Upgrading and reuse of glass fibre recycled from end-of-life composites

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Abstract. The value of recycled glass fibres is significantly reduced due to the loss of fibre strength and surface functionality that occurs during recycling. Results are presented from the ReCoVeR project on the regeneration of the strength of thermally conditioned glass fibres. Thermal recycling of end-of-life glass fibre reinforced composites or composite manufacturing waste delivers fibres with virtually no residual strength or value. Composites produced from such fibres also have extremely poor mechanical performance. Data is presented showing that a short hot alkali treatment of glass fibres which have been heat treated at typical composite recycling temperatures can more than triple their strength and restore their ability to act as an effective reinforcement in second life composite materials. Glass fibre recovered from fluidised bed recycling of composite materials exhibited much greater levels of mechanical abrasion damage. However, the strength of these fibres could also be increased to levels required in composite reinforcement by longer or more aggressive alkali treatment. The implications of these results for real materials reuse of recycled glass fibres as replacement for pristine reinforcement fibres are discussed.

1. Introduction

The disposal of composite manufacturing waste and end-of-life composite products in an environmentally friendly manner is one of the most important challenges currently facing the industrial and academic composites community. Glass fibre was the reinforcement of choice in more than 90% of all these composites and more than six million tons of reinforcement grade E-glass fibre was used in composite production in 2019. This will have potentially been associated with the production of up to one million tons of glass fibre manufacturing waste [1] most of which is landfilled. Approximately 70% of reinforcement glass fibre is used to manufacture thermoset based glass reinforced plastics (GRP) which also produces approximately 15% manufacturing waste. Such GRP materials (both end-of-life and manufacturing waste) are difficult to recycle in an efficient manner and have historically also been disposed of in landfills. Such landfilling is rapidly becoming untenable due to legislative and landfill cost developments. The perspectives on this issue have been recently highlighted due to the accelerating growth in the use of such composite materials in transportation and wind energy sectors [2-4]. A number of processes are available for recycling such composites and thermal recycling is probably the most technologically advanced [2,3]. However, nearly all options deliver recycled glass fibres (RGF) that suffer from a lack of cost competitiveness with pristine first-pass materials.
A critical technical challenge in the development of GRP recycling technology is the 80-90 % drop in the performance (and value) of RGF in comparison to its original state [5-9]. Recent studies have confirmed that the room temperature glass fibre strength can be drastically reduced by exposure to temperatures in the 300°C-600 °C temperature range [5,6] typical of the many different potential GRP recycling processes (see Figure 1). Similar behaviour has also been observed in silica and basalt reinforcement fibres [6]. Consequently, RGF have a very poor performance to cost ratio, and in most cases are unsuitable for reprocessing and reuse as a valuable reinforcement for composites. A breakthrough in this field could enable RGF to compete with pristine materials in high volume discontinuous fibre reinforced composite applications which would have major technological, societal, economic and environmental impacts. Yang et al. [7] recently demonstrated that the reinforcement potential of RGF could be significantly improved by a post treatment with hydrogen fluoride (HF) to remove the “damaged” fibre surface layer. However, given the very aggressive nature of HF and the associated safety issues, it seems unlikely that a cost-effective regeneration process could be based on HF treatment. We have been researching less aggressive chemical routes to achieve similar results. The literature generally refers to the effects of acid or alkali treatments in terms of their deleterious effects on glass fibres and the resultant lowering of composite strength. However, this body of work is almost universally predicated on maintaining the performance of strong fibres. Little if any work has been reported on the use of acid or alkali treatments to change the strength of very weak fibres. However, it is well known that a silica network can be dissolved by the use of hot alkali solutions [1].

The ReCoVeR project (Regenerated Composite Value Reinforcement) focuses on enabling cost-effective recycling of glass fibre thermosetting composites. In particular ReCoVeR investigates the possibility of regenerating the strength of recycled glass fibres to enable their reuse as a composite reinforcement replacing the use of pristine fibre materials. In this paper we report some recent research results on the use of hot alkali solutions and subsequent silane sizing to modify the surface of thermally recycled glass fibres and the resultant effects on glass fibre and composite strength.

