

Solubility of graphite and the efficacy of using its dissolved form as a conductive paste

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SUMMARY

Graphite is an inexpensive, common, and environmentally friendly compound with many unique properties, one being conductivity. With the world becoming more dependent on electricity, conductive materials have become increasingly relevant, and with them, graphite. This study aimed to take advantage of graphite's conductivity to evaluate its efficacy for use in circuit boards and other applications by using the solution resulting from exfoliation as a conductive paste. We hypothesized that using organic solvents and physical agitation in the form of sonication would increase the solubility of graphite and the solution's overall conductivity. Specifically, we hypothesized that sonication would interfere with enough of the π -stacking interactions to increase the surface area and conductivity of the solute, and the organic solvents would allow for a more consistent dispersion of the graphite solute than water due to graphite's hydrophobic nature. Dissolving graphite in acetone combined with sonication was the most successful method of creating a conductive paste because the solution that was created maintained a high conductivity and had other favorable properties such as a low boiling point (and thus a quicker evaporation time). We found that using a conductive paste for a customizable circuit could be practical as long as a few key engineering challenges are met—mainly, protecting the paste from the external environment and making good connections to the coating. This paste may not replace wires, but as a result of its properties, could have many future uses in the field of electronics or even infrastructure.

INTRODUCTION

Graphite is one of the many allotropes of carbon (1). It consists of stacked layers of graphene, conjoined with weak intermolecular forces such as π - π stacking interactions, and as a result, it retains many of the useful properties of this lattice structure (1). One of these properties is conductivity; any given carbon atom is bonded to three others in a graphene layer, and since carbon has four valence electrons, one is free to flow through the structure (1).

Graphene has been a center of focus in the field of materials science for quite a while because of its possible uses in various nanomaterials and technologies, including

batteries, capacitors, and sensors (2). Graphene is extremely tough, flexible, and light, so devising methods for its efficient production is of utmost importance to researchers (3). One such method is known as solvent-based exfoliation. This process entails the use of a solvent (often organic) combined with aggressive sonication to shear off graphene flakes from bulk graphite (4).

The objective of this experiment was to utilize solvent-based exfoliation methods not to generate graphene layers but to determine the efficacy of using graphite powder, a relatively common substance, to create a conductive coating or paste. A paste with these conductive properties could have many possible applications, ranging from conductive concrete in infrastructure or use as a material in 3D printing and design. In the case of this investigation, the processed graphite solution was used for the purpose of making customizable circuit boards without the use of techniques such as acid etching – a case that not only has real world uses but would be easy to test by constructing a simple prototype.

It is somewhat difficult to apply the term “dissolving” to graphene or graphite because it is a covalent network; attempting to free individual carbon atoms in water to form a paste would be extremely difficult, if not impossible, because the covalent carbon bonds are very strong and the polar forces in water are not strong enough to separate them and increase the surface area of the solute (5). Instead, the graphite layers were exfoliated with the goal of dispersing it through a solvent, so in this case dissolving would consist of breaking the intermolecular forces between layers (6). Specifically, we explored the effect of physical agitation in the form of ultrasonication and different organic solvents on the solubility of graphite and its conductivity. We hypothesized that dispersing graphite into a solution using these techniques would increase the solubility of graphite and the solution's overall conductivity. We came to this conclusion based off the prediction that the sonication would interfere with some of the π - π stacking interactions between layers, increasing surface area and conductivity of the solution (perhaps also freeing some electrons for movement through the structure). We also believed that organic solvents would allow for better dispersion of the graphite layers than water because solvent-solute interactions would not be discouraged by graphite's hydrophobic nature (and possibly prevent the formation of any form of hydrophobic clathrate structures). A solvent like water, because of its extremely polar nature, may be prone to encouraging re-agglomeration.

We concluded that the use of acetone as a solvent, combined with sonication, was the most successful method to create a graphite paste. The paste that was created

