Effects of alcohols versus water addition on the bulk solids frictional behaviour of flax shive

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Parsons, R. V., Cenkowski, S. and Zhang, Q. 2009. **Effects of alcohols versus water addition on the bulk solids frictional behaviour of flax shive**. Canadian Biosystems Engineering/Le génie des biosystèms au Canada **50**: 3.67–3.75. Understanding the frictional behaviour of biomass fibre is critical for its effective utilization. There is growing research on the handling of fibrous biomass, including the effects of moisture content; however, the impacts of non-water solvents on frictional behaviour have not been addressed in the literature. In particular, the implications of adding alcohol solvents to flax shive, as could occur as part of a high-value product recovery process, remain undefined. The bulk solids frictional behaviour of flax shive was investigated using a standard shear test system. The angles of internal friction and wall friction were evaluated to determine the effects of adding varying proportions of one of three different liquids, methanol, water or isopropanol. The shear strength of flax shive, as measured by the angle of internal friction, was increased by the addition of any of the three liquids, but did not depend on which liquid or what quantity of liquid was added over the range tested. Given differences in surface tension and viscosity of the three liquids, neither property appeared to affect the angle of internal friction. The frictional behaviour of flax shive against a standard galvanized-metal wall material was measured by the angle of wall friction. Changes in the angle of wall friction were different, with a much larger increase found in the case of water addition than for either of the alcohols. Change in the angle of wall friction appeared to be related to the surface tension of the added liquid. **Keywords:** frictional behaviour, bulk solids, flax shive, biomass, fibre, internal friction, wall friction, alcohol, methanol, isopropanol.

Il est essentiel comprendre les caractéristiques de frottement de biomasses fibreuses afin de pouvoir les utiliser efficacement. Il y a de plus en plus de recherche faite sur la manipulation de la biomasse fibreuse, incluant les effets de la teneur en eau, cependant les impacts de l'addition de solvants non-hydriques sur les caractéristiques de frottement n'ont pas été documentés dans la littérature. En particulier, les implications de l'addition de solvants à base d'alcool à de la chènevotte (addition pouvant être faite lors de procédés de récupération de produits de haute valeur) ne sont pas encore determinées. Le comportement de frottement de la chènevotte en vrac a été étudié en utilisant un appareillage standard pour des essais en cisaillement. Les angles de frottement interne et de frottement contre une paroi ont été évalués pour déterminer les effets liés à l'addition en proportions variées d'un des trois liquides suivant: le méthanol, l'eau ou l'isopropanol. Le cisaillement de la chènevotte, mesuré par l'angle de frottement interne, a été augmenté par l'addition de chacun des trois liquides mais ne dépendait pas du type de liquide ou de la quantité de liquide ajouté dans la plage de tests réalisés. Malgré les différences au niveau de la tension de surface et de la viscosité des trois liquides, aucune propriété ne semble avoir changé l'angle de frottement interne. Le comportement de frottement de la chènevotte contre une surface de métal galvanisé standard a été évalué en mesurant l'angle de frottement de paroi. Les changements dans l'angle de frottement de paroi étaient différents avec une augmentation de la valeur lorsque de l'eau était ajoutée comparativement à l'ajout des deux alcools. Le changement dans l'angle de frottement de paroi semblait être relié à la tension de surface du liquide ajouté. **Mots clés:** comportement de frottement, solides en vrac, chènevotte, biomasse, fibre, frottement interne, frottement de paroi, alcool, méthanol, isopropanol.

INTRODUCTION

Fibrous ligno-cellulosic biomass, which consists of residual straw or other plant materials left over after harvesting, has been recognized as a potential renewable source for the production of fuels, materials, and high-value compounds (BioProducts Canada 2004). For these varied uses, it will be necessary to significantly process the biomass feedstock using unit operations such as fractionation and chemical modification that will include the use of water and in some instances non-water solvents, particularly alcohols. Ileleji (2005) and Zhou and Ileleji (2005) noted that the processing of biomass can be difficult and costly if handling characteristics are not properly understood. There is a growing body of research on the handling of fibrous biomass, including the effects of moisture content, however, the impacts of non-water solvents on the bulk solids frictional behaviour of biomass have not been addressed in the literature, and remain ill defined.

