RECYCLED GLASS FIBRE/POLYESTER COMPOSITES – PROCESSING AND MECHANICAL CHARACTERIZATION

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ABSTRACT

The present article demonstrates the recycling and reuse of waste glass fibre fabrics. The recycled regenerated long glass fibres are impregnated with polyester resin to study the mechanical performance under tensile loading conditions. The specimens are prepared from the laminates made by using vacuum assisted resin infusion technique. Therefore high quality specimens are chosen for this study. Several processing issues were identified and also evaluated the quality of specimen before the test and evaluated fracture surfaces of the tested sample to predict the failure modes. The research study includes the development of silane treatments for the recycled glass fibres to regenerate the glass fibre surface. Experiments are conducted for the reference laminate (CSM as received), Heat Treated glass fibres without any sizings on surface, and silane treated glass fibres with heat treatment. Scanned electron microscopy demonstrated the differences in the fibre surface patterns. The results demonstrate the comparison of tensile performances both at single fibre and composite levels.

1 INTRODUCTION

Glass fibre reinforced polymers (GFRP) are increasingly being used in engineering structures due to their light weight, tailor made properties, corrosion resistance, and cost effective. For large structures like wind turbine blades, boat hulls, aircraft structures consumption of fibre reinforcements are huge generally in terms of tonnes. FRP industry produces several thousand tonnes of products every year increasing the use of polymer and fibre reinforcements. After the life cycle of any FRP structures, one needs to have technologies to implement waste management (in order of preference – waste minimisation, reuse, recycling, incineration with energy recovery) and reuse the material and develop some useful market using recycled fibres/polymers for the new product design and developments.

Several researchers [1-2] investigated the reuse of plastics (thermoplastics). Thermoplastic polymers are inexpensive, lightweight, and durable and can be easily moulded into a variety of products that find use in a wide range of applications. Whereas polymers like thermosets cannot be recycled, but researchers investigated methods to separate fibre reinforcements from thermoset composites [3-5] after its use. Fluidized bed process is one technique to separate the glass fibres recycled from scrap composites [6]. Another technique for recycling of GFRP composites is by mechanical methods, i.e. to grind the composite to be recycled and to use the recyclate that is obtained as filler [7]. In the present study, pyrolysis technique was implemented to separate glass fibres from GFRP waste. Recovery of glass fibre from waste composites was carried out at 450–600°C.

In the present study, we have demonstrated the recycling and reuse of waste glass fibre fabrics. The recycled glass fibres are regenerated by applying suitable sizing chemicals, where long recycled fibres are impregnated with polyester resin to study the mechanical performance under tensile loading conditions. The specimens are prepared from the laminates made by using vacuum assisted resin infusion technique. Experiments are conducted both at single fibre level and laminate level. The properties are evaluated for the three cases i.e., the reference laminate (CSM as received), heat treated glass fibres without any sizings on surface, and silane treated glass fibres reinforced with polyester resin. Scanned electron microscopy demonstrated the differences in the fibre surface patterns. The results demonstrate the comparison of tensile performances of recycled and non-recycled glass fibre laminates.

2 EXPERIMENTAL

2.1 Materials

CRYSTIC 701 PAX polyester infusion resin and its associated catalyst (methyl ethyl ketone peroxide) were acquired from East Coast Fibreglass Supplier. The chopped strand mat 92 was provided by PPG industries with average fibre diameter of 13µm.

2.2 Pyrolysis

The recycling was made through burn-off process, in a Carbolite Furnace. A sample was placed in the centre of an aluminium tray and then put into the furnace, at room temperature (25°C). The furnace heats up to 500°C and stays at this temperature until the end of the experiment. The sample stays inside the furnace until the entire polymer is burned-off and only clean fibres remain, this time may vary according to the sample conditions. Glass fibres from pyrolysis are observed under SEM, see Figure 1.