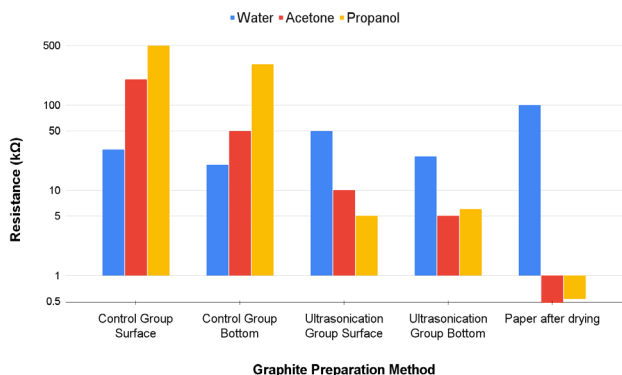


Figure 1: The effect of solvent type and sonication on resistance. The resistance of each graphite solution (water, acetone, propanol) was measured for the control group (no sonication) and the test group (sonication for 60 minutes). This was done using a multimeter and the measurement was taken at two locations, the surface and bottom of each beaker with leads on opposite ends (n=1). Keep in mind that the scale in Figure 1 is logarithmic and the difference of one gridline represents a change by a factor of 10.

maintained a high conductivity. In addition, the use of acetone imparted other favorable properties such as a low boiling point (and thus a quick evaporation time of the solvent), allowing the paste to dry more quickly. We found that using a conductive paste for a customizable circuit could be practical as long as a few key engineering challenges are met.

RESULTS

We analyzed two independent variables in the experiment: the type of solvent used (water, acetone, and 1-propanol, hereafter simply propanol), and the agitation technique (whether sonicated for one hour or left alone, no agitation acting as the control group). We prepared each of the solutions twice and tested under both agitation conditions. To gauge the effect these changes had on the solubility and conductivity of graphite, we took resistance and UV-vis absorption measurements, using a multimeter and a spectrophotometer, respectively.

Thus far, the term “conductivity” has been used, but during the experiment the properties were measured and the data was displayed in terms of resistance. Conductivity varies inversely with resistance, meaning that a higher conductivity correlates to a lower resistance (Table 1, Figure 1). We measured resistance in ohms (Ω) using a multimeter, and measurements were taken at the surface and bottom of each

Resistance	Organic Solvent Type		
	Water	Acetone	Propanol
Non-Sonicated Group Surface (kΩ)	30	200	500
Non-Sonicated Group Bottom (kΩ)	20	50	300
Sonicated Group Surface (kΩ)	50	10	5
Sonicated Group Bottom (kΩ)	25	5	6
Paste After Drying (kΩ)	100	0.48	0.53
Acetone Residue (kΩ)	N/A	0.06	N/A
Pure Solvent (kΩ)	> 2000	> 2000	> 2000

Table 1: The effect of solvent type and sonication on conductivity. The different resistance values for the solutions in different circumstances. Solutions were measured with a multimeter and placed on opposite ends of the beaker (5 cm in the case of the paper).

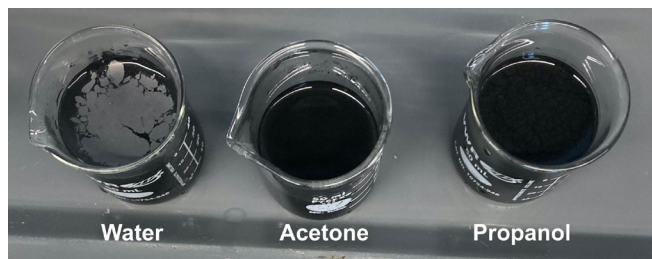


Figure 2: The appearance of sonicated solutions. The beakers contain the ultrasonicated (60 min) solutions of graphite (1 g) mixed with water, acetone, and propanol, listed from left to right. The water solution has flakes of an unidentified substance on the top. All solutions were quite dark (compared to the solutions without sonication, which were mostly transparent) demonstrating qualitatively that the sonication process helped to disperse some of the graphite powder throughout the solution.

beaker. All of the solvents on their own were insulative as demonstrated by the measured resistance in excess of 2000 kΩ (Table 1). The graphite solutions in acetone and propanol had similar measurements: the bottom of the solution had a resistance lower than or similar to the surface regardless of physical agitation, the ultrasonicated group had a lower resistance than the non-sonicated group regardless of depth, and the paste after drying had the lowest resistance of them all (Table 1, Figure 1). The water solution initially had a lower resistance than the other two solutions. However, sonication had little effect on it, and the water solution ended up having a much higher resistance than the others in the sonicated group after the agitation. For the water, the dried paste had more resistance than the solution (Table 1, Figure 1). Overall, the use of the ultrasonic bath greatly decreased the resistance of the graphite in the organic solvents.

The graphite solutions that used water as a solvent (in both the non-sonicated and sonicated groups) also developed a thin, silver-gray film on their surface, which seemed to be as conductive as the rest of the solution (Figure 2). This film was not apparent when any of the other solvents were used.

