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DEBATE

Energy Harvesting or Merely Electrochemistry? An Investigation Into Energy Sourced From Graphene Oxide–Metal Junctions

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ABSTRACT In recent years, there has been growing interest in energy harvesting from ambient moisture utilizing devices constructed using materials such as graphene oxide (GO), reduced graphene oxide (rGO), and various metals including copper, aluminum, titanium and silver. Although many papers report remarkable energy harvesting results, we contend that not all of the possible energy sources have been considered in many experiments. We argue that a significant amount of the energy claimed as harvesting is actually due to irreversible REDOX reactions between the GO and the metal electrodes. In this paper, we identify some example studies and carry out an experiment in which we characterize the charge and energy available from a GO-metal sample in a stable homogeneous atmosphere that excludes nearly all known sources of harvestable energy. We then utilize AFM IR spectroscopy to observe irreversible reduction in the graphene oxide that has been in contact with copper in configurations similar to published energy harvesting devices, showing significant reduction in contacted regions. We also observe physical changes at the copper-GO junction that offers further indication that GO undergoes chemical changes, which may potentially explain some of the energy that is claimed to have been harvested from environmental moisture or moisture gradients.

INDEX TERMS Electrochemistry, energy harvesting, graphene oxide, reduced graphene oxide.

I. INTRODUCTION

Kinetic ambient energy harvesting, utilizing graphene oxide (GO) interactions with local humidity and temperature gradients, has been widely accepted as genuine mechanisms for energy generation [1], [2]. Our experiments, in the case of GO, suggest that additional previously overlooked energy sources may play a role in the tiny amounts of energy

observed in many published device configurations. We argue that in many device configurations this energy originates not only from the environment, but also from irreversible changes in chemical or ionic potential in the metal–GO junction, or within the GO itself. In such cases, the observed electrical output originates from stored chemical energy rather than from true ambient energy harvesting. This distinction is critical because it challenges the interpretation of numerous experimental results. In this paper, we review a sample of the large number of published works in which consideration

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of this distinction is missing, where energy harvesting has been claimed with various metals including copper [3], aluminum [4], titanium [5] and silver [6] in contact with GO, and where stored energy may be playing a role in the observed experimental results. We present a method to differentiate between these two energy sources, and introduce a rigorous mathematical model based on empirical system behavior, enabling a more accurate characterization of energy generation mechanisms from some metal-GO based devices. Utilizing this method, we characterize the claimed energy harvesting, together with the ionic diode effects in GO-rGO junctions, as well as the ionic conductivity dependence on ambient humidity. This is in order to understand the phenomenon and identify the source of significant energy observed from these devices. We identify, isolate, and characterize three separate phenomena to explain all of the reported observations of these junctions. The characterization includes the asymmetric, rectifying current–voltage response of the GO-rGO junction, measurement of the electrochemical cell potentials of the rGO-GO-metal junctions, and the complex electron-ion conductivity of GO as a function of relative humidity. Our results indicate that the energy embodied entirely in the GO-metal interface may sometimes wholly explain the ‘harvested’ energy claim, with the observed energy released by electrochemical reduction of GO on the rGO interface, with the second half-cell being formed by the metal-GO interface. We show that GO behaves like an electrolyte with increasing ion conductivity as a function of humidity, suppressing ion conduction and REDOX in dry atmospheres, which can also be confused with the recharging process in hygroelectric energy harvesting when the stored energy becomes available again after rehydration.¹

II. GRAPHENE ENERGY HARVESTING

With the rapid expansion of ultralow-power electronics, driven by applications such as wearable health monitors, the Internet of Things (IoT), structural health monitoring systems, and implantable biomedical devices [7], energy harvesting has emerged as a critical area of research and development. These systems often operate in environments where battery replacement is impractical or impossible, demanding solutions that can autonomously and sustainably extract energy from gradients in the surrounding environment. In response, the past decade has seen a surge in efforts to identify and exploit novel ambient energy sources. A broad array of energy harvesting mechanisms have been proposed and experimentally demonstrated. For instance, the triboelectric effect harnesses mechanical energy, typically from motion or contact-induced charge separation between dissimilar materials [8], while piezoelectric, thermoelectric, and pyroelectric mechanisms convert vibration and heat flow into electrical energy [9]. Similarly, the streaming potential

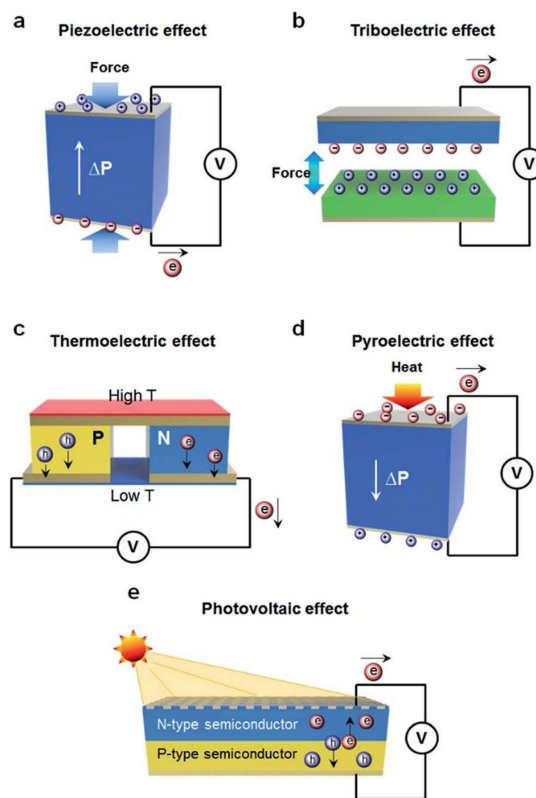


FIGURE 1. Mechanisms illustrating physical energy. After [9].

exploits the movement of water through nanoscale or capillary channels [10], often driven by evaporation, to generate an electric potential through ionic displacement [11]. All this is in addition to the more conventional photovoltaic, thermoelectric, pyroelectric, as well as the classical mechanical and vibrational forms of energy harvesting.

What unites these examples is the presence of a clearly defined external energy source, mechanical, thermal, or electromagnetic, which is converted into electrical energy. This traceable energy pathway is essential for validating a device as a genuine harvester of environmental energy, rather than just as a system discharging pre-existing internal stored energy like a battery. Furthermore, most of the device types pictured in Figure 1 do not undergo significant physical or chemical degradation with use. As the field matures, the ability to distinguish between these two categories becomes increasingly important for accurate evaluation of new technologies and their real-world viability.

In recent years, a new class of so-called energy-harvesting devices has emerged in the literature, in which the source of energy is either not clearly identified, appearing physically implausible, or remains fundamentally uncertain. One of the earliest and most widely cited examples is the graphene oxide–reduced graphene oxide (GO–rGO) junction device reported by Huang et al. [1]. This was followed by a series of similar works (e.g., Liu et al., 2020) [2], all of which claim to harvest electrical energy solely from the presence of ambient

¹rGO is reduced graphene oxide that is GO from which most of the oxygen-containing functional groups have been removed, partially restoring the electrical conductivity of the original graphene lattice, though residual lattice defects and disorder introduced during oxidation are never recovered.

atmospheric moisture—without any apparent external gradient of moisture, temperature, pressure, or motion. Since these early demonstrations, the approach has developed rapidly into a broad field of moisture electricity generators spanning carbon, polymer, hydrogel, and bio-derived materials [12], yet the precise origin of the measured output remains contested.

