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Material Characterization of Buried GFRP Pipeline Failed during Hydrostatic Testing

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Abstract - *Glass Fiber Reinforced Plastic (GFRP) pipe is made up of diverse materials, the qualities of the different materials combine to provide superior properties. GRP pipes consist of glass fibers, vinyl ester resins, or unsaturated polyester and different reinforcing agents. There was premature failure of GFRP composite pipes during hydrostatic (hydro) testing while laying out in the construction site. The hydro test is a process where components are tested for strength and leaks before putting them in service. Two pipe samples, one referred to as good pipe and another referred to as leaked or used pipe, were investigated to find out the root cause of failure. The investigation consisted of field-visit, studying the layout procedure, visual observation, microstructural characterization, Raman spectroscopy analysis, Thermogravimetric analysis (TGA) and Fourier transform infrared spectroscopy (FTIR) analysis analyses. Visual observations of the leaked or used pipe sample reveals some abnormal features like delamination of layers, circumferential cracks, air pockets, adherent foreign/ earthy materials, visible fibers, and heterogeneity in colors. Adherence of earthy material on pipe's surface indicates that there is incomplete curing of polymer. Microstructural analysis exhibits that the volume fraction of fibers was very less (particularly in used pipe) in resin matrix and their distribution was also uneven. Overall analysis suggests a defective manufacturing process, and usage of expired polymer and sand along with non-prescribed fiber loading which have initiated the failure of pipes during hydro test.*

Key Words: Glass Fiber Reinforced Plastic pipe, Hydrostatic testing, Resin, Raman spectroscopy analysis, Glass transition temperature.

1.INTRODUCTION

Glass reinforced polymer (GRP) pipes are gradually more used and have become a crucial class of engineering materials for a wide range of products. These types of components can be used as gas-liquid transfer pipes, high pressure containers, steel industrial pipe system, and marine and offshore for good corrosion resistance. Despite the advantages of GRP pipes, the product is not effectively used due to inadequate knowledge of different mode of failure and its mechanism for evaluating the means of deterioration. Long-term endurance of glass-epoxy composite pipeline in service is an industrial important issue mainly because the consequences of failure are severe in marine, oil, gas and (Petro)chemical applications. It is also a challenge because it includes many parameters (design, resins, fibers, manufacturing process, and environment). The resin plays an important role in pipe manufacturing, it helps in transfer stress between the reinforcing fibers, and helps in holding the fibers together, and preserve the fibers from mechanical and environmental damage. Due to the cost effectiveness impure resin are mixed during pipe manufacturing process which resulted in premature failure during service. Some of the those problems that occur during the manufacturing and after installation of GRP are such as formation of air bubbles between the polyester resin layer and the surface film or the mould surface, Moisture formed in between the tubing outer and inner layers after installation, heat released in between the layers of E-Glass GRP Pipes due to exothermic reaction which consequences in the formation of surface defect in forms of crack in the pipe [1]. The manufacturing temperature is another crucial parameter that influences the degradation resistance of the GRP pipes. The temperature determines various factors such as air bubbles, moisture, heat generated within the pipes. The failure of composite pipes falls into two main categories of short-term and long-term requirements in accordance with international rules and regulations. For the long-term considerations, fatigue failure, creep phenomenon and other environmental issues are the most important topics. Due to the cost effectiveness, based on ASTM standards for GRP pipes, sand aggregate was incorporated into the structural wall composition. The pipe was designed for a pipe stiffness C (248 kPa) and quartz sand was added to meet this requirement by increasing the wall thickness [2-4]. The sand was inserted between the filament-wound layers, as part of the shell wall. The structural wall of the pipe was formed by an inner layer of quartz sand-filled polyester resin, followed by a layer of filament wound continuous fibrous glass strand roving, saturated with polyester resin, then another ply of sand-filled resin and, finally, four layers of filament wound continuous glass fiber composite. Since the sand is added simultaneously with the glass fibers, the manufacturing time is not affected, and the overall cost is reduced. After the lay-up was complete, in view of the gravitational attraction force on the resin being cured, the pipe was kept rotating for 2-3 h, at 4 rpm, thus avoiding resin flow towards a particular bottom side of the cross section and resulted in uniform wall thickness. There

