

Non-Toxic Processable Solution Up-Cycling of Spent Electrical Anode: Environmental Sustainability

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Abstract:

Electric vehicles (EVs) are becoming widely used worldwide to reduce emissions of greenhouse gases from transportation. Managing the enormous quantities of waste from decommissioned lithium-ion batteries, which include rare earth and vital minerals, is becoming more problematic as this rapid increase continues. They were able to transform the graphite anodes that were wasted in an electric vehicle into graphene dispersions by using higher level shearing exfoliation in aqueous surfactants. Using used graphite anodes instead of high-purity graphite flakes increases process yield by 38.6% under the same hydrodynamic circumstances. Similar to high-purity precursors, surfactant concentration decreases average atomic layer number. This study shows that some layer graphene made from graphite flake has a better electrical conductivity, and we pinpoint the drawbacks of exfoliating wasted graphite anode material using aqueous surfactant solutions. An environmentally sustainable end-to-end upcycling of spent EV anodes into new technologies is shown by the creation of working paper-based electronic circuit boards using these non-toxic solution-processable nanomaterial dispersion.

Keywords: Electric Vehicle, Greenhouse Gas, Graphite, Atomic Layer, Electric Circuit.

1. Introduction

Reduced emissions of greenhouse gases from transportation, particularly from smaller automobiles, have made electric vehicles (EVs) the preferred choice in recent years [1]. As governments and manufacturers throughout the world have acknowledged the dangerous need to address climate change, the number of electric vehicles (EVs) on the road has increased dramatically in the last decade [2], going from almost nothing to several million. There is room for improvement in the current material recovery techniques and ways to make the whole battery production lifecycle more sustainable [3]. Since graphite anodes are considered to have a poor recovery value [4], pyrometallurgy and hydrometallurgy are limited to recovering high-value elements like lithium, cobalt, and nickel, copper, and aluminium [5-7]. Although resources are concentrated in some areas, graphite is more plentiful. It

has risen to the level of a premeditated mineral and is on the serious mineral tilts of several countries and regions due to its dependency on imports [8]. Even though it constitutes 25% of the weight of lithium-ion batteries and is on the list of important minerals, graphite is nevertheless used as fuel [9-11], as a reduction agent in metallurgical processes, and then thrown away. Graphite is in high demand for lithium-ion battery applications because to the increasing number of electric vehicles on the road [12]. To address this demand and ensure environmental and economic sustainability, it is essential to find solutions that promote a circular materials system [13].

A new area of attention in the fight against this waste is the development of efficient ways for recycling graphite anodes for use in new batteries [14-16]. This study will also investigate the possibility of upcycling graphite anodes into multipurpose graphitic nanomaterials. Nanosheets of size-selected graphene were printed by Large et al. to produce thin-film electronics and radio frequency antennas [17]. Shear exfoliated graphene nanosheets were disseminated in poly (PET) in tiny volume fractions, using melt mixing, increasing PET's strength by 45%. Graphene nanosheets may be used in 2D/2D hetero structures to enhance charge carrier separation from the parent semi-conducting photocatalyst [18], which in turn improves visible-light photocatalytic processes for water treatment.

Each layer of graphite is composed of separate graphene sheets that are held together by the forces of van der Waals [19-21]. The synthesis of graphene and similar materials from graphite has been accomplished by a number of top-down liquid exfoliation techniques, some of which are chemical while others are nonoxidizing [22]. The massive amounts of garbage produced by discarded electric vehicle batteries could be amenable to scale upcycling techniques if borrow certain tricks from the world of two-dimensional materials. The synthesis of graphite, graphene oxide (GO) [23], and reduced graphene oxide, or (rGO), has been achieved by the use of liquid-phase exfoliation methods to graphite based on anode retrieved from electronic device lithium-ion batteries [24]. The methods range from chemical exfoliation procedures to those that are aided by ultrasonication or a combination of chemical and mechanical processes. But there are still obstacles to overcome, especially with regard to the ecological friendliness of the procedures that might transform lithium-ion anodes into graphene, graphene oxide, and rGO.