![Figure 1. Average glass fibre strength after 25 min elevated temperature conditioning](image)

2. Experimental materials and methods

2.1. Materials
Thermal and chemical treatments were carried out on aminosilane coated (OC-APS) boron-free E-glass fibres supplied by Owens Corning [5]. Glass mat thermoplastic (GMT) composites were prepared using PPG 8069 chopped glass fibres, a product designed for easy dispersion in aqueous media for the production of wet-laid non-wovens, supplied by PPG Fibreglass (now part of NEG) combined with DA3/60 chopped homopolymer polypropylene (PP) fibres purchased in the form of Goonvean. Injection moulded composites were prepared using DS 2200-13P chopped glass fibres provided by 3B Fibreglass company, SABIC® PP 579 S polypropylene (PP) pellets with 1 % added Polybond 3200 maleic anhydride-grafted polypropylene pellets. Glass fibre reinforced epoxy (GF-EP) composites for recycling were produced in-house by infusing an E-glass fibres tri-axial 3-ply mat supplied by Hexcel Reinforcements with PRIME 27 Resin and PRIME 20LV extra slow hardener supplied by Gurit. After ashing in a furnace, it was established that the resulting GF-EP had a fibre weight fraction of 60%. The cured GF-EP feedstock was cut to 50 × 15 mm so the recycled fibres were long enough to be tested with a 20 mm gauge length.

2.2. Fibre treatments
Fibre bundles were arranged in a steel rig for thermal conditioning, which was carried out in air. A Carbolite furnace was used to treat the fibres at a range of temperatures up to 600 °C for 25 minutes. The rig was then extracted from the furnace and left to cool at room temperature, before the heat-treated fibre bundles were removed for further chemical treatments. These fibres are defined are glass fibres recycled thermally (rGFt). More details of the heat conditioning procedure are given in [5]. NaOH solutions were prepared according to the desired molarity and heated to 95 °C before treating the fibre bundles for the required times. After the fibres had been treated in alkaline solution, they were rinsed in 37 % HCl and deionised water for 1 minute respectively. The purpose of this rinsing procedure was to ensure the effective removal of residual deposits which developed on the fibre surface as a result of interaction with alkaline solution [1]. The SEM micrograph in Figure 2a shows a typical example of these deposits on an NaOH treated heat conditioned fibre. Where required, a 1 vol% γ-aminopropyltriethoxysilane (APS) aqueous solution was used to regenerate the surface functionality of the heat treated and alkali treated fibres. The fibres were immersed in the APS solution for 15 minutes. Samples for fibre testing were dried, however samples for GMT wet production were processed directly into the composites. Figure 2b shows the fibre surface of a heat conditioned fibre after NaOH treatment and silane treatment. This fibre exhibits the virtually featureless surface which is typically observed with new glass fibres and confirms the removal of the deposits seen in Figure 2a which can be very detrimental to the efficacy of the silane treatment [1].

Figure 2. SEM images of rGFt after a) NaOH treatment, b) NaOH + HCl rinse + APS coating.
2.3. Recycling glass fibre reinforced epoxy composites using fluidised bed

The GF-EP composites were recycled using an in-house built fluidised bed recycling process [10]. The thermosetting polymer matrix was thermally decomposed within the fluidised bed reactor, which comprises silica sand fluidised with air pre-heated using an electric heater. The composite feedstock was fed into the reactor through an access pipe, located above the expanded bed. The fluidising air was supplied to the reactor via a centrifugal fan and was sufficient to entrain the liberated glass fibres from the bed and into the reactor freeboard. An induced draft fan was used to draw the glass fibres and polymer volatiles from the freeboard and to keep the reactor under a slight negative pressure. The glass fibres were recovered from the gas stream using a cyclone separator. The bed temperature, static bed height and fluidisation velocity remained constant throughout this work at 500 °C, 150 mm and 1.5 m/s respectively. These fibres are defined as glass fibres recycled from composites (rGFc).

