DOI: http://doi.org/10.4038/jgssl.v22i2.71

SYNTHESIS OF REDUCED GRAPHENE OXIDE THROUGH MICROWAVE IRRADIATION OF GRAPHENE OXIDE DERIVED FROM VEIN GRAPHITE FOR THE ANODE APPLICATION IN LI-ION RECHARGEABLE BATTERIES

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(Received: 8th August 2021 Accepted: 28th November 2021)

ABSTRACT

Among several methods being pursued to synthesize graphene, thermal reduction of Graphene Oxide (GO) has been identified as a straightforward and highly efficient method for scalable graphene production. However, high energy consumption is a significant limitation faced by this method. Microwaves, an alternative energy input source, have been widely used in many technological fields. Although graphene is an excellent microwave absorbent, GO has a poor microwave absorption capacity. Therefore, it needs a microwave absorbent like graphene to carry out the microwave reduction of Graphene Oxide. Therefore, the objective of this study is to synthesize reduced Graphene Oxide (rGO) using purified Sri Lankan vein graphite as the starting material through microwave irradiation without the use of a microwave absorbent such as graphene. First, Graphene Oxide was prepared using Tour's method, and Graphite Oxide Intermediate (GOI) was collected before washing and neutralizing the product. It was then treated separately in a domestic microwave oven at 900W power for 15 and 20 minutes. GOI turned black from brown after microwave irradiation took place for 20 min. The reduction of functional groups was confirmed by Fourier Transformed Infrared (FTIR) spectroscopy and X-ray diffraction study confirmed the formation of the reduced graphene oxide with the significant peak d_{002} reappearing at 24.95 $^{\circ}$, corresponding to an inter-planer spacing of 0.365 nm, which is comparable to that of the thermally reduced graphene oxide. Scanning electron microscopic analysis showed evidences of the existence of an expanded 'worm-like'' morphology of the graphite layers. These characterizations proved that the microwave irradiation had reduced Graphene Oxide without the addition of a microwave absorbent. In an electrochemical performance investigation conducted by assembling Li-ion half-cells, the anode electrode fabricated from the synthesized microwave reduced graphene oxide material showed a promising capacity of 266.7 mAh g^{-1} Vs. Li/Li^{+}. This study revealed the ability to successfully synthesize reduced graphene oxide through microwave irradiation without using a microwave absorbent starting with natural vein graphite for the anode application in Li-ion rechargeable batteries.

Keywords: *Vein graphite, Graphene Oxide, Reduction, Microwave irradiation, Li-ion battery*

1. INTRODUCTION

The development of numerous portable electrical equipment, involving consumer electronics, electric tools, and electric vehicles, has lead to a heavy demand for rechargeable energy sources. Lithium ion batteries (LIB), characterized by high specific energy, high efficiency, and long life, have been the common power source of choice for the consumer electronics market with a production of the order of billions of units per year (Scrosati &

Garche, 2010). Due to the advantages they offer (Deng, 2015), it is expected that LIBs will still be dominant in the rechargeable battery market, at least the next decade. However, due to the limitations of the availability of lithium reserves, it is not feasible to rely on this for longer term or in the event of a dramatic, unexpected increase in resource demand. One way to address this issue is by increasing the energy density of batteries (i.e., the amount of energy stored per liter or kilogram of battery). This not only offers important benefits for battery performance. It reduces pressure on resources and the impacts caused by battery production, as fewer materials are needed to produce the same power.

In a conventional LIB, the anode material is synthetic graphite, with an interlayer spacing of 3.35 4 Å (Hewathilake et al., 2018; C. Yang et al., 2020). Lithium ions are gradually inserted between single graphite layers. A maximum of one lithium per six carbons (LiC_6) is expected to be intercalated under ambient conditions in the van der Waals gaps above and below a carbon hexagon (Hewathilake et al., 2017). As a result, its theoretical capacity in LIBs is 372 mAh g-1 vs. Li/Li+ (Amaraweera et al., 2014; Balasooriya et al., 2006; Hewathilake et al., 2017; Yang et al., 2020). There is a likelihood of increasing the energy density of LIB, if the graphite anode can be modified to intercalate more Li^+ ions into its domain.

