Final Report (12/15/2023) Submitted by Surojit Gupta PhD (PI) Yun Ji PhD (co-PI) Dept. of Mechanical Engineering University of North Dakota, Grand Forks, ND 58201

Submitted to:

Miki Miheguli Research Programs Coordinator

> North Dakota Soybean Council Our World Is Crowing of

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1. Research Overview and Objectives: background information and research gaps

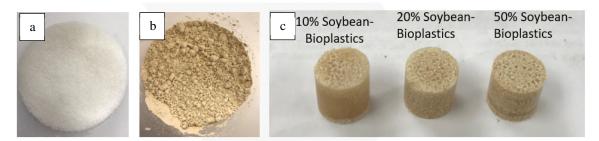
1.1 Background on Soybean Meal (SM)

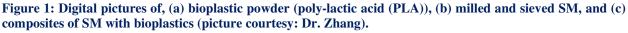
World's 85% of Soybean crop is processed into Soybean Meal (SM) and oil [1, 2]. The oil component is used for human consumption, bio-diesel component etc. Comparatively, SM component is predominantly used for animal feed. In 2020, US used 34 million metric tons and exported 11.88 million metric tons [3]. Currently, SB has a low valuation of 0.42 USD/Kg [4]. Solvent extraction is the common method for extraction of oil from soybean with 1.5% oil being left in SM during the process [5].

1.2 Alternative Usage of SM – A Literature Survey

SM has some inherent advantages like low cost, biodegradability, and processability [6] which makes it a unique materials system for advanced materials research which is underutilized. Proteinrich SB has some disadvantages like poor mechanical behavior and poor water resistance due to presence of hydrophilic groups which has hindered design of materials from this precursor [6-8]. Recently, some studies have focused on designing and manufacturing SM-based adhesives, but the process used during manufacturing is tedious and involves intermediate steps like chelation [6], cross-linking [7], and hybridization [8]. These intermediate steps further increase the difficulty in commercialization of these materials. In addition, the authors have not found any background study where the material system has beneficially used 1.5% residual oil in the SM-matrix for further applications. Furthermore, the beneficial and synergistic usage of environmentally friendly SM for designing high performance functional materials with high profit margin is underutilized. **1.3 Preliminary Data**

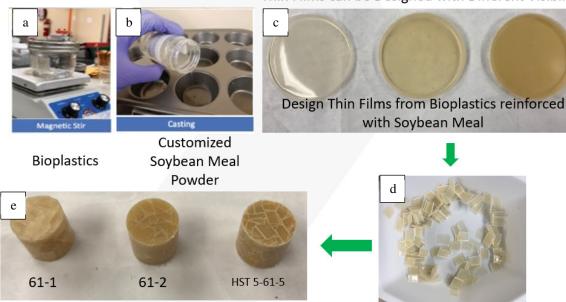
Dr. Gupta's team is in the process of developing collaboration with Mr. Mike Keller from ADM which was facilitated by Dr. Scott Korom. During this process, ADM supplied SM to Dr. Gupta's team at UND. We were successfully able to process it into particles of different size-fractions (Fig. 1b) (Fig. 1a shows example of bioplastics feedstock powder). In addition, we used hot pressing to design and manufacture bioplastics (Poly-lactic Acid (PLA))-SM composites (Fig. 1c) where PLA is a bioplastic processed from Corn (we have designed technology to design different types of bioplastics which will further give us competitive edge for commercialization). These composites have unique surface appearance which can be further leveraged for different





types of applications like packaging and infrastructure. Figure 2 shows the technology to design solvent-cast thin films by using SM-bioplastics with different visual appearance. It is also possible to machine these films and consolidate them into pressed-samples (Fig. 2e). As a background, bioplastics are renewable and biodegradable polymers, for example PLA is derived from renewable sources [9]. Previously, Soybean oil has been integrated with PLA to design biodegradable materials [9]. Thus, the integration of SM with bioplastics will create *unique renewable material systems for large-scale valorization and commercialization*.

Design of novel SM-based renewable products will be a win-win situation for all the stakeholders from commercialization perspective as traditional polymers are not sustainable and as high as 40% of the produced plastics is thrown away [10].



