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Characterisation and Use of Glass Fibre Reinforced Plastic Waste Powder as Filler in Styrene-Butadiene Rubber

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Abstract

Glass fibre reinforced plastic (GRP) wastes are often disposed of in landfill, incinerated or processed into powders. GRP waste powders can be recycled as filler in virgin polymers and should be characterised before they are added to avoid processing problems. A GRP waste powder was characterised using advanced measuring and analytical techniques. These included, scanning electron microscopy, Fourier transform infrared spectrometry, particle size analyser, differential scanning calorimetry, X-ray photo-electron spectroscopy and energy dispersive X-ray microanalyser. The results showed that the waste powder consisted of irregular shaped particles and glass fibre fragments up to 700 μm in size. Moreover, the waste powder was a thermoset polyester resin and its chemical constituents were calcium, oxygen, aluminium, silica, chlorine, bromine and carbon. When up to 25 parts per hundred rubber by weight of the GRP waste powder was mixed with a sulphur cure-based styrene-butadiene rubber, the viscosity, scorch and optimum cure times increased, and the rate of cure decreased. The tearing energy, elongation at break, tensile strength, stored energy density at break, and Young's modulus of the vulcanisate improved as the loading of the waste powder was raised.

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INTRODUCTION

Thermoset polymers reinforced with glass and carbon fibres are used to manufacture a wide range of products for the automotive, aerospace, electrical, wind energy, civil engineering, prototyping and marine industries.¹ These materials are lightweight and possess excellent mechanical properties for example tensile strength, Young's modulus and strain at break.^{2,3} The substantial improvement in these properties is due to the inclusion of glass and carbon fibres in the polymer matrix.^{4,5} Fibres are often treated with adhesive or silane coupling agent to maximise the fibre/polymer interfacial adhesion strength which enhances mechanical properties.^{5,6} At the end of their usable service life, these materials are discarded as waste, causing significant environmental pollution. For example, in Europe each year 9 million tonnes of waste is generated by End-of-life vehicles, which are landfilled, incinerated, recycled or reused.⁷ However, landfill tax in UK will be increasing by more than 30% per tonne per year until 2010-11.⁸ Although landfill is a relatively cheap method of waste disposal and the amount of waste going into landfill has been falling in recent years, the leaching of contaminants into local water supplies is causing major concern.⁷ Therefore, more efficient methods for waste management are now needed.

One alternative method is to recycle solid wastes for use in virgin polymers as filler.

Recycling of thermoset composite material wastes involves mechanically breaking down the solid to reduce the size of the scrap to produce recyclates; and use thermal processes to break the scrap down into materials and energy.⁸ In a mechanical recycling process, discarded solid waste is reduced in size into recyclates, which contain a mixture of fine polymer and filler powder and coarse fragments of fibre. The size reduction stage produces a product ranging from typically 10 mm in size down to particles less than 50 nm in size. Finally, recyclates are graded into fractions of different sizes by sieves.⁹

Solid polymer composite waste powders have been recycled as filler in virgin polymers in the past. Perrin and co-workers¹⁰ studied effects of recycled sheet moulding compounds consisting of unsaturated polyester filled with glass fibres and calcium carbonate on the mechanical properties of polypropylene thermoplastic and reported improvement in tensile strength and yield strength.

Before waste powders are added to virgin polymers, it is essential to characterise them to avoid processing problems. Improvement in mechanical properties is mainly due to strong adhesion between the filler and polymer matrix.¹¹ However, without

proper measurement of the filler surface chemical composition, it will not be possible to optimise the filler/polymer interaction to achieve good mechanical properties.⁶

Furthermore, filler particle size and particle size distribution also affect the mechanical properties of polymers. For example, fine fillers such as carbon blacks and silicas with particle sizes ranging from 20 to 500 nm are very effective in improving the rubber properties. The finer the particle size, the more reinforcing the filler is.¹²⁻¹⁴

The aim of this study was to characterise a GRP waste powder of unknown chemical composition and physical properties using advanced measuring and analytical techniques. The properties measured included particle size, particle size distribution, particle shape, glass transition temperature and surface chemical composition. After the waste powder was characterised, it was mixed with a sulphur cure-based styrene-butadiene rubber to measure its effect on the viscosity, cure properties, hardness, tensile strength, elongation at break, tearing energy, Young's modulus, and stored energy density at break of the rubber. The results were subsequently evaluated to determine a potentially new recycling route for the waste powder in rubber.

EXPERIMENTAL

Materials

The material used was a glass fibre reinforced plastic solid powder, referred to as waste powder. The process for producing the waste powder consisted of primary size reduction of the post consumer and manufacturing waste and feeding it into a grinding machine where the original waste was reduced to a fine resin/glass powder and strands of glass fibre up to 75 mm in length. These components were separated by several mechanical methods and then bagged. The waste powder was generated from post production material at Hambleside Danelaw Rooflights and Cladding Limited, UK, where the grinding machine for processing the waste was originally developed .

The chemical composition and physical characteristics of the waste powder were unknown. However, the waste powder contained large chunks of glass fibre when it was originally supplied. These were separated from the powder by a 200 µm size sieve before characterisation began.

