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BATTERY APPLICATION OF SILVER MOLYBDATE MATERIALS

by

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Honors Work in Chemistry
For CHEM-498
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INTRODUCTION

As the demand for renewable energy grows, the investigation of potential battery materials continues to progress hastily. Many devices and machines of present day are operated by battery technology, and their energy requirements warrant which kinds of batteries will power them. The battery in a phone requires hundreds, possibly thousands, of uses and needs to be able to recharge quickly. The battery in the implantable cardiac defibrillator (ICD), the battery by which this product was inspired, is only used once, but it must last years before it is replaced. The cathode of the ICD battery is comprised of a silver and vanadium compound. Silver has a large affinity for electrons, making it a popular candidate for battery studies, and vanadium has shown tremendous promise in numerous other battery studies to date.¹⁻⁴ Instead of silver-vanadium compounds, this project examines four silver-molybdenum complexes referred to as silver molybdates ($\text{Ag}_2\text{Mo}_x\text{O}_y$). The four complexes are shown in **Figure 1**.

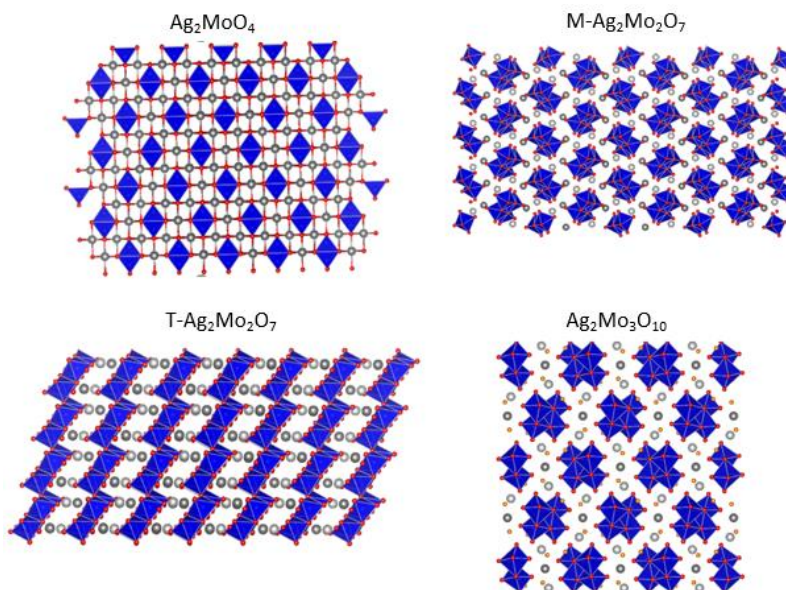


Figure 1: The four silver molybdate complexes. Blue = Mo, Silver = Ag, Red = O.

Molybdenum has received much less attention than vanadium in battery research, but its higher oxidation state potentially permits coordination with more silver atoms, which could very well improve on the current structure of the ICD battery. Even if these compounds are not destined to be the next ICD battery, this project proposes a new, energy-related perspective on molybdenum-based materials and a chance to further understanding of uncharted territory in chemistry.

BACKGROUND

The basic principles of a battery are important for understanding the desired properties scientists look for in a material and the common procedures they run to test candidates with promise. A battery consists mainly of a cathode and an anode separated by a liquid electrolyte and a separator (typically made of a special filter paper). When the battery operates, positively charged ions move through the electrolyte and separator to the cathode. Simultaneously, electrons travel from the anode via a connector of some kind (steel, wires, etc.). The electrons perform the desired electrical work, such as powering a phone, before they move to the cathode, where they reduce the cation in the active material and plate out a new solid (throughout which they can move and exist freely). Where cations are removed, ions from the anode take their place. **Figure 2** illustrates this process with molybdenum and silver to better portray how the cathodes in this particular study will be functioning. This process shown in Figure 2 is known as a “discharge,” whereas the reverse of this process, during which electrons and ions move the opposite way, is referred to as “recharge.”

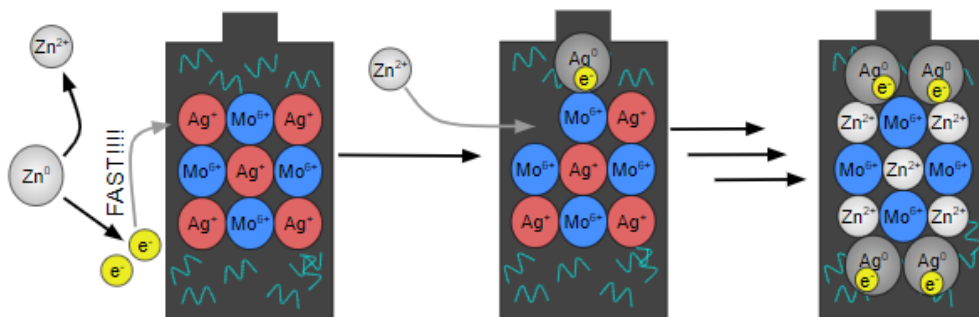


Figure 2: A simplified schematic of the discharging process an operating battery undergoes.

The most desirable batteries are fast, long-lasting, and quickly rechargeable. Finding the material(s) that are characterized by all three is the fundamental goal of battery research, but it is no small feat or easy search. Since the conventional battery structure is composed of several parts, finding the perfect setup is a challenge. However, having various modifications to explore keeps the field busy and invites viewpoints from many angles.

While studies considering the most optimal separator or electrolyte are important, the investigation of the electrodes and how to prepare them is the heart and soul of current research. The most popular anodes are lithium, sodium, and zinc, but there are many other groups exploring ways to optimize this half of the battery. This project is less concerned with the choice of anode; however, it is still essential to note that zinc was the anode for all significant experiments throughout this project. In a lab environment, stable, inexpensive zinc is favored over the flammable, highly reactive lithium. Moreover, it is still frequently seen in many other battery investigations.⁵⁻⁷

The focus of this project was directed towards the preparation and testing of four new cathodes composed of novel silver molybdate structures. The goal was not only to have a quantitative understanding of how these batteries performed, but there was also a need to explain

why one battery worked better than another. Explaining the relative performances of the cathode was broken down into a narrative with several parts.

Material Synthesis

Logically, the project began with making the materials. **Table 1** describes the general syntheses of the four silver molybdate compounds. Prior to any testing, significant differences in the procedures allowed for preliminary assumptions about how the materials might perform. According to general acid-base chemistry, Lewis acids are great electron pair acceptors. Applying the same thinking to the pH conditions of the materials, it may be predicted that the $\text{Ag}_2\text{Mo}_3\text{O}_{10}$ is synthesized in the ideal environment (with the lowest, most acidic pH) for hosting an electron pair acceptor. Moreover, if silver is the primary electron acceptor in the structure, then the highest possible Ag:Mo ratio might be preferred over a lower ratio. This thinking would predict that Ag_2MoO_4 offers the most advantageous properties. However, the greatest Ag:Mo ratio exists in the substance synthesized in the the highest (most basic) pH, and the lowest ratio exists in the substance made in the lowest (most acidic) pH; therefore, a perfect correlation cannot be observed prior to further testing.

Table 1: Synthesis procedures of the four silver molybdate materials.⁸⁻¹²

Final Product (Ag:Mo Ratio)	Synthesis Procedure
Silver Monomolybdate: Ag_2MoO_4 (2:1)	A solution of Na_2MoO_4 is added dropwise to a solution of AgNO_3 and the resulting final product is vacuum filtered at pH 10.
Monoclinic Silver Molybdate: M- $\text{Ag}_2\text{Mo}_2\text{O}_7$ (1:1)	AgNO_3 and MoO_3 are mixed in 40 mL of water at pH 4.5 and heated for 5 hours at 70°C .
Triclinic Silver Molybdate: T- $\text{Ag}_2\text{Mo}_2\text{O}_7$ (1:1)	Monoclinic silver molybdate (M- $\text{Ag}_2\text{Mo}_2\text{O}_7$) is heated to 450°C for 5 hours.

