

# Recycling Diesel Soot Particles for Use as Activated Carbon in Li Ion Batteries

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<b>16. Abstract</b> This report documents the successful capture and reuse of diesel exhaust soot particles as a conductive additive in lithium manganese oxide (LMO) and lithium iron phosphate (LFP) cathodes in Li-ion batteries. This approach enables an abundant toxic pollutant to be converted into a valuable material for energy storage devices. This study consists of an initial characterization of the diesel soot particles, a high-temperature annealing step to remove residual organics and unburned hydrocarbons, and characterization of the electrical performance in a Li-ion battery configuration. Here, composite electrodes are fabricated by mixing active materials (LFP or LMO) with conductive carbon and binders. The performance of the diesel soot particles as conductive additives is compared with that of commercially available activated carbon (i.e., Super P®). The current evolution of the composite electrode made with diesel soot particles demonstrates comparable performance to the electrodes containing the Super P® carbon. Based on high-resolution transmission electron microscope (HRTEM) images and scanning mobility particle sizer (SMPS) spectra, it is found that these diesel soot nanoparticles follow a narrow log-normal distribution centered around 100 nm in diameter and consist of highly porous amorphous carbon, which provide a large surface-to-volume ratio, making them ideal candidates for electrode materials in Li ion batteries.			
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August 2022

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# Recycling Diesel Soot Nanoparticles for Use as Activated Carbon in Li Ion Batteries

## EXECUTIVE SUMMARY

This documents the successful capture and reuse of diesel exhaust soot particles as a conductive additive in lithium manganese oxide (LMO) and lithium iron phosphate (LFP) cathodes in Li-ion batteries. This approach enables an abundant toxic pollutant to be converted into a valuable material for energy storage devices. This study consists of an initial characterization of the diesel soot particles, a high-temperature annealing step to remove residual organics and unburned hydrocarbons, and characterization of the electrical performance in a Li-ion battery configuration. Here, composite electrodes are fabricated by mixing active materials (LFP or LMO) with conductive carbon and binders. The performance of the diesel soot particles as conductive additives is compared with that of commercially available activated carbon (i.e., Super P®). The current evolution of the composite electrode made with diesel soot particles demonstrates comparable performance to the electrodes containing the Super P® carbon. Based on high-resolution transmission electron microscope (HRTEM) images and scanning mobility particle sizer (SMPS) spectra, it is found that these diesel soot nanoparticles follow a narrow log-normal distribution centered around 100 nm in diameter and consist of highly porous amorphous carbon, which provide a large surface-to-volume ratio, making them ideal candidates for electrode materials in Li ion batteries.

## Introduction

The wide acceptance of the serious health effects associated with nanoscale particulate matter (PM) has led to an extensive range of nanotoxicology studies of the environmental emissions from the combustion of fossil fuels, cigarette smoke, and even emissions from commercial cooking processes (e.g., charbroiling).<sup>1-8</sup> During the past couple of decades, the adverse health effects of diesel particulate emissions have been firmly established by many toxicological studies.<sup>9-12</sup> These ultrafine particulates have been linked to premature cardiovascular and respiratory deaths in metropolitan areas, as well as lung cancer.<sup>1, 3-5</sup> For example, a 1993 study published by Dockery et al. has been cited more than 4,600 times as of the time of this writing, demonstrating the broad impact of this problem.<sup>2</sup> Diesel engines and vehicles represent major sources of PM and are responsible for approximately 8% of the PM<sub>2.5</sub> found in outdoor air, and they emit more than 70 tons of PM per day in California alone (25,000 tons of PM per year).<sup>13-19</sup> The Air Resources Board (ARB) and U. S. Environmental Protection Agency (EPA) have stated that an estimated 3,500 premature deaths per year are affiliated with respiratory and cardiovascular diseases that can be attributed to diesel PM in California.<sup>19-22</sup>

