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# Use of Empty Fruit Bunches from the Oil Palm for bioethanol production: A thorough comparison between dilute acid and dilute alkali pretreatment

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## HIGHLIGHTS

- Oil palm Empty Fruit Bunches shown as good candidates for bioethanol production.
- Two pretreatment techniques compared (acid/alkali): the first option is superior.
- Optimized dilute acid treatment (≈161 °C, 10 min, 1.5% acid): 85% glucose yield.
- Alkali treatment is seriously hampered by high lignin content of the feedstock.
- Fate of the different components of biomass monitored during each treatment.

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## ABSTRACT

In the present work, two pretreatment techniques using either dilute acid ($H_2SO_4$) or dilute alkali (NaOH) have been compared for producing bioethanol from Empty Fruit Bunches (EFBs) from oil palm tree, a relevant feedstock for tropical countries. Treatments' performances under different conditions have been assessed and statistically optimized with respect to the response upon standardized enzymatic saccharification. The dilute acid treatment performed at optimal conditions (161.5 °C, 9.44 min and 1.51% acid loading) gave 85.5% glucose yield, comparable to those of other commonly investigated feedstocks. Besides, the possibility of using fibers instead of finely ground biomass may be of economic interest. Oppositely, treatment with dilute alkali has shown lower performances under the conditions explored, most likely given the relatively significant lignin content, suggesting that the use of stronger alkali regime (with the associated drawbacks) is unavoidable to improve the performance of this treatment.

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## 1. Introduction

Over the last decades, novel forms of energy alternative to petroleum based products have been sought for, principally to cope with the growing energy demand and environmental concern caused by fossil fuels. Lignocellulosic ethanol, i.e., alcohol obtained from plant residues, has emerged as an attractive option. This is mainly due to its ease of use in the transportation sector, its renewable character and its relatively even distribution worldwide.

In several tropical countries in Africa, Asia and South America, relevant amounts of biomass residues are generated from the palm oil industry. Among these, in particular, the leftovers obtained when oil palm fruitlets are stripped from the bunches holding them together have been given growing attention over the last few years. Palm trees are principally cultivated for their fruits from which an edible oil can be extracted. Beyond local consumption in traditional dishes, palm oil is widely used in the food industry worldwide, given its moderate price combined with a semi-liquid status, which is highly desirable for industrial applications. Some of the residues that are generated during the processing of the fruits at the oil plant are already used to generate power for the plant itself. Others, however, are considered as a waste and remain at present underused. Oil Palm tree Empty Fruit Bunches belong to this second category. They are humid as they undergo a sterilization process in autoclave before stripping. Noteworthy, EFBs seasonality is not particularly pronounced, and fruits may be available, depending on the country, for several months of the year. Also, this feedstock is already available at the chemical plant where palm oil is produced, and it is natural to think of an extension of

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the industrial plant that could process this residue to produce ethanol.

A rational use of EFB involves using its high polysaccharide content, mainly cellulose, to produce bioethanol by fermentation. As any other substrate for 2nd generation ethanol production, EFB contain sugar polymers that must be broken down to fermentable sugars by enzymes (saccharification) before being fermented via ordinary techniques (fermentation). Unfortunately cellulose is located in cell walls whose resistant structure must be disrupted to make cellulose attack possible by enzymes: therefore, a physico-chemical pretreatment must be performed at the beginning of the ethanol production chain. Industrially, the pretreatment step is critical both for its cost and for the impact on all following operations performed. This operation must be studied in detail and optimized for each particular plant residue. Particular emphasis should be given to those treatments which could reasonably be scaled to a real industrial context.

EFB have been recently investigated for bioethanol production using various techniques (Lau et al., 2010; Hassan et al., 2013; Park et al., 2013). Some difficulties arise in interpreting the results, possibly given the dissimilarity of the material used. Among the different studies, geographical origin and the composition of the samples vary, and sample preparation is not always thoroughly described. EFBs are peculiar in that they are usually left in the fields for variable times (usually a few days) after harvesting, and they undergo a sterilization step before separation of the fruits from the bunch takes place. These operations, which are demanded by the oil production, can presumably have an impact on the composition and susceptibility to successive treatments of the feedstock; this point, however, has not been given much attention in the literature. Also, a complete characterization of the biomass in terms of its chemical composition is not always given.

