Ceylon Graphite Outperforms Other Graphite Materials in a Groundbreaking Graphene-based Sensing Platform for Chemical Analysis Study Conducted by Imperial College London

19.07.2023 | GlobeNewswire

Vancouver, July 19, 2023 - Ceylon Graphite Corp. ("Ceylon") (TSX-V: CYL) (OTC: CYLYF) (FSE: CCY) is pleased to announce that Ceylon graphite achieved new concentration and conductivity records when studied in the manufacture of an adaptable sensing platform for chemical sensing. The research, published in the Royal Society of Chemistry's "Nanoscale" Journal, was conducted by partners at the Molecular Sciences Research Hub at Imperial College London and specifically incorporated Ceylon's vein graphite to produce a low-surface-tension sprayable graphene ink that was key to the sensor's functionality.

A summary of the test results, completed in January 2023[1] is highlighted below:

- Ceylon graphite was used to create high concentration, graphene/polyvinylpyrrolidone inks, with record-breaking concentrations as high 3.2 mg mL?[1].
- Raman spectroscopy was used to show high-quality graphene flakes produced via liquid phase exfoliation.
- The Ceylon-based graphene device was successfully used to detect for pH within the range of pH 3 - 11.
- These results demonstrate the potential of high-quality graphite to empower the next generation of nanomaterial-based diagnostics for biological and chemical sensing.

"Our findings highlight the promising pH sensing capabilities of the Ceylon graphene-based devices for pH sensing, which can be deployed for a variety of medical and environmental applications," said Dr. Felice Torrisi, corresponding author of this work and principal investigator. "In particular, the sprayed "Electrolyte-gated Graphene Field-effect transistor" (EG-GFET) fabricated using Ceylon graphite outperforms any other EG-GFET prepared by any other technique, demonstrating the unique characteristics of Ceylon graphite for high quality graphene inks with electronic grade suitable for large area printed electronics, integrated circuits and sensing. We see this as a breakthrough with Ceylon vein graphite aiming to uncap the potential of graphene inks for printed electronics by demonstrating high-performance devices suitable for applications ranging from flexible and wearable electronics to sensing and automotive."

"We are thrilled that world-leading researchers have discovered the advantages of our high-carbon vein graphite and its potential applications in the worlds of graphene and nano-technologies," said Ceylon CEO, Sasha Jacob. "This is a key area of our future development and one that provides high-margin value-added products to our portfolio."

Dr. Felice Torrisi is a Senior Lecturer in Chemistry of 2D materials and Wearable Electronics in the Department of Chemistry at Imperial College London and Fellow of Trinity College, Cambridge. He previously held a University Lectureship in Graphene Technology in the Department of Engineering at the University of Cambridge, where he jointly managed the Centre for Doctoral Training in Graphene Technology and the Cambridge Graphene Centre.

Results in Summary:

Graphene ink formulation

Graphene inks have emerged as a new revolutionary element for high-performance printed, flexible and wearable electronics.[2] Among the various methods available for preparing graphene ink, sonication-assisted liquid-phase exfoliation (LPE) has been chosen due to its simplicity and compatibility with low-boiling solvents. This process involves subjecting graphite (in powder or flakes) and low-boiling point solvents, such as 2-propanol (IPA), along with small amounts (20 mg) of the polymer stabiliser, resulting in a graphene ink with desired electronic propertied for printed electronics and significantly enhanced shelf life of the ink. IPA was selected as the solvent for the graphene ink as it has a boiling point of 82 °C and impressively low surface tension of only 20.34 mN $m^{?1}$, satisfying the criteria for a scalable spray-coating as well as inkjet printing of the optimised ink.[3] The sonication process lasts for 9 hours, ensuring thorough exfoliation of the graphite flakes. Centrifugation at 2.000 - 13,000 g is then employed to further refine the ink and effectively eliminate any remaining unexfoliated flakes.

The optical absorption spectrum (OAS) of the graphene ink, as depicted in Figure 1a, exhibits the characteristic profile associated with graphene inks, a flat absorption pattern in the visible spectrum and a distinctive peak in the UV region, confirms the ink is mainly composed by high-quality graphene flakes. The concentration of graphene flakes in the ink is estimated to be ~ 1 mg mL^{-1} when centrifuged at 13,000 g and as high as 3.2 mg $mL^{?1}$ when centrifuged at 2,300 g (Figure 1b). This concentration surpasses those reported in the literature for graphene inks stabilised by polyvinylpyrrolidone (PVP) by an order of magnitude, underscoring the exceptional quality and potential of this formulation.

Please click here to view image

Figure 1: Ceylon inks show optimal graphene/PVP ink concentrations. a) OAS data for Ceylon-based graphene/PVP ink. b) Ceylon-based graphene/PVP ink can accommodate > × 3 the flake concentration than previously reported inks when prepared under similar conditions.

