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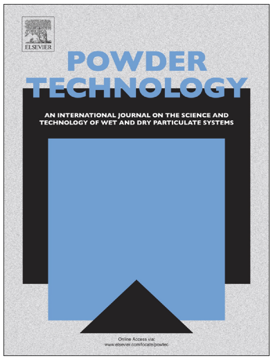

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Impacts of Caking on Corn Stover – An Assessment of Moisture Content and Consolidating Pressure

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Abstract

Caking or time consolidation of powders is a serious problem which can hinder productivity and overall feasibility of various industrial processes. This study focuses on the impacts moisture content and consolidating pressures can have on 2 mm corn stover samples after undergoing a drying treatment at different times. Individually, moisture content and consolidating pressure did not exhibit any significant changes throughout the modified variable flow rate tests using the FT4 powder rheometer. However, applying both variables simultaneously yielded higher-than-usual energy outputs from the corn stover, indicative of biomass agglomeration due to the moisture and its subsequent evaporation while under pressure via the induced consolidating pressure. However, despite the increase in energy usage, time dried did not have a direct effect and demonstrated no trend.

Keywords

Caking, Corn Stover, Moisture Content, Consolidating Pressure

Nomenclature

CP	Consolidating Pressure
CS	Corn Stover
DSC	Differential Scanning Calorimetry
DVS	Dynamic Vapor Sorption
FT4	Freeman Rheometer Model
GAB	Guggenheim-Anderson-deBoer
MC	Moisture Content
T2	Spin-spin relaxation time
VFR	Variable Flow Rate
xCS_z^y	Sample label: x is the moisture content in %, y is the consolidation pressure in kPa, and z is the time the vessel was dried in days

1. Introduction

Increasing population and economic growth forecast a rise in global energy demand by about 50% by 2050. [1] Meeting these energy needs and doing so in carbon neutral/negative way is the basis for many of the Department of Energy's Grand Challenges. [2-4] Many great strides have been made for diversifying our energy portfolio with reduced carbon have been made, including a 13% growth in solar and 18% in wind energy production since 2010. [5] For certain sectors, however, the Department of Energy's Bioenergy Technology Office has identified biofuels as the best way to replace their fossil fuel counterparts. [6] This is especially true for complex fuels, like those formulations used in aviation which may be produced by pivoting off the diversity of biomass. [7] Although an onerous task, a fundamental change towards the production of sustainable bioenergy is vital, primarily due to the finiteness of non-renewable resources among other socioeconomic, environmental, and political factors. These factors place a priority on addressing the ongoing strive for sustainable development which are urgently required.

Biomass as an energy source is a promising feedstock to replace fossil fuels owing to its compositional versatility and their ubiquity. Furthermore, biomass may be converted into a plethora of platform chemicals that can be used as additives for diesel fuels or other building blocks; this wide array of bio-based products and bioenergy production are carried out by biorefineries. Biorefineries, however, can suffer from operational downtime, particularly due to the feeding and handling of the biomass; [8] the primary contributor is feedstock variability, requiring comminution, pretreatment steps, customized hopper designs, and computational modeling. [9,10] Additionally, biomass can pose negative effects on prolonged operational windows due to their supply seasonality. [11] Therefore, biomass storage is fundamental to prevent any process downtime throughout different periods. Whilst various methods of storage exist and have their own advantages and disadvantages, several factors such as degradation, self-heating and subsequent fire/explosion by controlling dust or oxygen concentrations, and controlling moisture have been identified. [12]

Similar to other bulk powders, biomass can present a phenomenon called caking. Caking or time consolidation is a common problem in various industries, for example, food, pharmaceuticals, detergents, and chemicals; it consists in the agglomeration of solid particles, having deleterious effects to its flowability and causing it to create large lumps and overall impacting profitability. [13-15] The caking of powders can be influenced by different mechanisms, such as mechanical, thermal, environmental, and chemical. [16] One of the more common mechanisms involves moisture, creating liquid bridges and increasing the capillary forces and surface tension of the powders. [14,17] Moisture content can also impact the conversion performance as well as pretreatment processes such as dry grinding which will reflect on its final particle size distribution. [18-22] This phenomenon has been observed in biomass using different levels of moisture content, increasing the compressibility and cohesion—vital properties for the flowability of the material. [23-25] Some disadvantages include the loss of active storage space (material adhering to the walls of the storage container), erratic flow, and material bridging over the outlet of the storage container, preventing it from flowing out. [26]

There are numerous caking test methods, from the reasonably simple, empirical approaches such as throwing a bag from waist height and calculating the amount of free flowing powder[27,28] to correlating the caking using spin-spin relaxation time (T_2) as a function of temperature using NMR spectroscopy. [29,30] Other methods consist of Differential Scanning Calorimetry (DSC) to observe the glass transition and evaluate the cohesiveness of the sample, [31,32] uniaxial consolidation tests to measure the unconfined yield strength of the caked powder,[4] and powder rheometers such as Schulze ring shear tester or FT4 powder rheometer. [16,17,33,34] Other studies involving biomass which are tied to caking and cohesion include in the assessment their surface energetic properties to determine their hydrophobicity after their degradation caused by self-heating. [35]

Herein, we evaluate the impacts of caking in bulk (unseparated) 2 mm corn stover using the FT4 powder rheometer. Tests were conducted using different moisture content and were dried with different consolidation weights; this allowed us to assess the significance of moisture and the applied pressure has upon drying.

