

**STRUCTURALLY ENGINEERED AND AGROCHEMICALS-LOADED
BIODEGRADABLE POLYMER-BASED ELECTROSPUN ASSEMBLIES AS
SUSTAINABLE AGRO-AUGMENTING CONSTRUCTS**

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AS SUSTAINABLE AGRO-AUGMENTING CONSTRUCTS**

by

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Submitted

*in fulfilment of the requirements of the degree of **Doctor of Philosophy***

to the



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CERTIFICATE

This is to certify that the thesis entitled “Structurally engineered and agrochemicals-loaded biodegradable polymer-based electrospun assemblies as sustainable agro-augmenting constructs” submitted to the Indian Institute of Technology Delhi by Ms. Krishna Priyadarshini Das, for the award of the degree of Doctor of Philosophy in the Department of Materials Science and Engineering, is a bonafide record of original research work carried out by the candidate. The results contained in the thesis have not been submitted, in part or full, to any other university or institute for the award of any degree.

I further certify that Ms. Krishna Priyadarshini Das has fulfilled all the requirements for the submission of the thesis.



Bhabani. K. Satapathy

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Krishna Priyadarshini Das

ABSTRACT

Meeting agricultural requirements, without a significant impact on the soil-water ecosystem in terms of delivering agrochemicals for seed germination and growth of plants necessitates the development of a sustainable and multifunctional controlled-release agrochemicals carrier. Thus, the current thesis investigates the development and performance evaluation of structurally stable and mechanically robust biodegradable polymer-based electrospun micro/nanofibers and their structural assemblies for sustainable agricultural practices, that not only promote nutrient release kinetics but also improve soil health by increasing the water-holding capacity, and microbial activity while reducing the ecological footprint of conventional agricultural practices. The research consists of four interconnected studies, each focused on the fabrication and performance evaluation of agro-augmenting mats for potential applications in controlled/slow-release fertilizers, seed coating, and controlled herbicide carriers. In the first phase, our research focused on the fabrication of uniform ($\sim 0.208 \mu\text{m}$), bead-free and urea-loaded polyvinyl alcohol (PVA)/sodium alginate (SA)-based electrospun mats (EMs) with minimum diametral variation ($\pm 0.017 \mu\text{m}$) using Taguchi design-based optimal level of control parameters i.e., urea loading of ~ 45 phr (Parts per Hundred Parts of Resin), flow rate of ~ 1 ml/h, polymeric concentration of ~ 8 wt.%, and an applied voltage of ~ 25 kV. The contact angle and thermogravimetric analysis indicated an increase in hydrophobicity (from $\sim 30.64^\circ$ to $\sim 74.13^\circ$) and thermal stability (from 164°C to $\sim 290^\circ\text{C}$) for PVA/SA-based EM upon urea incorporation and post-crosslinking, respectively. Further, the effect of urea loading and crosslinking on swelling and degradation behavior indicated a sequential increase in water absorption capacity up to $\sim 296\%$ accompanied by a decrease in degradation rate up to $\sim 13\%$. Spectroscopic analysis revealed a non-Fickian diffusion-induced sustained release of urea in water (> 21 days) and soil (> 30 days) media. Soil burial studies of crosslinked urea-loaded EM exhibited excellent biodegradability ($> 80\%$ in 60 days) and structural stability (~ 30 days). Subsequently, the next phase of the research work focuses on the development of highly porous urea-biochar/ Polylactic Acid (PLA)-based agro-augmenting bead-free EM with improved physicochemical performance. The method involved the hydrothermal synthesis of walnut shell-derived biochar, followed by ball milling and urea loading and subsequent incorporation of urea-loaded ball-milled biochar into porous PLA-based electrospun fibers. The impacts of ball milling and urea loading were evaluated by using morphological (FESEM and TEM), microstructural (FTIR and XRD), and physiochemical (BET and BJH) attributes. To enhance the surface hydrophilicity, PLA-based porous EM was fabricated by altering the concentration

