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Onwudili, JA, Insura, N and Williams, PT (2013) *Autoclave pyrolysis of carbon reinforced composite plastic waste for carbon fibre and chemicals recovery.* Journal of the Energy Institute, 86 (4). 227 - 232. ISSN 1743-9671

http://dx.doi.org/10.1179/1743967113Z.00000000066

Autoclave pyrolysis of reinforced composite plastic waste for carbon fibre and chemicals recovery

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Abstract

Carbon reinforced composite plastic (CRCP) waste has been pyrolyzed under nitrogen atmosphere in a batch stainless steel autoclave between temperatures of 350 °C and 500 °C and reaction times of up to 60 minutes. The reaction products were dominated by solid residue (72 – 77 wt%), gas (2 – 4 wt%) and liquid (22 – 25 wt%). The solid residue consisted mainly of carbon fibre, giving up to 98 wt% recovery. However, the mechanical properties of the recovered fibres were lower when compared with a sample of virgin carbon fibre. The gas products contained mainly carbon dioxide and some hydrocarbon gases. The proportion of the combustible gases gave calorific values of up to 35MJ/m^3. The liquid products consisted of 15 – 20 wt% water, possibly from the degradation of the amide/ester linkages in the resin, and the rest was dark-brownish oils, soluble in dichloromethane. The TGA/DTG analysis of the CRCP waste indicated that the primary resin present could be based on polybenzoxazine polymer. The major components of the oils included aniline, methyl aniline and phenols. Apparently, these chemical products could be regarded as the monomers for making the resin and its curing agents (e.g. bisphenol-A). Hence, the autoclave pyrolysis allowed complete degradation of the resin fraction of the CRCP at moderately low temperature, along with the recovery of carbon fibre and chemical feedstocks.

Keywords: Pyrolysis; Waste; Reinforced composite plastics, Carbon fibre, Recycling

1.0. Introduction

Reinforced composite plastics contain between 5 and 60 wt % of reinforcing fibres which are usually glass, carbon, aramid or cellulose. These composite construction materials made of fibres enclosed in a plastic matrix, which are usually thermosetting plastics. Examples of reinforced composite plastics include complex structures such as carbon fibre composites, glass fibre composites, cellulose fibre composites and printed circuit boards. These reinforced plastics have desirable construction properties such as high durability, high stiffness, steady dimensions, appropriate structural strength and are resistant to corrosion [1-3]. They have been used in the construction of transportation and space applications, sports tools and electronics [2, 4-5]. The popular use of these fibres in automotive construction and other industrial sectors has been actively developed over the past few decades as they contribute to a significant total weight reduction in their applications [6-7].

The main thermosetting plastics utilized in composites such as matrix are epoxy resins and polyesters. Epoxy resins include many different types, but are mostly produced from epichlorohydrins and bisphenol. Some examples of the most common resins used include, diglycidyl ether of bisphenol A (DGEBA), N,N,N',N'-tetraglycidyl-4,4'-diaminodiphenylmethane (TGDDM) and N,N,O-triglycidyl-p-aminophenol [8]. These plastic types form high mechanical property composites that are utilized in structural applications [9]. The monomers are connected by fixed links, producing large molecules that begin as soon as curing agents such as amines and phthalic acid anhydrides are added. One of the amines used as a hardening agent is 4,4-di-amino diphenyl sulfonate [8]. In addition, polyesters such as ortho phthalic polyester [5] and iso-neopentyl glycol [9], straight chain glycols, branched glycols, ether-containing glycols and isophthalic acid and tetraphthalic acid based polyesters [10]. Thermosetting plastics cannot be re-melted and remoulded into new products. Currently, the main option for composite plastic waste disposal is landfill [6]. However, with the EU Directive for End-Of-Life Vehicle (Directive 200/53/EC) on the disposal of vehicles, only 15% of the total weight of a vehicle can be disposed in landfills. The remainder must be reused, recycled or treated for energy recovery with effect from 2006. In addition, from 2015 only 5 % could be sent to landfills, 10% treated for energy recovery and the remaining 85% reused or recycled [11].

Pyrolysis offers a potential route for the conversion and recovery of the organic fraction of reinforced composite plastic wastes. Many studies have investigated the thermal recycling of composite plastic wastes by pyrolysis [6, 12-13]. Depending on the type of plastic matrix present, different compositions of oil products can be obtained, which relate original resin, even in cases where different resin mixtures are pyrolyzed together [14-15]. Plastic materials can be converted into oil product containing short chain hydrocarbons that could be used as fuel or chemical feedstock. Cunliffe et al [6] investigated the pyrolysis of glass fibres, reinforced with thermosetting polyester/styrene copolymer plastic at a temperature of 450 °C in a fixed bed reactor. Oil product contained a high ratio of styrene and 40 % of it was within fuel oil range. The wax product included mainly phthalic anhydride.