Silver Trimolybdate: $\text{Ag}_2\text{Mo}_3\text{O}_{10}\cdot 2\text{H}_2\text{O}$ (2:3)	Na_2MoO_4 is mixed with methylamine HCl. The pH of the solution is adjusted to ~ 1.3 and heated in a hydrothermal vessel for 6 hours. The precipitate is collected via filtration and refluxed with AgNO_3 in 1 M HNO_3 overnight. The final product is collected through filtration.
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Material Characterization

Following syntheses, multiple techniques were used to confirm that the materials had been synthesized correctly and ensure there were no impurities present. X-ray fluorescence (XRF) made sure the appropriate elements – silver and molybdenum – were the only ones present in the product of each synthesis. In this method, X-rays excite the electrons in an element, and the photon it produces is read by a detector. The photon produced is specific to each element since every element has a different orbital configuration. **Figure 3** depicts the resulting XRF spectra of the of the silver molybdates and a reference spectrum for the predicted peaks.

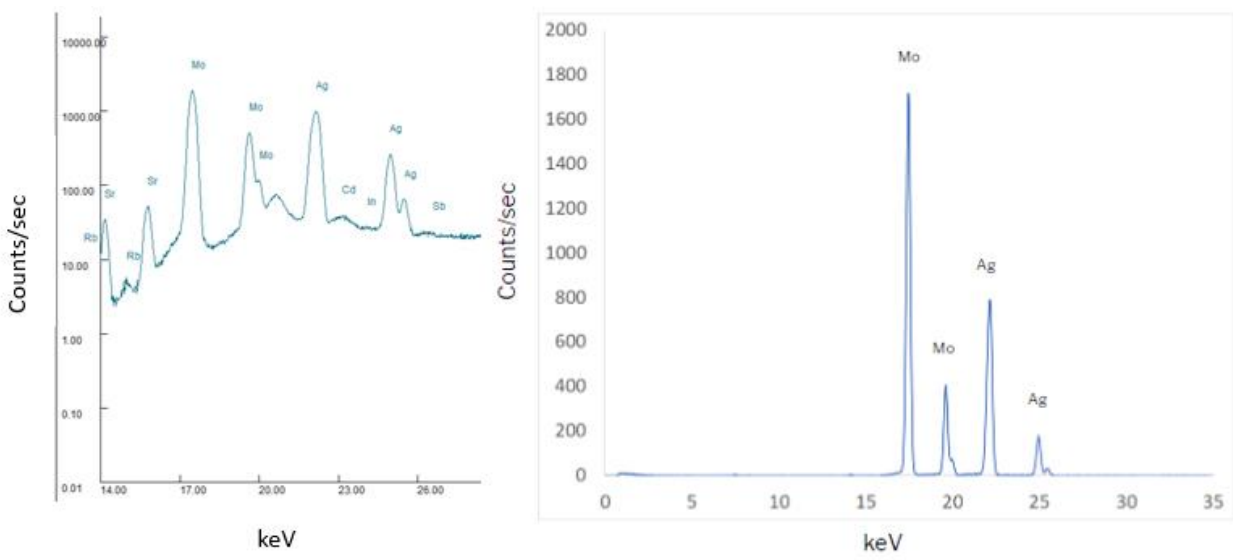


Figure 3: XRF spectra of the reference peaks of elements (left) and silver molybdate products (right). Oxygen is not shown because it is not large enough to be detected by XRF.

XRF established that only the anticipated elements were present in the products, but X-ray diffraction (XRD) techniques proved that the structures of the products formed as expected. In this technique, the angles at which X-rays diffract provide a spectrum of the distances between the atoms. Comparing the experimentally obtained spectrum to a reference spectrum checks for any impurities or structural issues in the yields of the final products (**Fig 4**).

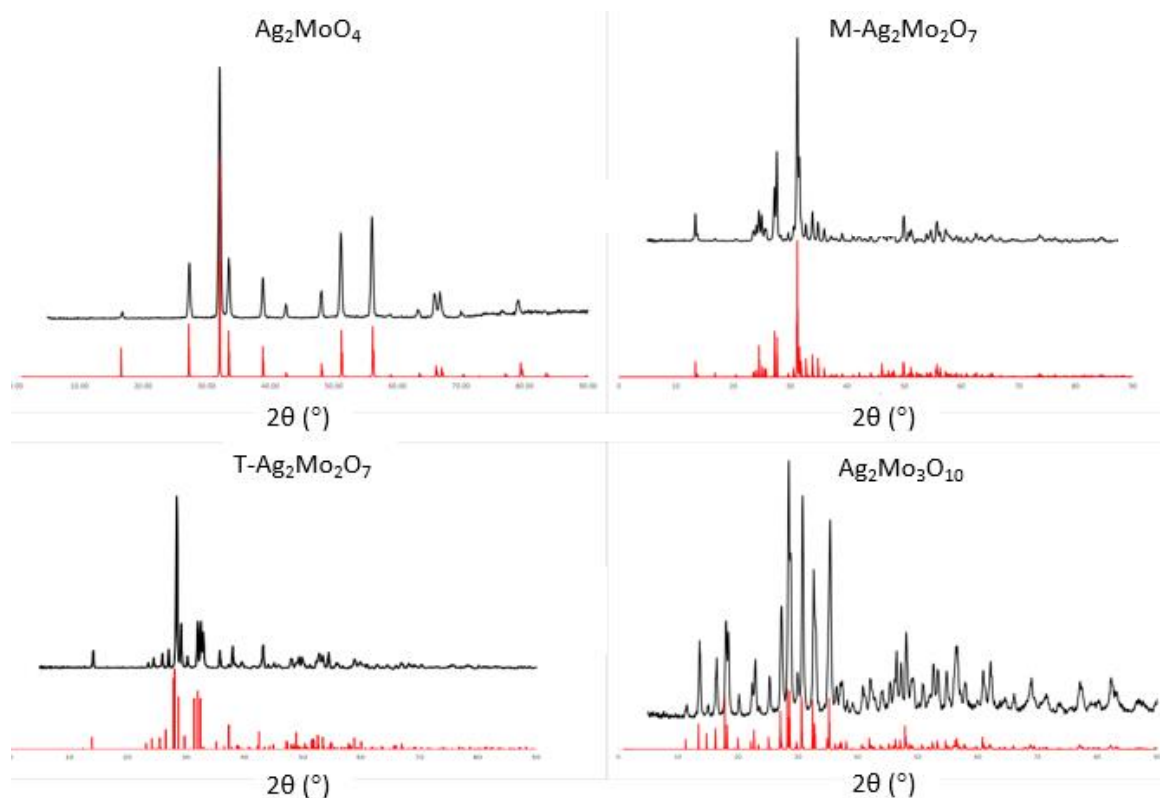


Figure 4: XRD spectra of the four silver molybdates. The black patterns represent the synthesized products, and the red patterns denote reference spectra.

Electrochemical Prepping and Testing

After XRF and XRD characterization had deemed the syntheses successful and without impurity, electrochemical testing began. The original cathode preparation procedure called for the mixing of the silver molybdates – also known as “active material” – with carbon black, and a liquid, organic binder in an 8:1:1 ratio. The slurry this mixture produced was then coated onto a

piece of thin steel, from which small, circular cathodes could be hole-punched after drying. The carbon assisted with the movement of electrons through the substance, and the binder kept the mixture sealed to the steel surface. A circle punched from a well-coated area of the steel would then be assembled into an electrochemical cell (**Fig 5**) to make a testable battery.

