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Thermodynamic Properties and Mold Appearance on Selected Corn Stover Components

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Abstract. *Thermodynamic properties and mold appearance analysis of major corn stover components such as leaf, stalk skin, and stalk pith were determined from the sorption isotherms data in the temperature range of 10°C to 40°C. Brunauer-Emmet-Teller (BET) monolayer moistures decreased with an increase in temperature. Net isosteric heat of sorption and differential entropy values reduced exponentially with moisture increase and approached the latent heat of vaporization of pure water. Leaf had the highest spreading pressure followed by stalk skin and stalk pith. Spreading pressures increased with increase in water activity and reduced with temperature increase. Net integral enthalpy increased with moisture content to a maximum and decreased afterwards, whereas net integral entropy displayed a reverse trend. Mean values of net integral enthalpy and entropy of stalk pith was the maximum followed by leaf and stalk skin. Thermodynamic properties of corn stover find application in moisture-material interaction, sorption and desorption kinetics, and energy calculations that in turn lead to the development of efficient processing and handling systems. Spoilage status of samples of isotherm experiment was assessed by visual observation of mold growth. Mold affected all stover components at water activity > 0.90. High temperatures were conducive for mold growth and stalk pith was the least resistant to mold growth followed by stalk skin and leaf. Mold free days were predicted using a new three-parameter (temperature (T), water activity (a_w), and $T \times a_w$) model (R^2 = 0.99), with results comparable with existing exponential model (R^2 = 0.95). The advantage of the developed model was to predict the safe storage period of corn stover from the storage environmental conditions.*

Keywords. Corn, Enthalpy, Entropy, Isotherms, Mold, Stover, Thermodynamic, Water activity.

Introduction

Thermodynamic properties of corn stover (a major crop based biomass) aide the quantifications of moisture interaction with this material that in turn lead to development of efficient processing and handling systems. Thermodynamic properties were derived from moisture isotherms provided information about water-material interaction, sorption kinetic parameter, microstructure, and physical phenomenon on material surface (Iglesias et al., 1976; Rizvi and Benado, 1984; Aviara and Ajibola, 2002, Kaya and Kahyaoglu, 2005). Among corn stover components, stalk (72.6%) and leaf (20.7%) dominated stover mass (Igathinathane et al., 2004). Stalk can be further divided into distinct stalk skin and stalk pith components. This division of stalk components was essential because the combine harvesters subject corn stalks to crushing, twisting, and pulling actions and expose stalk pith to surroundings for moisture exchange.

Microbial activity leads to spoilage of biomass, especially when stored under high humidity environments. Storage characteristics based on microbial growth on corn stover components were assessed simultaneously during the sorption isotherm experiments.

Studies on equilibrium moisture relations of biomass materials are highly limited, though several works were reported on food materials. Literature on thermodynamic properties of biomass is even scarcer. To obtain the needed information, a study was formulated with objectives as follows:

1. Determine thermodynamic properties including monolayer moisture content, isosteric heat of sorption, differential entropy, spreading pressure, net integral enthalpy, and net integral entropy for the selected corn stover components.
2. Develop models to predict the mold-free days based on visual observation of mold infestation.

Materials and Methods

Sorption isotherm data (10°C to 40°C) of corn stover leaf, stalk skin, and stalk pith collected earlier (Igathinathane et al., 2005) were utilized to determine various thermodynamic properties and assess storage stability in this study. Appropriately prepared samples were used in static gravimetric method that employed saturated salt solutions in glass desiccators (fig. 1).