The objective of this work was to determine the effects of adding selected alcohols versus water on the angle of internal friction and the angle of wall friction for flax shive, in particular reflecting the conditions of both the feed material and liquid addition that would be encountered in a pretreatment process for the recovery of high-value constituents, such as oligosaccharides. At the same time, alcohol solvents with differing viscosity properties were included, permitting the opportunity to also assess the impacts of variations in the surface tension and the viscosity of the added liquid on frictional behaviour.

Research has been underway at the University of Manitoba on the potential recovery of nutraceutical constituents from flax shive, including recovery through the use of alcohol solvents. The selection of flax shive as a candidate feedstock was straightforward. Kozlowski et al. (2005) identified that Western Canada is a leading world production area for flax (*Linum ussitatissimum*), albeit dominated primarily by oilseed flax. Significant quantities of flax straw are processed, nevertheless, to recover bast fibre from the outer portion of the flax stem. This generates large volumes of flax shive, a waste residual composed primarily of the woody core of the flax stem. The scutching or decortication process, described in general by Salmon-Minotte and Franck (2005), produces a size-reduced and relatively consistent shive fraction, potentially suitable as a feedstock for further processing.

The use of alcohols was also straightforward. As described by Wyman (1999), alcohol solvents have been well identified for use in organosolv fractionation to separate ligno-cellulosic biomass into major components. More specifically, Vazquez et al. (2000) noted that pretreatment of raw biomass materials with alcohols can be a potential initial step in the manufacture of selected oligosaccharides. Being able to exploit alcohols for this purpose, however, requires that their effects on the frictional behaviour of flax shive be quantified.

LITERATURE REVIEW

The basic theory of bulk solids frictional behaviour has been well established, described for example in Mohsenin (1986) or Shamlou (1988). Bulk solids frictional behaviour is characterized using plasticity theory and evaluated in terms of the classical, linear Mohr-Coulomb relationship:

$$\tau = \mu\,\sigma + C \tag{1}$$

where: τ is the shear stress (kPa), σ is the normal stress (kPa), μ is the slope or coefficient of friction (dimensionless), and C is the intercept (cohesion or adhesion) (kPa).

The slope term represents the coefficient of internal friction (μ_i) or the coefficient of wall friction (μ_w), depending on whether friction is assessed against the solid material itself or against a wall surface. Similarly, the meaning of the intercept term depends on which type of frictional interaction is being assessed. In the case of internal friction, it represents cohesion, the sticking together of particles, while in the case of wall friction, it represents adhesion, the sticking of particles to another surface material. The coefficients of friction are often expressed as angles of friction (ϕ), whether angle of internal friction (ϕ_i) or angle of wall friction (ϕ_w), by calculating the arctangent of the respective coefficient of friction:

$$\phi = \text{arctangent}\ (\mu) \tag{2}$$

In terms of liquid addition to bulk solids, the formation of liquid bridges between particles has been long recognized as an important and sometimes problematic factor determining bulk solids behaviour (Peleg 1977). Shamlou (1988) indicated that as the content of liquid in a dry bulk solid is increased, attractive forces arising from physically absorbed liquid films can readily dominate the interactive forces between individual particles or between particles and an adjacent surface. The magnitude of these forces, termed capillary forces, is related to the surface tension of the liquid. If the content of liquid in a bulk solid continues to be increased, as described by Fekete et al. (2007), at some level the porosity of the solid material will be exceeded and it will not be able to retain internally any additional liquid. Up to this saturation point, wall friction at an adjacent surface will be a combination of solid particle sliding friction and liquid capillary forces. Beyond a saturation level, the adjacent surface will be completely wetted by liquid alone, and friction will be dominated entirely by the liquid and its viscosity. Thus, both the surface tension and viscosity properties of the liquid can be important under different conditions.

As summarized by Schulze (2006), it is not possible to theoretically deduce the frictional behaviour of a specific bulk solid material or the extent to which it may be able to freely flow. This is firstly because behaviour depends on too many variables, both of the handling system and the particulate material itself, including size, size distribution, shape, and composition, and, secondly, because of potential changes in the dominant forces. It makes sense to empirically derive the necessary quantitative information. In considering the frictional behaviour of flax shive, three general groups of materials that may have potential relevance have been extensively evaluated in the literature. These are other agricultural bulk materials, including seed crops and foods; powders, particularly food products; and, most relevant, other ground fibrous biomass.