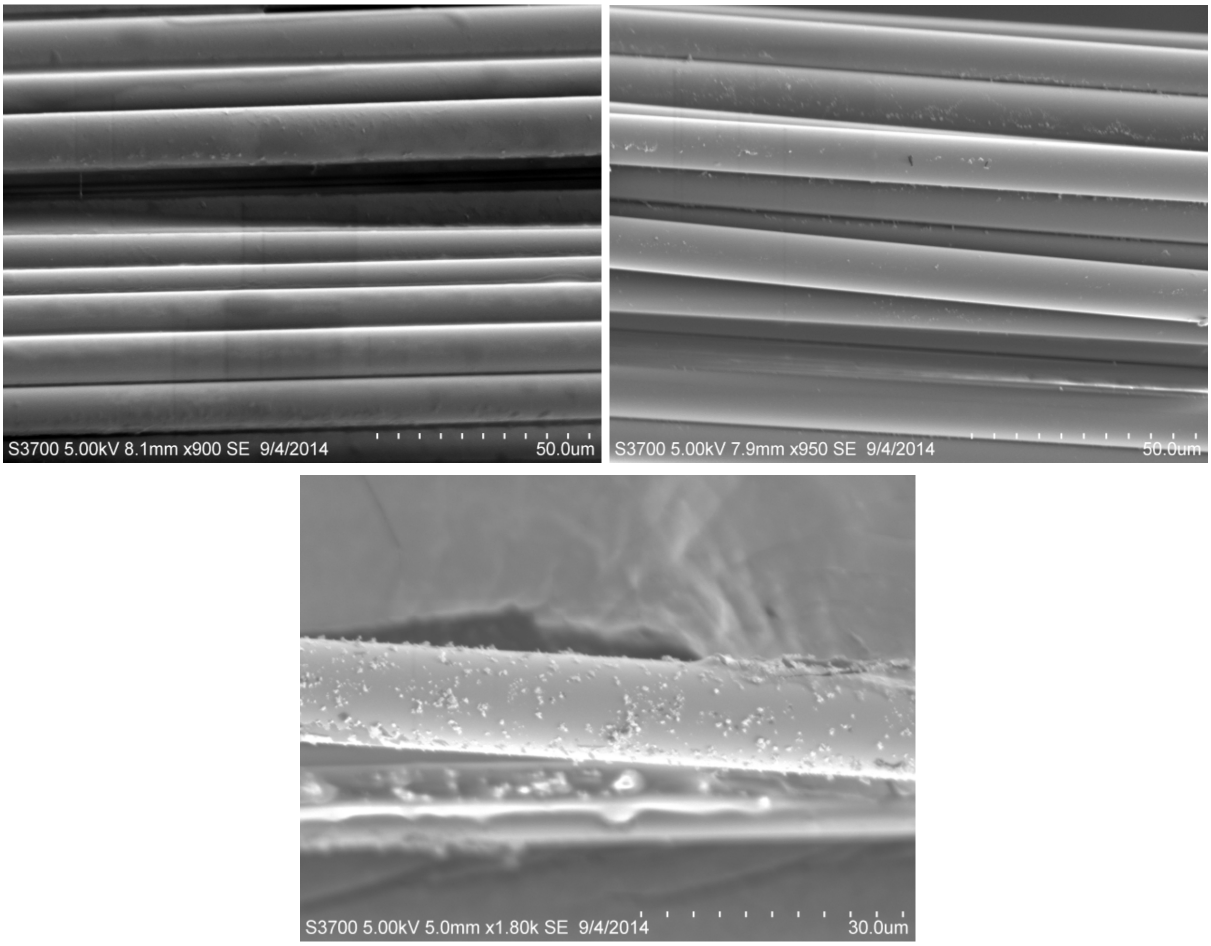

Figure 1. Scanned Electron Micrographs (SEM) – Glass Fibres as Received (CSM), Heat Treated, Silane Treated Fibres

2.3 Preparation of Composites

For experimental characterization laminates are made by the Vacuum Infusion Process (VIP), where the processing technique uses vacuum pressure to drive resin into a glass fabric layup. Three laminates are made by standard polyester resin and glass fabrics (CSM as received), heat treated glass fibres without any sizings on surface, and silane treated glass fibres. The glass fibres used are standard chopped strand mat (CSM) have E-glass fibres randomly oriented with a length 80mm. The infusion setup shown in the Figure 2 is used for the laminate trials. Laminates are made at room temperature under vacuum, where polyester cure under vacuum bag for a 24 hours and later the laminate is post cured in oven for a period of 4 hours around 80°C.