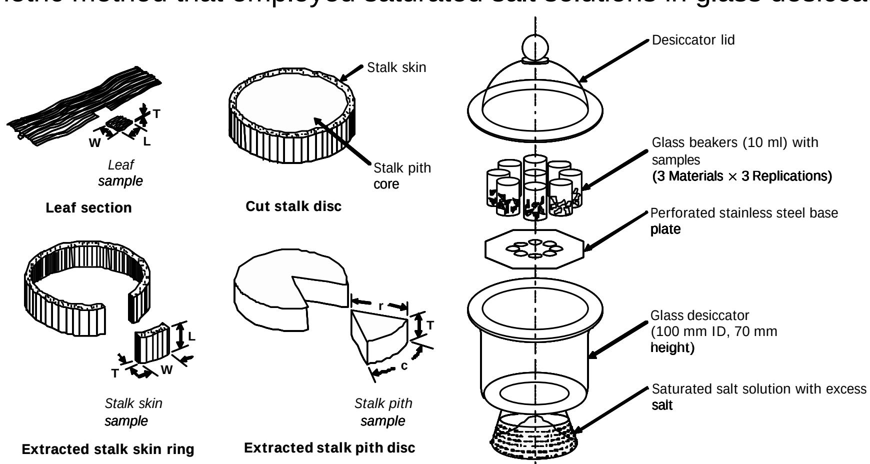

Figure 1. Sample preparation and static method of sorption isotherm data collection for selected corn stover components

Thermodynamic Properties Determination

Monolayer moisture content

Monolayer moisture contents of stover components in dry basis (d.b.) were obtained from standard Brunauer-Emmet-Teller (BET) (Brunauer et al., 1938; Menkov, 2000) and modified BET (Jayas and Mazza, 1993; Menkov, 2000) equations. The modified BET equation incorporating temperature effects is given as:

$$M = \frac{(A + BT)Ca_w}{(1 - a_w)(1 - a_w + Ca_w)} \tag{1}$$

$$M_m = A + BT_c \tag{2}$$

where

M = EMC (%) dry basis (d.b.), A, B, C = model constants, T_c = temperature (°C),
a_w = water activity (decimal), M_m = monolayer moisture content (% d.b.)

Non-linear regression using PROC NLIN of SAS (2002) evaluated the constants (A, B, and C) of equation 1, using the experimental data combining all temperatures at a_w < 0.45. Monolayer moisture values were expressed as a function of temperature (eq. 2) and were compared with fitted BET equation monolayer moistures.

Net isosteric heat of sorption

Net isosteric heat of sorption was determined using Clausius-Clapeyron equation relating the water activities and temperatures at a fixed moisture content (Rizvi, 1986; Bell and Labuza, 2000).

$$\left.\frac{\partial[\ln(a_w)]}{\partial[1/T]}\right|_M = -\frac{q_{st}}{R} \tag{3}$$

where

q_{st} = isosteric heat of sorption (kJ·mol^{-1}), T = temperature (K),
R = universal gas constant (0.00831434 kJ·mol^{-1}·K^{-1}).

This procedure assumes that heat of sorption is independent of temperature change. Slope of plot of $\ln(a_w)$ versus 1/T at constant M gave the net isosteric heat of sorption (eq. 3). Cubic spline interpolation determined the required a_w values at specified moisture (M) at various temperatures from the isotherm data.

Differential entropy

Substituting free Gibbs energy in Gibbs-Helmholtz equation, the differential entropy was calculated (McMinn and Magee, 2003; Kaya and Kahyaoglu, 2005) using the following equation:

$$-\ln(a_w)\big|_M = \frac{-(q_{st} + l)}{RT} - \frac{S_d}{R} \tag{4}$$

where

l = latent heat of vaporization of pure water (kJ·mol^{-1}),
S_d = differential entropy of sorption (J·mol^{-1}·K^{-1}).

From the intercept of plot of $\ln(a_w)$ versus $1/T$ at specified moisture levels, the differential entropy was evaluated (eq. 4).

Spreading pressure

Spreading pressure was evaluated based on the analytical procedure described by Iglesias et al. (1976) and Fasina et al. (1999), as shown in the equation:

$$p = \frac{KT}{A_m} \int_0^{a_w} \frac{M}{M_m a_w} \tag{5}$$

where

p = spreading pressure, (J·m^{-2}), K = Boltzman's constant (1.380 × 10^{-23} J·K^{-1}),
T = temperature (K), A_m = surface area of a water molecule (1.06×10^{-19} m^2).