Several technologies have been developed to treat diesel particulate emissions, including diesel particulate filters (DPFs), wet scrubbers, and electrostatic precipitators (ESP).<sup>23-25</sup> Many of these have been developed in response to increasingly stringent air quality regulations, such as those imposed by the EPA, California Air Resources Board (CARB), and local air quality air management districts (AQMD). Most recently, there has been a popular trend towards zero-emissions systems. However, for most practical applications, the combustion of diesel fuel will remain a dominant source of power, requiring more advanced pollution control devices to be developed. Diesel particulate filters provide a relatively cheap solution that works well for small size engines. Over time, however, these filters become clogged, which creates engine backpressure and requires regeneration at regular maintenance intervals. Electrostatic precipitators are generally too large for vehicles and present the problem that they eventually need to be cleaned out, which raises the issue of how to dispose of this now highly concentrated hazardous carcinogenic material.

Li ion batteries have become a ubiquitous part of our society, often going unnoticed. As the global Li ion battery capacity continues to grow at its current exponential rate of growth, innovations in high surface area materials will be needed in order to sustain this growth and meet the economic pressures of supply and demand. By 2030, it is estimated that the global Li ion battery capacity will exceed 1 TWh, and cheap rechargeable batteries remain a key enabling technology for electronic vehicles and solar farms. The present composite electrodes in commercialized batteries consist of three main components: active materials, conductive additives, and polymer binders. Active materials intercalate with Li ions during the charging/discharging cycles of the Li-ion battery operation. Common active materials in commercialized cathode electrodes are lithium iron phosphate (LFP), lithium manganese oxide (LMO), lithium cobalt oxide (LCO), and lithium nickel manganese cobalt oxide (NMC). These cathode electrodes, however, have a poor electrical conductivity. For example, the electronic conductivity of LMO and LFP are  $\sim 10^{-6}$  and  $10^{-9}$  S/cm, respectively.<sup>26, 27</sup> Carbon additives play

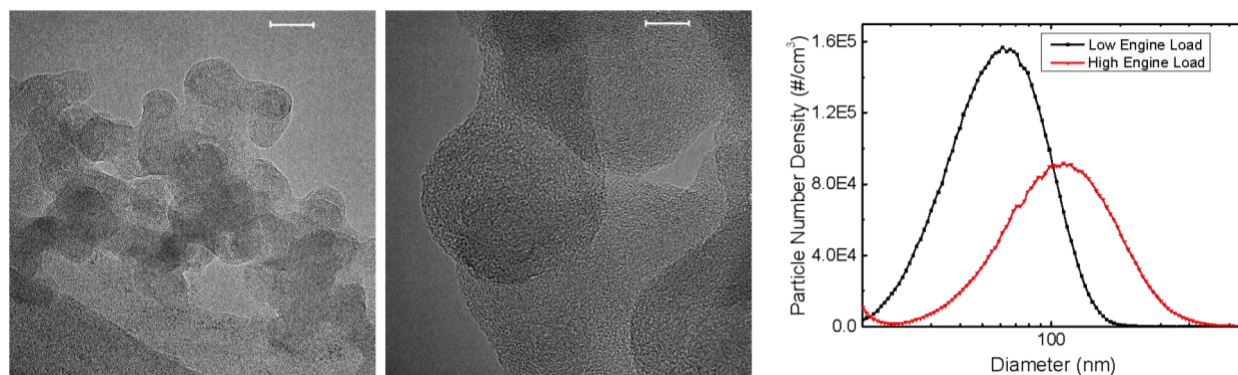


an important role in creating conductive networks in composite electrode structures in order to improve battery performance, and the impact of conductive carbon on the capacity profile of electrodes is significant. Previously, the contribution of the conductive carbons on the practical capacity of these electrodes was studied, but only using commercially available conductive carbons.<sup>28, 29</sup> In this study, we evaluate the electrochemical performance of conductive carbon derived from diesel soot particles.

