*, 2000). The coating/base interface adherence energy can be calculated from the dimensions of the blister and the subjected pressure. During the test, the coating may fracture before debonding from the base or a tragic debonding which usually takes place at critical pressure which is considered the major drawback of this standard procedure.

A blister test was improved by Malyshev and Salganik (Malyshev and Salganik, 1965) where a shaft-loaded blister was used, as the load is applied on the puncture by a shaft to cause delamination at the coating/base interface, as shown in Fig. 1 (De Barros et al., 2019). Chen et. al. (Chen et al., 2014) found out that this procedure is easier than the pressurized procedure.

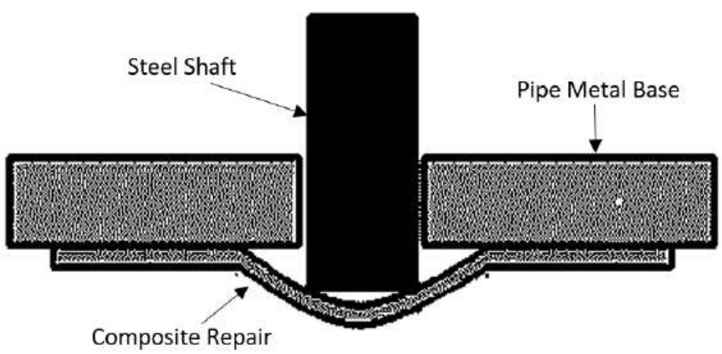


Fig. 1. Blister test arrangement, source: Author's own creation.

When a little leaking flaw occurs, the pressure creates blisters below the composite repair layer. If the adherence is frail, a crack will grow in the interface between the composite and the pipe until it reaches the border of the composite, which results in the formation of more leaking. A well-prepared surface and a good matrix are more important than the fiber content (Linden et al., 2016).

Different arithmetic models were derived to represent the adherence stiffness of composite repair using blister which is carried out through a point force or pressure loading (Sofla *et al.*, 2010) (Malyshev and Salganik, 1965) (Wang and Tong, 2016). The mechanical resistance of brazed juncture of metal with ceramic materials was measured by using the blister test (Kozlova *et al.*, 2008). The elastic strain energy was presented theoretically by evaluating the composite repair/metal base interface (Sun *et al.*, 2013). Adherence to interfacial failure of composite repair to pipe metal base was evaluated by shaft-loaded blister tests.

Calculation of the overall energy release rate G_T is calculated by a load of failure F, eq. (1) (De Barros *et al.*, 2019).

$$G_T = \frac{F^2}{32\pi^2 D} \ldots\ldots (1)$$

were:

D is the stiffness of bending in GPa measured depending on the characteristics of the laminate

E is Young's modulus in GPa.

ν is the Poisons ratio,

t is the plate thickness in mm, D can be calculated in eq. (2), as below (De Barros *et al.*, 2019):

$$D = \frac{Et^3}{12(1-\nu^2)} \ldots.. (2)$$

Zugliani et al. (Zugliani *et al.*, 2019) developed a method of repairing pipes with bonded joints by wrapping glass fiber reinforced polymer around the pipe which became standard practice in the sector. A shaft blister test was utilized by De Barros et al. (De Barros *et al.*, 2019) to evaluate the projected failure pressure of glass reinforced Epoxy composite repairs. The full-scale pipeline burst tests and finite element calculations on a damaged pipe and a composite-repaired pipe were explored by Lim et al. (Lim *et al.*, 2019). Deao et al. (Deao *et al.*, 2020) utilized Carbon fiber reinforced polymer to repair defects in pipes with various hoop lengths, and various putties to cover the flaws. Zhang et al. (Zhang *et al.*, 2021) used tests to investigate the durability of Carbon Fiber Reinforced Polymer (CFRP) which was used to repair corroded maritime pipes with the bending moment and seawater immersion. To assess the failure behavior and capacity of grouted composite repair systems, Shamsuddoha et al. (Shamsuddoha *et al.*, 2021) created a three-dimensional (3D) Finite Element Analysis (FEA) of a full-scale pipe with varying amounts of metal loss. The researcher used carbon and glass reinforced polymer composites to repair the cracked pipeline. No research has been found in the literature review that focused on improving the behavior of Epoxy (used as a matrix in composite repair) by adding MWCNT, as such, this indicates to the main contribution and inputs of the current study in developing the crack repairing in petroleum pipelines.