In these studies, the central claim is that the device spontaneously generates a persistent open-circuit voltage and is capable of recharging itself indefinitely when the electrical load is removed with nothing more than a stable and humid atmosphere. This steady-state behavior, in the presence of constant humidity and absence of any obvious energy flux, is interpreted as continuous energy harvesting from the ambient environment. However, such behavior raises critical questions about the actual origin of the energy, particularly when the observed electrical output is maintained over long durations with no detectable input of mechanical, thermal, or electromagnetic energy.

We propose that the apparent self-recharging behavior and sustained voltage under open-circuit conditions is not necessarily evidence of genuine energy-harvesting systems, as these phenomena may also suggest the possibility of latent internal energy sources, such as chemical potential differences across material interfaces (e.g., GO–metal contacts), being discharged rather than energy being continuously extracted from the environment. Without rigorous thermodynamic accounting and definitive identification of the energy input, these systems risk being mischaracterized as harvesters when, in fact, they may function more like chemical cells slowly depleting internal reservoirs.

In this study, we replicate key aspects of previously reported experiments by fabricating graphene oxide (GO)–metal junction devices, and measuring their electrical behavior under controlled environmental conditions, and then use near-field AFM spectroscopy to compare carbon-oxygen bond density in pristine parts of the sample, and regions contacted with copper during the experiment. The experiment subjects two separate devices to a stable atmosphere with constant temperature and humidity, specifically chosen to reduce most of the external energy input. Under these conditions, there is little thermal, mechanical, or photonic gradients available to drive continuous energy harvesting. Therefore, any sustained electrical output arises mostly from internal processes rather than from ambient energy sources.

If these devices truly function as energy harvesters, they would be expected, over time, to accumulate and deliver a total charge (coulomb output) that exceeds the finite energy capacity of an electrochemical storage device such as a battery or capacitor. However, our experimental results reveal that the devices likely exhibit a finite space-charge capacity, with discharge behavior closely resembling that of a conventional primary electrochemical cell. Specifically, we observe a characteristic exponential decay in short-circuit current over time, approaching zero asymptotically, a hallmark of systems discharging stored internal energy.

We present detailed experimental data along with analytical modeling to support this interpretation. Our model accurately describes the observed electrical discharge as the depletion of a fixed quantity of charge stored at the GO–metal interface, consistent with electrochemical potential rather than ongoing ambient energy harvesting, with only a tiny allowance for actual energy harvesting due to experimental noise and ambient variability. By estimating the volumetric charge density, we show that the energy capacity of these devices is on the same order of magnitude as conventional thin-film electrochemical cells. Moreover, we critically review some of the existing literature, as summarized in Table 1, and find that no GO-copper based energy harvesting devices demonstrate charge or energy densities that exceed the bounds of our findings. In every case, the reported performance is consistent with the discharge of finite stored chemical energy, rather than with continuous environmental energy harvesting. This raises important questions about the true nature of the mechanisms at play in these devices, and calls for greater rigor in distinguishing between genuine ambient energy harvesters and systems driven by transient electrochemical processes.

In this paper, we do not aim to conclusively determine the energy source or provide a resounding refutation of claims made. We aim only to point out deficiencies in the assumptions made and identify alternative explanations for at least some of the reported energy.

III. ELECTROCHEMISTRY OF GRAPHENE OXIDE (GO)

Graphene oxide (GO) deposited films, as well as bulk GO, consist of stacked assemblies of single- and few-layer flakes of chemically modified graphene, which typically adopt a lamellar structure after solvent evaporation. In these structures, some of the covalent bonds within the aromatic carbon lattice are disrupted and functionalized by various oxygen-containing groups. It is a particularly complex non-uniform material comprising a patchwork of oxidized (sp^3 -bonded) and graphitic (sp^2 -bonded) domains with various functional groups (epoxide, hydroxyl, carboxyl, etc.) creating negatively-charged surfaces. These chemical groups are what distinguishes graphene from GO. These introduce a range of local electronic states, defects, and charge trapping sites, all of which impact GO's electronic structure and chemical potential. This provides for a number of possible mechanisms by which stored electrochemical energy could become manifested through the motion of charges, movement of charged functional groups, catalytic reduction of GO, as well as oxidation-reduction reactions with other materials such as chemical reagents, or the metal electrodes [5], [13]. Furthermore, GO has been shown to be able to spontaneously become reduced at room temperature under various catalytic conditions [14], [15], which may also release latent energy.

The fabrication of GO is an energy-intensive chemical process that requires a substantial input of chemical potential energy to oxidize graphite and to separate it into GO [16]. As a result, GO possesses a considerable amount of stored

TABLE 1. Reported electrical output and measurement duration of representative GO/rGO-based moisture energy-harvesting devices, compared with the present work. Headline voltages are frequently open-circuit values, which deliver negligible charge; the distinguishing test of a genuine harvester is whether current is sustained as the device is run toward depletion.

Reference	Material / electrodes	V_{oc}	I or I_{sc}	Current-delivery duration	Reservoir depleted?
Huang <i>et al.</i> 2018 [1]	Bulk GO, Schottky-junction electrodes	~ 1.5 V (unit)	—	Brief duration	Not demonstrated
Liu <i>et al.</i> 2020 [2]	Protein nanowires, Au electrode	~ 0.5 V	$\sim 17 \mu\text{A}/\text{cm}^2$	≥ 20 h before self-recharging	Not demonstrated
Qi <i>et al.</i> 2020 [3]	PEDOT:PSS/GO aerogel	sub-volt	low	Thermal/humidity transients	Not demonstrated
Zhao <i>et al.</i> 2016 [4]	Gradient GO framework (g-GOF)	~ 0.4 – 0.5 V	μA -scale	Hours	Not demonstrated
Cho <i>et al.</i> 2024 [5]	Water-induced p–n rGO junction	sub-volt	μA -scale	Hours	Not demonstrated
Huang <i>et al.</i> 2019 [6]	GO composite	~ 0.6 V (held > 120 h)	μA -scale	V_{oc} held > 120 h; brief current duration	Not demonstrated
This work	GO-rGO, Cu and Zn electrodes	0.5 V (Cu), 1.3 V (Zn)	$30 \mu\text{A}$ (peak)	$> 1,300$ h continuous I_{sc}	Yes, fitted to $I \rightarrow 0$ finite Q

Gibbs free energy, which can be readily released through various chemical, thermal, and electrochemical mechanisms.

Graphene oxide is a more thoroughly exfoliated form of graphite oxide, which was first synthesized in 1859 by Brodie [17]. Despite the similar composition to graphene oxide produced using Hummers' method, its precise chemical structure is still not clear [15], contributing to the complexity of a rigorous understanding of its chemistry, as well as its amorphous, semiconducting, ionic, and non-stoichiometric characteristics. The situation is further complicated by the variability in the produced GO, which can have different chemical structures and compositions of the oxygen rich groups, e.g. hydroxyl, carboxyl, carbonyl, and epoxy functional groups, which have different binding energies, as well as total carbon-to-oxygen ratios that vary by more than a factor of two, depending on the preparation method and technique [17], [18]. Furthermore, there are also variations in the concentrations and types of lattice defects in the carbon plane. The unconventional electrochemical activity of GO may also explain its sometimes counterintuitive behavior, which is contrary to standard conventions and likely also contributes to confusion about what is actually taking place. For example, the electrical polarity of oxidation and reduction can be reversed in an electrochemical cell, and GO has been observed to reduce on the anode of an electrolysis cell, where only oxidation is conventionally expected to occur [19].