Graphene is the most studied material for charge storage due to its superior properties, such as high surface area, low weight, chemically inert and low price (El-Kady et al., 2016). The high surface area of graphene is (2,630 m^2 g^{-1}) is hugely favorable for energy storage applications (Pumera, 2011). Due to this property, it could be considered a potential anode material for high density LIBs. For preparing graphene, five different standard methods have mainly been reported namely; chemical vapor deposition (CVD), micromechanical exfoliation of graphite, epitaxial growth on an electrically insulating surface, solvothermal synthesis and the reduction of graphene oxide (GO) (Chen et al., 2010). Among these methods pursued to synthesize graphene sheets, reduction of Graphene Oxide (GO) at high temperature has been identified as a highly efficient green method where almost no hazardous reductants are used for scalable graphene production (Huh, 2011). Reduced graphene oxide (rGO) is thought to have similar properties to graphene and has thus been studied as a substitute for graphene in several publications (Gao, 2012; Liu, 2017; Pei & Cheng, 2012; Voiry et al., 2016). However, this reduction process requires rapid heating (>2000 °C min^{-1}) up to 1050 °C in an oven under argon gas or up to 800 °C in the presence of hydrogen gas, which is a significant limitation faced in this method (Chen et al., 2010).

Microwaves, an alternative source of energy, have been widely used in many fields. Although graphene is an excellent microwave absorbent, Graphene Oxide (GO) has poor microwave absorption capacity (Hu et al., 2012). Therefore, it needs the addition of a microwave absorbent to carry out the microwave reduction (Hu et al., 2012). Earlier studies have used graphene, a costly material, as a microwave absorbent to reduce GO(Hu et al., 2012). These studies show that microwave radiation can be used to reduce Graphene Oxide with the addition of a microwave absorbent. However, reduction of Graphene Oxide prepared with natural vein graphite without adding a microwave absorbent has never been reported. The present study prepares reduced Graphene Oxide, starting with natural vein graphite, without adding a microwave absorbent for a lithium-ion rechargeable battery (LIB).

2. MATERIALS AND METHODS

Sri Lankan vein graphite was collected from a deep underground mine. It was then crushed using a laboratory disk mill, and a particle size fraction <53 μm was separated by mechanical sieving. The collected sample was purified by the acid leaching method as described elsewhere (Amaraweera et al., 2013, 2018).

Then graphite was oxidized according to Tour's method (Marcano et al., 2010). Purified raw vein Graphite (3 g) and 18 g KMnO4 (Sigma Aldrich) were mixed well with 360 ml of conc. H2SO4 (Sigma Aldrich) and 40 ml of H3PO4 (Sigma Aldrich) to get a homogeneous mixture. The mixture was then heated to 50 °C and stirred for 12 h. Then it was cooled down to room temperature. At this stage, graphite was fully oxidized and converted to Graphite Oxide

(Dimiev & Tour, 2014). The resulting graphite oxide was collected and will be referred to as “Graphite Oxide Intermediate” (GOI) from now on. According to Tour’s method to prepare Graphene Oxide, the next three steps involve; 1) adding H_2O_2 to remove the excess $KMnO_4$, and 2) washing the mixture with water to neutralize the remaining acid 3) Sonicating to disperse the coagulated materials were not performed on the collected GOI because the remaining excess $KMnO_4$ and acid could aid in the Microwave reduction adapted in the next step (Pei & Cheng, 2012).

The collected GOI was separated into three parts where two parts were used as a starting material for the microwave reduction. One part of that GOI was then treated in a domestic microwave oven at 900 W for 15 min (MWrGO- 15) and the second part for 20 min (MWrGO- 20). They were then washed with water several times to neutralize the resulting material and sonicated to obtain Microwave reduced Graphene Oxide (MWrGO). Third part of the GOI was used to synthesize Graphene Oxide by adding 30% H_2O_2 (3 mL) and washing the resultant several times with water and sonicating it according to the conventional Tour’s method for comparison.