Torresa et al. [5], pyrolysed sheet moulding compound waste, which consisted of glass fibres and ortho-phthalic polyester plastic, at a temperature of 30-700 ºC for 30 min. The oil product had a high calorific value of 34-37 MJ/kg and its composition consisted of C_5-C_{20} complex organic compounds that were mostly aromatic and oxygenated hydrocarbons. About 40 wt % of the oil product was distributed within the gasoline range, while the remainder was within the fuel oil. Lum et al. [16] investigated the thermal analysis of graphite and carbon phenolic composite using pyrolysis-mass spectrometry. At a temperature of 400 °C, the oil product contained low molecular weight species, the major species observed were phenol and methyl phenols. Smaller concentrations of formaldehyde, xylenol and toluene were also produced.

In this paper, the pyrolysis of a sample of carbon reinforced composite plastic (CRCP) waste has been carried in a closed batch autoclave reactor. Reactions have been studied at temperatures between 350 °C and 500 °C with reaction times between 0 and 60 minutes. The use of an autoclave reactor could enable secondary reactions, which could in turn lead to the decomposition of the plastic fraction of the CRCP at moderately low temperatures, leading to the recovery of carbon fibres and chemical feedstocks

2.0. Experimental

2.1. Materials

A 300 ml mini bench-top stirred pressure reactor was used for pyrolysis and was supplied by the Parr Instrument Company, IL, USA. Details of the autoclave reactor have been provided in a previous paper [17]. Briefly, the reactor was made of stainless steel, with a maximum working temperature of 500 °C. It is fitted with 3 valves comprising of a gas in-let valve, gas outlet valve and liquid sampling valve. The reactor was heated by means of a Watlow microprocessor based to the designated temperature and held for pre-determined reaction time ranging from zero to 60 min. The reactor temperature was monitor by means of J type thermocouples, while a gauge mounted on the reactor head was used to monitor the running pressure. Carbon fibre composite plastic (CRCP) waste used in this study was in the form of flat sheets of woven carbon fibres reinforced by plastic materials. The ultimate and proximate compositions of the CRCP waste are shown in Table 1.

2.2. Methods

2.2.1. Proximate and elemental analysis of the CRCP waste

Three replicate samples of the CRCP waste were analyzed on thermogravimetric analyzer to determine the proximate composition of the material. The instrument used was a Metler Toledo TGA/DSC 1 Star System. Approximately 20 mg (± 0.50mg) was loaded into the sample pan and heated under nitrogen atmosphere from 50 °C to 1000 °C at a constant rate of 20 °C min^{-1}. The elemental compositions (CHNS) of the CRCP waste of the solid products were determined by using between 3 -5 mg of the sample on a Carlo Erba Flash EA 1112 compact analyser for automated determination [18].

2.2.2. Pyrolysis procedure

For batch pyrolysis experiments, the CRCP sample was cut into small pieces of around 3-4 cm in length and 1 cm width before it was introduced to the pyrolysis reactor in order to maintain a suitable length for mechanical analysis. The reactor was tightly sealed by means of six screws and purged with nitrogen gas for 10 minutes. The reactor was then charged with 0.5 (0.05MPa) bar of nitrogen pressure prior to heating. Experiments were carried out in the closed autoclave reactor between the temperature range of 350 and 500 ºC. The reaction times at the designated temperatures ranged from zero to 60 min. The operating pressures due to the hot gases released during the pyrolysis reached a maximum of approximately 90 bar (0.90MPa) at 500 °C. After reaction, the reactor was quickly cooled to room temperature, the cooled gas pressure noted and the gaseous effluent sampled for analysis. The gas samples were quickly analyzed offline by means of two Varian 3380 gas chromatographs for permanent gases and hydrocarbons, respectively. The analytical procedure used has been previous reported in a previous publication [17]. Using the general gas equation, the yield of each gas component was obtained by applying the volume % obtained from the GC and the pressure exerted by the gas products after cooling the reactor to room temperature. The higher heating value (HHV) of the gas product was estimated as the sum of products of the volume/mole fractions and calorific values of individual combustible components present.

The solid and liquid products were separated via vacuum filtration. This was followed by rinsing of the solid products left on the filter paper with dichloromethane (DCM) under vacuum to extract any remaining liquid product. Since the yields of gas products and solid products could be measured experimentally, the yield of the liquid product was then obtained by difference. The liquid product consisted of a mixture of aqueous and organic phases. The liquid product was analyzed by Dean-Stark Method [19], to determine the proportions of aqueous phase and oil phase, respectively. The DCM-soluble organic phase was separated from the aqueous phase by liquid-liquid extraction and analyzed by gas chromatography-mass spectrometry. The GC/MS instrument used was a ThermoQuest CE Instrument GC 2000 series with a Restek RTX-5MS 30 m x 0.25 mm, 0.1 mm film depth column, of fused silica, 5 % diphenyl and 95 % dimethyl polysiloxane. The injector was held at 250 °C and the oven temperature was held at 40 °C for 4 min then ramped at 5 °C min^{-1} to 100 °C for 1 min, and further ramped at 10 °C min^{-1} to 280 °C, and then held at 280 °C for another 38 minutes. The GC is fitted with Thermo Scientific AS 2000 auto sampler and is coupled to a Fisons MD800 single-quad mass detector through the transfer line held at 250 °C. The carrier gas used was helium. The MS source was held at 200 °C with an emission current of 150 µA. The detector was operated at full scan from 50 to 500 amu.