The raw elastomer used was a styrene-butadiene rubber (SBR) (23.5 wt % styrene; Intol 1712, Polimeri Europa UK Ltd., Hythe, UK). SBR Intol 1712 is a cold emulsion co-polymer, polymerized using a mixture of fatty acid and rosin acid soaps

as emulsifiers. It is extended with 37.5 phr of highly aromatic oil and contains a styrenated phenol as a non-staining anti-oxidant. It has approximately 4.8% by weight organic acid and a viscosity of 32 Mooney units. In addition to the raw rubber and waste powder, the other ingredients were *N*-cyclohexy-2-benzothiazole sulphemamide (CBS, an accelerator), zinc oxide (ZnO; an activator), stearic acid (an activator), elemental sulphur (curing agent), and *N*-(1,3-dimethylbutyl)-*N'*-phenyl-*p*-phenylenediamine (Santoflex 13, an antidegradant).

Mixing

Compounds 1-3 (Table 1) were prepared in a HAAKE RHEOCORD 90 (Berlin, Germany), a small-size laboratory mixer with counter rotating Banbury rotors. In these experiments, the rotors and the mixing chamber were maintained at 25°C and the rotor speed was 45 r.p.m. The volume of the mixing Chamber was 78 cm³, and it was 60% full. HAAKE Software version 1.9.1. was used for controlling the mixing conditions and storing data. To prepare the compounds, the raw rubber and the waste powder were placed into the mixing chamber and mixed for 6 minutes. The rotors were then stopped to allow rubber to cool down to about 40-45°C to avoid scorch before the chemical curatives were added. The rotors were started and CBS, zinc

oxide, stearic acid, sulphur and antidegradant were added and mixed for an extra 6 min. The total mixing time for the compounds was 12 min.

Finally, when the mixing ended the rubber was recovered from the mixer and milled to a thickness of about 6 mm for further work. The compounds were kept at ambient temperature ($\sim 22^{\circ}\text{C}$) for at least 24 h before their viscosity and cure properties were measured.

Characterisation of the Waste Powder

The particle size, particle size distribution and particle shape of the waste powder were measured by a LEO 1530 VP Field emission gun scanning electron microscope (SEM) (Carl Zeiss SMT, Ltd., Cambridge, UK) and a low angle laser light scattering Malvern Mastersizer 2000 Ver. 5. particle size analyser with a range of 0.02 to 2000 μm (Malvern Instruments Ltd, UK), respectively. 0.5 g of the powder was placed on the sticky side of a sellotape 25 mm wide and coated with gold, examined and then photographed in the SEM to determine the size, size distribution and shape of the particles. To test the powder in the Mastersizer, a sample approximately 20 mg in weight was mixed with distilled water in the bath of the machine and stirred to produce a good mixture. The machine was then switched on and the mixture was

exposed to laser scattering. The data was collected and processed by Malvern Mastersizer 2000 Software to produce a range of particle sizes and a particle size distribution profile by plotting the volume fraction of the particles against the corresponding particle sizes for the sample.

The surface composition of the waste powder down to a depth of 4-5 μm in the sample was determined by an energy dispersive X-ray (EDX) microanalyser (EDAX Phoenix, Carl Zeiss SMT, Ltd, Cambridge, UK). These measurements were carried out at the same time the waste powder was tested in the SEM for particle size and particle shape determination.

To determine the composition of the waste powder, a Fourier transform infrared spectrometer (FTIR-8400S) (Shimadzu Scientific Instruments, USA) was used. A sample of powder approximately 1g in weight was mixed and grinded with KBr powder. The mixture was poured into a chamber and pressed manually to form a semi transparent disc 1 mm thick and 10 mm in diameter. The disc was then placed in a sample holder and tested. The test produced a spectrum which was printed out and compared with the standard spectra of commonly known polymers. The composition of the waste powder was subsequently identified.

In addition to FTIR, the surfaces of the waste powder were also chemically analysed by an X-ray photoelectron spectroscope (XPS) (VG Scientific, UK). This technique can detect the surface composition of materials down to a depth of 5-10 nm from the top surface of a sample. To perform these tests, an aluminium boat approximately 15 mm in diameter was used to hold a 2 mm thick sample of powder. The sample was irradiated with X-ray, normally aluminium or magnesium characteristic X-ray lines. The X-rays excited photoemission from the core levels of the atoms present in the sample and the resultant photoelectrons emerging from the sample were collected and energy analysed to yield the photoelectron spectrum. Since the kinetic energy of the photoelectron depended on the binding energy of electrons in the core levels from which photoemission was excited, each element gave rise to a set of peaks at characteristic energies in the photoelectron spectrum. Measurement of these energies therefore allowed the chemical elements present in the sample surface to be determined. Quantitative analysis was subsequently obtained from the measurement of the relative intensities of the photoelectron peaks.

The glass transition temperature (T_g) of the waste powder was measured by a differential scanning calorimeter (DSC 2010) (TA Instruments, USA). A sample of

the powder, 10.4 mg in weight was placed in an aluminium pan with lid. The pan was then placed in the furnace of the DSC machine and the temperature was increased from 20 to 200°C at 10°Cmin⁻¹. A Heat flow versus temperature trace was produced from which the T_g of the powder was determined.