Seed kernels, fruits and other related products share compositional similarities with fibrous biomass, but have quite different particle characteristics, being generally larger in size, more regular in shape, and less fibrous in character. Of greatest relevance is the impact of liquid addition on such agricultural materials, with only water being considered in the literature. The general indication for agricultural materials is that adding moisture has a monotonic increasing impact, i.e., both internal friction and wall friction increase as moisture content is increased (Mohsenin 1986), although the precise nature of variation with moisture over a range of values remains less certain. Mohsenin (1986) summarized various earlier studies in this area for agricultural materials in general, both relevant to wall friction and internal friction, but suggested no consistent relationships with moisture content.

Fine powders have been extensively addressed in the literature, and, as described by Prescott and Barnum (2000), moisture content is well recognized as an important issue in their handling, even to the extent of impeding the ability of powders to flow. However, the nature of powders is not necessarily similar to fibrous biomass. Powder products are generally much finer in particle size and also can be vastly different in composition.

An important insight from fine powder handling is in the investigation of viscosity and surface tension modifiers. The use of bulk solid lubricant additives, particularly magnesium stearate, is common in the pharmaceutical industry, as described by Faqih et al. (2007). The established effect is to provide a surface coating to reduce inter-particle friction and particle-wall friction, however, the precise causative action of the lubricant is less clear, i.e., whether the effect results from viscosity or from surface tension. Scoville and Peleg (1981) specifically considered the impacts of liquid films on the bulk properties of model powders of glass beads. They observed:

> “Perhaps the most interesting result of this work is that neither the film viscosity in the range of up to 2000 mPa·s nor its surface tension at 35–75 mN/m had a decisive effect on the powder bulk characteristics. This was found in all the tested powders regardless of their particle size and also in sand where the shape of the particle was more irregular. Apparently, the presence of the liquid film by itself was sufficient to cause drastic modifications in the bulk properties.”

For flax shive, the frictional behaviour of ground ligno-cellulosic biomass residuals is most relevant, having both similar composition and fibre-like particle nature. However, only recently has the behaviour of such biomass begun to be studied significantly. As observed by Ileleji (2005), “There is limited basic research that has been conducted to characterize their physical and flow properties with respect to handling because, until recently, they were not considered a major feedstock for conversion.”

Mani et al. (2003, 2004) evaluated wall friction and adhesion coefficients for corn stover, based on variations in both grind size and moisture content. The coefficient of wall friction was found to increase with increased moisture content, with the relationship in this case suggested to be quadratic in nature. However, the moisture content was relatively low, ranging from 7.2 to only 15.2% wb. Shaw and Tabil (2006) compared properties for a broader range of materials including peat moss, oat hulls, wheat straw, and flax shive, but did not study the effect of moisture content or grind size. For sample moisture content in the range of 9–10% wb, they found ground flax shive to have a similar coefficient of wall friction as ground wheat straw or oat hulls, but with a relatively higher adhesion value.

Zhou and Ileleji (2005, 2006) assessed the frictional behaviour of various grinds of corn stover and switchgrass, specifically samples ground through hammermill screen sizes of 6.4, 3.2 and 1.6 mm. They identified moisture content and particle size as key variables. Zhou and Ileleji (2005) evaluated the angle of repose (ϕ°), which, as noted by Reimbert and Reimbert (1987), is closely related to the angle of internal friction. Only corn stover was assessed at different moisture conditions by Zhou and Ileleji (2005). They consistently found an increase in the angle of repose for higher moisture content. However, for these samples only two moisture conditions were assessed, a dry condition, in the range of 5.4 to 6.0% wb, and a wet condition, in the range of 25.6 to 48.5% wb. No comprehensive relationship could be deduced, given the lack of intermediate data points. Zhou and Ileleji (2005) also found the angle of repose to be generally higher for a larger grind size for both corn stover and switch grass, i.e., higher for 6.4 mm grind than smaller grinds. At the same time, they found that the angles of repose for both ground corn stover and switch grass were generally much higher than for grains, with the expectation that these material would be problematic for transport and storage.