The separated solid residue was dried for 2 hours in an oven at 105 °C to a constant weight. However, the solid residues obtained from the pyrolysis experiments consisted of both recovered carbon fibre and char. The char was removed by burn-off in a muffle furnace at 450

°C to obtain the amount of carbon fibre. The maximum burn-off at this temperature occurred after 2 hours, after which the carbon fibre was cooled and weighed.

3.0. Results and Discussion

3.1. Thermogravimetric analysis of CRCP

The TGA and DTG profiles of the CRCP waste shown in Fig. 1, shows that degradation of the plastic fraction started just under 300 °C and the maximum degradation temperature (T_{max}) was about 380 °C. The weight loss between 290 °C and 480 °C on the TGA thermograms was calculated to obtain the percentage of volatile components in the sample. From this result, the temperature for the char burn-off process earlier mentioned in the 'Methods' section was decided. The results from the burn-off process showed that the CRCP had a composition ratio of around 55-60 wt% carbon fibres and 40-45 wt% resin (thermosetting plastic). The TGA/DTG data along with FTIR data (not shown) and the elemental data in Table 1 appear to suggest that the main resin used in the CRCP waste may be based on polybenzoxazine.

Low and Ishida [20] reported that bisphenol-A had the lowest stable linkage among the structures of polybenzoxazine-based resins. In their work with the polybenzoxazine polymer based on bisphenol-A and aniline, the bisphenol-A, initial thermal degradation was attributed to the cleavage of the isopropylidine group of bisphenol-A that occurred at approximately 380 °C. However, the higher thermal stability of the 2, 2-biphenol linkage delayed the main derivative fragmentation peak to 500 ºC [20]. Considering the results of the TGA in the work, there were striking features between the degradation of this present sample and polybenzoxazine resin, indicating that this may be the main polymer in the CRCP waste used in this work.

3.2. Pyrolysis of CRCP

3.2.1. Effect of reaction temperature: The effect of temperature on the pyrolytic degradation of the carbon reinforced composite plastic waste was investigated between 350 °C and 500 °C for a reaction time of 60 min. The results presented in Fig. 2 shows that solid residue (comprising recovered carbon fibre and pyrolytic char) accounted for over 70 wt% of the reaction products.

This was not surprising as the reinforcing carbon fibre was not expected to degrade under the pyrolysis conditions. However, a combination of reaction temperature and extended reaction time of 60 min ensured that considerable degradation of the plastic fraction of the CRCP was achieved even at 350 °C.

Gas production was relatively generally low but tended to increase with increasing temperature. Interestingly, the proportion of liquid products was highest at 400 and 450 °C but fell at 500 °C. It would appear that the decrease in the yield of liquid products corresponded to increase in both gas and solid residue yields due to secondary pyrolysis reactions of the components of the liquid products. Evidence for the possible conversion of the liquid products could be obtained from the molar compositions of the gas products in relation to reaction temperature as shown in Fig. 3. It can be seen that at lower temperatures, carbon dioxide was the dominant gas species as a result of primary degradation of the resin. However, the yields of hydrocarbon gases, especially the C1-C4 alkanes increased considerably with temperature. This increase in hydrocarbon gas yields corresponded to increase in the overall calorific value of the gas products. The stripping reactions that led to the formation of hydrocarbon gases, would partly lead to condensation of carbon atoms to form pyrolytic char. Hence, the slight increase in the yield of solid residue between 450 and 500 °C.

The liquid product was found to contain about 15-20 wt % water with the remainder consisting of dark-brownish oil. The presence of water in the liquid product from pyrolysis of different plastics such as polyester, polyurethane and epoxy resins has been reported by several studies [5, 20]. Water formation has been attributed to the oxygen content in the polymer functional groups such as -COOH, -COO and -OH [5, 20].

Table 2 shows the composition of the oils derived from the pyrolysis of the CRCP in relation to temperature. Clearly, the proportion of aniline produced increased with temperature and could indicate its stability under the pyrolytic conditions. Nahil and Williams [21] carried out pyrolysis of CRCP samples in a fixed bed reactor at 600 °C, and also found aniline to be the major component of the oil product. The formation of aniline and phenols can be attributed to the decomposition of the polybenzoxazine-based resin, possibly cured with bisphenol-A, present in the reinforced plastic waste [17, 22]. However, that work was carried out in a nitrogen flow reactor, whereas this work used a closed autoclave reactor where the evolved gases remain under pressure and temperature within the reactor.