This work consists of three stages; first, studying the effect of adding MWCNT to the epoxy by flexural, impact, and hardness tests. Second, finding/adding the best amount of MWCNT from the first stage to the woven carbon reinforced epoxy and studying the effect of this addition on the properties of the composite repair used for the pipeline through blister test numerically by finite element method. The third comparison between the experimental results of the composite repair with the finite element model under the blister test.

2. Materials and Methods

This section consists of using multiwall carbon nanotube MWCNT to improve fracture toughness and decrease delamination of woven carbon reinforced epoxy used in pipeline repair. Flexural, hardness, and Izod impact tests were used to evaluate the MWCNT effects on the epoxy by adding MWCNT with 0.2, 0.4, 0.6, 0.8, and 1 wt. % and comparing them with pure epoxy, as a reference. Then, identify the best content of MWCNT to the woven carbon reinforced epoxy and use a blistering test to evaluate its behavior. A scanning electron microscope (SEM) was used to show the dispersion and agglomeration of the MWCNT in the epoxy.

2.1. Materials and Testing

Epoxy resin from Don Construction Products (DCP) company-Quickmast type105 base was used as a matrix, which is a low viscosity liquid mixed with a hardener, ratio 3:1, Table I shows the matrix properties. Table II shows the properties of MWCNT reinforcement provided by Henan Huier Nano Technology company.

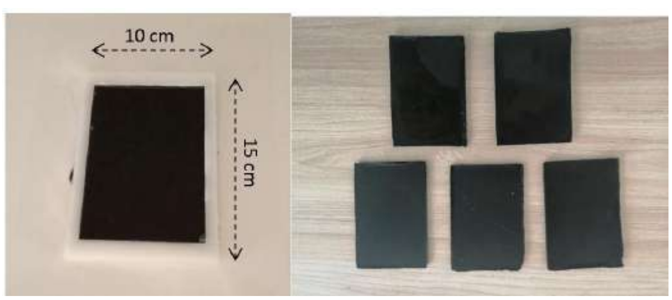
Table I. properties of epoxy matrix (DCP Company, 2020).

Table II. Properties of MWCNT (provided by the supplier).

Table III shows the chemical composition of the steel plates, 6mm thickness, used in this study. The ultimate tensile and yield strengths were 470 MPa and 355 MPa, respectively.

Table III. The Chemical Composition of steel (s355) is used in this study.

The MWCNT reinforced epoxy molds were prepared (see Fig. 2) to study the effect of adding the nano reinforcement where the hardener and carbon nanotube mixture, was manually stirred for 10 min to form a homogeneous suspension by using a simple low shear mixing technique since repairing pipeline requires simple and quick preparation steps in practice and avoiding nanocomposite preparation methods such as using sonication with additives (where this method need additional steps to remove the additives, lowering the molecular weight of the polymer which lead to softening problem and reagglomeration problem in the time of removal procedure (Mirsalehi *et al.*, 2021)), followed by adding the epoxy resin to the mixture. Then the composite mixture was added from one corner into the mold - to avoid bubble formation - and left to be cured at room temperature for 7 days (Fadhil *et al.*, 2016). The molds were cut to be ready for the mechanical tests: flexural (ASTM D790) (ASTM D790, 2016), impact (ISO-180 2019) (ISO-180, 2019) and hardness (ASTM D2240) (ASTM D2240, 2017).