This integral becomes indeterminate at a_w = 0.0. Therefore, spreading pressure was evaluated by dividing the a_w limits (eq. 5) into two intervals of 0.05 to a_w and 0.0 to 0.05.

Iglesias et al. (1976) used Halsey equation (eq. 6) to fit isotherm data and evaluated the spreading pressure in the interval of 0.05 to a_w (eq. 7).

$$M = \left(\frac{-a}{\ln(a_w)} \right)^{1/r} \tag{6}$$

$$p = \frac{KT}{A_m} a^{\frac{1}{r}} \left[\frac{1}{\left(\frac{1}{r} - 1 \right) \left(-\ln(a_w) \right)^{\frac{1}{r}-1}} \right]_{0.05}^{a_w} \tag{7}$$

where

a, r = Halsey isotherm models constants.

The remainder of the a_w range of 0.0 to 0.05 was evaluated assuming a linear relationship (Henry's law) between M and a_w (Fasina et al., 1999) as:

$$p = \frac{KTM}{A_m M_m} \tag{8}$$

Total spreading pressures were obtained by combining the results (eqs. 7 and 8).

Net integral enthalpy and entropy

Net integral enthalpy was analogous to isosteric heat of sorption. The net integral enthalpy based on Gibbs equation (Hill and Rizvi, 1982) evaluated at constant spreading pressure is expressed as:

$$\left.\frac{\partial[\ln(a_w)]}{\partial[1/T]}\right|_p \approx -\frac{Q_{in}}{R} \tag{9}$$

where

Q_{in} = net integral enthalpy (kJ·mol^{-1}).

Slope from the plot of $\ln(a_w)$ versus $1/T$ at constant spreading pressure (p) produced the net integral enthalpy (eq. 9). For plotting, a_w values at specified p were obtained by interpolation at various temperatures.

Net integral entropy for a thermodynamic system was based on the following relationship (Benado and Rizvi, 1985):

$$S_{in} = \frac{-Q_{in}}{T} - R\ln(a_w^*) \tag{10}$$

where

S_{in} = net integral entropy (J·mol^{-1}·K^{-1}), a_w^* = geometric mean water activity at constant p .

Similar to a_w^*, a geometric mean temperature was used to find the S_{in} (eq. 10).

Mold Free Days Analysis

Day of first appearance of mold in the form of visible network of mycelium (cloudy formation) on the samples, counted from the start of experiment, was recorded. Samples before the day of mold appearance were considered to be free of molds and this period was termed as mold free days (MFD). Numerically, the day of first appearance of mold less one is MFD. To assess MFD of stover components in storage, the mold growth on the samples during sorption experiments was observed. Although mold spore formation as small black dots was observed few days earlier to the mold appearance, those days were not considered in the analysis. Research work of Herrman and Loughin (2003) on corn pelletized feed and Sokhansanj et al. (2003) on alfalfa cubes, proposed an exponential spoilage model for MFD in terms of temperature and water activity. In this study, few models with linear combinations of temperature and water activity along with exponential model were compared.

Results and Discussion

BET Monolayer Moisture Contents

Figure 2 shows a plot of standard BET and modified BET (eq. 1) monolayer moisture contents of corn stover components at different temperatures. In general, leaf was found to have the highest monolayer moisture content followed by stalk skin and stalk pith. The mean standard BET monolayer moisture contents at temperatures of 10°C to 40°C were estimated as 4.87±0.91%, 4.87±0.72%, and 3.74±1.43% d.b. for leaf, stalk skin and stalk pith, respectively. Whereas the initial moisture content of corn leaf, stalk skin, and stalk pith were determined as 8.11%, 12.19%, and 6.09% d.b., respectively. Modified BET monolayer moisture contents as a function of temperature for corn stover components are also given (fig.2). Negative coefficients of temperature indicate reduction in monolayer moisture with temperature increase. Iglesias and Chirife (1976) and Rahman and Labuza (1999) reported after studying several (100 to 142) foods and food components that monolayer values decreased significantly with increasing temperature. This reduction in monolayer moisture content was attributed to the reduction in the number of active sites due to physical and chemical changes induced by the temperature increase (Aviara et al., 2004). Monolayer moisture level is a limit below which rate of quality loss is negligible (Bell and Labuza, 2000).