Fasina (2006) and Fasina et al. (2006) investigated flow properties for various grinds of peanut hulls, switchgrass, and poultry litter, but in this case using relatively smaller hammermill screen sizes of 3.2, 1.6 and 0.8 mm. They did not include the effect of moisture content. They found, in contrast, that screen size did not significantly affect the unconfined yield strength or the cohesion of samples in this case. For all samples, they found the angle of internal friction and cohesion were greater than critical values of 30° and 2 kPa, respectively, such that none of the materials would be expected to flow under gravity discharge from a bin or silo.

Available literature suggests that flax shive, like other fibrous biomass, likely would be a problematic bulk solid. Both the angle of internal friction and wall friction would be expected to be relatively high. Flow behaviour would be expected to significantly worsen through the addition of moisture.

An important consideration in recent literature where moisture content was a variable is that higher moisture content levels were obtained by using harvested wet material stored at reduced temperatures until used, adding distilled water to relatively dry samples followed by equilibration periods of up to several days, or exposing samples to a very high relative humidity environment, again for periods of up to several days. Such procedures to set or adjust moisture content prior to bulk solids frictional evaluation, however, do not reflect the anticipated conditions in a pretreatment process, whereby relatively dry biomass feedstock would be dosed with liquid and then processed within a relatively short period of time, with processing likely to begin within about 3 h and concluded in under 24 h.

Additional issues in the handling and evaluation of fibrous biomass have been identified in the literature. In conventional shear testing for frictional behaviour, particularly for powders, it has been typical for samples to be preconsolidated (Shamlou 1988). However, as described by O’Dogherty (1989), compressed fibrous straw exhibits elastic recovery, rendering preconsolidation impractical. Sieving of biomass samples is also recognized to be problematic, whether for separation or analysis. Sieves are only two-dimensional in nature while individual fibrous particles are three-dimensional, potentially having varying lengths, and as noted by Bartl et al. (2004), sieves cannot distinguish between width and length of biomass fibres. Sieving also represents effectively a form of staged gravity-flow through openings of decreasing size. As outlined by Paulrud et al. (2002) the highly irregular and often elongated particle shape associated with size-reduced biomass has a significant impact on the passage of materials through sieves, in particular increasing the potential for bridging. Naita et al. (1998) found in the measurement of particle size distribution for non-biomass materials by various analytical techniques that shape had a much more important impact for rod-like particles than for block-shaped particles. Yang et al. (2006) specifically compared image analysis versus standard sieves for ground biomass, and confirmed that caution must be exercised in the use of conventional sieve methods given the highly irregular nature of the fibrous biomass. Concerns regarding sieving were considered in this research in the separation of the flax shive samples.

METHODS

Shive sample preparation

Sufficient shive for testing was obtained during the early summer of 2006 from the production line of the flax decortication facility of Schweitzer-Mauduit Canada Inc., located near Carman, Manitoba. The material from the decortication line was double-sealed in plastic bags and stored in a cool, dry location until used in order to minimize changes in moisture content or other properties.

Schweitzer-Mauduit Canada Inc. staff described their decortication waste as composed of three roughly equal mass components: mid-core shive itself; residual short waste fibre, termed tow; and dust. Shive was segregated prior to testing using standard sieves (Canadian Standard Sieve Series, Tyler, St. Catharines, ON). Only selected sieves were used, and in the following order: No 6, No 8, No 16, No 14, No 14 and then the pan. The material was shaken for approximately 120 s in a shaker (Model CL 305-A-1, Soil Test Engineering, Evanston, IL). The material retrieved for testing was that passing through the No 8 mesh screen (nominal opening size of 2.38 mm), but retained on the No 16 mesh screen (nominal opening size of 1.19 mm). This pretreatment was not done for analysis purposes, but rather ensured that the samples for testing consisted predominantly of the spongy stalk mid-core, with the residual longer fibres largely excluded and the fine dust removed.