(a) (b)

Fig. 2. (a) preparation mold of MWCNT reinforced Epoxy, (b) molds of the five reinforcements, source: Author's own creation.

A field emission scanning electron microscope FESEM-MIRA 3 LMU was used to find out the MWCNT dispersion and if there is an agglomeration in the nanopowder.

Plates of woven carbon reinforced composite materials with 0.8 wt.% MWCNT repairs have been prepared by hand layup and prepreg method. The composite repair was made of four woven carbon fiber plies and an epoxy matrix containing 0.8 wt.% MWCNT. Figure 3 shows that the layers of the composite are placed on a piece of glass, followed by adding the epoxy matrix to each layer then another window glass part is placed over the composite layers to push the bubbles outside the composite plate and then fixed tightly by 4 clamps.

Fig. 3. Composite plate preparation by hand layup and prepreg method, source: Author's own creation.

Steel plates, 9.5×14× 0.6 cm in dimensions, were drilled to make a hole, 10 mm in diameter, in the center to let the shaft goes through. The upper surface of the plate was cleaned by a wire brush grinder to remove any dirt or scratches and get the required roughness. Following, cleaning with acetone, using wet cotton, to remove grease or dust, then wax is used to fill the hole in the center to avoid the adhesive to go inside. The epoxy is added to the surface to stick the composite plates with the metal base. Fig. 4 shows the woven carbon-reinforced epoxy-containing MWCNT plates, 8×8×0.1cm in dimension, were applied to the steel plates and left for 7 days at room temperature, then the wax was removed. A disc with 8mm diameter and 3mm thickness is stuck over the composite repair inside the hole in the center of the steel plate before proceeding with the test, to ensure an axisymmetric formation through the test.

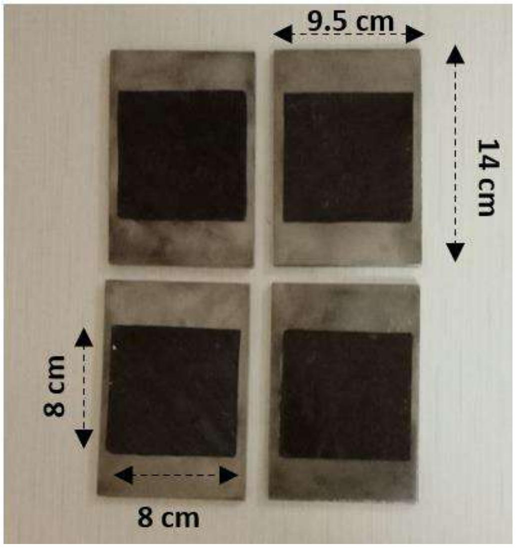


Fig. 4. Blister Test Samples, source: Author's own creation.

2.2. Blister test

A universal tensile machine with a capacity of 100kN was used to perform the blister test. The specimen was fixed horizontally and the repaired face by the composite was faced down (to produce tensile stresses) and fixed by two holders. The used cross-head velocity was 2 mm/min and the force was exerted on the shaft which was placed on a small disc to make sure that the load will transfer uniformly to the composite repair. Figure 5 shows the blister test arrangements, see also Fig. 1.

Fig. 5. Blister test sample under the tensile testing machine, source: Author's own creation.

3. Numerical model

Simulation of the blister test was carried out by building a model in the ANSYS workbench and using the finite element method to represent the interface between the metallic base and the composite repair by utilizing the Cohesive Zone Model (CZM). The used CZM model depends on a bilinear traction separation law (Alfano and Crisfield, 2001) with traction stress (T=8 MPa) and the displacement of the interface (δ = 0.09 mm). Sweep meshing is used with Quad/Tri free face mesh type were meshing and boundary conditions of the model, see Fig. 6. To reduce the run time of the analysis an axisymmetric geometry of the specimen symmetry boundary conditions was used by applying a fixed displacement on the area of the steel plate. A vertical displacement was exerted on the upper surface of the disc to represent the shaft (De Barros *et al.*, 2019). The mechanical properties of woven carbon reinforced epoxy with and without the MWCNT used in the numerical model are mentioned in table IV (Dalina *et al.*, 2014) (Mirsalehi *et al.*, 2021) (Jarali *et al.*, 2012).