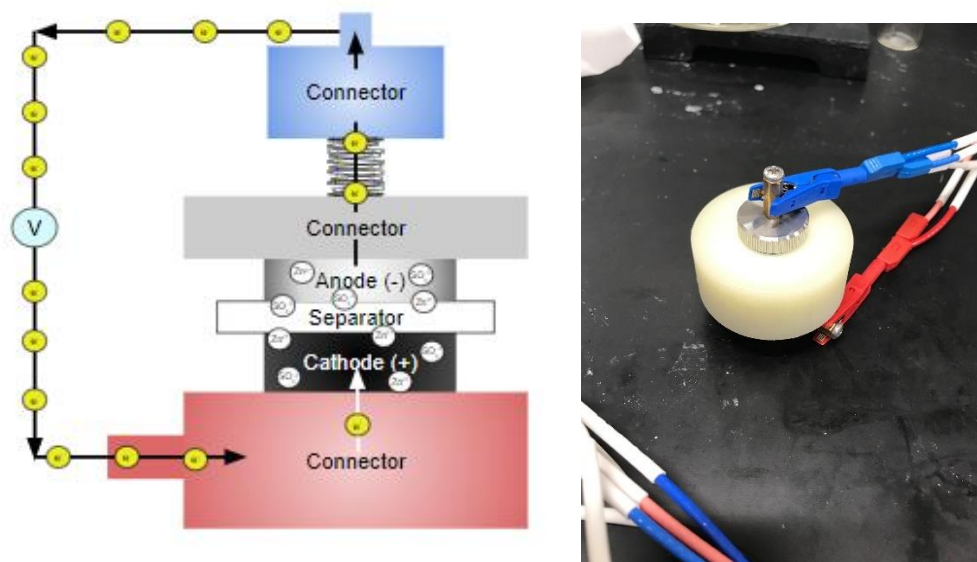


Figure 5: An animation and image of the cathode coating assembled into a functioning battery. In this setup, the coating acts as the cathode, and zinc acts as the anode. 2M ZnSO_4 is applied dropwise to the cathode and separator, and it functions as the electrolyte. Fiber glass filter paper is used as the separator. The connectors are all made of steel.

Over time, adjustments were made to this wet-coating procedure. With a hydraulic press, the dry pure materials (without carbon or binder) could be pressed onto steel mesh, and tested the same way shown in **Fig 5**. This was done to study the benefits of including carbon and binder in the mixes. Pressing was also helpful as a dry mixing procedure was explored and improved. Rather than creating a slurry, the active material, carbon, and binder were blended together and evenly dispersed by the addition of ethanol. Following the evaporation of ethanol, the remaining powder could also be dry pressed onto steel mesh and tested. This method poses an advantage over the wet-coating prep because it is less susceptible to detaching from the steel when the

electrolyte is added, and it still allows for the analysis of an 8:1:1 ratio of material, carbon, and binder.

Once the cathode had been assembled into a battery, “cyclic voltammetry,” or CV, was the first electrochemical test that needed to be conducted. This technique applies a sweeping energy to monitor current. Peaks in the CV indicate at which potentials elements are oxidizing and reducing. Prior to the procedure, it can be inferred that silver will begin to reduce near a potential of 1.0 volts and the molybdenum will reduce around a potential of 0.4 volts because the reference electrode (anode) was zinc. Silver peaks appearing at a value greater than the anticipated 1.0 V – like the silver trimolybdate peak in **Figure 6** – may be indicative of high-energy silver. This test determines the appropriate voltage range in which to operate the silver molybdate batteries. **Figure 6** provides example CVs obtained from wet coatings of Ag_2MoO_4 and $\text{Ag}_2\text{Mo}_3\text{O}_{10}$. The CVs of all four samples determined similar voltage ranges (0.1 V – 1.7 V) in which the batteries should be tested.

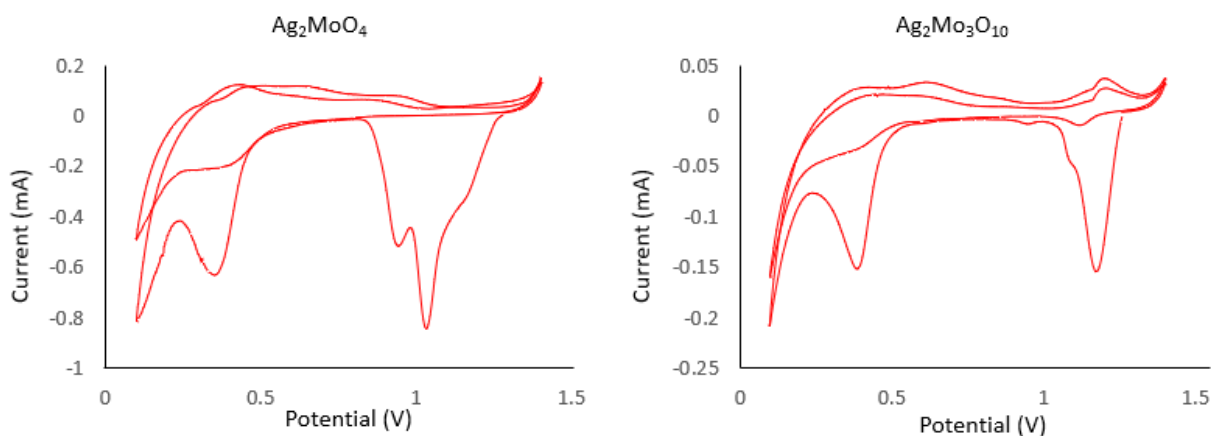


Figure 6: Cyclic voltammograms of wet-coatings. 8:1:1 mixtures of active material, carbon, and binder. The peak near 1.0 V represents the reduction of silver. The peak near 0.4 V represents the reduction of molybdenum.

Once this range was established, the discharging abilities of the batteries were tested in a technique known as chronopotentiometry (CP). This process harkens to **Figure 2**, which illustrates the movement of electrons and Zn^{2+} cations from the anode to the cathode. A battery's discharging ability is measured by the unit milliamp hours per gram (mAh/g). This is commonly referred to as capacity, which is essentially the number of electrons the cathode can hold at a certain rate in milliamperes per gram (mA/g). Mass data is included in the units to normalize performance to the amount of active material (excluding carbon and binder) in the material. The subsequent data provides potential (V) as a function of the material's capacity (mAh/g). Generally, great batteries demonstrate large capacities at fast rates. For frame of reference, the familiar Duracell® battery, which also operates with a zinc anode, exhibits an approximate capacity of 300 mAh/g. With these thoughts considered, the performances of the silver molybdates are not only comparable with each other, but they can be judged with respect to other battery candidates in papers from other projects.

The discharging capabilities of these compounds were assessed in several ways. The first analysis was done on the pure material without the aid of any carbon or binder. After the pure materials were pressed onto steel mesh and assembled into the electrochemical cell, they were all run at 50 mA/g of active material (**Fig 7**). The $\text{Ag}_2\text{Mo}_3\text{O}_{10}$ and T- $\text{Ag}_2\text{Mo}_2\text{O}_7$ materials performed the best, but this was only the first test. The following tests would add carbon and binder to the mixture. This is standard procedure in the battery community for research and commercial distribution. The carbon assists with the movement of electrons throughout the cathode, and the binder works to prevent the cathode from detaching from its metallic bottom. Since cations enter the cathode during battery operation, expansion will occur, and the binder will be necessary for keeping the material held down.

