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AN OVERVIEW OF GLASS FIBER EPOXY AND POLYETHYLENE HIGH DENSITY BURST FROM CAPV (COMPOSITE AIR PRESSURE VESSEL)

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ABSTRACT

Composite materials have numerous applications in the most varied areas of engineering as offshore, oil and gas industry, marine industries, aeronautics, and automaker. Used because they bring high rigidity and specific resistance, due to the advantages of being resistant to corrosion and having structural flexibility. The work is focused on fiberglass composites with epoxy matrix (FG) and no high-density polyethylene (HDPE) a decade after the incident. The approach was based in two stage, first on an investigation that took place on board of a semi-submersible drilling rig that operates offshore with a drilling capacity of 7500 meters in depth and a water depth of 2500 meters. a high pressure line that is connected to CAPV (composite air pressure vessel) that is part of to the high pressure system of the CMC (Crown mounted compensator). In a second stage, was took samples from CAPV to be analyzed, where impact tests (Charpy) were performed according to ASTM D6110-10 standard, flexion tests according to ASTM D790-17 standard, differential scanning calorimetry (DSC) according to ASTM D3418-12 standard, scanning electron microscopy (SEM) and thermogravimetric to have a more detailed study on the composite materials that made up those vessels, which are PEHD and FG with matrix epoxy. Showed the results of the investigation and finish the work with material analyses assays even after 10 years of manufacture and complications during the use of the vessels.

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INTRODUCTION

Glass fiber and polyethylene high density (PEHD) are widely used in many engineering fields, (offshore, oil and gas industry, marine industries, aeronautics, etc.…) and specific custom applications (cars, yachts, etc...). They bring both high specific stiffness and strength, due to the advantages of corrosion resistance and structural flexibility. The rapid growth of composite materials expands their feather almost to all the engineering applications. This is due to their unique advantage of lower weight to higher specific strength [Altin, 2018; Kumre, 2017]. The PEHD + Glass fiber epoxy composite materials are extensivel used in lightweight structural components because of their versatile behavior of reinforcements and polymer matrices [Jappes, 2012; Mayandi, 2015]. Also, those material are becoming more and more attractive mainly due to their higher moduli, weight savings and less installation costs [Rafiee, 2016; Shamsuddoha, 2013], properties such as high specific strength, good fatigue strength and excellent corrosion resistance are the essential requirements for the transportation of hot mediums [Rafiee, 2017; Jaipurkar, 2017; Boussetta, 2017]. In these circumstances, it is important to exploit more knowledge about the design and performance of PEHD + Glass fiber epoxy composite materials towards achieving enhanced properties. The concurrent engineering on material, design and fabrication needs to be well addressed to achieve better quality [Dai, 1988]. Accordingly, a better under-standing of the fabrication processes and its state-of-art technology are of great and prime importance. Generally, CAPV (composite air pressure vessel) that basically is manufactured with PEHD + Glass fibber epoxy, have been produced using distinct production method namely injection molding (IM). From an engineering point of view, composite parts undergo compressive stresses (compression or bending loadings). In the case of CAPV (composite air pressure vessel) bending loads will induce tension on one side and compression on the other side. Based on the observation that compressive strengths are usually lower than tension ones [Effendi, 1995], compressive failure should be considered carefully. Therefore, a proper estimation of compressive strength will allow for an efficient design of structures.

Fig. 1. CAPV's fabricated with Glass fiber epoxy and polyethylene high density

Experimental: This section has two objectives. Firstly, to introduce the composite material we used as an example of our experiment. Secondly, to present the set of experiments carried out and the results obtained.

MATERIALS AND MEDHOTS

The present study is focused on glass fiber epoxy and polyethylene high density (PEHD) used in CAPV, which are commonly used in oil and gas industries. Polyethylene (PEHD) is Borecene RM 7403 UV stabilized polyethylene (PE) grade. Exhibits good flow and high stiffness. Is suitable for processing by injection molding (IM). Recommended for thick walled applications, large containers and foamed articles. Glass fiber is T30 SE 1500 is also suitable for use in filament wound pipe, tubes, tanks and air pressure vessel.

Fig. 2. CAPV where the samples came from

Charpy tests: Charpy tests were performed following ASTM D6110-10 [12] which have dimensions of 127x12,7x12,7mm (length × width × thickness). This test method is used to determine the resistance of plastics to breakage by flexural shock as indicated by the energy extracted from standardized pendulum- type hammers, mounted in standardized machines, in breaking standard specimens with one pendulum swing.