of co-solvent (DCM: DMSO) and relative humidity (20-80%). Bead-free and uniform urea/biochar-loaded PLA EM were fabricated by incorporating urea/biochar into PLA precursor solution and the resultant EM showed improved surface hydrophilicity (with a contact angle of 98.4°), water absorption (~ 69.4%), retention capacity (~17 days), and effective release of urea in water (~11.6 %) and soil (~5.67 %). The thermal stability (degradation temperature from 334 to 413°C) and mechanical properties (from ~9.6-13.56 MPa) are improved for PLA-based EM upon incorporating urea-biochar. The efficacy of developed EM for promoting plant growth was validated by conducting germination and growth assessments using green gram (*Vigna radiata*) plants. The results demonstrated higher germination rate, plant height, root length, dry weight, and fresh weight for plants treated with the EM as compared to the control sample. After establishing these results, the next chapter involves the fabrication and comparative performance analysis of urea-loaded core-shell, multilayered, and sandwiched electrospun fibrous constructs designed for controlled-release fertilizer systems. Core-shell fibrous constructs were fabricated using a co-axial electrospinning process, which enables a prolonged urea release that begins with an initial burst, followed by a sustained, diffusion-controlled release, which further accelerates as the shell gradually degrades. Multilayered constructs involved the sequential deposition of hydrophilic (urea-loaded PVA/SA) and hydrophobic (urea/biochar-loaded PLA) fibrous layers, which exhibited a staggered urea release profile by creating a series of barriers to facilitate a tailored release profile depending on the specific layer composition. In contrast, the sandwiched constructs comprising of both urea-loaded hydrophilic and hydrophobic polymeric fibers demonstrated intermediate properties with a moderate release rate.

The research work further demonstrates the development of sustainable seed coatings by incorporating eco-friendly additives and designing polymers with tailored physicochemical properties, ensuring the resilience and sustainability of agricultural systems. A biodegradable and biocompatible polymeric framework based on polyvinyl pyrrolidone/carboxymethyl cellulose (PVP/CMC) blend electrospun fibers loaded with Mg-Zn-Biochar was successfully fabricated and characterized. Comparative studies between uncoated seeds and seeds (*Brassica oleracea*, *Oryza sativa*, and *Vigna radiata*) coated with pristine and micronutrient-loaded PVP/CMC fibers were conducted to assess germination parameters and subsequent seedling growth through targeted micronutrient delivery. The results demonstrated enhanced seed germination, improved early growth, and better seedling growth (plumule as well as radical), thereby exhibiting the potential of such an innovative seed coating platform in promoting sustainable agriculture.

Thereafter, atrazine/hydroxypropyl beta-cyclodextrin (ATZ-H- β CD) inclusion complex (IC)-impregnated PVA-based nanofibrous mats were successfully evaluated as a potential herbicide-controlled release formulation by facilitating the controlled release of Atrazine (ATZ) while maintaining the herbicidal activity. The IC was synthesized via the coprecipitation method in a 1:1 stoichiometric ratio, and successful encapsulation of ATZ into H- β CD led to the formation of a stable complex, as revealed from the results of thermal, morphological, and microstructural characterizations. The phase solubility profile revealed that the formation of IC was able to improve the solubility and dissolution profile of ATZ in water. Further, the synthesized IC was loaded into the PVA-based EM, and the influence of IC loading on the microstructural, thermal, mechanical, as morphological attributes was analyzed followed by their simultaneous correlation with their respective release efficacy. Further, the swelling properties, porosity, herbicidal activity, and release kinetics in both water and soil media were analyzed to appraise the feasibility of the developed fibrous mat as a controlled release formulation to augment sustainable agriculture. The herbicidal activity assay revealed that the IC-loaded PVA EM can improve the herbicidal effect of ATZ on *L. sativa* weeds. Thus, the thesis systematically dealt with the optimization, fabrication, and performance evaluation of new-age multi-functional biodegradable, mechanically stable micro-/nano-structured controlled release carriers (CRC) that not only enhance crop yield but also ensure environmental sustainability as undesirable nutrient-induced secondary complications such as eutrophication and soil quality deterioration possibilities are largely mitigated.