In the past, treatment and pretreatment steps have often included the use of harmful solvents (like NMP) or techniques derived from other chemical processes (such modified Hummer's method). To create solution-processable graphene nanoparticles for paper-based electronic device fabrication, investigate high-shear exfoliation as a potential alternative to hazardous solvents and harsh chemicals [25]. Using graphite taken from an electric vehicle's dead battery as an anode, test this upcycling method. That way, this quickly expanding economic sector may benefit greatly from the study methodologies and findings.

2. Methodology

2.1 EV Graphite Recovery

The first generation of Nissan Leaf batteries had its used graphite anode material recycled. The disintegration of the battery down to its individual cells is seen in Figure 1. Through a cell disassembly procedure, the graphite was extracted from the copper current collectors. After being depleted to a 1% level of charge, the car battery was deemed to have reached the end of its useful life. The powdered

anode, which was black in colour, included graphite and a binder, which was believed to be less than 8 weight percent poly (vinylidene fluoride, or PVDF). By using laser diffraction, a median particle size based on volume was determined. Scanning electron microscopy has previously shown that there are many particles within the anode dark mass with sizes.

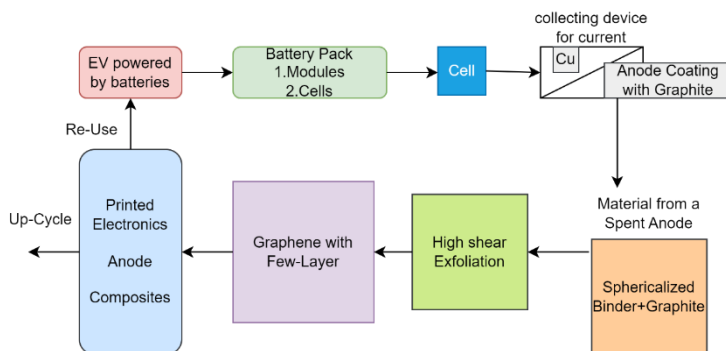


Figure 1 Disintegration of the Battery Down to its Individual Cells

2.2 Producing Graphene with Few Layers

Electric vehicle anode graphite was distributed in a surfactant solution made of deionised water along with sodium cholate. To study synthesis without pretreatment procedures that include harmful solvents like NMP to remove PVDF from the graphene, the binder was kept in the initial graphite material. The cylindrical stirred vessel measures 71 mm in diameter, 96 mm in height, and has a contoured base, all of which were used to submit the dispersion to high-shear exfoliation. The shear rates were $1.20 \times 10^6 \text{ s}^{-1}$, and the turbulent flow within the vessel was generated by a four-bladed impeller that was spun at a speed of $\bar{v} = 20,100 \pm 1600 \text{ rpm}$. The rotating speed of 105 s^{-1} was selected since it is higher than the crucial requirement needed to remove few-layer graphene from the graphite particles. After one minute of rotation, the impeller was switched off for six minutes to prevent the motor and liquid dispersion from being overheated. The vessel was kept in a freezer at $-30 \text{ }^\circ\text{C}$ and encased in ice throughout the off time. At the start of each one-minute processing period, the dispersion was maintained at room temperature in this way. Each material synthesis took a total of sixteen minutes due to the sixteen process intervals that were executed.

Finally, a similar set of experiments was run on pure graphite to see how well the shear exfoliation upcycling technique worked using used electric vehicle anode materials. Graphite flakes were selected due to their prevalence as a precursor material for few layers graphene synthesis in published works. Laser diffraction analyses showed that the flakes had a median particle size that was proportional to their volume. Preparation of materials, higher shearing exfoliation, and post-production followed identical protocols as described in the Methods section for graphite from spent electric vehicle anodes.

2.3 Analysing the Material

Following the liquid-phase exfoliation, the aqueous-surfactant solutions were transferred to 16 mL centrifugation tubes and spun at 245 g for 46 minutes. The top of the supernatant, which included just a few layers of graphene, were extracted for the purpose of conducting the experiment using UV vs. nIR spectroscopy. Averaging the graphene spectroscopic metrics and the extinction spectra of the

nanomaterial dispersions, they get the mean atomic layer number (N), which has a value equal to $25 \left(\frac{E_{550nm}}{E_{max}} \right) - 4.2$.