The resultant solids were characterized by Fourier Transformed Infrared (FTIR) spectroscopy (Nicolet 6700 spectrometer) to identify the functional groups present within the 500-4000 cm^{-1} region. UV-Vis spectroscopy (Shimadzu 2450) was used to understand the formation of graphite oxide and the reduction within the 220- 400 nm region. The crystal phases present in the synthesized samples were analyzed using X-ray Diffractometry (XRD) with the 'Rigaku-Ultima IV' X-ray diffractometer and Cu-K1 radiation (= 1.54). Scanning Electron Microscopy (SEM) (EEVO/LS 15 ZEISS) was used for morphological observation of the resultant samples.

Electrochemical performance investigations were conducted by assembling Li-ion half-cells with electrodes fabricated with both MWrGOs. Electrochemical performance investigations were conducted by assembling Li-ion half-cells with electrodes fabricated with both MWrGOs. To make electrodes, 80% MWrGO was mixed with 10% conductive carbon (Sigma Aldrich), 10% PVDF (Sigma Aldrich), and 10% N-Methyl-2-pyrrolidone (NMP, Sigma Aldrich). The resulting slurry was then cast on copper foil. The sample was dried at room temperature before being placed in a standard vacuum oven and kept at 100 °C overnight. In an argon-filled glove box, a coin cell (CR2032) was assembled from a fabricated electrode, a lithium metal as counter and reference electrode, a Celgard 3501 separator, and 1.0 M LiPF6 in ethylene carbonate and dimethyl carbonate (EC: DMC vol. 1:1/Sigma Aldrich) solvent liquid electrolyte. Galvanostatic charge/discharge tests were carried out using a Landt CT2001A battery testing station. The half cells were cycled between 0.002 and 1.5V (versus Li/Li^+) at a 0.2 C rate.

3. RESULTS AND DISCUSSION

The physical appearance of the Graphite Oxide Intermediate GOI(a), GO(b), MWrGO-15(c) and MWrGO-20(d) dispersions is shown in Fig. 1. GOI dispersion shows a dark brownish colour whereas GO dispersion shows its characteristic brown colour in dispersion (Marcano et al., 2010). However, MWrGO-15

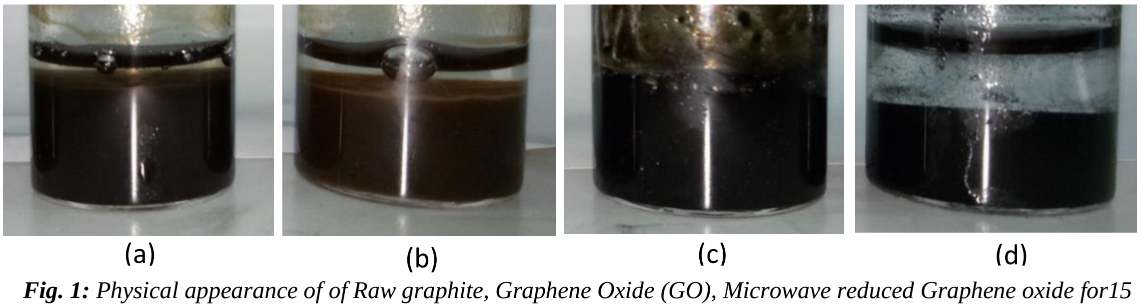

Fig. 1: *Physical appearance of of Raw graphite, Graphene Oxide (GO), Microwave reduced Graphene oxide for15 Minutes (MWrGO-15) and Microwave reduced Graphene oxide for 20 Minutes (MWrGO-20)*