Figure 2. Vacuum Infusion Setup – Recycled Glass Fibre/Polyester Resin Laminates

2.4 Characterization

The experimental study includes characterization of single glass fibres, recycled glass fibres, recycled and regenerated glass fibres, and composite laminates. The tests for characterization of materials are single fibre tensile tests (SFTT), single fibre/polyester interface tests (SFIT), and composite laminate tensile tests.

Single fibre tensile tests (SFTT) were chosen to evaluate the fibre properties [8]. The fibre strength was determined by using the single fibre tensile test based on ASTM D3379-75. Individual fibres were glued onto card tabs with a central cutout that matched the gauge length chosen for the test. Then the tab ends were gripped by the universal testing machine (Instron® Model 3342) with a 10N load cell. Tests were performed with a gauge length of 20mm and strain rate of 1.5%. All samples were photographed with a microscope lens of 500x, and then the diameter was obtained using ImageJ software with a scale of 113.5 pixel/µm. The mechanical performance of single fibres taken from CSM of well-defined sizing and binder and heat treated E-glass fibre samples, and heat treated and silane sized glass fibre were investigated at room temperature after thermal conditioning at temperatures up to 500°C.

Single fibre/polyester interface tests (SFIT) are performed using the microbond test. Specially designed fixture [9] with two movable knife edges controlled by a pair of micrometer heads with resolution to 1µm. The microbond tests were conducted with a free distance between fibre and knife edge of 20µm. A stereo-microscope was utilised to aid the positioning of knife edges and monitor the testing process. The same testing machine used in the single fibre tensile test with 10N load cell was employed to carry out the test with the rate of fibre end displacement set to 0.1mm/min. The fibre with bonded resin droplets was mounted in the machine. Some card frame was left taped to the bottom of the fibre to keep it under tension (~0.5mN). The fibre was pulled out of the droplet while the droplet was constrained by the knife edges. Where the load-extension from the sample is recorded and noted the peak load to estimate the Interfacial Shear Strength (IFSS).