IV. ELECTRICAL AND ELECTRONIC PROPERTIES OF GO

Moist GO has exceptional ion transport properties, offering fast and non-fluidic ionic transport in both horizontal (in-plane) and vertical (cross-plane) directions for positive and negative ionic charges [7]. This facilitates the galvanic extraction of electrical energy from the GO as well as any REDOX reactions with contact materials that may be in contact with it, with the presence of moisture. When placed in a dry environment, the GO becomes a poor ionic conductor, and energy production is almost entirely suspended, rendering it a poor conductor with some semiconducting properties.

As an illustration of the reactivity of GO with metals, it has been observed that stable bonds between numerous metals and chlorine are spontaneously broken in aqueous solution, and GO is spontaneously reduced at room temperature by the metal salts [20]. Furthermore, there is ample evidence from previous studies that GO can spontaneously breakdown oxygen-contained functional groups in aqueous room temperature conditions in the presence of stable organic compounds such as vitamin C [21], various amino acids [14], as well as by heating. Furthermore, GO has exceptional capacity for energy storage by charge separation. This effect can be isolated by sandwiching GO between two rGO electrodes, thus ensuring that there are no electrochemical processes that occur. The charge stored in such devices has been shown to be as high as $2.35 \text{ F}/\text{cm}^3$ [22], which is an order of magnitude greater than our electrochemical device, and may also offer an alternative explanation for the energy claimed to be harvested in many GO based devices.

V. EXAMPLES OF CLAIMED ENERGY HARVESTING USING GO-METAL JUNCTIONS

A review of published literature shows that there are only two physical mechanisms that require GO for energy harvesting [13]. This section describes the available mechanisms by which GO is known to be able to extract energy from the environment and convert it into electricity. These can be summarized as follows:

A. MOISTURE BASED THERMAL KINETIC ENERGY HARVESTING WITH GRADIENT GO FRAMEWORKS (G-GOF)

Utilizes the dehydration and rehydration of GO as the energy source. During the dehydration cycle, the thermal kinetic energy of the displaced water molecules is converted into a chemical potential, with the restoration of the COOH functional group in GO. During rehydration, COOH dissociates into COO^- and H^+ producing the electric potential. The transport of water extracts thermal energy from the environment, which is stored as chemical potential energy

in the GO, which becomes available after rehydration [4]. The amount of water taken up by the GO film, and hence the magnitude of this stored chemical potential, depends on its exceptional water-adsorption capacity [23].

B. MECHANICAL KINETIC ENERGY EXTRACTION ENHANCED VAN DER WAALS HETEROSTRUCTURES

Utilizes electrostatic forces (Coulomb drag) between negatively charged GO with another, positively charged material such as graphene film, to convert mechanical motion into electricity [24].

Other energy harvesting phenomena including triboelectric systems (utilizing friction to convert mechanical energy into electricity), piezoelectric-graphene hybrid systems (inelastic stress and strain conversion to electricity), are not GO-specific and not relevant to the following analysis.

Many papers utilizing GO as part of a claimed energy harvesting system do not consider, or properly explain the recharging process, particularly with moisture based harvesting. It is often claimed that once the electrical load is removed from the device, the open-circuit voltage *recovers*, which is claimed to demonstrate energy harvesting. Our experiment illustrates that this is merely the expected time-response of an RC ladder, or the diffusion element of a Randles-type [25] equivalent circuit for a capacitor or battery model, as illustrated in Figure 2, undergoing intermittent rest followed by discharge, and not in itself evidence of any type of energy harvesting phenomena.

When the GO dehydrates, it is unclear how much of the subsequently observed energy comes from the recharging process during the dehydration, and how much of the observed energy is simply due to the restoration of the ionic conductivity, and the subsequent continuation of a REDOX process once the sample rehydrates. In other words, the well characterized property of graphene oxide having a strong dependence of conductivity on relative humidity [26], has not been adequately separated from the actual energy recharging process that requires both dehydration and rehydration.

Any moisture-based thermal kinetic energy harvesting with gradient GO frameworks (g-GOF) devices requires the dehydration or removal of water molecules from the GO matrix. However, very few papers on the subject include this part of the cycle in their experiment, and also claim that devices maintain a significant amount of energy after a number of claimed charge-discharge cycles. This may be a confusion between the relaxation discharge and the actual recharge by harvesting environmental energy during the dehydration process.

Furthermore, it has been claimed that the increase of open-circuit voltage with ambient moisture illustrates that energy is somehow being harvested from the ambient environment. When water is added, GO becomes simultaneously more chemically active as the functional groups such as COOH dissociate into COO^- and H^+ , as well as increasing its ionic conductivity. As a result of these changes, the open

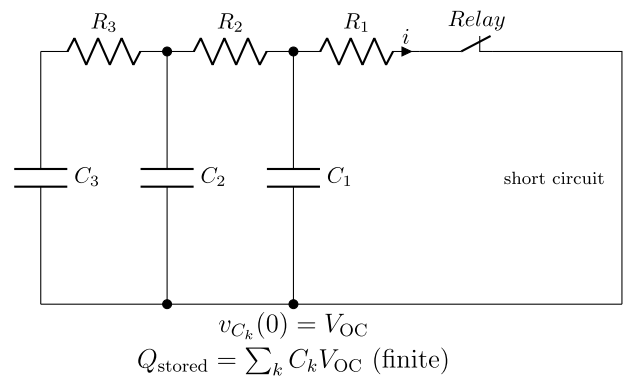


FIGURE 2. The device modeled as a finite charge reservoir: an open-terminated RC ladder pre-charged to V_{OC} . With the relay closed, the reservoir discharges through R_1 ; with it open, charge redistributes internally through R_2 and R_3 and the terminal voltage recovers without any environmental energy input.

circuit voltage increases in response. As noted previously, this may be confused with the energy harvesting part of the cycle, which actually occurs during the drying-out process of the GO when thermal kinetic energy is absorbed and COOH functional groups in GO are restored. Conversely, the decrease in the open circuit voltage with the reduced humidity can also be explained by the reduced ion mobility in GO as a function of lower moisture content. It has been well established that GO essentially becomes an insulator when moisture is removed, and therefore this does not by itself provide evidence that when a voltage is present, that it originates from harvested energy and not from the stored energy.