Fig.3 Sample of polyethylene

Fig.4. Sample of glass fiber

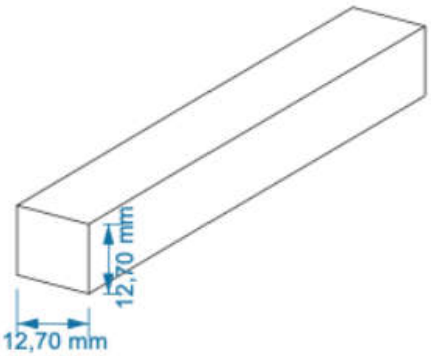


Fig.5 Dimension of specimens used on Charpy test

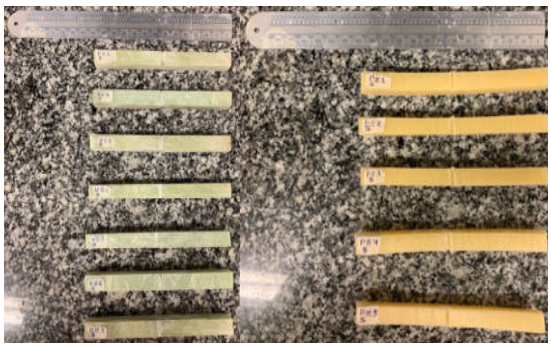


Fig.6 Specimens of glass fiber and polyethylene used on Charpy test

Fig.7 Specimens #5 of glass fiber after Charpy test

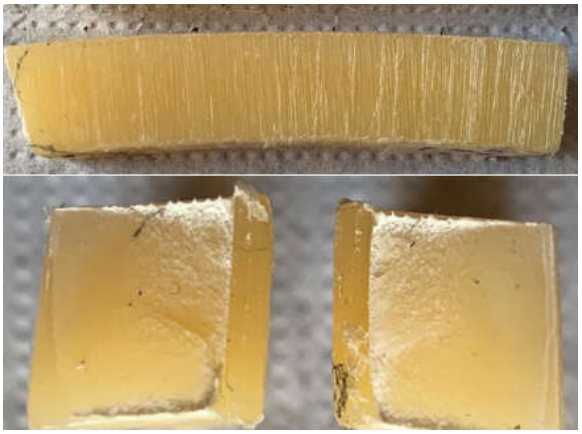


Fig. 8. Specimens #1 of PEHD after Charpy test

Fig.9 Impact machine used during the tests

Flexural tests: Flexural tests were performed following ASTM D790-17 [13] which have dimensions of 128x13x4 mm (length × width × thickness). These test methods are used to determine the flexural properties of unreinforced and reinforced plastics, including high modulus composites and electrical insulating materials utilizing a three-point loading system to apply a load to a simply supported beam (specimen).

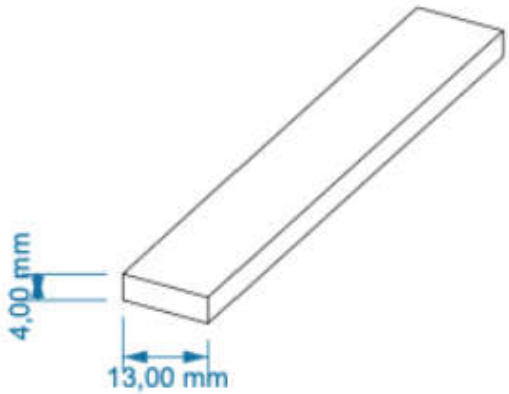


Fig.10. Dimension of specimens used on flexural test

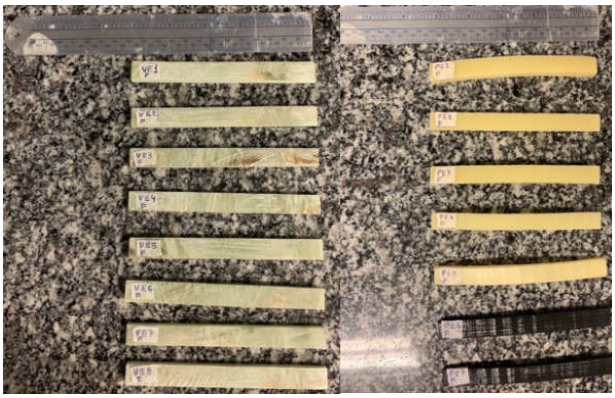
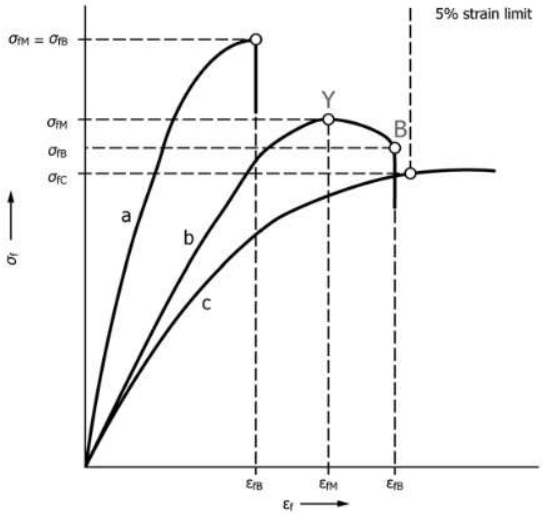


Fig.11 Specimens of glass fiber and polyethylene used on flexural test



NOTE 1—Curve a: Specimen that breaks before yielding.
Curve b: Specimen that yields and then breaks before the 5 % strain limit.
Curve c: Specimen that neither yields nor breaks before the 5 % strain limit.