are some important quality tests performed before and during installation of the pipe. One of the important quality tests is the hydrostatic test which is essential to confirm the hydraulic sealing of the system at the testing pressure and then, ultimately, its structural integrity. The internal hydrostatic pressure depends on the GRP pipe production process and includes aspects such as internal structure and volume fractions of the constituents, as well as distribution, sizes and shapes of the reinforcing phases, voids, and cracks. In this present work failure analysis of GRP pipe was carried out to find the geneses of failure and improve its service life.

2. Experimental Procedure and Results

2.1 Site Visit

During laying of GRP pipe of around 4500 m long pipeline network, leakage was observed during hydrostatic test. The diameter of pipe was 500 mm and thickness were around 7.4 mm. After laying down the pipeline underground, a hydrotest procedure (as per ASME B31) [5] was carried out to check any leakage through pipeline as per standard. In hydrotest, the pipeline is subjected to a pressure of 1.5 times the design pressure for approximately 1 hour and any drop in pressure during that time indicates a leakage. Pipelines of different length were laid down at 9-10 locations, and the hydrotest was conducted at all locations. At all locations, first a leakage through the joints was observed (Fig.1a-b). The manufacturer of the pipes provided lamination at all the joints where leakage was found. After providing lamination at joints, leakage from joints stopped. But hydrotest failed due to seepage from the main pipe body at some locations. To conduct the hydro test, lamination was provided on the seepage locations, but the test failed as the location of leakage could not be detected since most part of pipeline is underground and not visible, except joints.

Fig.1a: Leakage through main pipe body; Fig.1b: Seepage through main pipe body

2.2 Visual Observation

Two pipe samples were received for investigation to find out the root cause of failure. Leaked or used sample refers to the part of the pipe which was in service, and leakage was found through it during hydro test (Fig.1c). However, the leaked sample has a coupler joint within it and a thick layer of lamination was observed on it. Therefore, the location of leakage is not clear. Good pipe refers to the part of pipe which was pass though hydro test.

Fig.1c: Pipe samples received for investigation; Fig.1d: Air pockets and adherent materials; Fig. 1e: Heterogeneity of colors

Visual observations of leaked pipe sample revealed some abnormal features like air pockets, adherent foreign materials, visible fibers and heterogeneity of colors (Fig 1d-e). When the Curing speed is too high (styrene is entrapped in surface) air pockets were observed in the surface. Visual observations of good pipe sample didn't reveal any such abnormal features.

2.3 Microstructural Analysis

The samples were prepared in EXTEC Labcut 1010 Low Speed Diamond Saw for precision cutting of a GRP pipe micro sample. Samples were cut with minimal damage and deformation. Variable wheel rotation speeds were used with range from 100 to 200 rpm for cutting the GRP pipes. The microstructures of the composites were analysed by optical microscopy and scanning electron microscopy to observe the different component and presence of voids along with the distribution of the fibers. In case of Leaked sample, volume fraction of fibers was observed to be very less compared to resin matrix; distribution of fibers was observed to be non-uniform (Fig.2a). Numerous voids or porosities were observed in the sample; voids were found to be bigger in size and more in numbers (volume fraction of voids: Vv < 25% volume fraction of fibers: Vf ranging from 35-40%) compared to those of good GRP Pipe sample (volume fraction of voids: Vv < 10% volume fraction of fibers: Vf ranging from 50-65%) as shown in Table 1. Plenty of particle like features were observed in the matrix. Decohesion or separation between the matrix and particles were distinctly observed (Fig.2b-c). Both the samples were having non-uniform sand content which may result in false weight gain and reduced the strength of composite as they hinder the bonding between fibers and polymer.