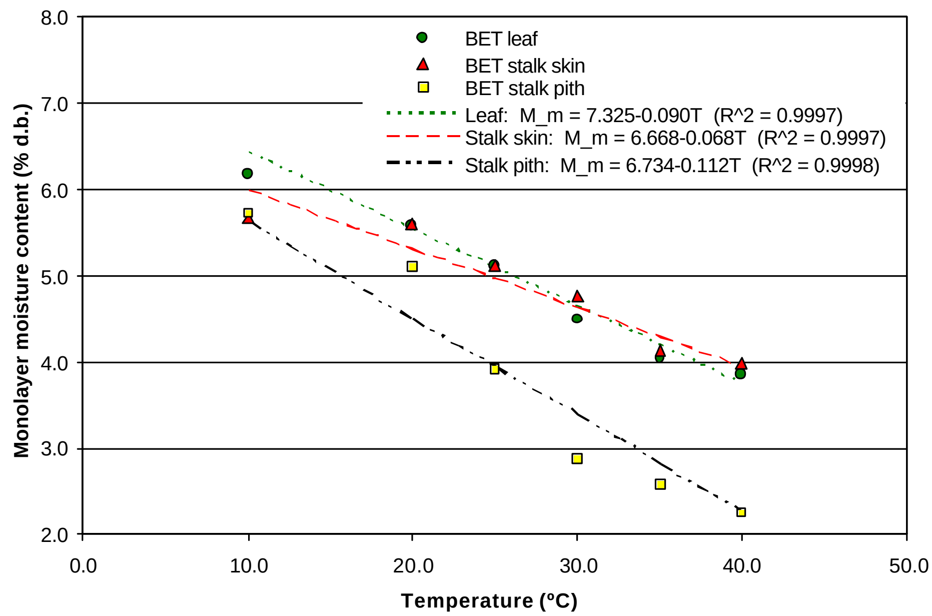

Figure 2. Monolayer moisture content of corn components at different temperatures by standard BET and direct modified BET equations

Net Isosteric Heat of Sorption and Differential Entropy

Calculated net isosteric heat of sorption and differential entropy are plotted in figure 3. Both parameters showed strong dependence on moisture content, with high values at low moisture contents.

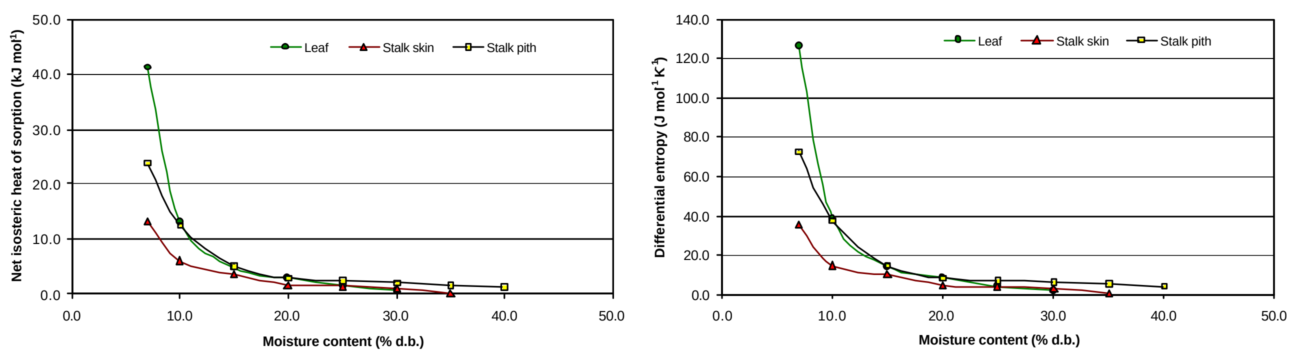

Figure 3. Net isosteric heat of sorption and differential entropy variations of corn stover components with moisture content

