



Structural properties and ion diffusion pathways in molybdenum sulfide materials of interest for Li-S batteries



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Abstract

Rechargeable battery systems, such as lithium-sulfur batteries, have garnered significant attention due to their high specific energies, lightweight form factors, and cost. However, challenges such as polysulfide shuttling and limited recharge cycles remain as shortcomings. Molybdenum sulfide-based chalcogels demonstrate potential to address these limitations as they show enhanced kinetics of lithium diffusion and the ability to mitigate polysulfide shuttling in lithium-sulfur (Li-S) systems. Here we are interested in developing structure–function relationships of molybdenum sulfide structures identified in the chalcogels through density functional theory (DFT) calculations and first-principles molecular dynamics simulations. We explore diverse sampling trajectories to extract structural factors, pair distribution functions, and bond structures to identify models that closely align with experiment. We then aim to elucidate ion diffusion pathways within the different molybdenum sulfide structures. The results of these studies provide fundamental understanding of the relationships among the composition, structure, and ion diffusion pathways in molybdenum sulfide-based chalcogels with the goal of delivering insights for future materials development.



Supercapacitor Electrodes Obtained from Hydrothermal Carbonized Bourbon Stillage



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Abstract

Bourbon whiskey is "a distinctive product of the USA," mainly made in Kentucky (95% of the world's production). The bourbon industry has filled around 2.4 million barrels annually since 2020. However, one of the industry's biggest challenges is the spent grain (stillage) problem. For every barrel of bourbon produced, it is estimated that six to ten barrels of stillage remain. This research aims to transform the stillage into supercapacitor electrodes. Supercapacitors are high-performance electrochemical energy storage devices frequently complementary to Li-batteries and ideal for acquiring energy from alternative sources. The stillage is hydrothermally carbonized at high pressure and temperature, followed by steam activation to enhance the surface area and porous structure. The resulting activated carbon interacted well with aqueous electrolytes, especially in 1 M sulfuric acid, where the equivalent series resistances were the lowest (ESR=0.92 m Ω). Supercapacitor electrodes displayed specific capacitances of 183.3 F/g at 10 A/g in 1M sulfuric acid, retaining up to 100% of its capacitance after 10,000 charge-discharge cycles. This research shows an innovative, potentially scalable, and sustainable strategy to repurpose the bourbon stillage into efficient, green, and high-performance materials for energy storage applications, potentially expandable to the whiskey and ethanol industries in the USA.

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Single Atom Catalysts: Understanding Synthesis and Catalytic Activity towards Oxygen Reduction Reaction



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Abstract

Single-atom catalysts (SACs) is emerging as a promising candidate for electrocatalysis, while offering several crucial advantages: (1) efficient utilization of costly and rare metals through achieving maximum metal utilization, and (2) selective activity through modification of metal-ligand configurations. A prevalent SAC structure involves metal-nitrogen-carbon (M-N-C) arrangements, where single metal (M) atoms serve as active sites for molecule adsorption and desorption, stabilized by coordination with nitrogen atoms. Despite extensive research on SACs, significant challenges persist: (1) inadequate anchoring of single metal atoms on catalyst supports leading to metal agglomeration, (2) difficulty in achieving uniform distribution of metal atoms on the support, and (3) restricted metal loading (<5wt%), hindering the commercialization of SAC-based technology.

In this symposium, we will unveil effective and adaptable methods for producing high metal loading Cu- and Fe-based Single-Atom Catalysts (SACs). Our synthesis predominantly yielded catalysts consisting of individual metals, where metal atoms were uniformly dispersed with high metal loading (Cu and Fe loadings of up to 20 wt.%). The initial synthesis procedure involved utilizing citric-acid-derived carbon nanodots (CNDs) to encapsulate individual metal atoms, followed by pyrolysis in the presence of urea. This process facilitated the incorporation of nitrogen and metal into graphitized carbon, resulting in the formation of metal-nitrogen coordination. In the second synthesis approach, ZIF-8 was employed as a precursor for capturing metal, followed by pyrolysis. The location and distribution of SACs were characterized using high-resolution scanning transmission electron microscopy (HR-STEM). Additionally, the chemical composition and loading of SACs were assessed through X-ray photoelectron spectroscopy (XPS) and thermogravimetric analysis (TGA), respectively. Both Cu- and Fe-based SACs underwent evaluation for their performance in the electrochemical oxygen reduction reaction (ORR) and were compared with commercial Pt/C catalysts. Notably, they exhibited overpotentials and limiting currents comparable with those of the commercial Pt/C catalyst.



