High On-Off Ratio Graphene Switch via Electrical Double Layer Gating

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ABSTRACT This paper discusses the production and investigation of novel graphene switches by gating through an electrical double layer (EDL). Controlled voltage biases across a liquid dielectric and graphene induce electrochemical reactions within the dielectric and produce high electric fields in an EDL at the surface of graphene. As the electrochemical reactions occur within the dielectric, the EDL strength separates the electrochemically-produced ions based on their polarity, and provides the necessary molecular activation and deactivation energies to form weak, reversible molecular bonds between the produced ions and graphene. The reversible bonds between the ions and graphene are used to dynamically alter the electronic transport through graphene, which introduces an exciting assortment of device possibilities. Whereas traditional graphene devices are unuseful for electronic switches or digital logic due to an insufficient bandgap of graphene, the presented graphene electrochemical field-effect transistors (GEC-FETs) exhibit ON-OFF ratios larger than 10^4 with OFF-resistances as high as 10 MΩ. Channel current, gate voltage, and dielectric medium are varied and compared to show their effect on device performance. The presented device and associated techniques show potential for integration in graphene digital-logic architectures.

INDEX TERMS Electrical double layer, electrochemical, field effect, graphene, liquid dielectric, reversible, switch, transistor.

I. INTRODUCTION

Graphene is renowned for its unique physical, electrical, and thermal properties. Its ultra-high carrier mobility, zero bandgap, and high tensile strength enable the development of novel devices such as flexible sensors, broadband detectors, and piezoresistive devices [1]–[3]. Despite these remarkable properties, there is still an abundance of unexplored research areas for graphene. A challenge for graphene in comparison to silicon-based electronics is the lack of a bandgap in graphene that makes it impossible to fully turn off and prevents switching applications that are fundamental to digital CMOS [4].
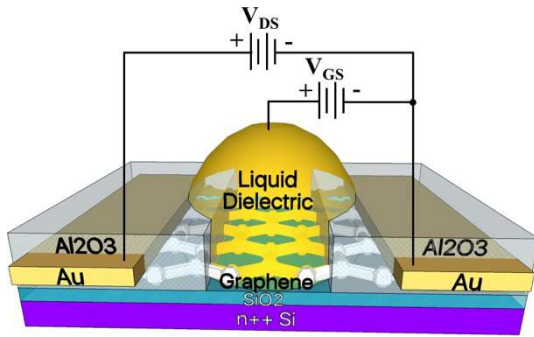
The monolayer structure of graphene allows it to be sensitive to electrostatic perturbations at its surface [5]. This observation has led to developments in graphene-based chemical

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and biological sensors that experience a change in electrical properties when exposed to the targeted chemical or biological agent [6], [7]. Researchers have purposely chemically modified graphene to form materials such as graphane, which is insulating rather than conductive [8], [9].

It has been demonstrated that graphene field-effect transistors utilizing 20nm-thick silicon-dioxide (SiO_2) dielectrics can be used to electrochemically modify graphene in order to create a nominal bandgap and enable electrical switching [10]. Although this method is reversible and can be used for non-volatile memory, the switching times were slow ($\sim$10s), and reliability was an issue. Furthermore, graphene-on-thin-SiO_2 has no utility for flexible device applications, which have experienced an increasing demand in the graphene electronics sector [11], [12].

Electrical double layer (EDL) dielectrics have seen a recent surge in interest due to their ability to produce large capacitances across nanometer-sized electrolyte-electrode

FIGURE 1. Cross-sectional view of graphene electrochemical field-effect transistor.

junctions [13], [14]. EDLs are able to form large electric fields ($\sim \mu F/cm^2$) and efficiently control the accumulation of charge carriers and ions in electronic devices [13], [14]. Furthermore, the liquid nature of EDL dielectrics provides a conformal structure that can be easily integrated into wearable and flexible electronics [2]. Different liquid dielectrics can be rapidly integrated and tested with graphene to optimize targeted performance characteristics [15].

Our team pioneers the research area of incorporating EDL dielectrics in Graphene Electrochemical Field-Effect Transistors (GEC-FETs) to produce faster electrical switches and reversible, reconfigurable graphene devices.

