World Academy of Science, Engineering and Technology
International Journal of Electrical and Computer Engineering
Vol:15, No:10, 2021

Separation of Composites for Recycling: Measurement of Electrostatic Charge of Carbon and Glass Fiber Particles

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Open Science Index, Electrical and Computer Engineering Vol:15, No:10, 2021 publications.waset.org/10012281.pdf

Abstract**—**Composite waste from manufacturing can consist of different fiber materials, including blends of different fiber. Commercially, the recycling of composite waste is currently limited to carbon fiber waste and recycling glass fiber waste is currently not economically viable due to the low cost of virgin glass fiber and the reduced mechanical properties of the recovered fibers. For this reason, the recycling of hybrid fiber materials, where carbon fiber is blended with glass fibers, cannot be processed economically. Therefore, a separation method is required to remove the glass fiber materials during the recycling process. An electrostatic separation method is chosen for this work because of the significant difference between carbon and glass fiber electrical properties. In this study, an experimental rig has been developed to measure the electrostatic charge achievable as the materials are passed through a tube. A range of particle lengths (80-100 µm, 6 mm and 12 mm), surface state conditions (0%SA, 2%SA and 6%SA), and several tube wall materials have been studied. A polytetrafluoroethylene (PTFE) tube and recycled fiber without sizing agent were identified as the most suitable parameters for the electrical separation method. It was also found that shorter fiber lengths helped to encourage particle flow and attain higher charge values. These findings can be used to develop a separation process to enable the cost-effective recycling of hybrid fiber composite waste.

Keywords**—**Electrostatic charging, hybrid fiber composite, recycling, short fiber composites.

I. INTRODUCTION

RECYCLING is essential to make a sustainable environment [1]. In recent years, European legislation has set out goals for improving waste management, promoting creative thinking in recycling, reducing landfill use and providing incentives to alter consumer behavior [2]. The waste hierarchy steps of the European Union's waste management system is prevention, reuse, recycling, and recovery, with disposal (including landfill and incineration) as the least preferred choice. [3], [4].

Composite waste has emerged as a serious environmental issue that has attracted widespread attention in recent years as increased waste has arisen. A Composites UK FRP Circular Economy report indicated that the generation of composite production waste is around 6,200 tons of glass fiber reinforced plastic (GFRP) and 1,600 tons of carbon fiber reinforced plastic (CFRP) in the UK each year [3]. Recycled glass fiber (rGF) has low economic value because of cheap virgin materials, and it loses its mechanical properties significantly during the thermal-chemical recycling processes. As a result of the low-cost industrial margin, several countries do not recycle glass fiber composite waste [4]. Hence, the reuse opportunities for rGF are limited, and it is currently used only as a raw material for cement production [5], [6].

The volume of available CFRP composite waste is low compared with GFRP, but it is more attractive to commercial recycling due to its higher economic value and higher mechanical properties [5]. Some of the commercial recycling operations and their capacity are ELG Carbon Fiber Ltd. in the United Kingdom - 2000 tons/year, CFK Valley Stade Recycling Gmbh in Germany - 1500 tons/year, Karboreck RCF in Italy - 1500 tons/year and Carbon Conversions in the United States - 2000 tons/year [6], [7]. For reuse opportunities, the recycled carbon fiber product is classified by length; milled fiber (80-100 µm), short fiber length (3 mm to 9 mm) and long fiber length (60 mm to 120 mm). Long fiber is currently used to make non-woven mats, which can be processed by compression molding. Milled and short fibers can be formed into pellets to use in injection molding applications. Although carbon fiber production is expanding year after to fulfil material demand, fiber production is still insufficient [8]. In 2020, the carbon fiber demand forecast is 150,200 metric tons, but the actual supply is 129,965 metric tons [4], [5]. Therefore, recycled carbon fiber could potentially fill the gap between the demand and supply.