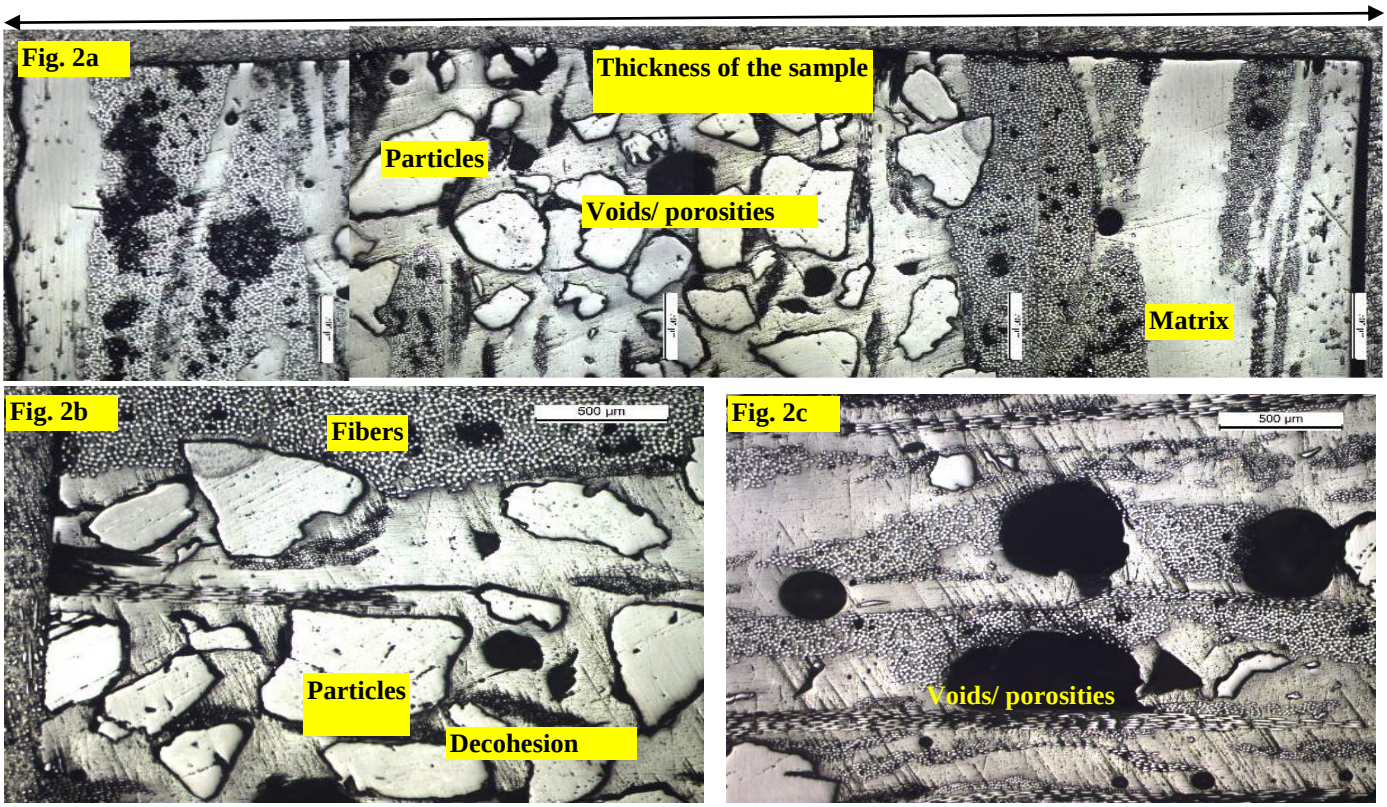

Fig.2a-c: Microstructural examination of leaked/used pipe at different magnifications.