## Materials and Methods

Here, we present a proof-of-concept study demonstrating the successful conversion of diesel engine exhaust soot particles to a high-surface area electrode material for rechargeable Li ion batteries. We characterize the size and shape of these particles using high-resolution transmission electron microscopy (HRTEM) and a scanning mobility particle sizer (SMPS), and then anneal the material in a pretreatment step before building a coin-cell lithium-ion battery using this material. In this controlled study, the performance of commercially available activated carbon material is compared to the pre-annealed and post-annealed diesel soot material. We characterize the current-voltage characteristics as well as the charging/discharging performance of these prototype LMO and LFP composite electrodes.

In our experimental setup, diesel soot particles are collected and then deposited on TEM-compatible substrates. The particle size, shape, and clustering are characterized using HRTEM. Figure 1 shows HRTEM images of nanoparticle size distributions in the 20-200nm diameter range, which is consistent with the diameters measured using an SMPS spectrometer. These particulates are then annealed at 250°C in air for 4 hours in order to purify them for use as activated carbon.



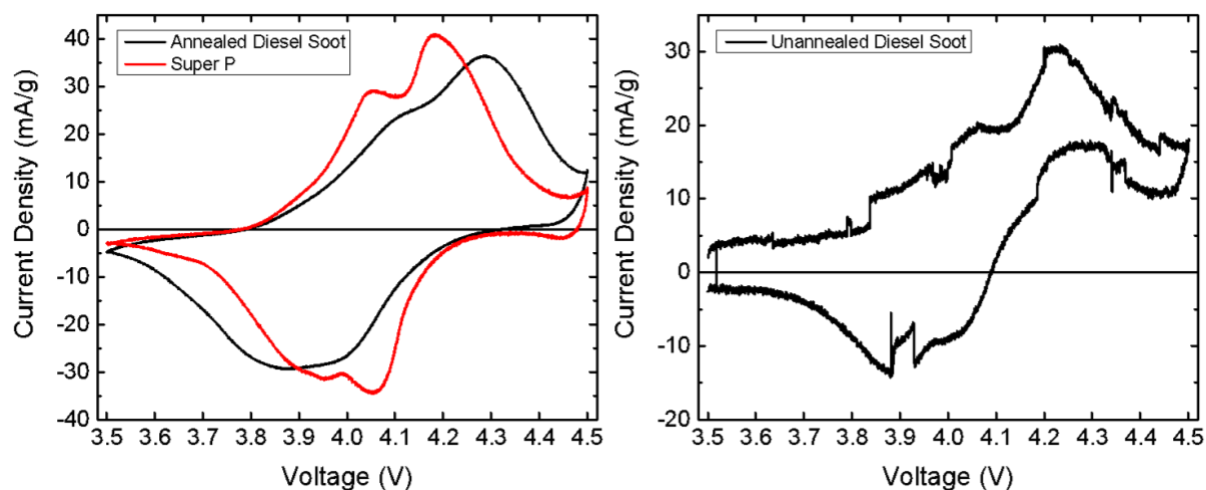
**Figure 1. (a,b) High-resolution transmission electron microscope (HRTEM) images and (c) scanning mobility particle sizer (SMPS) spectra of diesel soot particles (i.e., particulate matter).**

Composite electrodes were fabricated in order to characterize the electrochemical performance of the activated carbons. These electrodes were made by combining lithium manganese oxide ( $\text{LiMn}_2\text{O}_4$ , LMO, electrochemical grade, Sigma Aldrich) or lithium iron phosphate ( $\text{LiFePO}_4$ , LFP) with carboxymethyl cellulose sodium salt binder (CMC, Aldrich) and

