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End-of-life recycling options for glass fibre reinforced polymers

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End-of-life recycling options for glass fibre reinforced polymers

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Abstract

This report covers the possible recycling options for end-of-life glass fibre reinforced polymers. Two forms (jigsaw and diamond bladed wet saw) of composite size reduction were compared to determine the most viable option. It was determined that the use of a jigsaw is essential due to its portability and low cost; being $\approx 36\%$ less than the cost of the wet saw. Incineration with fibre recovery was performed at six different temperatures to determine which was the best to achieve complete resin decomposition. It was found that the most effective temperature, in terms of cost, burn-off time and effectiveness was 500°C . Incineration with energy recovery tests were performed using the bomb calorimeter. It was determined that the recoverable energy was 7MJ/kg . These two tests were conducted in order to determine the most feasible option for dealing with end-of-life waste composites. It was concluded that the most feasible option was incineration with energy recovery. This is due to its potential in aiding the process of cement production. The matrix has potential to be used as a fuel due to its high calorific content and the reinforcement could be integrated into the cement itself.

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Nomenclature

Symbol	Property	Unit
ACMC	Advanced Composites Manufacturing Centre	-
A_w	Areal Weight of the fabric	g/m ²
BMC	Bulk Moulding Compounds	-
CaCO₃	Calcium Carbonate	-
COSHH	Control of Substances Hazardous to Health	-
C_{pc}	Composite Specific Heat Capacity	J/kgK
C_{pf}	Fibre Specific Heat Capacity	J/kgK
C_{pm}	Matrix Specific Heat Capacity	J/kgK
DEFRA	Department for Environment Food and Rural Affairs	-
EC	European Commission	-
ECRC	European Composites Recycling Concept	-
ELV	End-of-Life Vehicle	-
EU	European Union	-
EOL	End-of-Life	-
EuCIA	European Composites Industry Association	-
EXP1	Experiment 1	-
EXP2	Experiment 2	-
EXP3	Experiment 3	-
FRP	Fibre Reinforced Polymer	-
GFRP	Glass Fibre Reinforced Polymer	-
LTCS	UK Landfill Tax Credit Scheme	-
NCF	Non-crimp fabric	-
ROM	Rule of Mixtures	-
SEM	Scanning Electron Microscope	-
SMC	Sheet Moulding Compound	-
TRL	Technology Readiness Level	-
V_f	Fibre Volume Fraction	%
V_{filler}	Filler Volume Fraction	%
V_m	Matrix Volume Fraction	%
WID	Waste Incineration Directive	-
n	Number of layers	-
t	Thickness	mm
ρ_f	Fibre Density	kg/m ³

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Aim

To assess the economic viability and End-of-Life options for Glass Fibre Reinforced Polymers (GFRP's).

Objectives

- To consider means to reduce large waste composites to a size which can be transported to appropriate recycling facilities.
- To calculate the energy consumption in the above process.
- To expose laminates to high temperatures and plot mass vs. time curves for complete resin elimination.
- To calculate the energy consumption within this process.
- To measure retrievable energy in waste GFRP through use of the bomb calorimeter.

1.0 Introduction

Composites have become increasingly popular due to their low weight, high mechanical properties and aesthetics (Otheguy, 2009). Globally, approximately 90% of the Fibre -Reinforced Polymers (FRP) are composed of glass fibres and a thermoset resin (Garcia, 2014). It is estimated that the European production volume, of Glass Fibre Reinforced Polymers (GFRP), increased to over 1 megatonne in 2015 (Dr. Witten, 2015).

With an increased quantity of GFRP reaching their end-of-life (EOL), the question of how to reuse, recycle, recover or dispose of the composites is growing in importance. The preference of dealing with EOL and waste composites is in accordance with the

waste hierarchy (Halliwell, 2006; Summerscales, 2015). The waste hierarchy (DEFRA, 2011) is as follows:

Prevention > Preparing for Reuse > **Recycling** > other **Recovery** > Disposal

For the disposal of GFRP, traditional routes such as landfill or incineration are becoming increasingly expensive or less available (Halliwell, 2006; Jacobs, 2011; Job, 2010), not least because the European Commission (EC) has implemented the Waste Framework Directive (2008/98/EC). Similarly, the reuse of waste composites is not always possible. With reuse not always possible and disposal being less favourable, it has given rise to waste composite recycling, particularly in the form of mechanical and thermal processing (Jacobs, 2011). The recycling of thermoset composites is difficult due to their three-dimensional intertwined, cross-linked network structure, which means they cannot normally be remoulded (Garcia, 2014; Pickering, 2006).

When using the mechanical processing techniques, the waste composites are ground or milled into a finer regrind. The regrind is separated into three grades of recyclate (coarse, medium and fine). These mechanical recycling by-products may then be used in various forms as filler or reinforcement within new composite products (Halliwell, 2006; Otheguy, 2009) or can be transported more easily to facilities for alternative treatments.

For thermal processing there are two principal techniques; recovery of fibres, and incineration with energy recovery (Summerscales, 2015). The recovery of fibres involves the waste composite being subject to high temperatures for specific periods of time in order to fully decompose the resin; leaving clean, useable recovered glass fibres. Incineration with energy recovery involves the waste composite being subject to incineration to recover embodied energy, e.g. waste composite being used as an alternative for fuel within a cement kiln (Jacobs, 2011).

All of the above methods, whether it be reuse, recycling or disposal, require significant energy inputs, so consideration of the economic viability of these processes will be considered (Summerscales, 2015).

2.0 Theory/ literature review

Halliwell (2006), Conroy et al. (2006), Pickering (2006), Yang et al. (2012), Palmer (2009), Garcia (2014), Otheguy et al. (2009), Job (2010), Summerscales (2016) and Jacobs (2011) have all reviewed the EOL options GFRP.

The literature reviewed in this report considered the legislation affecting EOL GFRP, and their thermal recycling techniques.

Halliwell (2006) has divided the recycling techniques for waste composites into four categories:

- *Primary recycling* – Conversion of waste into a material with properties equivalent to those of the virgin material
- *Secondary recycling* – Conversion of waste into a material with properties less than that of the virgin material
- *Tertiary recycling* – Conversion waste into fuel or chemicals
- *Quaternary recycling* – Conversion of the composite waste into energy (Halliwell, 2006; Palmer, 2009; Pickering, 2006; Summerscales, 2010)

There are further processes at an early stage of development as indicated by the Technology Readiness Level (TRL) (see Appendix H: TRL Figure). The TRL is a measurement system used to assess the maturity level of a particular technology/process. There are nine TRL's, TRL 1 is the lowest and TRL 9 is the highest. TRL 1 is a technology/process that is still at the research stage, whilst TRL 9 has already been successfully implemented and tested (Mai, 2015).

2.1 Legislation

With an increase of GFRP component manufacture and increasing penetration into renewable energy devices and construction sectors, EU directives will be putting more pressure on solving the GFRP waste management (Conroy et al., 2005; Halliwell, 2006). The key Directives are:

- EU Waste Framework Directive
 - Directive 2008/98/EC
- End-of-Life Vehicle (ELV) Directive for the automotive sector
 - Directive 2000/53/EC
- EU Directive on landfill of waste
 - Directive 1999/31/EC
- UK Landfill Tax Credit Scheme (LTCS)
- Waste Electrical and Electronic Equipment (WEEE)
 - Directive 2012/19/EU
- Waste Incineration Directive (WID)
 - Directive 2000/76/EC

(Halliwell, 2006; Palmer, 2009; Pickering, 2006; Summerscales, 2010)

The EU waste management regulation provides the main regulatory framework for the collection, transport, recycling/recovery and disposal of waste (DEFRA, 2014). The Directive requires that Member States take appropriate action to encourage the prevention and reduction of waste and its harmfulness (DEFRA, 2014). It also encourages the recovery of waste by means of recycling, reuse or the use of the waste as a source of energy (DEFRA, 2014; Halliwell, 2006).