To make the process costeffective, sand was insterted between the filament wounded layers, as a part of shell wall. The sturctural wall of the pipe was formed by an inner layer of quarz sand-filled polyester resin, followed by a layer continious fibrous glass strand roving saturated with polyester resin, then another ply of sand-filled resin and, finally four layers of wound continious glass fiber composite [6]

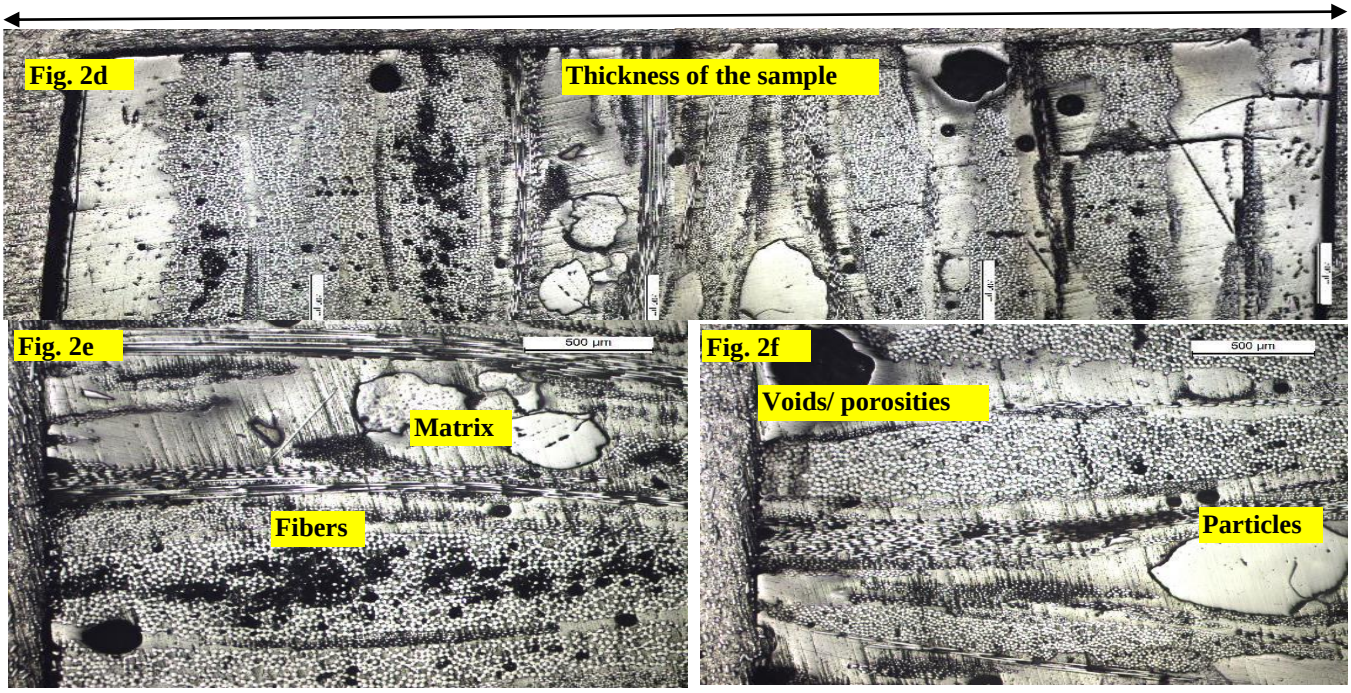

Fig.2d-f: Microstructural examination of leaked/used pipe at different magnifications.

Table 1: Size distribution of voids and fibers in GRP pipes

Parameter	Leaked/Used Pipe	Good Pipe
Average Void Dia. (microns)	547.19	287.45
Minimum Void Dia. (microns)	81.59	23.52
Maximum Void Dia. (microns)	1386.70	472.23
Volume fraction of voids (Vv)	< 25%	< 10%
Volume fraction of fibers (Vf)	35-40%	50-65%

The typical microscture of GRP pipe is shown in Fig.3.