conductive carbon additives at an 8:1:1 mass ratio. The LMO and LFP powders were used as-is, and their particle sizes are around 3  $\mu\text{m}$  and 250 nm, respectively.<sup>30, 31</sup> In our study, the conductive carbon consisted of either commercially available Super P<sup>®</sup> carbon (Timcal), annealed soot, or unannealed soot material. The CMC polymer binder was dissolved in deionized water at a 1:50 weight ratio. The LMO or LFP and conductive carbon additives were then added to the binder solution. The slurry was mixed for 30 minutes using a Thinky centrifugal mixer at 2000 RPM. Figures 5 and 6 of the Supplementary Information show pictures of these electrode manufacturing steps. The slurry is cast on an aluminum foil current collector using a doctor blade and dried under ambient conditions. The electrolyte for all electrochemical testing consisted of ethylene carbonate (EC, anhydrous, 99%, Sigma Aldrich) and dimethyl carbonate (DMC, anhydrous, >99%, Sigma Aldrich) at a 1:1 volume ratio. After that, 1 M LiPF<sub>6</sub> (98 %, Sigma Aldrich) was added to the EC:DMC solution. The electrolyte was mixed with a magnetic stirrer in a glove box for one day. A CR2032 coin-cell was fabricated with an LMO or LFP composite cathode as the working electrode, lithium metal as the anode counter electrode, and a Celgard polyethylene film as the separator. The composite electrodes were cycled either via cyclic voltammetry at 50  $\mu\text{V}/\text{sec}$  or galvanostatically at different charging rates (i.e., C-rates).

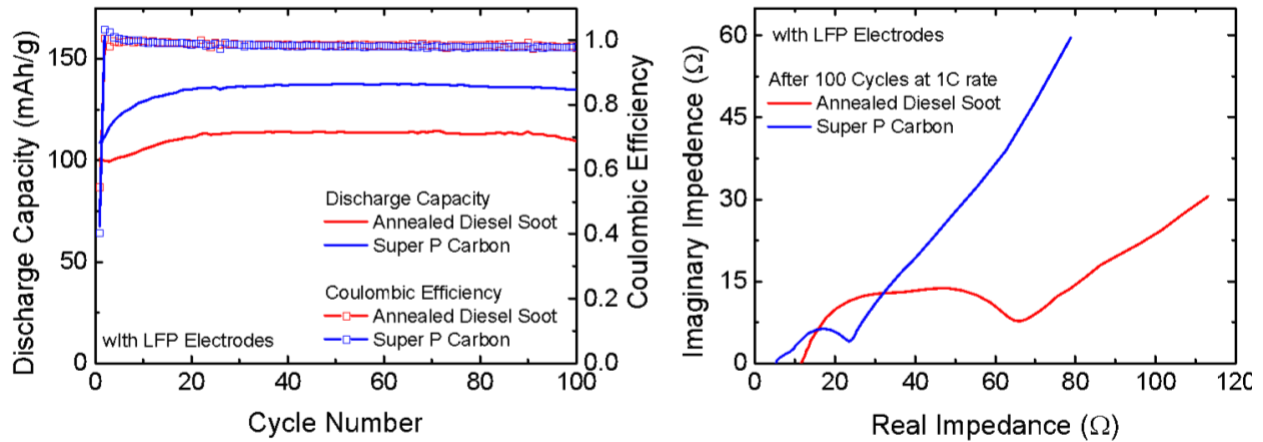
## Results and Discussion

Figure 2 shows the current density evolution of the composite LMO electrodes prepared with different conductive additives plotted as a function of voltage during the 5th cycle at a scan rate of 50  $\mu\text{V}/\text{sec}$ . As expected, two distinct current peaks are clearly observed corresponding to lithiation and delithiation in the Super P<sup>®</sup>-containing electrode. These current peaks correspond to the well-known two-phase transformation in LMO electrode structures.<sup>32</sup> We also observe two pairs of oxidation/reduction peaks in the annealed soot-containing electrode, which indicates that there are no additional redox reactions associated with the annealed soot particles. The current peaks for the annealed soot particle-containing electrode precede the current peaks for the Super P<sup>®</sup>-containing electrode by approximately 0.1 V during and after delithiation. The interval between the oxidation and reduction potentials are also greater for the annealed soot particle-containing electrode, indicating that there is polarization within the electrode due to the larger resistance in the annealed soot particle-containing electrode. While the annealed soot particles demonstrate larger resistance in the electrode, the maximum current density observed in the annealed soot particle-electrode is comparable to that of the Super P<sup>®</sup>-containing electrode. Here, the capacity of the electrode is calculated by taking the integral of current with time. The charging/discharging capacities of the LMO electrodes containing Super P<sup>®</sup> carbon and annealed soot particles were both around 70 mAh/g. As a comparison, the theoretical capacity of the LMO electrode is 148 mAh/g. Figure 2b shows the current evolution of the electrode prepared with unannealed soot particles. Less distinct current peaks with lower current density values are observed in the unannealed soot particle-containing electrode, which also exhibits highly unstable current responses in its cyclic voltammetry traces.