The ELV Directive requires that 85% (by weight) of end-of-life vehicles are recycled or reused, a further 10% being subject to energy recovery and only 5% being allowed to go to landfill (Conroy et al., 2005; Pickering, 2006; Stewart, 2010).

The EU Directive on landfill waste aims to prevent or reduce the negative effects on the environment and human health during the whole life cycle of a landfill (Halliwell, 2006). The Landfill Directive defines four categories of waste: municipal waste, hazardous waste, non-hazardous waste and inert waste (Halliwell, 2006). According to Halliwell (2006), composite waste is currently classed as 'Biodegradable wastes and other non-special waste which can give rise to organic or other contamination'. Halliwell (2006) continues to say that due to the 'organic' nature of the resins used in composites, the landfilling of waste composites within the near future will be forbidden in most EU countries.

The LTCS is to ensure that the price of the landfill reflects the impact it is having on the environment. The LTCS provides incentive for the landfilling of waste to be reduced and promote the processes higher in the waste hierarchy (see Section 1.0 Introduction).

The WID aims to prevent or limit negative effects of pollution by emissions into air, soil, surface and groundwater and the resulting risks to human health (Summerscales, 2016). This is achieved by setting emission limits for incineration plants.

The European Composites Industry Association (EuCIA) is proposing a European Composites Recycling Concept (ECRC) which will give composite manufacturers a 'Green label' if they adopt the scheme. The scheme will guarantee that waste composite is recycled appropriately, in accordance with legislative requirements at the time (Pickering, 2006).

3.0 Waste GFRP Recycling

Recycling is the act of (i) converting waste composite into a reusable material or (ii) returning the material to a previous stage in the cyclic process. Pickering (2006) and Garcia et al. (2014) confirm that due to the nature of thermoset composites, it is considered impossible for the initial materials to be recovered using conventional methods.

Recent changes in waste management legislation and the likely future directions for EOL composites mean that there is a need for recycling routes to be established in order for composite materials to have a place in the market (Conroy et al., 2005; Pickering, 2006; Summerscales, 2010).

Halliwell (2006) states that in order for recycling procedures to be easy and as cost effective as possible, waste composites need to be recovered in as clean and pure a condition as possible. Halliwell (2006) continues to say that this is not always possible due to the likelihood of composite materials being used in association with other materials. After their useful life, waste composites might be dirty, contaminated and/or require careful cleaning which is both time consuming and labour intensive.

3.1 Recycling Methods

According to Pickering (2006) there are fundamentally two categories of the recycling process for thermoset composites; those that involve mechanical comminution (milling, grinding) techniques in order to reduce the size of the waste composite to produce recyclates and those that use thermal processes to break down waste composites into recoverable materials or energy (Figure 1). This report will cover the thermal processing options.

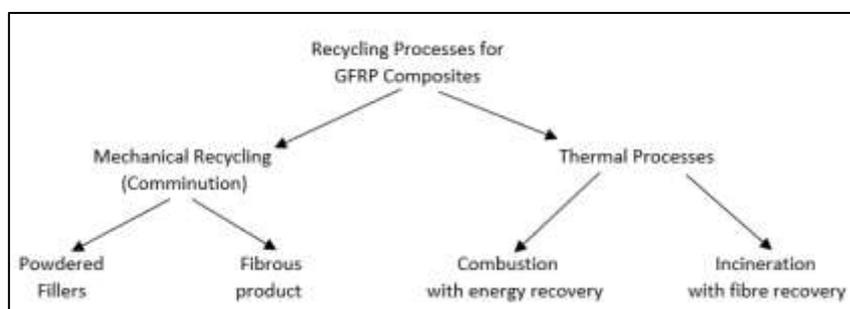


Figure 1 - Recycling Processes for GFRP Composites

3.1.1 Fibre Recovery

The recovery of fibres involves inserting waste composites into a furnace for a finite time. This process destroys the resin and useable, but degraded, clean glass fibres remain. The fibres that remain can then be reused in GFRP's which require lower

mechanical properties. The mechanical properties within the glass fibres are reduced due to their reduction in length and orientation within the process (Job, 2010; Summerscales, 2016). Glass fibres recovered using the fluidised bed process, by Pickering (2006) and Job (2010), reported a reduction in mechanical properties which increased with the temperature. These strength reductions were similar to those of an investigation performed by Jenkins (2015). Summerscales (2015) states that, in order for fibre recovery to be optimised a consistent, known source must be available to provide clean, uncontaminated waste composites (preferably manufacturing waste), e.g. recycling E- and S-glass together would compromise the re-spinning of the melt.

To fully decompose a range of resins using pyrolysis, Pickering (2006) found that the process temperature needed was between 500-550°C. As well as a reduction in mechanical properties, Conroy et al. (2006) report that recovered fibres are sometimes coated with char, if the resin has not been fully decomposed.

Pickering (2006) suggests that the most promising uses for the recovered fibres are the same as those discussed above in the mechanical recycling. However, Conroy et al. (2006) also state that fibres can be used as a form of low grade insulation, although the performance for recovered glass fibres used as insulation has not yet been measured.

Murphy (1998), Garcia (2014), Palmer (2009), Jacobs (2011), Pickering (2006), Halliwell (2006), Otheguy et al. (2009), Job (2010) and Reynolds et al. (2010) have all reviewed the use of the recyclate. Fine recyclate could be used as a replacement for calcium carbonate (CaCO_3) in moulding compounds. Halliwell (2006) says that there is no detrimental effect on the mechanical properties of the moulding compounds if the recyclate is used under specific ratios (20% for Sheet Moulding Compounds (SMC's) and 30% for Bulk Moulding Compounds (BMC's)). IFP CICOMP has developed a core using coarse grades of recyclate (Recycore) which allows a high permeability of resin during impregnation (Pickering, 2011). Coarse grades of recyclate have also been used in the manufacture of plastic lumber and asphalt, where the recyclate is used as an alternative to wood fibre (Pickering, 2011).

3.1.2 Energy Recovery

As in many other systems, there is a considerable amount of energy that goes into producing polymer composites (Summerscales, 2016) so EOL energy recovery is essential. Halliwell (2006) quotes a value of 36 MJ/kg of recoverable energy from waste composites. Since there is no material recovery, this is technically not classified as recycling, although residues left after combustion could be used in the cement industry (Yang et al. 2012).

Thermosetting polymers can be burned as a source of energy; to recover the embodied energy (Conroy et al., 2005; Pickering, 2006). As glass is incombustible the calorific value of the waste composite depends on the proportion of resin to fibre (Figure 17). This is supported by Otheguy et al. (2009). Some fillers, used within composites, may act as a fire retardant during combustion. Pickering (2006) states that in the presence of fire retardants, the energy absorbed during combustion is small compared to the calorific value of the resin.

Conroy et al. (2006) state that, if being incinerated in a domestic refuse incinerator, due to the high calorific content together with the toxic emissions, the waste composite tends to overload the incinerators, meaning low amount of domestic

refuse is dealt with. A balance must be struck between incinerating waste composites and domestic refuse because, as Conroy et al. (2006) say, if the incinerator is overloaded with composite waste there will be an increase in domestic refuse in landfill and vice-versa.