High heat of sorption values (in addition to latent heat of vaporization) at low moisture levels indicated high binding energy for removal of water. Increasing moisture content decreased the heat of sorption and the value tends to that of pure water indicating water existence in free form (Aviara and Ajibola, 2002; Kaya and Kahyaoglu, 2005). At low moisture levels (< 10% d.b.) leaf had the highest followed by stalk pith and stalk skin, and at high moisture levels stalk pith had the maximum followed by stalk skin and leaf. Estimated geometrical mean net isosteric heat of sorption for leaf, stalk skin and stalk pith were 2.77, 1.56, and 2.99 $kJ \cdot mol^{-1}$, respectively, in the temperature range of 10°C to 40°C. This shows that lesser energy is required to extract moisture from stalk skin, followed by leaf and stalk pith.

Some of the reported range of heat of sorption values for corn grain ranged from 65 to 3 $kJ \cdot mol^{-1}$ and sorghum 35 to 1 $kJ \cdot mol^{-1}$ (Cenkowski, et al. 1992), pineapple 30 to 5 $kJ \cdot mol^{-1}$ (Hossain, et al., 2001), and quinoa seeds 47 to 4 $kJ \cdot mol^{-1}$ (Tolaba et al., 2004) at various moisture contents. Differential entropy also followed similar variation as net isosteric heat of sorption. The corresponding geometric mean values of differential entropy were 7.79, 4.22, and 8.81 $J \cdot mol^{-1} \cdot K^{-1}$. Heat of sorption and differential entropy values, at specific moisture level, indicated the state of sorbed moisture, thereby providing a measure of physical, chemical, and microbiological stability of the material under specified storage conditions. Relative to the latent heat of vaporization of pure water, magnitude and variation of these values with moisture were useful in understanding the moisture and material interaction, energy consumption, and design of drying equipment (McMinn and Magee, 2003).

Spreading Pressure

Halsey isotherm equation modeled the sorption isotherm data of corn stover components adequately (R^2 > 0.983). In general, among the components leaf had the highest spreading pressure followed by stalk skin and stalk pith at temperatures < 35°C. Overall mean spreading pressures, obtained from the whole data were 0.74±0.33%, 0.71±0.31%, and 0.65±0.38% $J \cdot mol^{-2}$ for leaf, stalk skin, and stalk pith, respectively. Spreading pressure can be viewed as the force in the plane of the surface that must be exerted perpendicular to each unit length of edge to keep the surface from spreading (Smith et al., 2001). To visualize the trend, spreading pressures at temperature limits of 10°C and 40°C of corn stover components against water activities are plotted in figure 4.