II. METHODS

Graphene field-effect transistors (GFET-S20 for Sensing) were commercially acquired from Graphenea. As shown in Fig. 1, the circuit comprised of a top-gated GFET array on a silicon-on-insulator substrate. Gold (Au) electrodes formed the drain and source contacts, and they were encapsulated with alumina (Al_2O_3) to allow the drain-to-source current to transport through the graphene channel rather than electrically shorting through the top-gate dielectric. All of the devices were sized with channel dimensions of $100\mu m \times 100\mu m$.

Three liquid dielectrics were chosen to form the top-gate electrical double layers on graphene: honey, caramelized honey (c-Honey), and glycerin. All liquid dielectrics were acquired commercially. The dielectrics were chosen based on their widespread availability and non-toxicity, thereby demonstrating that these devices can be created with easily obtainable materials.

In all of the experiments, a particular liquid dielectric was deposited between the source and drain electrodes of the graphene device, as shown in Fig. 2. The source and drain electrodes for the device under test are annotated. A micromanipulator with a 500nm-diameter tungsten probe was dipped into the liquid dielectric of choice and then lowered onto the GFET active area until the liquid contacted the surface. Once the liquid contacted the surface, the micromanipulator was lifted up, and the surface tension of the liquid caused it to adhere to the large surface area of the device rather than the nanometer-scale probe tip. Since the fabricated
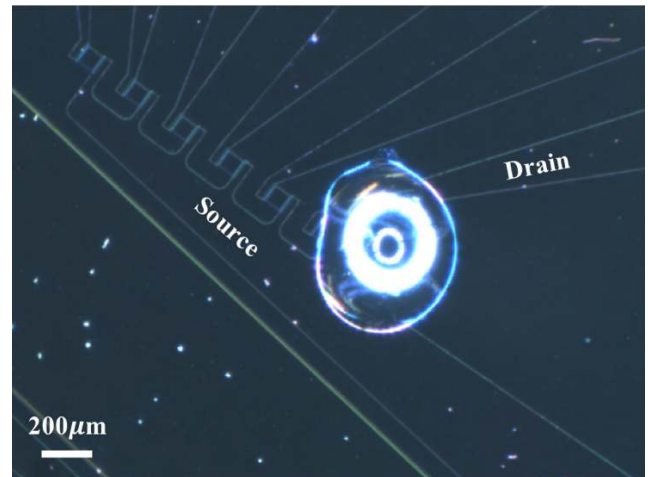
FIGURE 2. Microscope image of Graphene electrochemical field-effect transistor.
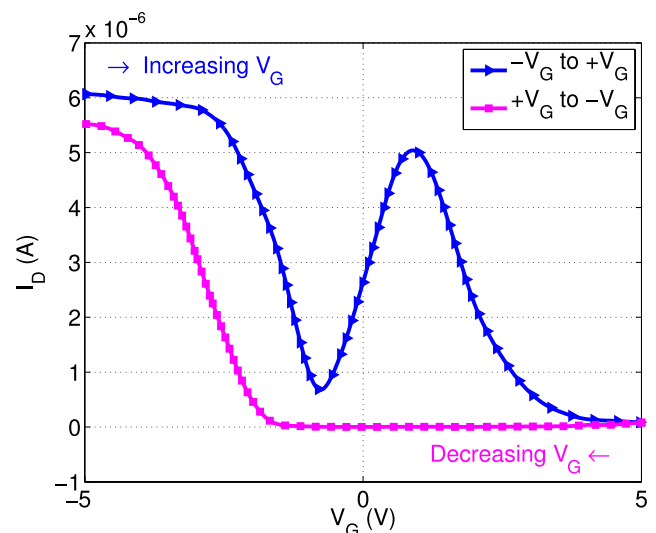
FIGURE 3. Top-gated GFET: Transfer curve with hysteresis.

device contained an array of GFETs, different dielectrics were deposited on each GFET for comparison.

The caramelized honey dielectric was produced by heating regular honey to the "soft-ball" point (110°C) for 5 minutes on a hot plate. During the "soft-ball" process, water in the honey begins to evaporate, which produces a more concentrated sugar solution [16]. The end product is a higher-resistance gate dielectric [17].

All electrical measurements were taken in ambient air with a Semiprobe Manual Probe Station and Keithley B1500A Semiconductor Parameter Analyzer.