सारांश

बीज के अंकुरण और पौधों के विकास के लिए एग्रोकैमिक्स प्रदान करने के मामले में मिट्टी-जल पारिस्थितिकी तंत्र पर महत्वपूर्ण प्रभाव डाले बिना कृषि आवश्यकताओं को पूरा करने के लिए एक स्थायी और बहुक्रियाशील नियंत्रित-रिलीज एग्रोकैमिक्स वाहक के विकास की आवश्यकता है। इस प्रकार, वर्तमान थीसिस संरचनात्मक रूप से स्थिर और यांत्रिक रूप से मजबूत बायोडिग्रेडेबल पॉलिमर-आधारित इलेक्ट्रोस्पून माइक्रो / नैनोफाइबर और टिकाऊ कृषि प्रथाओं के लिए उनकी संरचनात्मक विधानसभाओं के विकास और प्रदर्शन मूल्यांकन की जांच करती है, जो न केवल पोषक तत्वों की रिहाई कैनेटीक्स को बढ़ावा देती है बल्कि पारंपरिक के पारिस्थितिक पदचिह्न को कम करते हुए जल-धारण क्षमता और माइक्रोबियल गतिविधि को बढ़ाकर मिट्टी के स्वास्थ्य में सुधार करती है कृषि पद्धतियां। अनुसंधान में चार परस्पर जुड़े अध्ययन शामिल हैं, जिनमें से प्रत्येक नियंत्रित / धीमी गति से जारी उर्वरकों, बीज कोटिंग और नियंत्रित शाकनाशी वाहकों में संभावित अनुप्रयोगों के लिए कृषि-वृद्धि मैट के निर्माण और प्रदर्शन मूल्यांकन पर केंद्रित है। पहले चरण में, हमारे शोध ने वर्दी (~ 0.208 माइक्रोन), मनका मुक्त और यूरिया-लोडेड पॉलीविनाइल अल्कोहल (पीवीए) / सोडियम एल्गिनेट (एसए) आधारित इलेक्ट्रोस्पून मैट के निर्माण पर ध्यान केंद्रित किया, जिसमें न्यूनतम व्यास भिन्नता (± 0.017 माइक्रोन) के साथ टैगुची डिजाइन-आधारित इष्टतम स्तर नियंत्रण मापदंडों का उपयोग किया गया, यानी, ~ 45 phr की यूरिया लोडिंग, ~ 1 मिलीलीटर / घंटा की प्रवाह दर, ~ 8 wt.% की बहुलक एकाग्रता, और ~ 25 केवी का एक लागू वोल्टेज। संपर्क कोण और थर्मोग्रैविमेट्रिक विश्लेषण ने क्रमशः यूरिया निगमन और पोस्ट क्रॉसलिंग पर पीवीए/एसए-आधारित ईएम के लिए हाइड्रोफोबिसिटी (~ 30.64 डिग्री से ~ 74.13 डिग्री सेल्सियस) और थर्मल स्थिरता (164 डिग्री सेल्सियस से ~ 290 डिग्री सेल्सियस) में वृद्धि का संकेत दिया। इसके अलावा, सूजन और गिरावट व्यवहार पर यूरिया लोडिंग और क्रॉसलिंग के प्रभाव ने ~ 296% तक जल अवशोषण क्षमता में अनुक्रमिक वृद्धि का संकेत दिया, साथ ही ~ 13% तक गिरावट दर में कमी आई। स्पेक्ट्रोस्कोपिक विश्लेषण ने पानी (> 21 दिन) और मिट्टी (> 30 दिन) मीडिया में यूरिया की एक गैर-फिकियन प्रसार-प्रेरित निरंतर रिहाई का खुलासा किया। क्रॉसलिंगड यूरिया-लोडेड ईएम के मृदा दफन अध्ययनों ने उत्कृष्ट बायोडिग्रेडेबिलिटी (60 दिनों में 80% >) और संरचनात्मक स्थिरता (~ 30 दिन) का प्रदर्शन किया।

इसके बाद, अनुसंधान कार्य का अगला चरण बेहतर भौतिक प्रदर्शन के साथ अत्यधिक छिद्रपूर्ण यूरिया-बायोचार/पीएलए-आधारित कृषि-संवर्धक मनका-मुक्त इलेक्ट्रोस्पून मैट के विकास पर केंद्रित है। इस विधि में अखरोट के खोल-व्युत्पन्न बायोचार के हाइड्रोथर्मल संश्लेषण शामिल थे, इसके बाद बॉल मिलिंग