2. Materials and Methods

2.1. Sample Origins and Moisture

Bulk corn stover, comprising all anatomical fractions (leaf, husk, cob, and stalk) with an average particle size of 2 mm was used for the caking tests. Corn stover was procured from Idaho National Laboratory (INL). Initial drying and moisture introduction of the corn stover samples are reported elsewhere. [23] Moisture content was calculated using the following equation (Eq. 1):

$$MC = \frac{m_{H_2O}}{m_{H_2O} + m_{CS}} \ x \ 100 \tag{1}$$

where MC is the weight percentage of moisture content in the sample (within ~1-2 wt. %), m_{H_2O} is the mass of water, and m_{CS} is the mass of the bulk corn stover. In addition to a dry sample, two moisture levels (25 wt.% and 50 wt.%) were analyzed. The FT4 vessel (50 mm diameter) was loaded first with dry corn stover followed by the test sample at the top A copper grid separator (Fig. 2) is then placed on top to mitigate perturbation from the fins of the in-house machined stainless-steel weight which applies a consolidating pressure (CP) upon the biomass, as shown in Fig. 1.

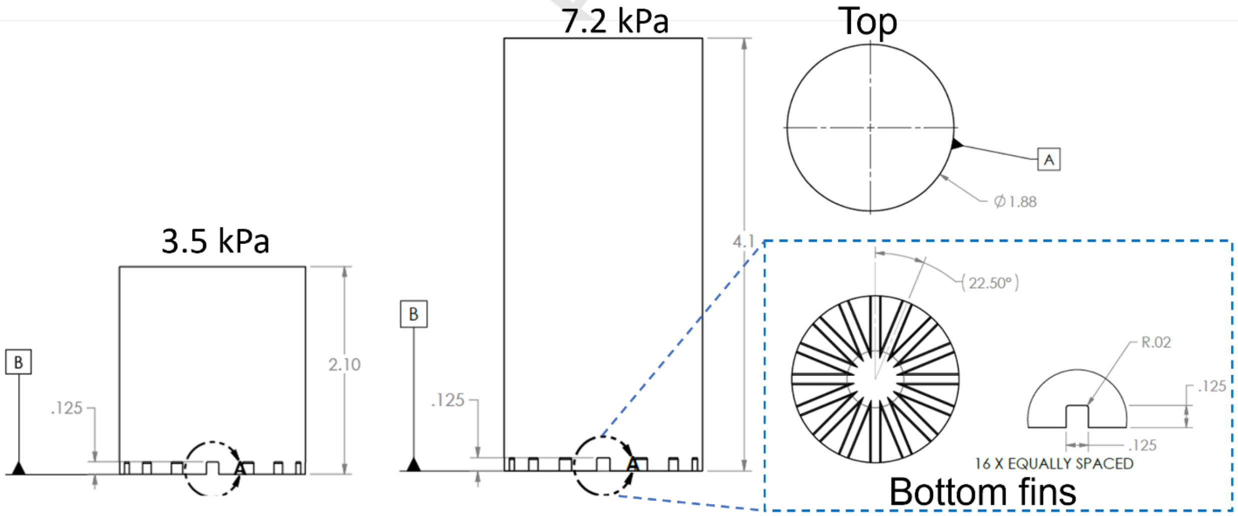

Fig. 1. Schematic of the 3.5 kPa and 7.2 kPa consolidating weights. The channels (denoted by the dashed circles) were incorporated to allow for moisture removal during drying.

Samples with and without the consolidating weights were subsequently placed in a convection oven at 45 °C for a determined number of days to remove the moisture content from the wet biomass, facilitated by the fins of the consolidated weight. Corn stover (CS) sample were labeled as xCS_z^y, where: *x* is the moisture content in %, y is the consolidation pressure in kPa, and z is the time the vessel was dried in days.

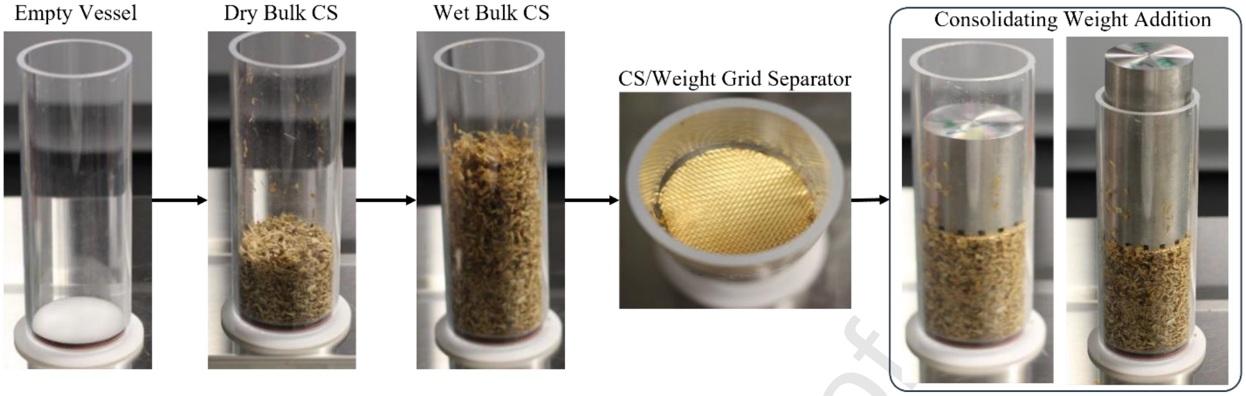

Fig. 2. Caking vessel preparation.