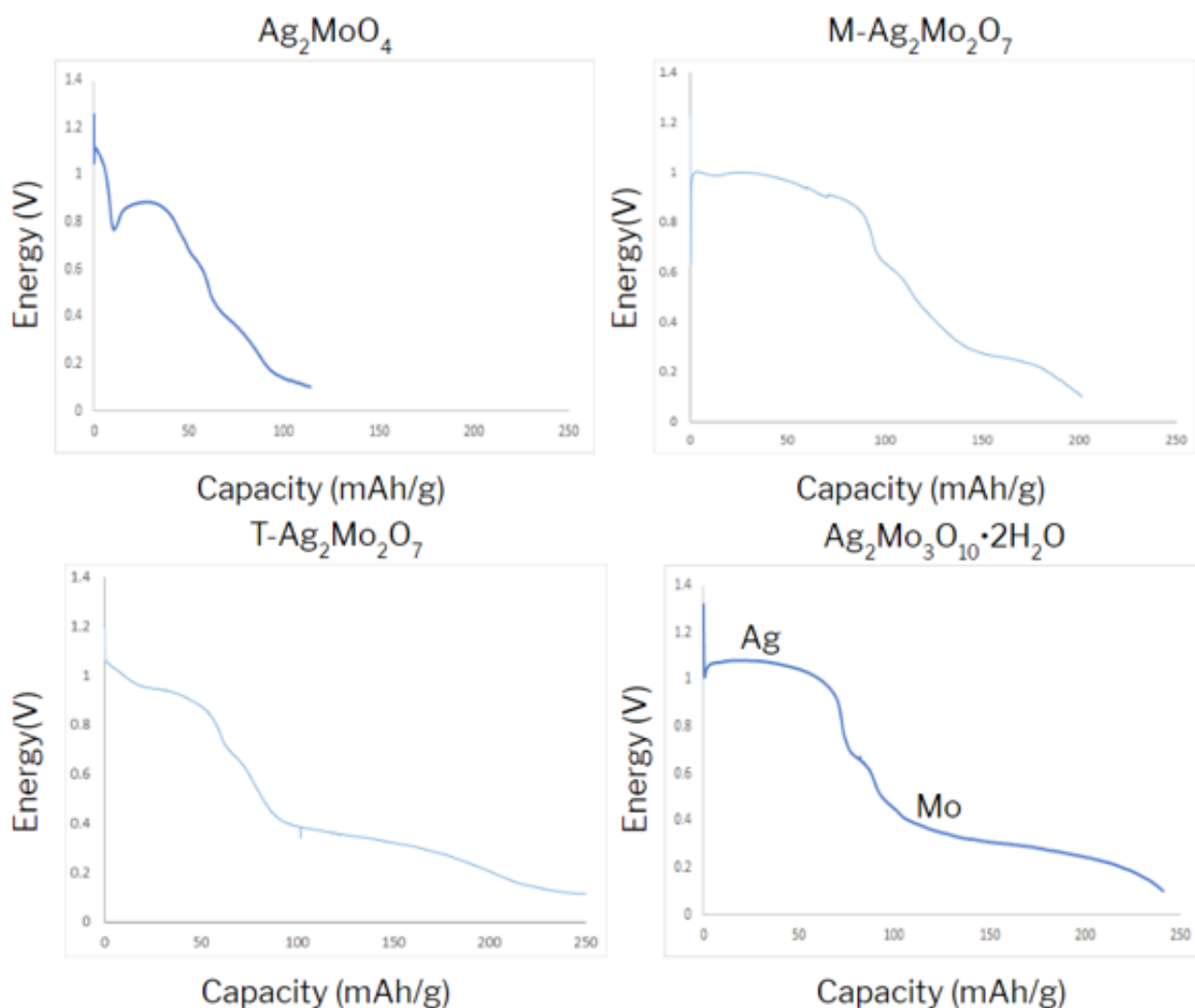


Figure 7: 50 mA/g discharges of the pure (no carbon or binder) materials.

The results were different when the materials were tested in the presence of carbon and binder. The discharges of all silver molybdates improved with the assistance from the carbon and binder, but the monoclinic material achieved a capacity around 300 mAh/g (**Fig 8**). When the rates were varied between 50 mA/g and 1000 mA/g, $\text{M-Ag}_2\text{Mo}_2\text{O}_7$ continued to run at capacities between 250-300 mAh/g (**Fig 9** and **10**). Low rates were an exception for all four samples. This

may suggest that the silver molybdates run most efficiently within a specific range. Aside from disappointing slow rate data, the results were clearly telling that the M- $\text{Ag}_2\text{Mo}_2\text{O}_7$ cathode had the greatest capacity. The task of determining why this material ran the best was the next step.

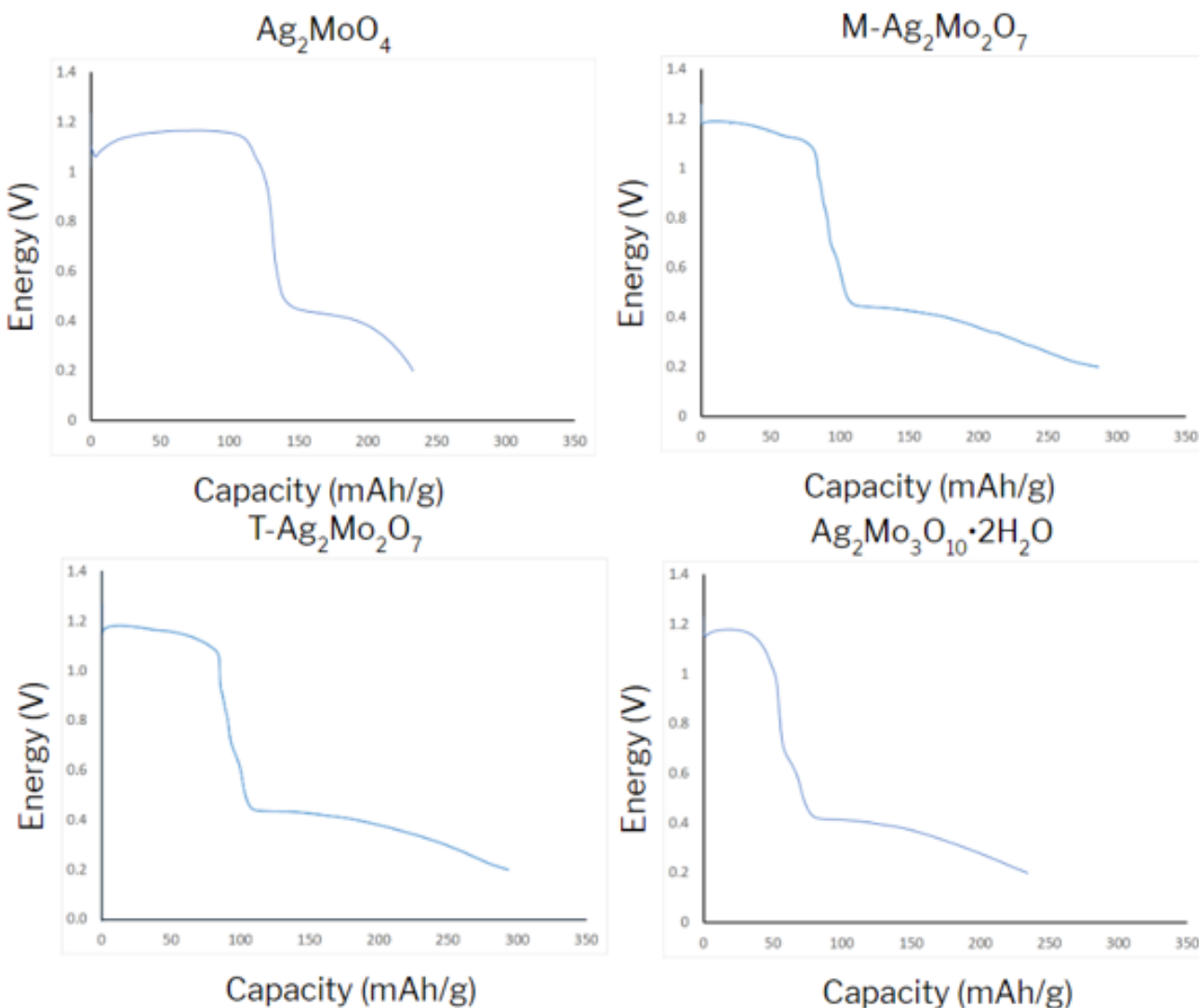


Figure 8: 50 mA/g discharges of the materials combined with carbon and binder (8:1:1).