The benefit of recycling carbon fiber is bringing down the material price due to the saving in energy relative to new fiber manufacture whilst making a reused product. For example, a kilogram of recycled carbon fiber is priced at around US$ 13-19 compared to US$ 33-66 for virgin carbon fiber [7], [9]. Pyrolysis is a commonly used thermal recycling method at an industrial scale; such as in ELG Carbon Fiber Ltd., UK [10], [11]. All types of carbon fiber composite waste can be recycled, i.e., bobbins, prepreg, and laminates. But hybrid materials in which glass fiber is mixed with carbon fiber composite cannot be processed [6]-[12]. Even though the volume of hybrid fiber composite waste is currently low, it is likely to increase in near the future due to the high demand from automotive and

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transportation, wind energy and sports and recreational sectors. It is expected that hybrid fabric market will rise from US$197 million in 2019 to US$ 415 million by 2024, at a CAGR of 16.0% during the forecast period [3]. The manufacturing waste generated from the production process (cutting and trimming) may account for up to 40% of material supplied. Disposing waste to landfill is undesirable; hence, a separation method is needed to remove the glass fiber via an efficient recovery process to enable recovery of the more valuable carbon fiber from hybrid fiber composite waste.

A separation method based on the electrical properties of material has been chosen due to carbon and glass fibers behaving differently on account of their different conductive properties. The principle is that each particle acquires an electrostatic charge and the particles are sorted through the application of an external electric field. The charging of particles can be achieved by contact electrification (the triboelectric effect), conductive induction, and ion bombardment [13]. The efficiency of the separation process primarily depends upon the charge obtained on the particles [14].

Electrostatic charge can be obtained when two different materials are brought into contact by rubbing against each other. The material with a higher affinity for electrons absorbs them and thus charges negatively, while the material with a lower affinity tends to become positively charged [13]-[15]. The amount of charge transferred between the particles can be expressed as a function of parameters such as particle shape, length ratio, sliding area and velocity, plane angle and relative humidity [16]. Li et al. used a two-stage separation process to recycle the end-of-life passenger vehicle part (PP, PU and PVC). In the first stage separation, PVC components and a combination of PP/PU mixtures were recovered. The recovery of PVC was 41% with a purity of 97%, and the recovery of the PP/PU mixture was 73% with a purity of 99%. The individual components of PP and PU were recovered in a second stage separation process. The recoverability of PP and PU was 74% and 94%, respectively, with a purity of 95% and 99%, respectively [17].

The aim of this research work is to measure the electrostatic charge of the carbon/glass fiber material while varying parameters such as surface condition, particle length and interaction with wall materials. The experiment carried out in this study measured the electrostatic charge with three levels of parameter variation, as indicated in Table I.

TABLE I
IDENTIFIED PARAMETERS AND THEIR VARIATION LEVEL

Parameters	Surface state condition	Tube wall material	Particle length
1	No Sizing Agent (0%SA)	Glass	80-100 µm
2	Sizing Agent as supplied (2%SA)	Steel	6 mm
3	Sizing Agent as modified (6%SA)	PTFE	9 mm

The tube materials were chosen from the triboelectric series shown in Fig. 1. The triboelectric series is a ranking of various materials based on their ability to gain or lose electrons. Generally, the list of materials in the order reference as the top is more likely to lose electrons, the bottom is more likely to gain electrons and the middle is not likely to do either. We have chosen the three selected materials (glass, steel and PTFE) to determine the unknown electrostatic charge for both carbon and glass fiber particles. The selected three materials are based on having the highest tendency to lose and gain electrons. A combination of a Faraday Cup and electrometer (charge sensor) is the most common method to measure the electrostatic charge of a material. The V-tube geometry was chosen to combine the different mechanisms leading to triboelectrification: (i) friction between the fibers, (ii) friction between the fibers and the wall, and (iii) fiber impact on the wall at the contact between the two tubes [4]-[18].