Fig. 3

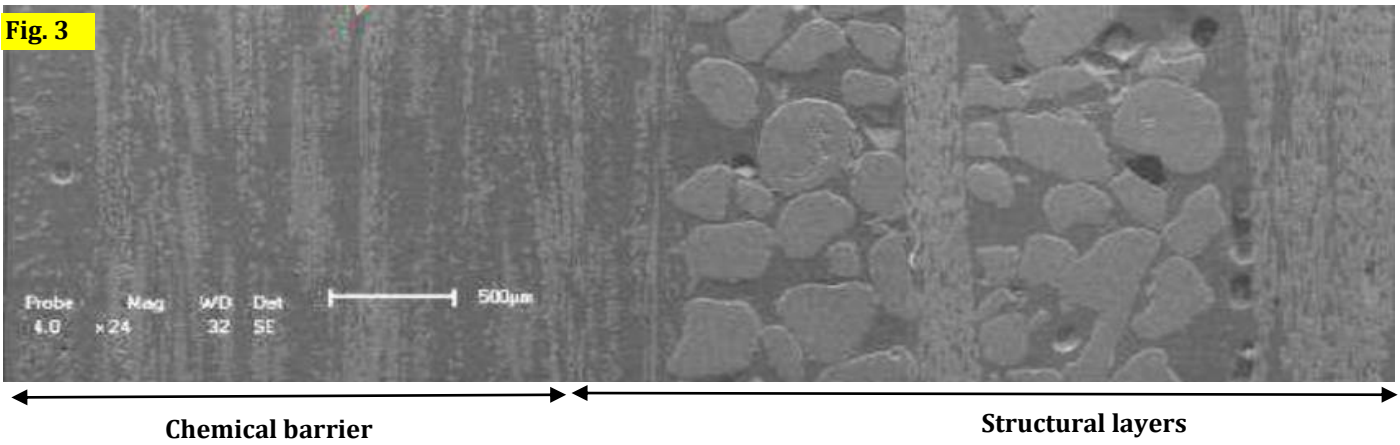

Fig.3: The innermost (at left) to the outermost surface microstructure of the GRP pipe wall

2.4 SEM and EDX Analysis

Two GRP pipes (Leaked and Good sample) were observed by Field Emission Gun Scanning Electron Microscope (FEG-SEM). The GRP pipes were observed directly without any coating. The accelerating voltage was 5 kV. EDX analysis of the GRP pipes was conducted using a JEOL JSM 6460LV SEM instrument (JEOL Ltd., Tokyo, Japan) with a silicon drift detector. The testing was performed at 5^{10-8} A probe current and 15 keV accelerating voltage. The GRP pipes were coated with gold using a vacuum coater.The gold input was manually withdrawn from the final EDX data. EDX analysis of particles revealed significant content of Si and O suggesting presence of sand (~95%) as shown in Table 2. Fibers were non-uniform and varying in size. Large voids with entangled fibers inside were observed (Fig.4). Voids present were very big (~1386 micron max.) in size.

Fig. 4

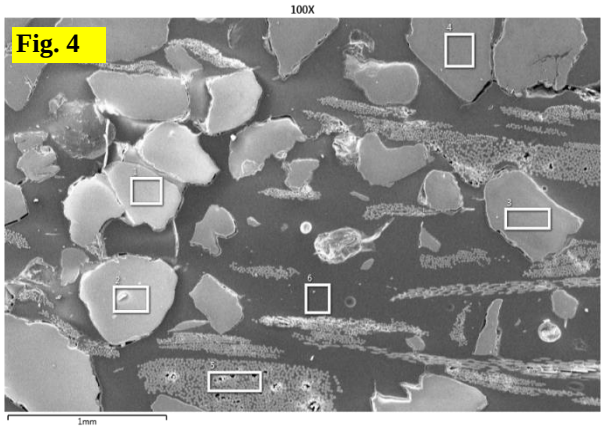

Fig.4: EDX analysis of the leaked pipe sample

Table 2: EDX analysis (wt.%) of particles in the matrix for Leaked sample:

Spectrum Label	1	2	3	4	5	6
C	5.19	7.63		4.87	40.86	75.32
O	50.43	49.62	50.79	50.28	33.27	24.68
Al	---	---	---	---	3.56	---
Si	44.38	42.75	49.21	44.85	13.87	---
Ca	---	---	---	---	8.43	---
Total	100.00	100.00	100.00	100.00	100.00	100.00