We used a spectrophotometer to measure the concentration of graphite in each solution (indicative of how much of the graphite added was dispersed throughout the solution rather than resting on the bottom of the beaker). We measured the concentration via absorbance using UV-vis spectrophotometry. The water solution had an absorbance value of 2 and both organic solutions had a value of 2.5 (Table 2).

Because we kept path length and molar absorption coefficient constant, the absorption varies linearly with concentration. While the exact concentration of graphite dispersed throughout each solution could not be calculated, the relative concentration could be by using this fact. The spectrophotometer measurements (Table 2) indicate that the organic solvents dissolved 1.25 times as much graphite as did the water.

We performed additional tests on the acetone solution as it displayed the most favorable properties. Not only did graphite dissolved in acetone have a similar resistance to graphite dissolved in propanol but the solution would also dry the fastest due to the lower boiling point of acetone compared to propanol (56 °C and 97 °C, respectively). We conducted one such test to determine if the resistance of the graphite

UV-vis spectrophotometry tests on Graphite Solutions	Organic Solvent Type		
	Water	Acetone	Propanol
Absorbance Value	2.0	2.5	2.5
Transmittance Value	0.010	0.003	0.003
Percent of Photons Absorbed	99.0	99.7	99.7

Table 2: The effect of solvent type on absorption and concentration. The different absorption values obtained from placing the graphite solutions in a spectrophotometer (340 nm) are displayed in this table, as well as the transmittance values that they correspond to.

in acetone solution would be maintained after the acetone solvent evaporated. After being made into a paste, placed on paper, and allowed to dry, the graphite powder from the acetone solution was scraped off the paper and transferred to another. While this powder did not conduct because the grains did not have good contact with each other, when we added water, the rehydrated paste had a resistance of between 0.05 k Ω and 0.10 k Ω .

We performed another test specifically on the acetone residue that was formed after leaving it in the fume hood in the beaker (not the dried paste directly from the solution). The residue had conductive properties superior to both the solution and the fully dried paste, with a viscosity that allowed it to be more easily shaped. Even after being applied to cardstock with a plunger syringe (**Figure 3A**), the residue continued to have a lower resistance than both the fully dried acetone paste and the acetone solution (**Table 1**).

The acetone residue was then used in construction of a prototype circuit. We put the viscous substance in a plunger syringe and applied it to the cardstock paper (**Figure 3A**). We added aluminum foil contacts to each end of the circuit and sealed the top of the graphite conductor with tape to protect the coating. We connected two AA batteries with alligator clips as well as a blue light-emitting diode, which was rated for ranges between 1.8 and 3.3 volts (**Figure 3B**). The diode successfully produced light and continued to do so even after the graphite coating was bent and twisted multiple times along each axis. In addition to this, the circuit continued to work even days later showing that as long as the paste continued to have a good contact with itself and the wires, it could continue to function, even after drying.

DISCUSSION

To understand how the process of creating a conductive paste out of graphite can be optimized, one must understand the effect that the type of solvent has on graphite conductivity and concentration. The high resistance of the non-sonicated groups can be explained relatively easily (**Figure 1, Table 1**). Each of the pure solvents were insulative (>2000 k Ω , **Table 1**). Because we allowed the solutions in the non-sonicated group to sit for an hour, the graphite powder quickly settled to the bottom and measuring the surface of each solution could only offer a marginal difference compared to the solvent itself. While they might have had a lower resistance than the solvent, the surface of the non-sonicated group for the acetone and propanol solutions remained the least conductive other than the pure solvents (**Figure 1, Table 1**). The bottom of each solution in the non-sonicated groups likely remained relatively insulative because, without sonication, the graphite was not suspended, instead merely resting at the bottom of the beaker and having minimal interaction with the solvent. This would discourage dispersion of graphite particles throughout

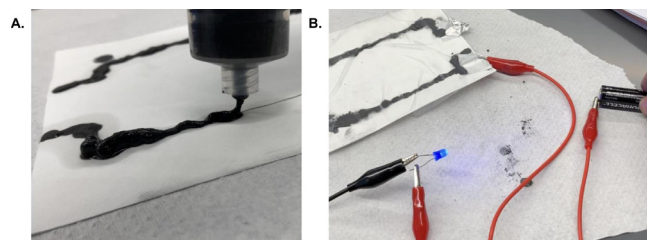


Figure 3: Application of the residue via the plunger syringe and a demonstration of the functional circuit. A) The acetone residue (sonicated) was taken from the bottom of the beaker. Using a plunger syringe, it was placed onto the paper. B) While the acetone residue was still partially wet and viscous, aluminum foil contacts were placed on either side so that alligator clips could be added. The circuit was wired through the paste such that two AA batteries were used in series, the positive side of the batteries connecting to the light-emitting diode's cathode and the other side to the LED's anode. The paste was conductive enough to light up the LED.

