



Regenerating the strength of thermally recycled glass fibres using hot sodium hydroxide



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ABSTRACT

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 sodium hydroxide solution treatment of such recycled fibres can more than triple their strength and restore their ability to act as an effective reinforcement in second life composite materials. The implications of these results for real materials reuse of recycled glass fibres as replacement for pristine reinforcement fibres are discussed.

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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. 2015 global production of fibre reinforced composite materials will significantly exceed 10 million tons [1]. Glass fibre (GF) is the reinforcement of choice in more than 90% of all these composites and more than 5 Mton of reinforcement grade E-glass fibre was produced in 2015 [2]. In fact, some available data indicates that E-glass consumption could already be as high as 7 Mton when yarns are included [3]. The manufacture of this volume of GF results in 0.5–1 million tons of GF manufacturing waste [4] most of which is landfilled. Furthermore, approximately 70% of reinforcement glass fibre is used to manufacture thermoset based composites (GRP) which also produces approximately 15% manufacturing waste [5]. 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 [6–9]. A number of processes are available for recycling such composites and thermal recycling is probably the most technologically advanced [6,9].

However, nearly all options deliver thermally recycled glass fibres (TRGF) 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 TRGF in comparison to its original state [9–13]. Processing temperatures in the production of glass fibre are significantly higher than GRP recycling temperatures. Nevertheless, earlier work has indicated that the room temperature tensile strength of glass fibre can be significantly reduced by annealing at a temperature as low as 150 °C [9]. More recent studies have also confirmed that the room temperature glass fibre strength can be drastically reduced by exposure to temperatures in the 300–600 °C temperature range [11–13] typical of the many different potential GRP recycling processes. Similar behaviour has also been observed in silica and basalt reinforcement fibres [14,15]. Consequently, TRGF 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 such TRGF to compete with pristine materials in many large volume discontinuous fibre reinforced composite applications. The development of an economically viable process for regenerating the properties of thermally recycled glass fibres would have major technological, societal, economic, environmental impacts. Fig. 1 indicates that there is an on-going situation where up to 50% of the global demand for glass fibre reinforcements could potentially be supplied by TRGF from end-of-life and manufacturing waste recycling. The reuse of these materials could result in a huge reduction in the environmental impact of the glass-fibre and composites

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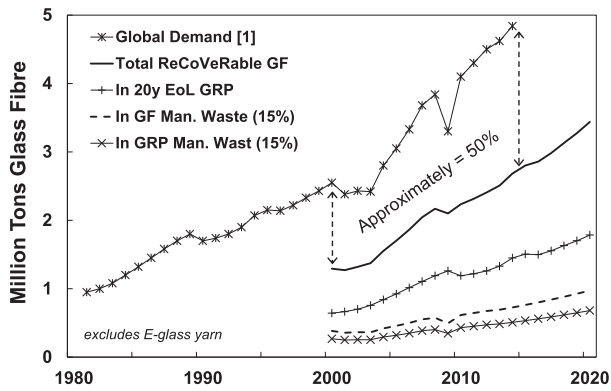


Fig. 1. Glass fibre global demand and availability for recycling.

industry where the replacement of 50% of current glass fibre products by TRGF products would equate to a global reduction in CO₂ production of 2 Mtons/annum from reduced melting energy requirements alone [16]. Furthermore, such a development would also vastly reduce the landfill requirements of the glass fibre and GRP industry. This would clearly be in line with the growing societal and environmental pressure to reduce landfill disposal, increase the reuse of valuable raw materials, and reduce CO₂ release to the atmosphere.

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 (sizing degradation and surface damage), or just below (“flaw” generation and growth, and dehydroxylation), the fibre surface. Yang et al. [17] recently demonstrated that the reinforcement potential of TRGF could be significantly improved by a post treatment with hydrogen fluoride (HF) to remove this “damaged” surface layer. Nevertheless, 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 [18] 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 under the appropriate conditions a silica network can be attacked, and in some cases dissolved, by the use of sodium hydroxide (NaOH) solutions [19,20].

