## Determination of drying behaviour and ethanol production from fresh and dried sweet sorghum stalks

# Ocreto, M. B.1\*, Elepaño, A. R.2 and Yaptenco, K. F.2

<sup>1</sup>Department of Engineering Science (DES), College of Engineering and Agro-Industrial Technology (CEAT), University of the Philippines Los Baños (UPLB), 4031 College, Laguna, Philippines; <sup>2</sup>Agricultural and Bio-Processing Division (ABPROD), Institute of Agricultural Engineering, College of Engineering and Agro-Industrial Technology (CEAT), University of the Philippines Los Baños (UPLB), Philippines.

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**Abstract** Sweet sorghum, with high photosynthetic efficiency, tolerant to drought and salinity, is rich in sugar and high in biomass which makes it suitable for bioethanol production. However, sweet sorghum is not available throughout the year and stalk can be stored for short period due to high sugar and high moisture content upon harvest. Drying process is known to prolong shelf life of commodity. The drying behaviour of sweet sorghum stalks was determined and ethanol production from sweet sorghum stalks was evaluated. In determining the drying behaviour of the stalks, the predicted shortest drying time using Page equation was 7.99 hours with samples cut into 1-cm chips. The maximum ethanol production was recovered from fresh 10-cm cut samples with cellulase at 221.92 ml per kg dry mass while 168.24 ml of ethanol per kg dry mass was recovered from dried 10-cm stalks. Employing dried and fresh stalk samples for fermentation was proven to be feasible. The fermentation process can be further improved by sugar extraction through agitation and addition of cellulase.

Keywords: Bioethanol; Drying; Fermentation; Sugar; Sweet sorghum

#### Introduction

Sweet sorghum or *Sorghum bicolor* L. Moench as described by Zhao *et al.* (2012) is considered to be one of the most promising renewable source for biofuel production because of its high photosynthetic efficiency, high biomass and sugar yields, low Nitrogen fertilizer and irrigation requirement, wide adaptability, and tolerance to drought and salinity. Moreover, the stalks contain ample amount of fermentable sugars, cellulose and hemicellulose.

The sweet sorghum grains are also rich in starch. Thus making sweet sorghum a favourable substrate for both first and second generations of biofuels since all its contents can be possibly utilized for ethanol processing.

<sup>\*</sup> Corresponding Author: Ocreto, M. B.; Email: melanieocreto@gmail.com

Lignocellulosic materials will be used as feedstock for ethanol production in the near future. Various studies have been recently carried out to enhance the energy potential of sweet sorghum for biofuels (Zhao *et al.*, 2012).

Bioethanol producing industries in the Philippines use starch sources such as cassava and corn as well as sugar cane molasses as substrates. However, these commodities are consumed as food which imposes competition against the basic need of man for nourishment. Reddy *et al.* (2011) stated that sweet sorghum in the Philippines opens opportunities since its temperature and relative humidity is suitable for its cultivation. Sweet sorghum provides grains for food or feed and also offers alternative substrate for ethanol production because of its sugar-rich stalks.

Shen et al. (2011) stated that the primary disadvantages of using sweet sorghum for ethanol production are its seasonal availability and short shelf life of the stalk. Storage must be initiated immediately after harvest, because the stalk or juice can easily deteriorate in natural conditions. Drying offers practical and effective solution for stalk storage. There were studies conducted on drying chopped sweet sorghum stalks using sun drying and forced drying for emergency cases because of unstable weather (Shen et al., 2011). A study was conducted by Mercer et al. (2011) to investigate the drying kinetics of energy sorghum. The thick stalk of sorghum with its high moisture content upon harvest limits the moisture removal causing drying times to be lengthy and prolonging exposure to environmental conditions. Lowering of moisture content of the freshly harvested stalk in a shorter period of time would result in reduction of sugar loss during storage (Mercer et al., 2011). About four months of successful storage is necessary to provide the amount of raw material during off-season to keep the plant in operation. The growing season for sweet sorghum is from February to October when daytime is longer than night time. Sweet sorghum is a photoperiodic plant.

However, there are no studies conducted on drying sweet sorghum chips, 5-cm cut stalks and split 10-cm stalks. There were studies on drying with sorghum internodes and cut internodes using energy sorghum variety. Studies on drying kinetics were already conducted on crushed and ground sweet sorghum.

The study aimed to determine the drying behaviour of sweet sorghum stalks of various sizes using heated air at ambient conditions and required drying time to reach the moisture content for safe storage and ethanol production from dried and fresh sweet sorghum stalks.