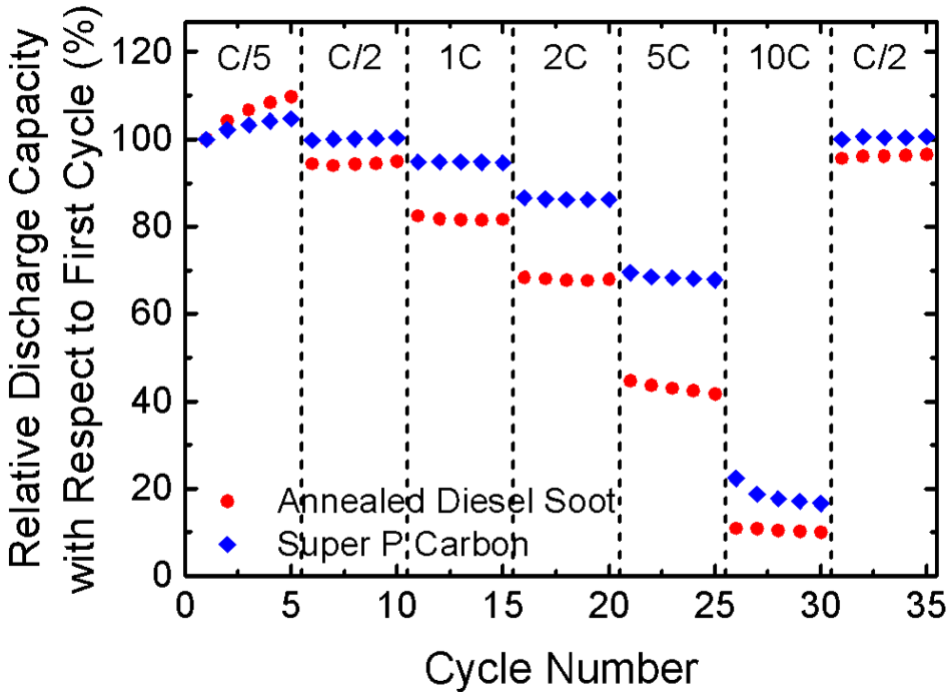
**Figure 2. Current-voltage curves taken from LMO electrodes containing (a) annealed diesel soot, Super-P® (commercially available activated carbon, typically used in Li-ion batteries) and (b) unannealed diesel soot.**

Figure 3a shows the discharging capacity of the LFP composite electrodes cycled 100 times at 1C rate. The initial discharge capacities of the electrodes containing the Super P® carbon and annealed soot carbon were 92.2 and 108.7 mAh/g, respectively. After 100 cycles, the discharge capacities of the electrodes containing the Super P® carbon and annealed soot carbon became 134.9 and 98.3 mAh/g, respectively. The Coulombic efficiencies for the LFP electrodes containing Super P® carbon and annealed soot carbon after 100 cycles was 97.9% and 98.2%, respectively. Electrochemical impedance spectroscopy (EIS) of the composite electrodes containing different conductive carbons was performed before and after cycling with a sinusoidal amplitude of 5 mV from 100 kHz to 10 MHz, as shown in Figure 3b. These EIS measurements were conducted using a Biologic potentiostat equipped with EC-EC-lab® acquisition software. The high-to-mid frequency region obtained from EIS is represented as Nyquist plots with real ( $Z'$ ) and imaginary ( $-Z''$ ) parts of the impedance plotted on the x- and y-axes, respectively. The radius of the semicircle at high frequency (low  $Z'$ ) can be associated with the charge transfer resistance of the electrode.<sup>33</sup> Before cycling, the charge transfer resistance of electrodes containing the annealed soot carbon and is found to be much lower than the electrodes containing Super P® carbon. Since impedance measurements are highly sensitive to the effective surface area of these electrodes, it is more informative to compare the change in electrode resistance with cycling. Overall, the electrochemical resistance decreases with cycling for all electrodes.