the solution and have a minimal effect on surface area (compared to the analogs in the sonicated group) and thereby conductivity (**Figure 1, Table 1**). The reason that the water solution initially had a lower resistance than the other two solvents tested may be attributed to graphite's hydrophobicity (specifically because water is polar and graphite is not). While the other solvents likely interfered with the graphite, a film formed on top of the water and a layer formed at the bottom of the beaker, which may have increased the graphite conductivity in the non-sonicated water group compared to the non-sonicated acetone and propanol groups (**Figure 1, Table 1**). This may also explain how sonication increased the resistance in the water solution, as these layers were likely disrupted.

Even with sonication, it is extremely unlikely that a significant amount of carbon-carbon bonds were broken, having a dissociation energy of approximately 607 kJ/mol (7). That being said, even weak shear forces have the ability to meet the cleavage energy of graphite; at around 0.36 J/m² the intermolecular π - π stacking interactions holding the graphene layers together are broken (8). The kinetic energy being added into the system in from the ultrasonic bath certainly is enough to exceed this threshold in localized areas, as evidenced by the fact that the solutions became noticeably hotter (6). This separation of graphene layers greatly increases the surface area, which allows for greater conductivity and better contact to other materials (such as multimeter pins or wires). This is not to say that every single graphene layer was separated into pure, undamaged, single-layer graphene in this study, but a sufficient percentage of π - π interactions were dissociated. This would increase the total surface area and assist in dispersing the graphite throughout the solution.

This increase in surface area does help to explain how ultrasound affects the structure of a graphite solution on the nanoscale. Yet, in order to make a suggestion on how the organic solvents affect the conductivity (compared to water) and how the chemical's conductivity is affected even after drying, the solvent's ability to disperse the layers must be factored in as well.

The absorption value for each of the ultrasonicated samples was dependent on solution type. The graphite solution that used water as a solvent had a lower absorbance than the graphite solution that used the organic solvents.

Specifically, the graphite in the water-based solvent blocked 99% of light and the organic solutions blocked 99.7% (**Table 2**). This suggests that the organic solvents were able to disperse a higher concentration of graphite throughout the solution compared to the water. It should be noted that this data is at the upper limit of the spectrophotometer used for experimentation (perhaps a different wavelength would have yielded better results, but our instrument had a limited range in this regard). It should also be noted that this information reveals that solution may not be the best term to use to describe the graphite dispersion. The extremely dark color hints that it should probably be classified instead as a colloid (which contains larger particles than a solution would), due to the larger particles blocking a greater amount of light; in addition to this, the exfoliated layers that were successfully dispersed did not seem to be settling out of the solvent, suggesting that the particles were also too small to form a suspension, putting the substance in the range to be a colloid (**Figure 2**).

Thus far, the structural changes in the solutions have been viewed through the lens of differences in surface area and concentration. Yet, there is another perspective which may better explain the role of organic solvents, and that is optimizing exfoliation and maintaining dispersion. In the water solution, ultrasonic waves broke up the graphite into smaller flakes. However, graphite is hydrophobic and was most probably held in place by the polar molecules, eventually reforming π -bonds with their neighboring sheets, in a re-agglomeration process. Water would have a hard time spreading between the graphene layers, a process known as intercalation, and an equally hard time overcoming the cohesion forces of the layers and keeping them apart. This is similar to our experimental observation as some of the graphite formed a precipitate (the film above the water and sedimentation at the bottom of the beaker) separate from the water (**Figure 2**). We posited that unlike water, the two organic solvents were particularly good at maintaining graphene suspension because they could intercalate the graphite, making it easier for the sonication to disrupt the bond energies and disperse the layers through the solution. This hypothesis is evidenced by the fact that the two organic solutions had considerably less precipitate settled on the bottom of the beakers than the water (**Table 2**). As the organic solvents evaporated, the graphite could be left behind with increased surface area, contact, and conductivity (**Table 1, Figure 1**).