और यूरिया लोडिंग, और बाद में यूरिया-लोडेड बॉल-मिल्ड बायोचार को झरझरा पीएलए-आधारित इलेक्ट्रोस्पून फाइबर में शामिल किया गया था। बॉल मिलिंग और यूरिया लोडिंग के प्रभावों का मूल्यांकन रूपात्मक (FESEM, और TEM), माइक्रोस्ट्रक्चरल (FTIR, और XRD), और फिजियोकेमिकल (BET, और BJH) विशेषताओं का उपयोग करके किया गया था। सतह हाइड्रोफिलिसिटी को बढ़ाने के लिए, पीएलए-आधारित झरझरा ईएम को सह-विलायक (डीसीएम: डीएमएसओ) और सापेक्ष आर्द्रता (20-80%) की एकाग्रता को बदलकर गढ़ा गया था। पीएलए अग्रदूत समाधान में यूरिया/बायोचार को शामिल करके मनका मुक्त और समान यूरिया/बायोचार-लोडेड पीएलए ईएम का निर्माण किया गया था और परिणामी ईएम ने बेहतर सतह हाइड्रोफिलिसिटी (98.4° के संपर्क कोण के साथ), जल अवशोषण (~ 69.4%), प्रतिधारण क्षमता (~ 17 दिन), और पानी में यूरिया की प्रभावी रिहाई (~ 11.6%) और मिट्टी (~ 5.67%) दिखाई। यूरिया-बायोचार को शामिल करने पर पीएलए-आधारित ईएम के लिए थर्मल स्थिरता (334 से 413°C तक गिरावट तापमान) और यांत्रिक गुणों (~ 9.6-13.56 एमपीए से) में सुधार किया जाता है। पौधों की वृद्धि को बढ़ावा देने के लिए विकसित ईएम की प्रभावकारिता को हरे चने (*विय्या रेडिएटा*) पौधों का उपयोग करके अंकुरण और विकास आकलन करके मान्य किया गया था। परिणामों ने नियंत्रण नमूने की तुलना में ईएम के साथ इलाज किए गए पौधों के लिए उच्च अंकुरण दर, पौधे की ऊंचाई, जड़ की लंबाई, शुष्क वजन और ताजा वजन का प्रदर्शन किया। इन परिणामों को स्थापित करने के बाद, अगले अध्याय में यूरिया-लोडेड कोर-शेल, बहुस्तरीय, और नियंत्रित-रिलीज उर्वरक प्रणालियों के लिए डिज़ाइन किए गए सैंडविच इलेक्ट्रोस्पून रेशेदार निर्माणों के निर्माण और तुलनात्मक प्रदर्शन विश्लेषण शामिल हैं। कोर-शेल रेशेदार निर्माणों को सह-अक्षीय इलेक्ट्रोस्पिनिंग प्रक्रिया का उपयोग करके गढ़ा गया था, जो लंबे समय तक यूरिया रिलीज को सक्षम बनाता है जो प्रारंभिक फट से शुरू होता है, इसके बाद एक निरंतर, प्रसार-नियंत्रित रिलीज होता है, जो धीरे-धीरे शेल के रूप में तेज हो जाता है। बहुस्तरीय निर्माणों में हाइड्रोफिलिक (यूरिया-लोडेड पीवीए / एसए) और हाइड्रोफोबिक (यूरिया / बायोचार-लोडेड पीएलए) रेशेदार परतों का अनुक्रमिक जमाव शामिल था, जिसने विशिष्ट परत संरचना के आधार पर एक अनुरूप रिलीज प्रोफाइल की सुविधा के लिए बाधाओं की एक श्रृंखला बनाकर कंपित यूरिया रिलीज प्रोफाइल का प्रदर्शन किया। इसके विपरीत, यूरिया-लोडेड हाइड्रोफिलिक और हाइड्रोफोबिक पॉलिमरिक फाइबर दोनों से युक्त सैंडविच निर्माणों ने मध्यम रिलीज दर के साथ मध्यवर्ती गुणों का प्रदर्शन किया।

अनुसंधान कार्य आगे पर्यावरण के अनुकूल योजक को शामिल करके और अनुरूप भौतिक रासायनिक गुणों के साथ पॉलिमर डिजाइन करके टिकाऊ बीज कोटिंग्स के विकास को प्रदर्शित करता है, जिससे कृषि प्रणालियों की लचीलापन और स्थिरता सुनिश्चित होती है। Mg-Zn-Biochar से भरे पॉलीविनाइल पाइरोलिडोन/कार्बोक्सी मिथाइल सेलुलोज (PVP/CMC) मिश्रण इलेक्ट्रोस्पून फाइबर पर आधारित एक