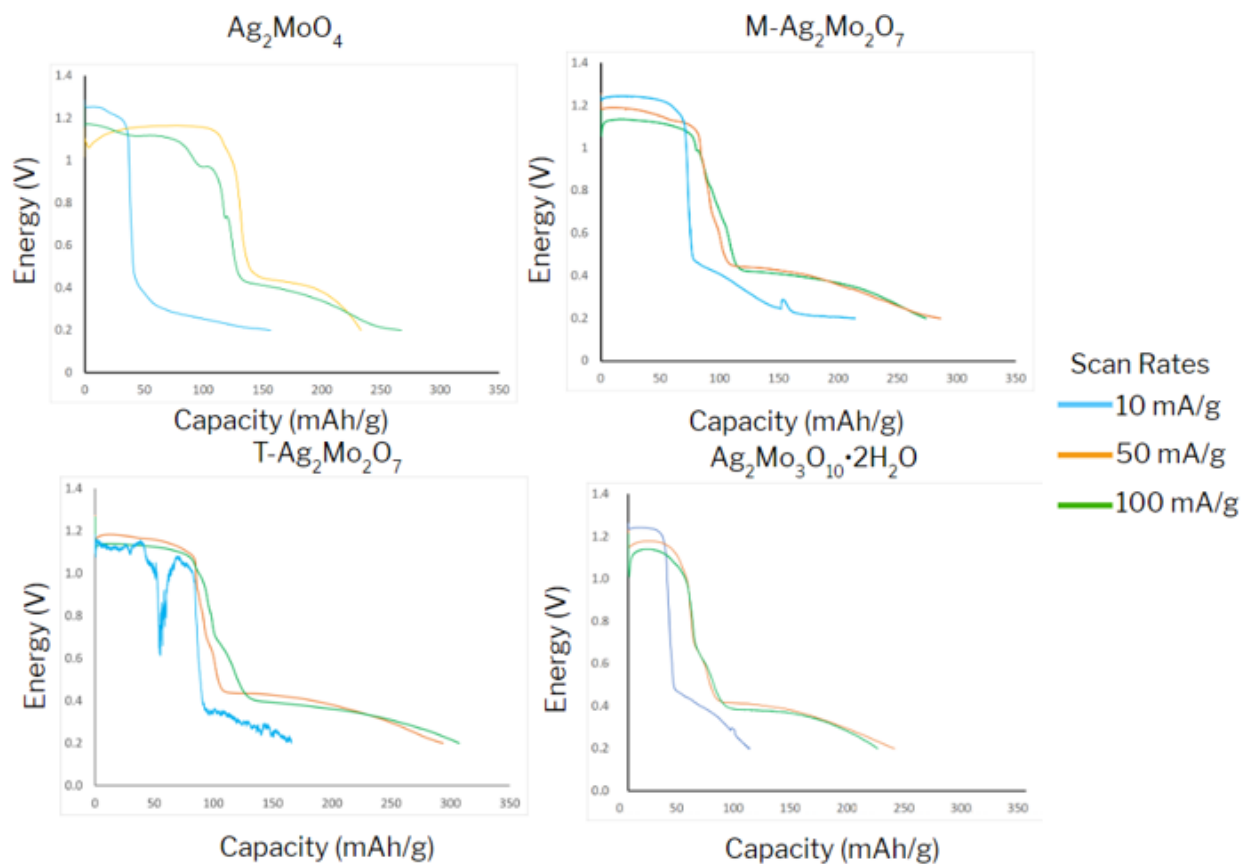


Figure 9: Discharges at different rates of the materials combined with carbon and binder (8:1:1).

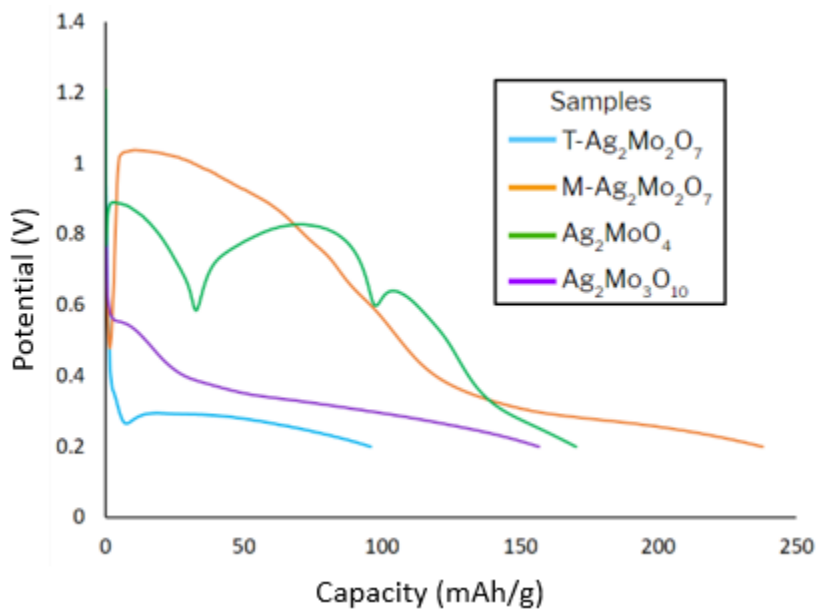


Figure 10: Discharges at 1 A/g of the materials combined with carbon and binder (8:1:1).

The CP program was set to run the battery to a certain voltage. A full discharge means that the battery operates until ~0.1 V, which ensures that both Ag and Mo were reduced. Having control over the stopping point allows for interval observations as the reaction occurs, and it makes it possible to explain why one battery operates more successfully than another. This technique not only tells which is quantitatively better, but it offers the opportunity to continue the narrative as to why one is best or worst. For example, suppose one of the silver molybdate batteries runs until capacity reaches 0.8 V, a point at which only silver could have reduced. By analyzing the partially discharged cathode with XRD, the formation of silver can be verified, and the crystal sizes of the silver could be calculated. With XRD, this crystal size analysis was conducted on partial discharges of the silver molybdates (**Table 2**).

Table 2: Crystal sizes of the materials before and after battery operation. Values were calculated with the Scherrer equation and peak widths from XRD spectra.

Material	Starting Material (nm)	Discharge to 0.8 V (nm)	Discharge to 0.2 V (nm)
Ag ₂ MoO ₄	29	8.7	17.4
M-Ag ₂ Mo ₂ O ₇	22	15	11
T-Ag ₂ Mo ₂ O ₇	12	14	16
Ag ₂ Mo ₃ O ₁₀ •2H ₂ O	23	27.4	20.4

Although a relationship was predicted, the results were not telling of a relationship between crystal sizes and capacity. Crystal size values demonstrated some variance from one compound to another, but there was no logical trend. Initially, one might have thought that if silver was the primary electron acceptor, then a larger crystal size would have been indicative of

a better battery material. However, this was not the case and inquiries would need to shift to a highly advanced technique known as scanning electron microscopy (SEM). In SEM, the sample is blasted with electrons, and the diffracting electrons create high-resolution images on the micron and nanometer scales of the surface of the material. This method is also capable of elemental mapping, when the electrons are projected deeper into the sample past its surface.

SEM was the next chapter in the narrative. If crystal sizes, pH conditions, and Ag:Mo ratios do not clarify the differences in the performances of the silver molybdates, then SEM may offer some reasoning. With SEM, the morphology of the materials and the grouping of the elements in the structures could be visualized. The distinct morphologies are immediately apparent when surface images are taken of each sample. In **Figure 11**, the best performing material, the M-Ag₂Mo₂O₇, showed a morphology composed of nanowires. The Ag₂Mo₃O₁₀ compound did not demonstrate nanowires that were as well-defined as those of the monoclinic, but its morphology was still much less globular than those of the T-Ag₂Mo₂O₇ and Ag₂MoO₄. These images of the pure materials taken before any battery assembly or reaction could be partially telling as to why monoclinic performs so well compared to the other three substances.