III. RESULTS AND DISCUSSION
A. TRANSFER CHARACTERISTICS AND OPERATION
Fig. 3 and Fig. 4 show the transfer curve of the honey top-gated graphene device with linear and semi-logarithmic scales respectively. The plots show the dependence of drain
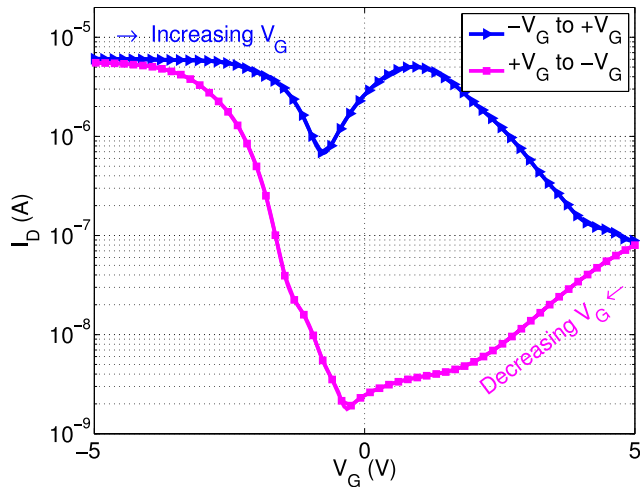
FIGURE 4. Top-gated GFET: Semi-logarithmic transfer curve with hysteresis.

FIGURE 5. Back-Gated (SiO$_2$) GFET drain-current vs. drain-voltage at various gate voltages.

FIGURE 6. Top-gated (honey) GFET drain-current vs. drain-voltage at various gate voltages.

current (I_D) on the top-gate voltage (V_G). The initial sweep from $-V_G$ to $+V_G$ depicts a conventional GFET response with the characteristic Dirac minimum conductivity point observed near $-1V$ [18]. However, at $V_G > 2V$ the device starts to operate differently, and exhibits a sharp reduction in channel current. This phenomenon continues until the gate voltage passes a certain threshold of opposite polarity ($-2V$); at this point the characteristic hole-conduction branch of graphene can be seen, and the graphene device returns to typical operation [19].

After repeated experimentation, it was evident that this phenomenon operated in two steps as follows: 1) SET- once a gate-voltage threshold was exceeded, the graphene resistance drastically increased orders of magnitude above its typical off-resistance; 2) RESET- a gate voltage of equal magnitude and opposite polarity was needed to reset the device and resume conventional GFET operation.

In order to investigate the cause of the unconventional device operation, the team performed a controlled experiment that compared the electrical performance of a GFET device when it was back-gated (SiO$_2$) versus top-gated (honey). The drain-current versus drain-voltage responses of the GFET with back-gate SiO$_2$ and top-gate honey are shown in Fig. 5 and Fig. 6 respectively. It is evident from these plots that there was a clear change in behavior between the back-gate and top-gate responses at higher gate voltages. Whereas the back-gated device showed conventional behavior, the resistance of the top-gated device at $V_G = 4V$ exhibited a resistance as high as 10MΩ, which corresponds to a conductivity far below the theoretical minimum conductivity point of graphene: e^2/h [20].