Thin Films can be Designed with Different Visibility

Figure 2: Digital pictures of, (a) stirring process), (b) liquid ink (images courtesy: Saud et al. JMEP), and (c) solvent-cast films, (d) machined coupons, and (e) hot-pressed samples (pictures courtesy: Dr. Zhang). **1.4 Integrated Research Program**

This proposal addressed the "New Use Research Priorities" of, (a) Soybean Meal (SM) Use and (b) Potential Commercialization. As integral component of this research, we are proposing a hypothesis driven design paradigm which beneficially use SM for manufacturing high performance materials with high valuation in high growth market areas. It is also proposed that the design will effectively use residual oil in SM-matrix for lubrication applications which will open potential avenue of synergistic usage of SM and Soybean oil for rapid commercialization.

During the research, we had proposed following objectives:

Objective 1: To create novel pathways of creating SM-based feedstock;

Objective 2: To create *transformative methods* for manufacturing novel sustainable composites by using SM;

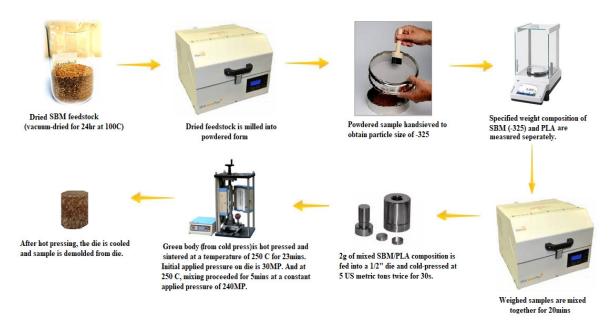
Objective 3: To understand the properties of the composites by experimental studies.

Objective 4: To *formulate strategy for commercializing* the materials developed during work in phase-II.

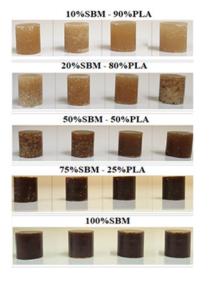
2. Materials and Methods

2.1.1 Scaffold Technology

In this project, a graduate student (Temofeh) and Dr. Zhang are working with the PIs. Figure 1 shows the manufacturing paradigm for manufacturing scaffolds. During this study, we designed scaffolds by integrating different fractions of Poly Lactic Acid (PLA) with Soybean Meal (SM).







- 10%SBM 90%PLA 0.2g SBM mixed with 1.8g PLA
- 20%SBM 80%PLA 0.4g SBM mixed with 1.6g PLA
- 50%SBM 50%PLA 0.5g SBM mixed with 0.5g PLA
- 75%SBM 25%PLA 1.5g SBM mixed with 0.5g PLA
- > 100% 2g SBM. No PLA added.

Figure 4: Different samples synthesized during this study

2.1.2 Solvent Cast Technology

Figure 2 shows the manufacturing process of solvent casting technology. The team was successfully able to fabricate PLA-SM composite films. Figure 10 shows the tensile strength behavior of solvent cast films. All the composites showed plastic behavior. Figure 11 summarizes the ultimate tensile strength behavior of SM-PLA composites. This work shows that we can add 40% SBM as additives in PLA matrix. Currently, we are characterizing these films for further evaluation as bioplastics for different applications like packaging etc.

3. Research Results/Outcomes and Discussion

3.1 Scaffold Samples

Figure 5 shows the compressive stress vs displacement plot of different SBM-PLA composites. Table 1 displays the summary of the mechanical properties of the blends. The Ultimate Compressive Strength (UCS) refers to the maximum stress that each material can withstand under compression before failure. By using SBM, it is possible to generate composites with greater than 55 MPa compressive strength which is the strength of high strength concrete.

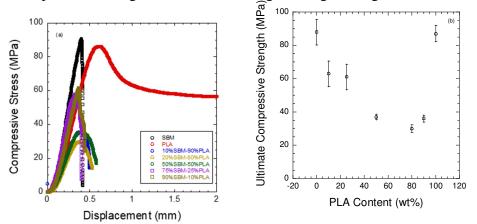


Figure 5: Plot of, (a) compressive stress vs displacement in SBM-PLA composites, and (b) the compressive stress vs displacement plot of PLA with varying compositions of SBM additive.