Filtering the dispersions using PTFE filtration with a minute opening size of 222 nm allowed us to calculate the few-layer graphite concentration (C_{gr}). The filters had a 26-mm diameter. The extinction coefficients, $\epsilon(\lambda)$, were calculated by measuring some layer mass graphene maintained on the filters. The extinction coefficient at $\lambda = 662$ nm is unaffected by the size and thickness of the nanosheet. This wavelength was selected for the Lambert-Beer relationship, which measures the density of well dispersed graphene nanoparticles as $C_{gr} = \left(\frac{E_{662nm}}{\epsilon_{662nmL}} \right) = E_{662} \text{ nm} / \epsilon_{662} \text{ nmL}$, where cuvette optical path length is L. The material constructed from the graphite flake predecessor had an extinction coefficient of $1522 \text{ L g}^{-1} \text{ m}^{-1}$, whereas the substantial made with the discarded anode precursor with EV had an extinction value of 662 nm, or $1015 \text{ L g}^{-1} \text{ m}^{-1}$. Different published publications provide various assumptions of the extinction coefficient. Nevertheless, the one for fewer layer graphene that is exfoliated from high-purity graphite flakes is consistent with earlier studies that employed fluid exfoliation of the identical precursors in aqueous-surfactant solutions. Graphene made from the old EV anode might have a lesser extinction coefficient since different graphite sources are of different grade. Take graphene flakes in water as an example; recent research has shown that their optical characteristics are significantly affected by material dependencies. Even when using the same solvents and liquid exfoliation conditions, our results show that extinction coefficient measurements should be taken into account when evaluating various graphite sources.

3. Results and Discussion

3.1 Upcycling with High-Shear

Results from high-shear exfoliation in water-based surfactants were compared to those from high-purity graphite flake precursors in order to gauge the upcycling efficiency of the process. At different surfactant concentrations, the concentration of fewer layer particles of graphene is shown in Figure 2. Using the regenerated EV graphite anode as a precursor was associated with consistently higher product concentrations across the substrate for the sodium cholate content range. This was the most striking, as reductions in surfactant concentration were associated with differences as much as 10 times larger. The concentrations of few-layer graphene obtained from the discarded anode antecedent were either somewhat higher than the level of graphite flake precursor or were similar to them when surfactants were present at amounts that varied from 1 to 20 g/L. See Figure 3 for the materials and surfactant concentration-dependent optical extinction spectra. The electronic conjugation of graphene was shown by the absorption peak at $\lambda = 268$ nm, which was seen in both samples.