2.4. Single fibre tensile testing

Single fibre tensile testing was performed following the standard ASTM C1557-03 at a gauge length of 20 mm. Fibre diameters were measured using an optical microscope before testing for tensile strength using a Testometric tensile testing machine at ambient environment. The load cell was 5 N with an applied strain rate of 1.5 %/min. At least 30 fibres of each sample were tested. Error bars associated with the strength measurements represent 95 % confidence limits [1,5].

2.5. GF-PP Composite Preparation and Testing

The influence of the properties of glass fibres after thermal recycling and chemical regeneration on composite performance was further investigated by the preparation of 30 % in-plane randomly oriented glass fibre reinforced polypropylene laminates (GMT) prepared by a wet-laid papermaking process and compression moulding as previously described [1]. A water jet cutter was used to cut dog bone shaped tensile test specimens. Tensile tests were performed following ISO 527 using an Instron 5969 universal testing machine equipped with a 50 kN load cell and a crosshead speed of 1 mm/min. For injection moulded composites a Betol BC25 extruder was first used to compound PP pellets with 30% by weight glass fibres. The extruded material was drawn through a water bath and cut into pellets using a rotary cutter. An Arburg 170-90/200 injection moulding machine was used to produce dog-bone shaped tensile test specimens according to ASTM 638. The processing temperatures were set between 170°C and 230°C. All samples were conditioned at room temperature for three weeks before mechanical testing. An Instron 596 testing machine was used to perform the tensile tests. Sample strain was recorded with a video extensometer. A constant head displacement rate of 1 mm/min was used to test the composites. The PP reference sample was tested with a head displacement rate of 1 mm/min up to 3 % strain. Then the head displacement rate was increased to 5 mm/min. Unnotched Charpy impact test specimens according to ISO 179-1 were cut from injection moulded tensile bars. A Tinius Olsen Impact 503 impact tester with a 25 J hammer was used to perform the impact tests.

3. Results

3.1. Fibre Strength after Thermal and Chemical Treatment

The results for the average single fibre tensile strength of the OC-APS fibres conditioned at 450, 500 and 600 °C and then treated in hot NaOH and APS are summarised in Figure 3 [1]. It can be seen that thermal conditioning caused a considerable reduction in fibre strength, with a loss of over 85% of the original strength. After a 10 minute treatment in hot NaOH solution the fibre strength initially increases significantly. It was also found that with prolonged treatment times and higher alkali concentrations the fibre strength improvement was significantly reduced [8]. It is generally accepted that the measured strength of glass fibres is dependent on their brittle nature and the state of the fibre surface region. Fundamental research on the thermally induced strength loss of glass fibre indicates that the phenomenon may have a complex nature but that it appears to be caused mainly by changes on the fibre surface. We have also recently demonstrated that the reinforcement potential of rGF can be
significantly improved by a post treatment with hydrogen fluoride (HF) to remove this “damaged” surface layer [7].

Figure 3. Influence of recycling temperature and ReCoVeR treatments on single fibre strength.

The results of a further investigation of fibre strength regeneration are also shown in Figure 3. OC-APS fibres were thermally conditioned at three different temperatures and subjected to either hot NaOH alone or hot NaOH followed by application of a simple APS sizing. The results obtained by Yang et al on the same fibre, thermally conditioned in an identical manner but then treated using HF etching, are also included in Figure 3 for comparison [7]. The large drop in average fibre strength (from an untreated reference value of 2.3 GPa) was observed after HT alone at all three temperatures. It was also noted that, after thermal conditioning, the fibres were extremely brittle and difficult to handle without breakage. Hence thermal recycling not only results in a very large drop in the glass fibre performance but also makes rGF virtually impossible to process in any standard composite production equipment. The results for the fibre strength recovery achieved with the hot NaOH treatment are impressive, with the strength regeneration for all three HT temperatures reaching approximately 1.5 GPa from the initial value of the HT fibre strength of (less than) 0.6 GPa. The addition of a simple APS sizing after the hot NaOH treatment leads to a further significant (at the 95 % confidence level) increase in fibre strength regeneration to well above 1.5 GPa. We consider this value of 1.5 GPa to be an important target for strength regeneration of recycled fibre in order to compete, on performance, with commercial first-pass glass fibre products [9].