Manufacturing Scale-Up of Supported Loose-Nanofiltration Polymeric Membranes with Eco-Friendly Solvents for Water Filtration



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Abstract

In this study, loose nanofiltration membranes made of polysulfone dissolved in co-solvents PolarClean and gamma-Valerolactone were prepared via slot die coating (SDC) on a roll-to-roll (R2R) system by directly coating them onto a support layer or free standing. A solution flow rate of 20 mL/min, substrate speed of 17.1 mm/s, and coating gap of 0.1 mm resulted in the formation of membranes without structural defects. Pre-wetting the support layer with dope solution minimized shrinkage of membrane layer thickness and improved interfacial adhesion. Membrane samples produced using SDC exhibited properties and performance consistent with bench-scale doctor blade extruded samples; pre-wetted and uncompressed samples (SDC-3) exhibited the highest rejection of bovine serum albumin ($99.20\% \pm 1.31\%$) and along with adequate mean permeability during filtration (70.5 ± 8.33 LMH/bar). This study shows that combining sustainable materials development with SDC provides a holistic approach to membrane separations to bridge materials discovery and membrane formation.



Synthesizing a Copper-Atom Catalyst for Use in Clean Energy Conversion Technologies



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Abstract

The electrocatalyst is pivotal in determining the cost and efficiency of clean energy conversion technologies. Current noble-metal catalysts have certain limitations preventing them from being implemented on a global scale. First, these catalysts are not cost effective due to low metal utilization. Second, they involve a large wastage of metal due to the oxidation and dissolving of bulk metal during electrolysis. Third, the surface-level attachment of metal to the carbon support is unstable shown by the removal of the metal during electrolysis. We proposed to combat these limitations by synthesizing a single-atom catalyst (SAC) using a common transition metal to replace noble- and bulk-metal catalysts. In our experiment, we used bottom-up synthesis and heat stabilization techniques to synthesize a copper-atom catalyst from a citric acid precursor. After confirming the SAC structure, we immobilized it onto a carbon backbone using a layer-by-layer technique. This technique allowed us to create a stable electrode with our SAC to undergo carbon dioxide reduction (CO₂RR). Preliminary data shows evidence of an effective and economically viable catalyst to be used in a variety of clean energy conversion technologies.



Investigation and Optimization of Isoeugenol Dimers in the Pursuit of a Novel Therapy for Triple Negative Breast Cancer



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Abstract

Triple negative breast cancer (TNBC) treatment lacks targeted therapies due to the minimal or non-existent expression of three receptors: estrogen, progesterone, and HER2. Non-targeted therapies include chemotherapy, mastectomies, and radiation, all of which gravely impact quality of life. It is imperative that we develop novel compounds and identify targets to improve treatment options. A recent study posited that the plant-derived compound Machilin D is an anti-tumorigenic agent that also exhibits anti-proliferative activity in breast cancer stem cells. This study isolated the compound from saururus chinensis, a plant commonly used in traditional Chinese medicine, but did not offer a synthetic route. I have successfully synthesized Machilin D through a convenient one-hour, one-step synthesis using an inexpensive starting material: isoeugenol. This synthetic route offers modification of the compound through inclusion of various hydroxy-containing reagents. By leveraging this modifiable site, we have identified a more potent compound with the potential for further optimization. Additionally, this novel synthetic route presents the opportunity for target identification towards the goal of targeted TNBC therapies.