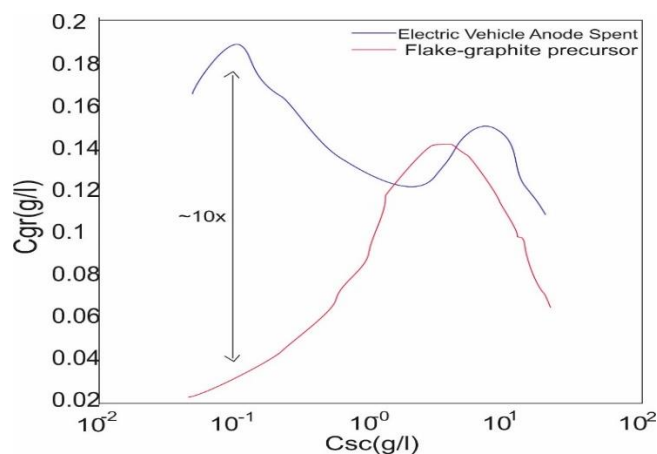


Figure 2 Concentration of Few-Layer Graphene Particles

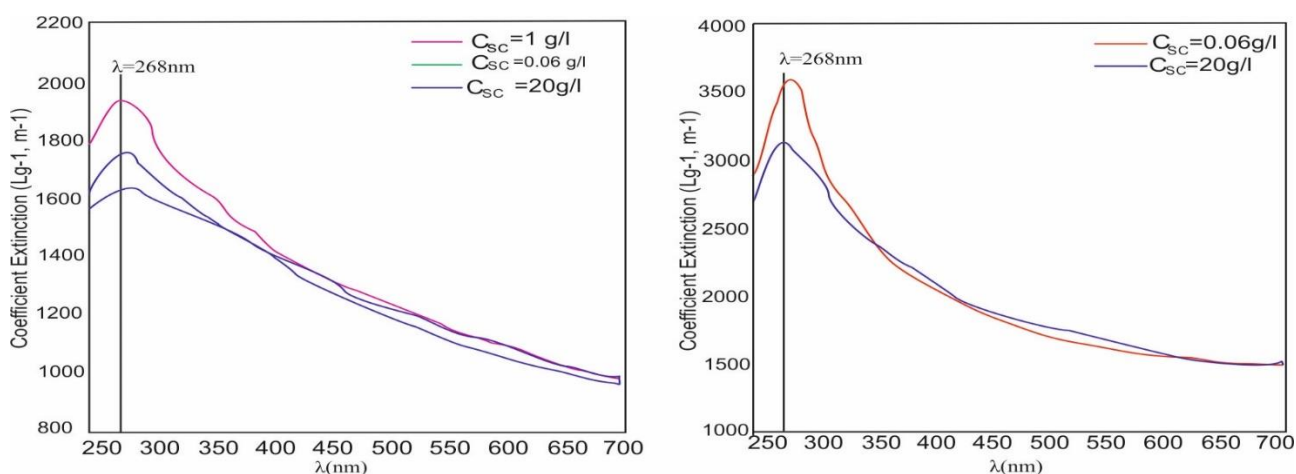


Figure 3 Optical Extinction Spectra of the Materials

After sixteen minutes of high-shear exfoliation, the upcycled EV anode reached a maximum yield of 1.88 weight percent, while the graphite flake reached a maximum yield of 1.37 weight percent. The absolute yields are far better than those associated with extra scalable non-oxidizing liquid exfoliation methods, such as ultrasound, microfluid, higher shear mixing devices, rotating disc, and Taylor-Couette-type methods, which result in yields between 0.1 and 3% weight percent and necessitate processing times of about 1 to 10 hours. Also, they tried the shear exfoliation process again after dispersing the unexfoliated anode based on EV sediment in a sodium cholate or fresh deionised water solutions to see whether they could get additional graphene nanomaterial out of the solvent exchange. Reprocessing graphite anode sediment many times may achieve yields of >4% in as little as 32 minutes. This is shown by the fact that 70% of the initial output was created in the identical 16-minute process period during this second exfoliation cycle.

The results of the process yield may be used to assess the rate of material upcycling. If $C_i = 100$ g/L and $V = 0.17$ L are used, this high-shear method might attain a production rate of around 1.3 g/h. Additionally, when compared to other batch shear exfoliation procedures, this performs well. In batch mode, high shear mixers may achieve production rates of around 5.4 g/h with $C_i = 100$ g/L and $V = 300$ L. The disparity between the two in terms of resources for input for processing waste is about ~ 104 times bigger, even if the latter has a higher absolute output rate. Another option is to look into

the necessary scale to reach the batch high-shear mixer's output rates. Based on this scaling, predict that the upcycling production rate is around 5 grammes per hour with a process capacity of 3 litres.

A further concentration measurement for higher level shear recycling in water alone yielded 0.70 wt %. Given the difficulties in removing residual surfactant from functional devices that have been solution-processed, high-shear upcycling offers hope for a sustainable way to synthesise few-layer graphene with minimal additive needs.

Each predecessor had two distinct traits, which is rather intriguing. A recent study shown that all ionic surfactants exhibit a decrease in nanosheet concentration at around 10 mM, using WS2 as a study system. Specifically, the scientists found that the aqueous-surfactant dispersions had reduced ionic conductivity in this area, which they attributed to screening which is electrostatic. A comparable decrease in concentration is seen in the less layer graphene that is generated during upcycling. It may be inferred from this that the procedure for destabilising nanosheets in aqueous surfactants during solution processing of used graphite anode materials is same for $C_{sc} = 10 \text{ mM}$.