Viscosity and Cure Properties of the Rubber Compounds

The viscosity of the rubber compounds was measured according to the British Standard¹⁵ and the results were expressed in Mooney Units (MU). The scorch time and the optimum cure time were determined from the cure traces generated at 140 ± 2 °C by an oscillating disc rheometer curemeter¹⁶. The cure rate index, which is a measure of the rate of cure in the rubber, was calculated using the method described in the British Standard¹⁷. The rheometer tests ran for up to one hour. Results from these experiments are summarized in Table 1.

Test Pieces and Test Procedure

After these measurements were completed, the rubber compounds were cured in a compression mould at 140°C with a pressure of 11 MPa. Pieces of rubber, each approximately 130 g in weight, were cut from the milled sheet. Each piece was placed in the centre of the mould to enable it to flow in all directions when pressure was

applied. This prevented anisotropy from forming in the cured rubber. For determining the mechanical properties of the rubbers, sheets 23 cm by 23 cm by approximately 2.6 mm thick were used, from which various samples for further tests were cut

Hardness, Cohesive Tear Strength, and Tensile Properties of the Rubber

Vulcanisates

The hardness of the rubber was measured in a Shore A durometer using cylindrical samples 12 mm thick and 28 mm in diameter according to the British Standard¹⁸. The cohesive tear strength was determined at an angle of 180°, at ambient temperature (22.5°C) and at a constant cross-head speed of 50 mm/min¹⁹. The tensile stress, elongation at break, stored energy density at break, and Young's modulus of the rubber were determined in uniaxial tension at 22.5°C and at a cross-head speed of 50 mm/min²⁰.

RESULTS AND DISCUSSION

Particle Size, Particle Size Distribution and Particle Shape of the Waste Powder

When the SEM photographs were examined, it was evident that the waste powder was made of particles and glass fibre fragments of different sizes and shapes. The glass fibre fragments were approximately 570 µm in length (Fig.1), whereas, the particles

had irregular shapes and were from 0.5 to 200 μm in size (Fig.2). The results from the particle size analyser indicated that the powder had particles from 1 to 700 μm in size (Fig.3). Note that particles less than 0.02 μm could not be measured and this method did not differentiate between the glass fibre fragments and particles. Moreover, no information about the shape of the particles could be attained from the results in Figure 3. It was therefore concluded that SEM was a more useful method for measuring the particle size and determining the particle shape of the waste powder. The SEM technique also differentiated between the particles and glass fibre fragments in the powder whereas, the particle size analyser did not.

Surface Chemical Composition and T_g of the Waste Powder

Figure 4 shows typical spectrum of a glass fibre fragment consisting of aluminium (Al), calcium (Ca), silica (Si) and oxygen (O). The spectrum of a particle showed that it was made of the same elements and chlorine (Cl) and carbon (C) were also present. Elements from the glass fibre were clearly evident within the sampling depth of EDX. Therefore, there was no gross contamination of the glass fibre. However, the lack of direct evidence of primer and/or coupling agent and the relatively large sampling depth of EDX, i.e. 4-5 μm , means that there might not be desirable chemistry present

on the surface of the glass fibres for interactions to occur if the waste powder was to be recycled as filler in virgin rubber. Similarly, there was no direct evidence for functional groups on the waste powder. An alternative technique such as static secondary ion mass spectroscopy (SSIMS) could provide such information. It is worth noting that different amounts of these elements were present in the surface of the two particles analysed (Table 2). For example, O was 48.51 and 18.13 wt%, and Al 7.99 and 11.62 wt%, respectively. Particle 1 had 27.46 wt% Si and 16.04 wt% Ca, and particle 2, 67.34 wt% C and 2.91 wt% Cl, respectively. The glass fibre had 44.95 wt% O, 7.31 wt% Al, 29.94 wt% Si and 17.79 wt% Ca. It was interesting that Si, O and Al (probably from aluminate) were also detected in the particle surfaces. These elements are normally present in glass fibres.³ The presence of Ca and C in the particle surfaces might have been from calcium carbonate which was incorporated in the virgin polymer.¹⁰ When the SEM photograph taken of the powder at a higher magnification was examined, it was evident that the glass fibre fragments were coated with residual polymer from the virgin material (Fig.5), hence some of the similarities between the surface composition of the glass fibres and particles.

The surface composition of the waste powder measured by XPS consisted of C

74.0, O 23.3, Br 1.6 and Cl 1.2 atom%. Br and Cl might have been from flame retardant materials, C from the virgin polymer and O from boric acid in the glass fibre.¹⁰ The carbon bonding was approximately 60% C-C or C-H bonds, 30% C-O bonds and 10% O-C=O or connected by single bonds to three oxygens. This also confirmed the polymeric nature of the waste powder. Limited information was obtained from the Br signal, and the Cl peak was too low in intensity to determine its chemical state. The reduced sampling depth of XPS of approximately 5-10 nm compared with 4-5 μm for EDX identified increased C and O along with lower Br and Cl levels. The presence of carbonyl and carboxylic acid groups and the lack of Si also indicated that the fibre and powder materials were coated with residual polymer from the previous virgin material.