3.2. 2. Effect of reaction time: The effect of reaction time on the pyrolysis of CRCP waste was investigated at 400 °C for reaction times of 0, 30 and 60 min, respectively. Table 3 shows the distribution of products during these experiments. Again, the solid residue, made up of char and carbon fibre, was the dominant product, accounting for over 72 wt%. There was a dramatic decrease in the yield of solid residue, which corresponded to slight increases in the yields of gas and liquid products, as a result of enhanced cracking of the resin. However, the increase in the yield of solid residue, and indeed other products, was not very significant between 30 min and 60 min reaction time, indicating that the degradation process was complete after 30 min.

Carbon dioxide remained the dominant component of the gas products as shown in Fig. 4; however the proportion of carbon dioxide in the gas decreased in terms of mole percent, as the formation of hydrocarbon gas increased as a result of secondary reactions. These secondary reactions could involve cracking of the components of the liquid products and also gas phase reactions including methanation. Similar to the trends obtained with increasing temperature, the proportion of hydrocarbon gases, especially the alkanes, increased with increasing reaction time at 400 °C, indicating increased pyrolytic degradation which would result in an increase in the calorific value of the gas products.

In essence, the conversion of thermally unstable intermediates in the liquid products to gas would leave a cleaner oil fraction containing mainly stable compounds as well as higher resin removal efficiency from the carbon fibre. This suggested that these ‘others’, could be actively involved in the secondary reactions to produce gas, thus enriching the oil fractions with simple but stable monomeric compounds such as aniline and toluidines (methyl anilines). The proportion of phenol also increased, seemingly at the expense of alkyl phenols, possibly due to a similar mechanism.

3.3. Recovered carbon fibre

3.3.1. Carbon fibre recovery: The solid residue obtained from the reactor was oxidized in a muffle at 450 °C for two hours to remove any pyrolytic carbon (char) left or deposited on the carbon fibre. Fig. 5 shows that in general, over 94% recovery of the carbon fibre was obtained from the pyrolysis work, after oxidation of the solid residues. The recovery was based on the wt% of carbon fibre obtained from the TGA analysis. Although, the percentage recoveries appear to slightly decrease with the severity of the reaction conditions, the results are not significantly different and could be due to random errors.

In Fig. 6, the transformations of the CRCP (Fig. 6a) after pyrolysis are presented. The photograph of the recovered material is shown in Fig. 6b. In comparison, there are hardly any physical differences between the textures of the recovered and virgin fibre. Furthermore, the SEM images (Figs. 6c and 6d) of the virgin and recovered carbon fibres, respectively at two different resolutions, did not show any significant difference in their physical appearances.

3.3.2. Mechanical properties: Further mechanical tests on the recovered carbon fibres were carried out to compare them with the strengths of virgin fibres. The results of these tests are presented in Fig. 7, which shows the changes in the mechanical properties of the recovered fibre after pyrolysis at 400 °C at different reaction times.

From Fig.7, it can be seen that mechanical properties such as breaking force (N), elongation (%) and tensile stress (GPa) all decreased with increasing reaction times, especially after pyrolysis for 30 min and 60 min, respectively. On the other hand, the Young’s Modulus of the recovered fibres, which is a measure of the stiffness of a material, increased with the increased reaction time. This could be linked to the reduction in their elongation, indicating some form of shrinking of the fibres after pyrolysis and/or oxidation.

4.0. Conclusions

Autoclave pyrolysis of carbon reinforced composite plastic (CRCP) was carried out between a temperature range of 350 °C to 500 °C and in a batch reactor, under nitrogen atmospheres. TGA results indicated that the resin present in the CRCP waste could be based on polybenzoxazine polymer made from bisphenol-A and aniline. Degradation of the CRCP was substantial even at 350 °C yielding a mixture of solid liquid and gaseous products. By far, solid residue was the dominant product due to the presence of carbon fibre, which would not degrade under experimental conditions.

The gas products consisted mainly of carbon dioxide due to the decarboxylation of the ester linkages in the polybenzoxazine-based resin. The decomposition of the ester and amide linkages may be responsible for the formation of about 15 – 20 wt% of water in the liquid products. However, increasing severity of the reaction conditions, in terms of reaction temperature and reaction times, led to an increase in the hydrocarbon contents of the gas products due to secondary cracking reactions. While the yield of solid residue remained fairly stable, investigation on the effect of reaction time, suggested that the liquid products were the source of the hydrocarbon gases. Aniline was the main organic components of the oil fraction. Substantial yields of o-toluidine, phenols and alkyl phenols were also found in the oil.

The solid residue consisted of char and carbon fibre. Further oxidative treatment of the residues showed that up to 98% of the original carbon fibre could be recovered. However, the mechanical properties of the recovered fibres were lower compared to a sample of virgin fibre. Further analysis of the surface properties of the recovered carbon fibre would be required in order to find suitable uses for them

Thus, autoclave pyrolysis has the potential for moderate temperature pyrolysis of CRCP waste to recover the carbon fibre and a part of the monomers used in making the resin, in the form of aniline and phenol. In addition, the gas product obtained at higher temperatures gave calorific values of up to 35 MJ/m^3, and could easily be used as fuel for the process.