बायोडिग्रेडेबल और बायोकंपैटिबल पॉलिमरिक फ्रेमवर्क सफलतापूर्वक गढ़ा और विशेषता बनाई गई थी। लक्षित सूक्ष्म पोषक तत्वों के वितरण के माध्यम से अंकुरण मापदंडों और बाद में अंकुर वृद्धि का आकलन करने के लिए प्राचीन और सूक्ष्म पोषक तत्वों से भरे पीवीपी/सीएमसी फाइबर के साथ लेपित अनकोटेड बीजों और बीजों (*ब्रैसिका ओलेरासिया*, *ओरिजा सैटिवा*, और *विया रेडिएटा*) के बीच तुलनात्मक अध्ययन किए गए थे। परिणामों ने उन्नत बीज अंकुरण, बेहतर प्रारंभिक विकास, और बेहतर अंकुर विकास (आलूबुखारा और साथ ही कट्टरपंथी) का प्रदर्शन किया, जिससे टिकाऊ कृषि को बढ़ावा देने में इस तरह के एक अभिनव बीज कोटिंग मंच की क्षमता का प्रदर्शन हुआ।

इसके बाद, एट्राज़ीन/हाइड्रॉक्सीप्रोपाइल बीटा-साइक्लोडेक्सट्रिन (एटीजेड-एच-सीडी) समावेशन परिसर (आईसी)-गर्भवती पीवीए-आधारित नैनोफाइबरस मैट का सफलतापूर्वक मूल्यांकन किया गया था, जो हर्बिसाइडल गतिविधि को बनाए रखते हुए एटीजेड की नियंत्रित रिलीज की सुविधा प्रदान करके संभावित हर्बिसाइड-नियंत्रित रिलीज फॉर्मूलेशन के रूप में मूल्यांकन किया गया था। आईसी को 1: 1 स्टोइकोमेट्रिक अनुपात में सह-वर्षा विधि के माध्यम से संश्लेषित किया गया था, और एच-सीडी में एटीजेड के सफल एनकैप्सुलेशन ने एक स्थिर परिसर के गठन का नेतृत्व किया, जैसा कि थर्मल, रूपात्मक और माइक्रोस्ट्रक्चरल लक्षण वर्णन के परिणामों से पता चला है। चरण घुलनशीलता प्रोफ़ाइल से पता चला कि आईसी का गठन पानी में एटीजेड की घुलनशीलता और विघटन प्रोफ़ाइल में सुधार करने में सक्षम था। इसके अलावा, संश्लेषित आईसी को पीवीए-आधारित इलेक्ट्रोस्पून मैट में लोड किया गया था, और माइक्रोस्ट्रक्चरल, थर्मल, मैकेनिकल के साथ-साथ रूपात्मक विशेषताओं पर आईसी लोडिंग के प्रभाव का विश्लेषण किया गया था, जिसके बाद उनके संबंधित रिलीज प्रभावकारिता के साथ एक साथ सहसंबंध था। इसके अलावा, टिकाऊ कृषि को बढ़ाने के लिए नियंत्रित रिलीज फॉर्मूलेशन के रूप में विकसित रेशेदार चटाई की व्यवहार्यता का मूल्यांकन करने के लिए पानी और मिट्टी मीडिया दोनों में सूजन गुणों, संरक्षता, शाकनाशी गतिविधि और रिलीज कैनेटीक्स का विश्लेषण किया गया था। हर्बिसाइडल गतिविधि परख से पता चला कि आईसी-लोडेड पीवीए इलेक्ट्रोस्पून मैट एल सैटिवा मातम पर एटीजेड के हर्बिसाइडल प्रभाव में सुधार कर सकते हैं। इस प्रकार, थीसिस व्यवस्थित रूप से नए युग के बहु-कार्यात्मक बायोडिग्रेडेबल, यांत्रिक रूप से स्थिर सूक्ष्म / नैनो-संरचित नियंत्रित रिलीज वाहकों के अनुकूलन, निर्माण और प्रदर्शन मूल्यांकन से निपटती है जो न केवल फसल की उपज को बढ़ाती है बल्कि पर्यावरणीय स्थिरता भी सुनिश्चित करती है क्योंकि अवांछनीय पोषक तत्व-प्रेरित माध्यमिक जटिलताओं जैसे यूट्रोफिकेशन और मिट्टी की गुणवत्ता ड्यूटेरिएशन संभावनाएं काफी हद तक कम हो जाती हैं।

