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Single particle mechanical characterization of ground switchgrass in air dry and wet states using a microextensometer

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ABSTRACT

To understand the mechanical process of pelletization, it is critical to study the particle level interactions that cause particles to bind together and form a pellet. A micromechanical extensometer device, inspired by the MEMS technology, was developed and used to perform tensile experiments to deduce the stress-strain response of single particles of ground biomass. The effect of moisture, which has a significant role in forming pellets, was examined based on the micromechanical characterization of moisture conditioned and unconditioned (control; air dried) switchgrass particles. Conditioned particles exhibited three phases sigmoidal shaped stress-strain response. The three phases include the first linear elastic zone, where the particle behaved linearly up to approximately 1.6% strain, the second linear elastic zone (strain ranging from 1.6 to 2.1%) with significantly increased elastic modulus (168.9–223.7%), and the third zone (strain beyond 2.1%), where elastic modulus declined sharply (down to 90.9% of the second zone). The modulus of elasticity up to 1.5% strain for unconditioned and conditioned switchgrass particles were 1.60 ± 0.33 GPa and 6.99 ± 1.66 GPa, respectively ($p = 0.00$). The nominal fracture strength of unconditioned (6.2%, w.b.) and conditioned (17.5%, w.b.) switchgrass particles were determined as 35.77 ± 14.99 MPa and 130.42 ± 87.56 MPa, respectively ($p = 0.08$). The nominal fracture strain of unconditioned and conditioned switchgrass particles were determined as 2.43 ± 0.70% and 1.51 ± 0.66%, respectively ($p = 0.06$). Increase in the stiffness of switchgrass particles is contributed to the bundling of fibers promoted by the activation of binders due to increased moisture content.

1. Introduction

Densification of an ensemble of particulate matter is achieved by forcing the particles together by applying mechanical force to create inter-particle binding, producing compacted particle systems of well-defined shapes and sizes such as briquettes, pellets, and cubes [1]. Pelletization is a process of compressing or densifying biomass material into pellets, which increases the bulk density of biomass about ten-fold [2]. The applied force during pelletization causes rearrangement, breakage, and binding of particles, which result in a stable and near contiguous assembly of the compacted biomass. Much work has been performed to identify the optimum densification parameters for various kinds of biomass feedstocks; aiming towards the improvement of the pellet qualities [3–7]. Most studies focused on the effect of factors affecting the densification process and pellet qualities including physical and mechanical properties of the biomass, preconditioning, pretreatment, and pelletization parameters [3,4,6,7]. However, these studies are empirical in a sense that they do not provide governing laws explaining the bulk properties based on the fundamental mechanical theories. Therefore, to implement a scientific and systematic approach in the pelletization process, it is critical to study particle level interactions that hold the particulate assembly together in a stable form. To that end, the overarching goal of the research team is to investigate particle-particle interaction in its natural state accompanied with the characterization of individual particles with the focus on the mechanical responses. The fundamental knowledge of the quantitative contribution of such inter-particle interactions at the microscale is essential to understanding how the mechanical behavior of particles of irregular shapes and sizes during the densification process affects the pellet quality.

1.1. Qualitative studies on particle binding

In recent years, limited studies have been performed to observe and qualitatively understand the role of binders during densification and binding and failure mechanisms in the pellet [8,9]. These studies attempted to examine proposed binding mechanisms through microstructural imaging analyses. Kaliyan and Morey [8] investigated the particle–particle binding and role of natural binders by microstructural analyses. They used Scanning Electron Microscopy (SEM) images to show that the binding between particles was created mainly through solid bridges, which were formed by natural binders in the biomass during the densification process. UV auto-fluorescence images of cross-sections of briquettes and pellets were also studied confirming that the

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solid bridges were made mainly by natural binders such as lignin and protein. This study suggested that activating the natural binders using moisture and temperature in the range of glass transition are important to make durable particle–particle bindings. In a study conducted by Stelte et al. [9], SEM images were used to study the binding mechanism of the prepared biomass pellets by fracture surface analysis of failed pellets. The study observed that the adjacent particles formed bridges due to lignin softening and inter-diffusion resulted in high energy absorbing mechanism during failure [9]. Both studies [8,9] investigated the presence of natural binders in biomass, which affect the binding mechanism and play an important role in the formation of particulate assemblies. However, the mechanical properties of these particle-particle bonds have not been characterized at the microscale level. The quantitative characterization of the strength of solid bridges formed during the densification will provide better understanding of the densification process and how pellets attain desirable qualities.

1.2. Micromechanical characterization

Most techniques, which are available for the mechanical characterization at the macroscale, cannot be used or translated directly to the microscale because of their unsuitability in manipulating samples with sub-millimeter size [10], which is also the subject of this study. Extensive researches have been performed to conduct mechanical testing with microscale samples providing enough accuracy, reliability, and simplicity. However, there are no standard methods or universally recognized fixtures for mechanical testing of materials at microscales [8]. Microelectromechanical systems (MEMS) is one such technique, which has been utilized to determine the mechanical properties at the microscale. MEMS devices have been widely used to characterize classical engineering materials as well as plant and biological samples at tissue or even at the cell wall fragment level [11–17]. To carry out an accurate mechanical characterization at the microscale, one of the requirements is a high resolution imaging system for displacement and force measurements [10], which is readily available with the recent development in the microscopy. Use of micromechanical extensometer device, inspired by MEMS device, in this field of biomass densification is a unique initiative. It is shown that the MEMS-based mechanical test can be successfully employed in measuring the mechanical behavior of microscale assemblies of particulate materials to determine their mechanical properties in tension and compression [14]. This method can provide invaluable information about binding properties of particles and bonds formed during the densification of biomass.