2.5 Raman Spectroscopy Analysis

The Raman Spectroscopy Analysis was performed at 785 nm with a Renishaw, inVia Qontor system having a Nd-YAG laser source and equipped with a Centrus detector OFQM98 and grating 1200 l/mm. The continuous scan was checked for the spectral range from 98.87 to 3199.04 Raman shift/cm^{-1}. Raman spectroscopy was used to study resin and to identify the purity of the resin material. Purity of the resin material can be related to the identification of peak assignments for the starting materials of the resin such as phthalic acid and ethylene glycol (EG) and the polymerized product, polyethylene terephthalate (PET) respectively. Here, it is noteworthy, that phthalic acid can exist in 3 isomeric forms such as terephthalic acid (TPA), isophthalic acid (IPA) and orthophthalic acid (OPA) based on the position of the carboxyl groups [7-8] on the benzene ring as shown in figure 5.

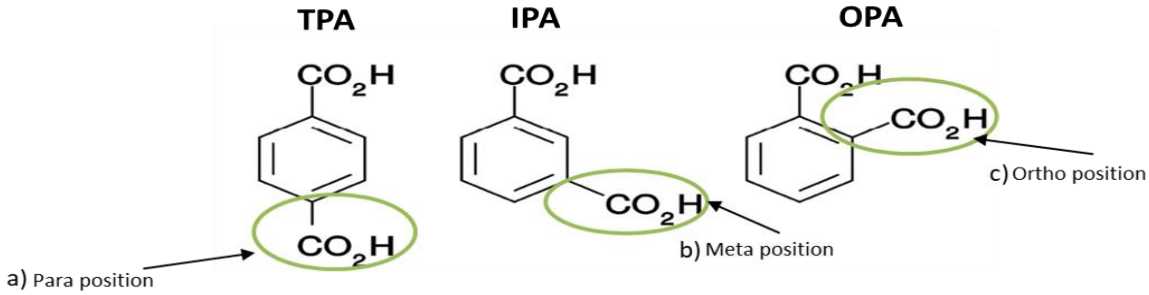

Fig. 5: Isomers of phthalic acid: a) TPA; b) IPA and c) OPA

In the present case, Raman analysis can be useful in indicating which isomer [9] is present and also in predicting the effectiveness of the polymerization. Figure 6 (a, b) presents the Raman spectra for the good pipe and leaked pipe samples.

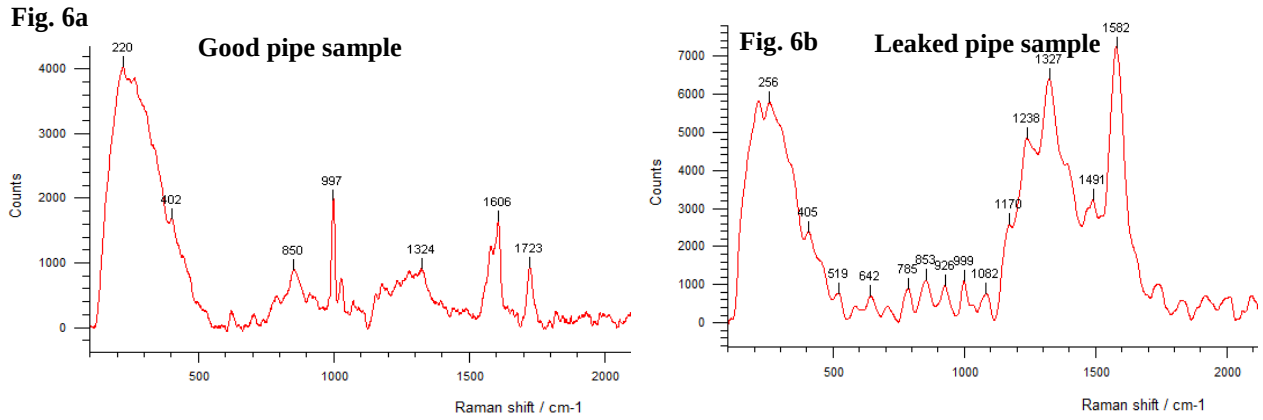

Fig.6a-b: Raman Spectroscopy Analysis of leaked and good pipe analysis

Table 3: Peak assignments in Raman spectra:

Sample type	Peak Position (cm^{-1})	Peak assigned to	References
Good pipe	220	Terephthalic acid (TPA)	Tellez S., et al. 2001
	850		Bardak et al., 2016
	1324		Tellez S., et al. 2001
	1606		Tellez S., et al. 2001
	1723	Polyethylene terephthalate (PET)	Adar et al. 2018
	997	Ethylene glycol (EG)	Krishnan et al., 2010
Leaked pipe	785	Orthophthalic acid (OPA)	Osterrothova and Jehlicka; 2010
	1582		
	853	Terephthalic acid (TPA)	Bardak et al., 2016
	1327		Tellez S., et al. 2001
	1491	Isophthalic acid (IPA)	Osterrothova and Jehlicka; 2010
	995	Ethylene glycol (EG)	Krishnan et al., 2010

Table 3 presents the peak assignments and it can be observed that peak for PET is not seen in case of the leaked pipe sample indicating a weaker polymerization. Furthermore, peak assignments of leaked pipe sample suggest the presence of OPA, IPA and TPA indicating a mixture of all the 3 isomers, whereas presence of only TPA is observed in case of the good pipe sample. Thus, in case of leaked pipe, it can be said that resin is contaminated, and polymerization is also weaker, which could cause failure of the sample during the end application.

2.6 Temperature-modulated differential scanning calorimetry test (TMDSC)

TMDSC is a thermal analysis technique that has been found to be an accurate tool for measuring the temperatures of various transitions occurring in materials. In the present investigation, the TMDSC was employed to determine the Glass Transition Temperature (Tg) value of the composite pipe materials. Tg is the temperature range during which the polymer shifts from a tough glassy state to a soft rubbery state. The test was conducted from 30°C to 250 °C at the heating rate of 10 °C/min. Tg value of leaked pipe material showed a relatively lower value than that of new pipe material Fig 7a-b and Table.4. At temperatures above Tg, a polymer experiences a sudden drop in its mechanical stiffness. Typically, when mechanical stiffness is desired, a polymer's service temperature should be below its Tg.

Fig. 7a

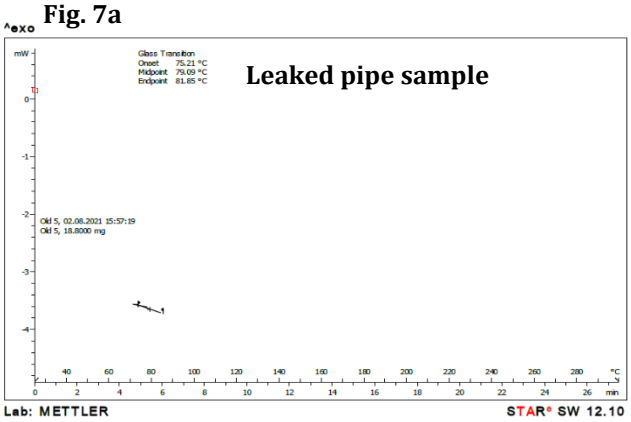

Fig. 7b

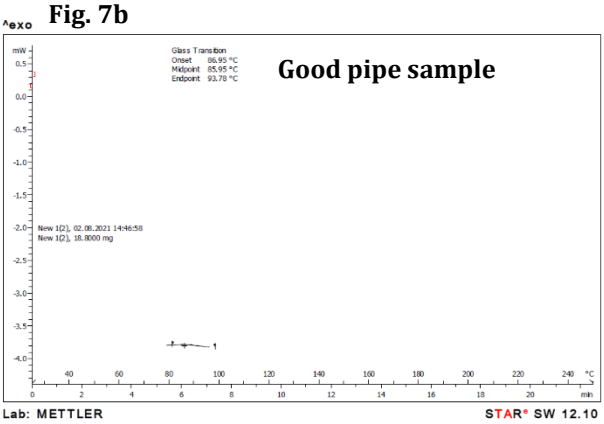

Table 4: Values of Glass Transition Temperature (Tg) for Different Samples

Material	Onset Tg (˚C)	Material	Onset Tg (˚C)
Leaked (Sample #1)	76.00	Good (Sample #1)	88.04
Leaked (Sample #2)	75.21	Good (Sample #2)	86.95
Average (Leaked)	75.60	Average (New)	87.49