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Table 1: Proximate and ultimate compositions of the CRCP waste used in this study

Parameter	**Proximate Analysis (wt%)**
Moisture	-
Volatiles	28.8
Fixed carbon	69.8
Ash	1.40
	Ultimate Analysis (wt%)
Carbon	68.3
Hydrogen	6.30
Nitrogen	8.15
$^{\alpha}$ Oxygen	16.5
Sulphur	1.80

$^{\alpha}$ calculated by difference

Table 2: Compositions of oil fractions from the pyrolysis of CRCP in relation to temperature

Compounds, % of total peak area	Pyrolysis Temperature, °C		
	400	450	500
Anisole	0.27	0.00	0.00
Phenol	4.93	5.12	6.79
Aniline	26.9	32.2	36.1
o-cresol	0.50	0.65	0.92
m/p-cresol	1.64	1.80	2.30
o/p-toluidine (methyl anilines)	7.68	7.98	8.80
Ethyl phenol	1.06	1.19	1.30
2-Dimethylphenol	0.28	0.50	1.70
Dimethyl aniline	0.42	0.45	0.61
2-Isopropylphenol	1.38	1.00	1.15
Trimethyl phenol	0.86	0.93	1.15
4-Isopropyl phenol	8.00	7.37	2.74
2-methyl-5-isopropyl phenol	1.20	1.15	1.09
2,5-Bis(1-methylethyl)- phenol	2.29	2.17	2.50
N-methyl indole	1.01	0.87	0.56
2-methyl naphthalene	0.99	1.26	1.22
Dimethyl indanone	1.25	2.92	3.26
8-methyl-quinoline,	0.76	1.19	1.73
2,6-dimethoxy phenol	1.60	0.53	0.47
Cyanophenol	0.97	1.12	1.22
Trimethyl-indanone	1.12	1.57	1.43
2,5-dimethyl indole	2.32	1.04	0.56
Bibenzyl	1.55	0.50	0.30
4-phenyl phenol	0.92	0.85	0.65
o-Terphenyl	1.09	1.59	0.81
Phenyl phthalate	2.20	0.78	0.20
Triphenyl benzene	1.70	0.68	0.15

Table 3: Product distribution in relation to reaction time at 400 °C

Reaction Time (min)	Solid Residue (wt%)	Gas Yield (wt%)	$^{\beta}$ Liquid Yield (wt%)
0	77.0	2.4	20.6
30	72.5	2.8	24.7
60	72.0	3.0	25.0

$^{\beta}$ Liquid yield obtained by difference

FIGURE CAPTIONS

Figure 1: TGA/DTG profiles of the carbon reinforced composite plastic waste

Figure 2: Product distribution in relation to reaction temperatures

Figure 3: Gas product composition in relation to reaction temperatures

Figure 4: Composition of the gas products in relation to reaction time at 400 °C

Figure 5: Carbon fibre recovery during autoclave pyrolysis of the CRCP sample

Figure 6: (a) Picture of CRCP waste (b) Picture of recovered carbon fibre after oxidation (c) SEM image of virgin carbon fibre; Low magnification(d) SEM image of recovered carbon fibre; Low magnification (e) SEM image of virgin carbon fibre; High magnification (f) SEM image of recovered carbon fibre; High magnification