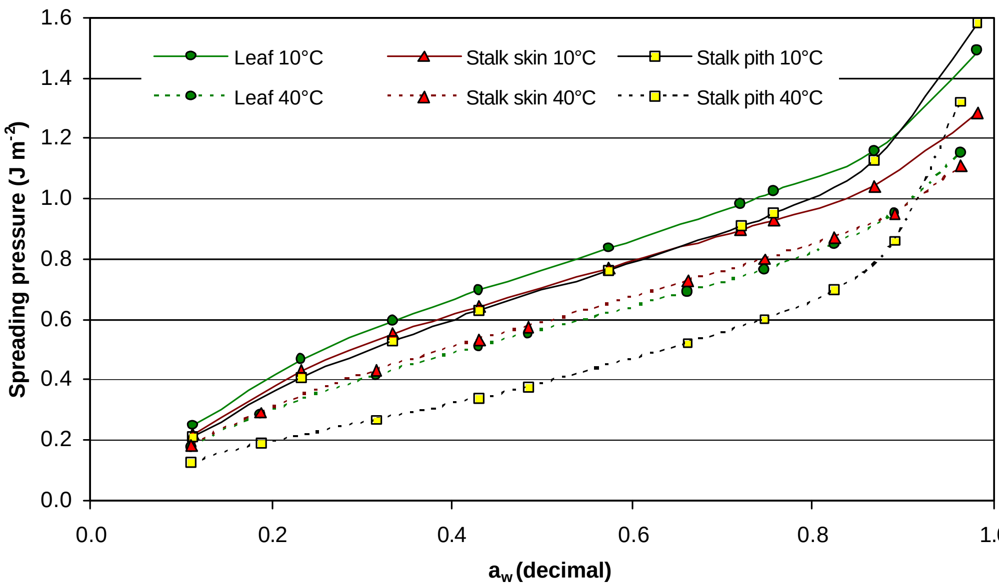

Figure 4. Spreading pressure variation of corn stover components with moisture content

The plot shows that the spreading pressure increased with water activity, and the curves have shapes of type II moisture isotherms. An increase in temperature reduced spreading pressure. The values of spreading pressures, water activities, and trends with temperature are comparable to those reported by Kaya and Kahyaoglu (2005) for pestil (grape leather).

Net Integral Enthalpy and Net Integral Entropy

Figure 5 shows the variations of net integral enthalpy and net integral entropy of corn stover components with moisture content. For all the stover components, net integral enthalpy initially increased as moisture content increased to a level of about 10% to 12% d.b. and decreased with further increase in moisture. Similar trends were reported for enthalpy of melon seed and cassava (Aviara and Ajibola, 2002), potato (McMinn and Magee, 2003), and pestil (Kaya and Kahyaoglu, 2005). Stalk pith registered the highest net integral enthalpy values followed by leaf and stalk skin. The corresponding mean values were 10.30±7.67%, 8.34±3.58%, and 5.58±2.31% kJ·mol^{-1}. The initial increase in enthalpy at low moisture contents was understood as water was absorbed on the most accessible sites on the solids exterior surface. Further increase in moisture made the material to swell and opened up new high-energy sites for water to get bound to, which caused increase in the net integral enthalpy. The increase at low moisture contents continued until binding sites were covered. The net integral enthalpy after reaching a maximum value declined with further increase in moisture levels as less favorable sites were covered and multiple layers of sorbed water formed (Aviara and Ajibola, 2002).

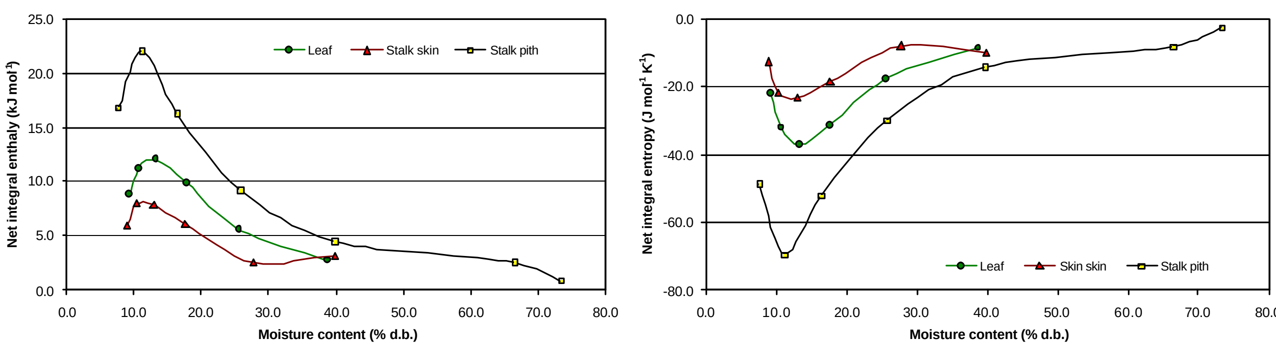

Figure 5. Net integral enthalpy and net integral entropy variations of corn stover components with moisture content

Variation of net integral entropy was found to be mirror image of net enthalpy variation about moisture content. Hence, the net integral entropy variation decreased initially and increased with increase in moisture contents. Estimated means of net integral entropy were -32.36±23.79%, -24.84±10.58%, and -15.83±6.36% J·mol^{-1}·K for stalk pith, leaf, and stalk skin, respectively. Several researchers (Aviara and Ajibola, 2002; McMinn and Magee, 2003; Kaya and Kahyaoglu, 2005) suggested the following explanation for this variation. The decrease of entropy in low moisture range was attributed to increased restriction in the movement (loss of rotational freedom or degree of randomness) of water molecules, as the readily available sites became saturated and the strongest binding sites were utilized. The minimum entropy occurred when the sorbed water became completely localized as the first layer was covered. Subsequent increase of entropy reflected availability of more freely held moisture and formation of multi-layers. At high moisture levels net integral entropy approached the value of free liquid water.