Catalytic Reduction of CO₂ Using Re(I) complexes with Pyridyl-Imidazoles



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Abstract

Carbon dioxide (CO₂) comprises a large amount of greenhouse gas emissions globally and is relatively difficult to convert into carbon monoxide (CO) under standard electrochemical conditions without the use of a catalyst. The CO that is produced from this catalysis can be utilized in the Fischer-Tropsch process in which CO is converted into hydrocarbon fuels, thus recycling the CO₂ from the atmosphere. Previous catalyst species have utilized rhenium pentacarbonylbromide (Re(CO)₅Br) to synthesize efficient and selective Rhenium (I) catalysts using ligands such as bipyridine with bidentate coordination to accomplish the reduction of CO₂ at much lower overpotentials. We utilize a pyridyl-imidazole core for coordination with Re(CO)₅Br and as a bridging point for the introduction of donor (D) and acceptor (A) moieties to improve the catalyst's ability to absorb light. The D- π -A structure is also known to introduce a charge-transfer (CT) band which could prove useful in this type of catalysis. We herein report a series of Re(I) complexes that act as electrocatalysts for the reduction of CO₂.



Monitoring the conformational ensembles of an Electron Transfer Flavoprotein in solution



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Abstract

Bifurcating electron transfer flavoproteins (Bf-ETFs) perform electron transfer bifurcation; in multiple microorganisms, including both bacteria and archaea. Bf-ETFs contain two flavin adenine dinucleotide (FAD) molecules. The bifurcating FAD accepts a pair of electrons from NADH and dispenses them to two separate pathways. An exothermic electron transfer (ET) reaction to a high-potential acceptor, mediated by the second FAD termed ET-FAD, drives the endothermic transfer of the second electron to a lower potential acceptor: ferredoxin or flavodoxin semiquinone. The open and closed states of *Acidaminococcus fermentans* (Afe) ETF have been observed by X-ray crystallography, however it is established that an ensemble of conformational intermediates is critical to maximize the energy efficiency of bifurcation. ^{19}F NMR provides high sensitivity spectra in which the observed chemical shifts are highly responsive to local chemical environment change, allowing observation of Bf-ETF's conformational dynamics. In the current study, incorporation of 5-Fluo-Trp in wild type and mutant AfeETF was accomplished, and ^{19}F NMR measured with the objective of monitoring the conformational ensembles. Future directions include investigating the spectral responses upon partner protein binding and/or different redox states. Additionally, we are assigning the observed signals to specific residues based on mutagenesis, to obtain insight into local structural features.



A single H-bond tunes flavin redox reactivity and activates it for modification



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Abstract

Flavin based electron bifurcation (FBEB) generates high energy reducing equivalents at the expense of mediocre and abundant reducing energy, and takes place in heterodimeric proteins, known as bifurcating electron transfer flavoproteins (Bf ETFs) comprising of two flavins- electron transfer FAD (ET-FAD) and bifurcating FAD (Bf-FAD).(1) It involves the splitting of a pair of electrons in two separate but tightly coupled pathways, an exergonic pathway which pays for the endergonic pathway. In FBEB, a two-electron source such as NADH reduces the bifurcating interface, the Bf-FAD from which the two electrons bifurcate, one goes downhill to a high potential acceptor the ET-FAD and the second reduces low potential acceptor flavodoxin or ferredoxin (Fld- or Fd-). Gating the second electron uphill to a low potential pathway is made possible due to 1e- reactivity of ET-FAD, and conformational dynamics which moves Bf-FAD further away (> 14 Å) from ET-FAD preventing the downhill flow. The Bf ETFs are self-contained to tune the reactivity of ET-FAD allowing it to do 1e- chemistry unlike the usual 2e- reactivity.(2, 3) Our hypothesis states that a His residue (H290 in *A. fermentans* ETF) preferably situated close to O2 of ET-FAD plays a significant role in its 1e- reactivity by stabilizing the ASQ (anionic semiquinone) state via H-bond donation and is also responsible for chemically modified ET-FAD at C8m position due to stabilization of the methide intermediate which also concentrates the electron density at O2 position.(4)

Herein we altered that specific interaction by generating H290 variants which retained the characteristics of Bf ETFs and demonstrated via reductive and potentiometric titrations that removal of this H-bond destabilizes the ET-FAD ASQ state relative to the oxidized state ($E^{\circ}ASQ/OX$) by 150 mV and averts the modification at C8m position confirmed via UV-visible spectroscopy and mass spectrometry. Thus, we confirmed that this H-bond donation by H290 is responsible for stabilizing the excess electron density at O2 position in either of the ET-FAD species (ASQ state or the ET-FAD methide intermediate). This study was supported by support of N.S.F. under award CHE 2108134.