Fig.12 Typical Curves of Flexural Stress (σ_f) Versus Flexural Strain (ε_f)

SEM (scanning electron microscopy analysis): Scanning electron microscopy (SEM) analysis was performed on FEI INSPECT S50. Analysis via SEM is a technique that characterizes the elements present in the sample, and not the compounds, and the detection limit can reach 0.1% by mass concentration.

DSC (differential scanning calorimetry) analysis: DSC analysis were performed following ASTM D3418-12 (Standard test method for transition temperatures and enthalpies of fusion and crystallization of polymers by differential scanning calorimetry, 2012) [14],

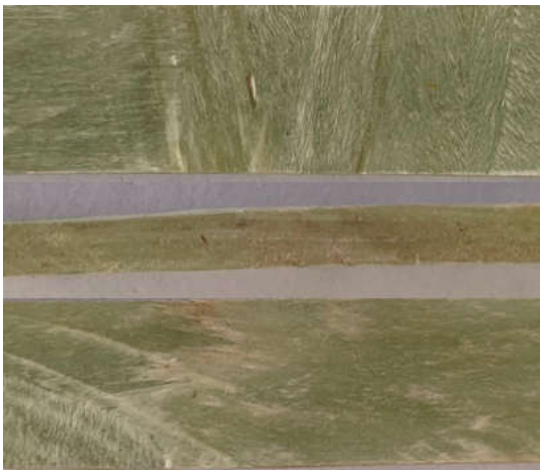


Fig.13 Specimens #4 of glass fiber after flexural test

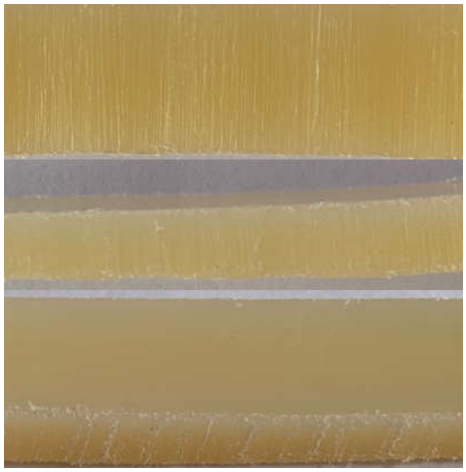


Fig.14 Specimens #5 of PEHD after flexural test

Fig.15 Flexural machine used during the tests

It consists of a test in which the temperature difference between the sample to be characterized and an inert substance (reference), when both are subjected to a controlled temperature program [CANEVAROLO, 2013]. The sample is subjected to a uniform heating ramp, with the temperature monitored by means of a thermocouple and compared to the temperature of the reference sample. Two samples were sent, one of fiber glass and the other of PEHD to the laboratory to carry out DSC, SEM and ash content.

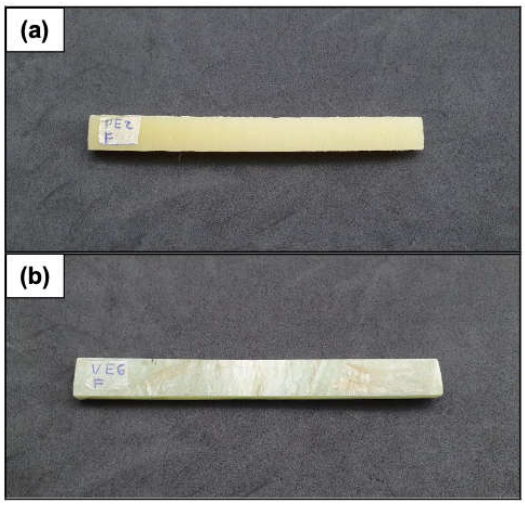


Fig.16 Specimens sent for analysis (a) AFK211230 e (b) AFK211231

Table 1. DSC test conditions

STEPS	Temperature range (°C)	Heating rate (°C/min)	Gas	Flow gas(mL/min)
1st Heating	25 a 300	10	N2	50
Isotherm 5 min.	300	0	N2	50
2nd Heating	25 a 300	10	N2	50
Sample holder	Aluminum Candlestick			
Completion date	04.26.2021			
Reference	Afinko methodology			
Equipment	DSC Shimadzu, DSC-60			
Sample masses (mg)	**AFK211230:** 5,010	**AFK211231:** 4,700		

Ash content: To determine the amount of inorganic material, the sample was calcined in a muffle, which basically consists of a high-temperature oven with controlled temperature. In this way, all organic material is eliminated and by weighing the sample before and after calcination, the amount of inorganic material present in the crucible after calcination is determined [ISO, 2019].