Composition	Compressive strength (MPa)
Pure PLA	86.57 ± 4.88
Pure SBM	87.90 ± 7.60
10 % SBM – 90 % PLA	36.03 ± 2.10
20 % SBM – 80 % PLA	29.50 ± 2.30
50 % SBM - 50 % PLA	37.02 ± 1.65
75 % SBM – 25 % PLA	60.57 ± 7.63
90 % SBM – 10 % PLA	62.77 ± 7.61

Table 1. Summary of compressive test results of SBM-PLA blends (Hot press)

Figure 6a illustrates the DSC findings for the SBM-PLA compositions. In the course of the DSC heating cycle, PLA exhibited a semi-crystalline behavior, with a glass transition temperature (T_g) and melting point (T_m) measuring approximately 69 and 167 °C, respectively. The amorphous

curve for the pure SBM displayed a single endothermic peak. The pure SBM sample exhibits a distinct glass transition temperature. The fully amorphous pure SBM demonstrates a glass transition temperature of approximately 119 °C. The addition of SBM to PLA did not result in significant changes in the glass transition temperature (T_g) and melting point (T_m) of the composites compared to pure PLA.

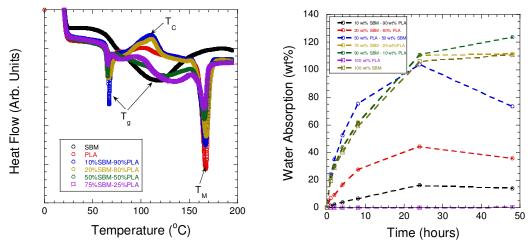


Figure 6: (a) DSC and (b) water absorption plot of hot-pressed SBM composites.

Here, in this study, we demonstrated that blending soybean meal with PLA can result in the formation of stable, less-porous structures. In Figure 6b, we can observe the graphical representation of water absorption capacity over time. The composites showed high water absorption which showed promise as water absorption sinks in plantation, filtration and other applications.

Figure 7 shows the SE-SEM (Secondary electrons) images of the SBM-PLA blends fabricated at 250 °C. Very little plastic deformation and a shiny fracture surface characterize brittle fractures as seen in the Figure. The examination of fracture surfaces resulting from these brittle failures revealed that these failures typically commence at a notch or stress concentration and propagate with minimal plastic deformation. In brittle fractures, the fracture process involves minimal energy absorption, as energy is absorbed through areas characterized by small plastic deformation. The separation of individual grains occurs through cleavage along specific crystallographic planes. The micro-surfaces also exhibit the presence of cracks and roughness. These cracks may arise from factors such as thermal stresses, inadequate dispersion of soybean meal within the PLA matrix, or insufficient interfacial adhesion between the components.

3.2 SBM-PLA Bioplastics Characterization by Solvent Casting

Figure 8 displays digital images of various solvent-cast samples. All the samples exhibit a uniform surface, indicating a well-dispersed arrangement of SBM fibers within the PLA polymer. These bioplastics can be used for packaging, structural and other applications.

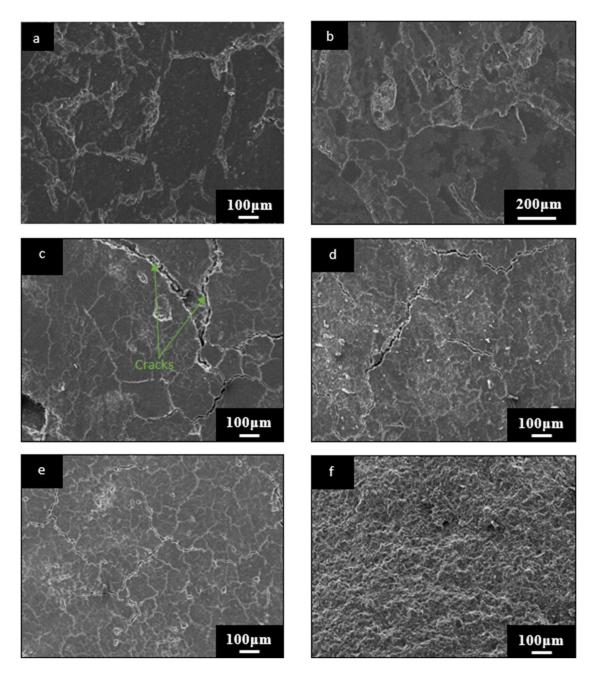


Figure 7: SE-SEM micrographs of hot press, (a) 10 % SBM – 90 % PLA, (b) 20 % SBM – 80 % PLA, (c) 50 % SBM – 50 % PLA, (d) 75 % SBM – 25 % PLA, (e) 100 % SBM, and (f) 100 % SBM (fractured).