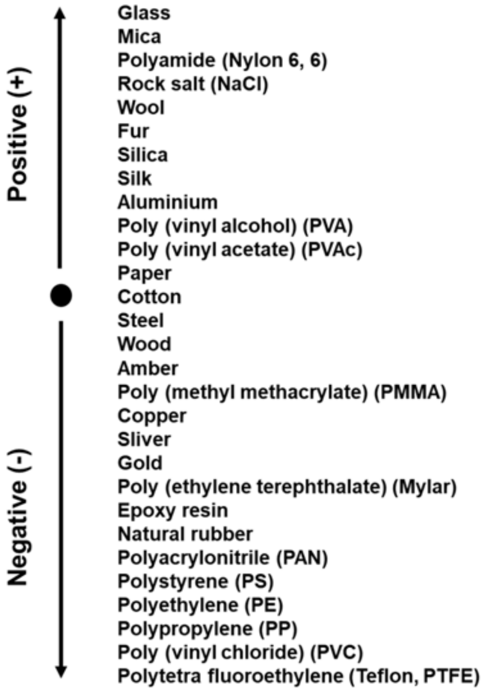

Fig. 1 Triboelectric series of common materials [19]

II. MATERIALS AND METHODS

A. Material Preparation Process

Short fiber products can be created by two methods: milling and chopping. Milled fibers are produced using a hammer mill which results in relatively broad length distribution with consistent output and an average length of 80 µm to 100 µm. Chopped fibers are produced by chopping a fiber tow into precise lengths. The length of chopped fibers is significantly greater than that of milled fibers. To produce the desired length of 6 mm and 12 mm chopped fiber tows, the E-glass 2400 Tex and 24 K carbon fiber tow rovings were used in this study. East Coast Fiberglass supplied the glass fiber products (milled fiber and fiber tow) for this analysis, whereas ELG Carbon Fiber Ltd. provided the carbon fiber products. A coating of polyurethane material was applied to understand the effect of a sizing agent on the particles on the electrostatic charge level. The process for producing chopped fiber tow particles is illustrated in the Fig. 2.

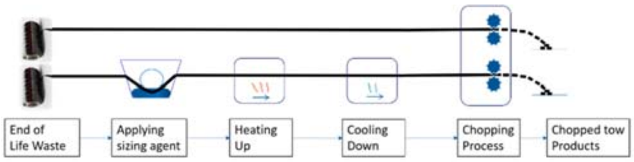

Fig. 2 Chopping fiber tow process @ELG

B. Burn off Method

The burn-off test was used to measure how much sizing agent was present on the particles' outer surface. The procedure was carried out according to the ASTM D3171-99 standard test method. An electric muffle furnace with a heating limit of 1100 °C was used to remove the sizing agent present in the outer surface of chopped fiber tows. The sizing agent was removed using a 500 °C heating temperature and a 20-minute heating period. For each test, the mass sample weight before and after the burn-off test was recorded. The experiment was run five times, with the average value given in Tables II and III.

TABLE II
RESULT FROM BURN OFF TEST FOR CF CHOPPED FIBER TOW 6 MM - SUPPLIED MATERIAL

S.No	Crucible weight (g)	Sample mass + Crucible weight before burn off (g)	Sample mass + Crucible weight after burn off (g)	Sample mass difference (g)	Sizing content (%)
1	43.958	48.658	48.576	0.082	1.617
2	44.456	48.622	48.517	0.105	2.514
3	44.509	48.645	48.562	0.076	2.007
4	43.349	47.549	47.491	0.078	1.848
5	42.939	46.984	46.874	0.109	2.695