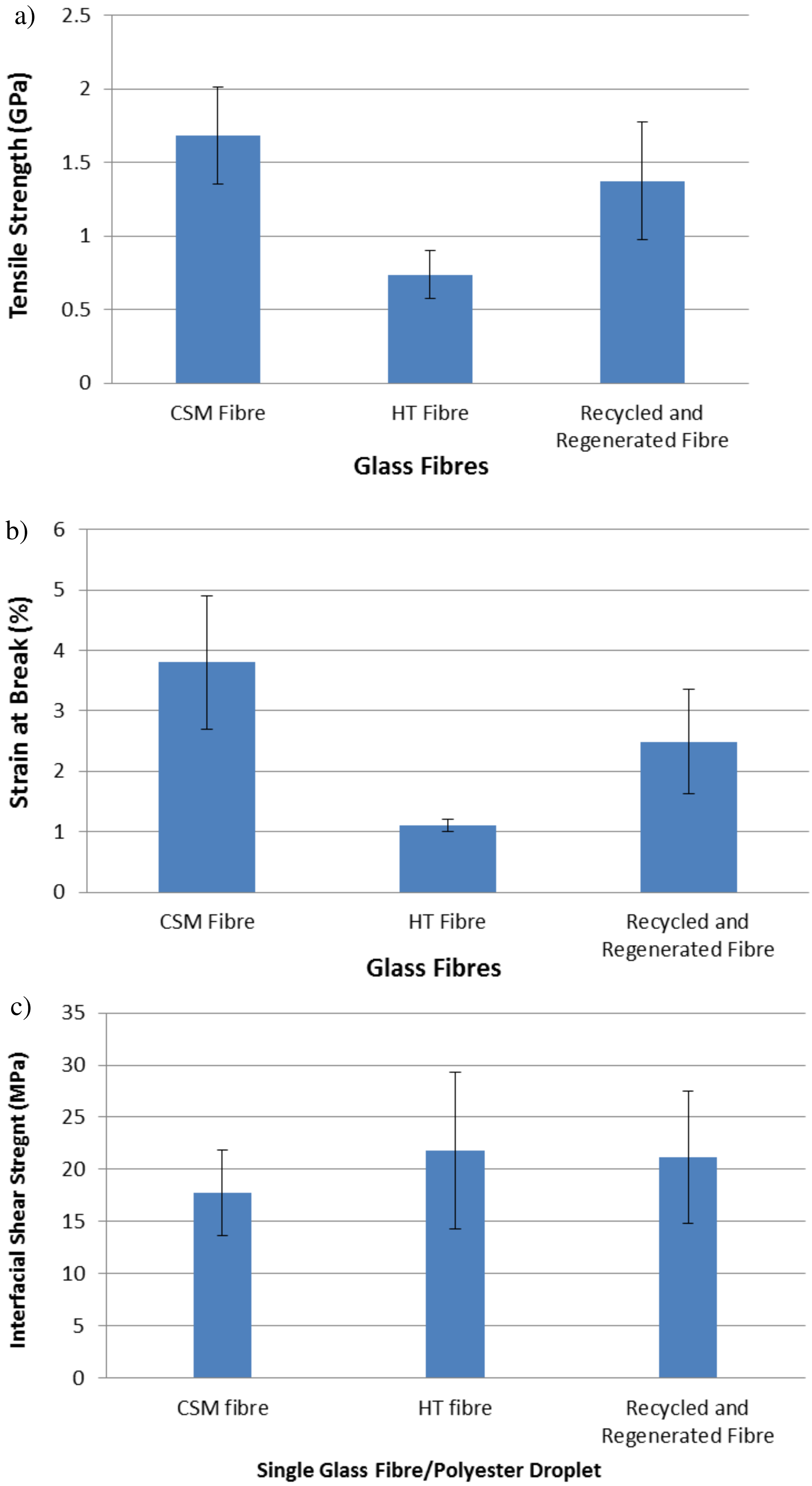

Figure 3. Single Glass Fibre Tensile and Interface Properties With Polyester Resin

Composite laminate tensile tests was carried out on a 100 kN servo-hydraulic Instron universal testing machine. The test sample size was 150mm x 15mm x 4 mm. The specimen geometry is as defined in standards, the samples were considered as dog-bone shape as per ISO standards. Machining composites is a challenging task specially dog-one shape, water-jet cutting is the best possible method to shape the specimen dimensions as per standards. All the samples were tested in tensile mode, the crosshead was programmed to apply the load at a constant displacement rate of 1.0 mm/min through the entire duration of the test. The tests were carried out under ambient conditions and the tensile properties measured in accordance with the procedures of standard ISO 527-5 [10]. The strain measurements were made with video extensometers of 60 mm gauge length.

3 RESULTS AND DISCUSSIONS

3.1 The recycled and regenerated fibres strength

The results for the average single fibre strength (at 20mm testing gauge length) of CSM fibre with standard sizings, CSM fibres after heat treatment, and heat treated fibres and regenerated with silane sized are shown in Figure 3. The results indicate that thermal conditioning can cause a considerable strength reduction for fibre samples after heat treatment, with a loss of over half of the original strength in the case of 30 minutes at 500°C. It can be seen that heat treated glass fibre types reduce in strength, with the silane sized glass falling by a greater percentage of its original strength. In general glass fibres can be extracted after thermal conditioning 500°C and above. Above this threshold temperature the average fibre strength is seen to decrease rapidly. Comparison with the results in Figure 3a indicates that the single fibre tensile strength results of recycled and regenerated/sized fibres recover its strength around 82% compared to fibre strengths of CSM glass fibres. The CSM fibres and fibres taken after heat treatment or during sizing treatments are having greater potential for damaging due to bundle-bundle interactions while separating from the fabrics (also damaging due to fibre-fibre interactions within the fibre bundles) taken from CSM (fibres separating from bundles are extremely difficult). In some cases the fibres after separating from the fibre bundles seems extremely difficult to handle or fixing single fibres on card frame which can also damage the fibres. The results shown in Figure 3b, indicates recycled and regenerated fibres can recover strain-to-failure around 65% compared to CSM fibres which is a good sign of tensile property improvement. Whereas the modulus measured for the three fibres show different trend. Fibres taken from CSM gave 60GPa, whereas for the heat treated fibres the modulus is bit higher than the modulus of CSM (63GPa). The recycled and regenerated fibres shown better performance and the modulus recorded is 68GPa. Therefore single fibre testing indicate that the strength of glass fibres is strongly influence by thermal conditioning at temperatures and times which may commonly be experience in the processing of such fibres in engineering composite materials.