#### **Materials and Methods**

## 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. Fresh samples were checked for sugar content in degree Brix using refractometer. A typical Brix measure for sweet sorghum sap is 85% sugar and 15% soluble starch (Wortmann and Regassa, 2011).

Three sizes of stalks were considered in the study which can be handled and stored conveniently. Grinder equipped with cutting blade was used for cutting the stalks in desired forms. These sizes were 1-cm chips, 5-cm stalk section and 10-cm section cut longitudinally.

## Drying time and drying behaviour of sweet sorghum stalks

The sample was dried at 60 degrees Celsius drying temperature, apparent air velocity of 2.91 meters per second and 14% relative humidity using a mechanical dryer. Conditions of the drying air were taken from the psychrometric chart using ambient conditions recorded during the experiment at 27°C and 75% relative humidity. Samples were weighed every 15 minutes for the first two hours of drying and every 30 minutes thereafter until weight was constant, without taking out the samples from the dryer. Weighing was done through a digital weighing scale attached to the tray containing the samples through a wire.

Drying models were employed for each configuration which included Newton, Page, Henderson-Pabis and Wang-Singh equations. Goodness of fit was computed using R-squared (R<sup>2</sup>), Root Mean Square Error (RMSE), Chi-Square and Mean Relative Deviation Modulus (E). Drying behaviour of each stalk configuration was considered by monitoring the changes in moisture content of the samples over a period of time.

### **Fermentation**

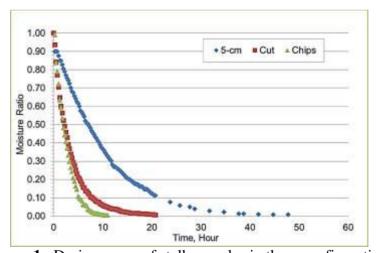
Dried and fresh samples were tested for fermentation using instant yeast. The first method was fermentation using the sweet sorghum juice. The juice was extracted from the stalks prior to fermentation. The juice was evaporated up to 26 degrees Brix for fermentation. Sugar extraction from fresh and dried

sweet sorghum stalks was also accomplished, prior to fermentation, using a shaker at ambient condition for 7 hours, with 1:4 ratio (w/w) of solid stalks to distilled water. Another method employed for fermentation was using fresh and dried powdered samples of sweet sorghum stalks. Fermentation was set up for cut stalks of fresh and dried samples using various sizes including 1-cm chips, 5-cm and 10-cm. Fermentation with cellulase enzyme was also conducted. Cellulase from BIOTECH-UPLB at 25 FPU was utilized per gram dry mass to convert stalk cellulose to fermentable sugars. Dried samples were utilized as substrates without extracting the juice from the stalk. Additives and distilled water were added to the substrates to facilitate the fermentation process. Ethanol concentration was analysed using gas chromatograph.

#### Results

## Drying behaviour of sweet sorghum stalk

Drying curve for each sizes were plotted in Figure 1 with changes in gram per moisture per gram dry mass of the sweet sorghum stalk samples. The drying was terminated until the equilibrium moisture content (EMC) of the samples was obtained.



**Figure 1.** Drying curve of stalk samples in three configurations

The average EMC (Equilibrium Moisture Content) for the sweet sorghum stalk samples was equal to 0.12 gram moisture per gram dry mass from initial 2.33 gram moisture per gram dry mass. For 5-cm samples, EMC was obtained after 50.75 hours, for 10-cm cut stalks EMC was achieved after 31.75 hours while for 1-cm chips EMC was attained after 12 hours of drying.

Drying equations are shown in Table 1. From computed R<sup>2</sup>, Chi-Square, RMSE and E (%), Newton's equation in Table 2 shows the best fit for 5-cm samples while Page equation indicates the best fit for both 10-cm cut and 1-cm chip samples. Predicted values at gram moisture per gram dry matter using Page equation and data gathered from the experiment are shown in Figure 2.