Our team believes the discrepancies between the back-gate and top-gate are due to the advantageous properties that liquid dielectrics provide towards the electrochemical effect [21]. Liquid dielectrics possess a significant amount of moisture, which can enable water-based chemical reactions that produce weakly-binding hydrogen (H+) and hydroxyl (OH-)
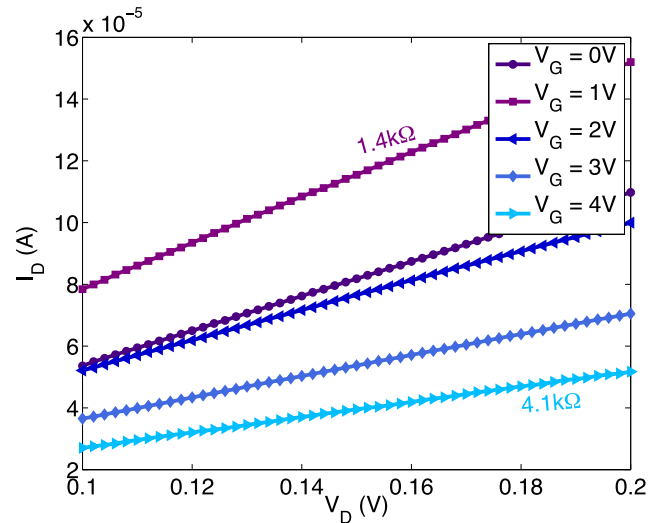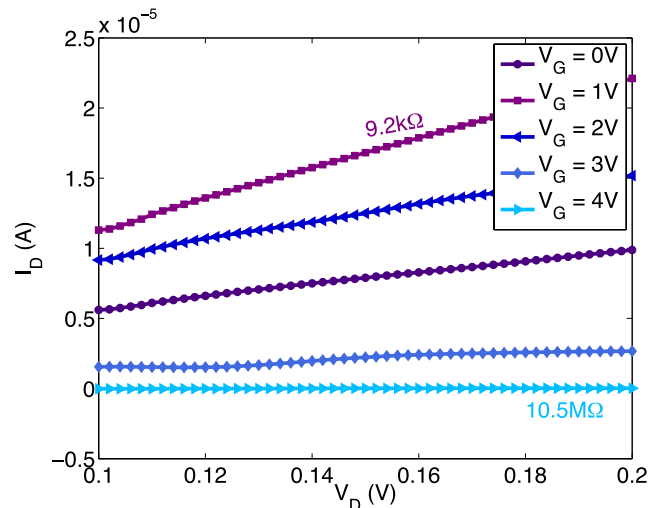
ions [10], [22]. In other work, it was shown that weak molecular bonds can form at the EDL interface, and nominal activation and deactivation energies can be used to control the molecular bonding between the ions and the substrate [23]. Furthermore, scattering experiments with hydrogen and graphene showed that hydrogen atoms possess a high sticking probability with graphene: C-H bonds formed within 10fs of interaction time [24]. Both of these studies support the notion of electrochemically generating ions within the liquid dielectric, providing an activation energy via electric field to form weak molecular bonds with graphene, and providing a deactivation energy to remove those molecular bonds.

Echtermeyer *et al.* noticed a similar phenomenon with graphene on a thin SiO$_2$ dielectric in humid conditions [22]. They performed a controlled series of experiments, and electrically characterized their device in varying conditions:

vacuum, nitrogen, and water vapor. Echtermeyer *et al.* found that the large ON-OFF ratios could only be achieved in the water vapor environment. Therefore, they believed electrolysis of water was crucial to achieving the high ON-OFF ratio. They believed that, after the hydrogen ions were produced from electrolysis, the hydrogen ions interacted with graphene to produce hydrogenated graphene.

Our team's findings support the theory for a water-based electrochemical reaction, as the phenomenon was observed with various liquid dielectrics, but not with the silicon dioxide dielectric. Furthermore, our electrical measurements support the theory that hydrogen ions are forming C-H bonds at the surface of graphene and hydrogenating graphene. Hydrogenation of graphene produces something closer to graphane, which is an insulator rather than a metal [10]. Our electrical transport measurements showed SET resistivities in the same range as the measured resistance of graphane: $\sim$MΩ [8].

The presented work provides a new approach of utilizing water-based reactions to improve electrical switching in graphene. Instead of utilizing a device with a solid dielectric, we present a graphene-based switch with a considerable improvement in response time. The presented device is more than 1,000x faster than the device with SiO_2 dielectric [10]. We believe the ionic concentration and nanometer-scale interface layer, as determined by the Debye length, of liquid dielectrics allow for faster transport of the H+ ions as compared with the solid dielectric example [25]. When the top-gate reaches a large enough RESET voltage of opposite polarity, the weakly-bound ions on the graphene can be released and redistributed in the liquid.

Since the presented device relied on an electrical double layer to distribute ions across the surface of graphene, a uniform EDL was required to produce the insulative state in graphene. ON-OFF switching was only evident when the graphene channel area was not significantly larger than the gate probe. Since there is limited conduction within liquid dielectrics, the electric field formed at the tip of the gate probe will only extend across a limited area of the graphene surface. Therefore, we believe large-area graphene devices contain areas located further away from the probe that do not experience electrochemical doping. The undoped graphene areas act as high-conductivity paths, and limit the overall change in off-conductivity. As a reference, the graphene channel area in this paper was 100μm $\times$ 100μm and the probe tip area was 500nm $\times$ 500nm. Graphene channel areas larger than 5mm $\times$ 5mm with a probe tip area of 500nm $\times$ 500nm did not demonstrate high ON-OFF switching.