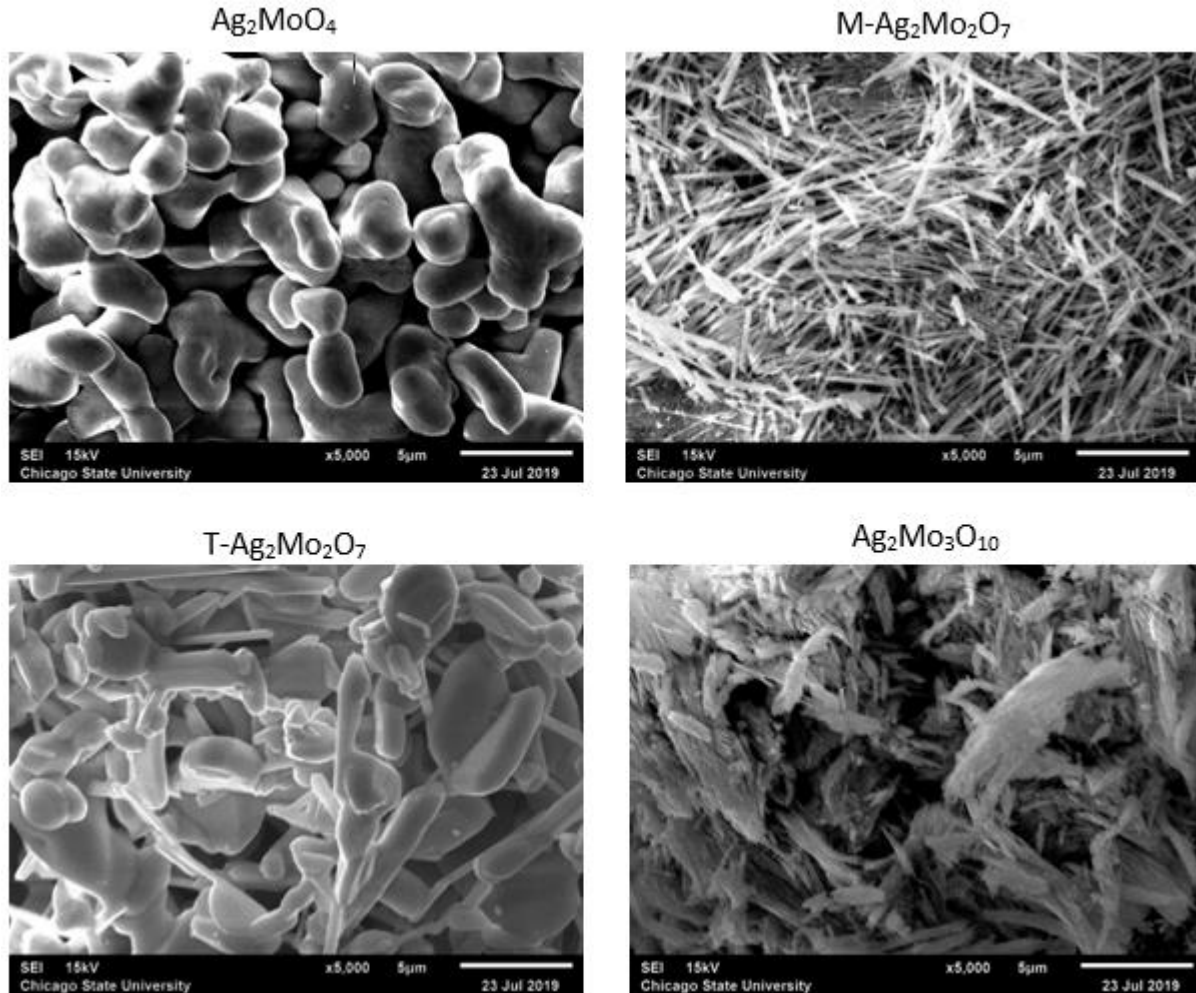


Figure 11: SEM images of pure materials.¹³

Furthermore, partial discharge data was especially helpful in describing why some of the materials worked better than others. To coincide nicely with the images from Chicago State University, partial discharge images acquired at Argonne National Labs showed a noticeable relationship between the morphology of the active material and the elemental grouping following the reduction of silver in a pure material cathode. When analyzing the complete discharge data, it is clear that all cathodes experience drastic elemental separation when they are operated to 0.1 V, and it is difficult to spot significant or telling differences among the electrodes and the segregations of their elements. However, analysis of an electrode, in which only silver was

reduced, finds differences between the materials that had been regularly successful and the materials that have normally struggled more times than not.

In the case of the best silver molybdate, M-Ag₂Mo₂O₇, elemental mappings of discharges to 0.8 V found that the various elements present did not spread out in any evident fashion. When zinc moves to the cathode, it plates out silver. When zinc does this in an M-Ag₂Mo₂O₇ cathode, silver remains close to the zinc that supposedly displaced it from the structure. When analyzing the middle-tier molybdates, Ag₂Mo₃O₁₀ and T-Ag₂Mo₂O₇, elemental migration is slightly more apparent. This movement is less obvious in the images of the Ag₂Mo₃O₁₀ (the material that achieved the greatest capacity during the pure discharge) and is possibly more similar to the lack of shifting shown in the M-Ag₂Mo₂O₇ figures, but there are a couple regions (**Fig 12, D and E**) in the T-Ag₂Mo₂O₇ images where there is clear separation between the silver and groupings of molybdenum and zinc. The most obvious cases of segregation are visible in the Ag₂MoO₄ images, where there are at least three regions (**Fig 12, A, B, and C**) in which the silver is clearly depleted, and the zinc and molybdenum are concentrated.

These noteworthy areas in the SEM figures, combined with the original morphologies of the pure substances, are likely the answers to the questions on performance. This is not the first instance where morphology has greatly enhanced or impaired a material's ability to operate. Other studies on battery materials, like β-AgVO₃, have found dramatic differences in recharging and discharging performances between different morphologies of the same material.¹⁻² Thus far, a nanowire morphology continues to beat an amorphous morphology in vanadium compounds and apparently the molybdenum compounds in this project.

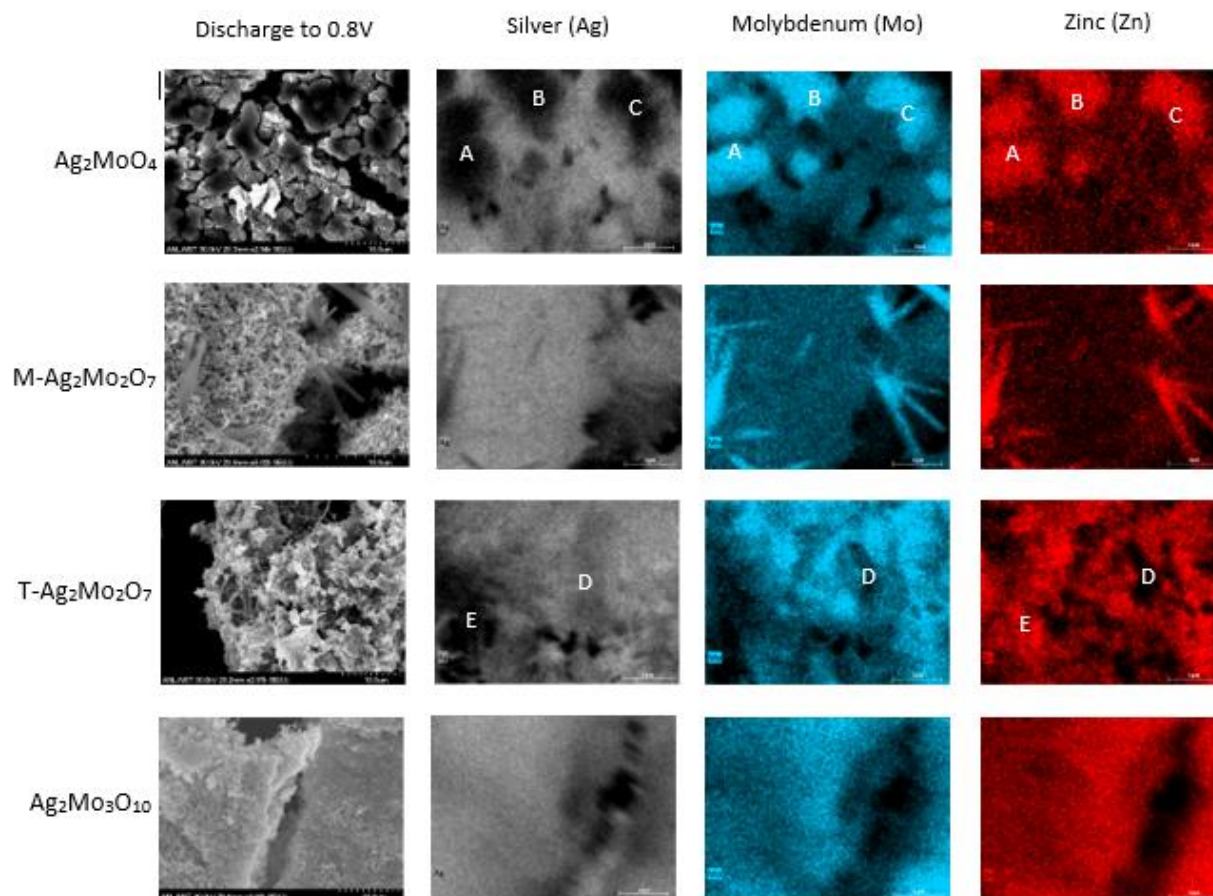


Figure 12: SEM images and elemental mappings of cathodes discharged to 0.8 V. Regions A-E represent spaces where there is a clear discrepancy between the concentration of silver and the concentration of molybdenum/zinc.¹⁴