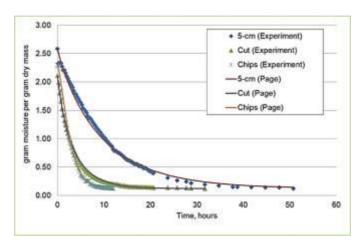


Figure 2. Moisture content g per gram dry mass employing Page Equation

**Table 1.** Model equations for drying curves of sweet sorghum stalk samples

MODEL	FORMS	EQUATIONS	PREVIOUS STUDIES
Newton	1-cm Chips	$MR = e^{-0.4868 t}$	Shen et al., 2011
	5-cm	$MR = e^{-0.1074 t}$	
	10-cm Cut	$MR = e^{-0.2471 t}$	
Page	1-cm Chips	$MR = e^{-0.2439 t^{1.3396}}$	Shen et al., 2011;
	5-cm	$MR = e^{-0.1293 t^{0.9132}}$	Mercer et al., 2011
	10-cm Cut	$MR = e^{-0.3439 t^{0.8952}}$	
Henderson-	1-cm Chips	$MR = 1.4888 e^{-0.5371 t}$	Shen et al., 2011
Pabis	5-cm	$MR = 1.0733 e^{-0.1108 t}$	
	10-cm Cut	$MR = 0.6332 e^{-0.9701 t}$	
Wang-Singh	1-cm Chips	$MR = 0.9129 - 0.2159 t + 0.0124 t^2$	Shen et al., 2011
	5-cm	$MR = 0.8823 - 0.0567 t + 0.0009 t^2$	
	10-cm Cut	$MR = 0.6685 - 0.0803 t + 0.0023 t^2$	

MR (Moisture Ratio) = 
$$\frac{M - M_e}{M_o - M_e}$$

M = moisture content at time t, % dry basis

 $\mathrm{M_{e}} = \mathrm{equilibrium}$  moisture content, % dry basis

 $M_0$  = initial moisture content, % dry basis

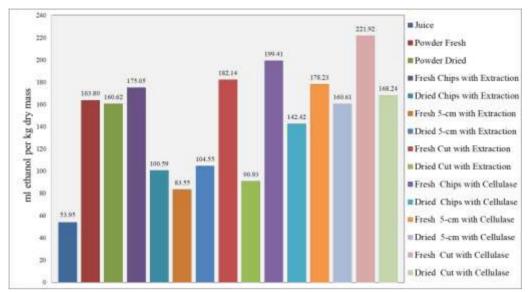
t = time, hours

**Table 2.** Statistical parameter for goodness of fit of drying equations

	STATISTICAL	FORMS		<del>, , , , , , , , , , , , , , , , , , , </del>	
MODEL		1-cm	5-cm	10-cm	AVERAGE
	PARAMATERS	Chips		Cut	
Newton	R squared	0.9786	0.9946	0.9448	0.9727
	RMSE	0.1251	0.0495	0.0869	0.0872
	Chi-square	0.0160	0.0025	0.0076	0.0087
	%E	10.9675	4.2972	12.9218	9.3955
Page	R squared	0.9544	0.9726	0.9927	0.9732
	RMSE	0.0586	0.0575	0.0181	0.0447
	Chi-square	0.0036	0.0034	0.0003	0.0024
	%E	3.9294	6.1016	3.6065	4.5458
Henderson & Pabis	R squared	0.9903	0.9961	0.9701	0.9855
	RMSE	0.2258	0.0743	0.3848	0.2283
	Chi-square	0.0532	0.0056	0.1515	0.0701
	%E	8.3525	3.5483	43.2997	18.4002
Wang & Singh	R squared	0.9778	0.9799	0.8578	0.9385
	RMSE	0.0966	0.1304	0.1963	0.1411
	Chi square	0.0099	0.0175	0.0399	0.0224
	%E	22.1938	9.2861	35.2201	22.2334

## **Fermentation**

Fermentation was carried out to test ethanol recovery of using fresh and dried stalk samples. Extracted juice was sterilized and evaporated up to 26 degrees Brix for fermentation. Various sample sizes were utilized for fermentation including powder, chips, 5-cm and split stalks. Some samples were agitated for sugar extraction prior to fermentation. Cellulase enzyme was also employed on some fermentation set-up. From Figure 3, highest ethanol recovery was obtained from fresh 10-cm cut sweet sorghum stalks with cellulase at 221.92 ml per kg dry mass, followed by fresh chips with cellulase at 199.41 ml of ethanol per kg dry mass, and 10-cm cut samples with sugar extraction at 182.14 ml per kg dry mass. Minimum ethanol recovered was from juice fermentation at 53.95 ml of ethanol per kg dry mass. When using sweet sorghum juice, large volume of stalks is necessary for juice extraction.



**Figure 3.** Ethanol production from various substrates during fermentation

For dried stalk samples, maximum amount of ethanol was recovered from 10-cm cut samples with cellulase at 168.24 ml per kg dry mass, followed by dried powder stalks at 160.62 ml per kg dry mass, and 5-cm with cellulase at 142.42 ml per kg dry mass. Minimum amount of ethanol was produced from dried 10-cm cut stalks with sugar extraction at 90.93 ml per kg dry mass. The 10-cm cut samples during sugar extraction were not completely submerged during the agitation and fermentation process.