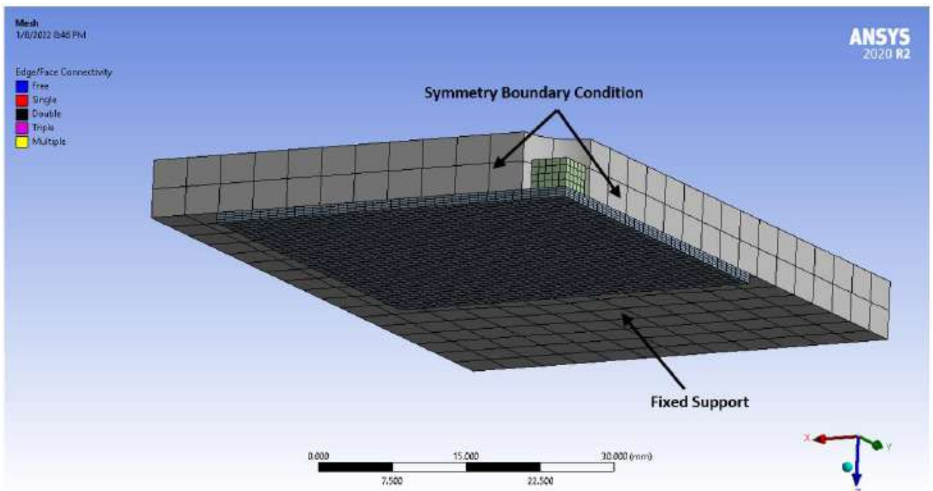


Fig. 6. Finite element model meshing and boundary conditions of the blister test sample, source: Author's own creation.

Table IV. The mechanical properties of 0 and 0.8 wt.% MWCNT woven carbon reinforced epoxy.

4. Results and Discussion

This section consists discussion of SEM images, mechanical (flexural, hardness and impact) tests results in addition to the numerical and experimental results of the blistering test.

4.1 SEM and Mechanical Test Results of MWCNT Reinforced Epoxy

Fig. 7 shows the effect of adding 0.2, 0.4, 0.6, 0.8, 1 wt.% MWCNT to epoxy on the flexural properties of the Epoxy. The content of MWCNTs (0.2-1 wt.%) enhances and increases the flexural strength and reaches the highest values at 0.8% MWCNT composite, flexural modulus was increased from 1 MPa for the epoxy resin to 2.78 MPa for 0.8 wt.% of MWCNT. Making desirable polar forces, and Van der-Waals bonding between chains and MWCNTs results in improving the restriction between MWCNTs/epoxy chains, tangles the resin chains and come closer to each other, consequently, lowering free volume space, which makes the epoxy chains bear additional loading because of nano reinforcement (Park *et al.*, 2004). A 0.2- 0.6 wt. % of MWCNTs was better than epoxy, but lower than 0.8 wt.%, due to the low content of the nanotubes, as did not provide the epoxy with the required strength. Adding 1 wt.% of MWCNT was better than that of epoxy, but lower than 0.8 wt.% because of forming agglomerates of MWCNT in the epoxy, which results in lowering the strengthening effects of the MWCNT (Fadhil *et al.*, 2016) where the agglomerates also serve as planes where cracks can easily spread and slip, which causes material breakage even at low mechanical loads (Mateab & Albozahid, 2022). Adding 0.8 wt.% of MWCNT to epoxy was the best among the others because this percentage was high enough to strengthen the matrix without causing tube tangling and agglomeration. Fig. 8a shows the field emission scanning electron microscope FE-SEM image of non-agglomerated MWCNTs content at 0.8 wt.% which is an approval of the strengthening of this percentage and Fig. 8b shows the FE-SEM image of agglomerated MWCNTs content at 1 wt.% that supports agglomeration of this percentage.