3.2 Interface strength

Interface strength for single glass fibre/polyester can been determined by using fibre pull-out and microbond methods. In the current study, microbond test is chosen to evaluate the interfacial properties. The apparent IFSS is an adequate quantitative parameter which can characterise the mechanism of interfacial failure in any fibre reinforced polymer composites. Sample preparation is one difficult task to use single fibres from CSM fabrics (as discussed previously). To replicate the composite processing technique, microbond test specimens are prepared exactly similar to vacuum infusion technique used for laminate trials (i.e. droplets are formed under vacuum for 24 hours and then dried over an oven at 80°C for 4 hours). The fibres are taken from before the fabrics placed in the vacuum infusion setup for the microbond trials. The average fibre tensile strength of CSM fabrics is 1.69±0.33GPa at 20 mm gauge length was obtained by the single fibre tensile test. The tests are conducted for 30 specimens each, and the average interfacial shear strength for each case is shown in

Figure 3c. Fibres taken from CSM and recycled/regenerated fibres were able to perform good interfacial tests i.e. shown constant interfacial friction after debonding. Whereas the heat treated fibres, as the fibre surface is not coated and the surface is damaged due to thermal conditioning to 500°C, where the droplets formed on the fibre surface could not show good bond strength with polyester and debonding similar to previous case. This indicates the specimens fail before droplet able to initiate debond and also observed specimen to encounter fibre breakage rather than fibre pull-out from the polyester droplet. The results shown in Figure 3c demonstrate the recycled and regenerated fibres show good interface strength compared to heat treated and as received (CSM fibres) case. In general the reference case (as received CSM) have sizing on glass fibre surface and binders to keep the fibre bundles to form a fabric structure. Therefore the IFSS values recorded in the microbond test for reference case shown less compared to other cases. The IFSS values recorded are in the range of 18-22 MPa, the values agrees well with the IFSS-range published in the literature for GF-thermoset polymer interface (evaluated by the single fibre pull-out technique).

3.3 Composites properties

In the current experimental study, vacuum infusion technique chosen to develop composites with CSM, heat treated CSM (at 500°C, 30min), and heat treated CSM (at 500°C, 30min) with a silane treatment. Standard polyester resin was used to infuse and develop composite plates of roughly 65% weight percent glass fibres. All three laminates contain a layup of 16 glass fabric layers of dimension 160mm x 230mm to process a 4 mm thick laminate. Laminates were machined by the waterjet technique, where specimens are dog-bone shaped as per ISO standards [10] later used to evaluate the tensile properties.

Figure 4 shows the tensile properties and comparison between the three laminates data. The stiffness for the three composites was in the range of 11.5 – 13.5GPa. Composites with recycled and regenerated fibres show good recovery of stiffness i.e. 85% of composite with CSM (as received). In general the heat treated fibres demonstrate higher modulus compared to standard case [8-9], which is repeated even in the current study. For most industrial products, composite strength is most important parameter for product design. Tensile strength for the laminates developed are shown in Figure 4b, where the test data shown are in the decreasing trend from the reference case to heat treated to recycled/regenerated ones. Strength recovery is only 48% compared to as received (CSM fibre/polyester case). Similar trend is observed for the tensile strain-to-failure as shown in Figure 4c.

CSM glass fibre/polyester strength values recorded from experiments are 196±15MPa, the values agree well with the values published in the literature. The tensile strength and IFSS at single fibre level showed much better recovery, whereas the strength recovery at laminate level is not an acceptable value. Therefore the experimental study demonstrates some more research efforts need to transfer the single fibre properties (recycled/regenerated fibre) to laminate level.

4 CONCLUSIONS

The article presents single fibre tensile properties and single fibre/polyester interface properties. Experimental data also include the composite properties tested under tensile loading. The results of single fibre testing presented here clearly show that the thermal conditioning (heat treatment at 500°C) likely cause damage to glass fibres and can potentially show decrease in the fibre properties compared to the room temperature fibre strength (i.e. CSM glass fibres). Similarly to tensile properties, single fibre/polyester interface strength for recycled and regenerated fibres shows full recovery of IFSS with silane treatment. Whereas strength recovery for a composite is not that easy with glass fibres (of 80mm length) reinforced polyester resin. Comparing the tensile properties, demonstrate composites with recycled and regenerated fibres show only 45% recovery to the standard case (i.e. as received CSM glass fibre/polyester laminate). This explains transferring fibre properties to composites level requires proper understanding and some additional research. Future work will focus on full recovery of composite properties from recycled glass fibres.