B. TRANSIENT SWITCHING

Once the phenomenon was analyzed, the team investigated methods to utilize the electrochemical doping effect to produce practical devices. Threshold SET and RESET voltages for the electrochemical reactions were obtained via the device transfer curve, and the device was biased appropriately to take transient switching measurements, as shown in Fig. 7.
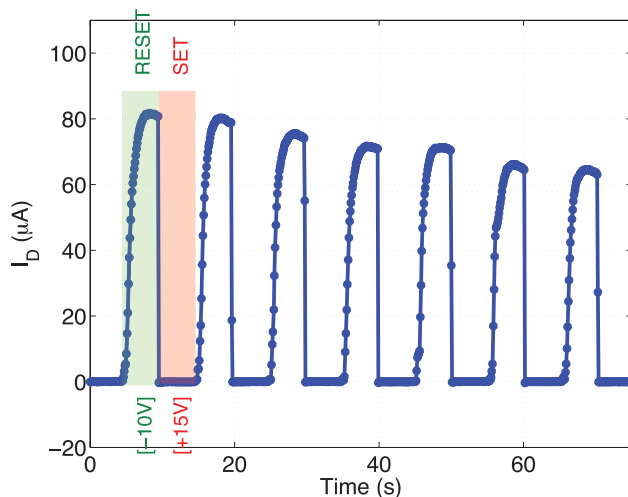
FIGURE 7. Top-Gated (honey) GEC-FET transient switching response with annotated SET (+15V), RESET (−10V) Biases. 0.5Hz switching.
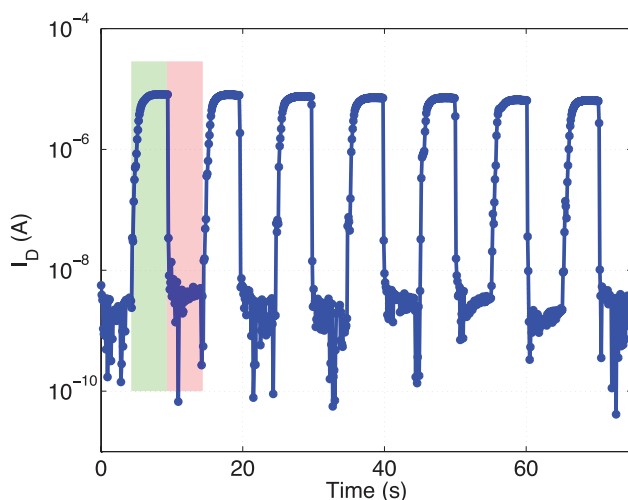
FIGURE 8. Top-Gated (honey) GEC-FET transient switching response with log-formatted drain current. 0.5Hz switching.

The team noticed a consistent degradation in the RESET ON-current over multiple cycles, as was also observed in [10]. Despite the consistent degradation in ON-current, the device maintained a high ON/OFF ratio ($> 10^4$) and remained reconfigurable throughout the full experiment as shown in Fig. 8.

In this device, the degradation was exacerbated at high gate-voltage magnitudes (15V+). Upon further inspection, the team noticed visible bubbles being produced during the electrochemical reaction, as illustrated in Fig. 9. We believe the physical stress and agitation from the electrolysis process can cause breaks and discontinuities in the graphene channel, as experienced in the common electrolysis graphene-transfer technique [26].