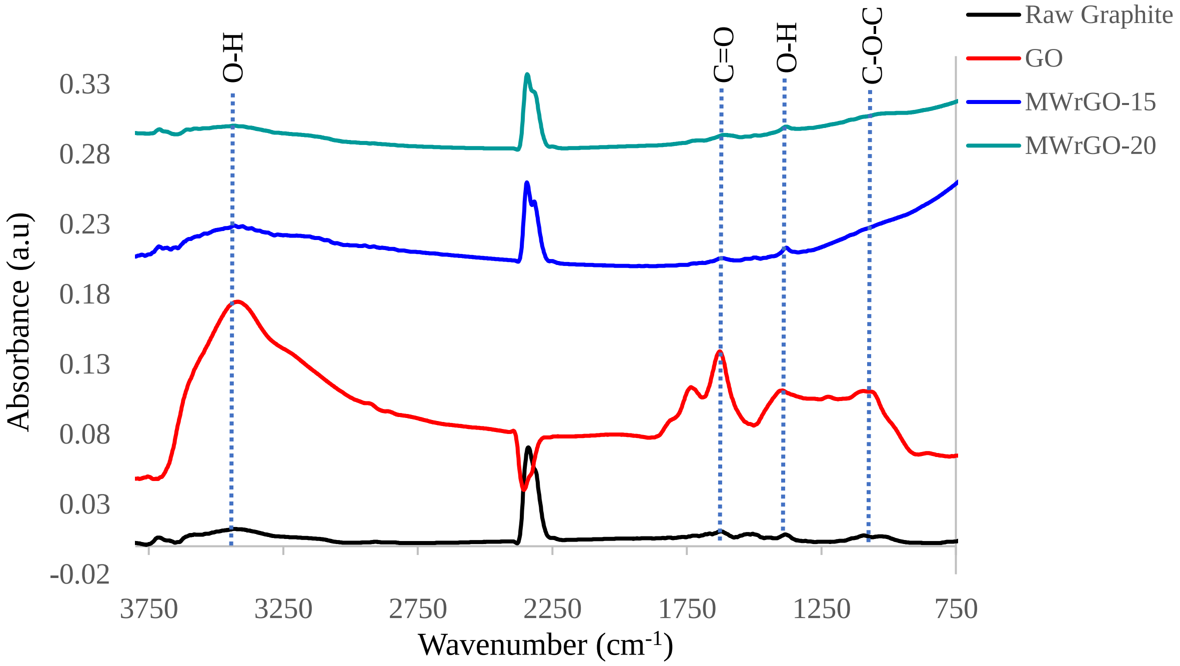

Fig. 2: *FTIR spectra of Raw graphite, Graphene Oxide (GO), Microwave reduced Graphene oxide for15 Minutes (MWrGO-15) and Microwave reduced Graphene oxide for 20 Minutes (MWrGO-20)*

showed a brownish-black colour after reduction, and MWrGO-20 showed a black

FTIR spectra of raw graphite, GO, MWrGO-15, and MWrGO-20 are shown in Fig. 2. The existence of molecular water with strong broad vibrational bands corresponding to O-H stretching from 2800-3700 cm-1 can be observed very clearly. The above peak in this region is responsible for stretching vibration of the hydroxyl group, where the hydroxyl groups may be from absorbed water molecules or phenolic OH or OH from carboxylic groups.; C-O stretching vibration of carboxylic acid at 1414 cm-1, vibrational bands corresponding to C=O stretching at ~1735 cm-1, O-H bending at ~1220 cm-1 and C-O-C stretching at ~1043 cm-1 imply the presence of carboxyl groups, alcoholic and epoxide groups, respectively. The peak appearing near ~2250 cm-1 could be attributed to atmospheric CO2 (Amaraweera et al., 2018, 2014; Hewathilake et al., 2017, 2018; Rolle & Sega, 2019). From the FTIR spectrum of MWrGO-15 and MWrGO-20, it is evident that the intensity of these peaks is relatively small in the microwave reduced material confirming the elimination of these functional groups from both MWrGO-15 and MWrGO-20, which is expected during the reduction of GO. Further, reduction of functional groups in MWrGO-20 compared to MWrGO-15 can also be observed from the FTIR spectra. These bands agree with those in the literature for reduced Graphene Oxide (Huh, 2011).