The upcycling process's main (and greatest) Cgr peak occurs in the low-surfactant-concentration zone. Graphite flakes that have been shear exfoliated and are very pure defy this tendency, with dispersions that are unstable and low concentrations of few-layer graphene. Although this discovery needs more research, the higher concentrations achieved with spent anode precursors could be because of three things: (1) the sphericalized particles have more edge sites where intercalation and delamination can happen; (2) graphite expands as a result of lithium intercalation among layers in charge or discharge batteries; and (3) the surfactant makes the PVDF binder more wettable.

Building on the comments made earlier (1 to 3), the graphite particles which are in spherical used in the EV anode have a much lower diameter of 22 μm in comparison to the flake graphite particles' 550 μm . The shape that emerges from spherization of these particles during jet milling offers additional opportunities for layer insertion and delamination compared to the graphite flakes. The van der Waals attraction is diminished when lithium ions integrate into the layers of graphite of the anode over the operating lifetime of the battery. The layer spacing can go up by an average of 3.6% and as much as 14.8% as a result of this process. Because graphite's biochemical and thermal expansion is a proven way to increase the output of graphene and graphite derived material synthesis, enlarged layer in the anode might help the exfoliation procedure. Lastly, the hydrophobic binder based on PVDF should make graphitic particles less soluble in water. On the other hand, in conditions under the critical micelle concentration (CMC), anionic surfactants enhance the wettability and have been shown to have an affinity for PVDF. Sodium cholate's improved anode material dispersibility below the CMC might be due, in part, to its increased wettability. However, more research in this low surfactant region would be beneficial in maximising concentration utilising used graphite anode material, and the strong recovery performance is encouraging.

Both graphene materials' UV extinction spectra were affected by changes in surfactant content. A modification in the normal number of atomic layers is shown by the change in the shape of few-layer graphene. Both materials, which had few graphene layers, followed a similar trend as shown in Figure 4, which plots the numeral layers which are atomic against the concentration of surfactant. In dispersions of low surfactant content, they may see thicker sheets. Up to a surfactant concentration of

around 1 g/L, the thickness is very steady, but it drops much more after that. These findings are in line with the prior findings for Cgr and other 2D materials made from very pure precursors, and they correlate to the threshold of Csc 10 mM. Exfoliated lesser layer graphene from recycled anodes based on graphite from EVs may have its atomic layer number modified using surfactants, as shown here.

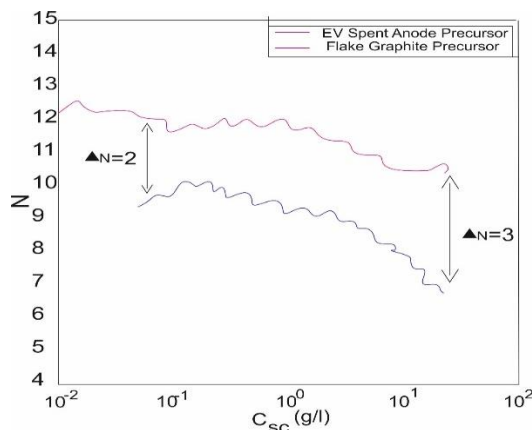


Figure 4 Plot of Number of Atomic Layers Vs. Concentration of Surfactant

After processing the upcycled material, researchers discovered that the layer number changed at a slower pace while using Csc. The variations in the number of layers rose from about 2 to 3 at the maximum concentration of 20 g/L (Csc). Recycled graphene seems to have a greater average layer counts than its few-layer graphite flakes-based counterpart. A fewer layer graphene is created by the process of shear exfoliation by means of turbulent fluxes, which degrade the outer surfaces of graphite particles. The resultant thin sheets with few layers are subject to rate-controlling mechanisms that extend to all flow structures containing turbulent energy up to the Kolmogorov length. Binder coatings on graphite anode particle and tiny exfoliated platelets may inhibit nanoscale peel and slide delamination.

The foundation of the spectroscopy metric used to determine N is a theoretical correlation that predicts the average layer count for numerous graphite precursors to within 16%. Figure 5's shaded areas show the total experimental and correlational uncertainty. Since the limitations of these inaccuracies in the normal layer's numbers for the various predecessor materials are comparable, the depth statistics would be validated by investigating distributions of particular nanosheet dimensions. These variations in material thickness, however, are consistent with the electrical property tests given later on.