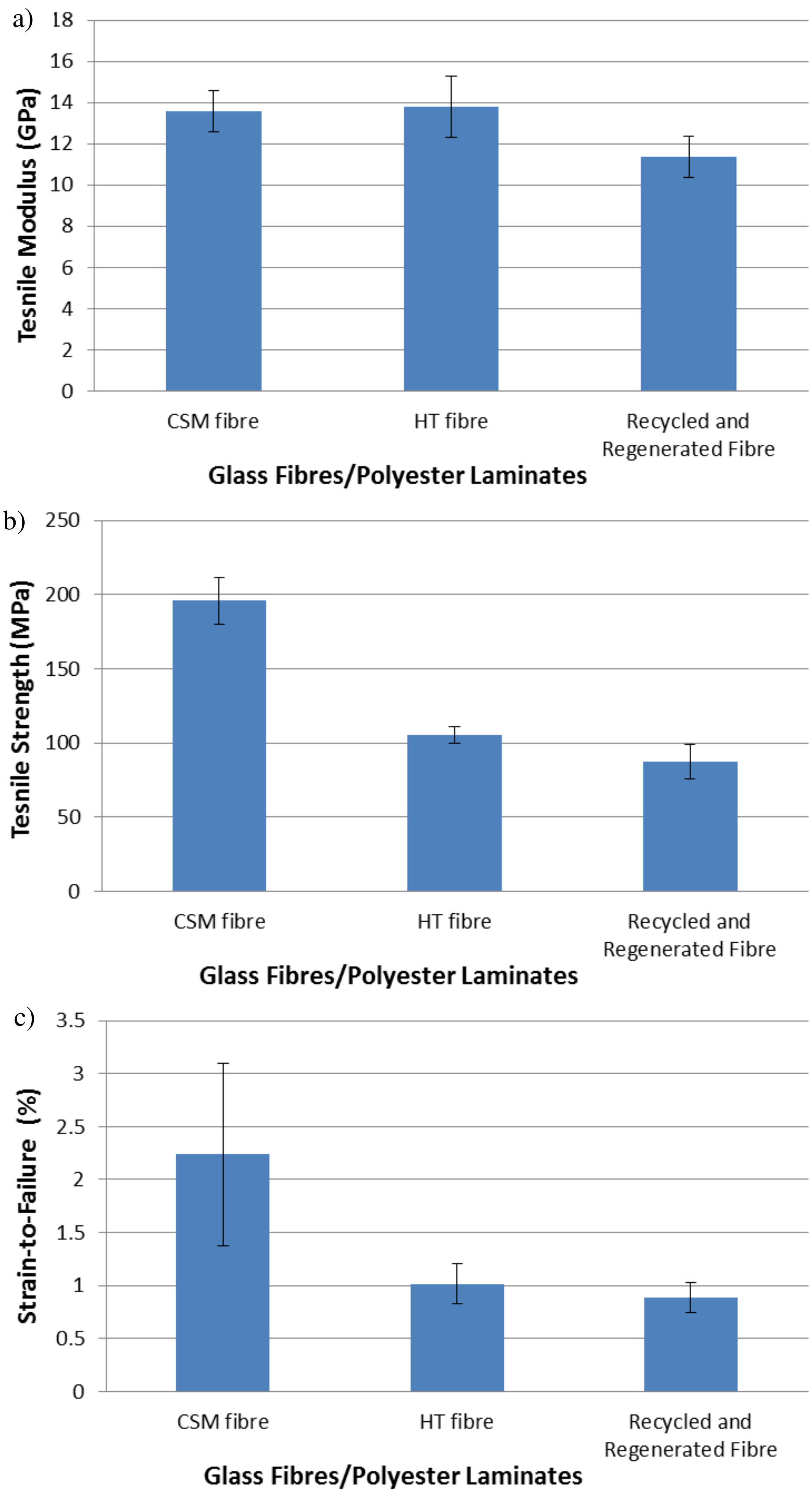

Figure 4. Summary of Tensile Properties - CSM Glass Fibre/Polyester Composites (As received, Recycled, and Regenerated glass fibres)

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