The X-ray diffractograms obtained on the raw graphite, Graphene Oxide (GO) and the Microwave reduced Graphene Oxide MWrGO-15 and MWrGO-20 are shown in Fig. 3. The major peak for graphite (002) and (004) phases can be seen around 26°. However, after oxidation, the major peak appears near 10.31°. Further, according to Fig. 3, the d-spacing value increases from 0.335 nm to 0.837 nm when graphite is fully oxidized in GO (Marcano et al., 2010). This indicates that the oxidation alters the crystalline structure of graphite thus resulting in an expanded structure. However, after reduction using microwave energy, the formation of rGO with its prominent peak of (002) reappearing at 24.95 ° corresponding to an interlayer spacing of 0.365 nm for both MWrGO-15 and MWrGO-20 can be observed. The major peak shift from 10.31 ° to 24.95 ° which is a characteristic of rGO, confirms that the rGO is achieving its structure. These observations are in agreement with the previously reported results on reduced graphene oxide (Huh, 2011; Loryuenyong et al., 2013).

According to the XRD analysis, it can be seen that after treating the GOI using microwave irradiation, the peaks shift towards 24.18 ° (Fig.

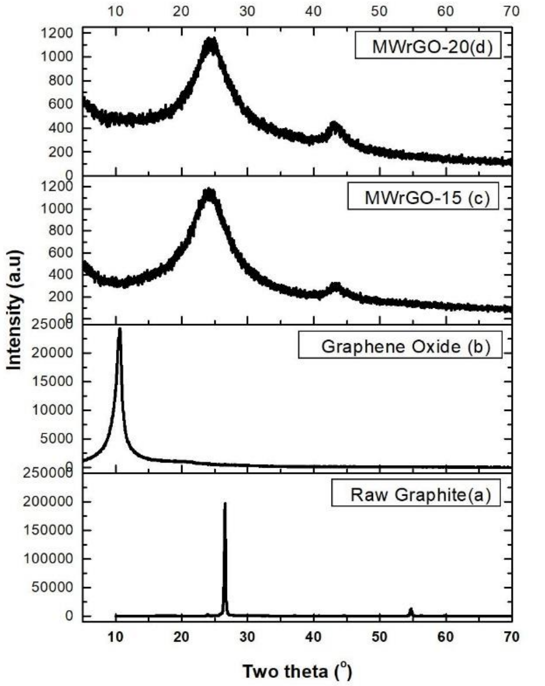

Fig. 4: *(a) X-ray diffraction of Raw graphite, (b) Graphene Oxide (GO), (c) Microwave reduced Graphene oxide for15 Minutes (MWrGO-15) and (d) Microwave reduced Graphene oxide for 20 Minutes (MWrGO-20)*

3). However, the peak broadening is relatively high reflecting its amorphous nature (Huh, 2011). It is also worth noting that X-ray diffractogram of the reduced graphene oxides does not show the existence of the peak corresponding to GO after the reduction after reduction (Fig. 3(c) and 3(d)). This can be interpreted as; the microwave irradiation has resulted in a reduction of GO into rGO (Alam et al., 2017; Loryuenyong et al., 2013; J. Yang & Gunasekaran, 2013). The FTIR also confirms that the peak intensity corresponding to epoxy,hydroxyl, and carbonyl groups in MWrGO-15 & MWrGO-20 has been reduced compared to GO (Fig. 2). When comparing the FTIR results of MWrGO-15 and MWrGO-20 with the XRD results, it can be seen that when the time of microwave irradiation was increased, some functional groups have been removed without changing the peak positions of the XRD spectrum. This could be because, when GOI was subjected to microwave treatment for 15 minutes, the structure of rGO was achieved. However, some remaining functional groups were removed when subjected to 20 minutes of microwave irradiation. Hence, microwave irradiation has resulted in the successful reduction of GO into rGO without the addition o f a microwave absorbent (Alam et al., 2017; Loryuenyong et al., 2013; J. Yang & Gunasekaran, 2013).