Table 2. Ash content test conditions

Reference	ISO 3451-1:2019 - "Plastics - Determination of ash Part 1: General methods"
Method	( )A-Rapid Ashing or ( )B or ( ) C
Lab temperature: 23,5 °C	Lab humidity: 42%
Calcination temperature: 600°C	Calcination time: 60 min
Heating rate:	20 °C/min)
Date:	04/12/2021
Equipment:	Muffle and analytical balance, AUW220D

TGA (Thermogravimetric analysis): Thermogravimetric analysis (TG or TGA) is a widely used tool for the characterization of materials for several applications, especially for the characterization of polymers. This analysis is based on the continuous measurement of sample mass as a function of temperature or time and under a controlled atmosphere [Canevarolo, 2003]. The results obtained from the thermogravimetric analysis are presented in curves of mass versus temperature variation and, from this information, it is possible to determine, for example, sample composition, thermal stability and quantification of inorganic residues [Mothé, 2009]. The equipment used in the analysis consists of a microbalance and an oven connected to a programmer responsible for controlling the temperature profile to which the sample will be submitted [LUCAS, 2001].

Table 3. TGA test conditions

STEPS	Temperature range (°C)	Heating rate (°C/min)	Gas	Flow gas(mL/min)
I	30 a 800	10	N2	20
Sample holder	Platinum			
Completion date	06.01.2021			
Reference	Afinko methodology			
Equipment	Shimadzu, TGA-50			
Sample masses (mg)	**AFK211230:** 12,426	**AFK211231:** 10,894		

RESULTS

In this section we present results obtained of the fiber glass and PEHD composite samples through tests carried out in laboratories. To better describe the mechanical characteristics of composites, a systematic study of the flexural and impact properties of composites was conducted in the present research. The strengths of the samples ranged from 138 MPa to 10835 MPa. Different specimen sizes made following their respective standards were employed to investigate the effects of fiber reinforcement index on flexural and impact properties, including mechanical properties and crack patterns. The relationships between the fiber reinforcement index and the polyethylene properties were established in all tests and compared with the previous experimental values.

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Test Report

Machine: **Emic DL2000** Load cell: **Trd 24** Extensometer: **-** Date: **08/06/2020** Time: **18:50:56** Job n. **0884**
Program: **Tesc version 3.01** Test Method: **Igor compósito**
Sample Ident.: >>flexao : **IGOR FLEXÃO PE**

Specimen	Area (mm2)	Gauge Length (mm)	Force @Max. Load (N)	Stress @Max. Load (MPa)	Deformation @Max. Load (mm)	Strain @Max. Load (mm/mm)	Elast.Modul. (MPa)
Spc 1	1	102	51	50	24	0	425
Spc 2	1	102	50	49	28	0	352
Spc 3	1	102	44	43	27	0	358
Spc 4	1	102	33	32	24	0	270
Spc 5	1	102	40	39	26	0	323
Spc 6	1	102	18	17	25	0	138
Spc 7	1	102	17	16	25	0	181
Number Spcs	7	7	7	7	7	7	7
Mean	1,026	102,1	36,02	35,11	25,66	0,2514	292,5
Median	1,026	102,0	39,61	38,61	25,21	0,2472	323,3
Std. Dev.	0,0000	0,1512	14,36	14,00	1,378	0,01359	102,8
Coeff.Var. %	0,0000	0,1481	39,87	39,87	5,371	5,404	35,14
Min. Value	1,026	102,0	16,53	16,11	23,98	0,2351	137,5
Max. Value	1,026	102,4	51,49	50,19	27,56	0,2702	425,1

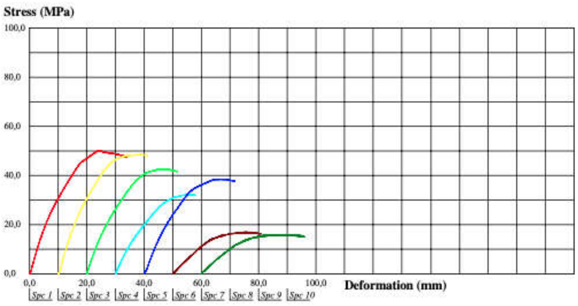

Fig.17. Graphic of Stress versus Deformation to PEHD results

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Test Report

Machine: **Emic DL2000** Load cell: **Trd 24** Extensometer: **-** Date: **08/06/2020** Time: **17:42:04** Job n. **0882**
Program: **Tesc version 3.01** Test Method: **Igor compósito**
Sample Ident.: >>> flexao : **Igor compósito vidroepoxi**

Specimen	Area (mm2)	Gauge Length (mm)	Force @Max. Load (N)	Stress @Max. Load (MPa)	Deformation @Max. Load (mm)	Strain @Max. Load (mm/mm)	Elast.Modul. (MPa)
Spc 1	1	102	260	253	7	0	6926
Spc 2	1	102	204	199	9	0	4574
Spc 3	1	102	411	400	6	0	8036
Spc 4	1	102	309	301	8	0	5425
Spc 5	1	102	236	230	14	0	4364
Spc 6	1	102	746	727	11	0	10793
Spc 7	1	102	207	201	4	0	5504
Number Spcs	7	7	7	7	7	7	7
Mean	1,026	102,1	338,8	330,3	8,415	0,08247	6517
Median	1,026	102,0	259,7	253,1	7,512	0,07365	5504
Std. Dev.	0,0000	0,1512	193,4	188,5	3,329	0,03268	2287
Coeff.Var. %	0,0000	0,1481	57,09	57,09	39,57	39,62	35,09
Min. Value	1,026	102,0	203,9	198,7	4,234	0,04135	4364
Max. Value	1,026	102,4	746,2	727,3	14,10	0,1383	10790