2.2. *Instrumentation – Freeman FT4 Rheometer*

To evaluate the effect of MC and/or CP on possible caking of the corn stover, tests were conducted in an FT4 powder rheometer instrument using the variable flow rate (VFR) method with modifications due to the instrument's susceptibility to overtorque while using the conventional conditions (i.e., impeller traversing through the sample clockwise). A typical VFR method involves the use of an impeller blade is rotated and is moved downwards the sample and yields energy values corresponding to the force required to traverse the sample. Energy is calculated using the following equation (Eq. 2):

$$E = R * D = (F * \tau) * D \tag{2}$$

where E is the energy, R is the resistance exerted from the blade, F is the force, and τ is the torque.

Due to the aforementioned overtorquing, tests were carried out with the conditioning step (i.e., counterclockwise impeller), using a tip speed of -60 mm/s and a helix angle of -2°. Table 1 (vide infra) displays the tests conducted, which were carried out a minimum of three times each.

2.1. *Dynamic Vapor Sorption (DVS)*

DVS was carried out using a Surface Measurement Systems DVS Resolution – a Dual Vapor Gravimetric Sorption Analyzer. DVS is a gravimetric technique that is employed to evaluate the hygroscopicity in addition to the solvent uptake. This is determined by the relative mass change between the sample under dry conditions to a certain partial pressure. In a typical experiment, measurements were performed using 5% P/P_0 (relative pressure) increments with a dm/dt (change of mass over time) of 0.005% within a range of 0-95%. All analyses were obtained at 25 °C.

3. Results and Discussion

3.1 *Rheological measurements – Variable Flow Rate*

VFR tests carried out at 0 days (Fig. 3) that required weights for their consolidation were subjected to them for about 10 minutes to ensure compression while minimizing the time under consolidation. Upon analyzing the samples, no energy increase was observed using high moisture content or consolidation weights by themselves, reaching average maximums 12±1.6 mJ throughout the three tests. Interestingly, sample $50\mathrm{CS}_{0d}^{0kPa}$ resulted in runs with fewer erratic changes in comparison to the $0\mathrm{CS}_{0d}^{0kPa}$ or consolidated $0\mathrm{CS}_{0d}^{6.7kPa}$ corn stover samples, as shown by the red area throughout the run, which might have played a role in decreasing any random particle-particle interactions throughout the run. Solvent evaporation is a common phenomenon that can induce caking via the creation of liquid bridges and the ensuing solute solidification after the evaporation of the liquid. [13] While this is a common occurrence in various powders such as lactose and other food powders, [36,37] caking of the biomass was not present in the control samples. This can be due to the increased particle size the biomass samples have which reduce the amount of surface contact they have with one another whereas finer powders would have an easier tendency of creating liquid bridges.

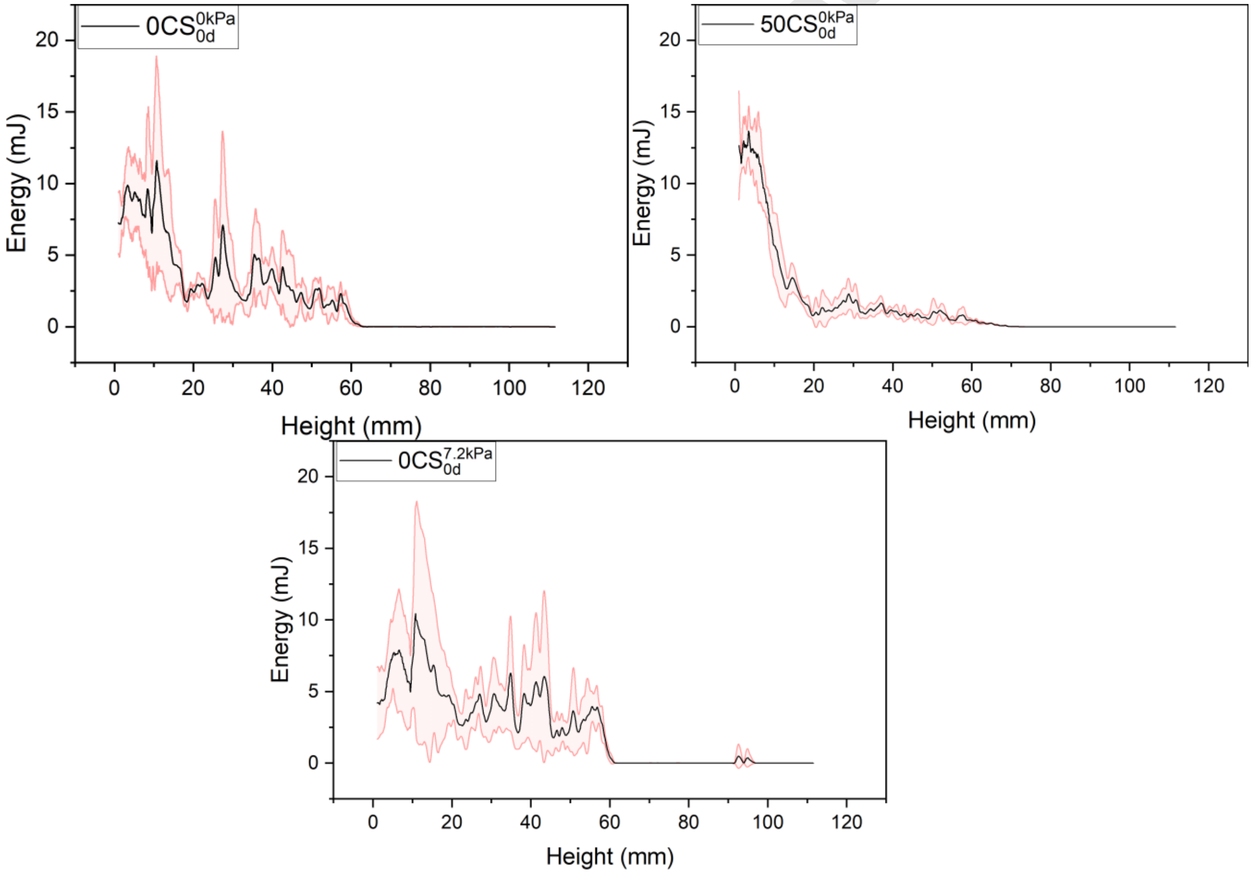

Fig. 3. Baseline Corn Stover VFR tests (0 days). Black trace is the average of 3 runs, the range of which is indicated by the red colored region.

