Determination of Moisture Sorption Isotherm Model for Dried and Fresh Sweet Sorghum [*Sorghum bicolor* (L.) Moench] Stalks

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***Abstract*—Sweet sorghum is rich in sugar and high in biomass which makes it suitable as substrate for bioethanol production. However, the problem with this crop is the short storage duration of the stalk due to high sugar content at high moisture content upon harvest. The study aimed to determine moisture sorption isotherm model for sorghum stalks and investigate the effect of drying on fresh sweet sorghum stalks. According to GAB model, the moisture content at which the microbial activity for the dried stalk becomes limited was computed as 0.24 gram moisture per gram dry mass or lower, considering the water activity at 0.70 where bacterial and fungal growth becomes limited. Moisture content of samples also exhibited changes during storage for both fresh and dried samples. The changes were brought by chemical and microbial activities occurring in the packed samples, respiration in particular, with water as the by-product. Stalks with lower water activity were proliferated by molds and yeasts while stalks with higher water activity were more suitable for bacterial growth.**

***Index Terms*—sweet sorghum, drying, water activity, bioethanol, sugar content**

I. Introduction

Sweet sorghum or *Sorghum bicolor* L. Moenchis considered to be one of the most promising renewable source used as substrate for biofuel production for its high photosynthetic efficiency, high biomass and sugar yields, low Nitrogen fertilizer and irrigation requirement, wide adaptability, and tolerance to drought and salinity [1]. Moreover, the stalks contains ample amount of fermentable sugars, cellulose and hemicellulose as well as the grains is rich in starch. This makes it as favourable as raw material for both first and second generation biofuels because all of the contents that can be utilized for ethanol processing including its bagasse. Lignocellulose materials will be used as feedstock for ethanol production in the very near future. Various studies have been recently carried out on the energy potential of sweet sorghum and recognized the crop as one of the most potential feedstocks for biofuels [1].

The primary disadvantages of using sweet sorghum for ethanol production are the seasonal availability and short storage duration of the stalk [2]. Especially, storage must be initiated immediately after harvest, because the stalk or juice is rich in soluble sugar and can be easily deteriorated in natural conditions. Drying offers practical and effective solution for stalk storage. There were studies on drying of chopped sweet sorghum stalk using open-air sun conditions and forced drying to meet the need of emergency from the unstable [2]. About four months of successful storage system is necessary to provide the amount of raw material during off-season thus keeping the plant in operation during such time. The growing season for sweet sorghum is from February to October when daytime is longer than night time since this plant is photoperiodic.

Reference [3] and [4] reviewed moisture sorption isotherm as mathematical relationship between Equilibrium Moisture Content (EMC) and water activity at constant equilibration and temperature. To define stability of a feedstock, water activity (aw) is commonly used base on the equilibrium moisture content and equilibrium relative humidity. A study using a cellulosic energy variety of *Sorghum bicolor* (L.) Moench, the predicted moisture content to limit microbial activity ranged from 0.16 to 0.19 gram moisture per gram dry mass for initial drying process and 0.12 to 0.14 gram moisture per gram dry mass for adsorption process across 40 to 15 ℃ [5]. That's why for stable long-term storage of cellulosic energy variety of sorghum, in-field or mechanical drying should be done before storing the biomass to avoid degradation and dry matter loss [5].

Reference [6] discussed that hermetic storage is an ancient way of storing grains in clay pots, underground pits of mudplastered structures. Hermetic conditions create a modified atmosphere high in carbon dioxide and low in oxygen thus controlling infestation.

The study aimed to determine moisture sorption isotherm of sweet sorghum stalks. This study further

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evaluated hermetic and conventional packaging of dried sweet sorghum stalks for changes in water activity during storage.

II. Materials and Methods

A. Preparation of Materials

SPV 422 variety of sweet sorghum grown at Nasugbu, Batangas was used as raw material for this study. It was planted last May 14, 2015 and harvested on September 22, 2015. The harvested stalks were transported to Agricultural and Bioprocess Division, Institute of Agricultural Engineering, University of the Philippines, Los Baños, Laguna.