The experiment involving the remains of the sonicated acetone residue being dried and rehydrated acts as additional evidence to show that the graphite solutions underwent a structural change (even after drying). When water was added to the powder to see if it could be turned back into a paste after being dried out, it could be shaped again, and it became conductive even after drying. While the resistance of the rehydrated paste was still below the 0.1 k Ω range, it is unclear how many times this process could be repeated without losing conductivity (**Table 1**).

To demonstrate that using graphite to produce an electrically conductive and malleable paste is not only possible but perhaps one day even practical, a simple circuit was constructed using the acetone residue (**Figure 3B**). Because this circuit was conductive enough to light an LED, the effect of the process in this experiment is both noticeable and relevant, though possible applications do come with

engineering challenges.

One problem with the coating is its fragility. While it is flexible, it is extremely easy to rub off, which breaks the circuit. In order to rectify this, the paste needs to be modified, or it needs to be protected (using a method more efficient than the tape used in this study). One way to seal it would be to surround the circuit loosely in a thin plastic sheet and then use heat to shrink it around the substrate. It is also possible that a glass or acrylic pane would work well, though this would take away the flexibility. A resin coating could also be used, which could protect the circuit while maintaining its flexibility.

It may also be beneficial to add a medium to the coating to make it more durable, flexible, or change its viscosity. Adding small amounts of insulative material would likely have a negligible effect on conductivity. Take, for example, the non-conducting solvents themselves which were present in much greater amounts than the graphite; these solvents only decreased the conductivity of the graphite by an order of magnitude (when compared to the dried paste), so another material present in much smaller amounts would likely have an even smaller effect than this (**Figure 1, Table 1**). What matters is that the graphite has complete contact across the circuit, so as long as there is a continuous path there should not be too great of a change. This arrangement of particles could be modeled using a percolation method, to predict the threshold of when the molecules of the other substance begin to interfere with the graphite connection (9).

When making aluminum foil contacts between the graphite 'wire' and the rest of the circuit, the way in which external wires that are attached to the graphite residue could be improved (**Figure 3B**). It is possible a portion of the resistance stemmed from a bad connection between the multimeter leads and the surface – the readout fluctuated frequently and often depended on the orientation and pressure of the connection. The measurement was made when the readout would eventually settle on a single value, which would usually occur a few seconds after contact was made with the circuit.

This study was preliminary and there is much to be done that could optimize the process. To begin, there were a few procedural limitations that, if removed, may provide more insight. For example, one limitation of this study was the small sample size ($n=1$), because the creation of the paste was rather time consuming. A larger sample size with the appropriate statistical analysis would be valuable. Were this paste to be made in an industrial setting, research into making the process more efficient or figuring out how to automate it would likely need to be performed due to the time and effort needed to produce a relatively small amount.

Another possible improvement concerns the process of sonication and would involve using sonication probes rather than an ultrasonic bath, as they provide a greater amount of energy in a shorter amount of time. Also, solvents such as benzene, heptane, tetrahydrofuran, diethyl ether, and propylene carbonate should be tested because they each have their own unique structure which could possibly affect graphite's dispersion through the solution. In particular, the aromaticity of benzene may allow benzene molecules to participate in π - π interactions with the graphene layers. This could improve the solubility of the graphite because of their more similar structures. It would have been good to test more hydrophobic solvents like heptane as graphite itself is hydrophobic, but unfortunately, this was not possible due to a

combination of time and material constraints.

There are a few more areas which could be researched in the future. The identity of the silver-gray film that formed on the top of the water solution is still unknown, and what caused it to form is unclear, though we speculate that it is related to hydrophobicity (**Figure 2**). Any nonpolar molecule might display this behavior of forming a film in a polar solvent like water, which would explain why none formed in the less polar organic solvents. The film seemed to be very conductive, so it is possible that it was just the graphite, or perhaps even graphene. There is another method of graphene synthesis involving solvent-surfactant interface interactions, and research from this field could possibly shed some light on the identity of this film (5). It would also be interesting to combine solvents to see if the effect would simply be an interpolation between the two or something completely different. Different salts might be added as well to see whether their polarity helps or hinders the dispersion process. Another topic that could be explored is optimizing the solvent by finding one with a similar surface free energy to that of graphene or graphite, allowing the layers to disperse through the solution as efficiently as possible (1). This may help to narrow down which of the solvents mentioned in the last paragraph should be tested. Analyzing this topic in terms of thermodynamics may be fruitful, as many interfacial interactions could be explored computationally or mathematically and the most interesting results could then be selected for an experiment (1).