The two-dimensional deficiency of sieving was recognized. To mitigate this issue and ensure consistency of test samples, batches of mid-core shive were prepared by aggregating and mixing multiple sieve retentate samples into a single batch. Three separate batches were prepared in this way, with batch sizes of approximately 1500 g for Batch #1 and Batch #2, and 3000 g for Batch #3. Comparative shear test runs were conducted using samples from the same mixed batch.

Moisture content

Schweitzer-Mauduit Canada Inc. staff indicated that the moisture content of feed flax material to the decortication plant tended to be in the range of 10 to 14% wb, prior to processing. Three random samples of shive were retained in sealed plastic bags and assessed afterward for moisture content using ASAE standard S358.2 (ASAE, 2004).

Liquid additions

Samples of approximately 450 g of shive were withdrawn from respective mixed batches and placed into 20 L sealed plastic buckets. The samples were dosed with one of three liquids: methanol; water; or isopropanol (2-propanol). The liquids involved are all hydrophilic in nature, but the two alcohols have much lower surface tension and the three liquids have varying viscosity properties.

Relevant liquid property data were obtained from the CRC Handbook (2006) for the three liquids, and linearly interpolated to 20°C, the approximate temperature of the laboratory. The resulting surface tension values were 22.5, 72.7, and 21.3 mN/m for methanol, water and isopropanol, respectively, and viscosity values were 0.59, 1.07, and 2.55 mPa·s in the same order, respectively. Industrial grade methanol and isopropanol (i.e., 99.9%) were obtained from Canadian Tire and Safeway Pharmacy, respectively. Commercially available distilled water was used for water-dosed samples, this also obtained from Safeway. Four levels of liquid addition were used:

- No liquid, representing the control;
- Approximately 203 g liquid, representing 45% of the shive sample mass;
- Approximately 405 g liquid, representing 90% of the shive sample mass; and
- Approximately 608 g liquid, representing 135% of the shive sample mass.

These liquid doses were in addition to the residual moisture content already present in the shive samples, but were set sufficiently high to be significantly dominant in all cases.

All liquid-dosed samples were left for 3 h in the sealed buckets prior to testing in order to approximate the conditions that could be experienced as part of an actual shive processing operation. Similarly, all shear testing of liquid-dosed samples was completed within 24 h of liquid addition.

Shear testing

The shear test unit used for the experiments had been earlier constructed internally at the Department of Biosystems Engineering at the University of Manitoba, and was described briefly in Zhang et al. (1994). This unit is consistent with the standard shear apparatus originally developed by Jenike for determination of flow properties, and described in more detail in Mohsenin (1986). Important features of the device and associated methods are noted as follows:

- Unit incorporated a square rather than round test cell, with two compartments constructed of clear plastic of a thickness of approximately 13 mm. As a result, no rotation of the test cell was employed during testing. The elevation-view of the unit when used to assess internal friction is represented schematically in Fig. 1. The plan-view internal dimensions of the square cell were approximately 127 × 127 mm, with the upper and lower compartments each 75 mm high.
- Upper cell compartment was pulled at a constant rate of 0.12 mm/s, as opposed to being pushed, with the shear force on the drawn load cell being data-logged at 1 s intervals using a data acquisition unit (Model 3852A, Hewlett-Packard, Santa Clara, CA).
- Normal loads on the cell were achieved using a mass slung underneath the unit by means of a cradle sled suspended at the centre of the cell top plate using a

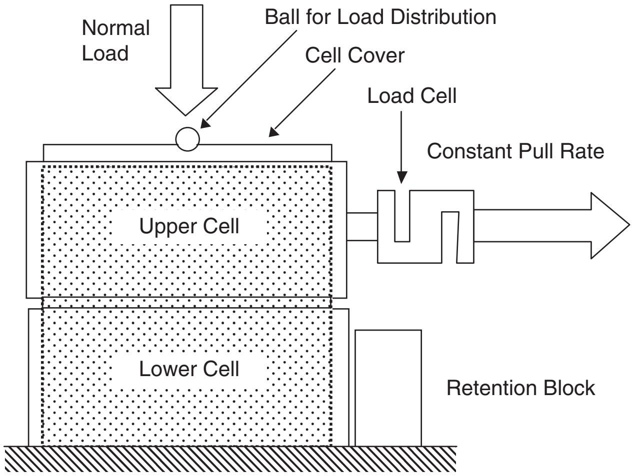

Fig. 1. Schematic elevation view of test unit as used for internal friction assessments.

stainless steel ball to ensure uniform weight distribution (Fig. 1).
- Lower cell portion was removable and could be replaced by a block with a standard flat galvanized sheet-metal top surface to permit testing for the angle of wall friction. The elevation-view of the unit when used to assess wall friction is represented schematically in Fig. 2.
- No pre-consolidation loading of the samples was undertaken.

