



## Co-extraction of soluble and insoluble sugars from energy sorghum based on a hydrothermal hydrolysis process



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### HIGHLIGHTS

- A co-extraction process of soluble and insoluble sugars from ES was developed.
- N series of ES presented a higher recalcitrance to sugar release than T series.
- More than 80% pentose and hexose was recovered for T series of ES.
- A negative correlation for the lignin content, CrI and G/S ratio to ED was observed.
- The heterogenization of chemical composition distribution for HH treated ES was broken.

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### ABSTRACT

A process for co-extraction of soluble and insoluble sugars from energy sorghum (ES) was developed based on hydrothermal hydrolysis (HH). Two series of ES were investigated: one (N) with a high biomass yield displayed a higher recalcitrance to sugar release, whereas the second (T) series was characterized by high sugar extraction. The highest total xylose recoveries of 87.2% and 98.7% were obtained for N-11 and T-106 under hydrolysis conditions of 180 °C for 50 min and 180 °C for 30 min, respectively. Moreover, the T series displayed higher enzymatic digestibility (ED) than the N series. The high degree of branching (arabinose/xylose ratio) and acetyl groups in the hemicellulose chains of T-106 would be expected to accelerate sugar release during the HH process. In addition, negative correlations between ED and the lignin content, crystallinity index (CrI) and syringyl/guaiacyl (S/G) lignin ratio were observed. Furthermore, finding ways to overcome the thickness of the cell wall and heterogeneity of its chemical composition distribution would make cellulose more accessible to the enzyme.

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### 1. Introduction

Energy sorghum (ES) is a C<sub>4</sub> graminaceous crop with a high biomass and sugar yield (Zhang et al., 2010). The juice extracted from the stalk is rich in soluble carbohydrates, including glucose, sucrose and fructose, so it is usually known as sweet sorghum (Regassa and Wortmann, 2014). Many studies have reported that ES is potentially a very good resource for fuel ethanol production by fermentation of the juice or the chopped stalks in a solid state process. Compared with the method of solid-state fermentation

(SSF) (Li et al., 2013b), the process of liquid-state fermentation (LSF) from juice is quite mature, but one of its inherent problems is minimization of sugar loss during storage of the juice (Ratnavathi et al., 2011). In addition, for both SSF and LSF, a major challenge for large-scale applications is how to deal with the solid residue (Yu et al., 2012a). Traditionally, this has been utilized as animal feed, in papermaking or for combustion. Recently, conversion of bagasse to fuel ethanol has attracted much global interest due to its high content of insoluble carbohydrates such as cellulose and hemicellulose (Jafari et al., 2016; Ostovareh et al., 2015; Rohovsky et al., 2013).

However, the chemical and structural properties of lignocellulosic materials protect the cellulose and hemicellulose from chemical and enzymatic attack (DeMartini et al., 2013; Meng and

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Ragauskas, 2014). This recalcitrance to sugar release is a major limitation to the cost-effective industrial conversion of bagasse to ethanol. Understanding and overcoming this recalcitrance are central research themes of the U.S. Department of Energy BioEnergy Science Center (BESC), where researchers hope to use a single microbe or microbial consortium to deconstruct lignocellulose and convert it directly to product. However, many literature reports have indicated that pretreatment, using catalysts, acids, bases, ionic liquids, water, heat, milling, microorganisms or other means, to break down complex cell wall structures composed of cellulose-hemicellulose-lignin is the inevitable course that will allow improvement in the release of fermentable sugars in the near future. An effective pretreatment can reduce downstream pressure by making cellulose more accessible to enzymes and minimize the formation of degradation products that inhibit the growth of fermentative microorganisms (Shen et al., 2012). In authors' previous work, a hydrothermal pretreatment (HP) process based on a step-change flow rate (Yu et al., 2011b) was developed for ES bagasse to enhance the sugar recovery from and enzymatic digestibility (ED) of cellulose. It was reported that less than 20% lignin was removed but ED of cellulose was more than 80%. The homogenization of the distribution of cellulose and lignin in the cell wall layer resulting from the removal of hemicellulose or lignin might contribute to the enhancement of accessibility to enzymatic attack (Yu et al., 2014).

As mentioned above, ethanol can be produced by fermenting both the extracted sugar juice (ESJ) and the liquid fraction of residual bagasse hydrolysis (LRBH), which would achieve a full ethanol conversion of sweet sorghum. Actually, such a model combining ESJ and LRBH might be impracticable for a cost-effective industrial operation because of the complexity of the process. In the present study, a co-extraction process that yields soluble and insoluble sugars from ES was developed based on hydrothermal hydrolysis (HH) to simplify the procedure of ethanol production from ES. It was expected that the soluble sugars derived from sucrose and starch would be dissolved and hemicellulose would be decomposed during this auto-hydrolysis process (Yu et al., 2013a). Hence, a systematic investigation of sugar release from ES using this hydrothermal and enzymatic hydrolysis process was performed. Furthermore, the possible structures or compositions that determine biomass recalcitrance were discussed here. The results provide insight into industrial applications of HH technology and the development of ES suitable for biofuel production.