TABLE III
RESULT FROM BURN OFF TEST FOR GF CHOPPED FIBER TOW 6 MM- SUPPLIED MATERIAL

S.No	Crucible weight (g)	Sample mass + Crucible weight before burn off (g)	Sample mass + Crucible weight after burn off (g)	Sample mass difference (g)	Sizing content (%)
1	47.631	48.724	48.667	0.097	1.398
2	44.182	48.319	48.274	0.045	1.087
3	43.928	47.986	47.931	0.055	1.335
4	44.270	48.212	48.142	0.070	1.776
5	42.942	46.916	46.848	0.068	1.771

C. Fiber Length Measurement

An automatic fiber shape system supplied by IST AG was used to measure the fiber length distribution. The system consists of a slide scanner equipped with a fully automatic image analysis tool to measure the fiber length in a range from 2 µm to 30 cm. The fibers were loaded and distributed into a tray filled with clean water. The image is taken from the scanner and analyzed by quantitative measurement with the XShape Fiver pro software. The measured lengths of the fibers are as described in Fig. 3.

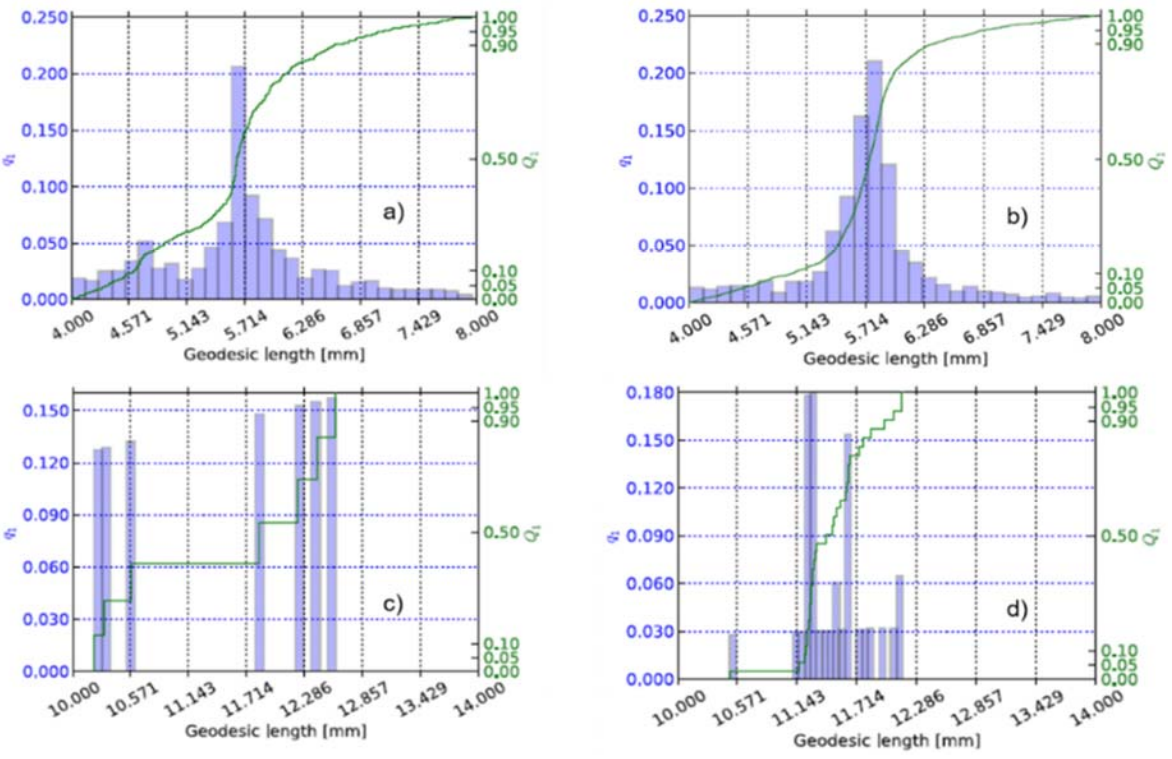

Fig. 3 (a) Chopped CF fiber length – 6 mm; (b) Chopped GF fiber length – 6 mm; (c) Chopped CF fiber length – 12 mm; and (d) Chopped GF fiber length -12 mm

D. Electrostatic Charge Rig Setup

The experiment rig has been developed to determine the electrostatic charge produced in a granular material during flow in contact with selected materials. The sample was poured into a V-shaped tube manually. The V-shaped tube was formed with an angle of 90° by connecting the two tubes of length L = 500 mm and internal diameter D = 50 mm.

