



Ethanol and lignin production from Brazilian empty fruit bunch biomass



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HIGHLIGHTS

- Brazil Government is promoting oil palm plantation on degraded land for biofuels.
- Brazil EFB has 33.5% glucan, 26.8% xylan, 21.2% lignin and 2.8% ash.
- Optimized dilute acid treatment conditions (160 °C, 1.025% acid, 10.5 min).
- Predicted maximum ethanol production was 51.1 g/kg dry EFB at low enzyme loading.
- 224 g lignin and 3.7 l xylose rich liquid per kg EFB are available as co-products.

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ABSTRACT

Brazil Government is promoting palm plantations to use degraded land for biofuels. Palm production is expected to increase 35 per cent in future and there would be profuse biomass available that needs to be handled efficiently. Therefore, in this study the potential of EFB from Brazil as raw material for biorefinery was explored by compositional analysis and pretreatment conditions optimization to produce ethanol and co-products. EFB from Brazil contains significant cellulose, hemicellulose, lignin and low ash content. The optimized dilute sulfuric acid pretreatment conditions for efficient cellulose and hemicellulose separation were 160 °C temperature, 1.025% v/v acid concentration, 10.5 min and 20% solid loading. Under optimum pretreatment process conditions, low enzyme loading (10 FPU, 20 IU cellulase and glucosidase enzyme/g glucan) and 15% solid loading, 51.1 g ethanol, 344.1 g solid residue (65% lignin and 24.87 MJ/kg LHV) and 3.7 l xylose rich liquid could be produced per kg dry EFB.

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1. Introduction

Increasing energy demand, security and environmental concerns are the reasons to explore more renewable energy resources. Among the renewable energy resources, biofuels have proved to be one of the options to replace fossil fuels in transport sector and at the same time mitigate carbon emission in the environment. There was an immense development in biofuel research and its commercialization in the last decade which led to use of blended biodiesel from vegetable oil and bioethanol from food crops with conventional fossil based diesel and petroleum in developed and developing countries. However, these advancements are seen as the primary reason for food price hike and deforestation in industrialized and less industrialized countries (Braun, 2007; Thompson and Meyer, 2013). To tackle this situation, the focus of biofuel research

has shifted towards non food based resources and agricultural waste like biomass residues for ethanol production and, non edible oil crops and algae for biodiesel production.

In case of bioethanol, though the technological advancement has been achieved to convert cellulose present in biomass residues to bioethanol via pretreatment, enzymatic saccharification and fermentation, the process does not seem to be economically viable for its commercialization (Carrquiry et al., 2011). However, a large amount of wastes containing xylose and lignin are generated in these processes that could be utilized to produce valuable products like furfural and lignin based products which could increase the overall economic status of the processes, and enable this technology (biorefinery) to be commercially available (European Commission, 2006).

Oil palm is cultivated in tropical regions mainly for its oil used in food, household products and biodiesel. In 2012, oil palm was cultivated in 17.2 million ha across the world (FAOSTAT, 2014). In Brazil, oil palm cultivation for biodiesel production is envisaged as a promising way to use degraded land and the Government is promoting oil palm plantations under the sustainable production

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of palm oil program (UNEP, 2011; Villela et al., 2014). Brazil produced 0.62 per cent of palm oil globally in 2012 and it is expected to increase by 35 per cent in future (UNEP, 2011; FAOSTAT, 2014) and if so there would be profuse biomass available that needs to be handled efficiently to avoid air pollution due to biomass burning (Sudiyani et al., 2013; Abdullah and Sulaiman, 2013).

Empty fruit bunch (EFB) is the biomass residue left over after extracting palm oil from fresh fruit bunches and it is rich in cellulose, hemicellulose and lignin. The composition of EFB varies based on the geographic conditions and the compositional analysis studies reported so far are from south-east Asia (Malaysia, Indonesia) (Sudiyani et al., 2013; Kim and Kim, 2013; Hamzah et al., 2011; Han et al., 2011; Tan et al., 2013; Jeon et al., 2014), Colombia (Piarpuzán et al., 2011) and Africa (Chiesa and Gnansounou, 2014), and there are no studies reported on EFB from Brazil. Hence, this study aims at exploring the compositional analysis and opportunity for bioethanol and co-products (lignin) production from Brazilian EFB. On the other hand, most of the studies in the past focused only on ethanol production from cellulose and hemicellulose. In this work a biorefinery approach capable of producing multiple products was examined to harness the benefits of other components which could be produced from EFB.