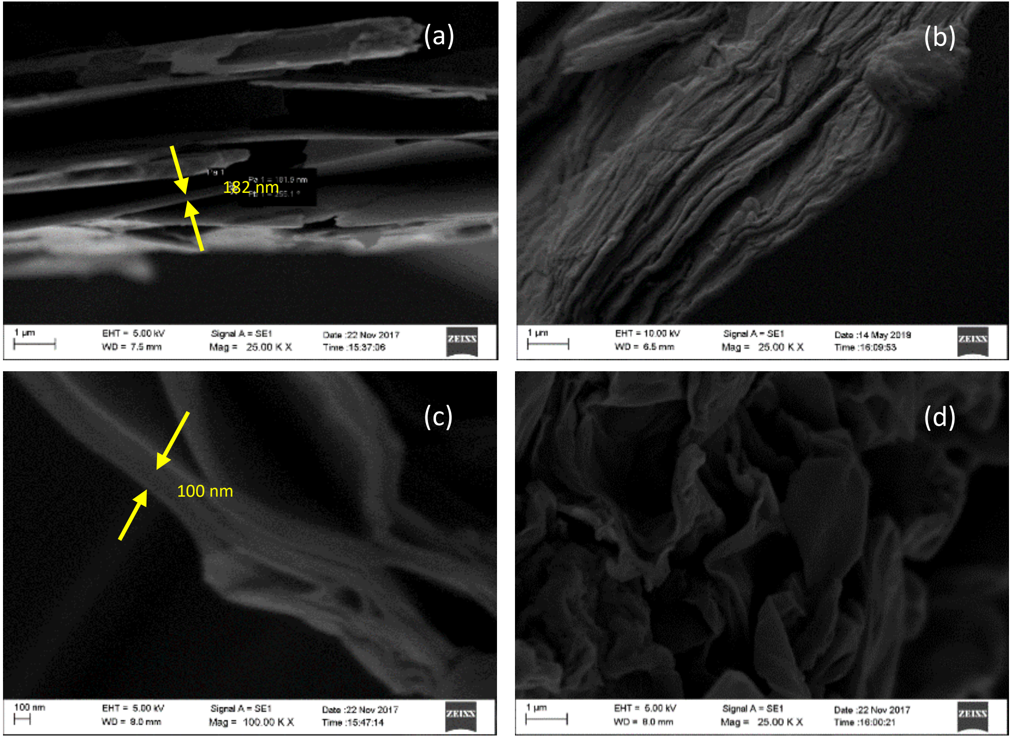

Fig. 3: *(a) SEM images obtained for Raw graphite, (b) Graphene Oxide (GO), (c) Microwave reduced Graphene oxide for15 Minutes (MWrGO-15) and (d) Microwave reduced Graphene oxide for 20 Minutes (MWrGO-20) (d)*