3.2 Paper and Electronics Application

Ensuring the focus on ecologically sustainable methods, they investigated the use of repurposed graphene materials in the production of paper circuit boards. Solution-processed graphene dispersions were used to make conductive thin films on paper to test electrical properties. The process of making graphene inks included centrifugation, spray deposition onto paper substrates, and shear exfoliation in a solution of deionised water and sodium cholate. Figure 5A depicts the spray deposition procedure. The amount of graphite that remains after a 15-minute exfoliation processing period is substantial, since few-layer graphene produces around 1%wt. Compared to non-centrifuged liquid-phase exfoliated dispersions, lesser layer graphene nanosheets with N greater than ten have a greater amount of remaining graphite particles. This points to the presence of a limited number of particles with poor

exfoliation dominating the graphite bulk. In order to get rid of them, the dispersions were spun at RCF = 116g for 20 minutes after preparation, same as the method that was previously detailed for biocompatible layer graphene inks. To differentiate these inks from the less layer graphene dispersals that are made at RCF = 245g, is called as graphene-related material since they include thicker nanoplatelets.

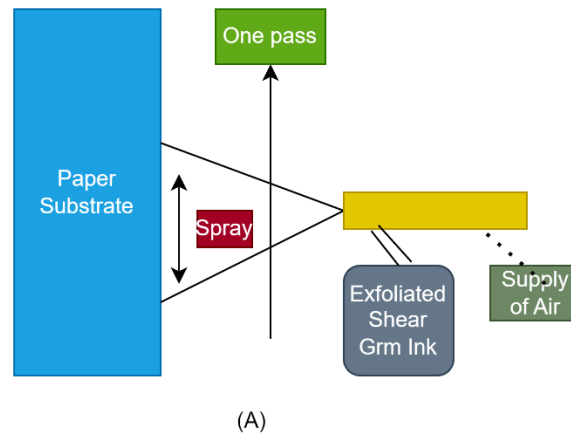


Figure 6 (A) Spray Deposition Procedure

In order to achieve an ink concentration more than 1 g/L, the graphite anode material was exfoliated at a concentration of 55 L/g with a surfactant concentration of 105 Csc/ Ci, which corresponds to the peak Cgr displayed in Figure 2. The resulting ink concentration was 1.67 g/L (CGrm). Spraying this 'flake Grm' substance onto the paper's substrate required adjusting its concentration to correspond with that of the regenerated 'EV Grm' substance.

Flexible paper substrates with electrical conductivity on one side and insulation on the other were made using several spray passes. In terms of physical robustness, the coatings were able to resist bending and folding and adhered strongly to the paper backing. A heat gun was used to blast 300 °C air over the paper, evaporating the water solvent with each spray pass, speeding up the drying process. Under the assumption of no pores and consistent thickness, the dried nanoplatelet sheet may be predicted to have a minimum thickness of around 9 µm after 105 spray passes. Nevertheless, it's probable that there will be some porosity, and the coating procedure will cause the actual film thickness to be bigger and variable. The cold-pressed paper substrate may add to this variability due to its visual roughness, but it also offers improved wetting and nanoplatelet film repeatability because to its higher substrate porosity. Figure 5C, which displays the electrical resistance standard deviations and low-magnitude error bars for the several nanoplatelet films tested, provides more evidence of this. All Grm coatings were determined to have electrical resistance variations below 13%.

Different spray-coated paper substrates had their sheet resistances tested using the transmission line technique. The Grm-coated paper was affixed to the copper tape electrodes in a parallel configuration utilising low contact resistance adhesive. Electrodes were placed at different distances (L) from one another in order to measure the electrical resistance. Figure 6B shows the trending linear among L or W and paper resistance; the slope of this trend was used to calculate the sheet resistance. Sheet

resistances range from around 1 to 10 kΩ/square, matching those found in previous research on graphene inks covered with spray on paper.

A tenfold increase in sheet resistance was found for the EV Grm covering over the flake Grm. Nanosheet defects, excessively thick or thin nanosheets, net are all potential causes of this. Under the same shear exfoliation conditions, the total amount of layer in the regenerated graphene dispersions is bigger than when using high-purity graphite flakes, as shown in Figure 5. In order to delve further into this, they conducted an extra centrifugation stage at 46 min and RCF = 245g to separate the percentage of fewer layer graphene from the shear exfoliated Grm inks (figure 5C).