As part of the EPSRC funded ReCoVeR project we have been focusing on enabling cost-effective recycling of glass fibre thermosetting composites. In particular we wish to show 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 present results showing the ability of hot NaOH and silane treatments to significantly increase the strength of thermally conditioned commercial continuous glass fibres. These chemical treatments are then shown to be equally effective in the strength regeneration of fibres thermally recycled out of model unidirectional continuous fibre reinforced thermoset composites. Finally, results are presented on the influence of fibre thermal conditioning and subsequent fibre strength regeneration through chemical treatments on the strength performance of laboratory produced discontinuous random in-plane reinforced polypropylene laminates.

2. Experimental

2.1. Materials

Initial screening work on the effect of NaOH treatment on the strength of glass fibre was carried out on boron-free E-glass fibres supplied by Owens Corning (OC). These OC-APS fibre rovings have been previously described in detail [12,13,21]. The rovings had a nominal tex of 1200 g/m and a nominal fibre diameter of 17 μm and had been coated with a 1% volume γ-aminopropyl triethoxysilane (APS) hydrolysed solution in deionised water. A similar glass fibre sample (3B-APS) of 14 μm diameter boron free E-glass coated with a 1% APS solution was supplied by 3B-Fibreglass. A boron containing E-glass roving (TufRov® 4599) supplied by PPG Fiber Glass was also used in fibre strength screening work. The 4599 roving is a 17 μm diameter continuous fibre product sized for polypropylene compatibility. To investigate the effect of fibre thermal conditional and subsequent chemical strength regeneration 30% weight discontinuous random in-plane fibre reinforced polypropylene laminates were prepared using a PPG supplied product. PPG 8069 is a chopped glass fibre product designed for easy dispersion in aqueous media for the production of wet-laid non-wovens. These 8069 fibres had a nominal diameter of 10 μm and were received as wet chopped bundles with a nominal fibre length of 9 mm. The homopolymer polypropylene (PP) for the production of composites was purchased in the form of Goonvean DA3/60 chopped PP fibres. The fibres were received chopped with a nominal length of 6 mm and a nominal linear mass of 3.3 dtex.

2.2. Glass fibre treatment

The procedure for thermal conditioning of glass fibres has been previously described in detail [12,13]. To imitate a composite recycling process, glass fibres were thermally conditioned in air. A ‘Carbolite CWF 12/13’ furnace was preheated to 450 °C, 500 °C, or 600 °C, and the fibres were placed in the preheated furnace for 25 min. After thermal conditioning the fibres were allowed to cool down at ambient temperature (21 ± 2 °C) outside of the furnace. The thermally conditioned fibres were subject to different treatments. The optimisation of the conditions of these chemical treatments has been described elsewhere and is not the subject of this report [22,23]. To regenerate the fibre strength the thermally conditioned fibres were immersed in a 3 M sodium hydroxide solution (NaOH) for 10 min at 90 °C. These fibres were then drained and rinsed in hydrochloric acid (HCl) to neutralise any remaining NaOH and assist in the removal of any water glass deposits. The SEM micrograph in Fig. 2a shows a typical example of these deposits on an NaOH treated heat conditioned fibre. These deposits are commonly observed after concentrated NaOH interacts with E-glass fibre surfaces [18]. Finally the fibres were rinsed three times in demineralised water and dried at 105 °C for approximately 6 h. A 1 vol% APS solution was used to regenerate the surface functionality of the heat treated and NaOH treated fibres. Deionised water was used to prepare the APS solution. Before the fibre treatment the solution was allowed to hydrolyse for 24 h at room temperature. The fibres were immersed in the APS solution for 15 min. Samples for fibre testing were dried, however samples for Glass Mat Thermoplastic (GMT) production were processed directly into the composites. This was because it was found that the APS treated fibres were more difficult to disperse if they were dried. Fig. 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

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