Figure 7: Mechanical properties of the recovered carbon fibres

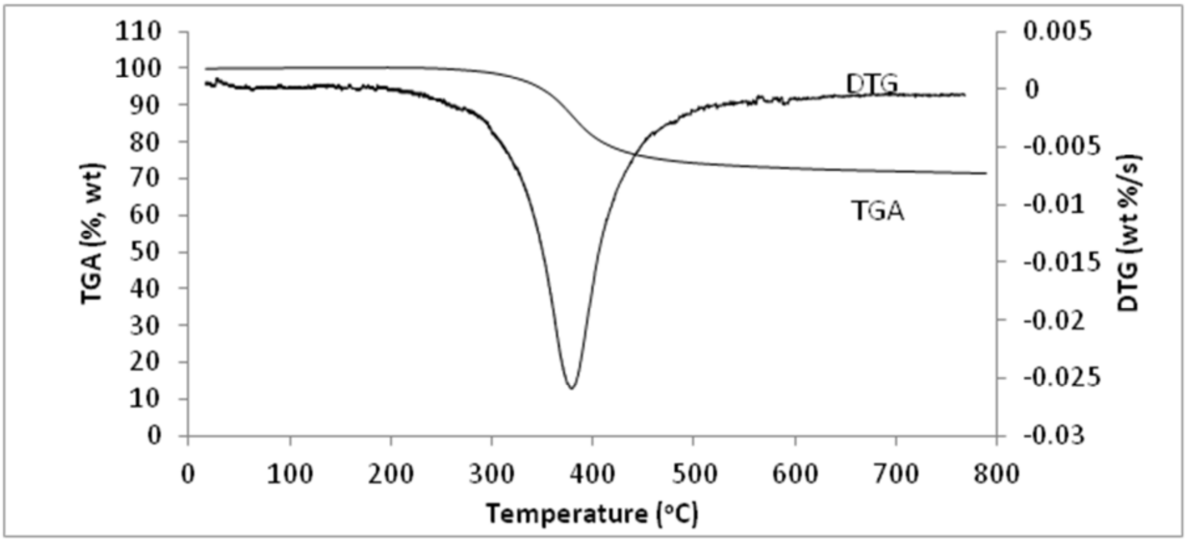
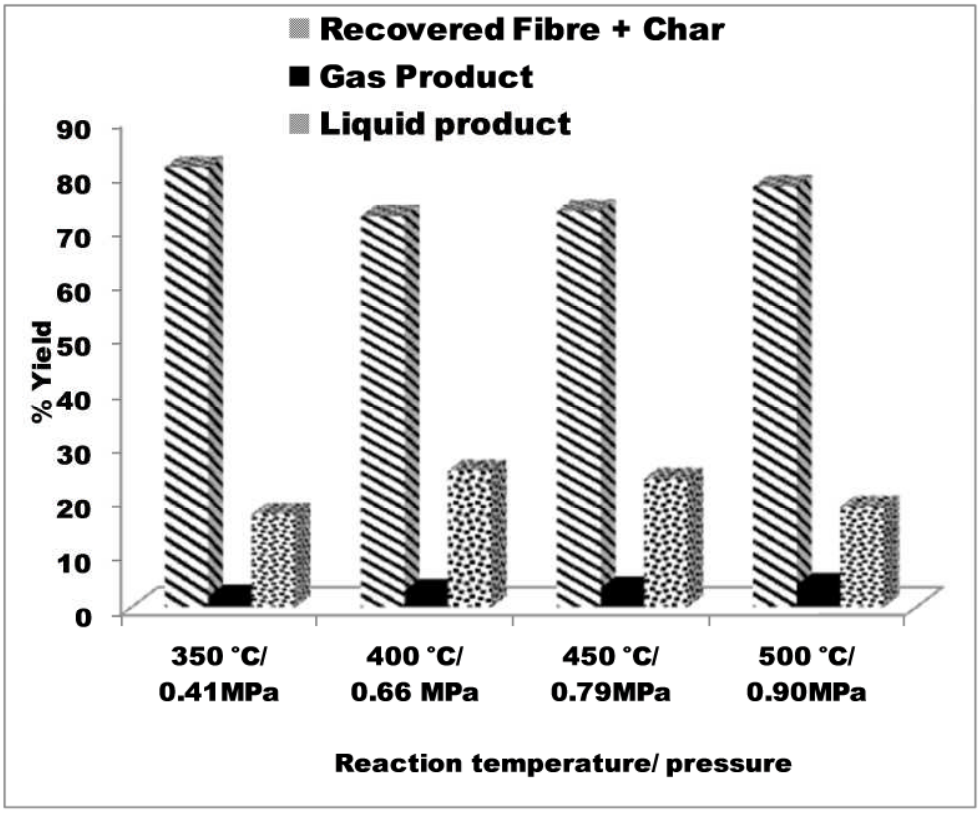
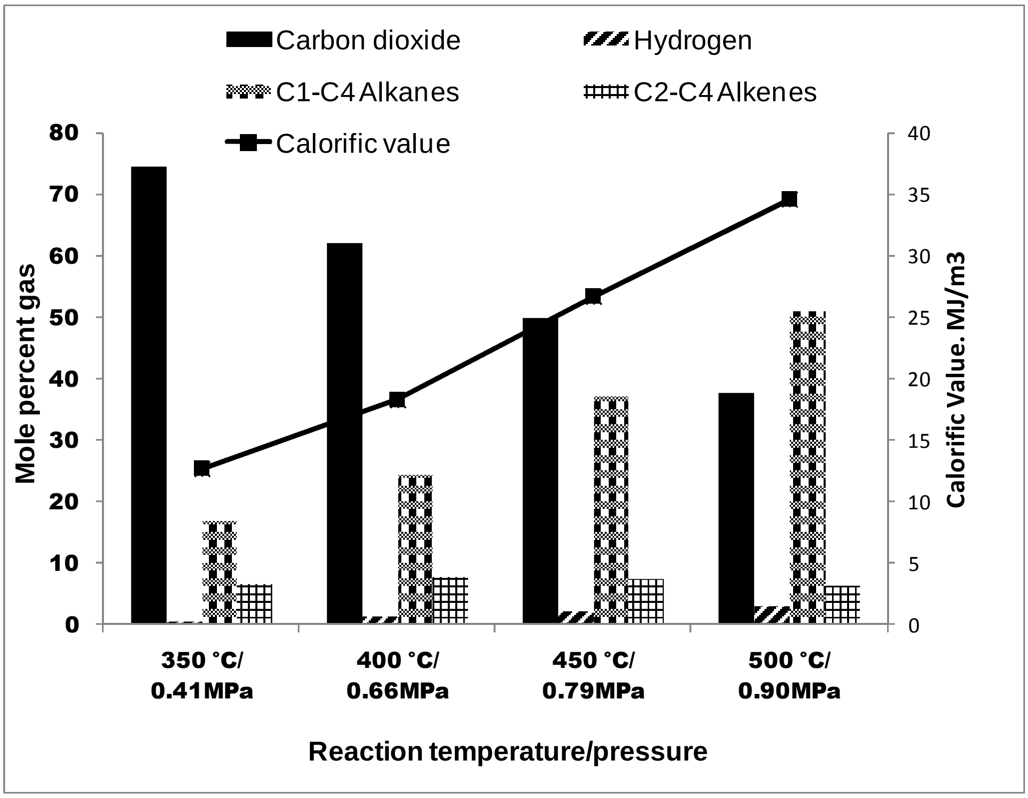