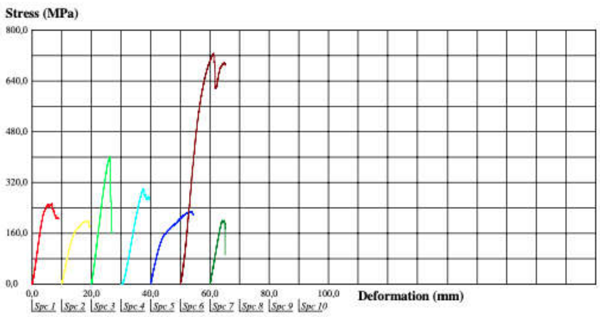

Fig.18 Graphic of Stress versus Deformation to glass fiber epoxy results

Flexural results: The incorporation of fibers that act as containment of cracks or barriers can change the failure mode of a specimen much more ductile. As the external loading increases, the microcrack appears and propagates in the matrix. The binding effect of the fibers was activated and provided a lateral barrier in the sample as the cracks extended [Linzhi, 2019]. The poor interface between the polymers increases the loss modulus and slightly decreases the impact strength of the composites [MARO, 2021]. The plastic deformation behavior of HDPE was experimentally examined. under different stress states. Macroscopic stress and volumetric strain were quantified while the transverse strain rate was controlled. Theoretical predictions and experimental results were found to be in good agreement, highlighting the relevance of constitutive representation for PEHD [*Mesbah, 2021*]. Figures 18 and 19 show the stress-strain graphs of a flexural test on specimens for composite materials and PEHD respectively. The main parameters highlighted during the analyzes were the following: (1) Force (N), (2) stress (MPa), (3) deformation (mm), (4) modulus of elasticity (MPa), (5) area (mm2) and (6) the length (mm). The performance of PEHD samples under cyclic loading conditions was evaluated and a physical scenario is proposed for the interpretation of the correlated macro-micro measurements: Structural deteriorations such as rupture, unraveling and lamella breaking are mainly induced by the first cycle. These structural deteriorations can induce modulus loss and compete with the orientation of stress/recrystallization transmitters associated with the strengthening effect [GUO, 2021].

Charpy results: The PEHD showed good mechanical strength during the tests, the flexural strength of PEHD ranged from 138 to 426 Mpa and the deformation in the Charpy test ranged from 8 to 11 Nm. The material used in CAPV's and which are commonly used in the oil and gas industry. PEHD is a UV stabilized polyethylene (PE) grade Borecene RM 7403. It has good flow and high rigidity. It is suitable for injection molding (IM) processing. Recommended for thick wall applications, large containers and foam articles.

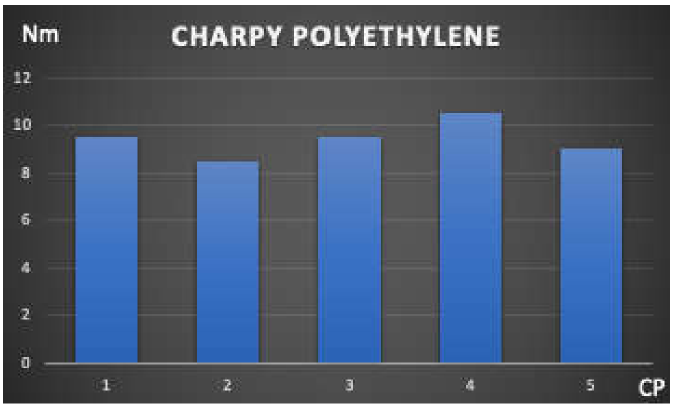

Fig.19. Values in Nm of Charpy PEHD results

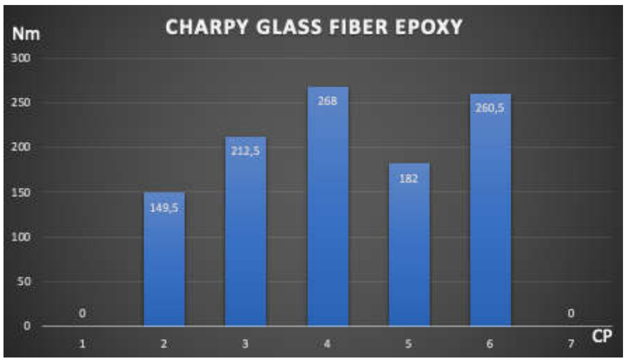

Fig.20 Values in Nm of Charpy Glass fiber epoxy results

M The FG composite demonstrates high mechanical strength during the tests, the flexural strength of the fiberglass ranged from 4364 to 10835 Mpa and the deformation in the Charpy test ranged from 149

to 268 Nm. The FG type is T30 SE 1500, it also It is suitable for use in filament tubes, tanks and atmospheric pressure vessels.