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Chiral Organic-Inorganic Halometallates for Piezoelectric Applications



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Abstract

The current state of the art for piezoelectricity are ceramics and polymers. Ceramics have a high piezoelectric response factor but are rigid and inflexible, while polymers are low in piezoelectric response factor but are flexible. The future of piezoelectric materials may lie in organic-inorganic halometallates (OIHM). These materials offer a high piezoelectric response and flexibility, making them ideal for medical devices in the human body, such as pacemakers. OIHM crystals must lack a center of inversion to exhibit piezoelectric properties. To ensure this, most researchers employ non-chiral cations, however, non-chiral materials cannot guarantee a non-centrosymmetric crystal lattice. To ensure a non-centrosymmetric crystal lattice, chiral molecules based on *S*-*a*-methylbenzylamine (MBA) and their enantiomers, were used for a proof-of-concept wherein we substitute at the para-position of the aromatic ring (an electron donor, electron acceptor, and inductive electron acceptor) to understand how electron density on the ring might impact the piezoelectric properties of the molecule. The derivatives of MBA were synthesized, characterized, and crystalized with and without the metallate anions.



Continuous Frontal Analysis to Study Lignan-Cyclodextrin Interaction



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Abstract

Lignin-first approach is a key to sustainable conversion of agricultural biomass to biofuels by generating valuable chemicals from the lignin portion. Cyclodextrin-functionalized particles are promising materials to selectively recover expensive lignans through guest-host complex formation from aqueous stream. Syringaresinol and pinosresinol are two such lignans with multiple therapeutic and industrial applications. This project aims to evaluate their interactions with cyclodextrins to facilitate the design of suitable recovery materials. Preliminary investigation was performed by high performance liquid chromatography using commercial C18 and beta-cyclodextrin packed columns. Beta-cyclodextrin demonstrated the ability to separate both lignans in highly aqueous conditions, unlike C18. It was also observed that more substituted analyte exhibited less retention suggesting that gamma-cyclodextrin could serve as a better host for syringaresinol because of its larger cavity. Further investigation was conducted by continuous frontal analysis capillary electrophoresis. A method was developed to determine binding constants of competitors and analytes which would provide insight into the analytes' binding mechanisms to cyclodextrins. The method was validated by calculating the binding constant of phenol and comparing the result to literature values. Future research will focus on determining the binding constants of lignans for cyclodextrins using 2-naphthalenesulfonate as competitor.



Multifunctional Nanocomposite Polysulfone Membranes using 2D nanomaterials for High Performance Water Separation



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Abstract

2D nanomaterials are presenting a promising avenue in confronting the challenge of water purification owing to their unique properties due to their ultrathin structures. These 2D nanomaterials affect the intrinsic properties of membranes including hydrophilicity, pore size, and surface charge to impart additive benefits such as adsorption or catalysis, to filtration. The research focus on non-carbon 2D nanomaterials, specifically phosphorene and hexagonal boron nitride (h-BN), which have shown promise for degrading emerging organic pollutants, such as per- and poly fluorinated alkyl substances (PFASs), through the generation of reactive oxygen species (ROS) under light irradiation. These nanomaterials are integrated in polyether ether ketone and polysulfone (PSF/SPEEK) matrix to impart advanced catalytic functionality targeting to increase the rejection and water flux to address the urgent issue water pollution. The SPEEK polymer in the composite membrane provides ion- exchange properties or water uptake ability, while the PSF imparts mechanical strength to enhance the overall stability of the membrane. The combination of these two materials with the functionalization on of 2D nanomaterials results in a multifunctional membrane capable of removing and degrading organic contaminants simultaneously. The synthesized membranes undergo comprehensive surface and structural characterizations, including evaluations of mechanical and chemical stability.