Figure 1: TGA/DTG profiles of the carbon reinforced composite plastic waste



Footnote: Pressure values stated are observed values during each experiment

Figure 2: Product distribution in relation to reaction temperatures



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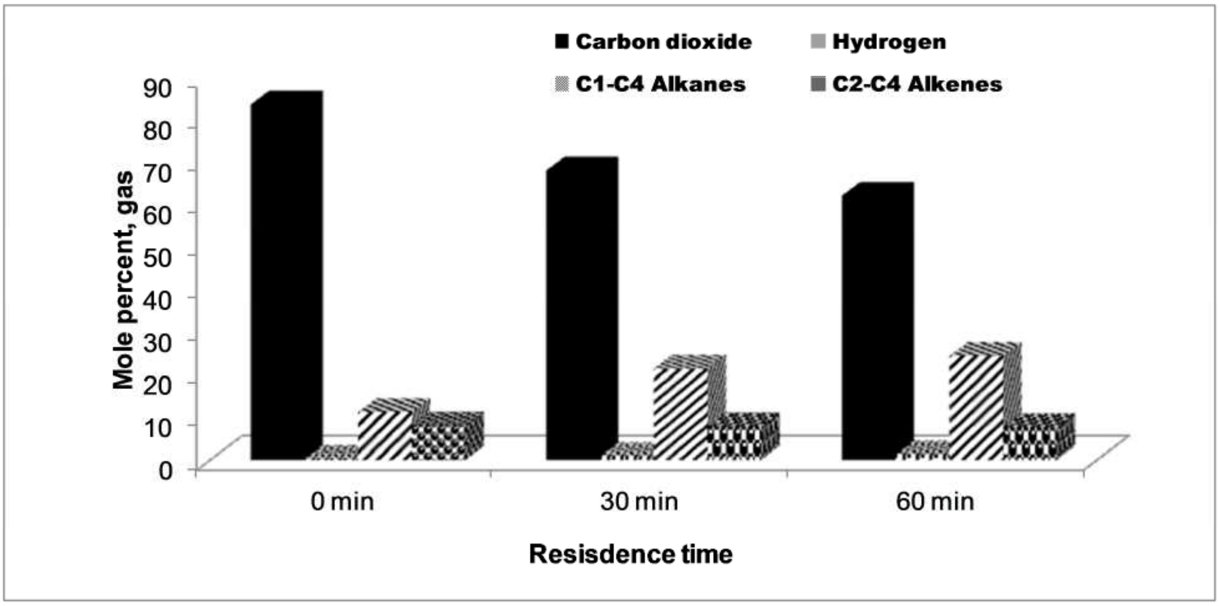