When 25% MC samples were dried for 3 days and then evaluated, average energy and peak energy was 5.11±2.5 mJ and 12.41 mJ for $25\mathrm{CS}_{3d}^{0kPa}$, and 13.95 ± 5.3 mJ and 24.61 mJ for $25\mathrm{CS}_{3d}^{3.5kPa}$, respectively (Fig. 4). The similar energies for $25\mathrm{CS}_{3d}^{0kPa}$ and $0\mathrm{CS}_{0d}^{0kPa}$ suggests moisture by itself does not impart an irreversible change in the material properties. In contrast, upon utilizing both MC and CP, a drastic increase in the energy from the originally wet corn stover was shown, increasing almost threefold the energy required to traverse through the sample, reaching values of over 30 mJ. In addition to the change in energy, another noticeable change was the height in which the blade observed the caked corn stover, shifting significantly due to the compaction of the corn stover.

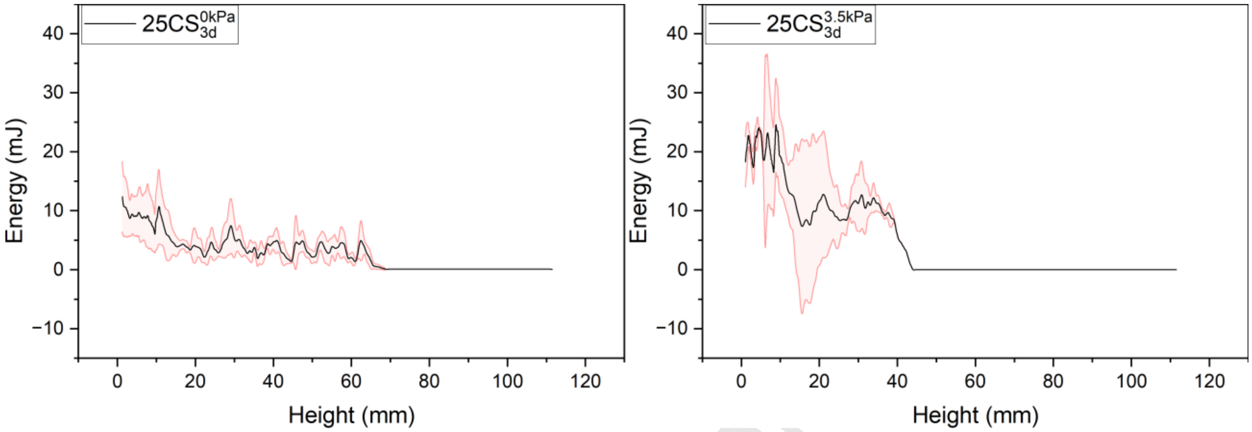

Fig. 4. Corn stover caking at 3 days and 25% moisture content, with (right) and without (left) consolidating pressure.

Similarly, high energy output from the blade was observed at 50MC under different consolidation pressures, averaging 32 mJ for $50\mathrm{CS}_{3d}^{3.5kPa}$, as can be seen in Fig. 5. The use of higher CP exhibited a significant increase in the overall energy throughout the caked sample, reaching values of over 60 mJ for $50\mathrm{CS}_{3d}^{3.5kPa}$. Higher CP ($50\mathrm{CS}_{3d}^{7.2kPa}$), while not showing a significant influence in the average energy being used from the blade, had a higher tendency to overtorque (i.e., abrupt end of analysis due to reaching maximum force/torque levels), indicative that the corn stover sample is highly caked.

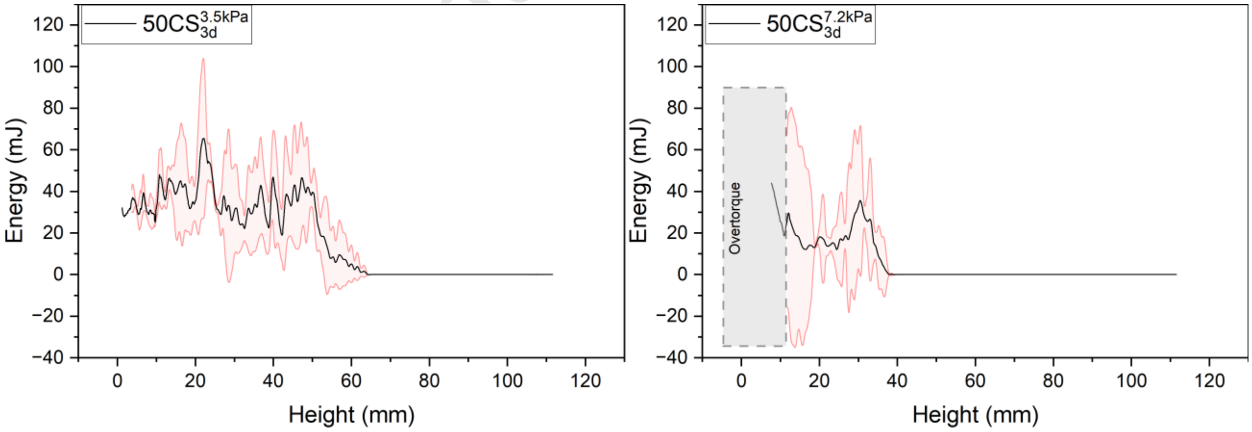

Fig. 5. Corn stover caking at 3 days and 50% moisture content. Gray dashed squares indicate a consistent overtorque on the instrument.