Individual test runs were conducted over a period of approximately 120 to 180 s each. For each individual batch of shive, random control samples prior to adding any liquids were withdrawn for shear testing, then returned and remixed into the batch.

It was not possible to completely prevent evaporation of alcohols during testing, given volatility, but exposure was limited. Samples incorporating liquids were removed quickly from a respective sealed bucket into the test cell. When the top plate of the cell was fitted, the total exposed area of the cell was only the gap between the upper and lower portions. This clearance was limited to no more than 2 mm, such that the total open area was less than 10^{-3} m^2, reducing potential evaporation of the alcohols. Once tests were completed, samples were then quickly returned back to their bucket, remixed into the remaining sample, and resealed.

At least four test runs were conducted using different compressive normal loads at each liquid-addition level for each liquid, and for each batch control sample to derive a linear regression based on Eq. 1. The mass of the entire sled apparatus was about 14 kg, to which additional increments of 20 kg were added. Four different normal stresses were thus employed: 8.5, 20.7, 32.8, and 45.0 kPa. The respective angle of internal friction or wall friction was calculated from each derived slope using Eq. 2.

One data point was missing from the results for both the angle of internal friction and wall friction. This was for the shive sample dosed with 45% isopropanol. A problem developed with the shear test unit preventing this final set of runs from being completed.

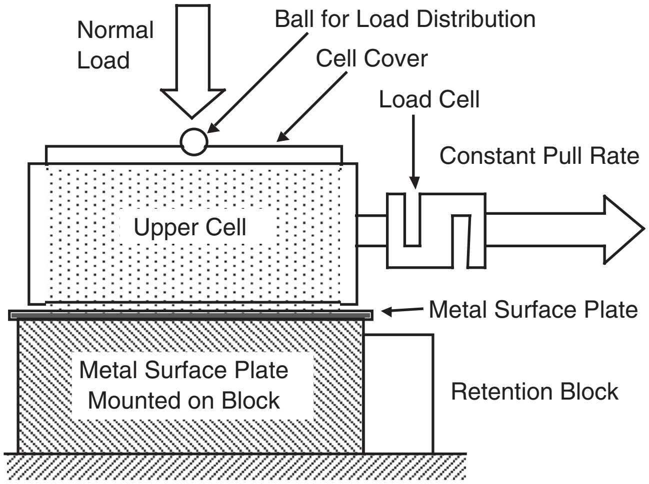

Fig. 2. Schematic elevation view of test unit as used for wall friction assessments.

Data analysis

Data analysis was performed using Microsoft Excel X for Mac, Service Release 1. Two key statistical analyses of data were conducted manually, given the small number of data points involved, to compare water-dosed versus alcohol-dosed samples for both angles of friction. These involved F-tests, calculating F statistics to assess the similarly of sample variances, and two-sample pooled t procedures, calculating two-sample pooled t statistics to assess the similarity of sample means. Procedures used are as outlined in Moore and McCabe (2006), with the use of the two-sample pooled t-test being based on the assumption of equivalent population standard deviations, verifiable from the corresponding F-test.

RESULT and DISCUSSION

The moisture content of three random shive samples was found to be 8.3% wb ± 0.1%. This was lower than the indicated moisture content of the flax straw prior to processing, but consistent with the findings of Zhou and Ileleji (2005), who found that the moisture content of biomass was reduced through hammermilling. These results also confirmed that the added liquids were the dominant liquids present, representing at minimum more than five times the residual moisture content.