Applications of Lewis Acidic [ZnCl₂]₂[ChCl] Deep Eutectic Solvents (DES) in the Synthesis of Unsymmetrical Ethers via Dehydrativ



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Abstract

Unsymmetrical ethers are generally synthesized via the Williamson ether method, but the unwanted formation of symmetrical ethers plus the basic and harsh conditions of the route pose a synthetic challenge. Transition metal-enabled Ullmann-type synthesis, on the other hand, comes with a high economic cost. In contrast, the electrophilic addition of alcohol to alkene presents a unique route to synthesize unsymmetrical ether. However, the formation of symmetrical ether as well as the use of toxic mineral acids, limits the large-scale application of this technique. Dehydration of alcohols in the presence of base-metal catalyst has, however, recently offered the greenest approach to synthesize unsymmetrical ethers, leaving water as by-product. In this contribution, we describe our use of Lewis acidic zinc chloride in combination with choline chloride deep eutectic solvent (DES) as catalyst for the dehydration of benzylic alcohol with other alcohols, to form unsymmetrical ethers. Experimental data obtained from the catalytic cross-etherification of benzylic alcohols with various alcohols functional group, such as- nitro, allyl, methoxy, halide, thienyl and sterically hindered substrates gave a 70-95 % yield of unsymmetrical ethers subsequently. Also, the zinc-DES catalyst went through a 5-cycle re-run and gave about 80-90% yield of the unsymmetrical ether showing the high stability of the catalyst system. The use of this DES catalyst mixture provides a better approach from the green chemistry standpoint as environmentally benign, efficient, and recyclable catalyst in the synthesis of unsymmetrical ethers for broader industrial applications in surfactants, liquid fuels, polymers, and pharmaceuticals, to name a few.



Performance and Environmental Assessment of Biochar-based Membranes Synthesized from Various Petroleum-derived and Eco-friendly



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Abstract

Water contamination resulting from coal spills is one of the largest environmental problems affecting communities in the Appalachia Region of the United States. This coal slurry contains potentially toxic substances, such as hydrocarbons, heavy metals, and coal cleaning chemicals, and its leakage into water bodies (lakes, rivers, and aquifers) can lead to adverse health effects not only for freshwater bodies and plant life but also for humans. In this project, two studies were conducted to remove organic contaminants from the waters. One study involved the use of biochar to create a biochar-polysulfone (BC-PSf) flat sheet multifunctional membrane to remove organic contaminants, and the other compared eco-friendly (Gamma-valerolactone – GVL, and Rhodiasolv® PolarClean – PC) and petroleum-derived solvents (i.e., N-methylpyrrolidone – NMP) in the fabrication of the biochar-polysulfone membranes. The resulting membranes were tested for their efficiency in removing both positively and negatively charged organic contaminants from the collected water at varying pH values. A comparative life cycle assessment (LCA) with accompanying uncertainty and sensitivity analyses was carried out to understand the global environmental impacts of incorporating biochar, NMP, GVL, and PC in the synthesis of PSf/NMP, BC-PSf/NMP, PSf/GVL, BC-PSf/GVL, PSf/PC, and BC-PSf/PC membranes at a set surface area of 1000 m². The results showed that the addition of biochar to the membrane matrix increased the surface area of the membranes and improved both their adsorptive and mechanical properties. The membranes with biochar incorporated in their matrix showed a higher potential for contaminant removal than those without biochar. The environmental impacts normalized to the BC-PSf/GVL membrane showed that the addition of biochar increased global warming impacts, eutrophication, and respiratory impacts by over 100% in all the membrane configurations with biochar. The environmental impacts were highly sensitive to biochar addition (Spearman's coefficient >0.8). The BC/PSf membrane with Rhodiasolv® PolarClean had the lowest associated global environmental impacts among all the membranes with biochar. Ultimately, this study highlighted potential tradeoffs between functional performance and global environmental impacts regarding choices for membrane fabrication.

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Investigation of the use of PolarClean as a green solvent alternative for PVDF membrane fabrication