7-day dried samples are shown in Fig. 6. Similar to the other two time points, minimal energy is required if only a consolidating pressure or introduction or moisture is used, as can be seen by $0\mathrm{CS}_{7d}^{3.5kPa}$, $0\mathrm{CS}_{7d}^{3.5kPa}$, and $50\mathrm{CS}_{7d}^{0kPa}$. $50\mathrm{CS}_{7d}^{3.5kPa}$ on the other hand, exhibited similar behavior to $50\mathrm{CS}_{3d}^{7.2kPa}$ albeit with a lower maximum energy, indicating that the amount of time dried can be considered the least impactful variable to create a more caked sample.

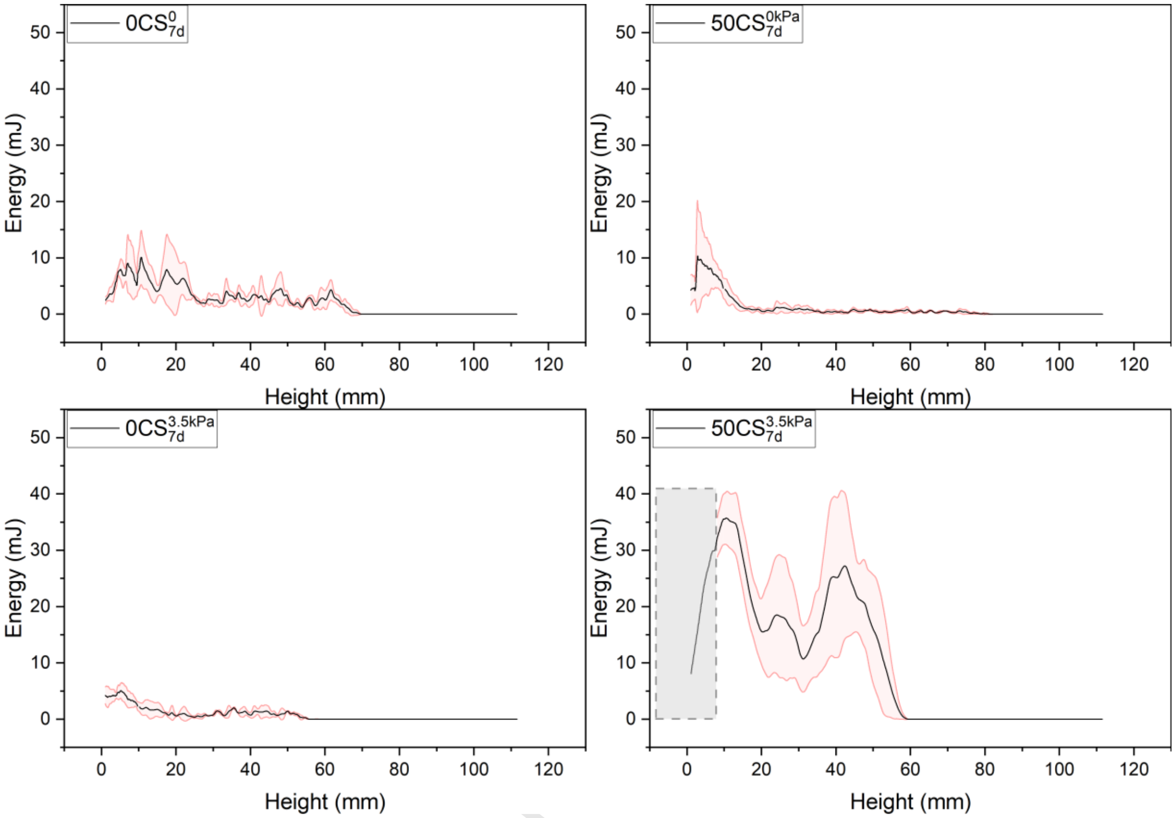

Fig. 6. Corn stover caking at 7 days. Gray dashed squares indicate a consistent overtorque on the instrument.

Maximum energies outputted and average energies partitioned at different heights for each run can be seen in Table 2, where an appreciable increase using both moisture and consolidating weights in the sample compared to using only one variable or in the absence of them is observed. Average energy output obtained through the runs using only one or no variable at 60 mm height was 3.68±1.27 mJ whereas with both MC and CP, energy values were 15.71±9.17 mJ—over five times the energy required to traverse the sample. Additionally, Fig. 7 shows a comparison between both the maximum and average (from 60 mm) energies obtained; the normalized values demonstrate that both the maximum and average energy values acquired are representative of each other.

Table 1. Average and maximum energy outputs obtained for each caking experiment conducted. All data used for the average energy values were obtained from a height of 60 mm and onward. Maximum values are determined with the average energy values obtained from a minimum of three runs.

Run	Days Dried	MC (%)	CP (kPa)	Maximum Energy (mJ)	Average Energy at 30 mm (mJ)	Average Energy at 60 mm (mJ)
1	0	0	0	11.59	6.15±2.79	4.429±2.87
2	0	50	0	13.66	5.74±4.65	3.518±4.19
3	0	0	7.2	10.44	5.53±1.90	4.55±1.99
4	3	25	0	12.41	6.55±2.63*	5.11±2.57
5	3	25	3.5	24.61	14.99±5.56	13.96±5.38
6	3	50	3.5	65.62	36.99±8.73	32.16±12.07
7	3	50	7.2	44.06	21.47±8.49	11.64±12.21
8	7	0	0	10.13	5.46±2.11	4.23±2.1
9	7	0	3.5	5.078	2.27±1.55	1.63±1.38
10	7	50	0	10.33	3.78±3.24	2.29±2.88
11	7	50	3.5	35.71	22.76±7.89	19.18±9.11

*Run 3 (Fig. 5) averaged at 40 cm height.