Although the recharging abilities of these silver molybdates were given some attention prior to the acquisition of SEM images, they are continuously receiving further evaluation as the coating method progresses and understanding of the materials improves. Even though the project has come a long way, the current framework of our battery systems is not ideal for recharge data. First, a lithium anode is more optimal when trying to demonstrate recharge, but it is less practical for this project considering the primary use of zinc and the hazards that come with a highly reactive substance. Second, silver is not renowned for its ability to recharge. In fact, other studies have shown that, because of silver's high affinity for electrons, it is less inclined to return them

to the cathode. Nevertheless, it is still important to explore rechargeability. Achieving ways to improve it or simply knowing what these molybdates are capable of are critical aspects of the narrative being constructed around these materials. Recharge data is often referred to as “cycling data.” The battery discharges to a selected voltage (electrons/ions move to the cathode), and then it recharges to a selected voltage (electrons/ions return to the anode). The combination of a discharge and its following recharge is called a “cycle.” As a battery runs, it often loses capacity from one cycle to the next. A decent rechargeable battery can recover most of its capacity as it cycles (continues to recharge and discharge), but the best rechargeable batteries can cycle many times before it struggles to reach its initial capacity.

Keeping in mind previous electrochemical results and SEM images, the trend remained relatively similar, according to **Figure 13**. It was no surprise to find that the monoclinic silver molybdate demonstrated the best recharge data when the original wet-coating procedure was tested. The trimolybdate and triclinic materials ran almost identically with their capacities topping off around 200 mAh/g on their first discharge and recovering approximately 50% of the capacity. The monomolybdate cathode only appears superior because the electrolyte began to reduce during the discharge. To this date, Ag_2MoO_4 struggles to deliver beyond the recharge phase of its first cycle.

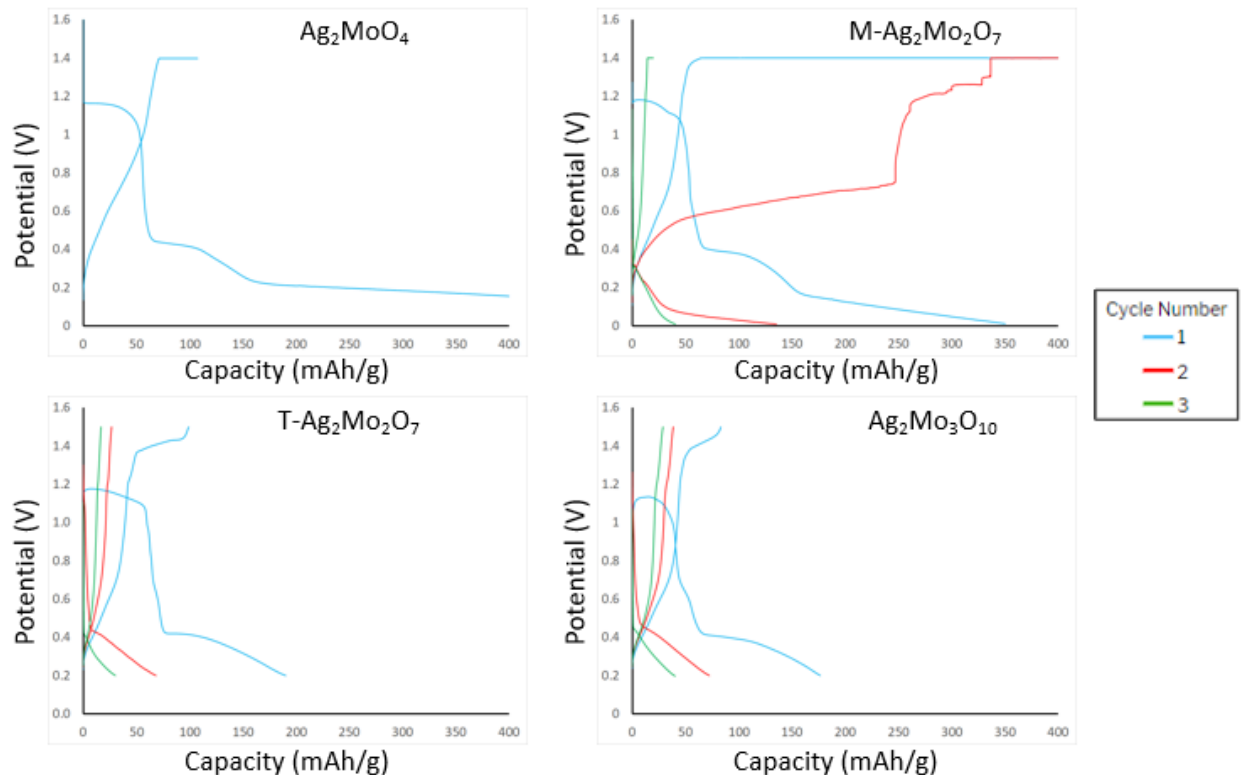


Figure 13: Recharge cycling data of the four silver molybdate compounds operated at 50 mA/g. All cathodes were made from a wet-coating procedure (8:1:1; active material, carbon, and binder). They were discharged to 0.2 V and recharged to 1.4 V.

Figure 14 and **Figure 15** present cycling data obtained from cathodes prepared through a dry-mixing procedure that were only partially discharged to 0.8 V and recharged to 1.7 V. The Ag_2MoO_4 continued to fall short, whether it was fully or partially discharged (**Fig 14, A and B**). The discharge half of its cycles were not entirely messy, but the battery appeared to short on the recharge and fail to reach 1.7 V. The $\text{Ag}_2\text{Mo}_3\text{O}_{10}$ cathode demonstrated impressive recovery percentages when it only discharged partially. The capacity of the first recharge recovers by about 60%, while recharges two through four appear to recover between 80-100% each (**Fig 14, C**). This level of success may be explained by the minimal elemental separation shown in the SEM images of the partially discharged $\text{Ag}_2\text{Mo}_3\text{O}_{10}$ electrode (**Fig 12**). If the zinc and silver remain local following the discharge, then reversing the process may be less difficult. This

argument is supported by the fewer cycles and low capacity recovery depicted in **Figure 14, D**, which shows the effect of reducing all the way past molybdenum.