Co-processing through the cement kiln is considered the best recycling option according to the EuCIA because the cement kilns are proving to be very cost effective and are generating valuable materials which helps to improve the carbon footprint of cement production.

Pickering (2006) and Jacobs (2011) state that to recover some of the incombustible materials, burning waste composite in cement kilns is a viable solution. Pickering (2006) says that the glass reinforcement and fillers can be incorporated into the cement. According to Job (2010) “approximately two thirds of the material is transferred into raw materials for cement and one third, the resin, generates energy”. Similarly Jacobs (2011) found that 67% of the material is integrated in the cement and 33% is used for fuel, allowing savings to be made. The EuCIA and ECRC have both put this solution forward as a viable answer which complies with the ELV Directive (Job, 2010). There is capacity to use 100% of the waste GFRP’s produced by Europe within the cement manufacturing. In 2015 there was only just over 1 million tonnes of GFRP produced in Europe (Dr. Witten, 2015) whilst there was 159 million tonnes of cement being produced within the European Union (EU) in 2014 (Cembureau, 2014).

4.0 Experimental Procedures

4.1 GFRP Waste Composite

A typical laminate from a marine composites company (Sunseeker International) was used for the experiments in this study.

The GFRP panel is an infused 8-ply, quadraxial non-crimp fabric (NCF), E-glass panel (reinforcement weight = 1652g/m^2) with a laminate thickness of $\approx 8\text{mm}$. The resin was Scott Bader 701PA isophthalic polyester.

The stacking sequence of the GFRP was analysed under the microscope. This was completed by casting resin (Sicommin SR8500) around a sample. Once cured, the specimen was polished following the polishing procedure and sequence in Appendix A: Stacking Sequence Determination. Once the desired surface finish had been achieved the specimen was examined under the Olympus SC50 Optical microscope to determine the stacking sequence.

Under the microscope fibres aligned at 0° are seen as small circles, fibres at 90° are continuous (if the fibres are perfectly aligned with the polished surface) and fibres at both -45° and 45° are elliptical in shape (Figure 3).

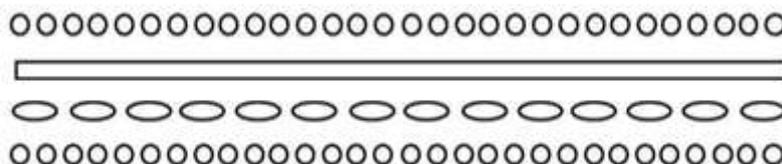


Figure 2 - Fibre Orientation Schematic

The above image shows an example composite with a 0, 90, 45 and a 0. The 45° plies within the schematic can either be positive or negative.

The fibre volume fraction (V_f) was calculated to be 60.4% by rearranging the laminate thickness calculation (see Appendix I, Equation 1), using an E-glass density of 2450 kg/m³.

4.1.2 GFRP Waste Composite Results

Figure 3 shows the stacking sequence of one NCF layer from the $[0^\circ, 90^\circ, \pm 45^\circ, 0^\circ]_8$ of the GFRP waste composite.

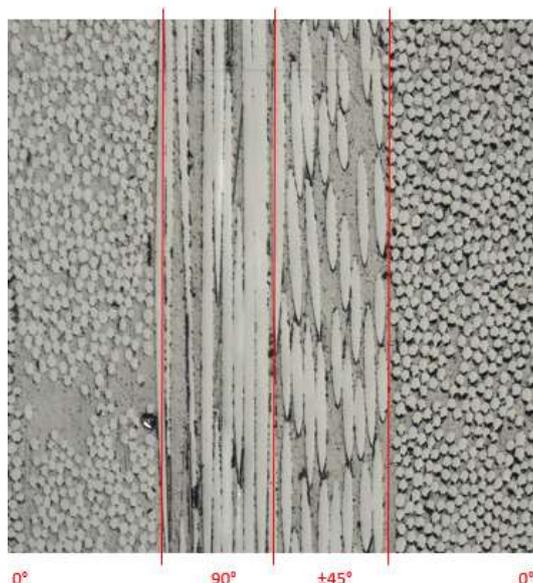


Figure 3 - GFRP waste Stacking Sequence of One NCF Layer (10x Magnification)

4.2 Mains Electric Supply

The supply voltage (V) and the supply energy (Hz) of the mains electric supply was determined using Energenie ENER007 (Table 1). This was recorded in two different locations both within the Brunel W007 laboratory. All energy recordings were determined using the same power meter.

Table 1 - Mains Electric Supply

Property	Unit	Location		Mean
		1	2	
Supply Voltage	V	219.1	218.2	218.65
Supply Energy	Hz	50	50	50

The energy tariff was set to £0.10/ kWh.

4.3 Composite Size Reduction

Thermal recycling methods require the waste GFRP to be reduced into smaller, more manageable sizes for processing to take place (Halliwell, 2006; Summerscales, 2016).

In order to reduce the GFRP waste to a suitable size for processing, both the wet saw (Tyslide diamond wheel slitting saw) and a jigsaw (DeWalt DW321-GB) were used. The cost and energy consumption of both methods were recorded and compared (Table 2).

A further test was conducted to determine the cost of the water which is required throughout the use of the wet saw. (1.2) metres of GFRP waste was cut and the water collected from the overflow container, the amount of water was then measured and the cost of that water was calculated (Table 3).

4.3.1 Composite Size Reduction Results

Table 2 - Wet Saw and Jigsaw Energy Consumption/ Cost Results

Property	Unit	Wet Saw	Jigsaw
Cut length	m	3.0	1.5
Cut duration	s	602	248
Minimum energy usage	W	32.2	3.9
Maximum energy usage	W	1039	425.4
Energy consumption	kWh	0.035	0.02
Tariff	£/kWh	0.10	0.10
Cost	£/km	1.17	1.33

Table 3 - Wet Saw Water Cost

Property	Unit	Value
Cut length	m	1.20
Cut duration	min	4.25
Water quantity	L	6.088
Water Cost	£/L	0.00195
Cost	£/km	9.89

The water cost was obtained from South West Water. Water costs for 2016/2017 are £1.9484/m³ (i.e. £0.00195/L) (South West Water, 2016).

4.4 Fibre recovery 1

In order to determine the temperatures at which resin elimination would occur it was necessary to conduct experiments at different temperatures. The first experiment (EXP 1) (Appendix C). The specimen was allowed to burn for 15 minute intervals before being removed, allowed to cool and weighed using the scales (Avery-Berkel WA205 analytical balance). After an hour at 300 °C the weight of the specimen had only been reduced by 0.07g. Due to the small reduction in weight, 300 °C was determined to be too low a temperature to eliminate the resin.

A second experiment (EXP 2) was conducted at 400 °C using the same specimen used in EXP 1 due to the low reduction in weight. The specimen was, again, allowed to burn for 15 minute intervals. After 75 minutes of burn-off time there was a weight reduction of 2.082g. Due to the significant weight loss 400 °C was considered a valid temperature to eliminate resin.

The third experiment (EXP 3) (Appendix C) was conducted on a 9.18g sample at 500 °C. The specimen was, again, allowed to cool and weighed every 15 minutes. The experiment revealed that after 75 minutes of burn-off time the specimen had reached a constant mass suggesting complete resin elimination had been achieved.