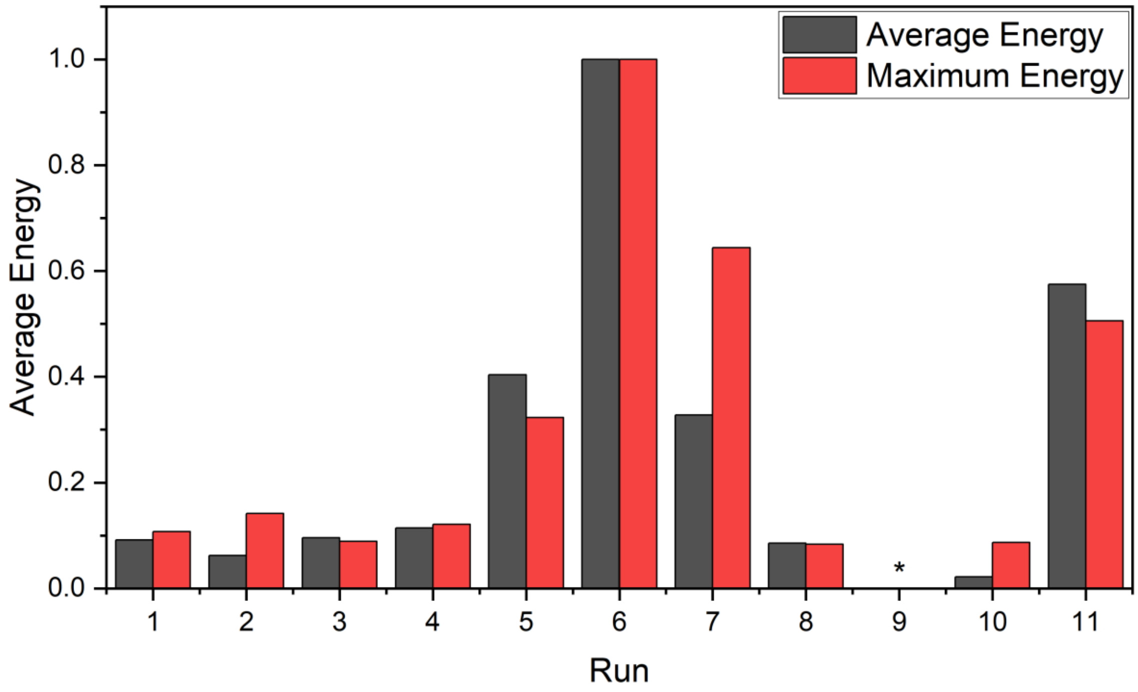

Fig. 7. Comparison of the normalized values obtained from the maximum and average energy values from the VFR tests. Average values used were from a height of 60 mm and downwards. Run 9 was observed to be the lowest on both values, hence, presenting a value of 0.

The handling of corn stover is significantly influenced by the simultaneous introduction of moisture and consolidating pressure. Corn stover, like other biomass, is mainly considered an amorphous powder, lacking long-range order and, where the glass transition temperature plays an important role when it comes to the caking of amorphous powders. [38,39] In the case of softwood lignin biomass such as corn stover, the glass transition temperature (T_g) is in the range 138-160 °C. The moisture content can have a substantial impact on its value, reducing the overall T_g, [40,41] as has been previously reported [42-44]

The decrease in T_g evidently affects the viscoelastic properties and plasticization of the material, making it more susceptible to caking. [41,45] Despite having less particle-particle interactions due the nominal size of the corn stover particles (2 mm) compared to fine powders, the increase in energy required for caking was apparent, albeit not necessarily linear; consolidating pressure and moisture content have an evident interplay, and as can be seen from runs 5-7 and run 11, no trend was found. It can be concluded that the consolidating pressure has a higher influence on the caking of the biomass than the moisture content due to the increased, albeit lower than run 6, maximum energy displayed from run 7, The number of days dried was also found to be inconsequential given that the moisture dries within the three-day mark, hence having negligible impact on the caking of the biomass upon drying more days. In conclusion, minimization of moisture content, which is the determining factor if the biomass will undergo caking. Additionally, other factors to consider is the pressure distribution of the storage system (i.e., silos), given that they behave in a non-uniform matter; [46,47] Moisture content will also influence the behavior of the static pressure observed in the silo, although the limits one should see should be significantly lower[47-49] and the MC values utilized here were to evaluate on any potential intricacies one might observe.

3.2 Dynamic Vapor Sorption of CS anatomical fractions

To elucidate which of the corn stover anatomical fractions may prove a more significant influence in water sorption, dynamic vapor sorption (DVS) was carried out on the bulk and the leaf, stalk, husk, and cob-enriched fractions of corn stover which were provided by INL. The assessment of water uptake is pivotal in evaluating storage concerns issues as well as their overall behavior in processes such as their discharging. Fig. 9 shows DVS isotherm for the 2 mm bulk corn stover, showing a type IV isotherm, i.e., a multilayer physisorption of water at low P/P_0 and a capillary condensation mechanism at the higher pressures, which is presumed to be filling the mesopores. [50] The maximum change observed at 95% relative humidity (RH) was of 28.55% (Fig. 8), indicating that over 40% of the water is physisorbed or adsorbed onto the surface. Despite excess introduced, this water still plays an important role in the creation of liquid bridges and consequently increasing the cohesive forces between particles, depending on the regime. [23,24,51]