The DSC trace indicated a T_g of 135.4°C for the waste powder (Fig.6). When the FTIR spectrum from the waste powder was matched against the standard spectra of commonly known polymer materials, it indicated a thermoset polyester resin (Fig.7).

As mentioned earlier,^{3,9,11} polymer composite wastes have been recycled as filler in virgin polymers such as raw rubbers. Fillers with surface area less than $400\text{ m}^2/\text{g}$

or particle size smaller than 500 nm offer the highest level of reinforcement and can improve tensile strength, tear strength, hardness and modulus.^{14,21} There are also extender fillers. For example talc and calcium carbonate (CaCO_3), which can replace a portion of the virgin polymer in compounds and reduce costs because they are cheaper than virgin polymers. The particle size of the waste powder was from 0.5 to 700 μm (Figs.1-3). Furthermore, the XPS analysis showed no direct evidence for functional groups on the waste powder and therefore, it was unlikely that the powder would have formed strong bonding with virgin rubber. This evidence and the large particle size of the powder (Fig.3) indicated that the waste powder could be used as a non-reinforcing or an extender filler in rubber. However, when the results in Table 1 were examined, it was clear that the inclusion of up to 25 phr waste powder had affected the processing and mechanical properties of the rubber differently. For example, the viscosity rose by 40%, the scorch and optimum cure times increased by 24 and 118%, respectively, and the rate of cure decreased by 60%. Δ torque, which is an indication of crosslink density changes in the rubber, also increased by 10%. The waste powder was beneficial to the crosslink density of the rubber. The tearing energy and tensile strength increased by 66%, respectively, stored energy density at break by

186%, elongation at break by 63%, Young's modulus by 50% and the hardness remained unchanged. This was in contrast to the results reported previously for natural rubber (NR) with the same formulation where the hardness and tearing energy were unchanged, and the tensile strength, elongation at break, and stored energy density at break deteriorated. The effect on the scorch and optimum cure times and cure rate index were similar to the ones recorded for the SBR rubber (Table 1). Since the waste powder improved the mechanical properties of the SBR rubber, it could be classified as a reinforcing filler. In view of the results reported previously for NR²² and the findings of this work, there is an early indication that the ability of a waste powder to reinforce mechanical properties of a vulcanisate will depend mainly on the composition of the raw rubber.

CONCLUSIONS

From this study, it was concluded that:

- 1 - The waste powder was made of particles and glass fibre fragments of different sizes and shapes. The SEM technique showed that the particles had irregular shapes and their sizes increased from 0.5 to 200 μm , and the glass fibre fragments were approximately 570 μm in length. The results from the particle size analyser

indicated that the waste powder had particles from 1 to 700 μm in size.

2 – The surface chemical composition of the glass fibre fragments was Al, Ca, Si and

O, and the particles Cl, C, O, Si, Ca and Al. However, the amounts of these

elements varied considerably between the particles and glass fibre fragments.

Furthermore, there was no direct evidence for functional groups on the powder phase.

3 – The waste powder was identified to be a thermoset polyester resin with a glass

transition temperature of 135.4°C.

4 – The tensile strength, elongation at break, tearing energy, stored energy density

at break, and Young's modulus of the rubber vulcanisate increased when the

waste powder was added. The waste powder can therefore be used potentially as

reinforcing filler in SBR. Notably, the waste powder had a detrimental effect on

the scorch and optimum cure times and rate of cure of the rubber.

In summary, advanced techniques such as SEM, particle size analyser, FTIR, XPS,

EDX and DSC can be used to determine particle size, particle size distribution,

particle shape, surface chemical composition and glass transition temperature of glass

fibre reinforced plastic solid waste powders. The information produced by these

techniques are useful but not sufficient to decide whether a waste powder can be used as reinforcing filler or extender one for rubber. This will depend on the composition of the raw rubber. Nevertheless, this will be a potentially useful recycling route for thermoset polyester resin wastes and will avoid disposal to landfill and incineration.

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FIGURE CAPTIONS

- 1 SEM photograph of the GRP powder showing irregular shaped particles and glass fibre fragments.
- 2 SEM photograph of the GRP powder showing irregular shaped particles of different sizes.
- 3 Volume percentage versus particle size data for the waste powder.
Data produced with Malvern Mastersizer 2000 Ver.5 particle size analyser.
- 4 EDX spectrum of a glass fibre shown in Figure 1.
5. SEM photograph of the waste powder showing particles and glass fibres. Note residual polymer on the long glass fibre surface.
- 6 DSC trace showing the glass transition temperature of the waste powder.
- 7 FTIR spectrum of the waste powder.