Lower gate-voltage biases and higher-resistance liquid dielectrics (i.e. caramelized honey) showed improvements in repeatability as shown in Fig. 10. It is believed that the low water content in the caramelized honey limited the amount
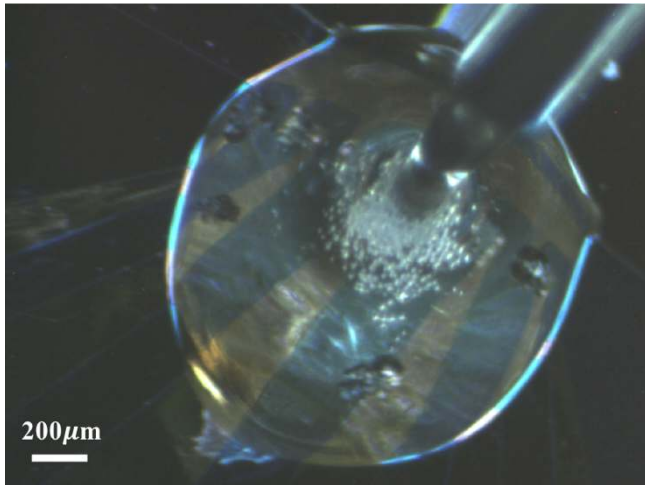
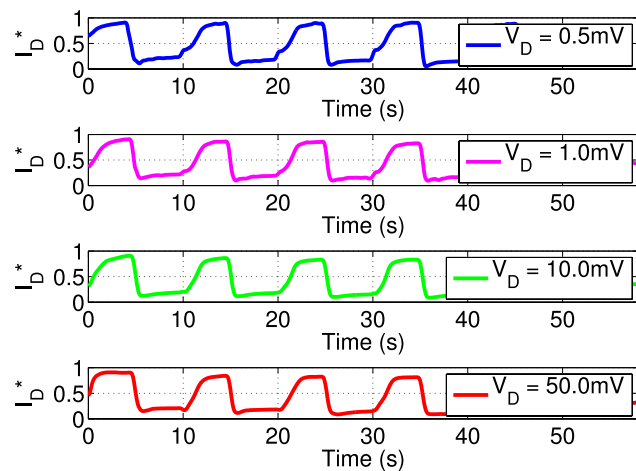**FIGURE 9.** Top-Gated (honey) GEC-FET with local electrolysis (+15V.)

FIGURE 10. Top-Gated (caramelized honey) GEC-FET: Normalized transient switching versus drain voltage.

FIGURE 11. Top-gated (caramelized honey) GEC-FET: Normalized transient switching versus gate voltage.

of electrolysis that occurred in comparison with the original honey dielectric. Despite the improvement in repeatability, further device optimization is required for feasible implementation in digital logic devices. We request for the readers to explore liquid dielectrics of varying pH and chemical composition to minimize the required switching threshold voltage and amount of electrolysis that occurs within the dielectric.

C. SWITCHING DEPENDENCE ON DRAIN VOLTAGE
Additional experiments were performed to gain a better understanding of the operation and relevant parameters of the graphene electrochemical device. A single top-gated device was switched with varying drain-voltage biases, and the normalized transient response was analyzed. Other than the expected linear scaling of the drain current with various drain voltages, there was no noticeable difference in switching operation or speed.

D. SWITCHING DEPENDENCE ON GATE VOLTAGE
Next, gate voltage was varied to observe its effect on the transient switching response. As shown in Fig. 11, gate-voltage
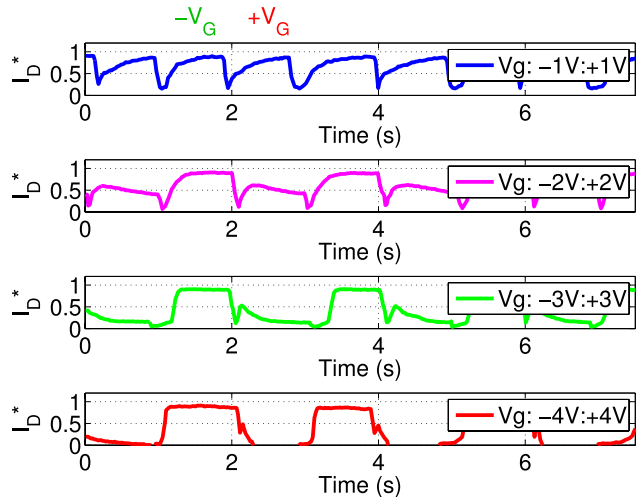
bias had a clear effect on the switching efficiency and response time. At lower gate voltages $+/-1V$, there were artifacts characteristic of charge injection at the SET step, and the switch response was relatively slow [11]. The non-uniform, slow switching was likely due to the electric field strength being below, or near, the activation energy threshold for molecular bonding [23].