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Abstract

Membrane technology has proven to be an effective tool for separation processes due to its lower material and fabrication costs, high automation and throughput, and reduced footprint. The most widely used technique for preparing polymeric membranes is phase inversion which involves dissolving a polymer in a suitable solvent and applying external or internal forces to induce phase separation. Solvents traditionally used for the fabrication of polymeric membranes, such as N-methyl-2-pyrrolidone (NMP) and dimethylacetamide (DMAc), have been found to lead to negative impacts on the environment and human health. When these solvents are used in membrane preparation via non-solvent induced phase separation (NIPS), toxins have been known leach out into the wastewater generated during the process. In efforts to make the membrane fabrication process more sustainable, Methyl-5-(dimethylamino)-2-methyl-5-oxopentanoate (Rhodiasolv® PolarClean) is used in this work as a bio-derived, biodegradable, non-flammable and non-volatile solvent for the fabrication of polyvinylidene fluoride (PVDF) flat sheet membranes. Solubility calculations and cloud point curves confirmed PolarClean is an adequate solvent for PVDF. Membranes were fabricated using PolarClean and characterized with respect to dope solution viscosity, membrane morphology, surface wettability and composition, permeability, and protein rejection. Membranes were also prepared using NMP as a solvent for comparison. The membranes prepared using PolarClean showed similar performance in terms of water flux and protein rejection rates to those prepared using NMP as a solvent. The results of this experiment indicate sustainable solvents show promise to replace traditional harmful solvents in the production of polymeric membranes for water treatment.



Iodide Oxidation at the Air-water Interface of Microdroplets



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Abstract

Microdroplets are tiny liquid particles that are ubiquitous in the atmosphere, where they provide the highly reactive air-water interface. Accelerating chemical processes in microdroplets that are unaccounted on existing air quality and climate models have the potential to influence atmospheric composition and dynamics. In this work, we present an experimental study of the catalytic environmental potential of reactions occurring in microdroplets in a homemade aerosolization system and correlate the size of droplets with reaction kinetics. Droplet sizes were determined using a micro-imaging technique for impacted droplets that were subsequently analyzed with ImageJ software. The activation of iodide anion (I^-) in this aerosolized system results in the production of triiodide ions (I_3^-), a phenomenon not observed in controls with bulk water. The production of hydroxyl radicals at the interface is registered with a radical scavenger, confirming this key oxidant drives the oxidation without any added catalyst or external stimuli, further validating the unique reactions that occur in the atmosphere. Considering key parameters such as reaction distance, liquid flow rate, and gas conditions, we established a new understanding of the effective fluid dynamics for microdroplet processing. In conclusion, the work indicates that reactions in microdroplets occur 10 times faster than in bulk water that is continuously agitated to introduce air bubbles. The enhanced reactivity, with profound implications for environmental systems, is attributed to the presence of a strong electric field at the air-water interface, partial solvation of this medium, and spontaneous hydroxyl radical production.



The Use of Biochar and Stillage for Remediation of Abandoned Construction/Urban Sites Around University of Kentucky



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Abstract

This proposal aims to evaluate the efficiency of utilizing biochar with stillage, byproducts from bourbon production, for soil remediation and plant growth promotion on waste soil. The waste soil, obtained from the Grounds department at the University of Kentucky (UK) campus, lacks soil organic matter and has predominantly been used for filling holes during construction or as road fill. A comprehensive soil restoration plan prioritizing soil health and vegetative growth is essential to address these issues.

Biochar acts as a biological agent, providing a habitat for microorganisms, promoting microbial growth, and improving soil health. Simultaneously, stillage, rich in organic matter and nutrients, complements biochar by offering valuable resources for soil restoration. Disposing of stillage from the expanding Kentucky bourbon industry presents a challenge, but when combined with biochar, it becomes a valuable resource for soil restoration, enhancing soil fertility and promoting crop growth. This innovative approach repurposes waste and supports sustainable agriculture, while also reducing greenhouse gas emissions to mitigate climate change.

Ongoing controlled experiments at the greenhouse are assessing the impact of biochar and stillage treatment on soil quality and plant growth. Soil quality indicators, including nutrient levels, pH, water-holding capacity, and visual quantification of wheat plants, are being measured to evaluate the effectiveness of the treatment as a sustainable and cost-effective strategy for soil remediation on waste soil near the UK campus and the broader bluegrass community. The result of this study provides valuable insights into the feasibility and effectiveness of using biochar and stillage as soil amendments for the long-term restoration of waste soil. These findings will inform the development of sustainable soil management practices and construction site rehabilitation strategies. By repurposing waste materials and promoting sustainable approaches, this research offers a promising solution to address the challenges associated with degraded soil, contributing to a more sustainable future in soil management and land reclamation efforts.