VI. CHEMICAL PROCESSES IN ENERGY HARVESTING DEVICES

Many papers claiming energy harvesting do not investigate the possibility that a chemical change has occurred. This change can potentially be characterized by corrosion of electrodes as well as chemical and physical changes in the GO material itself. Ours is not the only work in which electrode corrosion has been identified as a possible energy source and a cause of degradation in a harvesting device. For example, Fu et al. [27] attribute the gradual decay of their moisture generator to the formation of a passivation film on a metal electrode, and restore output only by chemically dissolving that film, expending chemical potential energy in the process and an implicit acknowledgment that the electrode reaction, rather than ambient harvesting, governs the observed current. The same concern has been raised for a range of non-GO moisture and evaporation generators. Wang et al. [28] do not specifically consider GO-metal corrosion, but they find that when active materials are used as electrodes, their contact with moisture can form a metal-air cell that irreversibly converts stored electrochemical energy into electricity, and that inert electrodes are preferable for mechanism studies because electrochemical output, unlike

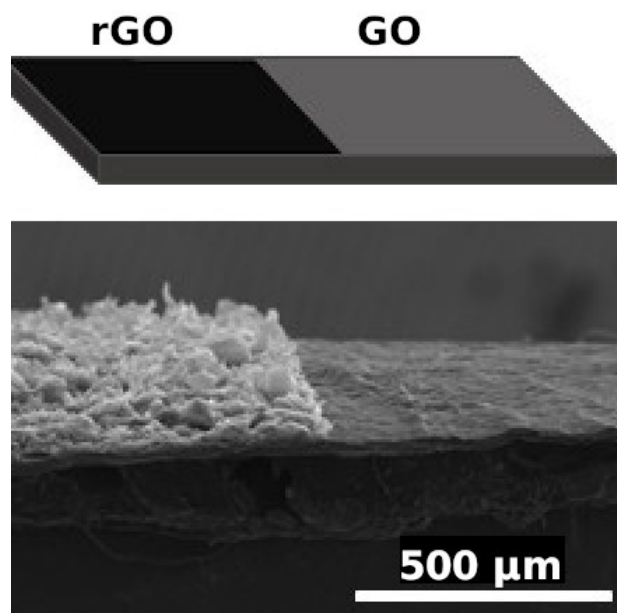


FIGURE 3. Schematic of GO-rGO junction (top) and its SEM micrograph (bottom) of one of the GO-rGO strips fabricated for this experiment. This junction ‘simulates’ many of the types of devices claiming to be demonstrating energy harvesting. Here, GO consists of layers of graphene sheets that are heavily functionalized with oxygen rich hydroxyl, epoxide, carbonyl and carboxyl groups. These groups disrupt the π -conjugated system and introduce disorder, but they also facilitate interlayer hydrogen bonding and van der Waals forces, which help maintain a compact layered structure. Reduction of GO restores sp^2 bonding, which weakens interlayer interactions and allows the layers to delaminate or separate. Furthermore, the fast reduction produced by rapid laser heating in this case also evolves gases resulting in voids in the rGO, producing the fluffy structure.

genuine harvesting, is insensitive to fluctuations in humidity or temperature. Consistent with this, several other groups have shown that replacing inert electrodes with active metals such as zinc, copper, or aluminum raises the measured output by orders of magnitude through galvanic corrosion rather than improved harvesting [29], [30], [31]. A recent review likewise lists electrode fouling among the principal degradation modes of these devices [12], conceding that reactive electrodes contribute to the measured output rather than merely collecting it.

There is a wide range of possible mechanisms by which electrochemical or ionic potentials may manifest as a tiny amount of observed energy in GO-metal devices. See [6] for examples. In the following results we illustrate not only the finite total charge, but also irreversible physical changes apparent in the GO-metal junctions after extended duration under short-circuit in the humid stable atmosphere.

VII. ENERGY EXPERIMENT

First, we synthesize graphene oxide (GO) by the modified (improved) Hummers’ method [16] and prepare the GO film by drop casting, with the thickness of the resulting films in the range of 150 μm to 200 μm . Then we use a CO_2 laser scribing technique on one half of the film’s surface to obtain

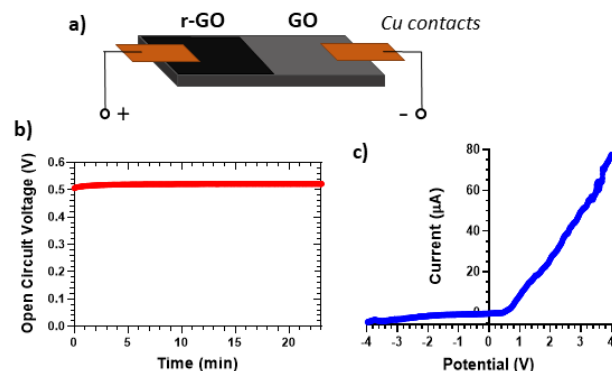


FIGURE 4. Following fabrication, the GO-rGO junction devices with an area of around 1 cm^2 are characterized using copper contacts (a), placed in a humid environment produce an open circuit voltage near 0.5 V (b). When an external potential is applied the current depends on the polarity and exhibits a polarity-dependent, rectifying diode-like response consistent with a Schottky-like interfacial barrier (c), verifying the junction structure required for our experiment.

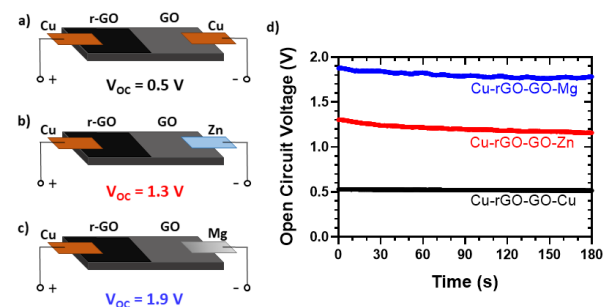


FIGURE 5. Open-circuit voltages with GO in contact with copper (Cu), zinc (Zn) and magnesium (Mg). We use the same metal with the rGO-metal junction, as the metallic nature of the rGO-metal contact makes no difference to the observed open-circuit voltage, and the different potentials are observed only with the various metal-GO contacts.

the reduced graphene oxide (rGO) [32] and therefore form the interface between GO and the rGO scribed with CO_2 laser, as schematically shown in Figure 3. The GO film contains abundant oxygen functional groups and structural defects, resulting in the material exhibiting inferior electrical/thermal conductivity and structural weakness (lower C/O ratio), with the ionic contact between the GO and the metal contact. However, fabrication using laser-induced reduction of GO, rGO is obtained with a higher C/O ratio, resulting in a much higher electron conductivity, resulting in metallic-like contact with the metal electrode. The expected asymmetric diode-like, rectifying current-voltage response of the fabricated junction is shown in Figure 4, consistent with the Schottky-like interfacial effects.