At the end of the flow, the sample material was collected in a Faraday cup. The Faraday cup was connected to a charge sensor. Einstein™World, Israel supplied the electrostatic charge sensor. The final charge obtained in the particle was calculated at the end of the flow, where m is the sample mass poured inside the tube and Q is the electrostatic charge obtained due to the triboelectric effect [20].

E. Design of Experiment Test

In this study, a general fractional factorial design was chosen for the design of experiment to minimize the number of tests. This resulted in a total of 135 runs for five replicates of each parameter. All selected parameters were set for three levels, except the particle length of 100 µm which has only one surface condition. The surface of the material, i.e., amount of sizing agent presented in the particle, varied from (0.1% to 6%). The amount of sizing agent (SA) presented in the material as supplied was 1% and modified with 6% SA applied. An additional test was performed in the burn off to achieve 0% of SA. The tube materials chosen were glass, stainless steel and PTFE. The length of the fibers was 80-100 µm, 6 mm and 12 mm.

III. RESULT

A. Electrostatic Charge Analysis

It is well known that humidity and room temperature can affect electrostatic charge measurement [18]-[20]. Therefore, the measurements were performed at controlled conditions with a relative air humidity of 60 ± 1% at a room temperature 18.5 ± 0.5 °C. The sample volume used for each experiment test was 15 g, and the sample was not reused after the measurement. At first, the sample initial charge value was recorded by introducing it into a Faraday cup. After the initial charge value recorded, the sample was poured into the selected tube to charge the particles and the sample was collected the cup. The final charge of the fibers was recorded as (nC/g). All values were recorded, and the mean calculated from the five repeated experiment tests. Stainless steel (316L) powder supplied by Höganäs, Sweden, was used to check the experiment rig calibration [21]. The calibration was used to show the comparison between reported electrostatic measurements and the measurements using our developed experimental rig. Table IV shows the electrostatic initial and final charge of the material.

The above equipment calibration work shows that the initial and final electrostatic charge values of the 316L stainless steel powder obtained are identical to the reported result. Therefore, it shows that our developed rig will produce the desired outcome. Several tube shape configurations were investigated to study the effect on receiving the maximum poured material into a faraday cup, and its obtained electrostatic charge value.

TABLE IV
EQUIPMENT CALIBRATION FOR ELECTROSTATIC CHARGE MEASUREMENT

Equipment	Sample mass (ml)	Initial charge value q0 (nC/g)	Final charge value qf(nC/g)	Difference in charge value Δ(qf-qo) (nC/g)	Std. dev. (%)
Reference rig [21]	50	-0.017	-0.127	-0.110	0.04
Developed rig	50	-0.027	-0.04	-0.067	0.02

The four different configurations were assessed by changing the shape position and angle of the tubes; i.e., two tubes placed as V-shaped at an angle of 45°, two tubes positioned as straight at an angle of 45°, one tube positioned as straight at an angle of 45° and one tube positioned as straight at an angle of 30°. Glass fiber, with varying three levels of particle length, was used for the assessment. The two tubes positioned as straight at 45° angle, as illustrated in Fig. 4, were selected to measure the electrostatic charge value among the four described configurations.