From the above results, it can be seen that significant increases of fibre strength were obtained through hot alkali regeneration treatments, achieving greater than a tripling of fibre strength over the thermally treated fibre values. Consequently, hot alkali treatments enable regeneration of fibre strength to a level which makes reusing these fibres as a composite reinforcement a viable technical option.

3.2. Performance of Regenerated Fibres in GF-PP Composites

Figure 4 presents the results for the tensile strength of the GF-PP GMTs produced using thermally conditioned (HT 25 min at 500°C) and chemically treated fibres (NaOH and APS) [1]. The sample containing the HT fibres exhibits a large drop in tensile strength due to the low strength of the fibres.
and the low level of interfacial adhesion in this composite. GMT tensile strength recovers by 44% when the HT fibres are given a hot NaOH treatment. Addition of silane coupling agent after the NaOH treatment results in a 74% recovery of the GMT tensile strength. To the best of our knowledge the result shown here is one of the first published demonstrations of a chemical treatment, other than HF etching, that significantly increases the strength of thermally recycled glass fibres. Moreover, the results on GMT laminates clearly show that this regeneration of glass fibre strength can be translated directly into composite strength. There are many non-technical factors which will play a role in the eventual commercial profitability of any GRP recycling process. However, maximizing the performance regeneration of rGF will, enable replacement of pristine fibre products from a performance viewpoint, maximize the value of such a recycled fibre product, and increase the economic attraction of composite recycling. The development of a non-HF based glass fibre strength regeneration treatment is an exciting development in the progress towards a cost-effective GRP recycling technology.

Figure 5 summarises the data on the tensile strength and unnotched Charpy impact performance of the injection moulded, glass reinforced polypropylene composites [11]. These composites were prepared with glass fibres that had been thermally conditioned for 25 min, across a range of temperatures. It can be seen that the tensile strength of these composites is also significantly reduced by the glass fibre thermal conditioning prior to composite manufacture. The glass fibre heat-treatment at 300°C caused a tensile strength reduction of more than 40%. A glass fibre heat treatment at higher temperatures caused a drop of the tensile strength of greater than 50%. The tensile strength of unreinforced PP was measured as 35.8 MPa. Consequently, the thermal conditioning of the glass fibres above 300°C essentially removed 100% of the reinforcement effect of the fibres in terms of increasing tensile strength of the composite in comparison with the tensile strength of the PP polymer alone. Similarly, the thermal conditioning of the glass fibres resulted in a loss of more than 50% of the unnotched impact performance of the composite. These results clearly reveal the potential poor reinforcement performance of any glass fibres obtained by thermal recycling of composites. The stiffness of the GF-PP composites was not seriously comprised by the thermal conditioning but the large drop in strength means that there is unlikely to be any significant commercial interest in large-scale application of rGF unless the properties of the rGF can be cost-effectively regenerated.

![Figure 4](image1.png)  **Figure 4.** Influence of ReCoVeR fibre treatments on GF-PP GMT tensile strength

![Figure 5](image2.png)  **Figure 5.** Influence of ReCoVeR treatments on injection moulded GF-PP performance

Injection moulded glass fibre reinforced PP composites were produced where the heat-treated fibres were also subjected to NaOH regeneration treatment and APS coating prior to extrusion and moulding. Data on the tensile strength of these composites is also shown in Figure 5. It can be seen that 71% of the composite strength loss due to thermal preconditioning of the glass fibres at 500°C was recovered by use of this treatment. The unnotched impact strength recovery of these composites was even more impressive with 87% of the heat treatment loss in composite impact strength...
performance recovered. This level of composite performance recovery exceeds the 50-70% regeneration of the properties of GF-epoxy composites reported by Yang et al [7] when using HF treatment to regenerate the strength of heat-treated glass fibres. The application of a full commercial PP compatible sizing [12,13] to the ReCoVeR treated fibres may well increase the final composite performance further.