#### **Discussion**

The study utilized sweet sorghum stalks which were cut into three sizes including 1-cm chips, 5-cm section and 10-cm split section. Drying behaviour was determined for these samples using drying model equations. Sugar extraction by agitation and fermentation were employed to evaluate ethanol recovery.

Drying behaviour was determined through thin layer drying at 60 degrees Celsius drying temperature, 14% relative humidity and 2.91 meters per second air velocity. Drying at 60 degrees Celsius or higher reduced growth of most microorganisms and prevented enzymatic browning from an experiment conducted by Shen *et al.* (2011). However, higher drying temperature increases the risk of non-enzymatic browning and produces fermentation inhibitors which could result in reduction of ethanol yield. According to Sutton (2011), major classes of inhibitors include sugar degradation products, weak acids, and

phenolic compounds. High concentrations of these inhibitors negatively affect ethanol production (Sutton, 2011). Shen *et al.* (2011) further suggested that optimum drying temperature for sweet sorghum is 50 to 60 degrees Celsius.

Stalks cut into chips had more surface area exposed to drying air thus resulted in shortest drying period. Split samples at 10-cm length dried faster than the 5-cm section since the outer rind of the stalk was removed. The hard covering rind of the stalk protects the plant from moisture loss. A study conducted by Mercer *et al.* (2011) utilized three configurations of energy sorghum including 20 cm long sections with sealed ends to duplicate stalks of infinite length, 20 cm section with open ends and 20 cm sections with longitudinal splitting. Mercer *et al.* (2011) used 50 degrees Celsius and 0.5 meter per second air velocity resulting in drying period ranging from 200 hours with sealed ends to 15 hours with split stalks to reach the final moisture at 10% (wet basis).

Page Equation as drying model was considered due to goodness of fit with an average values of R<sup>2</sup> at 0.9732, RMSE of 0.0447, Chi-square of 0.0024 and %E of 4.5458. Page model is a modified Newton's equation which is based on Fick's law of diffusion. Page model is introduced to provide better representation of the drying behaviour of most agricultural materials at drying temperature of 40 degrees Celsius and higher. Page model modified the Newton's model by adding n as an empirical constant which varies for each material being considered. Newton's model uses the first term of a general series solution for Fick's second law of diffusion (Yadollahinia et al., 2008). An experiment conducted by Mercer et al. (2011) Page model was utilized for energy sorghum with four different configurations of stalks which resulted in R<sup>2</sup> ranging from 0.997 to 0.999. Shen et al. (2011) utilized dried chopped sweet sorghum stalks. From an experiment by Shen et al. (2011), Wang-Singh's model showed the best fit from the gathered data. Drying time to achieve the moisture content for safe storage of sweet sorghum stalk at 16.28%, dry basis (14%, wet basis) using Page Equation were 43.88 hours for 5-cm sample, 14.95 hours for cut samples, and 7.99 hours for 1-cm chips, at 60°C drying temperature, 14% relative humidity and 2.91 meters per second air velocity.

Ethanol recovery from fermentation process proved that using solid stalk samples is feasible without juice extraction or size reduction. The maximum ethanol concentration was recovered from using fresh 10-cm samples with cellulase at 221.92 ml per kg dry mass while ethanol recovery from dried 10-cm cut stalk samples was 168.24 ml per kg dry mass. It was observed that cellulase provided more fermentable sugars for fermentation through enzymatic hydrolysis of cellulose from biomass substrate. Matsakas and Christakopoulos (2013) utilized cellulase for liquefaction step that resulted in lower viscosity of

high solid content substrate which allowed better mixing during fermentation. The results also show that ethanol from fresh samples were higher than the dried samples. Dried samples were depicted to have lower ethanol recovery since drying produced inhibiting compounds that affected the fermentation process. Possible inhibitors include furfural and hydroxymethyl furfural, weak acids and phenolic compounds (Sutton, 2011). It was also observed that chip samples with cellulase had lower ethanol production compared to other stalk samples. Glucose acts as inhibitor to cellulase enzyme and high initial concentration of this sugar affected hydrolysis of cellulose (Sutton, 2011).

From the average values of each statistical parameter for goodness of fit, Page drying equation best represent the drying behaviour of sweet sorghum stalks. From the results of this study, employing dried and fresh stalk samples without juice extraction is feasible for ethanol production. The process of introducing sweet sorghum stalks can be further improved by sugar extraction through agitation and enzymatic hydrolysis.

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