TABLE 1. RECIPES, VISCOSITY, CURE AND MECHANICAL PROPERTIES OF THE RUBBER COMPOUNDS

Formulation (p.h.r.)	Compound number		
	1*	2	3
SBR Intol 1712	100	100	100
Waste powder	0	15	25
Stearic acid	2	2	2
Santocure CBS ^a	1	1	1
Sulphur	2.5	2.5	2.5
Zinc oxide	5	5	5
Santoflex 13 ^b	1	1	1
Mooney viscosity ML (1+4, 100°C)	32	43	45
	ODR Results		
Scorch time, t_{s2} (min)	16.5	18.5	20.5
Optimum cure time, t_{95} (min)	49	84	107
Cure rate index (min^{-1})	3	1.5	1.2
Δ torque (dN m)	69	69	76
	Mechanical properties		
Hardness (shore A)	48	50	50
Tensile strength (MPa)	1.8	2.3	3.0
Elongation at break (%)	411	441	669
Stored energy density at break (MJ/m^3)	4.2	6.3	12.0
Young's modulus (MPa)	1.2	1.7	1.8
Tearing energy (kJ/m^2)	3	4.6	5

1* Control or unfilled compound

a N-cyclohexyl-2-benzothiazole sulphenamide (CBS)

b N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylenediamine

TABLE 2. SURFACE COMPOSITIONS OF PARTICLES AND GLASS FIBRES IN THE WASTE POWDER

Element	Particle 1 wt%	Particle 2 wt%	Glass fibre wt%
O	48.51	18.13	44.95
Al	7.99	11.62	7.31
Si	27.46	-	29.94
Ca	16.04	-	17.79
C	-	67.34	-
Cl	-	2.91	-

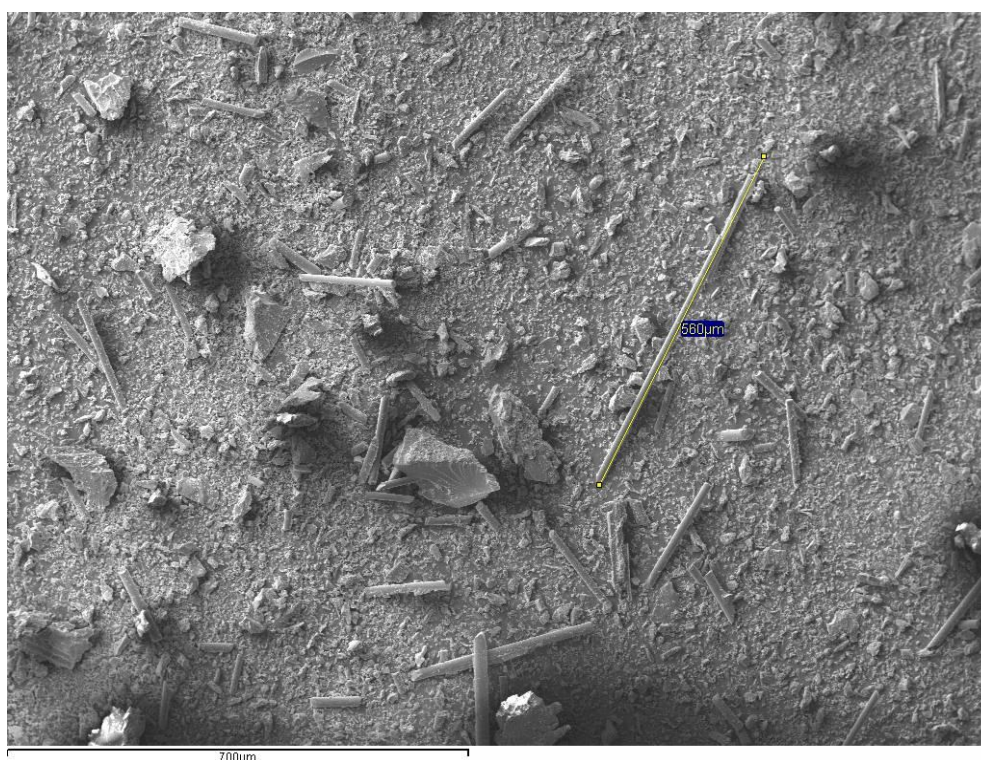


Figure 1 SEM photograph of the waste powder showing irregular shaped particles and glass fibre fragments.