A graphite paste could have many applications in a wide range of sectors from electronics to infrastructure. The ability of the paste to create a custom and flexible circuit capable of lighting an LED (**Figure 3B**) demonstrates that making electronics like this with a graphite paste is possible and may be practical for flexible circuits after engineering challenges are solved. In fact, the paste could potentially be printed into a circuit by an inkjet or screen printer (10). The viscosity of the graphite paste could be manipulated as needed, and the printer could apply the coating to easily generate an inexpensive custom circuit board. In addition, the hypothetical printed circuit board could have the option of being flexible and the paste could be combined with other materials to make them conductive, such as concrete (9, 10). Conductive concrete could be feasible as a result of the paste being able to remain functioning when dry and as a result of its relatively low resistance (**Figure 1, Table 1**). Conductive concrete could have a multitude of possible uses in infrastructure (10). There are many future possibilities for graphite, just as there are many avenues for further research and exploration.

MATERIALS AND METHODS

Setup and Procedure

To test the hypothesis that ultrasonication would decrease the resistance of the solution and that the organic solvents would dissolve and disperse the graphite powder better than water, two groups of three solvents were needed. The first was deionized water, and the two organic compounds were acetone and 1-propanol. The organic solvents were obtained from Sigma-Aldrich Corporation. The graphite was sourced from Carolina Biological Supply and was listed as 99% pure. An ultrasonic bath (Magnasonic brand), was used to provide physical agitation rather than a sonication probe. Studies by

Goncalves, et al. indicated that samples in an ultrasonic bath can still be agitated successfully so long as the duration is enough (6). A spectrophotometer (Thermo Fisher Scientific Spectronic 200) was used for measurement, along with handheld multimeters. The majority of this experiment was conducted in a fume hood to minimize vapors (especially from the acetone).

40 mL of each solvent was poured into two 50 mL beakers, one for a non-sonicated group and one for the sonicated group. Then, 1 g of graphite powder was added to each beaker. The non-sonicated control group beakers were allowed to sit for 1–2 hours. The sonicated group of beakers were placed in the ultrasonic bath for 60 minutes. Throughout this process, visual characteristics of each solution were documented – amount of graphite in separation versus in suspension, whether films or flakes formed on the top, the homogeneity of the solution and more generally features related to its turbidity were recorded (**Figure 2**).

Conductivity and Absorbance Measurements

One of the variables measured throughout the investigation was resistance. For each of the solvents both groups (non-sonicated and sonicated), multimeter leads were placed on opposite ends of the beaker and measured at two different depths, the surface and the bottom. Resistance was measured in the non-sonicated group after an hour, and after the ultrasonication in the experimental group at these two depths (**Table 1, Figure 1**).

The second measurement that was taken was UV-visible light absorbance, at 340 nm. The absorbance of each of the samples that underwent the ultrasonic treatment was measured using the spectrophotometer and zeroed according to the absorbance values of the pure solvent. In this way, the absorbance values of the suspended graphite were isolated from any interference caused by the solvent (**Table 2**).

Further Tests on Pastes and Acetone Residue

Upon the completion of the conductivity and light absorbance measurements, a graphite paste was produced by taking a small amount of each solution and, using a transfer pipette, spreading it in a line on a piece of paper. After drying for approximately 1–2 hours, the resistance of each paste was measured (with a distance of 5 cm between contact points), and its visual characteristics observed.

Further testing was conducted specifically on the acetone solution of the sonicated group. In one test, the line formed with the graphite-acetone mix was allowed to dry. The resulting dried powder was scraped off the paper it had dried on and placed on another paper. A few milliliters of water were added to this powder using a pipette, forming a new, rehydrated paste. The resistance of this paste was measured using a multimeter.

The beaker of acetone solution was left open in the fume hood, and due to the solvent's low boiling point, the majority of the acetone in the solution had vaporized in a short amount of time. What was left was a dark, viscous residue. The resulting residue was administered to a piece of cardstock using a plunger syringe and allowed to dry (**Figure 3A**). Because this residue was prepared using a different method than the paste (in that it was allowed to partially dry in the beaker before being applied to paper) resistance data is under the "Acetone Residue" row rather than the "Paste After Drying" row (**Table**

1). The line was covered with tape and aluminum foil was attached to either ends of the circuit to act as contacts. Two AA batteries were used to power a light emitting diode through the graphite 'wire' to test if the circuit could conduct enough electricity for practical purposes (**Figure 3B**).

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