Charpy results with Glass fiber epoxy: The Differential Scanning Calorimetry (DSC) results can be observed through the DSC curves as shown in the following figures:

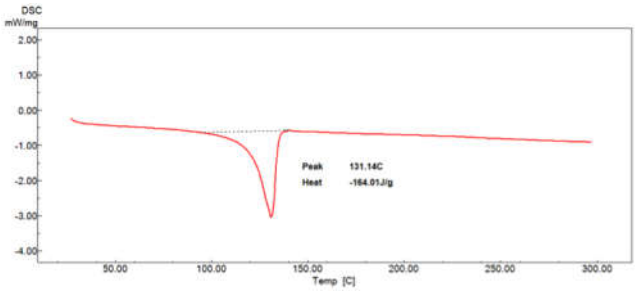

Fig.21 DSC curve of specimen AFK211230 – 1st heating

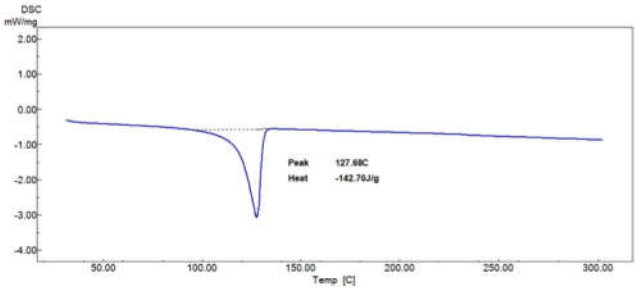

Fig.22 DSC curve of specimen AFK211230 – 2nd heating

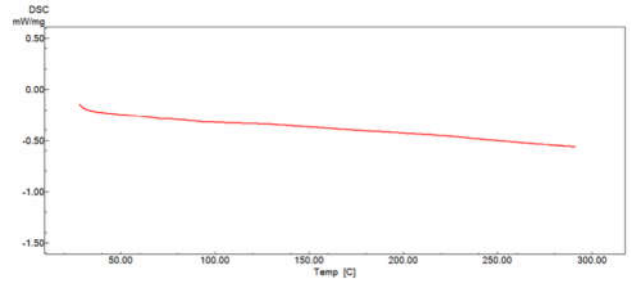

Fig.23 DSC curve of specimen AFK211231 – 1st heating

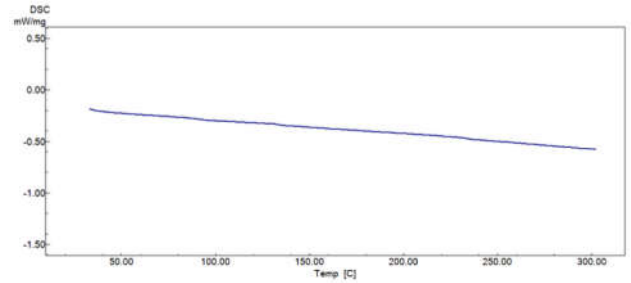

Fig.24 DSC curve of specimen AFK211231 – 2nd heating

We can indeed claim that DSC played a key role in clarifying fundamental scientific issues related to the glass transition and the glassy state. The glass transition is still considered one of the most challenging and interesting problems in condensed matter science [ZHENG, 2019].

SEM results: SEM micrographs showed that the fillers are finely distributed within the PEHD matrix. However, a direct correlation can be observed between the filler content and the tendency to form agglomerates [SALEH, 2020]. The figure below shows the micrographs of the AFK211230 specimen, showing the points where the EDS (Energy Dispersive Spectroscopy) analyzes were performed.

Table 4. DSC results

Specimen	1st heating Tm°C	2nd heating Tm°C
AFK211230	131,14	127,68
AFK211231	---	---

Table 5. Ash content results

Specimen	Ash Content		
	Measurements(%)	Avarage(%)	Avarage(mg/Kg)
AFK211230	0,168	0,142	1423,65
	0,116		
AFK211231	79,378	78,862	788618,56
	78,346		

The figure below shows the micrographs of the specimen AFK211231, showing the points where the EDS analyzes were performed.