2.7 Fourier Transform Infrared spectrophotometers (FTIR) analysis

The FTIR analysis was done using a Nicolet Magna 550 Fourier transform infrared spectroscopy (FT-IR) analyzer with a resolution of 4 cm-1 in the range of 400-4,000 cm-1 wave number. FTIR analysis shows that the intensity of FTIR peaks for leaked or used pipe was lower than that of new or unused pipe. Peak assignments for both the samples are presented in Table 5. From these observations, it is seen that peak position 1643 cm-1 is present in case of the leaked pipe sample which is a characteristic assignment for TPA [10-12]. Thus, the leaked pipe sample is not pure in form which can also be correlated to observations of Raman analysis and is prone to failure.

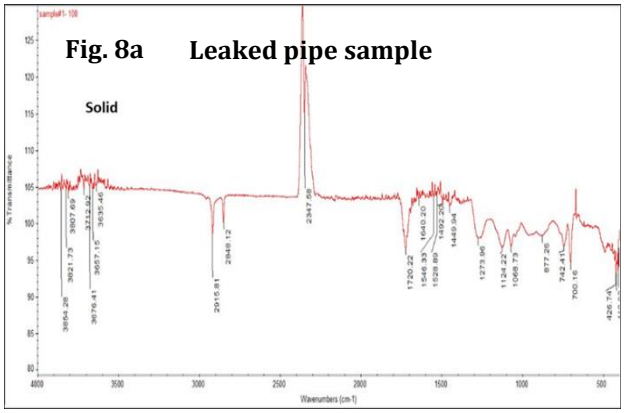

Fig. 8a Leaked pipe sample

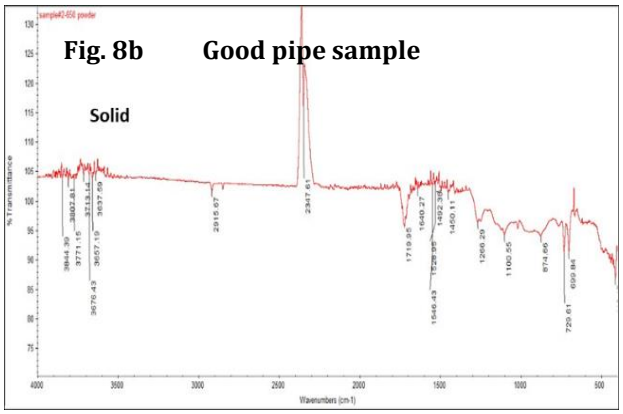

Fig. 8b Good pipe sample

Table 3: Peak assignments in Raman spectra:

Table 5 : Peak assignments in FTIR analysis

Description of peak	Peak Position (cm-1)	Good sample	Leaked sample	Reference
ν(C=O) carboxyl stretch	1715	√	√	Osterrothova and Jehlicka; 2010
ν(C=C), C=C stretch	1449	√	√	
ν(C-O), carboxyl stretch	1263	√	√	
νas (C=O) & δ(COH) antisymetric stretch and in plane deformation in benzene ring	1642	√	√	Tellez S. et al.; 2001
ν(CO) & δ(COH), strech and in plane deformation in benzene ring	1117		√	
γ(CCC) & δ(COH), out of plane and in plance deformation in benzene ring	1017			
δ(CCC), in plane vibration	700	√	√	Bardak et al.; 2016
δ(CC), in plane deformation	1073		√	Loring et al., 2001

3. Conclusion

The GFRP composite pipes failed due to leakage during hydro test while laying out in the site due to defective manufacturing process, and usage of improper polymer/resin material and non-uniform sand content along with non-prescribed fiber loading which might have initiated the failure of pipes during hydro test.

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