As the gate voltage was increased, separation of charges across the EDL became more significant, molecular bonding increased, charge injection became negligible, and the response time significantly improved. This shows that there exists a tradeoff that designers will need to account for: lower gate-voltage biases will allow for improved device repeatability with slower operation, while larger gate voltage biases will provide low-repeatability devices with faster switching speeds.

E. SWITCHING DEPENDENCE ON LIQUID DIELECTRIC
Based on the results of the gate-voltage sweep, our team decided to investigate the effect of several different types of liquid dielectrics. As there is an endless array of liquid dielectric combinations and possibilities [27], we chose to show there is a large area of unexplored territory for graphene electrochemical field-effect transistors.

Easily accessible liquid dielectrics—caramelized honey, honey, and glycerin—were evaluated for response times with a voltage-bias range of $+/-10V$. The transient response of a single switch cycle is shown below in Fig. 12, and the dielectric-dependent device performances are shown in Table 1.

Unsurprisingly, the time constants ($\tau = RC$) of the dielectrics were directly related to the response times. The charge/discharge time of the EDL directly affected how well the produced ions could attach and detach with the graphene sheet. Fig. 13 and Fig. 14 show the rise and fall times, respectively, of the device with the smallest τ: the top-gated
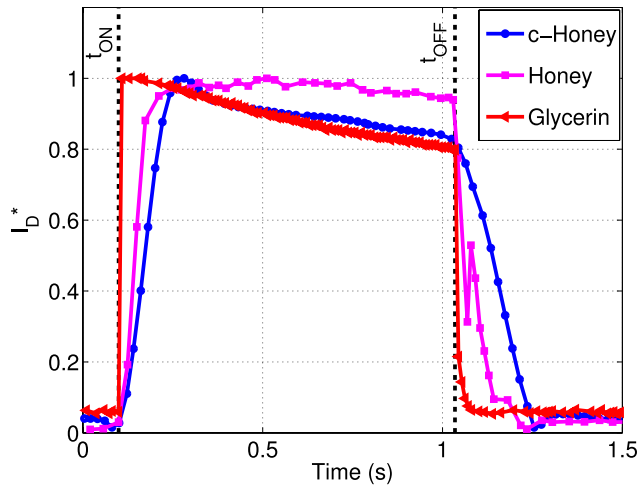
FIGURE 12. Top-gated GEC-FET switching performance with various liquid dielectrics.

TABLE 1. Comparison of GEC-FET performances.

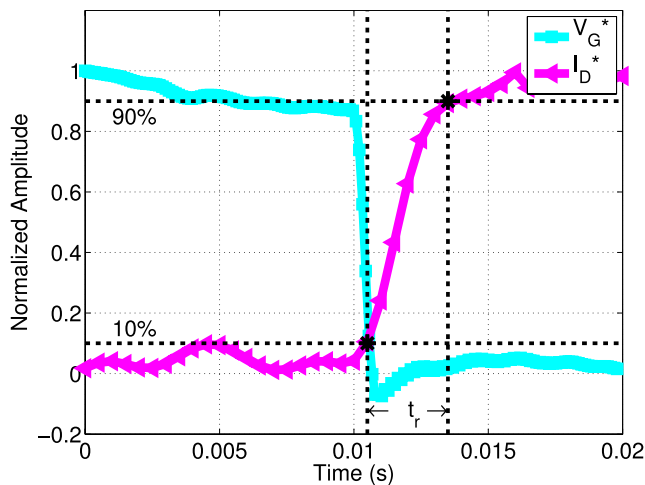| Liquid Dielectric	R	C	τ	Rise Time (t_r)	Fall Time (t_f)
c-Honey	24MΩ	3.36nF	80.6 ms	120ms	192ms
Honey	10MΩ	3.36nF	33.6 ms	74ms	99ms
Glycerin	0.6MΩ	4.16nF	2.50 ms	2ms	14ms

FIGURE 13. Top-gated (Glycerin) GEC-FET switching response: Rise time (t_r).

glycerin GEC-FET. The glycerin GEC-FET demonstrated a response time as fast as 2ms, which is 1,000x faster than in the thin-film SiO$_2$ device.