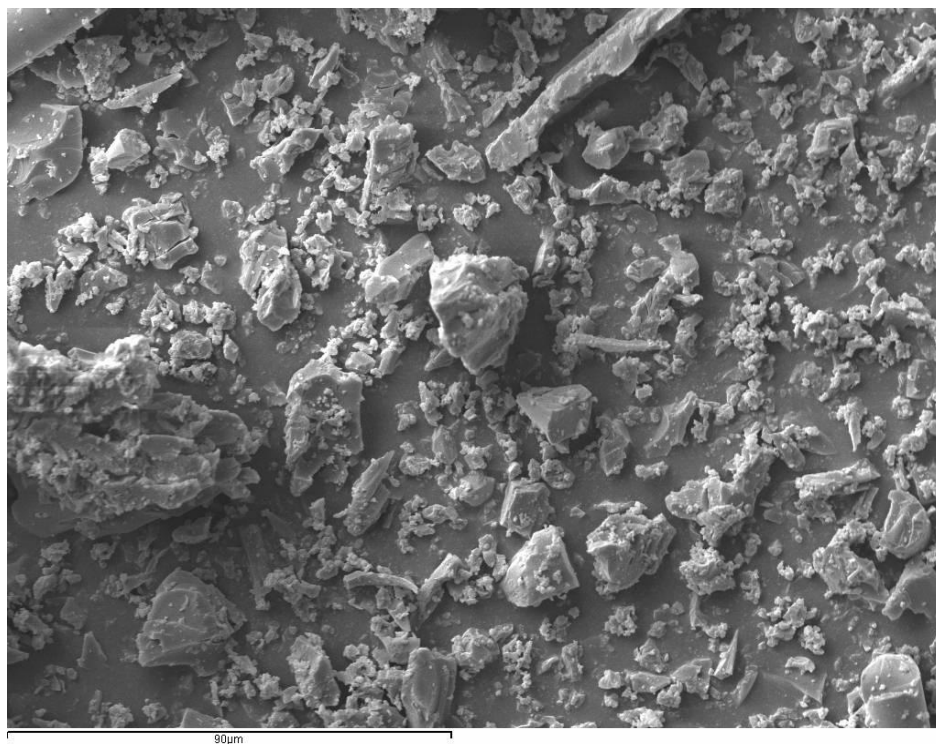


Figure 2 SEM photograph of the waste powder showing irregular shaped particles of different sizes.

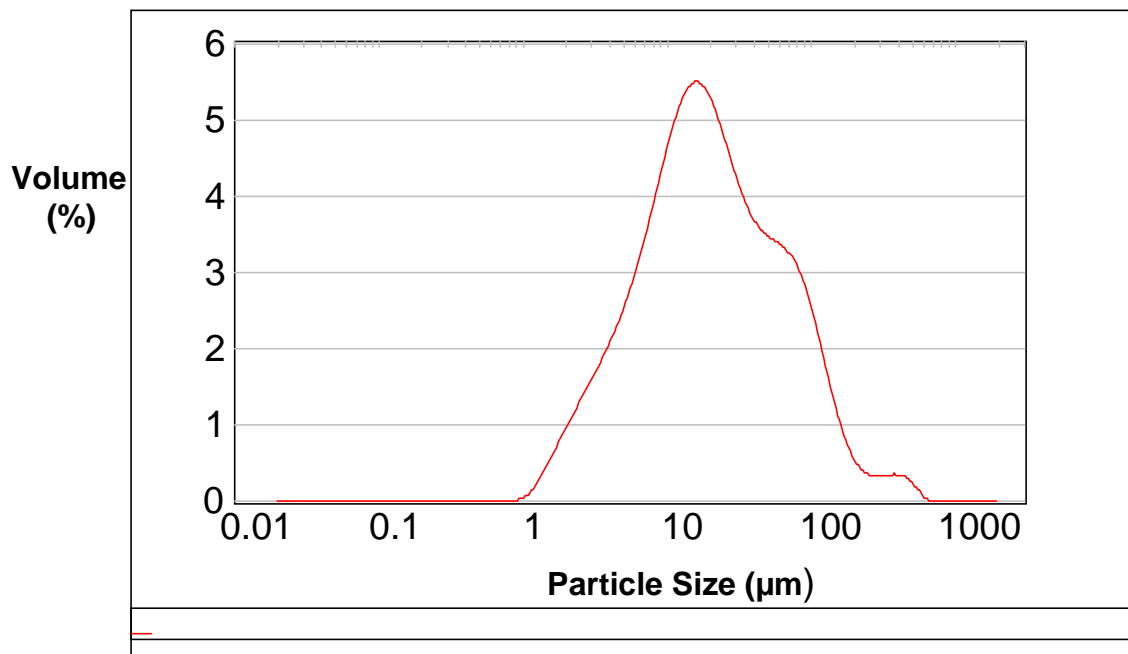


Figure 3 Volume percentage versus particle size distribution profile of the waste powder. Data produced with Particle Size Analyser.