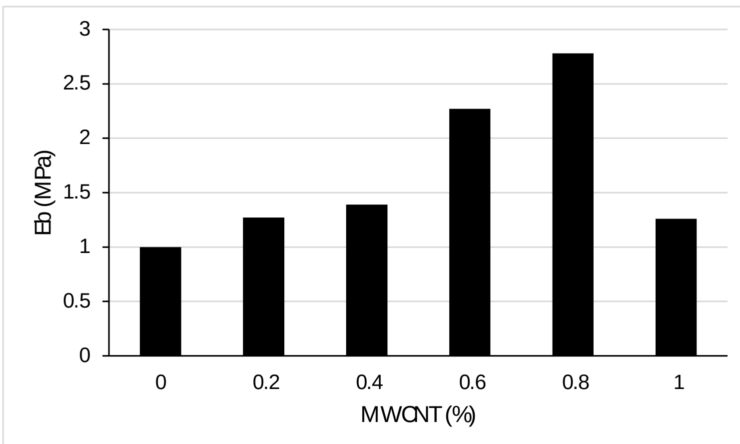


Fig. 7. Effect of adding 0.2-1 wt.% on the flexural modulus of Epoxy, source: Author's own creation.

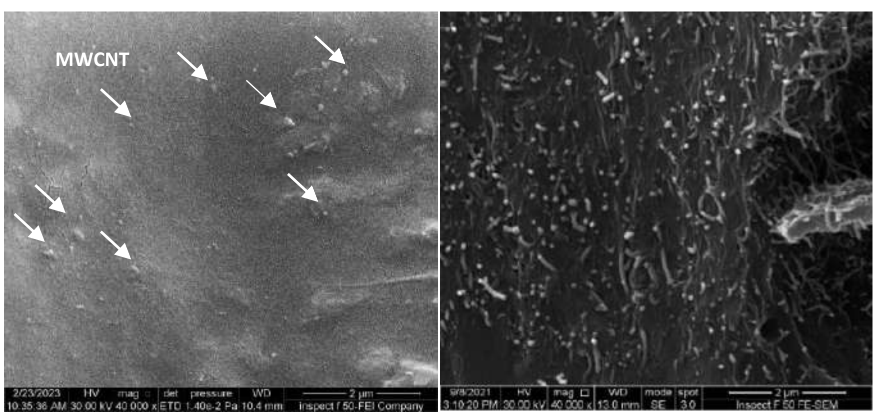


Fig. 8. FE-SEM image from impact fracture surface of (a) 0.8 (b) 1 wt.% MWCNTs, source: Author's own creation.

The same effect can be seen in the impact of epoxy in Figs. 9. Adding 0.6 wt.% MWCNT to epoxy gives good improvement against impact but not good as 0.8 wt.%. Impact toughness (K_{Ic}) is increased as a result of the debonding of the chain segments from the filler surface, which enables the matrix entanglement structure to relax when the load is transmitted to the matrix-filler physical network. Low impact energy, or the capacity of composite materials to absorb and reduce energy during fracture propagation, is influenced by the filler content. However, in the case of a thermoset-toughened polymer, the thermoset's presence effectively causes stress redistribution in the composite, leading to microcracking or crazing at various areas, resulting in a more efficient energy dissipation mechanism (Mateab & Albozahid, 2022).