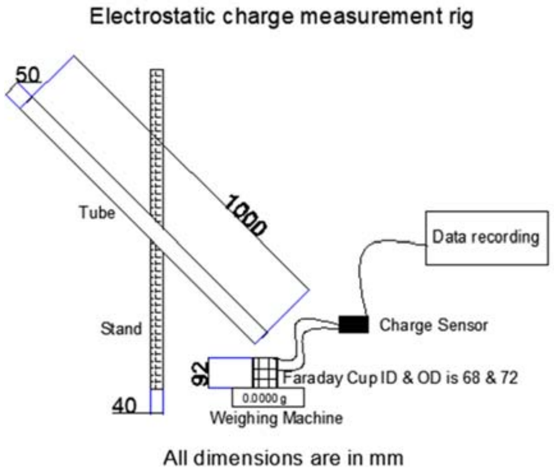

Fig. 4 Developed experimental rig for charge measurement

B. Design of Experiment Test

The DoE test was performed to investigate the influence of the critical parameters associated with the electrostatic charge value of the particle (surface state condition, fiber length and tube wall material). Based on the material preparation process used in this study, the samples were classified into two stages: virgin and recycled materials, as shown in Table I. The particle shape of glass/carbon chopped fiber tows was rectangular, and milled fibers were approximately circular. The experimental results of electrostatic charge for virgin materials are shown in Fig. 6. Generally, the initial charge value for virgin materials was CF (0.30 nC/g) and GF (0.37 nC/g). There was a minimal difference in the initial charge value for CF/GF with respect to particle length from 6 mm to 12 mm. However, the final charge value obtained differs significantly with the three tube materials. From these results, it is clear that the electrostatic charge for both CF and GF increased positively in all three tubes (PTFE, Glass and Steel). The electrostatic charge of the virgin materials is shown in Fig. 5. The steel tube gave the lowest charge compared to the PTFE and glass tubes. Interestingly, different initial charge values for recycled milled fibers of CF (-0.08 nC/g) and GF (0.38 nC/g) were observed. In contrast, with increasing fiber length, the initial charge tends to be positive due to the particle shape and size. After recycling, the recovered fibers from the tow became fluffier and stick to each other as shown in Fig. 6 (d).

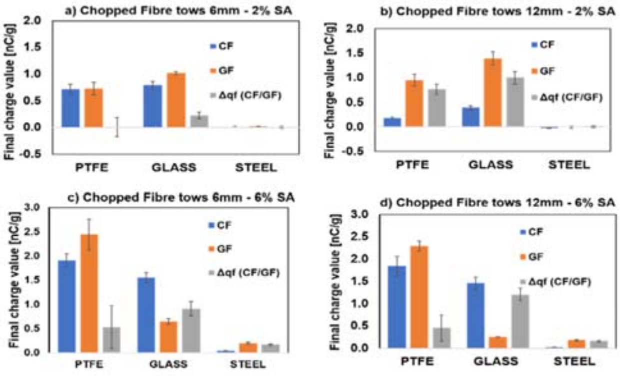

Fig. 5 Final electrostatic charge obtained for virgin materials; (a) Particle length – 6 mm, 2%SA; (b) Particle length - 12 mm, 2%SA; (c) Particle length - 6 mm, 6%SA; and (d) Particle length - 12 mm, 6%SA

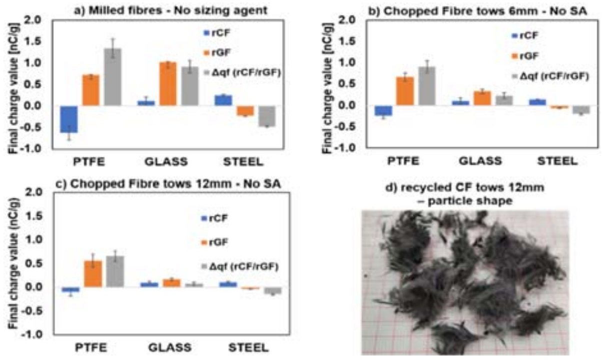

Fig. 6 Final electrostatic charge obtained for recycled materials; (a) Particle length – 100 µm, 0%SA; (b) Particle length - 6 mm, 0%SA; (c) Particle length – 12 mm, 0%SA; and (d) Particle shape for CF tows – 12 mm

For the recycled materials the carbon fibers charged negatively in the PTFE tube and positively in both glass and steel tubes. Glass fibers charged positively in both PTFE and glass tubes and charged negatively in the steel tube. Together, the findings confirm that the PTFE tube leads to a higher opposite sign polarity charge between the recycled carbon and glass fiber particles, followed by the steel tube.