## 2. Materials and methods

### 2.1. Materials

Two series of ES, including one with a high biomass yield (N) and another characterized by high sugar extraction (T), were kindly

**Table 1**  
Chemical composition changes and sugar recovery for different ES.

| ES    | Xylan (%)     |                                  |                        | Glucan (%)    |                           |                        | Lignin (%)    |                        |      |
|-------|---------------|----------------------------------|------------------------|---------------|---------------------------|------------------------|---------------|------------------------|------|
|       | Raw materials | Highest total xylose yield in HH | Removal ratio after HH | Raw materials | Total glucose yield in HH | Removal ratio after HH | Raw materials | Removal ratio after HH |      |
| N-1   | 20.7          | 83.1                             | 86.3                   | 35.6          | 14.0                      | 21.2                   | 22.3          | 31.5                   |      |
|       |               | 180 °C–50 min                    | 60.8                   |               | 14.4                      | 17.9                   |               | 31.4                   |      |
|       |               | 51.0                             |                        |               |                           |                        |               |                        |      |
| N-2   | 20.8          | 70.5                             | 75.6                   | 34.3          | 11.2                      | 24.2                   | 25.0          | 46.7                   |      |
|       |               | 180 °C–50 min                    | 47.1                   |               | 53.1                      | 14.0                   |               | 17.3                   | 40.2 |
|       |               | 160 °C–60 min                    | 79.7                   |               | 100                       | 19.4                   |               | 27.5                   | 26.8 |
| N-4   | 27.1          | 180 °C–50 min                    | 36.2                   | 38.6          | 19.9                      | 26.5                   | 50.0          |                        |      |
|       |               | 160 °C–60 min                    | 74.8                   | 100           | 35.8                      | 11.8                   | 27.9          | 29.5                   | 45.6 |
|       |               | 180 °C–50 min                    | 50.8                   | 54.4          | 14.0                      | 15.7                   | 41.1          |                        |      |
| N-10  | 21.2          | 160 °C–60 min                    | 87.2                   | 100           | 37.8                      | 19.1                   | 22.9          | 45.1                   |      |
|       |               | 180 °C–50 min                    | 52.4                   | 55.9          | 18.9                      | 19.2                   | 41.6          |                        |      |
|       |               | 160 °C–60 min                    | 84.2                   | 100           | 35.6                      | 13.4                   | 21.2          | 26.1                   | 49.1 |
| N-16  | 20.8          | 180 °C–50 min                    | 58.9                   | 62.3          | 15.6                      | 18.0                   | 39.4          |                        |      |
|       |               | 160 °C–60 min                    | 94.6                   | 100           | 38.6                      | 32.3                   | 48.7          | 20.8                   | 51.2 |
|       |               | 180 °C–40 min                    | 57.5                   | 63.6          | 38.7                      | 44.1                   | 49.1          |                        |      |
| T-3   | 19.3          | 160 °C–60 min                    | 98.3                   | 100           | 38.5                      | 33.4                   | 20.7          | 50.1                   |      |
|       |               | 180 °C–50 min                    | 52.5                   | 56.5          | 34.1                      | 39.4                   | 43.6          |                        |      |
|       |               | 160 °C–60 min                    | 97.1                   | 100           | 40.8                      | 35.6                   | 45.3          | 19.4                   | 44.1 |
| T-7   | 18.8          | 180 °C–30 min                    | 67.2                   | 71.3          | 35.5                      | 36.9                   | 39.6          |                        |      |
|       |               | 160 °C–60 min                    | 98.7                   | 100           | 41.5                      | 51.7                   | 62.5          | 15.3                   | 45.0 |
|       |               | 180 °C–30 min                    | 64.6                   | 67.5          | 39.4                      | 56.6                   | 40.8          |                        |      |
| T-8   | 19.9          | 160 °C–60 min                    | 98.7                   | 100           | 41.5                      | 51.7                   | 62.5          | 40.8                   |      |
|       |               | 180 °C–30 min                    | 64.6                   | 67.5          | 39.4                      | 56.6                   | 40.8          |                        |      |
|       |               | 160 °C–60 min                    | 98.7                   | 100           | 41.5                      | 51.7                   | 62.5          | 15.3                   | 45.0 |
| T-106 | 24.1          | 180 °C–30 min                    | 64.6                   | 67.5          | 39.4                      | 56.6                   | 40.8          |                        |      |
|       |               | 160 °C–60 min                    | 98.7                   | 100           | 41.5                      | 51.7                   | 62.5          | 15.3                   | 45.0 |
|       |               | 180 °C–30 min                    | 64.6                   | 67.5          | 39.4                      | 56.6                   | 40.8          |                        |      |

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