A number of metal combinations were tested in contact with both the GO and rGO faces of the sample. As expected, we observed no difference in open circuit potential when different metals are in contact with rGO because of its metallic conductive nature. However, when different metals were contacted with the GO face, we observe different

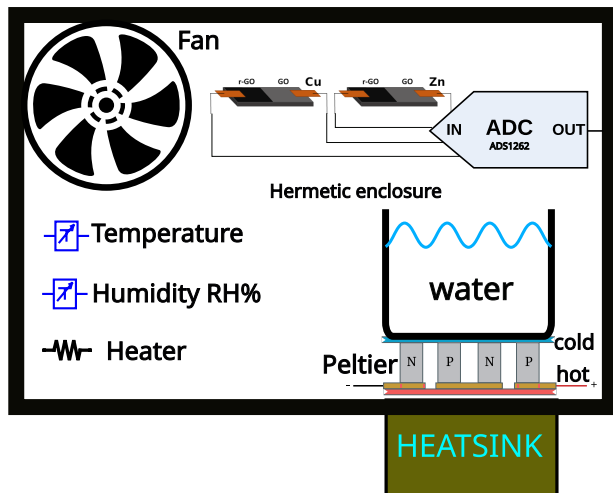


FIGURE 6. Block diagram illustrating the experimental setup. The fan ensures a homogeneous thermal and humid environment inside a sealed chamber with the humidity actively controlled using the cold side of a cycling Peltier. An SHT31 RH sensor [33] and microcontroller measure the RH inside the chamber and cycle the Peltier to keep the internal humidity near a constant 95% RH. The temperature regulation comes from the external environment. Two experiments are carried out simultaneously, measuring the charge produced by copper and zinc-GO-rGO junctions inside the controlled chamber. We found that a zero-offset amplifier in a A-D converter with a shunt resistor (10 k Ω) was better for measuring the small signals over the extended time intervals than a conventional transimpedance amplifier since its drift offset could not be calibrated, because it is a function of the electrical impedance of the sample that changes wildly during the experiment.

potentials in accordance with galvanic potential of the different metals. Figure 5 illustrates the different open-circuit potentials for copper, zinc and magnesium of 0.5V, 1.3V and 1.9V respectively. In other words, the potentials only depend on the various metal-GO contacts, with no potential differences observed between the various metals and the rGO side of the films.

Two separate GO-rGO metal junction devices that use copper and zinc in GO contact are placed in a sealed environmentally controlled chamber where relative humidity is measured and regulated at 95% using a humidity and temperature sensor (SHT31) [33], a water reservoir and a feedback loop driving a Peltier device cooling the water reservoir, as illustrated in the block diagram Figure 6. Each device has an effective short-circuit consisting of a resistive dummy load, with the potentials measured using a 32-bit ADC containing a zero-drift chopper-stabilized amplifier set to its maximum gain of 32. This serves both experiments simultaneously using the ADC's internal multiplexer. The control and data acquisition system utilizes an Arduino board with an ATmega328 microcontroller programmed using ATMEL-ICE and VScode, with the source code available as supplementary material, hosted by Codeberg [34]. Peripheral communication using I2C also included data from the SHT31 temperature and humidity transducer simultaneously acquired and stored with a ~ 5 -second time interval.

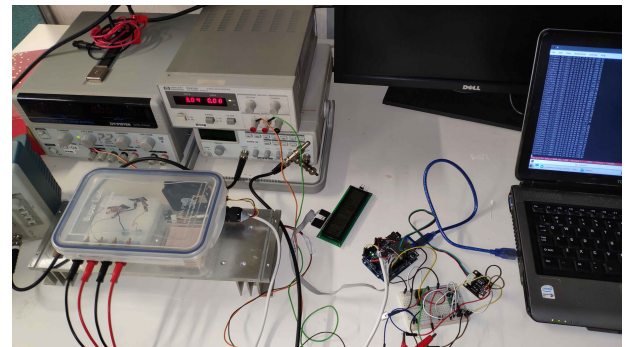


FIGURE 7. Experimental setup showing the sealed container on top of a heatsink, containing two separate metal-GO devices, with signal conditioning and data acquisition electronics shown. For redundancy, the data is simultaneously saved to a computer and an SD card.

The experiment utilizes a hermetically sealed plastic enclosure with a Peltier device protruding through the bottom. The hot side is mounted onto a heatsink on which the enclosure rests, where the cold side is in thermal contact with an open water container inside the sealed enclosure, as illustrated in Figure 7. The enclosed atmosphere is continuously homogenized using a small fan that operates at low power so as not to significantly contribute to internal heating. Since we are measuring very small voltages with temperature differences, the entire data acquisition system consisting of a Texas Instruments ADS1262 ADC board is placed inside the temperature homogenized enclosure in order to eliminate any voltage offsets caused by the Seebeck effect, which could otherwise be producing voltage offsets due to dissimilar metal contacts at different temperatures.

Although a transimpedance amplifier arrangement would provide an ideal short circuit load for this experiment, we found that using a resistive dummy load providing an effective, although non-ideal short circuit, to be a more practical solution to the difficulty of measuring very small signals over a long time interval. The problem with the transimpedance amplifier with a high and variable source impedance, is that any offset due to the operational amplifier is determined by the resistance of the current source, which in our case is the metal-GO junction. If we try a transimpedance amplifier, we observe varying offset due to the junction impedance increasing by an order of magnitude, to over 1 M Ω , as the experiment progresses. Therefore, we utilized a stable 10 k Ω load as a suitable approximation to a short circuit for this experiment, and measuring the voltage directly using a zero-offset amplifier and ADC. As a calibration for the offset, we include a relay contact, K1, as illustrated in Figure 8, which is operated remotely to check system offset without significantly disturbing the experiment.

The experiment is carried out over an extended period of time, running continuously for more than 1,300 hours, or nearly two months. The experiment began by measuring the open-circuit voltage of a rGO-GO-metal device in a stable 95% RH humid environment, followed by short-circuit

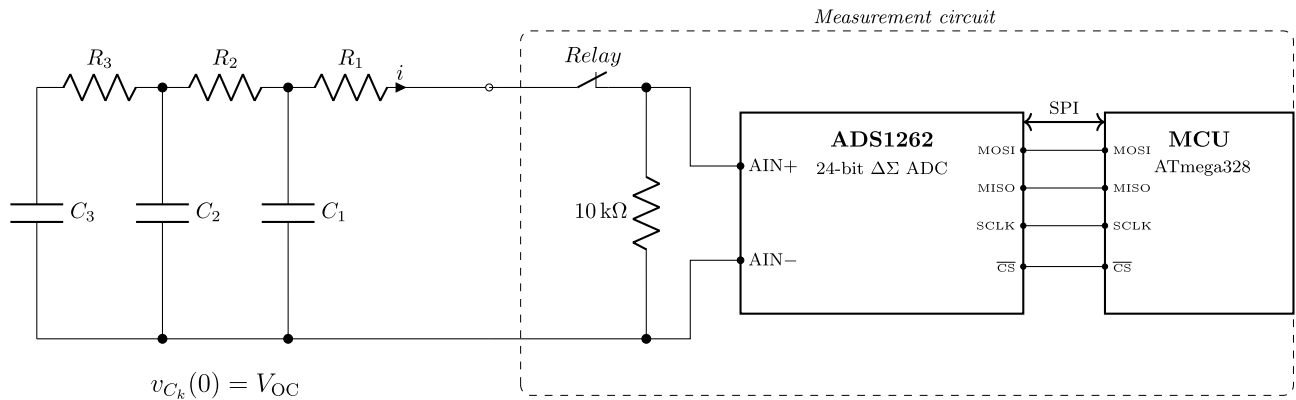


FIGURE 8. Equivalent circuit and measurement system. The device is an open-terminated RC ladder of N sections (R_k, C_k), discharging through a $10\text{ k}\Omega$ sense resistor sampled by the ADS1262 ($N=3$ for zinc, $N=2$ for copper). The model contains no sustained EMF element, and the experimental results show the fitted current decaying to zero.

current measurement over the extended time period, as illustrated in Figures 9 and 10. During this time, the short-circuit current produced by two metal-GO-rGO junctions are measured, representing the total charge produced by each device. In other words, we simultaneously carry out two separate experiments: One utilizing copper, while the other zinc.