From the above experiments, it was decided that the temperatures at which the testing would be undertaken were:

- 400 °C (673 K) (Specimen Group C)
- 425 °C (698 K) (Specimen Group D)
- 450 °C (723 K) (Specimen Group E)
- 475 °C (748 K) (Specimen Group F)
- 500 °C (773 K) (Specimen Group G)

(see Appendix E: Test Specimen Details)

The process of which would be exactly the same as mentioned above (see Section 4.4). Each specimen group contained 5 specimens (1C, 2C, 3C, etc.).

4.4.1 Fibre Recovery 1 Results

Table 4 - Fibre Recovery Test Results

Property	Unit	Specimen Group				
		C	D	E	F	G
Temperature	°C	400	425	450	475	500
Furnace run time	h	12.8	7.17	4.03	2.70	2.05
Burn-off time	h	10.8	5.5	2.75	1.5	1.0
Minimum energy usage	W	13.7	10.6	9.90	8.20	6.90
Maximum energy usage	W	2054	2012	2006	2063	2023
Energy consumption	kWh	6.64	1.81	2.62	1.97	1.54
Tariff	£/kWh	0.10	0.10	0.10	0.10	0.10
Cost	£	0.664	0.181	0.262	0.197	0.154
Corrected Cost	£	0.558	0.139	0.162	0.109	0.075

Table 4 shows the temperatures at which the resin burn-off was performed along with the time of burn-off and the final cost, which is related to the energy consumption in kWh. The results showed that there was a direct correlation between burn-off time and cost, where the burn-off time is determined by the process temperature (Figure 4, Figure 5 and Figure 6). The corrected costs were calculated using the burn-off time of each specimen group.

Figures 5 and 6 show that Specimen group D does not follow the trend set by the other Specimen groups due to it consuming a lot less energy than predictable but the burn-off time was as predicted. The energy consumption was smaller due to the furnace initial temperature being above ambient before the test was started which meant that the furnace did not use as much energy when reheating to the test temperature.

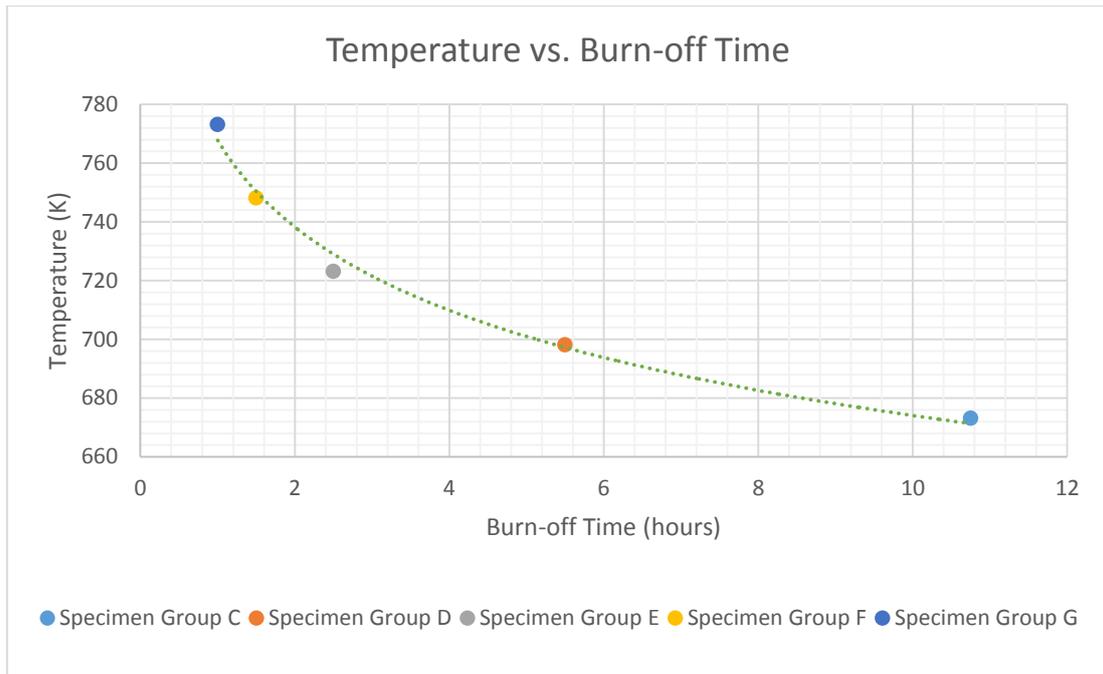


Figure 4 -Temperature vs. Burn-off Time (Specimen Groups C - G)

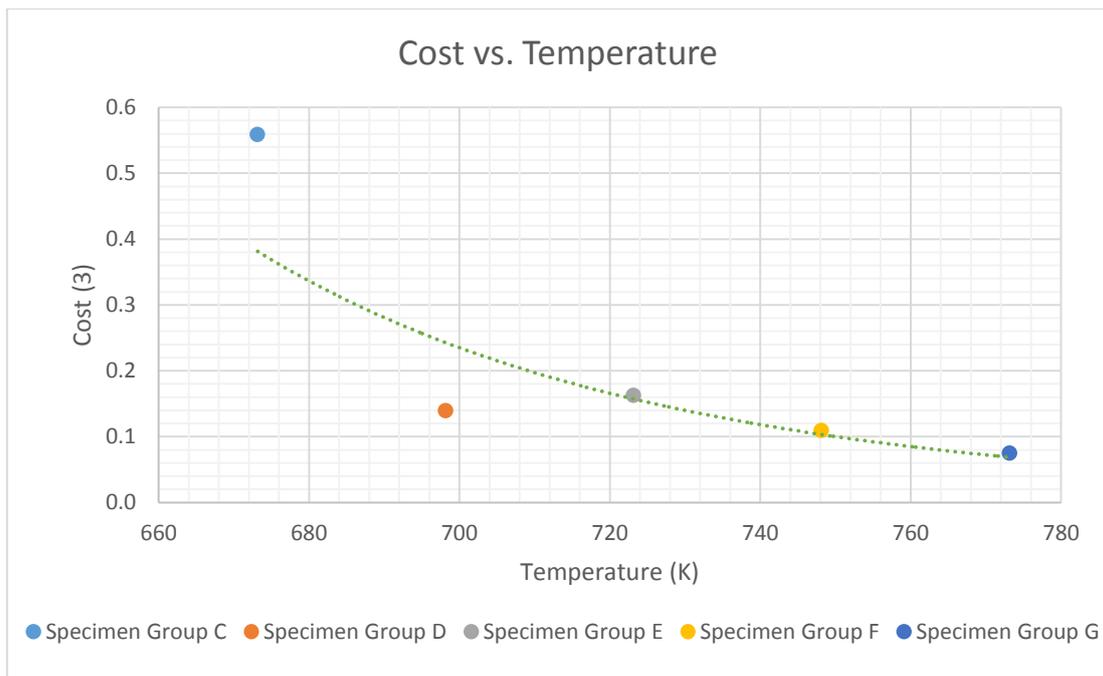


Figure 5 - Cost vs. Temperature (Specimen Groups C - G)