B. Moisture Sorption Isotherm

Moisture sorption isotherms of fresh and dried powder stalk samples were taken using static method. Equilibrium Moisture Contents of the stalks were obtained using sealed mason. The initial moisture contents of the samples were also determined. Saturated salt solutions were used to generate constant relative humidity values at room temperature in Table I were utilized. The containers were closed and the setup was placed in ambient conditions. For two weeks, weights of the samples were monitored until they reached constant values. Data were gathered, computed and plotted. Several models of sorption isotherms were used including B.E.T. (Brunauer-Emmet-Teller), G.A.B. (Guggenheim-Anderson-De Boer), and Oswin equations. Goodness of fit was also determined considering R-squared values.

TABLE I. Salts Utilized for Moisture Sorption Isotherm

SALTS	WATER ACTIVITY
Sodium hydroxide	0.08
Lithium chloride	0.11
Potassium acetate	0.23
Potassium carbonate	0.42
Sodium chloride	0.75
Ammonium sulfate	0.79
Potassium chloride	0.84
Potassium sulfate	0.97

Source: Smith, P.R. (1971); Greenspan L. (1976)

C. Analysis of Sweet Sorghum Stalks

Some samples were dried at 60 ℃ as suggested until moisture content of 14%, wet basis as suggested in reference [2]. The dried and fresh samples in chips, 5-cm section and 10-cm cut were stored in sacks and hermetic storage for 16 weeks. For hermetic storage system, GrainPro Superbags™ was used for some samples. For the storage conditions, some samples were placed in room temperature and others was kept frozen using chest-type freezers. Stalk samples were also analysed for moisture content using oven method.

Dried samples in sacks and hermetic bags at ambient condition were evaluated for Total Bacterial Count (TBC) and Total Yeast and Mold Count (TYMC) for 16 weeks of storage. Water activity was also computed for dried samples at ambient during the storage period.

The sampling periods were 0, 1, 2, 4, 6, 10 and 16 weeks of storage.

III. Results and Discussions

A. Moisture Sorption Isotherm

The adsorption and desorption curve as shown in the Fig. 1 follow sigmoid shape of a type II isotherm which is usually describing porous materials and other agricultural products which is the same as the sigmoid curve observed using energy sorghum variety according to the experiment conducted [5]. Fresh sample with initial moisture of 0.91 gram moisture per gram dry mass exhibited moisture desorption. Dried sample initially at 0.16 gram moisture per gram dry mass adsorbed and desorbed moisture.

Among the models, GAB (Guggenheim-Anderson-de Boer) equation has the best fit with R2 equal to 0.9826 and 0.9997 for both fresh and dried samples, respectively. The curve using the GAB equation almost followed the curve according to the data plotted from the experiment as observed in Fig. 1. Models from different equations are shown in Tables II and Table III with their respective R^2. GAB model was used for plotting equilibrium moisture content (% dry basis) with reference to water activity as shown in Fig. 1. Data taken from the experiment were also plotted in the Fig. 1.

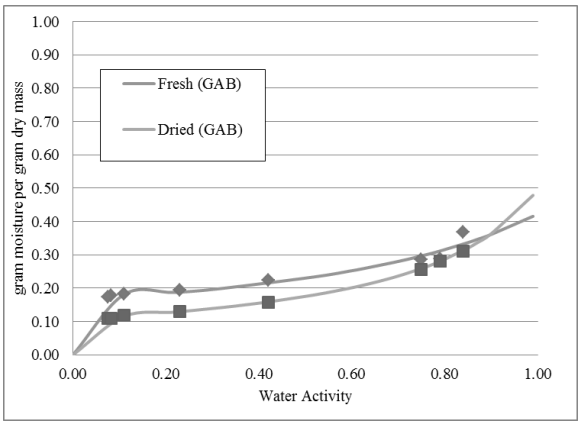

Figure 1. Moisture sorption isotherm of sweet sorghum stalks from experiment and GAB equation

GAB model is a refinement of Langmuir and BET theories of physical adsorption. The model postulates that the state of sorbate molecules in the second layer is identical to the one in superior layers but different from those of the liquid state. The constants in GAB model are the adsorption constants which are related to the energies of interaction between the first and the further sorbed molecules at the individual sorption sites [4].