Given the sensitivity of graphene to ambient humidity in terms of its ability to harvest energy from the displacement and restoration of water molecules in the GO, as well as its ionic and electronic conductivity, the relative humidity (%RH) inside the enclosed container has to be controlled. This is achieved using a chilled water bath, with the temperature of the water regulated to keep the %RH at a constant 95%.

We can demonstrate this method by considering the saturation vapor pressure of liquid water at a given temperature, T , that can be empirically calculated using the improved simple formula developed by Huang [35],

$$P_s = \frac{\exp\left(34.494 - \frac{4924.99}{t+237.1}\right)}{(t+105)^{1.57}} \quad (t > 0^\circ\text{C}) \quad (1)$$

where P_s is saturation pressure in Pascal and t is temperature in $^\circ\text{C}$. By cooling the water reservoir inside the enclosure, the humidity is controlled according to the principles of psychrometrics and the relationship between saturation vapor pressure and temperature given by Equation 1.

Using this equation, the required water surface temperature to maintain a relative humidity of precisely 95% inside the sealed chamber was calculated to be $\sim 1^\circ\text{C}$ below the ambient air temperature of $\sim 22^\circ\text{C}$. Without active cooling, the enclosed air in contact with the water surface would naturally equilibrate close to 100% RH. To avoid condensation and maintain a stable, well-defined humidity environment, a setpoint of 95% RH was selected, achieved using the Peltier cooling device described above.

Humidity stabilization is implemented using a simple on/off control loop: the Peltier chiller was activated whenever

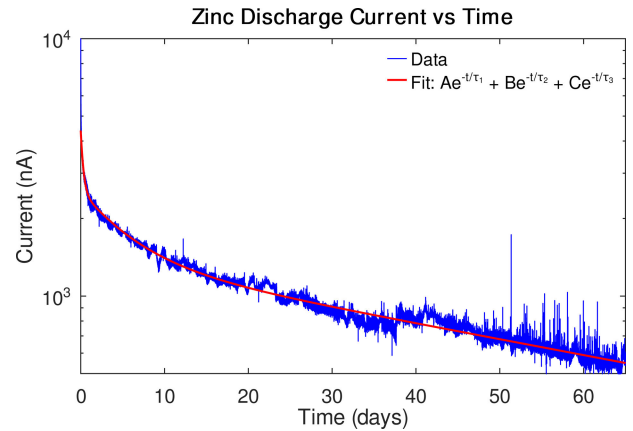


FIGURE 9. A comparison of the zinc data with a fitted model.

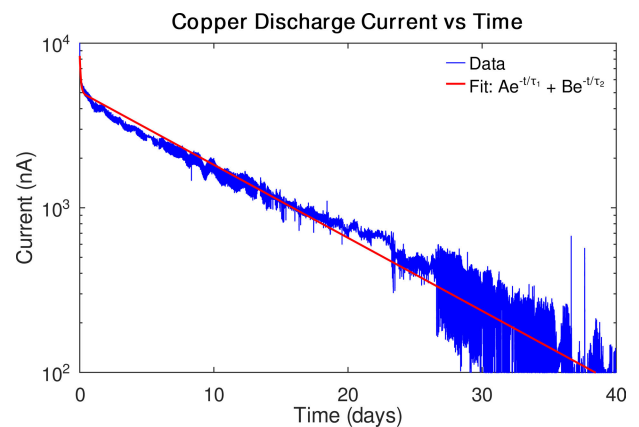


FIGURE 10. A comparison of the copper data with a fitted model. Note how the junction begins to fail and becomes unstable after ~ 25 days.

the RH exceeded the 95% setpoint, causing the water surface temperature to drop and the RH to fall accordingly, and is switched off once the RH returns below the setpoint. Direct measurement and control of the RH makes it unnecessary



FIGURE 11. GO-rGO sample where some areas of the GO face on the left half of the sample were in contact with copper under short circuit, while other areas do not contact the metal. We compare the carbonyl absorption of these two areas.

to monitor the water temperature independently. A small fan operating at low speed gently circulated the air within the chamber to ensure uniform humidity distribution, with negligible contribution to the overall thermal load. Over the course of the experiments, the temperature inside the enclosure fluctuated by less than 2°C due to ambient laboratory conditions, while the RH is maintained to within $\pm 0.5\%$.

VIII. COMPOSITION EXPERIMENT

A GO-rGO sample with copper contacts is held in a stable humid environment with a short circuit for two weeks in a prior experiment, and subsequently analyzed using IR AFM spectroscopy spanning $5.5\ \mu\text{m}$ to $8.3\ \mu\text{m}$ using the Attocube Atomic Force Microscope (AFM) with IR-neasCOPE+s from Neaspec [36]. This spectral range is selected to target the carbonyl absorption resonance, where differences in oxygen content and chemical composition are evident in GO. Eight surface locations are analyzed: four points from copper-contacted regions and four points from pristine areas, with the points randomly selected. Figure 11 shows the actual sample we used for this measurement, with GO on the left and rGO on the right. Slight surface morphology changes are visible where the copper alligator clip was in contact with the GO on the left side of the sample to create the depleted region, while the un-contacted region of the GO side of the sample was considered pristine for the purpose of this measurement.

As a further indication of the the electrochemical changes that result from the extended 2-month discharge experiment, Figure 12 illustrates the physical changes in the metal and GO with which it was in contact, at the end of the extended experiment.

Figures 13 and 14 show broadband IR images and the surface reflection, as well as the AFM surface textures. Unlike the long-term two month experiment, there was no adhesion between the metal and GO.

Figures 15 and 16 show the reflection amplitude and phase of each of the four points in the two regions. The spectra from