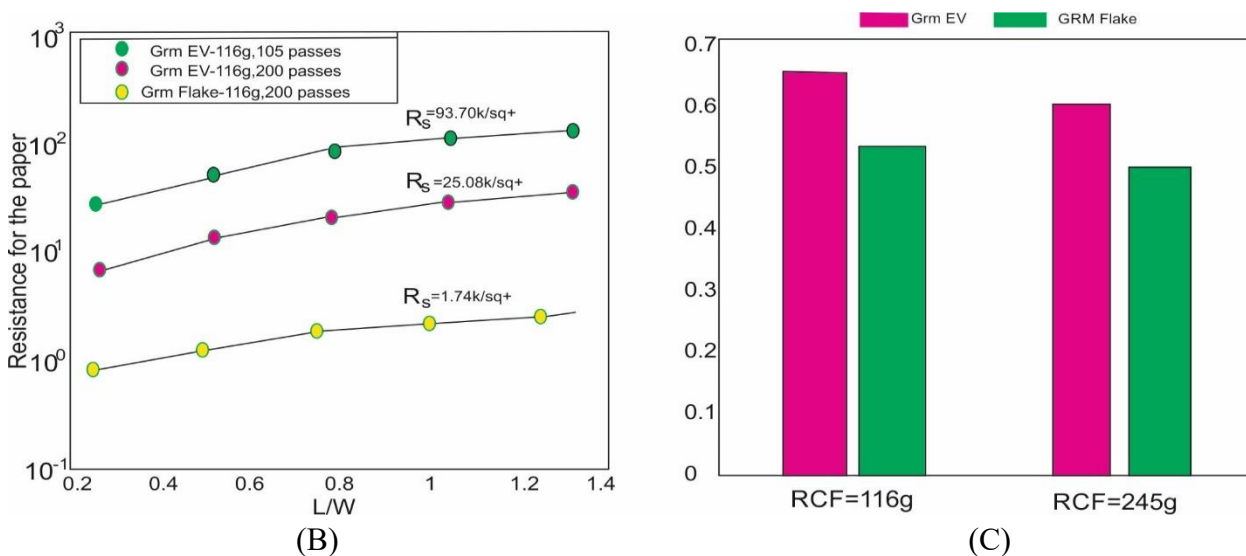


Figure 6 (B) Standard Deviation of Electrical Resistance, (C) Percentage of layers graphene from the exfoliated shear inks comparison

4. Conclusion

The results of this work suggest that high-shear exfoliation using water-soluble surfactants might be a good way to convert used graphite anodes from EVs into solution-processable graphene. Using higher purity graphite flakes as a predecessor resulted in nanomaterial yields that were on par with, or even greater than, those from another method. The highest absorption that could be salvaged by this method of upcycling was 38.6 percent more than the top amount of few-layer graphene that could be extracted from graphite flakes. This study demonstrates the viability of shear exfoliation, which means that additional shear exfoliation methods may treat waste electric car anodes with high yields. These approaches have produced high yields utilising natural graphite precursors. To achieve cost-effective upcycling of electric car used anodes, substantial modifications to this approach are acknowledged. The use of high-purity graphite flakes as a predecessor outcomes in aqueous surfactant dispersions with a few less atomic layers, according to spectroscopic facts. When the surfactant concentration is less than 10 mM, the PVDF binder and spherical graphite shape both affect the production yield. Surfactant affects the concentration and number of layers of the recycled product at concentrations higher than this, much as it does with few-layer graphene that is exfoliated from graphite. Our investigation concluded with a look at paper electronics made from recycled graphene-related materials. Alternative solvents for removing the binder and additives may be able to resolve the much-

increased sheet resistance of thin films made from recycled materials. The suggested water-based liquid processing methods provide environmental benefits over chemical treatments and the conventional usage of harmful solvents. Additionally, lithium-ion batteries may be made more sustainable via the use of graphite sources (natural or synthetic) and various water-soluble binders (carboxymethyl cellulose, for example), which opens up possibilities for improvement in the high-shear recycling process.

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