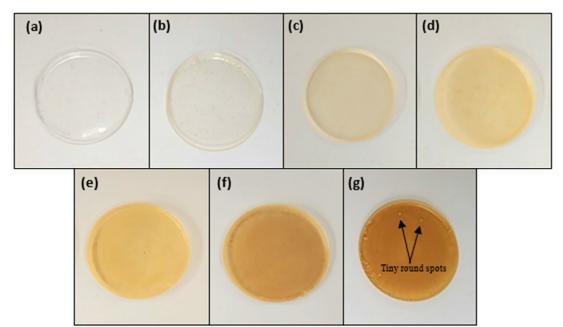


Figure 8: Digital pictures of solvent cast samples, (a) pure PLA (b) 2% SBM – 98 % PLA, (c) 5% SBM – 95 % PLA, (d) 10% SBM – 90 % PLA, (e) 20% SBM – 80% PLA, (f) 30% SBM – 70 % PLA, and (g) 40% SBM – 60 % PLA.

Figure 9 presents the SE-SEM (Secondary Electron Scanning Electron Microscopy) micrograph showcasing the fractured surface of the solvent-casted SBM-PLA samples following the tensile testing. Figure 10 presents the results obtained from Differential Scanning Calorimetry (DSC) for SBM-PLA compositions.

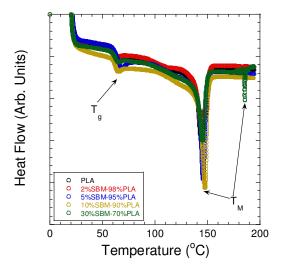


Figure 10: DSC plot of SBM-PLA composite (solvent casting)

In the heating cycle (refer to Fig. 10), PLA exhibited semi-crystalline behavior with a glass transition temperature (T_g) of 63 °C and a melting point (T_m) of 144 °C. With the addition of 2% SBM to the sample composition, the SBM-PLA composite displayed a T_g of 64 °C and a T_m of 143 °C, suggesting no significant difference when compared with the corresponding values of pure PLA. The similarity in glass transition temperature (T_g) and melting point (T_m) of the blends to that of PLA suggests that soybean meal exhibits a certain degree of compatibility with PLA. This similarity also implies that the blend is likely homogeneous, and the soybean meal is well-dispersed within the PLA matrix. Figure 10a illustrates the typical tensile stress versus displacement plot for PLA and various blends of SBM-PLA. PLA exhibited higher tensile strength in comparison to SBM-PLA blends. Nevertheless, the tensile strength remains consistent as soybean meal (SBM) is incorporated until approximately 5%. Beyond this threshold, a continual decline in strength is observed with increased addition of SBM. The variations in strength may be attributed to the influence of soybean meal on the composite's mechanical properties.

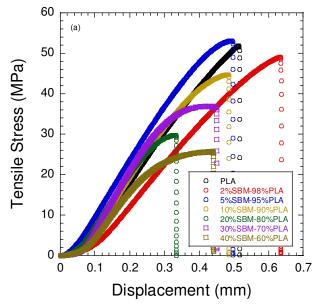


Figure 10: Plot of tensile stress vs displacement in solvent cast composites.

4. Listings of any disclosures of inventions or plant varieties

We have published the results in Mr. Temofeh Nathaniel Esenamunjor MS Thesis. Currently, we are collaborating with Dr. Carrie Miranda group's to design scaffolds from tailored Soybean Meal.

5. Conclusion/Benefits to the North Dakota Soybean Farmers and the Industry

Phase-I of this project is very successful. We have established that by using Soybean Meal, we can manufacture materials with high strength. These materials can be used in different highperformance applications. Based on the result presented, we think that the cementitious nature of composites are because of the protein-based nature of the Soybean Meal. We are planning to perform further research with Dr. Miranda's group to understand whether these properties can be further tailored by using crops with different concentrations of protein concentration. We are planning to submit a follow-up proposal. Based on these results, we can strongly conclude that Soybean Meal is a effective source for making alternative materials. It will directly benefit the industry and Soybean farmers by finding alternative avenues for selling their product.

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