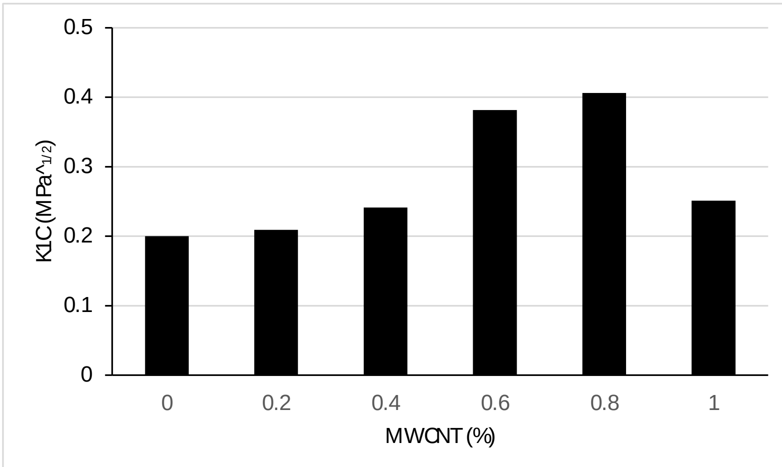


Fig. 9. Effect of adding 0.2- 1 wt.% MWCNT on the K_{1c} of Epoxy, source: Author's own creation.

Fig. 10 shows the effect of adding 0.2- 1 wt.% MWCNT on the Shore D hardness of epoxy. Adding both 0.8 and 1 wt.% of MWCNT were improving the hardness due to the improvement in the restriction between MWCNTs/matrix chains by these high percentages. Tangling of epoxy chains that come closer to one another and lowering the free volume space when adding 1wt% leads to improving the hardness despite the agglomeration.

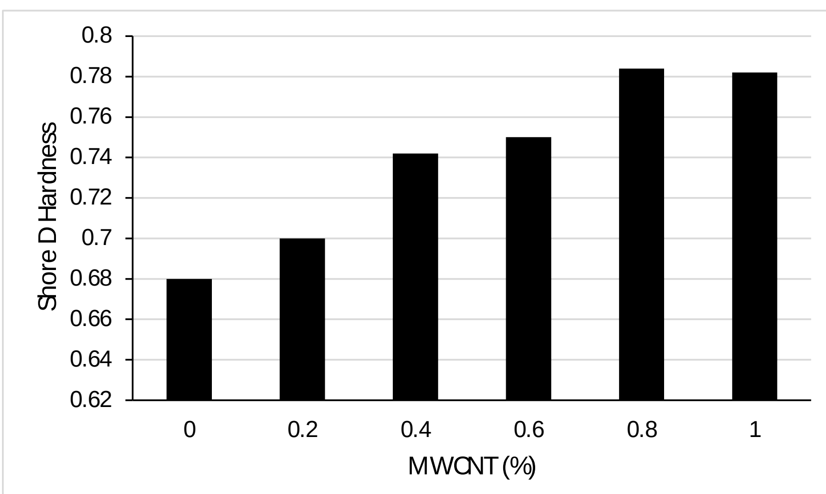


Fig. 10. Effect of adding 0.2- 1 wt.% MWCNT on the Shore D hardness of Epoxy, source: Author's own creation.

4.2. Blister Test Results

The finite element model was used to check if the MWCNT reinforcement is useful in improving the behavior of the composite against the blister test and to decrease the number of experiments to reduce the consumed time. Fig. 11. shows the load-displacement curves of the blister test for 0 and 0.8 wt. % MWCNT, first the curve is increased until the point of the critical load which indicates that the initial debonding of the repair is taking place. Then the interfacial debonding grew as a second stage, and finally, the tearing of the composite repair occurred. The total energy release rate (G_T) can be calculated from the critical load seen at the end of the 1st Region (Mohammad and Liyong, 2015). The figure shows that 0.8% improves the behavior of the composite and increases the value of the critical load to 1353.44 N compared to 446.24 N without nano reinforcement, and G_T was calculated from the critical load according to equation 1 where its value is increased from (0.238 N/mm) to (0.522N/mm). The reason behind this is the creation of desirable polar forces and Van der Waals bonding between the chains and MWCNTs which improves the restriction between the MWCNTs and epoxy chains, tangles the resin chains and forces them closer together, reducing the free volume space, and forcing the epoxy chains to bear additional loading due to nano reinforcement.