Analysis of MFD

Observed MFD and corresponding conditions are presented in table 1. Mold growth was noticed as early as 10 days, and the growth was sustained in the samples throughout the experimental period. High values of temperature and water activity of the environment were found to be more conducive to mold growth.

Only samples subjected to high water activity (a_w > 0.9) had the mold growth, irrespective of temperatures. Mold growth was noticed at lower a_w values when the temperature was high and vice versa. Therefore, the product of temperature and water activity ($T \times a_w$) would serve as a comprehensive measure to model the spoilage status of material based on mold infestation. The product decreased from 35.60°C to 9.82°C as MFD increased from 10 to 26. Based on this observation, for mold-free long duration storage of the corn stover components, value of $T \times a_w$ product should be kept small within reasonable practical limits.

Table 1. Storage conditions of corn stover components one day after MFD

MFD	T	a_w[a]	$T \times a_w$	Moisture content (% d.b.) of corn stover component one day after MFD		
	(°C)	(decimal)	(°C)	Leaf	Stalk skin	Stalk pith
10	40	0.89	35.60	46.82	43.24	42.63
11	35	0.967	33.85	40.62	38.35	35.79
12	30	0.97	29.10	38.32	35.81	34.43
13	25	0.973	24.33	36.12	34.29	32.86
15	20	0.976	19.52	32.63	30.37	28.88
26	10	0.982	9.82	19.90	18.22	17.79

[a] Minimum a_w at which mold growth was observed; any conditions > a_w also sustained mold growth and at a_w < 0.90 all components did not show any visible mold growth throughout the experimental period

Based on the moisture contents of components (table 1), it takes more moisture in leaf and relatively less moisture in stalk pith at a given value of $T \times a_w$ product for mold appearance. In other words, stalk pith was the least resistant component to mold growth followed by stalk skin and leaf based on the residual moisture in the material. As $T \times a_w$ product increased, mold growth had occurred at low moisture levels.

The developed models to estimate MFD from temperature and water activity are plotted in figure 6 for comparison. Since all samples inside desiccators were found infested with mold around the same period, the developed relationships apply equally to all corn stover components.