3.3. Tensile strength of fibres recycled from composites

Thomason has recently reviewed the available literature on the thermally induced strength loss in glass fibres and concluded that the mechanism of strength loss in thermally recycled glass fibre involves a combination of thermal and mechanical damage. Clearly the potential for greater levels of mechanical damage are present when considering potential large-scale industrial recycling processes [6,14]. Figure 6 shows the single fibre tensile strength for the same glass fibres after only thermal conditioning rGFt compared to fully recycled rGFc from fluidised bed processed composites, both following treatment in the NaOH solutions. The NaOH treatment time was kept constant at 10 min and solution concentrations of 1.5, 3 and 5 mol/L were used. The strength of glass fibre after thermal conditioning increases with NaOH concentration. Higher concentrations may continue to improve the strength of these fibres since a plateau does not appear to have been reached in the range of concentrations studied. However, these NaOH treatments showed little effect on the strength of rGFc, with a relatively consistent single fibre strength of 0.5 - 0.6 GPa in all cases. It has been hypothesised that the reaction of silica in the glass fibre with hydroxide ions from the NaOH solution blunts and/or removes the severe surface flaws that manifest during exposure to elevated temperatures, and thus increases the tensile strength of the fibre [1,8]. Given their significantly lower strength, it seems likely that there are more severe flaws on the surface of the rGFc compared to the rGFt.

![Figure 6. Tensile strength of rGFt and rGFc after NaOH treatment at different concentrations](image)

The surface of fibres recycled in the fluidised bed was further studied under SEM, as seen in Figure 7. The presence of apparent damage was found on the surface of rGFc in the form of scratches and depressions. No such features were observed on the surface of glass fibres simply thermally conditioned within a furnace at the same temperature, suggesting the damage is a result of mechanical attrition during recycling. Given the high hardness of silica sand, abrasion from this sand in the fluidised bed may be a likely cause. Pickering et al. [3], Fenwick [15] and Kennerley [16,17] reported the properties of glass fibres recycled using a fluidised bed, however, no such features were discussed.
The depth of the damage cannot be determined using SEM; however the topography of the fibre surface could be further explored using atomic force microscopy.

Figure 7. SEM images comparing surface features of (a) rGFt, (b) rGFc (c) rGFc

Given the lack in strength increase under the conditions used in Figure 6, rGFc were treated with NaOH for extended times. Figure 8 gives the strength and relative diameter of rGFc after treatment with a 5 mol/L solution of NaOH for times up to 180 min. There is a clear increase in tensile strength with treatment time when treating for longer than 20 min. The fibre strength appears to begin to plateau at 1.25 GPa after treating for 180 min, suggesting only minimal additional strength gain can be achieved by extending treatment duration. The reduction in rGFc diameter with treatment time is more pronounced when extending the range of NaOH treatment times. Similar to the tensile strength data, significant effect of the NaOH on rGFc diameter was not observed for treatments shorter than 60 min.

Figure 8. Tensile strength and relative diameter of rGFc treated with NaOH at 5 mol/L

It was anticipated that removing additional damaged surface material could lead to re-strengthening of recycled fibres; therefore, additional concentrations of NaOH solutions were applied to rGFc at extended treatment times. Figure 9 gives the tensile strength of rGFc then treated with NaOH at the
various concentrations and times. All concentrations investigated, except 1.5 mol/L NaOH solution, increased the strength of rGFc. In agreement with the rGFt data in Figure 6, the effectiveness of NaOH on rGFc strength tends to increase with NaOH concentration at these longer treatment times. rGFc treated with 1.5 mol/L NaOH solution may exhibit an increase in strength if the treatment times were extended further [8,18]. It appears there is a maximum strength increase range of 1.2-1.4 GPa for 7 and 10 mol/L treatments, where extending the treatment time does not improve the fibre strength. This plateau occurs more rapidly when higher concentrations are used. Figure 9 shows the strength of fibres treated at 7 and 10 mol/L appear to plateau after treatments lasting 120 and 60 min respectively. The 5 mol/L treatment also appears to be reaching the plateau level and may reach its maximum level for about 4 hr treatment time. No plateau in regenerated fibre strength is observed for 3 mol/L treatments which approximate a linear increase in strength within the treatment times studied. Extended treatments at 3 and 5 mol/L are required to determine if/when the fibre strength plateaus when using these concentrations. The maximum possible strength increase may in fact be the same for all concentrations, with lower concentrations requiring longer treatments to converge.