IV. DISCUSSION

In this section, the effect of the electrostatic charge obtained by varying the SA and the length of the fiber particles will be discussed.

Fig. 7 (a) shows the electrostatic charge obtained for carbon and glass fibers in a PTFE tube with increasing the amount of sizing on the fiber surface. The result shows that increasing the sizing content for both carbon and glass fibers tends to give a more positive. Therefore, sizing on the fiber surface is more influential than the fiber core material. It is therefore concluded that electrostatic separation is more easily achievable after a thermal fiber recycling process where the SA and any other surface coating has been removed.

Fig. 7 (b) demonstrates the electrostatic charge obtained from the experimental test with varying recycled fiber lengths. In order to attain a consistent result, the wall material inside the tube was properly cleaned to minimize the effect of the coated materials from the previous test.

This experimental study shows that the milled fiber particle performs well and gives a higher charge value difference in the recycled surface state condition. Therefore, shorter fibers charge better than those of longer length. Overall, these results are in accordance with findings reported for a similar investigation with plastic granules [17]. However, during the experiment, it was found that longer recycled fibers were more difficult to feed within the tube as the fibers tend to be fluffy and agglomerate. However, in the case of milled fiber, the fibers flow easily inside the tube. Therefore, milled fiber is a more favorable particle length for the separation of the materials. The triboelectric separation process is the most feasible for segregating glass fiber contamination in recycling composite waste. A similar conclusion was reached with previous studies in the recycling of mixed plastic waste [22].

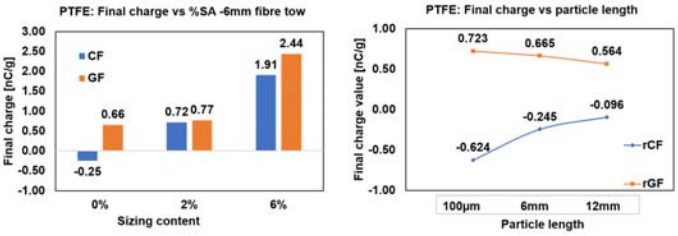

Fig. 7 Electrostatic charged value with PTFE; (a) Final charge value vs. %SA – 6 mm chopped fiber tow; (b) Final charge value vs. particle length – recycled material

V. CONCLUSION

In this research, an experiment rig was developed, and the electrostatic charges of carbon and glass fiber materials were measured during the interaction with various tube wall materials. From the results it is concluded that fibers with a recycled surface condition and the PTFE tube material gave the favorable conditions for attaining the largest electrostatic charge difference between carbon and glass fiber materials. This is an important finding in the understanding of triboelectric charge on fibrous particles. The results obtained show the potential for glass and carbon fiber separation.

Further work is now required to develop a viable fiber separation process by addressing these challenges.

- Recycled surface state condition is an essential parameter for fiber separation. To produce the controlled surface state condition of the recovered fiber requires optimizing the recycling process conditions for each composite waste specific to laminate configurations.

- Optimal parameter settings are necessary to recover the purity of the carbon fiber product and improve sorting efficiency. The optimum parameter condition is calculated using numerical modelling of particle trajectory that accounts for electrostatic, gravitational, and drag forces acting on the particles.

ACKNOWLEDGMENT

This research work was performed within the framework of the HyFiSyn project received funding from the European Union's Horizon 2020 research and innovation programme under the Marie Sklodowska-Curie grant arguments No765881.

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