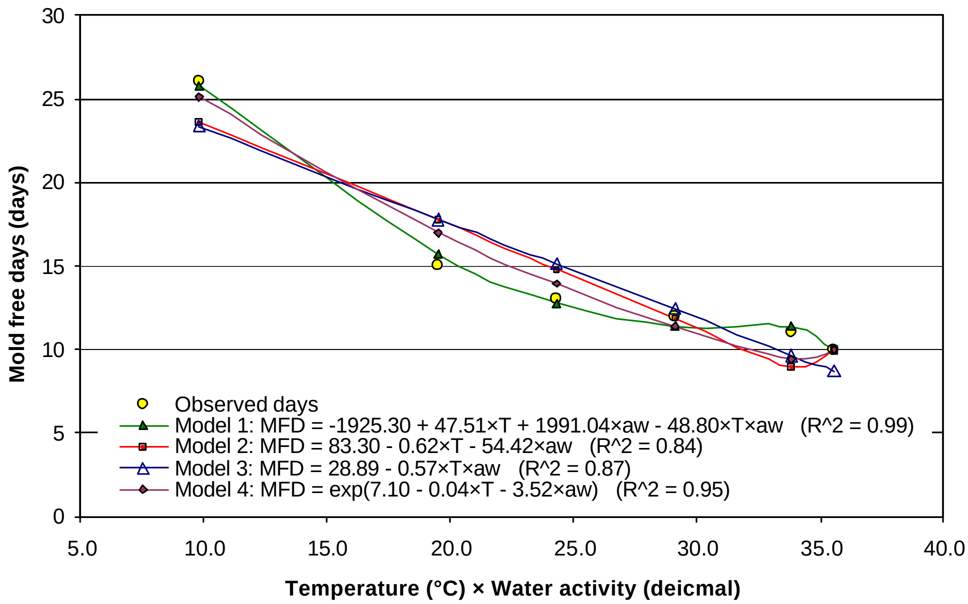

Figure 6. Comparison of observed and predicted mold free days of the corn stover components

The new three-parameter (T, a_w, and $T \times a_w$) model (Model 1; $R^2 = 0.99$) gave the best prediction than the exponential model (Model 4; $R^2 = 0.95$), and the simple two parameter (T and a_w) model (Model 2; $R^2 = 0.84$). The single parameter ($T \times a_w$ product) model (Model 3), though the simplest and had linear variation and produced slightly better results ($R^2 = 0.87$) when compared to Model 2 ($R^2 = 0.84$). The t-statistics revealed that the combined $T \times a_w$ product variable ($t = -5.11$, $p = 0.0069$) was found to be more significant followed by temperature ($t = -4.43$, $p = 0.0114$) and the least significant variable was water activity ($t = 1.15$, $p = 0.3140$) in modeling MFD. MFD models can be used to predict the safe storage period of corn stover from temperature and relative humidity of the storage environments.

Summary

- BET monolayer moisture contents of the corn stover components decreased with increase in temperature, and the estimated mean values were 4.87±0.91%, 4.87±0.72%, and 3.74±1.43 % d.b. for leaf, stalk skin and stalk pith, respectively in the temperature range of 10°C to 40°C.
- Net isosteric heat of sorption reduced exponentially with moisture increase and approached the latent heat of vaporization of pure water. Differential entropy of stover components also followed similar reduction trends. Geometrical means of net isosteric heat of sorption were 2.77, 1.56, and 2.99 $kJ \cdot mol^{-1}$ and differential entropy were 7.79, 4.22, and 8.81 $J \cdot mol^{-1} \cdot K^{-1}$ for leaf, stalk skin and stalk pith, respectively in the temperature range of 10°C to 40°C.
- Halsey isotherm equation modeled the sorption isotherm data of corn stover components adequately ($R^2 > 0.983$).
- Spreading pressures of leaf were the highest followed by stalk skin and stalk pith at temperatures < 35°C, with corresponding overall mean values of 0.74±0.33%, 0.71±0.31%, and 0.65±0.38% $J \cdot mol^{-2}$. Spreading pressures increased with increasing water activity, and decreased with temperature increase.

- Net integral enthalpy increased with moisture content to a maximum and decreased thereafter. Estimated mean values were 10.30±7.67%, 8.34±3.58%, and 5.58±2.31% $kJ \cdot mol^{-1}$ for stalk pith, leaf and stalk skin, respectively.
- Net integral entropy decreased with moisture increase to a minimum and increased thereafter, and displayed a reverse trend with net integral enthalpy. Estimated means of net integral entropy were -32.36±23.79%, -24.84±10.58%, and -15.83±6.36% $J \cdot mol^{-1} \cdot K^{-1}$ for stalk pith, leaf, and stalk skin, respectively.
- All stover components were affected by mold at water activity > 0.90. For a given water activity, high temperatures were found to be conducive to mold growth in the studied temperature range of 10°C to 40°C.
- Stalk pith was the least resistant to mold growth followed by stalk skin and leaf.
- In modeling mold free days on stover components during storage, temperature and water activity product (p = 0.0069) was the most significant variable followed by temperature (p =0.0114) and water activity (p = 0.3140).
- Developed three-parameter multiple-regression MFD model gave the best prediction (R^2 =0.99) and the existing exponential model also produced comparable prediction (R^2 =0.95).

Acknowledgements

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