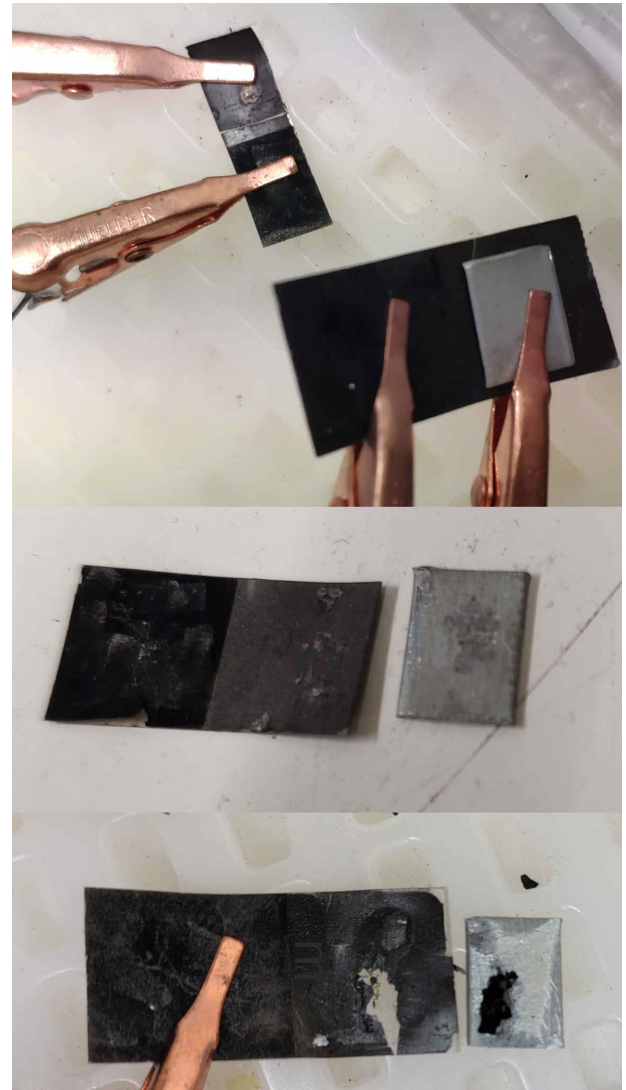


FIGURE 12. Indication of physical changes observed over time with metal in contact with graphene oxide under short circuit. The top image shows corrosion of the GO after contact with copper over extended period of time. The middle image shows corrosion on the zinc as well as the GO after only a week of contact. The bottom image shows complete fusion and delamination of the GO on zinc after 8 weeks. The copper-rGO junction is a metallic contact with no potential or electrochemical activity.

the pristine regions also show weaker reflection, while the phase delay indicates stronger absorption and hence a higher carbonyl concentration than the depleted region where copper is contacted in the humid, shorted state.

IX. DISCHARGE EXPERIMENT

The distinct open-circuit potentials observed across the different metal-GO junctions, illustrated in Figure 5, already suggest a significant electrochemical contribution to the measured electrical output. In this experiment we use total charge as a proxy for energy, exploiting the fact that charge, like energy, is also a fundamentally conserved quantity. Since copper-GO is the electrode configuration most commonly employed in published moisture-electric generator studies,

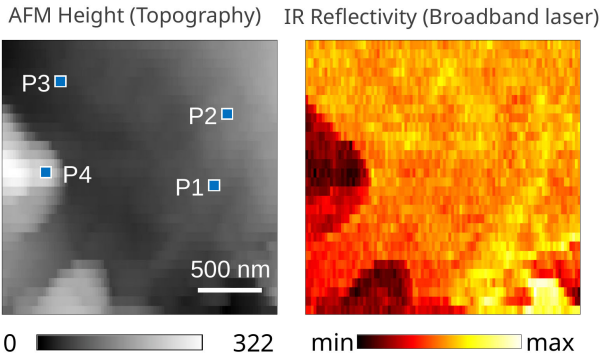


FIGURE 13. GO IR image and profile of GO pristine region showing relatively weaker overall reflection than the depleted GO region.

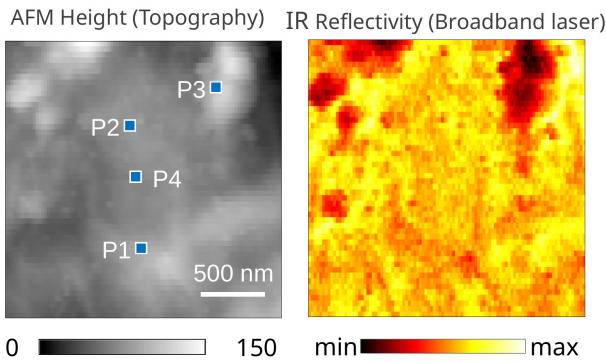


FIGURE 14. GO IR image and profile of the depleted GO region after copper contact, showing relatively stronger overall reflection than the pristine region.

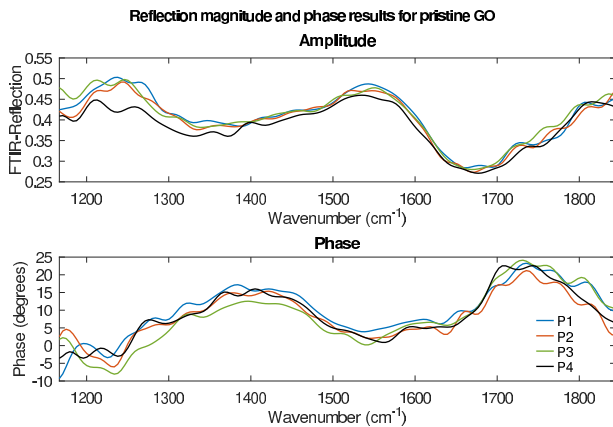


FIGURE 15. Spectra of pristine GO region showing more absorption with phase lag up to 25° and lower IR reflection than the depleted GO region with four points selected at random as a representative sample of surface variations.

it is the primary focus here, though qualitatively similar discharge behavior is observed with zinc-GO junctions. In both cases, the short-circuit current decreases monotonically with time, with only minor fluctuations consistent with changes in electrochemical activity as described by the Nernst equation.

The total charge observed at the copper-GO junction is approximately 0.4 mAh/mm³ as calculated in Appendix A.

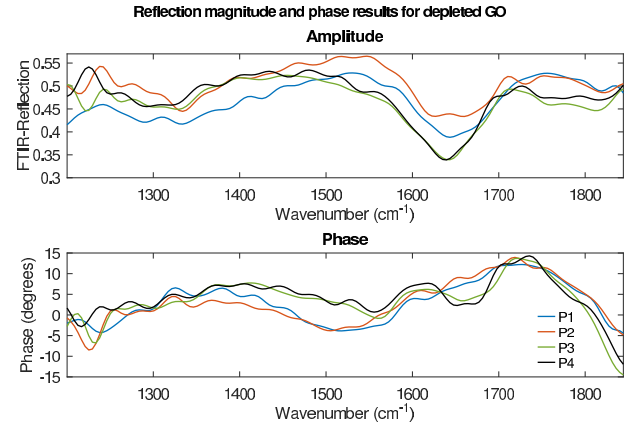


FIGURE 16. Spectra of depleted GO region showing less absorption with phase lag less than 15° and higher IR reflection than the pristine GO region with four points selected at random as a representative sample of surface variations.

Interestingly, this is similar to an AA alkaline battery that is approximately 0.34 mAh/mm³. See Appendix B.

X. ANALYSIS OF THE MEASUREMENTS AND DEVICE MODEL

The purpose of this section is to fit an equivalent circuit model to the acquired discharge data in order to identify which electronic model best describes the observed behavior. We find that the data is well described by the electrochemical model due to Randles [25], who proposed an equivalent circuit comprising a Thévenin source, a solution resistance, a parallel RC network, and a diffusion impedance element, as also discussed in Bockris et al. [37]. Here we retain only the diffusion element of this circuit, in the form of the open-terminated RC ladder shown in Figures 2 and 8.

The discharge current evolves as a superposition of exponentially decaying components, each corresponding to a distinct RC time constant in the equivalent circuit. This multi-exponential form arises from the ladder network structure of the circuit based on a reduced Randles model, where each RC stage introduces an additional time constant.

The monotonically decreasing short-circuit current observed over nearly two months, with no evidence of recovery or sustained baseline, is the expected temporal signature of a finite electrochemical reservoir discharging through a resistive load, and is inconsistent with continuous energy harvesting, which would require a non-zero and sustained output. We fit the data to a sum of decaying exponential components, using two terms for the copper channel and three for the zinc channel,

$$i(t) = C + \sum_{k=1}^N A_k \exp(-t/B_k) \quad (2)$$

where $N = 2$ for copper and $N = 3$ for zinc, t is time, A_k are amplitude coefficients, B_k are the characteristic decay time constants, and C is a constant offset representing any residual steady-state current.