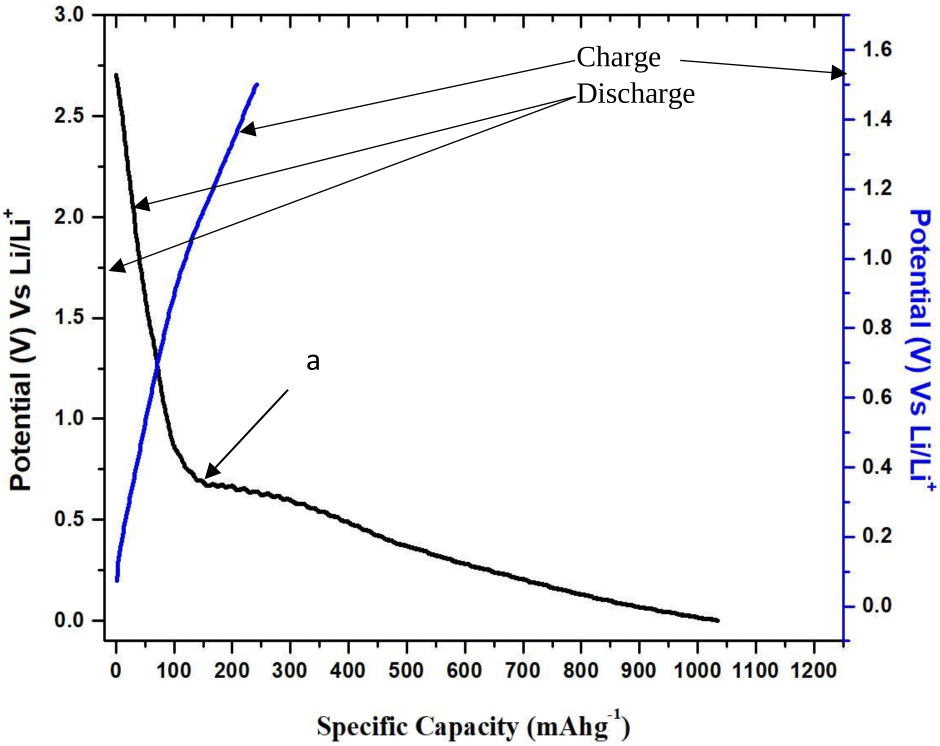

Fig. 5: *First discharge and charge cycle curve of the assembled cell using MWrGO versus Li/Li$^+$ between 0.002-2.0V at 0.2C rate*

Fig. 4 shows SEM images obtained for raw, Graphene Oxide (GO), MWrGO-15 and MWrGO-20. The SEM image of raw graphite shows a compact layered structure (**Error! Reference source not found.**a). However, SEM image of a GO shows a compact wrinkled wave-like structure (**Error! Reference source not found.**b). After reduction, MWrGO-15 and MWrGO-20 show an expanded 'worm-like'' morphology comparable to the previous literature (Zhu et al., 2010). This observation gives further shreds of evidence for the successful formation of the reduced graphene oxide through the Microwave reduction technique used in this study.

All the material characterization discussed above showed that both MWrGO-15 and MWrGO-20 have been reduced successfully. Relatively, MWrGO-20 had a puffier structure than MWrGO-15 through visual and SEM identification. Due to its puffy structure, MWrGO-20 may have the ability to serve as an anode material in high density batteries (Jian et al., 2012; Yuan, 2006). Hence, it was selected for the electrochemical performance analysis of the Li-ion rechargeable battery.

4. Electrochemical Performance analysis of the microwave reduced Graphene Oxide in Li-ion cell

The electrochemical performance of this Microwave Reduced Graphene Oxide (MWrGO-20) as the anode material of the Li-ion rechargeable battery was investigated by carrying out electrochemical charge-discharge cycle tests. The typical loading amount of active material was 4.2 mg. Cells were cycled between 0.002 and 1.5V (versus Li/Li$^+$) at 0.2 C rate. The capacity was calculated based on the mass of the active material. Fig. 5 shows the first discharge and charge cycle curve of the assembled cell.

The battery's discharge specific capacity and Columbic efficiency, assembled with MWrGO-20 anode and Li metallic cathode, against the number of cycles at 0.2 C rate between 0.002 and 1.5 V are shown in Fig. 5. The battery assembled in the present study, with MWrGO, showed a first specific discharge capacity of 1290 mAh g^{-1} with an irreversible capacity of 1050 mAh g^{-1} for the first cycle. The higher irreversible capacity during the first cycle is mainly due to the decomposition of solvent and