TABLE II. MOISTURE SORPTION ISOTHERM MODELS FOR FRESH SWEET SORGHUM STALKS

MODEL	EQUATION	R^2
G.A.B.	$M = \frac{-1639.3439 a_w}{(1-0.6316 a_w)(1-106.1914\, a_w)}$	0.9826
Oswin	$M = 24.9157 \left[\frac{a_w}{1-a_w}\right]^{0.1540}$	0.9387

$a_w - wateractivity$
$M - moisturecontent, \%\ drybasis$

TABLE III. MOISTURE SORPTION ISOTHERM MODELS FOR DRIED SWEET SORGHUM STALKS

MODEL	EQUATION	R^2
G.A.B.	$M = \frac{8064.5157 a_w}{(1-0.7851 a_w)(1+755.0271 a_w)}$	0.9997
Oswin	$M = 19.4180 \left[\frac{a_w}{1-a_w}\right]^{0.2480}$	0.9827

$a_w - wateractivity$
$M - moisturecontent, \%\ drybasis$

Taking the data gathered from the experiment using sweet sorghum variety, the monolayer values for fresh sample is equal to 0.13 gram moisture per gram dry mass while 0.09 gram moisture per dry mass for dried samples employing BET model.

Using GAB model for predicting moisture content at 0.70 water activity at which the growth of microorganisms becomes limited, the dried sweet sorghum sample can be safely stored when the moisture content is 0.24 gram moisture per gram dry mass or lower. As compared to reference [5] using energy sorghum variety, the range at which microbial activity becomes limited were found to range from 0.12 to 0.18 gram moisture per gram dry mass which is observed to be lower than that of sweet sorghum variety utilized in this experiment. The difference in equilibrium moisture content at 0.70 aw is due to the high sugar content of the sweet sorghum variety. Dried material stored or placed in a condition might encounter readsorption of moisture due to higher relative humidity in the surroundings. Maintaining the moisture content of the dried samples at safe storage conditions could be done by proper packaging or temperature control.

B. Moisture Content

The changes in moisture of the stalks were plotted as shown in Fig. 2. The maximum moisture change for dried frozen 5-cm samples in hermetic bags was equal to 21.19% after 4 weeks of storage from the initial 14.62% moisture all on wet basis with a difference of 6.57%. For hermetically packed samples on ambient conditions, the 10-cm cut dried samples reached 22.08% from an initial of 13.10% moisture on wet basis with an increase of 8.98% after 10 weeks of storage. For dried samples in sacks at freezer, an increase of moisture content equal to 5.35% at maximum was observed from 10-cm cut dried stalks from 15.99% to 21.34% moisture content wet basis after 10 weeks of storage. Also, an increase of 13.10% in moisture content was observed from an initially 14.83% to 27.93% wet basis after 10 weeks of storing the dried samples in sacks at ambient condition. For frozen dried samples, changes in moisture content were still evident due to the changes in temperature and relative humidity of the freezer used. Changes in moisture content were higher on dried samples at ambient conditions than in freezer, for both stalks packed in sacks and hermetic bags, since ambient conditions were unstable.

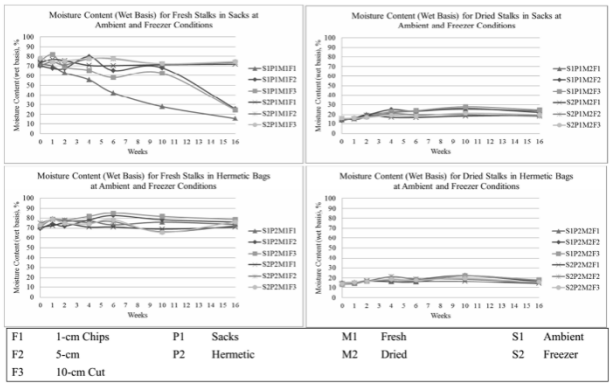

Figure 2. Moisture sorption isotherm of sweet sorghum stalks from experiment and GAB equation

Maximum observed moisture contents of samples were taken after 10 weeks of storage due to the flooding in the place where the samples were stored causing rise in the relative humidity of the surroundings.