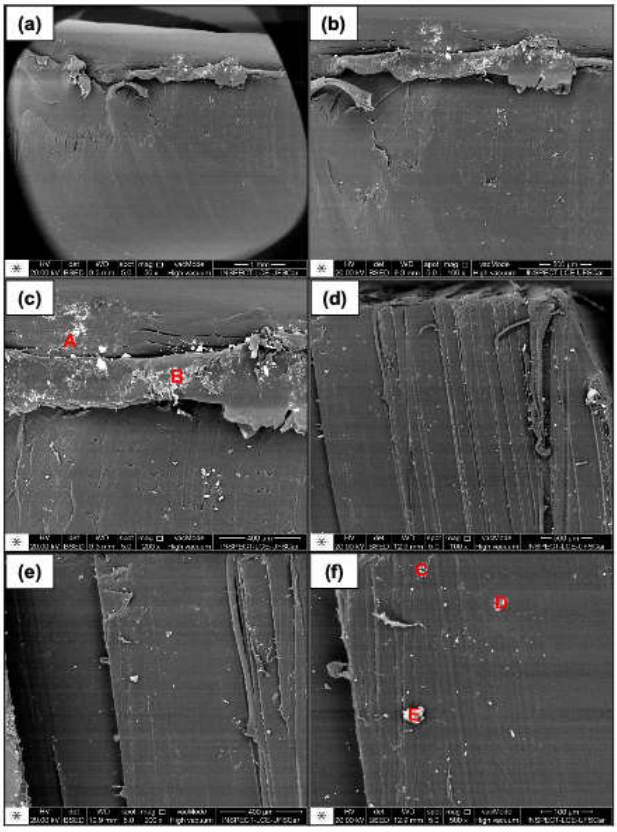

Fig. 25 Micrographs of specimen AFK211230, with magnifications of: 50X, (b) 100X, (c) 200X, (d) 100X, (e) 200X and (f) 500X

Ash Content results: The crystalline results around 128°C for the specimen AFK211230, considering the second heating, which reflects intrinsic properties of the material, which is characteristic of PEHD. The specimen AFK211231 did not present thermal transitions in the analyzed temperature range. The results of the SEM/EDS analysis of the specimen AFK211230 showed Carbon (C) as the major element, with low fractions of the elements Oxygen (O), Sodium (Na), Silicon (Si) and Chlorine (Cl), as well as traces of the elements Magnesium (Mg), Aluminum (Al), Iron (Fe), Nickel (Ni), Copper (Cu), Zinc (Zn), Potassium (K) and Calcium (Ca). As for the specimen AFK211231, where it is possible to observe the presence of fibers, the SEM/EDS results showed major fractions of Carbon (C) and Oxygen (O), with high fractions of Silicon (Si) and Calcium (Ca), in addition to several low fractions of Sodium (Na), Magnesium (Mg), Aluminum (Al) and

Chlorine (Cl). Still, this sample presented in some points the element Iron (Fe) with relatively high fractions and remnants of the elements Potassium (K) and Titanium (Ti).

Table 6. EDS of AFK211230 results

Element	(%) Mass				
	A	B	C	D	E
C	86,64	83,70	91,18	84,42	81,97
O	5,14	5,60	6,83	10,55	12,17
Na	0,46	0,42	1,00	2,18	0,54
Mg	---	---	---	---	0,19
Al	---	---	---	---	0,82
Si	0,49	0,59		0,33	2,00
Cl	---	---	0,99	2,52	0,71
Fe	3,18	3,39	---	---	---
Ni	2,06	1,79	---	---	---
Cu		2,80	---	---	---
Zn	2,02	1,72	---	---	---
K	---	---	---	---	0,51
Ca	---	---	---	---	1,12

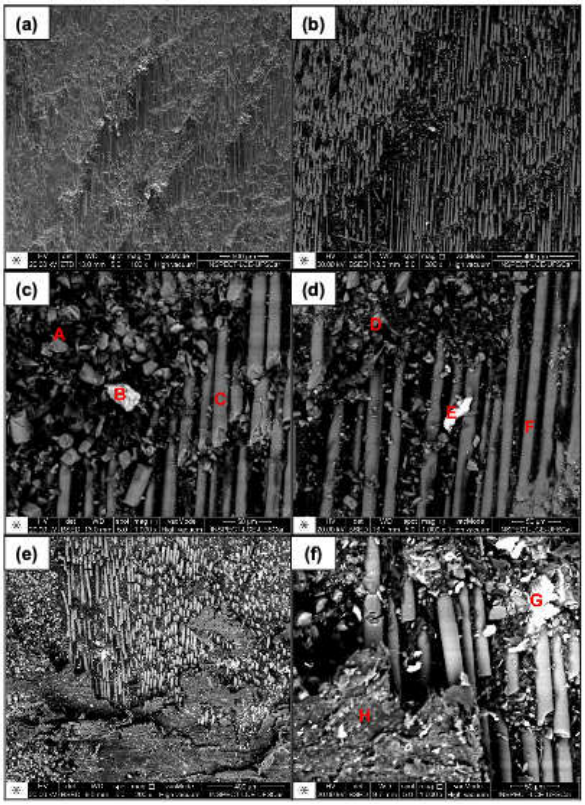

Fig. 26 Micrographs of specimen AFK211231, with magnifications of: 100X, (b) 200X, (c) 1000X, (d) 1000X, (e) 200X and (f) 1000X