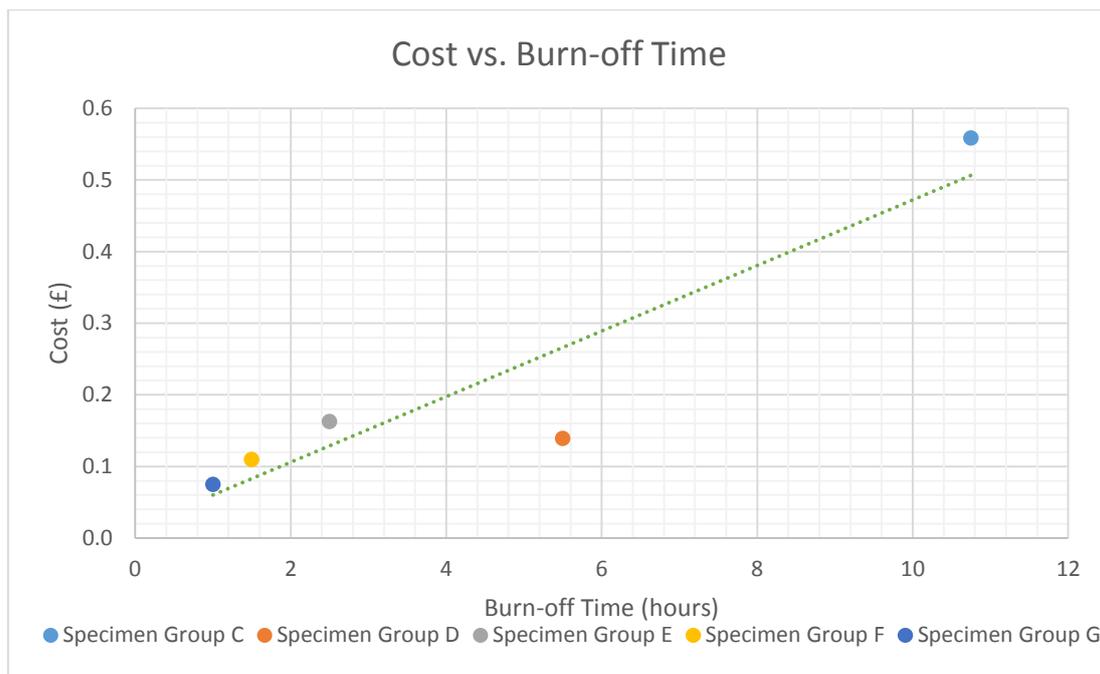


Figure 6 – Cost vs. Burn-off Time (Specimen Groups C - G)

4.5 Fibre Recovery 2

Due to the nature of the tests conducted using Specimen groups C – G, the cost and energy consumption were skewed due to the opening and closing of the furnace during the resin burn-off process. Further, it was noted that every time the furnace door was opened the temperature dropped drastically. This meant that once the door was closed the furnace consumed additional energy in order to reach the desired temperature.

To obtain a more accurate cost and energy consumption value, specimens from group H (Appendix E) were tested using the same temperatures but allowed to burn for 75% of the predetermined burn-off time. The reduced time accounts for there being no energy loss every time the specimens were removed from the furnace and allowed to cool. The specimens were burnt off at 400°C, 425°C, 450°C, 475°C, and 500°C with a further sample at 600°C for 10 minutes.

4.5.1 Fibre Recovery 2 Results

Table 5 shows the results from heating at 75% of the predetermined burn-off time, which gives more accurate estimates of the cost and energy consumption of each process temperature. Specimen 6H was removed before complete resin elimination had occurred.

Table 5 - Specimen Group H Fibre Recovery Test Results

Property	Unit	Specimen No.					
		5H	4H	3H	2H	1H	6H
Temperature	°C	400	425	450	475	500	600
Furnace run time	h	8.92	6.2	3.57	1.66	1.17	0.57
Burn-off time	h	8.5	5.75	3.00	1.25	0.75	0.17
Minimum energy usage	W	22.5	22.6	22.9	6.7	22.0	8.5
Maximum energy usage	W	2081	2090	2033	2023	2046	2103
Energy consumption	kWh	4.086	3.052	1.997	1.277	1.111	0.973
Tariff	£/kWh	0.1	0.10	0.10	0.10	0.10	0.10
Cost	£	0.408	0.305	0.200	0.128	0.111	0.10

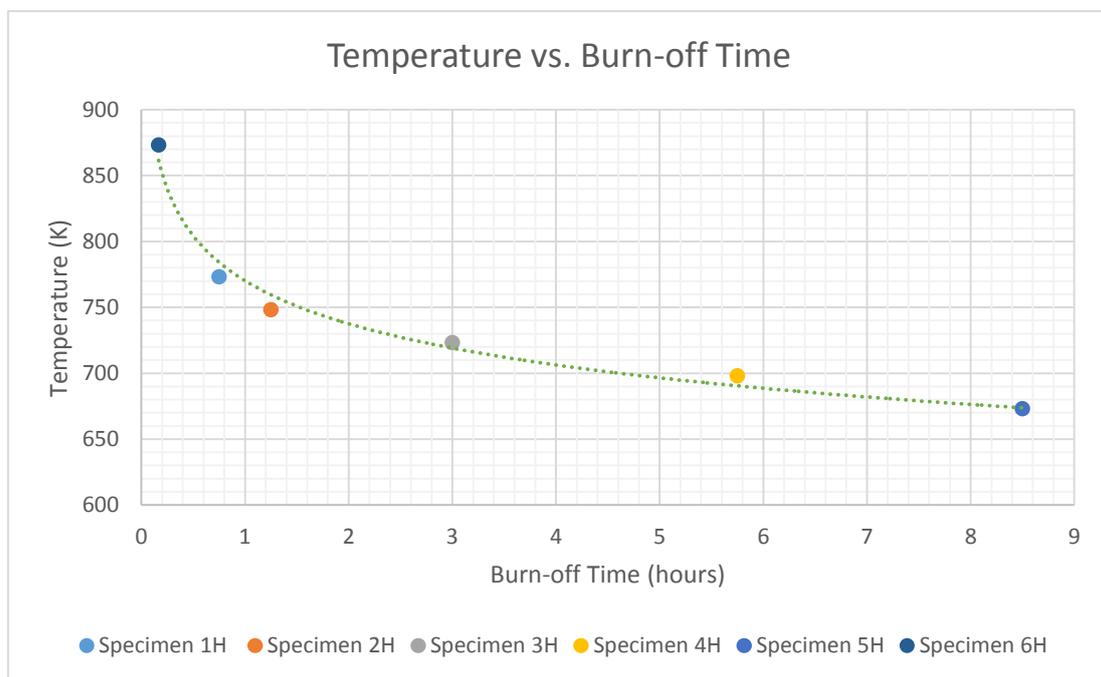


Figure 7 - Temperature vs. Burn-off Time (Specimen Group H)