1.3. Proposed quantitative study to understand mechanism

Bridging the link between the microscale particle bonds' properties and pellet qualities will contribute to science and engineering immensely. In order to gain this knowledge, there is a need to develop a methodology for the mechanical characterization of a single particle and the particle-particle interaction. To date, no experiment has been reported to study the mechanical behavior of sub-millimeter scale single particles of ground biomass under direct tensile/compressive loading. Since particles are the fundamental building blocks leading to the formation of the final assembly, it is of utmost importance to understand the mechanism of the densification of ground biomass from the microscale up to the macroscale. The stepping stone to such study would be an investigation on the feasibility of adapting MEMS inspired device for ground biomass particle. Therefore, the specific aim of this study is to characterize the mechanical behavior of single particles of ground switchgrass by using micromechanical devices. This article reports a novel experimental protocol overcoming the challenges in characterizing single biomass ground particles of irregular shapes. Since moisture content of biomass particles plays an important role in pelleting, we tested the hypothesis that the mechanical behavior of individual particles measured under a direct tensile loading is significantly affected by the moisture content. Ultimately, sub-millimeter size single particle mechanical properties will be used in the mechanical characterization of interactions of bonded particles.

2. Material and methods

Because of its importance as a renewable and sustainable bioenergy material, switchgrass was chosen as the test material [6,18]. Switchgrass, a perennial warm-season grass, can be grown on marginal land or rotated with other crops and has an advantage of lower ash and greater energy content over other crops [19]. Pretreatment of biomass improves both its physical and chemical properties and makes the material easier to densify, which has been documented to reduce the cost of production [5]. Size reduction of switchgrass was performed and particle passing through one screen size of 3.175 mm was collected [6]. The moisture content of size reduced switchgrass was determined by using the oven drying method [20]. Unconditioned (control) and conditioned switchgrass particles at moisture contents $6.2 \pm 0.3\%$ (w.b.) and 17.5% (w.b.), respectively, were used for experiments. A 17.5% (w.b.) moisture content level was chosen for conditioning of ground switchgrass since it was reported to be an optimum condition for forming pellets [6]. Samples of 25–50 g of switchgrass were conditioned in one batch by adding required water and mixing with a manual Mini-Inversina (Bioengineering AG, Switzerland) that is capable of giving 360° motion. The conditioned material was kept covered for 24 h for moisture equilibration [6].

The characterization of ground switchgrass single particles was carried out using a microextensometer device (Fig. 1), whose working principle was similar to MEMS based tensile-test device designed by Zamil et al. [13,17]. Microextensometer device consists of a piezoelectric motor-driven actuator (AG LS25, Newport, Bozeman, MT, USA) and a 3D-printed force sensor beam (FSB) (Proto3000, Woodbridge, Ontario, Canada). The obtained force-extension dataset was used in calculating the stress-strain relation, which was more useful and intrinsic information; i.e., as stress-strain analysis is independent of physical dimensions of samples. Tensile tests and subsequent data analyses were performed in five sequential steps as briefly summarized below.

1. *Calibration of the force sensor beam*: A FSB was the essential part of a microextensometer device in characterization of the biomass particle specimen, which acts as a transducer in the test set-up to measure the force applied during the test. The spring constant of the force sensor beam depends on the dimension of the beam and its material properties [13], therefore, the FSBs for biomass testing were designed considering these factors. 3D printed FSBs were manufactured by a polyjet process using VeroWhitePlus (Proto3000, Woodbridge, Ontario). The FSB spring constant, i.e. stiffness of FSB, decreased after every experiment due to stretching in the material, therefore, it was of utmost importance to measure stiffness after every single run, i.e., just prior to the subsequent run, for accurate data analysis. A calibrating device was an ensemble of a piezoelectric actuator and a precision weighing scale [15]. Theoretically, the displacement resolution of this piezoelectric device was 0.05 μm, however, the actual resolution of this actuator was determined as 0.45 μm displacement for each step. The displacement for each step was measured under the optical microscope. The actuator was connected to a piezo motor controller (AG-UC2, Newport, Bozeman, MT, USA), a push button controller, which provided power supply to the actuator. To calibrate the FSB, which was attached to the actuator, was given one step at a time and the respective load value was measured by a precision weighing scale. Obtained data was analyzed to determine the spring constant of the 3D printed FSB. The stiffness values of FSBs were in the range of 700–1000 N m^{-1}.
2. *Particle selection*: The criteria of particle selection were length and nominal width, where the particle should be long enough to be gripped on the testing device and the nominal width should be within the FSB testing limits and physical size of device. Based on