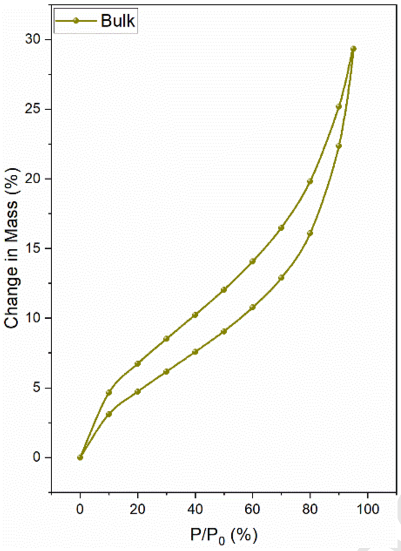

Fig. 8. DVS isotherm of 2 mm bulk corn stover.

In addition to the bulk corn stover, anatomically-enriched fractions (leaf, husk, cob, and stalk) were studied to determine any exhibited differences in terms of their uptake. As can be seen in Fig. 9, all fractions displayed analogous sorption isotherms, with distinct maximum changes at 95% RH. [50]

Unlike most biomass samples, [52,53] the corn stover fractions do not have a well fit for the classic GAB (Guggenheim-Anderson-deBoer) equation, [54] as has been reported previously. [55,56] Although similar in terms of their sorption isotherms, the stem fraction was shown to exhibit a particularly lower weight percent increase at the highest P/P_0, with a maximum value of 24.5%, whereas the remaining three were within ±1.5% of their values, showing a 28.8% weight increase for the leaf fraction – the highest value presented of the 4 fractions. The elucidation on the difference between the moisture uptake of each anatomical fraction can be insightful for other potential studies such as the flowability of complex mixture for flowability optimization and to determine which fraction can be likely bottle-necking the discharging or plugging of the feed in the silo. [57,58] The relatively hydrophobic nature of corn stover may also prove to be vital depending on the conversion pathway; high-temperature conversion would be advantageous at every stage whereas at low-temperature can be detrimental by cause and effect of restricting wettability and subsequently inhibiting pretreatment and enzymatic hydrolysis. [57]

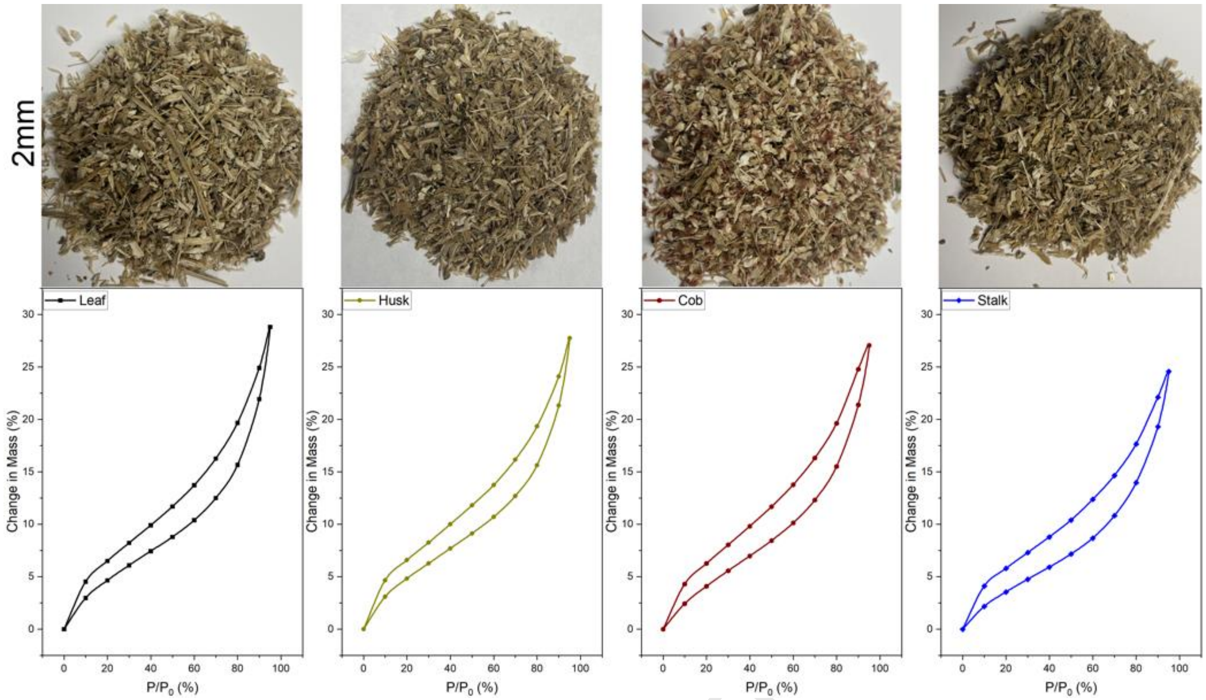

Fig.9. (Top) 2 mm corn stover anatomical fractions and (bottom) Dynamic Vapor Sorption of the 2 mm corn stover fractions.