As seen in MOSFETs and other field-effect transistors, parasitic capacitances at the gate can have a strong effect on the switching performance [28]. Parasitic capacitance was likely the reason for the asymmetric rise and fall times in all
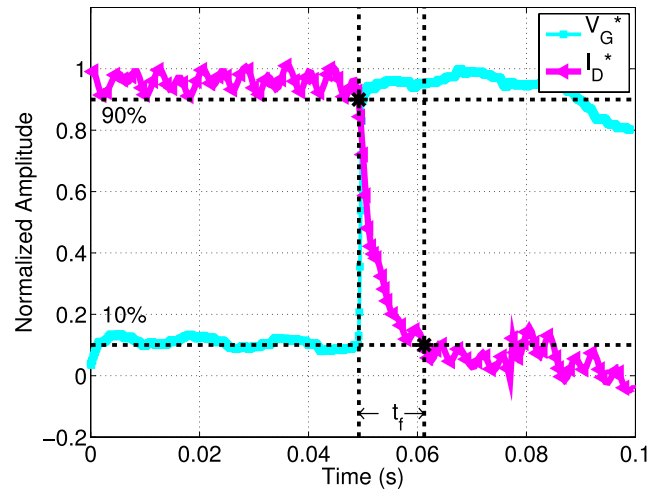

FIGURE 14. Top-gated (Glycerin) GEC-FET switching response: Fall time (t_f).

three devices: when the device was not conducting, there was a huge load seen at the gate, and the large resistance formed a large time constant with the parasitic gate capacitance.

The dependence of switching time on device resistance and capacitance shows that there is the potential for significant performance improvements with the GEC-FET architecture, particularly in GEC-FETs with smaller channel lengths. We investigated the general concepts with a non-optimized, relatively large (100μm $\times$ 100μm) device. CMOS has shown that switching speed can scale quadratically with channel length, as gate capacitance and channel resistance both scale linearly with smaller channel lengths [29]. Recent efforts in GFET fabrication have shown that it is possible to develop GFET channels as small as 30nm [30], which corresponds to a theoretical GEC-FET switching speed in the GHz range. We request for readers to continue the exploration of various liquid dielectrics with miniaturized, nanometer-scale GEC-FETs.

IV. CONCLUSION

This paper discusses the first ever attempt, to our knowledge, of utilizing electrochemical reactions between liquid dielectrics and graphene to produce high on-off ratio electrical switches. Furthermore, it advances past graphene electrochemical switching work by providing a significant improvement in switching speed and an in-depth study of how one can optimize the performance of the device. Despite clear tradeoffs in GEC-FET speed and reliability, we show that novel graphene devices can be developed and optimized with this technique. As shown in the liquid-dielectric comparison, there is a vast area that has yet to be explored with the remaining liquid dielectrics. The comparison of the switch time and charge dynamics with GEC-FETs versus CMOS suggests that there are significant speed improvements that may be realized as GEC-FETs are scaled down.

This technique enables high ON-OFF ratio ($> 10^4$) digital switching in graphene, which had previously been one of the

main limitations keeping graphene from overtaking silicon. While the current device demonstrates limited repeatability, the device is still a promising candidate for non-volatile memory or reconfigurable devices with the ability to reconfigure up to ten times. Pending further device optimizations to improve long-term repeatability, the devices can begin to be incorporated and characterized in digital logic devices. Researchers can utilize the presented techniques to develop digital standard cell libraries for graphene, including inverters, NAND gates, and NOR gates. Due to the ultrahigh carrier mobility of graphene, these devices may even be able to operate at lower subthreshold levels than what is currently possible with CMOS.

As an alternative to traditional digital circuits, this technique can also be applied in asynchronous/dynamic logic circuits. Topologies such as pseudo-NMOS or domino logic can take advantage of the fast rise-time of the GEC-FETs, and also help to limit the drain-to-source voltage across of each GFET. The incorporation of GEC-FETs into pseudo-NMOS or domino logic topologies may ultimately enable full-swing graphene-based dynamic logic.

Current graphene sensor technologies can immediately benefit from having an inherent switching capability. Currently, graphene sensors rely on external silicon-based electronics to perform signal conditioning and digitization of the sensor's analog output. The discussed switching technique may remove the need for external silicon electronics, and, instead, allow for all of the components to be made of graphene. This all-graphene device fabrication can provide significant improvements in device performance and manufacturing costs.

ACKNOWLEDGMENT

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