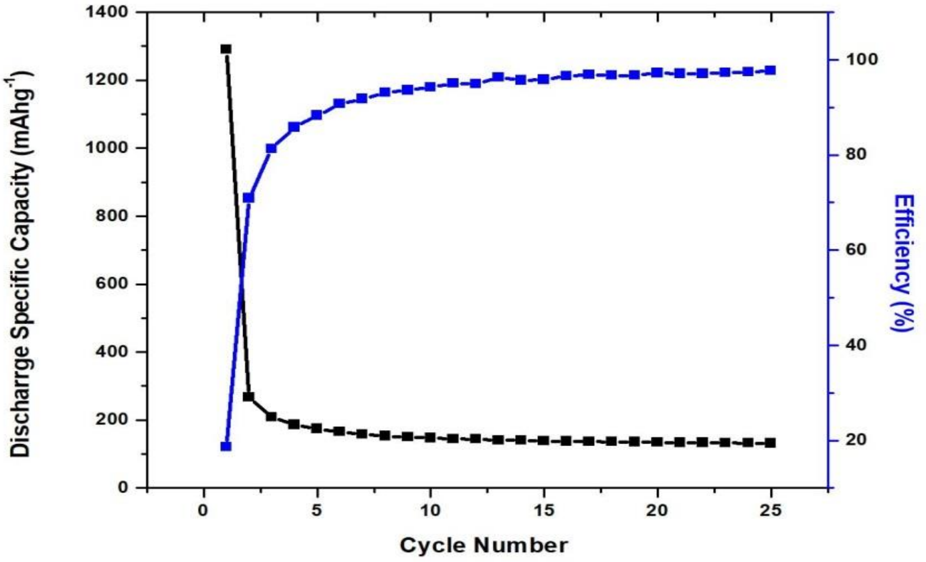

***Fig. 6:** Discharge specific capacity and Columbic efficiency of the battery, assembled with MWrGO versus Li/Li$^+$*

the simultaneous formation of a solid electrolyte interface (SEI) film. The irreversible capacity in a battery, in the first cycle, is usually attributed to the decomposition of solvent and the simultaneous formation of a solid electrolyte interface (SEI) film on the graphite surface (Amaraweera et al., 2018). MWrGO electrodes show a plateau near 0.70-0.80 V vs. Li/Li$^+$ (marked as 'A' in Fig. 5), which is characteristic of a passivation layer formed on the graphite surface (Hewathilake et al., 2017). The second discharge capacity was 266.7 mAh g^{-1}. Although the theoretical discharge capacity of graphite is 372 mAh g^{-1}, the second cycle discharge capacity was low in the present study. However, in a study by Alshareah *et al.*, they showed that the discharge capacity of anode material prepared using rGO, reduced using $AgNO_3$ and Hydrazine, resulted in 164 mAh g^{-1} for the 1st cycle. Compared to that, the present study shows a significant improvement. This observation shows that the microwave reduction of graphene Oxide has a higher potential for Li-ion intercalation than chemically reduced graphene oxide. Their study reports that superior performance of an anode could be achieved by using holey reduced graphene oxide (HRGO). The holes provide better access for the electrolyte into the entire HRGO structure and hence better Li-ion transport and intercalation mechanisms (Alsharaeh et al., 2016). This suggests that along with the puffy structure, if MWrGO has surface defects with interconnected pores, it could result in high capacitance.

5. CONCLUSIONS

Graphene Oxide was fully reduced through microwave irradiation without using a microwave absorbent from natural vein graphite as the starting material. In the electrochemical performance investigations conducted by assembling electrodes fabricated from the synthesized microwave reduced graphene oxide into Li-ion half-cells, it showed a promising discharge specific capacity of 266.7 mA h g^{-1}, which is higher than that of chemically rGO. This observation shows that the microwave reduced graphene oxide has a high potential for Li-ion intercalation than chemically reduced graphene oxide.

ACKNOWLEDGEMENT

This work was supported by Research grant on Development of Sri Lankan Graphite for Rechargeable Batteries funded by the General Treasury under a Cabinet Paper 17/1907/716/038 of 2017-08-09 of Sri Lanka and National Research Council of Sri Lanka, through Grant Number 15-007.

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