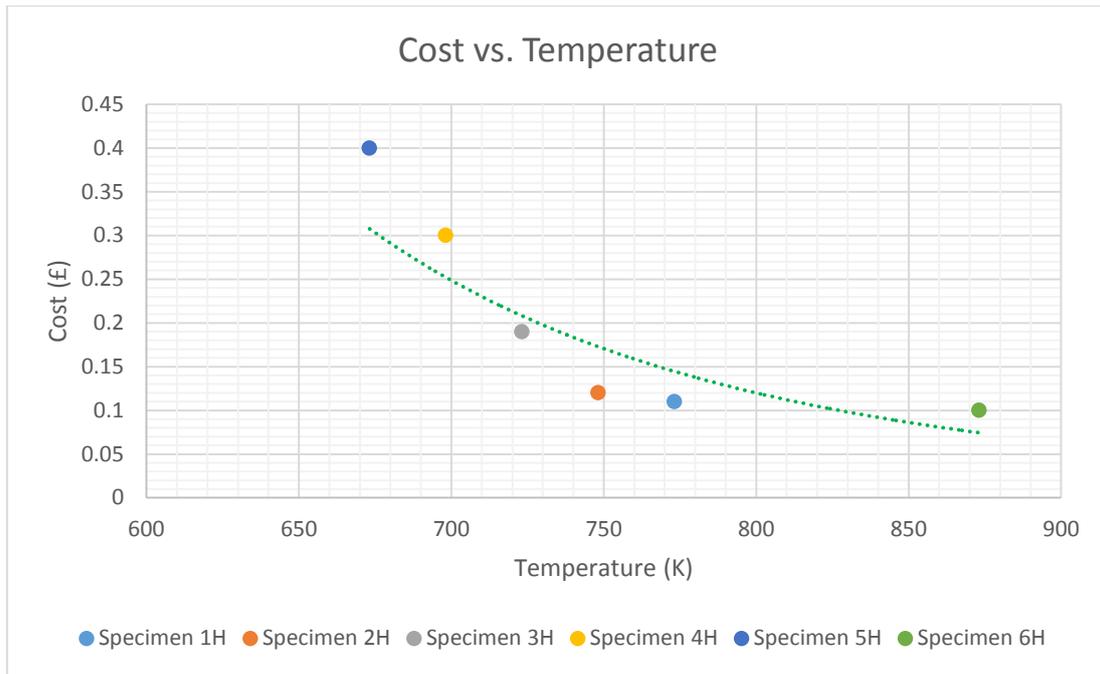


Figure 8 – Cost vs. Temperature (Specimen Group H)

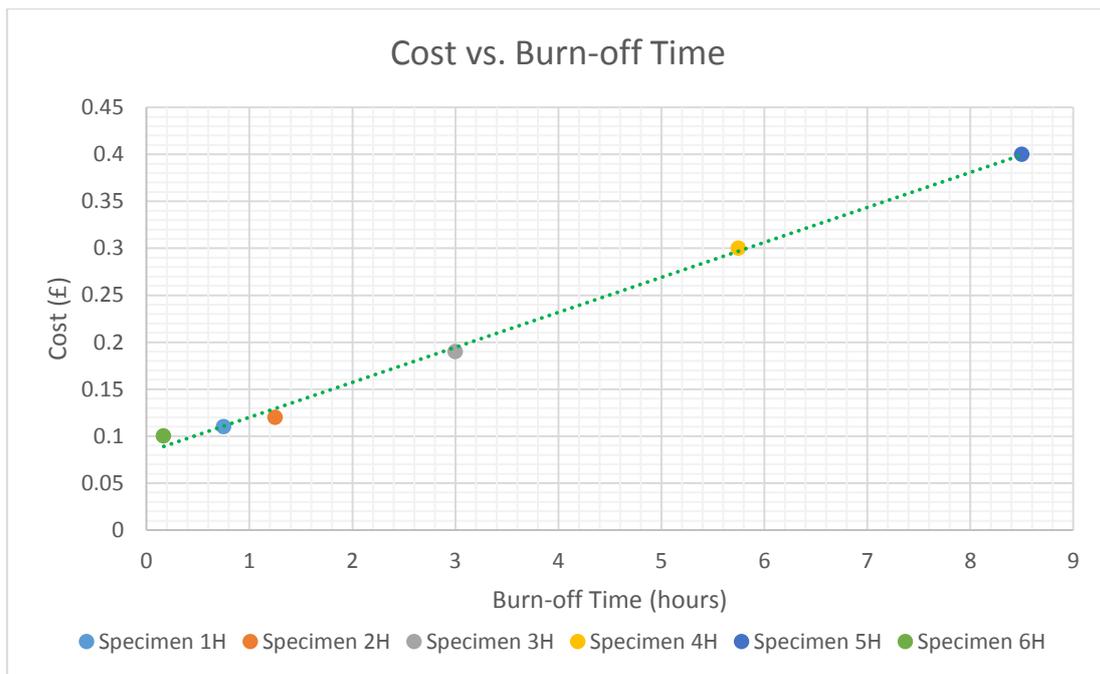


Figure 9 - Cost vs. Burn-off Time (Specimen Group H)

Figure 6 shows the burn-off time is related to the temperature at which the test was conducted. Figure 8 shows cost vs. temperature; it can be seen that as the temperature increases the cost of the fibre recovery decreases. There is a direct correlation between burn-off times and their costs (Figure 9).

4.6 Scanning Electron Microscopy

To determine the level of resin burn-off, fibres from each test were placed on an aluminium disc ($\approx 80\text{mm}$) and stuck in place using double sided tape and Agar silver paint (Batch: OW51451017) to secure the ends. The specimens were then placed in the gold sputter coating machine (Emitech K550). The thin gold coating applied to the surface of the specimens allows the electrons to be conducted away to produce the image and prevent charging artefacts. The specimens were then placed into the Scanning Electron Microscope (SEM) (Jeol JSM_6610LV) (calibrated/serviced annually). Scanning was completed at 5kV and the specimens were set at a height of 7mm.

4.6.1 Scanning Electron Microscope Results

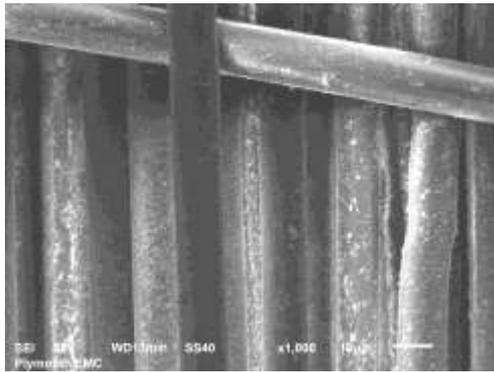


Figure 10 - Specimen 5H (400° C)

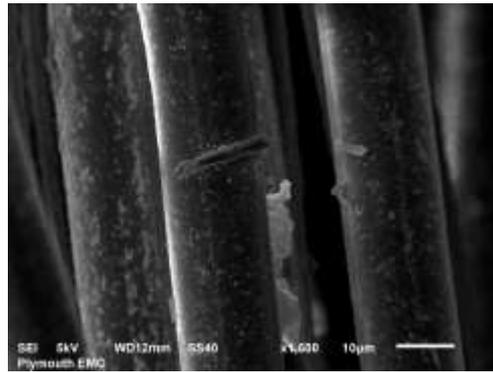


Figure 11 - Specimen 4H (425° C)

As can be seen from Figure 10, there is a large quantity of resin which remains on the surface of the recovered fibres.

Figure 11 shows large fragments of resin along with finer resin on the recovered fibres surface.

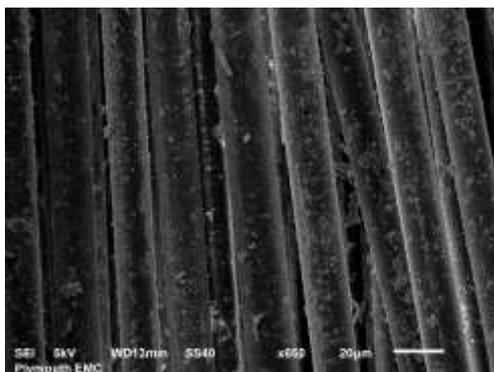


Figure 12 - Specimen 3H (450° C)

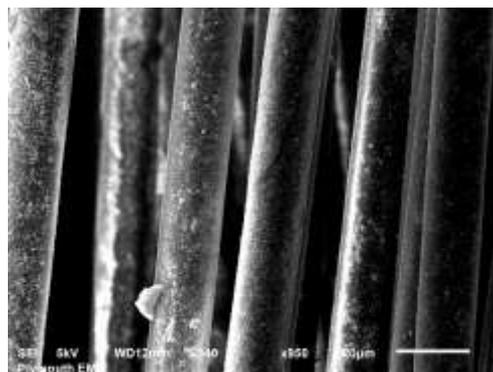


Figure 13 - Specimen 2H (475° C)

Large quantities of resin are visible on the surface of the recovered fibres (Figure 12).