Table 7. EDS of AFK211231 results

Element	(%) Mass							
	A	B	C	D	E	F	G	H
C	44,77	24,71	33,56	47,53	30,87	39,08	30,37	40,35
O	26,03	28,42	28,99	24,01	16,22	26,77	12,05	11,92
Na	0,71	0,30	0,37	0,73	0,64	0,69	---	0,95
Mg	0,84	0,81	1,14	0,88	1,18	1,06	0,77	0,72
Al	3,28	2,84	4,91	3,31	3,99	4,04	2,22	1,69
Si	14,29	10,54	20,48	14,25	17,42	18,54	10,47	4,44
Cl	0,61	0,37	---	0,77	---	---	2,03	16,30
K	0,33	---	---	---	---	---	0,31	---
Ca	9,14	6,28	10,55	8,51	9,06	9,82	5,99	6,05
Fe	---	25,74	---	---	20,61	---	27,34	5,89
Ti	---	---	---	---	---	---	8,46	11,70

Table 8. EDS of AFK211231 results

Property	AFK211230	AFK211231
Ash Content(%)	0,14	78,86

TGA results: Thermogravimetry (TG) results can be observed through the TG and DTG curves as shown in the following figures:

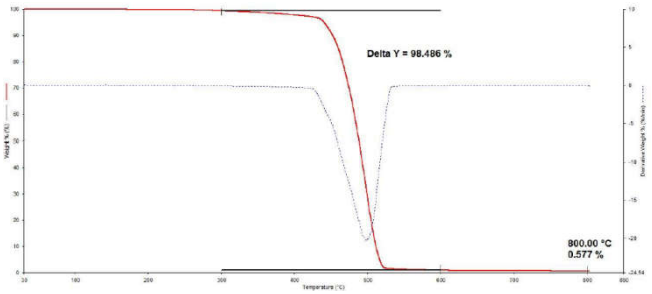

Fig.27 TG and DTG curves referring to specimen AFK211230

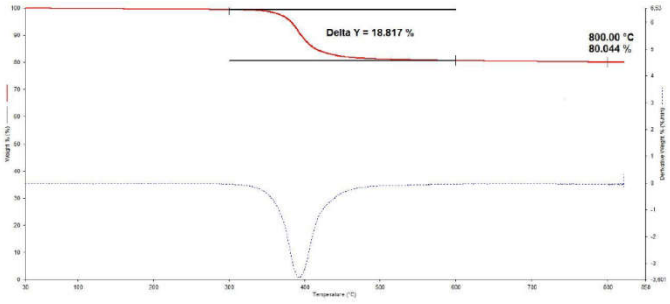

Fig.28 TG and DTG curves referring to specimen AFK211231

Table 9. TG results

Specimen	Temperature range (°C)	Weight loss (%)	Residue 800 °C
AFK211230	300 a 600	98,486	0,577
AFK211231	300 a 600	18,817	80,044

The results of the TG analysis indicated that for the specimen AFK211230 it presented a mass loss in the range of 300°C to 600°C around 98.5% and a stable residue at 800°C of approximately 0.57%, referring to the content of inorganic material in its composition. The specimen AFK211230 showed a loss of mass in the range of 300°C to 600°C and stable residue at 800°C of approximately 80.04%, referring to the content of inorganic material in its composition.

CONCLUSION

CAPV (composite air pressure vessel) have been used in several industries in the last two decades to storage fluids (liquids and gases), water, petroleum products and chemicals, among others. Lightweight CAPV are getting more attractive in the oil and natural gas industries, due to their significant advantages such as corrosion resistance and structural flexibility, lower transport and production cost, lower weight to higher specific strength, ease of repairs or maintenance and high durability against unfavorable weather condition. These inherent properties of injection molding (IM) to CAPV are extremely difficult to obtain in metallic, steel and concrete materials. The best manufacturing technique of fabricating CAPV is injection molding method. In addition to the properties of the fiber and PEHD, temperature, rotational speed and orientation/sequence are significant factors that determine the strength of the CAPV. There are several research studies on the CAPV. However, there is a very limited extensive review study on the raw materials used in the CAPV, production, utilization, repair and recovery of the CAPV, as well as recent developments and innovations.

Therefore, this study is to contribute to knowledge in aforementioned and neglected areas as well as proposed optimum CAPV for different areas of application in various industries. The composite materials used to CAPV fabrication and their applicability have been critically reviewed and reported within the scope of this paper. It was evident that external, internal and hydrostatic pressures are mainly used to analyze the leaks of the CAPV. Also, the cracks of CAPV occurs due a emptying or fill up operations that resulted in temperatures outside the limitations of the pressure vessels. The low temperature may result in shrinkage of the polyethylene inner liner and then leaks happens through the cracks, but it easily solved through correct operation to empty or fill CAPV, where the operator must to be awareness of differential pressure used in the system and also follow manufacture procedure to operations. Moreover, the durability properties of CAPV depend on their environmental conditions. The durability performances of CAPV reduce due to the adverse effects of environmental ageing or harsh conditions: ultraviolet rays from solar energy, hot medium, differential pressure, among others. These are worse if the fiber is an uncoated and untreated. The detrimental consequences, such as cracks, shrinkage of the polyethylene inner liner absorption, swelling, change in surface morphology, loss of aesthetics, week fiber–matrix interfacial adhesion which eventually reduces the mechanical and other structural properties of the CAPV.

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