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List of Abbreviations

- AIC: Akaike Information Criterion
- A_{663} : absorbances at ~ 663 nm
- A_{645} : absorbances at ~ 645 nm
- amoA: Ammonia Monooxygenase
- ANOVA: Analysis of Variance
- ATR-FTIR: Attenuated Total Reflection Fourier Transform Infrared
- ATZ: Atrazine
- BH β CD: Berberine-Hydroxypropyl- β -Cyclodextrin
- BET: Brunauer-Emmett-Teller
- BJH: Barrett-Joyner-Halenda
- C_a : chlorophyll a content
- C_b : chlorophyll b content
- CA: Cellulose Acetate
- CA: Citric Acid
- CEC: Cation Exchange Capacity
- CH₄: Methane
- CLSD: Laser Supersonic Drawing
- CMC: Carboxymethyl Cellulose
- CO₂: Carbon Dioxide
- CoNPs: Cobalt nanoparticles
- CRC: Controlled Release Carriers
- CRAs: Controlled Release Agrochemical Systems
- CRFs: Controlled release fertilizer systems
- Dt: The time in days corresponding to Gt.
- DCM: Dichloromethane
- DI: Deionized Water
- DI water: Deionized Water
- DMF: Dimethylformamide
- DMSO: Dimethyl Sulfoxide
- DOE: Design of Experiment
- DSC: Differential Scanning Calorimetry
- DW: Distilled Water
- EDX: Energy-Dispersive X-ray
- EM: Electrospun Mats
- E_E: Encapsulation efficiency
- FAO: Food and Agriculture Organization of the United Nations
- FAOSTAT: Food and Agriculture Organization Corporate Statistical Database
- FD: Fiber Diameter
- FESEM: Field Emission Scanning Electron Microscopy
- FTIR: Fourier-Transform Infrared Spectroscopy
- Gt: The number of germinated seeds on day t
- ha: Hectar
- HACTP: 2,2,4,4,6,6-Hexaaminocyclotriphosphatriene
- H- β CD: Hydroxypropyl- β -Cyclodextrin
- IC: Inclusion Complex
- IoT: Internet of Things
- L. sativa: Lactuca Sativa
- L_c : loading capacity
- m : Total weight of the tray containing the soil mixture (control sample)
- m_o : Weights of the plastic container before the addition of water
- m_i : Weights of the plastic container after the addition of water
- M_t: Amount of urea released at time
- M _{∞} : The mass of urea released as time approaches ∞ .
- MA: Maleic acid
- N₂O: Nitrous oxide
- NMR: Nuclear Magnetic Resonance

- NPK: Nitrogen, Phosphorus, Potassium
- OA: Orthogonal Array
- PAN: Polyacrylonitrile
- PAM: Polyacrylamide
- PCL: Polycaprolactone
- PEG: Polyethylene Glycol
- PEO: Polyethylene Oxide
- PGA: Polyglycolic Acid
- P_h: plant height
- PHB: Polyhydroxy Butyrate
- PVA: Polyvinyl Alcohol
- PVP: Polyvinylpyrrolidone
- R_L: root length
- S: the plant's dry weight.
- S/N: Signal-to-Noise
- SA: Sodium Alginate
- SD: Standard Deviation
- SSA: Specific Surface Area
- TGA: Thermogravimetric Analysis
- UN: United Nations
- USDA: United States Department of Agriculture
- VOC: Volatile Organic component
- phr: Parts per Hundred Parts of Resin
- PLA: Polylactic Acid
- PLGA: Poly(lactic-co-glycolic) Acid
- PLLA: Poly-L-Lactic Acid
- PM: Physical Mixture
- PP: Polypropylene
- PPZ: Polyphosphazene
- PV: Pore Volume
- WB: Walnut Shell-Derived Biochar
- WCA: Water Contact Angle
- W_d: dry weight
- W_w: Wet weight
- W_d: dry weight
- W_i: Initial weight
- W_f/F_w fresh weight
- WIOD: World Input-Output Database
- WS: Walnut Shell
- XRD: X-Ray Diffraction
- ZnO: Zinc oxide