Resin is still visible on the recovered fibres but is less dense than the residual resin on Specimens 2H – 5H (Figure 13).

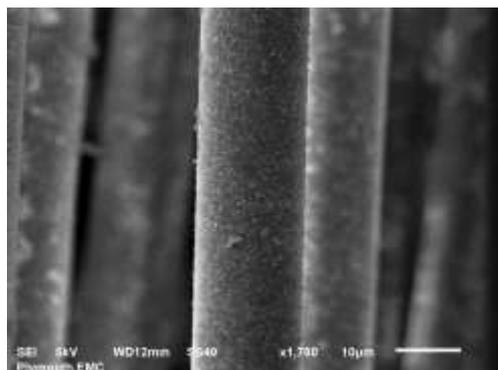


Figure 14 - Specimen 1H (500° C)

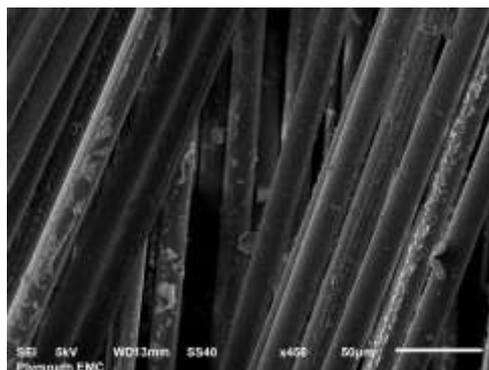


Figure 15 - Specimen 6H (600° C)

The resin which can be seen in Figure 14 is sparse. The figure shows that to view the resin clearly a magnification of x1700 on the SEM display was required.

The recovered fibres from Specimen 6H show a large quantity of resin remaining on the surface of the fibres (Figure 15).

4.7 Energy Recovery Determination in Bomb Calorimeter

In order to calculate the potential recoverable energy within the GFRP waste it was first necessary to grind the composite into a fine powder. This was achieved using a belt sander (Scheppach BTS 900X). A catchment tray was setup to catch the powder before it became contaminated by contact with other surfaces. The cost and energy consumption were recorded (see Section 4.7.1).

A bomb calorimeter (Parr 1356) was used to calculate the recoverable energy. The initial process was to weigh the sample, then place it into the bomb head with the fuse wire in contact with the sample. The jacket was filled with 2 litres (2kg) of water and the bomb filled with ≈1g of water. The bomb head and bomb were screwed together ensuring that the sample and fuse wire were not disturbed. The bomb was then filled with 30 bar of oxygen (99.5% purity) and placed into the jacket. The calorimeter was then closed and the bomb fired (Appendix G). The bomb was then removed from the calorimeter, the oxygen was allowed to escape from the bomb and the crucible was removed. The crucible was weighed again to get the weight of the residue (Table 5).

4.7.1 Energy Recovery Results

Table 6 - Bomb Calorimetry Results

Test	ID no.	Sample Weight	Water Jacket Weight	Gross Heat Output	Residue Weight
Unit	/	g	g	MJ/kg	g
1	44	0.7490	2000.08	7.254	0.411
2	45	0.7506	1999.96	6.917	0.398
3	46	1.007	2000.13	7.049	0.534
Mean	-	-	2000.06	7.07	-
St. Dev	-	-	0.0713	0.139	-

Table 15 shows the recoverable energy values for the GFRP. The first two tests were run with a specimen weight of ≈0.75g and the third test was run with a specimen weight of ≈1g. This was performed in order to validate gross heat output and with a

maximum percentage difference of $\approx 5\%$, between tests 1 and 2, the results were considered valid. The recovered energy from the waste GFRP had an average value of 7 MJ/kg.

5.0 Discussion

5.1 Assumptions Violated

Discrepancies in results may have been obtained through the use of the power meter. The power meter used for the energy consumption reading is of a fairly low grade and has a maximum tolerance of 2% on its energy display (see Appendix K: Energenie ENER007).

Another assumption violated during testing is that the crucibles in which the specimens were placed did not consume any of the energy produced by the furnace. Variability in the weighing of the specimens could have been a result of the Avery-Berkel WA205 Analytical Balance scales last been calibrated on the 10/04/2006.

To obtain better results, re-calibration of the scales would be essential and a full investigation into the readings produced by the power meter would need to be performed.

5.2 Discussion for Experiments Conducted

Table 7 - Relative Costs of Cutting Processes (easycomposites, 2015)* (DK Holdings Ltd, 2012)#

Both size reduction processes resulted in very low costs (**Error! Reference source not found.**).

	<i>Jigsaw</i>	<i>Wet-saw</i>
<i>Blade Material</i>	Tungsten Carbide (*)	Diamond Grit (ESC 165) (#)
<i>Cost (£)</i>	4.50	47.20
<i>Power (Wh)</i>	2.00	35.0
<i>Power Cost (£/km)</i>	1.33	1.17
<i>Water Used (£/km)</i>	0.00	9.89
<i>Total Cost (£/km)</i>	1.33	11.10

The use of water for the wet saw is required to prolong the life of the blade, reduce dust becoming airborne and provide a smoother cut. Jigsaw dust is extracted through the use of a vacuum line. Assuming the jigsaw blade life is 1/8 that of the wet saw blade due to the cutting surface length being ≈ 8 times smaller (Appendix I). The cost of the wet saw is $\approx 44\%$ cheaper than that of the jigsaw. As most large waste composites need to be reduced before they are processed, and often on site, the use of the jigsaw would be essential due to its portability and cost effectiveness. However diamond blades are normally recognised as having a longer cutting life.

The process of recovering the material required for the energy recovery process (see Section 4.7) resulted in $\approx 11\text{g}$ of recyclate being recovered. The test results indicate it would cost £182.00 to produce one tonne of recyclate. Filler grade CaCO_3 costs just £123.20/ tonne (Alibaba, 2016). Otheguy (2009) states that even if the process of using recyclate as a replacement for CaCO_3 became economically viable, it would represent a very low cost saving to GFRP manufacturers. With the cost of CaCO_3

being 38.5% cheaper than the cost of producing the recyclate, without considering the cost of labour to produce the recyclate, it proves that the use of CaCO_3 would be more attractive to GFRP manufacturers. However, if the cost of sending the waste composite to landfill was more than that of sending it to the incinerator both the waste composite originator and the incinerator operator would benefit.

With an average percentage difference between Fibre Recovery 1 and 2 of 40.1% (Appendix I), clearly the costs incurred in Fibre Recovery 1 were higher due to the time out of furnace for weighing of the specimens and the opening/ closing of the furnace.