**Figure 3. (a) Discharging capacity (line) and Coulombic efficiency (open square symbol w/ line) of LFP electrodes containing Super P<sup>®</sup> carbon or annealed soot. The composite electrodes were cycled at 1C rate. (b) Impedance plots of the electrodes after 100 cycles at 1C rate.**

The impact of the scan rates on the discharge capacity was also tested for the LFP electrodes containing Super P<sup>®</sup> carbon and annealed soot carbon, as plotted in Figure 4. The initial capacity at C/5 rate was 112 and 140 mAh/g for the electrodes containing annealed soot carbon and Super P<sup>®</sup> carbon, respectively. The discharge capacity decreased when the electrode was charged at faster rates regardless of the conductive carbon. When the rate was set back to C/2, the relative capacity become 99.9% and 95.5% with respect to the first cycle for the electrodes containing annealed soot carbon and Super P<sup>®</sup> carbon, respectively.



**Figure 4. Relative discharge capacity of LFP electrodes containing Super P® carbon (blue color) or annealed soot carbon (red color) cycled at C/5, C/2, 1C, 2C, 5C and 10C for four cycles each.**

In conclusion, we demonstrate that diesel exhaust soot particles can be recycled and used as activated carbon in Li ion batteries. Here, an abundant carcinogenic pollutant is converted into a value-add material that can be used in Li ion energy storage devices. The diesel soot nanoparticles follow a log-normal distribution centered around 100nm, as characterized by electron microscopy and scanning mobility particle sizer spectroscopy. Once collected, high-temperature annealing is used to remove residual organics and unburned hydrocarbons from the diesel soot particles. Composite LFP and LMO electrodes containing the recycled material performs competitively with the commercially available activated carbon (i.e., Super P®) typically used in Li ion batteries. The unannealed diesel soot particulate material, however, performs much worse than the annealed material with highly unstable current-voltage characteristics and low discharging capacities. These diesel soot particulates consist of highly porous amorphous carbon, which provide a large surface-to-volume ratio, making them an ideal candidate for electrode materials in Li ion batteries.

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## Data Summary

### Products of Research

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Figure 5. (a) Electrode slurry containing annealed diesel soot particles in Thinky Mixer Containers. (b) Picture after the slurry was casted on an aluminum current collector and dried. (c) Picture of electrode punched for coin-cell manufacturing.

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Figure 7. (a) Discharging capacity of LMO electrodes containing Super P® carbon, annealed soot or unannealed soot. The composite electrodes were cycled at 1C rate. (b) Impedance plots of the electrodes before and after 100 cycles at 1C rate.

Figure 8. Raman spectra (normalized) of Super P® carbon, annealed soot and unannealed soot.

### Data Format and Content

All text is in Doc files and all figures are in PDF and origin.

### Data Access and Sharing

Replication Data for: Recycling Diesel Soot Particles for Use as Activated Carbon in Li Ion Batteries is available at <https://doi.org/10.7910/DVN/MTDI25>.

### Reuse and Redistribution

Access to databases and associated software tools generated under the project was made available for educational, research and non-profit purposes. Such access was provided using web-based applications, as appropriate. Results generated under the project were disseminated in accordance with University/Participating institutional policies. Depending on

such policies, materials were transferred to others under the terms of a material transfer agreement. The results of the research performed under this proposal was disseminated primarily through publication in peer reviewed journals and conference presentations. Periodic updates on the research were posted on the group websites. Research data that documents, supports, and validates research findings were made available after the main findings from the final research data set were accepted for publication. Such research data were redacted to prevent the disclosure of personal identifiers.