Application: A graphene field-effect transistor as a scalable and low-cost high-performance biosensor

The EG-GFET channel is formed using an automatic spray-coating process, ensuring consistent and scalable deposition of the graphene ink onto the PCB test strip. The graphene ink exhibits excellent wetting properties that contribute to film uniformity. As the individual ink droplets merge into a thin film before evaporating, this wetting behaviour plays a vital role in achieving uniformity.

While the addition of PVP stabilizer enhances the concentration and stability of the ink, it is important to note that PVP is known to adversely affect the electrical conductivity of nanostructured graphene thin films due to its insulating properties. However, a solution has been found by utilizing a xenon intense pulsed light (IPL) source, which effectively degrades the PVP polymer without subjecting the PCB substrate to temperatures exceeding its decomposition threshold. This method proves to be the most suitable for this specific application. The spray coated graphene ink achieved an approximate channel resistivity of 100 ? after IPL annealing suitable for flexible and plastic electronics and currently used in industry.

Please click here to view image

Figure 2: Photonic annealing of Ceylon-based graphene improves electrical properties. a) Raman spectroscopy data indicating the absence of noticeable modification upon photonic annealing. b) IPL annealing causes a decrease in resistance of the graphene film.

The quality of the graphene flakes is assessed using Raman spectroscopy. Figure 2a displays typical Raman spectra of the graphene ink deposited on Si/SiO_2, before and after photonic annealing (black and red curves, respectively), to monitor any potential effects on the SLG/FLG flakes. The red and black curves in Figure 2a exhibit the characteristic D peaks at approximately 1346 $cm^{?1}$, 2D peaks at approximately 2690 $cm^{?1}$, and G peaks at approximately 1581 $cm^{?1}$ (red) and 1580 $cm^{?1}$ (black). The D peak displays a full-width at half maximum (FWHM) of 37.9 $cm^{?1}$ (red) and 38.9 $cm^{?1}$ (black). These values align with those reported for LPE graphene inks indicating the high quality of the SLG and FLG flakes in the graphene ink and the absence of noticeable modifications upon photonic annealing.

To assess the impact of photonic annealing on the electrical resistance of the EG-GFET channel, the PCB test strip is exposed to three different intensities of xenon IPL (IPL) energy. The exposure at 2.5 J $cm^{?2}$, 3.75 J $cm^{?2}$, and 5.0 J $cm^{?2}$ results in a similar decrease in resistance from 310 ? (not annealed, red curve) to 112 ?, 108 ?, and 115 ?, respectively. Consequently, the lowest IPL energy (2.5 J $cm^{?2}$) is employed for all subsequent experiments, ensuring an enhanced channel resistance while minimising the risk of PCB damage.

The response of the EG-GFETs to variations in pH was investigated by conducting experiments that involved altering the pH of the solution using a strong base or acid, while monitoring the corresponding response. The obtained results reveal valuable insights into the pH sensitivity of the EG-GFETs. Figure 3a illustrates the relationship between the drain current (I_D) and the gate-source voltage (V_{GS}) for the EG-GFETs exposed to pH values ranging from 3 to 11. Notably, the plot exhibits a discernible shift of the Dirac point from 60 mV to 270 mV, indicating the sensitivity of the devices to changes in pH. It is important to highlight that this pH sensitivity is attributed to the type and density of unintentional defects introduced during the LPE process.

Please click here to view image

Figure 3: pH response of Ceylon-based graphene chemical sensor. a) Shift in characteristic electrical transfer curves of EG-GFETs to changing solution pH. b) Transient pH change within the range of 7.2 - 7.5 pH units.

The corresponding pH-dependent shift in the Dirac point is depicted in Figure 3a, showcasing a maximum Dirac point of 270 mV at pH 11. A linear fit analysis of the Dirac point values (red dashed line) reveals a sensitivity of 25.8 ± 0.5 mV per pH within the linear pH range of 11 to 3. While this sensitivity falls below the theoretical maximum predicted by the Nernst equation (59.16 mV per pH), it outperforms graphene pH sensors prepared using alternative graphene fabrication techniques, such as chemical vapor deposition (CVD)-grown graphene on SiO2 (21-22 mV per pH), suspended graphene (17 mV per pH), epitaxial graphene on silicon carbide (19 mV per pH), and mechanically exfoliated graphene on SiO_2 (20 mV per pH). Moreover, transient pH changes observed in Figure 3b, show how the graphene devices respond to pH changes in < 10s, with a resolution as small as 0.04 pH units. These findings highlight the promising pH sensing capabilities of the Ceylon graphene-based devices for pH sensing, which can be deployed for a variety of medical and environmental applications.