In the case of ethanol production from cellulose and hemicelluloses, the pretreatment process conditions have to be optimized to retain the maximum amount of cellulose and hemicellulose with less inhibitors such as furfural and acetic acid. A severe pretreatment condition would generate high inhibitors, therefore, the pretreatment conditions have to be less severe to limit the inhibitors but components separation may not be efficient at that conditions. Whereas, if severe conditions are used to make the separation efficient, the inhibitors generated would be more and a specific type of microbial strain which has high tolerance to inhibitors has to be used to ferment glucose and xylose or the inhibitors have to be reduced by other processes. Whereas in biorefinery context, only cellulose fraction would be used for ethanol production and as the hemicelluloses fraction would be used for products like furfural, there is no concern of such inhibitors problems and pretreatment process conditions capable to separate the components efficiently could be achieved. Therefore in general the negative effect of pretreatment process over the subsequent processes would be less for that biorefinery process (multiproduct) compared to the ethanol production process (single product) and this could be an advantage of biorefinery approach. As this study is being conducted under the frame work of a larger project involving the synthesis of multiple products (Bioethanol, lignin and furfural) only results of bioethanol and lignin production streams using dilute acid pretreatment are presented here.

Bioethanol and co-products from biochemical method involves pretreatment, enzymatic saccharification and fermentation. Pretreatment is one of the most important processes in the biorefinery approach where the strong linkages between the biomass components are disintegrated to be raw materials for individual product through the subsequent processes. Several pretreatment methods such as dilute acid treatment, alkali treatment, steam explosion, hot water treatment have been reported and each process has its own advantages and disadvantages (Alvira et al., 2010). The objective of this study is to separate the cellulose, hemicellulose and lignin fraction present in the EFB in order to convert them in specific products in the framework of a biorefinery scheme. Therefore dilute acid pretreatment was chosen owing to its ability to separate cellulose and hemicellulose fraction apart in milder reaction conditions (Chiesa and Gnansounou, 2014; Hu et al., 2010). However, the optimum conditions (Temperature, time and acid concentration) of dilute sulfuric acid pretreatment are not general for all the biomass and all goals, as it depends on the nature of the biomass and to the desirability function of the process. The conditions

have to be optimized for each particular plant residue and desirability function (Chiesa and Gnansounou, 2014). Consequently, in this research work, where the objective is to maximize both the ethanol and furfural production the pretreatment process parameters were optimized using a statistical design to determine the best operational conditions. The optimal conditions would not be the same compared to the case where only the total yield of monomeric sugars has to be maximized. Hence one of the novelties of the paper is to find out the optimal conditions of the whole process when both ethanol and furfural have to be produced and the feedstock is the oil palm EFB.

Following the pretreatment, the biomass rich in cellulose and lignin were subject to enzymatic saccharification at low enzyme loading to hydrolyze the cellulose fraction to glucose, which was further fermented by *Saccharomyces cerevisiae* to bioethanol. Finally, the sensitivity of the optimal conditions to solid loading at pretreatment and the enzyme loading on saccharification were studied, and the composition and heating value of lignin rich solid residue from saccharification were analyzed, and the mass balance of whole process was presented.

2. Methods

2.1. Materials

Dried oil palm EFB (*Elaeis guineensis*) was provided by Federal University of Pará, Brazil. Fresh fruit bunch (approximate tree age-20–25 years) was harvested from the palm plantation located between Thailand and Moju (about 2°45'S and 48°50'W) for palm oil production by the AGROPALMA company. EFB (moisture content 20–25%) collected after pressing from the palm oil production plant was washed, dried for 24 h at 60 °C, ground in a mill to a size range 3–4 cm and stored in a polythene bag until sending to the Ecole Polytechnique Fédérale de Lausanne (EPFL), Switzerland. At EPFL, the sample was further milled to pass through 80–20 mesh and stored in a zip lock bag until use. Sugars, ethanol, organic acid standards, Cellulase (Cellulast® 1.5L) and β -glucosidase (powder from almonds) enzymes were procured from Sigma–Aldrich, Switzerland. Other chemicals used in this study were of analytical grade.

2.2. Compositional analysis

Compositional analysis of raw, pretreated, saccharified EFB samples were carried out according to the standard procedures for biomass compositional analysis of National Renewable Energy Laboratory (NREL) (Sluiter et al., 2010; NREL, 2014). Sugars were analyzed by high performance liquid chromatography (HPLC) (Agilent Technologies, Germany) equipped with an Aminex HPX-87P column and refractive index detector (RID) with Millipore water as the mobile phase at flow rate 0.5 ml/min. The column and detector temperature were maintained at 80 and 55 °C respectively. Ethanol, furfural, hydroxymethylfurfural (HMF), acetic acid were analyzed by same HPLC equipped with an Aminex HPX-87H column and RID detector with 5 mM sulfuric acid as the mobile phase at flow rate 0.6 ml/min. The column and detector temperature were maintained at 65 °C and 55 °C respectively. Moisture, ash and insoluble lignin were analyzed by gravimetric method. Soluble lignin was analyzed using spectrophotometer method at 320 using 30 l/g cm as the absorptivity coefficient (Sluiter et al., 2011).

2.3. Dilute acid pretreatment

Design expert v 9 was used to create a Box–Behnken design (Tables 2 and 3) for the dilute acid pretreatment experiments. Three parameters temperature (100–180 °C), residence time

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