## Supplementary Information



**Figure 5. (a) Electrode slurry containing annealed diesel soot particles in Thinky Mixer Containers. (b) Picture after the slurry was casted on an aluminum current collector and dried. (c) Picture of electrode punched for coin-cell manufacturing.**



**Figure 6. (a) Electrode slurry containing Super P<sup>®</sup> carbon in Thinky Mixer Containers. (b) Picture after the slurry was casted on an aluminum current collector and dried. (c) Picture of electrode punched for coin-cell manufacturing.**

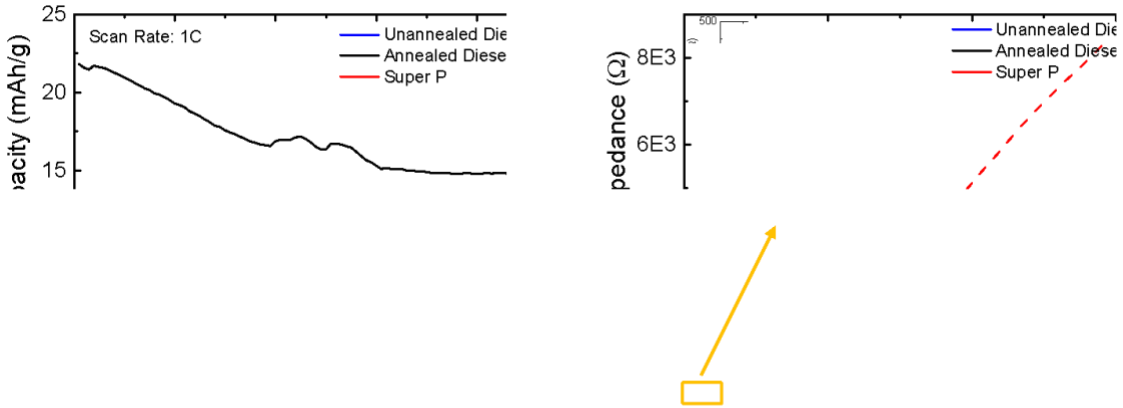


Figure 7. (a) Discharging capacity of LMO electrodes containing Super P<sup>®</sup> carbon, annealed soot or unannealed soot. The composite electrodes were cycled at 1C rate. (b) Impedance plots of the electrodes before and after 100 cycles at 1C rate.

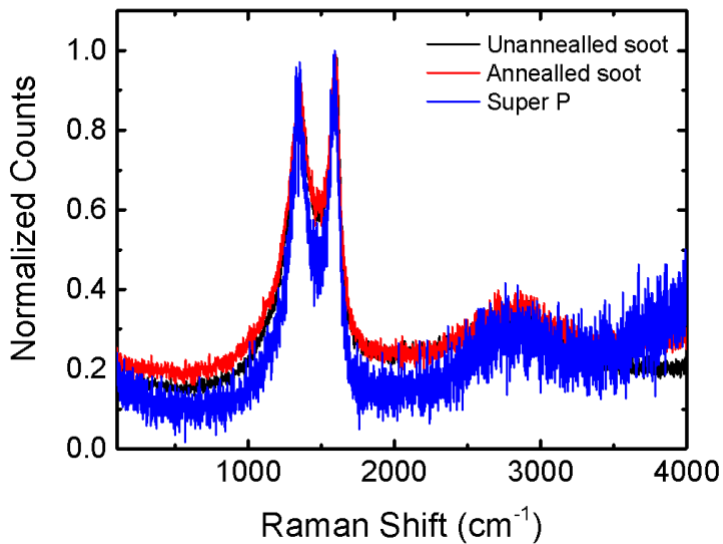


Figure 8. Raman spectra (normalized) of Super P<sup>®</sup> carbon, annealed soot and unannealed soot.