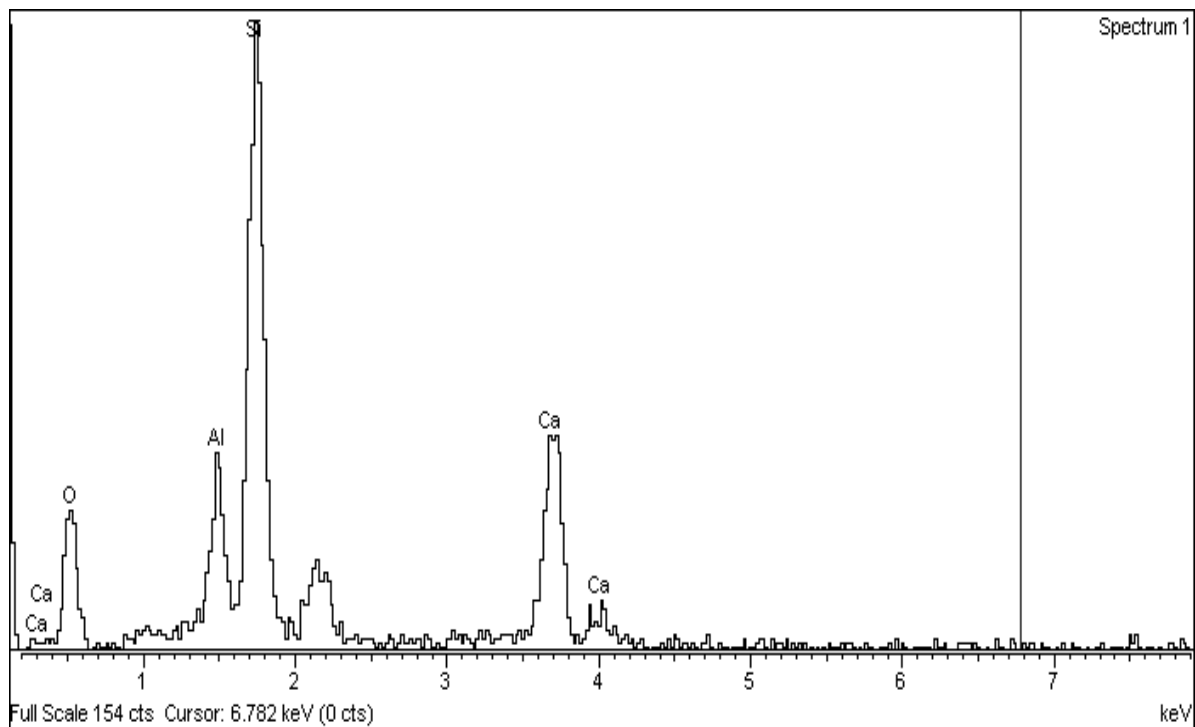


Figure 4 EDX spectrum of a glass fibre shown in Figure 1.

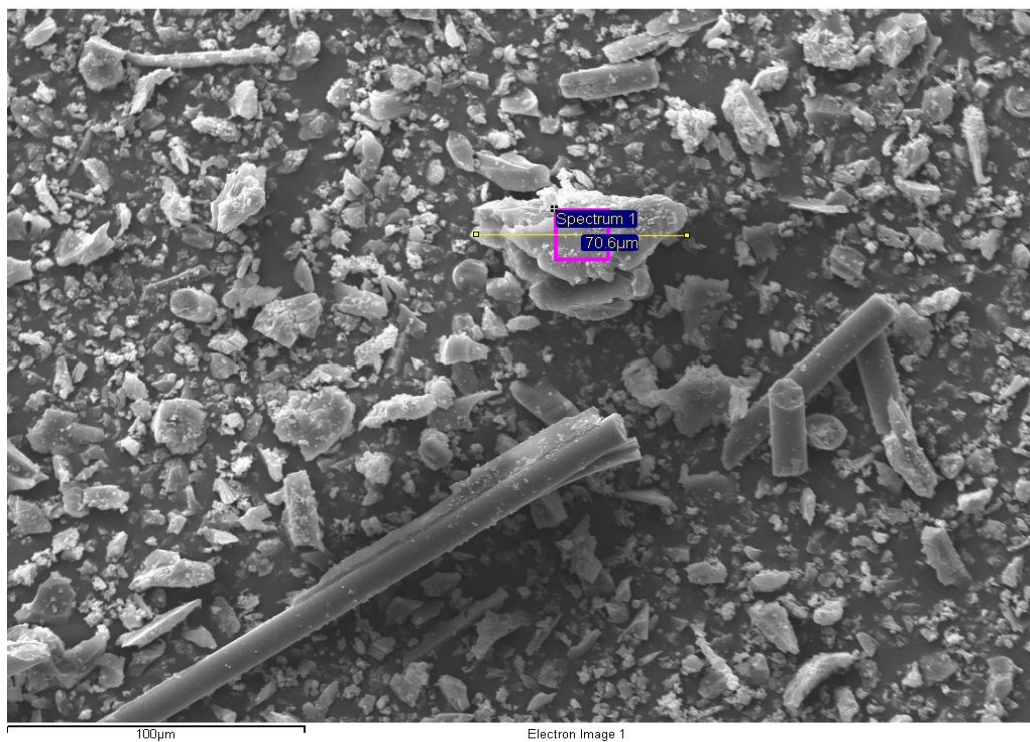


Figure 5 SEM photograph of the waste powder showing particles and glass fibres. Note polymer residual on the glass fibre surfaces after grinding.