![Figure 9](image1.png)  
**Figure 9.** Tensile strength of rGFc treated with NaOH at different concentrations and times

![Figure 10](image2.png)  
**Figure 10.** Diameter of rGFc treated with NaOH at different concentrations and times

Figure 10 shows the rGFc diameter after NaOH treatment at the various times and concentrations used in Figure 9. The trend lines represent second order polynomials with a minimum R^2 value of 0.91. rGFc treated with 1.5 mol/L solution exhibit no significant change in diameter until treated for 180 min. All treatments which exhibit an increase in fibre strength in Figure 9, also show a significant reduction in fibre diameter. Both fibre diameter reduction and rate of reduction tend to increase with treatment time as shown in Figure 10. Yang et al. observed an inverse relation between rate of fibre reduction and treatment time when etching glass fibre with HF [7]. This was attributed to the build-up of insoluble residue particles on the fibre surface masking the fibre surface and impeding the etching process over time. However, this does not seem to occur when using NaOH within the treatment times studied. The observations made in Figure 10 are in line with Bashir et al. who examined the kinetics of dissolution of glass fibres in NaOH solution, finding that a radial diffusion model does not describe the reaction well [18].

Under the conditions investigated, the maximum rGFc tensile strength attained was approximately 1.4 GPa after treating for 120 min at 7 mol/L. This is comparable to the strength level obtained for the heat treated fibres shown in Figure 3 which could be further processed to give the excellent composite performance discussed in Figures 4 and 5. It was previously demonstrated by Thomason and Kalinka that the actual average strength of fibres in a commercial 4 mm chopped glass product used in extrusion and injection moulding was as low as 1.5 GPa at a gauge length of 2 mm [9]. It is well established that there is an inverse relation between fibre tensile strength and gauge length, therefore, the regenerated strength of 1.4 GPa at 20 mm gauge length found in this work may provide a fibre strength distribution that is higher than that obtained in typical commercial discontinuous glass fibre products. It seems likely that injection moulded composite applications requiring short glass fibres will
be a better starting point for the use of recycled fibres than those requiring strength at longer lengths, such as the 10-20 mm fibres used in long fibre thermoplastic processes as these will have a higher probability of still having a critical flaw on their larger surface.

4. Conclusions
Glass fibres lose 80 % or more of their strength when exposed to temperatures used in GRP thermal recycling processes making them unsuitable for reuse as a composite reinforcement. The results of single fibre tensile testing clearly show that more than 50% of that strength loss can be recovered by a short treatment of the fibres using hot alkali solution. This strength recovery can be maintained and improved upon by a further application of a silane sizing which also acts to recover the compatibility of the recycled fibre surface with a polymer matrix. The regeneration of the performance of thermally treated glass fibres by these simple chemical methods resulted in GF-PP GMT and injection moulded composites which exhibited a 70-90 % property recovery compared to composites based on thermally recycled fibres which had received no further treatment. It is proposed that further optimization of these chemical treatments can lead to further improvements in performance enabling then to be used to replace glass fibre products used in many discontinuous glass-fibre reinforced composite applications. However, real-life recycling of glass fibres from composites exposes fibres to both thermal and mechanically abrasive conditions. Consequently, the treatment time and/or NaOH solution concentration required for strength regeneration was found to be significantly higher for glass fibres recycled from composites in a fluidised bed, compared to fibres thermally conditioned in a furnace at the same temperature. This was attributed to fibres sustaining a greater degree of damage during the recycling processes, therefore, requiring a greater degree of surface etching to remove/sufficiently modify the surface flaws. It was found that treating such fibres from fluidised bed composite recycling in 7 mol/L NaOH solution for 2 hr yielded approximately a 130% increase in fibre tensile strength.

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