A few studies (Rahman et al., 2007; Yunus et al., 2010) have performed a dilute acid hydrolysis of this substrate, but then have focused exclusively on the fermentation of the liquid stream obtained upon pretreatment, neglecting the cellulose-rich solid residue. In other instances, no statistical optimization of the processes has been carried out (Millati et al., 2011; Jung et al., 2013a,b), although good glucose yields have been obtained from the solid pretreated at temperatures around 190 °C. Alkali treatments have given far less impressive results. This could be partially interpreted considering the composition of EFB, which have relatively high lignin content, generally between 20% and 30%: lignin is known to reduce the efficiency of treatments using basic solutions. In the case of ammonia fiber explosion (AFEX) (Lau et al., 2010), a post-treatment size reduction was essential to later reach good glucose yields: such behavior is peculiar, as this operation is not demanded by other feedstocks, and is probably justified by the fibers' toughness. The economic impact of this post-AFEX operation should be thus assessed. A number of studies have used NaOH solutions of relatively high concentrations, reporting variable outcomes. Hassan et al. (2013) could obtain glucose yields around 50% after a NaOH-enhanced (5%) steam pretreatment at 110 °C. Han et al. (2011) reported higher glucose conversion (85% of the original glucan) upon enzymatic saccharification following a relatively concentrated (almost 3 M NaOH) alkali treatment at 128 °C and 22 min. Similarly, Park et al. (2013) claim to have attained an economically attractive (>40 g/L) ethanol concentrations with simultaneous saccharification and fermentation of NaOH-pretreated EFBs at 121 °C: although in this case the alkali loading used was milder (1 M), the use of a very basic solutions raises concern about possible economic and environmental impacts. Discrete results (Choi et al., 2013) have been obtained with prolonged overnight soaking in a 3% NaOH solution before pretreatment: the main drawback of such an approach is given by the longer times.

The present study was conducted in the frame of a larger project involving the comparison of three different strategies for pretreatment (namely using pure water, dilute acid or dilute alkali): in the present work the results from the dilute acid and dilute alkali treatments are presented, whereas those from the pure water treatment will be the object of another publication. These pretreatments have been chosen as they are considered of interest for a potential future extension to the industrial case (Mosier et al., 2005), particularly as the use of exotic reagents is not needed. At present, indeed, most of the commercial plants do not employ any reagent at all, preferring hydrothermal approaches. In this optics, the dilute acid and alkaline treatments have been carried out in this work with the cheapest chemicals available (sulfuric acid and sodium hydroxide) and a particularly dilute regime has been chosen for the solutions to be employed. This point is crucial for limiting both the economic and the environmental impacts. We incidentally note that oil palm cultivation is already bitterly criticized on the ground of its impact on the environment. Dilute regimes must be clearly coupled with higher temperatures than in other studies (Han et al., 2011; Park et al., 2013). This would also allow to increase the hourly productivity in an hypothetical industrial context, although the generalization of laboratory results, especially concerning the optimal working conditions, is generally very complex as results critically depend on the set-up adopted.

Rigorous compositional analyses have been employed to fully characterize the feedstock during each of the stages studied (pretreatment and saccharification). Mass balances have been used to measure the compositions of both the solid and liquid streams generated during biomass processing. Pretreatment processes have been standardized to ensure reproducible conditions and to limit external influences. In particular, special attention has been paid to the temperature profile of each treatment, a point which is often underestimated in the literature and that can nonetheless induce variations in the response to the treatment. Enzymatic saccharification of pretreated samples has been carried out to assess the pretreatment performance. A statistical design for the pretreatment runs was employed, and optimization was performed for each treatment in order to find the best operational conditions.

Finally, the feedstock used in this study was collected in Africa, which is also the region of origin of the oil palm. This is different from mostly of the works in the literature that employ EFBs from South-Eastern Asia. Also, EFBs underwent a preliminary preparation similar to that encountered in the industrial context. In this study, in fact, we considered as raw feedstock EFBs that have been harvested, left a few days in the fields and then sterilized for 1 h at 134 °C. These parameters are very similar to those employed in the case of industrial processing: the residue thus mimics at best what could be reasonably available for a hypothetical industrial plant.

## 2. Methods

### 2.1. EFB samples

The EFBs from oil palm used in this study were collected in the nearby of Sakété, Republic of Benin (Africa), in the month of June 2011, almost at the end of the harvesting season. They had been previously collected in the nearby palm fields and left a few days on the ground to start fermentation; then, the fruits were separated by hands and empty bunches set aside. EFBs were carried to Switzerland the day following their collection and sterilized in the laboratory at the EPFL using an autoclave operating at 134 °C for 1 h. Samples were air-dried to avoid spoilage, then defibered by hand to a typical length of 4–5 cm; the fibers from different bunches have then been mixed thoroughly and stored in closed zip plastic bags screened from the sunlight. A small portion has

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