Figure 4: Composition of the gas products in relation to reaction time at 400 °C

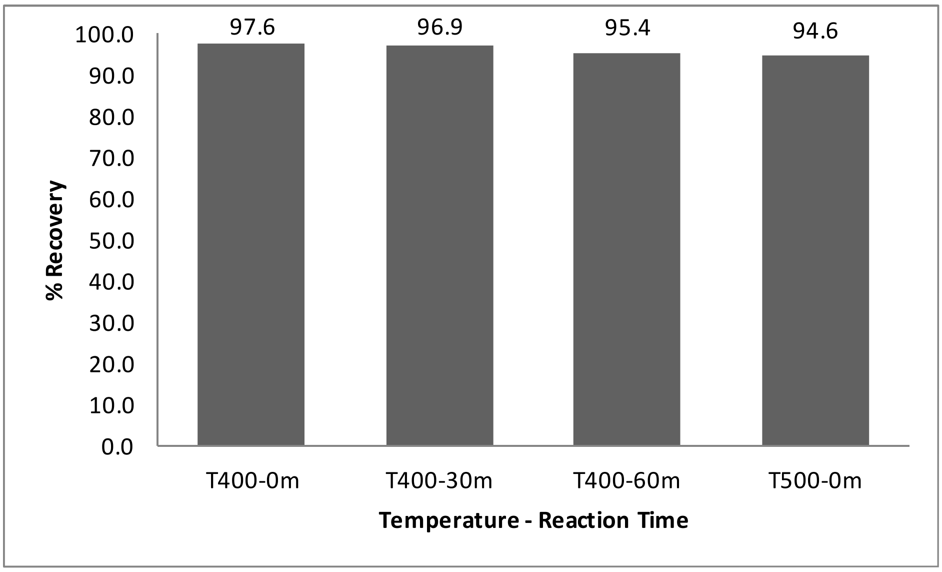
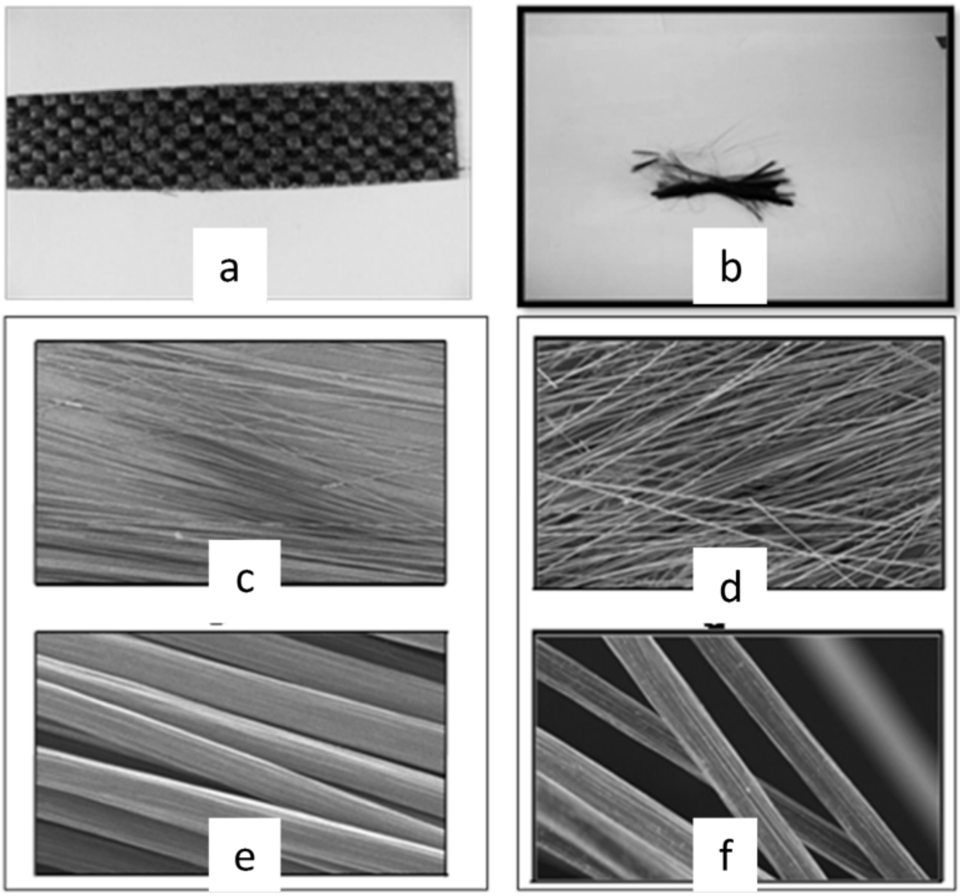


Figure 5: Carbon fibre recovery during autoclave pyrolysis of the CRCP sample



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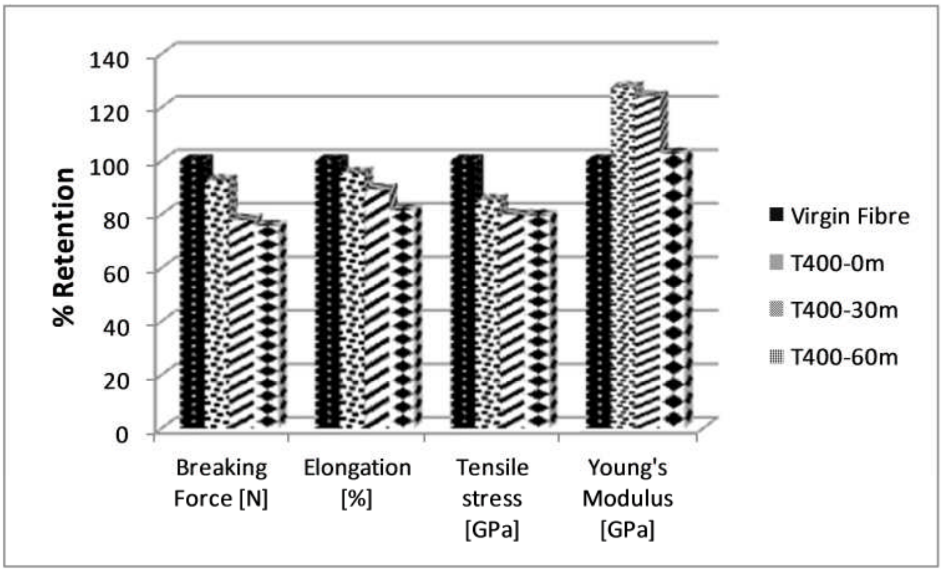

Figure 7: Mechanical properties of the recovered carbon fibres