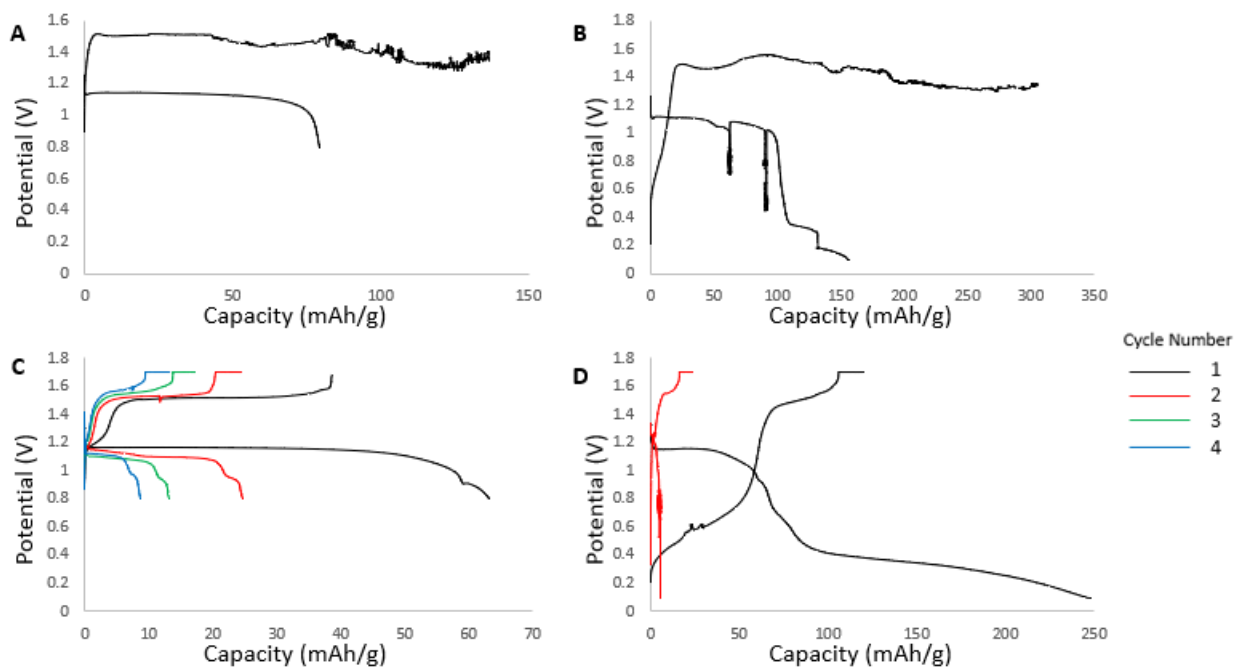


Figure 14: Recharge cycling data of (A) Ag_2MoO_4 cathode discharged to 0.8 V, (B) Ag_2MoO_4 cathode discharged to 0.1 V, (C) $\text{Ag}_2\text{Mo}_3\text{O}_{10}$ cathode discharged to 0.8 V, and (D) $\text{Ag}_2\text{Mo}_3\text{O}_{10}$ cathode discharged to 0.1 V. All cathodes were made from a dry mix (8:1:1; active material, carbon, and binder) pressed onto steel mesh. All were recharged to 1.7 V and operated at 50 mA/g.

In **Figure 15**, monoclinic and triclinic showed promise when asked to perform one cycle in the dry mix, averaging a capacity recovery around 70% (**Fig 15, A and C**). Issues arose when multiple cycles were attempted. The capacity recoveries of the triclinic cathode were respectable, and three cycles were managed. However, there was a sizeable capacity difference between the first discharge and the two that followed (**Fig 15, D**). The standard front runner, monoclinic, shorted on both of its recharges (**Fig 15, B**). Considering the success this material has shown previously, these shortages are best viewed as mistrials, and the results are best held

inconclusive. More recharge data will need to be gathered, and this type of coating will need to see more trials in the lab.

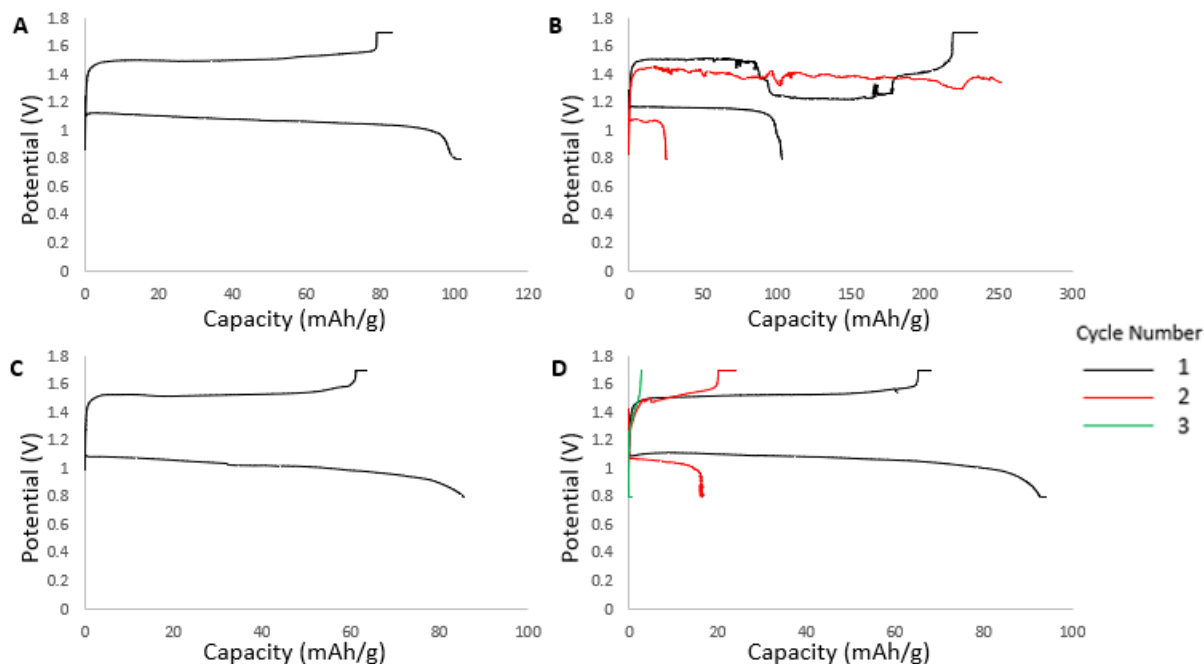


Figure 15: Recharge cycling data of (A) M-Ag₂Mo₂O₇ cathode operated at 50 mA/g through one cycle, (B) M-Ag₂Mo₂O₇ cathode operated at 40 mA/g through (an attempted) three cycles, (C) T-Ag₂Mo₂O₇ cathode operated at 50 mA/g through one cycle (D) T-Ag₂Mo₂O₇ cathode operated at 40 mA/g through three cycles. All cathodes were made from a dry mix (8:1:1; active material, carbon, and binder) pressed onto steel mesh. All were discharged to 0.8 V and recharged to 1.7 V

CONCLUSION

After successfully synthesizing and characterizing the four silver molybdate materials of interest, electrochemical testing provided answers as to which compound made the best battery. Enough data had been procured to establish a hierarchy for the battery candidates, and the order from least optimal to most optimal went Ag₂MoO₄ < T-Ag₂Mo₂O₇ ~ Ag₂Mo₃O₁₀ < M-Ag₂O₂O₇. The pursuit for answers as to why this was the order began with a consideration of the pH and Ag:Mo ratios. The investigation would eventually lead to a look at the crystal sizes of the silver

particles. Finally, SEM images would offer suggestions as to why the electrochemical data were falling in the aforementioned hierarchy. The top batteries, according to our research and the research of other groups, have an organized morphology and contained elemental packing. The data indicates that an orderly nanowire will run more efficiently than a blob of material, and it will discharge in a manner that can more readily reverse itself. This is not to say pH, Ag:Mo ratios, and crystal sizes are complete nonfactors, but the results are saying that a well-ordered morphology is fundamental in the success of these silver molybdates as batteries.

FUTURE WORK

Although the project has come a long way, there are still several ways to continue exploring these silver molybdate materials. The recharging capacities are still not fully understood, especially when the batteries are only partially reduced. More trials with the new cathode prepping procedure would be ideal as well. Argonne National Labs has already permitted more imaging with transmission electron microscopy (TEM), a technique even more advanced than SEM. Lastly, it was recently found that the silver monomolybdate, Ag_2MoO_4 , may actually have two forms (referred to as alpha and beta). This came to light when a cathode of Ag_2MoO_4 made from the new dry-mix prepping procedure ran to a capacity of approximately 297 mAh/g (**Fig 16**). This is the best this material has performed since the beginning of the project, and it would be valuable to know which of the forms can handle this level of capacity.

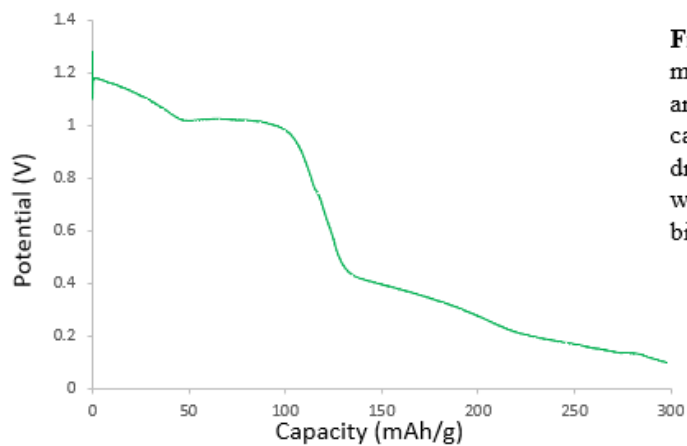


Figure 16: 40 mA/g discharge of an Ag_2MoO_4 cathode made from dry coating method with carbon and binder (8:1:1).

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I have neither given or received, nor have I tolerated others' use of unauthorized aid.