The selection of shear testing within a period of 3 to 24 h after liquid addition was dictated by process considerations. It is important to note that when shear testing was started after the 3 h period, all shive samples were found to have completely absorbed all added liquid, with no evidence of free liquid in any bucket. More importantly, there was no exuding of liquids evident from any of the samples even under the highest compressive loads. In all cases, the liquid added to the shive was completely

Table 1. Data for internal friction assessments of liquid-dosed flax shive samples(n = 4 data points in all cases, except * where n = 5 data points).

Batch number	Liquid Type	Liquid Addition (%)	Slope or coefficient of internal friction (μ_i)	Intercept or cohesion (kPa)	r^2
#1	Control	0	0.43	2.10	0.99
	Methanol	90	0.70	1.41	0.99
	Water	90	0.73	1.17	0.99
	Isopropanol	90	0.78	−0.61	0.99
#2	Control*	0	0.53	0.95	0.98
	Methanol	90	0.79	−1.05	0.99
	Water	90	0.75	1.13	0.99
	Isopropanol	90	0.72	2.14	0.99
#3	Control	0	0.56	1.38	0.98
	Control*	0	0.59	0.10	0.99
	Methanol	135	0.72	1.13	0.99
	Water	135	0.82	−0.99	0.99
	Isopropanol	135	0.66	4.32	0.97
	Methanol	45	0.74	1.37	0.99
	Water	45	0.75	1.68	0.99

absorbed, and none of the samples achieved a liquid saturation condition.

The slope data for the internal friction assessments are presented in Table 1 in the order conducted. Shear stress versus normal stress data provided excellent fit in all cases, with the lowest value of the bivariate coefficient of determination (r^2) being 0.97 (i.e., for Batch #3 test using 135% isopropanol). A total of four control runs were conducted without liquid addition. The mean value of the angle of internal friction for these control tests translated to approximately 28°, with a standard deviation of 3° based on a sample size n = 4.

The calculated angles of internal friction for the various liquid-dosed samples are summarized in Table 2. An increase in the angle of internal friction clearly occurred due to the addition of water or either of the alcohols. However, the results for all the liquid-dosed samples were relatively similar, despite differences in the type of liquid added or the level of dosing. Verification is provided later in Table 5. The mean value of the angle of internal friction for all the liquid-dosed samples was approximately 37°, with a standard deviation of 2° based on a sample size $n = 11$. The increase in the angle of internal friction appeared to result from liquid addition, and not depend on which liquid or what quantity of liquid was added over the range tested. Given differences in surface tension and viscosity properties for the three liquids, the further implication is that neither of these liquid properties had an important impact on the angle of internal friction. This result is consistent with the earlier findings of Scoville and Peleg (1981).

Table 2. Summary of angles of internal friction for liquid-dosed tests (For explanation of missing data point * see Methods section).

Liquid addition (%)	Angle of internal friction (ϕ_i,°) Methanol	Water	Isopropanol
45	37	37	*
90	35	36	38
90	38	37	36
135	36	39	33

The slope data for the wall friction assessments are presented in Table 3 in the order conducted. Again, the shear stress versus normal stress data provided excellent fit in all cases, with the lowest value of r^2 being 0.89 (i.e., for Batch #2 control test). A total of three control runs were conducted without liquid addition. The mean value of the angle of wall friction for these control tests translated to approximately 12°, with a standard deviation of 2° based on a sample size $n = 3$.

The calculated angles of wall friction for the various liquid-dosed samples are summarized in Table 4. An increase in the angle of wall friction occurred due to liquid addition, but was more complex. In this case, the values for the addition of water were clearly higher and different from those for the two alcohols. The mean value of the angle of wall friction for the water-dosed samples translated to approximately 28°, while the mean value of the angle of wall friction for all alcohol-dosed samples translated to approximately 23° (Table 5). Given differences in surface tension and viscosity properties for the three liquids, the further implication is that the surface tension, but not the viscosity, of the added liquid affected the angle of wall friction. At the same time, the angle of wall friction appeared to be unaffected by the quantity of

Table 3. Data for wall friction assessments of liquid-dosed flax shive samples on galvanized metal (n = 4 data points in all cases, except * where n = 5 data points).