2. Conclusions

In conclusion, the caking of biomass, exemplified by corn stover, is notably influenced by the interplay of moisture content and consolidating pressure. Key notes observed were the following:

- No significant increase in energy from the FT4 blade observed when moisture or consolidating pressure were introduced individually.
- Noticeable increase in energy usage observed by FT4 when both moisture and consolidating pressures were introduced simultaneously.
- The number of days being dried had the least impact on the energy output, requiring only three days to fully undergo caking.
- MC and CP in hoppers and silos are inevitable factors which will be present to some extent in addition to the inherent moisture observed from the biomass. Due to its screen size, 2mm corn stover demonstrated a higher susceptibility towards caking due to a closer particle-particle interlocking, making liquid-capillary bridge formation more likely; this was observed throughout all stages of the caking procedure where both MC and CP were simultaneously involved. The effect of drying was also an important factor, where it created a condensation of the particles and consequently the caked material more easily. Therefore, the use of bigger screen size can potentially mitigate caking effects from moisture, reducing overall condensation in addition to liquid bridging. Increasing the screen size during comminution will also lead to reduced operational costs, thereby enhancing the overall feasibility of the process.

Most of the variances observed can be contributed to the inherent heterogeneity the samples have, which will give them a higher degree of variance in their analyses. Nonetheless, the elucidation on how these materials behave in both MC and CP, independently and concurrently, can provide a behavioral baseline for potential feedstock that will be subsequently stored in discharge hoppers with varied MC. Further studies will be implemented tackling different feedstocks as well as screen sizes to determine an optimal condition for future biomass handling, such as the use of calorimetric studies to assess the changes in T_g.

Declaration of Competing Interest

The authors declare they have no known competing financial interest or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

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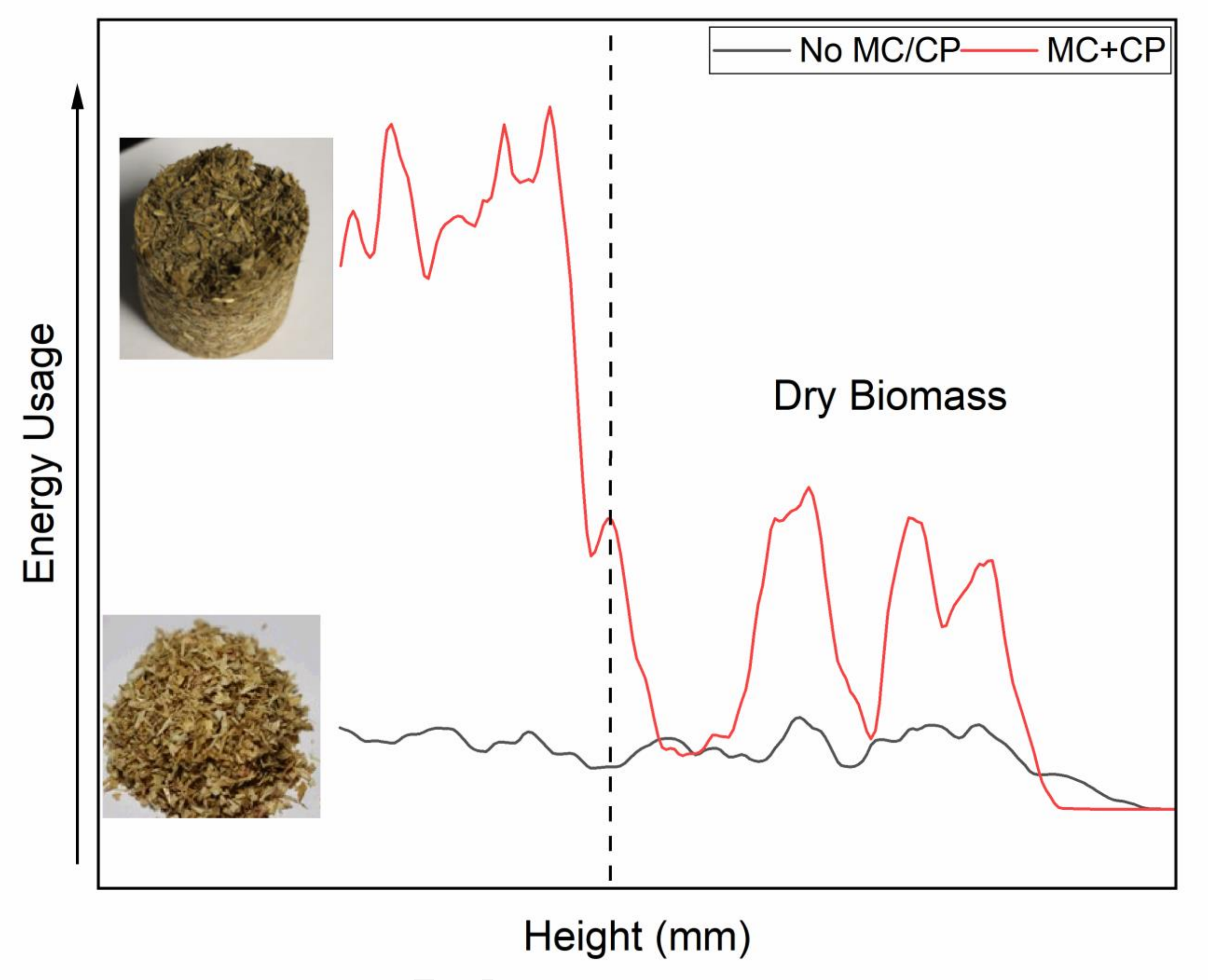
Declaration of Competing Interest

The authors declare they have no known competing financial interest or personal relationships that could have appeared to influence the work reported in this paper.


No MC/CP
MC+CP
Dry Biomass
Energy Usage
Height (mm)

Highlights

- Either moisture content or consolidating pressure imparted minimal energy changes in variable flow rate (VFR) tests.
- A combination of moisture and consolidating pressure resulted in the highest energy required, suggesting a caked material.
- Stalk anatomical fraction presented the lowest water uptake via dynamic vapor sorption (DVS).