Concluding remarks:

Ceylon has the correct characteristics to become a key player in graphene ink preparation, and achieved the highest concentrations of graphene flakes in the inks, as estimated using OAS. This high concentration of graphene flakes offers several advantages during the spray coating process. Firstly, it promotes improved uniformity in the deposition of the graphene ink. Additionally, the increase in graphene to PVP ratio leads to enhanced flake conductivity, as excessive PVP deposition can hinder conductivity. Raman spectroscopy analysis of the functionalized graphene ink demonstrated results consistent with those observed in LPE graphene, indicating a low defect area. This characteristic further enhances the overall quality and performance of the graphene ink.

The combination of these unique properties enabled the successful detection of pH using a Lab-on-PCB architecture for the first time. The graphene-based sensor exhibited a pH sensitivity of 25 mV per pH unit, showcasing its ability to precisely measure pH variations. Furthermore, the response times of the sensor were found to be less than 10 seconds, highlighting its rapid and efficient performance. This breakthrough in pH sensing utilizing the Lab-on-PCB platform demonstrates the potential of Ceylon-derived graphene in enabling advanced sensing technologies

About Ceylon Graphite Corp.

Ceylon is a public company listed on the TSX Venture Exchange, that is in the business of mining for graphite, and developing and commercializing innovative graphene and graphite applications and products. Graphite mined in Sri Lanka is known to be some of the highest grade in the world and has been confirmed to be suitable to be easily upgradable for a range of applications including the high-growth electric vehicle and battery storage markets as well as construction, healthcare and paints and coatings sectors. The Government of Sri Lanka has granted the Ceylon's wholly owned subsidiary Sarcon Development (Pvt) Ltd. an IML Category A license for its K1 mine and exploration rights in a land package of over 120km^2. These exploration grids (each one square kilometer in area) cover areas of historic graphite production from the early twentieth century and represent a majority of the known graphite occurrences in Sri Lanka.

Further information regarding Ceylon is available at www.ceylongraphite.com

Sasha Jacob, Chief Executive Officer and Rita Thiel, Chair of the Board of Directors

info@ceylongraphite.com

Corporate Communications

+1(604) 924-8695

Neither TSX Venture Exchange nor its Regulation Services Provider (as that term is defined in policies of the TSX Venture Exchange) accepts responsibility for the adequacy or accuracy of this release

FORWARD LOOKING STATEMENTS:

This news release contains forward-looking information as such term is defined in applicable securities laws, which relate to future events or future performance and reflect management's current expectations and assumptions. The forward-looking information includes statements about the potential value of graphene inks produced with Ceylon graphite, applications for future graphene ink technologies, Ceylon's role as a potential market leader in graphene ink technology preparation, expectations related to development of Ceylon's properties, strategic partnerships, potential customers and sales, plans for Ceylon's subsidiaries and Ceylon's mining operations. Such forward-looking statements reflect management's current beliefs and are based on assumptions made by and information currently available to Ceylon e, including the assumption that, there are no material adverse changes effecting development and production at the M1 mine or on other properties, testing related to the performance of Ceylon's vein graphite material are accurate, there will be no material adverse change in graphite and metal prices, there will be continued demand for graphite powered batteries, all necessary consents, licenses, permits and approvals will be obtained, including various Local Government Licenses. Investors are cautioned that these forward-looking statements are neither promises nor guarantees and are subject to risks and uncertainties that may cause future results to differ materially from those expected. Risk factors that could cause actual results to differ materially from the results expressed or implied by the forward-looking information include, among other things, the results of Ceylon's graphite testing being inaccurate or incomplete, the market for graphene ink related technologies not developing as expected, failure to obtain or maintain patents and proprietary technology, loss or failure to acquire available high quality graphite, any failures to obtain or delays in obtaining required regulatory licenses, permits, approvals and consents, an inability to access financing as needed, a general economic downturn, a volatile stock price, labour strikes, political unrest, changes in the mining regulatory regime governing Ceylon, a failure to comply with environmental regulations and a weakening of market and industry reliance on high quality graphite. Ceylon cautions the reader that the above list of risk factors is not exhaustive.

---

[1] Fenech-Salerno, B., Holicky, M., Yao, C., Cass, A. E. G. & Torrisi, F. A Sprayed Graphene Transistor Platform for Rapid and Low-Cost Chemical Sensing. *Nanoscale* 15, 3243-3254. (2023).

[2] Torrisi, F. & Carey, T. Graphene, related two-dimensional crystals and hybrid systems for printed and wearable electronics. *Nano Today* 23, 73-96 (2018).

[3] Lefebvre, A. H. & Mcdonell, V. G. General Considerations. in *Atomization and Sprays* (eds. Brenn, G., Hung, D. L. S., Herrmann, M. & Chigier, N.) 1-16 (Taylor & Francis Group, 2017).