For frozen fresh samples, increasing and decreasing moisture contents were observed for both stalks placed on sacks and hermetic bags due to the changes in the temperature and relative humidity inside the freezer. Maximum changes were seen from frozen hermetically stored 5-cm stalks with a decrease of 8.47% after 10 weeks of storage from an initial moisture of 74.99% to 66.52% wet basis, while maximum decrease for frozen 10-cm cut samples in sacks is equal to 5.61% after 10-week storage from 78.01% to 72.40% wet basis. The largest decrease of 56.39% in moisture content was also observed in fresh chip samples in sacks at ambient condition from 72.24% to 15.85% moisture content wet basis after 16 weeks of storage. The decrease in moisture of stalks was attributed to the lower relative humidity of the surroundings at 81.32% taking into account the very high moisture content of fresh samples ranging from 70% to 78% wet basis. However, for fresh samples stored in hermetic bags at ambient condition, an increase in moisture content was noticed with 10-cm cut fresh samples having the maximum increase of 13.89% after 6 weeks of storage from initial moisture content of 71.37% to 85.27% wet basis. The increase in moisture content of the hermetically packed fresh samples at ambient condition could be attributed to the respiration of the microorganisms with carbon dioxide and water as products. Changes in the moisture content of fresh samples in hermetic bags at ambient condition could also be the result of anaerobic processes which may exist within the hermetically packed samples which might include acetogenesis with water as the product of reactions [7] and [8].

C. Microbial Analysis

Dried samples in sacks and hermetic bags at ambient condition were evaluated for Total Bacterial Count (TBC) and Total Yeast and Mold Count (TYMC) for 16 weeks of storage. Water activity was also computed for dried samples at ambient during the storage period. Partial vapour pressure of samples and ambient condition were also computed as shownin Fig. 3.

Rise in vapour pressure of the dried samples could be attributed to change in water activity due to increase in moisture content from respiration. The vapour pressures of dried samples in sacks increased from initial up to 4 weeks of storage and started to decline and equilibrated with vapour pressure of the surroundings. GrainPro®SuperGrain™bag acted as barrier between the surroundings and the sample which resulted in lower vapour pressure of the hermetically packed dried samples. From Fig. 3, microbial analysis of the dried samples revealed the presence of microorganisms contributing to biological and chemical activities in the stalk samples. The results of microbial analysis of the dried samples in sacks and hermetic bags stored at ambient conditions are shown in Fig. 3. Frozen samples were assumed to have very little activity. Recorded freezer temperature ranged from -10 to -24 ℃elsius.

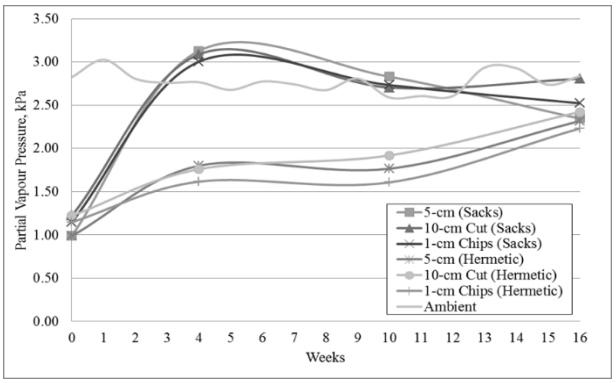

Figure 3. Computed partial vapour pressure at ambient conditions with corresponding vapour pressure of dried samples

From initial results, the population count showed presence of bacteria, yeasts and molds after the drying process. The temperature for drying utilized was 60 ℃ which was not enough to eradicate microorganisms present in the samples.

During the 4th week of storage, TBC increased for chips and 5-cm samples but decreased count in 10-cm cut samples packed in sacks with increase in TYMC for all these samples. The computed water activity for samples in sacks at ambient condition ranged from 0.90 to 0.93 which was suitable for bacteria, molds and yeasts. For hermetically packed dried samples, average increase in TBC for all samples at ambient condition was observed from 3.42 to 4.07 log of colony forming units per gram of sample. Increase in average TYMC was shown in 5-cm from and 10-cm cut samples at 1.30 to 1.51 log of colony forming units per gram of stalks while there was a decrease in count for chip samples from 1.43 to 0.18 log of colony forming units per gram of sample. Water activity for dried samples in hermetic bags at ambient conditions had lower computed values at 0.48 to 0.54 where microorganisms were almost inactive.