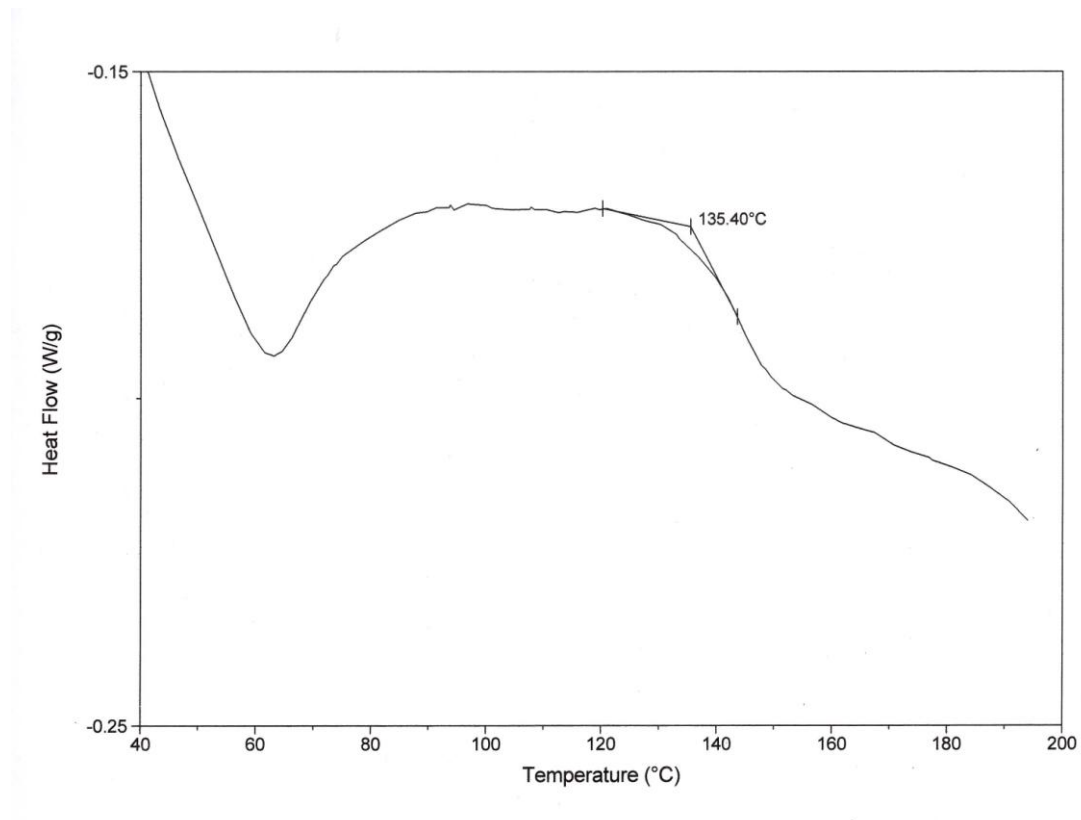


Figure 6 DSC trace showing the glass transition temperature of the waste powder

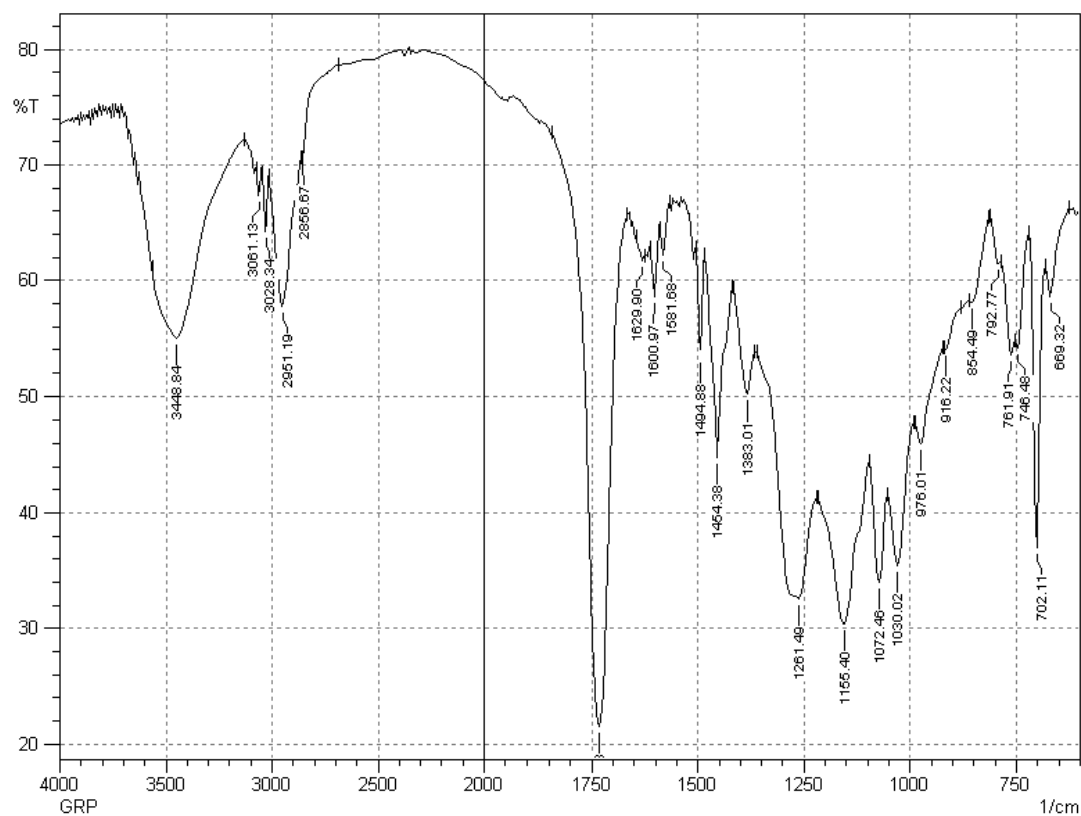


Figure 7 FTIR spectrum of the waste powder