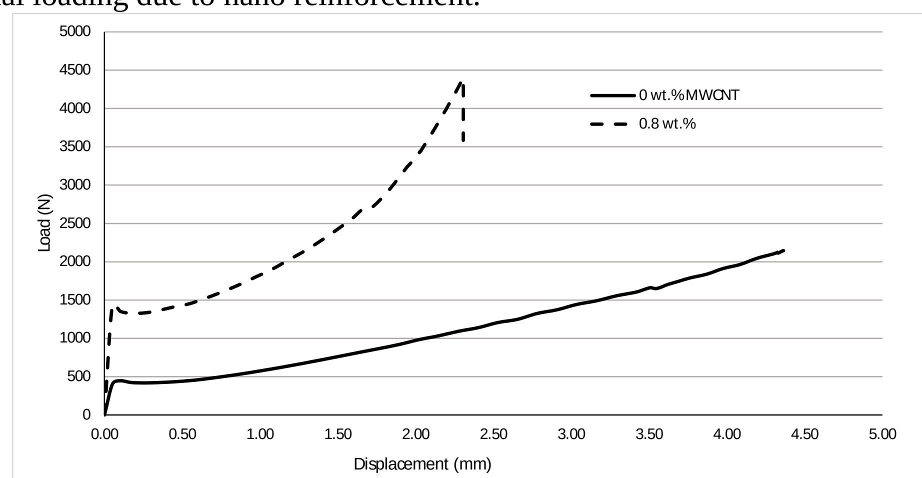


Fig. 11. Finite element Load-Displacement curve of blister test, source: Author's own creation.

Fig. 12. shows similarities in the behavior of finite elements and the experimental Load-Displacement curve of the blister test, an average of six tests was taken to draw the experimental blister test curve due to the diverse behavior of composite materials in real tests. This indicates that the bilinear traction separation law is successful in presenting the CZM zone of blister test. The bilinear cohesive traction-separation law is vastly used in finite element modeling because of its clarity and flexibility. This law is known as consisting of three regions 1st an elastic region up to full strength then 2nd region of softening till the 3rd region of full nodal pair separation on 0 tractions (ISO/TS 24817, 2017).

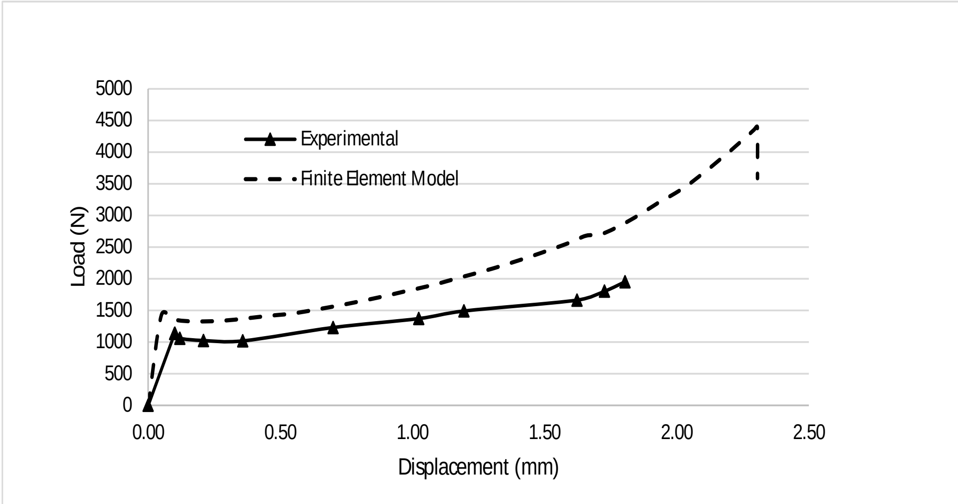


Fig. 12. Finite element and experimental Load-Displacement curve of blister test of 0.8 wt.% MWCNT, source: Author's own creation.