The data collected during the Fibre Recovery 2 tests indicated that the best temperature, in terms of cost and energy consumption, at which resin burn-off should be performed is 500°C (773.15 K), This is backed up by Pickering (2006) where he states that to decompose the resin, process temperatures between $500\text{-}550^\circ\text{C}$ were required. Table 14 (see Section 4.5.1) shows a cost of the burn-off for Specimen 6H (600°C) is lower than that of Specimen 1H (500°C). But the test was aborted after 10 minutes of burn-off time (due to the cost approaching that of Specimen 1H) with a large quantity of resin remaining within the fibres. This is supported by Conroy et al. (2006) when they state that fibres may be coated with char if resin has not been fully decomposed. Figure 16 (A) shows char coated recovered fibres.



Figure 16 - Specimen 6H (Recovered Fibres)

The SEM results show that the recovered fibres from Specimen 1H are the cleanest fibres recovered (see Section 4.6.1). It can be seen that the resin has not been completely eliminated from the recovered fibre. In order to obtain 100% clean fibres, they would have to undergo a cleaning process to remove any loose resin which may remain on the recovered fibres.

The energy recovered from the samples was 7MJ/kg (see Section 4.7.1 – Energy Recovery Results). Halliwell (2006) quotes a calorific value of 36MJ/kg . However Pickering (2006) reports that as the V_f increases the calorific value of the Composite reduces (Figure 17). This is due to the glass being incombustible. Young et al. (2009) reports that polyester resins have $63\text{-}78\text{ MJ/kg}$ embodied energy. With 40% matrix, the expected recoverable energy for the waste GFRP is $\approx 28\text{MJ/kg}$.

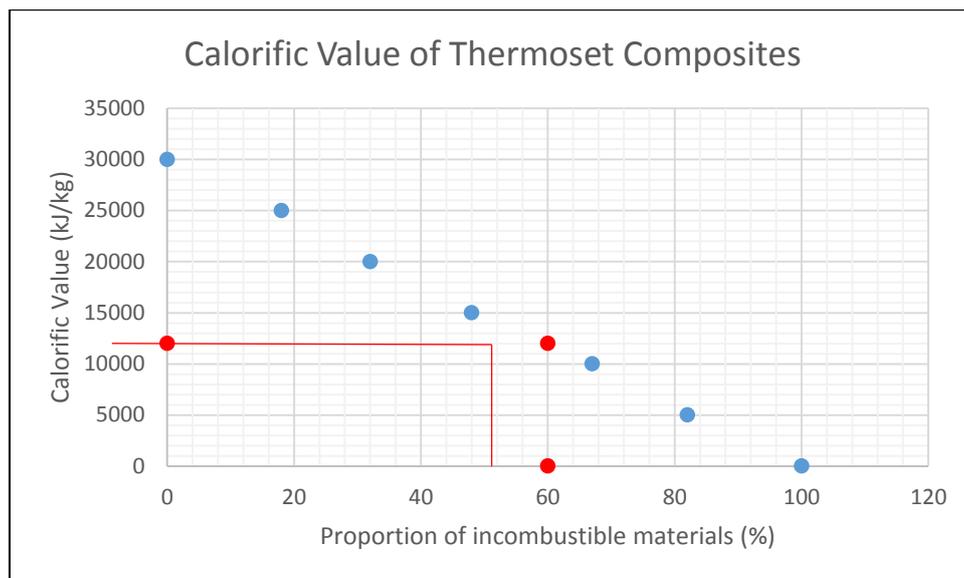


Figure 17 - Calorific Value of Thermoset Composites

Figure 17 shows that a GFRP with a V_f of 60.4%, the recoverable energy should be $\approx 12\text{MJ/kg}$. This is a 55% difference from the measured calorific value.

The differences between the values may be a result of a different matrix being used for the two tests, a high inert filler volume fraction (V_{filler}) which may displace polymer mass during the bomb calorimetry or abrasive from the grinding process transferred to the sample. During bomb calorimetry the fine powder used as the fuel, may have been displaced upon ignition giving smaller readings that were predicted by Halliwell (2006), Pickering (2006), and Young et al. (2009). The bomb calorimeter was also last serviced/ calibrated in August 2014 which may have skewed results.

5.3 Discussion of Scaling Implications

Scaling the process of energy recovery to an industrial level requires a few key aspects to be considered in order for the process to be feasible.

Large waste composites (i.e. wind turbine blade) will need to be reduced in size in order for transportation to take place. This initial size reduction could be performed through the use of a jigsaw. This size reduction process is restricted to the cutting length of the blade, meaning composites that are thicker than 50mm will need to be reduced using a different process. The waste GFRP will then be transported to facilities where further size reduction may be required. Since the wastes aesthetics are no longer important, this second stage of reduction can be performed using a crusher.

Scaling the process of fibre recovery to an industrial level would require the same initial stage as above (initial size reduction and transport). However, the size of the waste composite as it enters the furnace is crucial. The length and orientation of the fibres in the component will affect the mechanical properties of the recovered fibres as discussed by Job (2010) and Summerscales (2016). The temperature at which resin decomposition occurs will also affect the mechanical properties of the fibres.

This temperature will be determined by a number of variables (i.e. GFRP's specific heat, volume, resin-matrix composition, etc.) (Appendix I). For resin decomposition composites with a low V_f are preferable due to the matrix having a higher specific

heat capacity. But, due to composite design, composites with a low V_f are infrequent in structural composites as a higher V_f will give better mechanical properties. Once the ideal temperature for the waste GFRP is determined, resin elimination can take place.

Resin elimination can be conducted through the use of a furnace, through which waste is carried on a conveyor belt. The furnace would need to be designed so it allows access for the waste to and from the furnace without excessive loss of heat.

Further development into both fibre recovery and energy recovery is required. Cost implications such as transport, separation of waste GFRP from cores, Sorting of waste and cleaning of recovered fibres are all restrictive on the development of the processes. Hence, currently most GFRP waste is landfilled. However, as landfill costs climb and is made more difficult, alternative options for EOL GFRP's will become more appealing.

6.0 Conclusions

- The most practical process to reduce the size of GFRP waste composites is through the use of a Jigsaw. This is due to its portability (Battery powered options available), low cost consumables and it being 44% cheaper than the use of a wet saw. If waste composite is being transported after initial size reduction, the use of a generator on the truck to perform size reduction is also possible.
- The optimal temperature at which to recover glass fibres, from a polyester/ glass laminate is around 500°C. Although subsequent cleaning of the fibres is necessary to completely eliminate the matrix.
- The use of waste GFRP as a fuel in cement kilns is a viable solution. Where the matrix is used as a fuel and the recovered fibres are integrated into the cement itself.
- The most feasible recycling option for EOL GFRP's would be incineration with energy recovery. This is due to 100% of the waste composite being used.
- Further development into both energy and fibre recovery is required for them to become economically viable or present enough of a saving for companies to undertake.

Recommendations for Further Work

To gain further knowledge of the costs of the jigsaw and wet saw usage, research into the blades life needs to be conducted in order to calculate a more accurate cost per metre cut.

To determine the feasibility of the recovered fibres being reused in the manufacture of further composites, their mechanical properties should be determined following Grafil Test Methods for filament tensile strength and modulus (Grafil Test Methods, 1980).

The recycleate should be used as a filler within GFRP panels and tested for flexural strength, Inter Laminar Shear Strength (ILSS) and tensile strength; following BS EN ISO14125:1998, BS EN ISO 14130:1998 (British Standards Institution, 1998) and BS EN ISO 527:1997 (British Standards Institution, 1997) respectively.

An investigation into the use of a diamond bladed circular saw for waste composite size reduction should be performed to ascertain a feasible method in which composites thicker than 50mm could be reduced in size.

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