The near-perfect agreement between the fitted model and the measured discharge curves spanning more than six orders of magnitude in time constant, constitutes strong quantitative evidence that the device behaves as a conventional multi-stage electrochemical cell.

We find that $N = 2$ for copper and $N = 3$ for zinc were the lowest-order models that reproduced the measured discharge to within the noise, with higher orders yielding no significant improvement in fit. Within these models, the least-squares fit returned $C = 0$ to within the noise floor in both channels, so the integral of Equation 2 to $t \rightarrow \infty$ converges to a finite charge; That the freely-fitted offset C vanishes is thus not an assumption but the empirical result on which the finite-reservoir interpretation rests, consistent with complete depletion of a finite stored charge. The equivalent circuit is shown in Figure 2, with $V_{oc} \approx 0$ at $t = 0$.

A. ZINC CHANNEL

Parameters in Equation 2 estimated by least squares are:

$$\begin{aligned} A &= [1831.97, 1176.95, 1382.49] \text{ (nA)}, \\ B &= [25643, 497508, 6084642] \text{ seconds}, \\ C &= 0. \end{aligned}$$

Integration of Equation 2 with the above values for $t \rightarrow \infty$ yields

$$Q = \int_0^\infty i(t) dt \approx 2.51 \text{ mAh} \quad (3)$$

B. COPPER CHANNEL

Parameters in equation 2 estimated by least squares are:

$$\begin{aligned} A &= [3305.51, 5062.28] \text{ (nA)}, \\ B &= [6688, 846125] \text{ seconds}, \\ C &= 0. \end{aligned}$$

Integration of Equation 2 with the above values for $t \rightarrow \infty$ yields

$$Q = \int_0^\infty i(t) dt \approx 1.19 \text{ mAh} \quad (4)$$

The equivalent circuit of Randles-type [25] is selected because it is the physically motivated model for a metal-GO half-cell of the kind realized here and is independently supported by the measured galvanic open-circuit potentials, the observed electrode corrosion, and the AFM-IR evidence of GO reduction. We acknowledge, however, that a sum of decaying exponentials is a flexible functional form that will fit most smooth monotonic decays, and that the quality of the fit alone does not establish this circuit as the unique mechanism, as other decay models carrying different physical interpretations may fit comparably well. However, our central conclusion does not depend on this choice. The distinction between stored and harvested energy established in Equation 5 rests only on whether the integrated charge converges to a finite value as $t \rightarrow \infty$, which holds for any model in which the output decays without a sustained baseline. The exponential fit serves here as a convenient

means of extrapolating the measured tail to estimate the total charge, not as a unique identification of the underlying physics.

XI. DETECTION OF HARVESTED ENERGY

The instantaneous generation from any device can be expressed as Equation 5, Here, P_t is generated power (watts) or current (A), K_m is a positive scale factor specific to each device or technology, $Z(t)$ is a normalized positive random variable that is a function of time, such as, e.g. wind or solar. Note that $F(t)$ is a normalized function of power or current, and therefore $\frac{1}{2}(F(t) + 1)$ spans the interval between zero and unity. We can now write down the energy or charge produced over an interval of time (τ) as $E(\tau) = \int_0^\tau P(t) dt$ where

$$P(t) = K_m \cdot Z(t) \cdot \frac{1}{2} (F(t) + 1). \quad (5)$$

where $Z(t)$ represents the instantaneous availability of the external ambient resource (e.g., wind or insolation), a normalized quantity that fluctuates but is not depleted by the device. In contrast, $F(t)$ represents the state of any internal reservoir: for a genuine harvester $F(t)$ and therefore $\frac{1}{2}(F(t) + 1)$ remain constant over time, whereas for a device drawing on stored energy $\frac{1}{2}(F(t) + 1)$ decreases toward zero as the reservoir is depleted. The factor $\frac{1}{2}(F(t) + 1)$ thus spans the interval between full depletion and full availability. Assuming that the efficiency of the device remains constant over time, the device constant K_m is independent of time. Therefore, by integrating Equation 5 for τ approaching infinity, we can show that for a true energy harvesting device the total amount of energy (or charge) harvested must also approach infinity. However, for a stored energy device, the total amount of energy (or charge) must always approach a finite value. In other words, as time tends to infinity, so will the energy harvested, i.e. $E(\tau) \xrightarrow{\tau \rightarrow \infty} \infty$. Using this principle to distinguish between stored and harvested energy by fitting the measured function $P(t)$ to a mathematical model, we can thus formally distinguish between energy that is stored and released from a limited finite reservoir, and unlimited energy harvested from the external environment.

XII. CONCLUSION

In this paper we measure the total charge produced in a stable homogeneous environment, and find that negligible harvesting is taking place, and that the energy generated most likely originates from an electrochemical process that also produces some physical changes in the materials. We also carry out spectroscopic imaging and analysis indicating reduction of GO. These results cast some doubt on some of the claims about harvested energy from devices that employ non-noble metal junctions such as copper, silver and aluminum in contact with graphene, and that further work needs to be carried out on the stability and longevity of these devices. Furthermore, a clearer understanding of the energy sources in energy harvesting experiments needs to be communicated.

APPENDIX A

CALCULATION OF CHARGE DENSITY GO-METAL DEVICE

Let us consider only the GO taking part in the reaction, ignoring the metal oxidation entirely and underestimating the total volume involved, which overestimates the charge density. The volume of GO is approximately,

$$V = 0.15 \text{ mm} \times 20 \text{ mm}^2 = 3 \text{ mm}^3.$$

Integrating the short-circuit current to infinity using either the Zn and Cu battery model fits for the acquired data, we obtain;

$$\int_0^\infty I \, dt \approx 2.51 \text{ mAh, for Zn and}$$

$$\int_0^\infty I \, dt \approx 1.19 \text{ mAh, for Cu,}$$

therefore $\rho \approx 0.8 \text{ mAh/mm}^3$ for Zn and $\rho \approx 0.4 \text{ mAh/mm}^3$ for Cu.

APPENDIX B

CALCULATION OF CHARGE DENSITY OF AA ALKALINE BATTERY

The charge density ρ is the specified charge Q divided by the volume or $\rho = Q/V$, where the volume V of AA type battery $V = \pi r^2 L$, with $r = 7.25 \text{ mm}$ and $L = 50 \text{ mm}$, which yields $V = 7.7 \times 10^3 \text{ mm}^3$. Given the capacity of a AA alkaline battery $Q = 2800 \text{ mAh}$ [38], gives a charge density of $\rho = 0.3391 \text{ mAh/mm}^3$. It is interesting to note that this is of the order of the charge density calculated for GO, above in Appendix A.

DECLARATION OF GENERATIVE AI AND AI-ASSISTED TECHNOLOGIES

The authors declare that no generative AI was used to form the prose of this manuscript. However, GTP-5 was used as a productivity tool for assisting in L^AT_EX debugging and sorting references into correct numerical order. It was used for suggesting a manuscript structure in terms of section headings, but this was modified and corrected by the authors. It was also used as a research tool for finding references and structuring the bibliography.

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