The above blister test results could be used to calculate the failure pressure P of the utilized composites to repair the pipes by using the formula eq. (3) (ISO/TS 24817:2017 (ISO/TS 24817, 2017) and ASME PCC-2-2015(ASME, 2015)):

$$P = \sqrt{\frac{G_T}{\frac{(1-v^2)}{E_{ac}}\left(\frac{3}{512t^3}d^4+\frac{1}{\pi}d\right)+\frac{3}{64Gt}d^2}} \;\ldots\ldots (3)$$

Where $E_{ac} = \sqrt{E_{11}E_{22}}$ (Köpple *et al.*, 2013), d represents the diameter of the hole and G is the shear modulus.

The failure pressure is increased from 7.91 MPa without MWCNT reinforcement to 16.4034 MPa with 0.8 wt% of MWCNT which is a reasonable improvement if compared with previous studies when taking into consideration the material and thickness of the composite repair. These findings demonstrated that the addition of MWCNTs that have been uniformly dispersed enhances the mechanical characteristics of the constructed composite by increasing the number of MWCNT surfaces that are available for interacting with the surrounding epoxy and strengthening the epoxy resin matrix (Mirsalihi *et al.*, 2021) which made the composites bear more pressure.

5. Conclusions

This work studied the effect of carbon nanotube on repairing cracked pipelines where different percentages of the nanotube reinforcement were added to the Epoxy matrix of composite materials that were used to heal a crack on a steel plate to resemble the real pipe, the following findings are concluded from this study:

- All mechanical tests revealed that using MWCNTs in pipeline repair is beneficial because the epoxy chains bear additional loading due to the nano reinforcement in the woven carbon reinforced epoxy.
- Adding 0.2- 0.6 wt.% of MWCNT is not sufficient to strengthen the epoxy while 0.8 wt.% addition gave the highest strength and caused stress redistribution in the

composite, leading to microcracking or crazing at various areas, resulting in a more efficient energy dissipation mechanism.

- Adding 1 wt.% of MWCNT leads to agglomerates forming, which causes sample breakage even at low mechanical loads.
- Similarities in the behaviors of finite elements and the experimental Load-Displacement curve of the blister test were observed. This indicates that the bilinear traction separation law is successful in presenting the CZM zone of the blister test because of its clarity and flexibility.
- Other materials can be used in repairing pipelines as a future scope of this study like earth mineral nano powders and natural fibers to lower the cost and use environment-friendly materials.

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Table I. properties of epoxy matrix (DCP Company, 2020), source: Author's own creation.

Compression Strength	Bending Strength	Tension Strength	Specific Gravity	Viscosity
72 MPa	50 MPa	20 MPa	1.1	3-5 Poise

Table II. Properties of MWCNT (provided by the supplier), source: Author's own creation.

Carbon tube Length (nm)	Tube Outer diameter (nm)	Tube wall thickness (nm)	Number of layers
3 to 12	12.9	4.1	5 to 12

Table III. The Chemical Composition of steel (s355) is used in this study (provided by the supplier), source: Author's own creation.

C%	Mn%	Si%	S%	P%
0.23%	1.6%	0.05%	0.05%	0.05%

Table IV. The mechanical properties of 0 and 0.8 wt.% MWCNT woven carbon reinforced epoxy (provided by the supplier), source: Author's own creation.

	0% MWCNT	0.8% MWCNT
Longitudinal Modulus of Elasticity E11 (GPa)	29	29.5
Transverse Modulus of Elasticity E22 (GPa)	29	29.5
Shear Modulus G (GPa)	2.51	2.57
Poisson's Ratio υ	0.3	0.27
Density gm/cm^3	1.22	1.28