Batch number	Liquid		Slope or coefficient of wall friction (μ_w)	Intercept or adhesion (kPa)	r^2
	Type	Addition (%)			
#2	Control	0	0.22	1.16	0.89
	Methanol	90	0.48	1.06	0.98
	Water	90	0.56	0.42	0.99
	Isopropanol	90	0.48	0.77	0.99
#3	Control	0	0.24	−0.20	0.99
	Control*	0	0.18	0.67	0.98
	Methanol	135	0.38	1.05	0.99
	Water	135	0.53	0.29	0.99
	Isopropanol	135	0.43	0.94	0.99
	Methanol	45	0.36	0.86	0.99
	Water	45	0.54	0.16	0.99

Table 4. Summary of angles of wall friction for liquid-dosed tests (For explanation of missing data point * see Methods section).

Liquid addition (%)	Angle of wall friction (ϕ_w,°)		
	Methanol	Water	Isopropanol
45	20	28	*
90	26	29	26
135	21	28	23

liquid added over the range tested, just as in the case for the angle of internal friction.

The experiments showed a clear difference between the nature of changes in the angle of wall friction versus the angle of internal friction when an alcohol versus water was added. As illustrated by the two-sample pooled t statistics presented in Table 5, for a 95% confidence interval the difference between the mean angles of wall friction for water versus alcohol addition was sufficient to show distinct behaviour, while the difference between the mean angles of internal friction for water versus alcohol addition was due to random variation. The associated F statistics in both cases indicated that the assumption of

Table 5. Evaluation of differences between water-dosed versus alcohol-dosed flax shive samples based on F statistic and two-sample pooled t statistic.

	Parameter	Internal friction evaluation	Wall friction evaluation
Water-dosed tests	Mean angle of friction	37°	28°
	Standard deviation	1°	1°
	Sample size (*n*)	4	3
Alcohol-dosed tests	Mean angle of friction	36°	23°
	Standard deviation	2°	3°
	Sample size (*n*)	7	5
F-test	F statistic	4.0	9.0
	Confidence interval	95%	95%
	Degrees of freedom	6, 3	4, 2
	Critical F statistic	8.9	19.3
	Analysis result	Null hypothesis accepted	Null hypothesis accepted
	Implication	Population variances the same	Population variances the same
t-test	Difference in mean	1°	5°
	Pooled t statistic	0.9	2.7
	Confidence interval	95%	95%
	Degrees of freedom	9	6
	Critical t statistic	2.3	2.4
	Analysis result	Null hypothesis accepted.	Null hypothesis rejected.
	Implication	Population means the same	Population means different

equivalent population standard deviations was reasonable (Table 5).

It would appear from the results that the angle of wall friction depends on the surface tension of the added liquid, but the angle of internal friction does not. This situation was verified by qualitative observation. Both the alcohol- and the water-dosed samples formed solid plugs in the test cell as a result of compaction. However, while the water-dosed samples tended to cling to the wall, forming a stable bridge that was difficult to remove, the alcohol-dosed samples all tended to slip out easily as a whole plug. This may represent a useful behaviour that could be exploited. Given test results, further investigation of the impacts of adding alcohols on the frictional behaviour of flax shive and other biomass is warranted.

CONCLUSIONS

The shear strength of bulk solid flax shive, as measured by the angle of internal friction, was increased by the addition of any of the three liquids tested, whether methanol, water, or isopropanol. The angle of internal friction increased consistently by about 9° as a result of liquid addition, but with no relationship to which liquid or what quantity of liquid was added over the range tested. Neither the liquid properties of surface tension nor viscosity appeared to have any effect on the angle of internal friction.

The frictional behaviour of flax shive against a standard flat galvanized-metal wall material was measured by the angle of wall friction. Changes in the angle of wall friction were found to be higher by about 5° for the addition of water than for the addition of either of the two alcohols. In this case the surface tension, but not the viscosity, of the added liquid appeared to affect the angle of wall friction.

A clear difference was found to exist between the nature of changes in the angle of wall friction versus the angle of internal friction as different liquids were added. This appeared to occur because the angle of wall friction depended on the surface tension of the liquid, whereas the angle of internal friction did not. It may be possible to usefully exploit this difference in behaviour in the handling of flax shive or other fibrous biomass bulk solids, in particular as part of a process to recover high-value constituents.

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