For 10 weeks of storage, samples in sacks at ambient condition exhibited decrease in TBC for all samples accompanied by increase in TYMC. Water activity computed ranged from 0.82 to 0.86 which was much suitable for molds and some strains of yeasts, but not for most bacteria. For hermetically packed samples, increase in TBC for 5-cm was noticed but decrease in chips and 10-cm cut samples while TYMC resulted in increased count for all samples. Computed values for water activity for hermetically packed samples at ambient condition ranged from 0.49 to 0.58 which was suitable for molds and few yeast strains but not for bacteria. Water activity for hermetically stored samples at ambient condition was lower compared to stalks in sacks. The molds and yeasts population were much lower for samples in hermetic storage.

During the 16 weeks of storage, samples in sacks at ambient condition resulted in decreased TBC and TYMC due to also decreased in water activity. The computed water activity was equal to 0.67 to 0.80 where most strains of molds and few strains of yeasts could survive, but not suitable for most bacteria. For hermetically packed samples, TBC and TYMC decreased with computed water activity at 0.64 to 0.69 which was suitable for few strains of molds and yeasts but not for most bacteria.

For fresh samples stored in sacks and hermetic bags at ambient conditions, TBC and TYMC were analysed after 4 weeks of storage. From the Fig. 4, the population of the microorganisms were higher for fresh samples because of its high-water activity. For samples in sacks, there was an increase for both TBC and TYMC. While for samples in hermetic bags, decrease in TBC for all samples and TYMC chips and 10-cm cut stalks were observed, but there was an increase in TYMC for 5-cm samples. The oxygen and carbon dioxide levels were to be considered for these differences in which the microorganisms that could thrive for aerobic and anaerobic conditions were not similar.

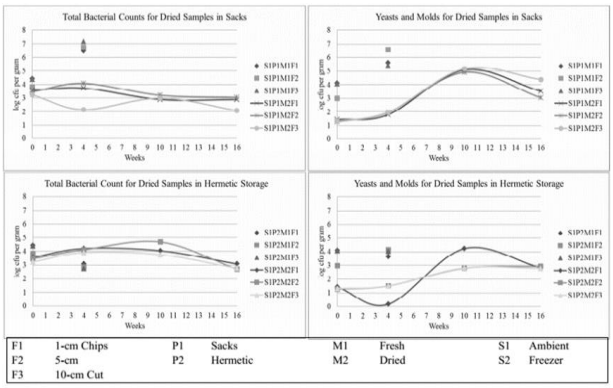

Figure 4. Total microbial count for sweet sorghum stalk samples

IV. Summary and Conclusion

The study utilized sweet sorghum stalk samples. Effect of drying method on stalks was investigated through

color analysis. Water activities of the samples were also determined to identify the critical moisture content for safe storage of the samples.

Among the isotherm models, GAB (Guggenheim-Anderson-de Boer) equation was utilized since it had the best fit with R2 equal to 0.9997 and 0.9823 for both dried and fresh samples respectively, as well as considering the model's theoretical bases and mathematical simplicity. Using GAB model for predicting moisture content at 0.70 water activity at which the growth of microorganisms becomes limited, the dried sweet sorghum sample can be safely stored when the moisture content is reduced to 0.24 gram moisture per gram dry mass or lower.

Moisture content of samples also exhibited changes during storage for both fresh and dried samples. The changes were brought by chemical and microbial activities occurring in the packed samples, respiration in particular, with water as the by-product. These changes in moisture content especially from samples stored in sacks at ambient conditions were caused by changes in relative humidity of the surroundings. Changes in moisture contents of samples in freezer were caused by the unstable temperature and relative humidity inside the freezer.

Microbial analysis revealed that water activity was an important factor showing the type of microorganisms present in the stalk samples. Changes in water activity of samples were accompanied by changes in moisture content of samples. Stalks with lower water activity were proliferated by molds and yeasts while stalks with higher water activity were more suitable for bacterial growth.

V. Recommendations

It is recommended for future research that test should be conducted on formation of inhibiting compounds and evaluation on changes in sucrose, glucose and fructose content during drying process.

Further studies should also include optimization on agitated sugar extraction considering speed of rotation, temperature, sample loading, dilution factor and time of extraction.

Research should also be conducted on the design, fabrication and evaluation of chipping or cutting machine for sweet sorghum stalks and same materials with hard rind and succulent pith.

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