Study of valorization routes of spent coffee grounds

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Introduction

Global coffee production in 2017–2018 has been estimated by the International Coffee Organization (ICO) at 9.6 billion kilograms and 1.4 billion cups of coffee are prepared each day (ICO, 2018). Coffee remains one of the most traded products in the world. Since brewing of coffee consists of extracting a small number of selected compounds from the bean, the industry generates massive waste streams in the form of spent coffee grounds (SCG), which is the term used for the grinds remaining after the desirable compounds have been removed in the brewing process. Unlike many other organic wastes, coffee is high in several compounds detrimental to the environment and therefore is mostly disposed of in landfills as opposed to being used as compost. The economic and environmental costs of disposing of SCG in this way are undesirable, and for this reason alternative methods for dealing with SCG are needed. Moreover, the SCG supply is more stable than its edible and nonedible crop counterparts. To this end, this paper aims to assess valorization routes of coffee spent ground towards biodiesel and bioethanol production.

Materials and Methods

Spend coffee grounds**:** Samples of SCG were collected over two days from the university cafeteria in the Chemical Engineering School, in the National Technical University of Athens, Greece. The SCG samples from freshly brewed coffee were subjected to drying processes to hinder any possible microbial development. They were heated overnight in an oven at 103°C, cooled in a desiccator, and repeatedly weighed until they reached a constant mass (±0.1 g). Then they were physico-chemically characterised. They were mainly composed of 12.89% oil, s9.87% cellulose, 0.85% hemicellulose, 24.06% lignin (23.46% Klason lignin and 1.60% acid-soluble lignin), and 1.00% ash.

Oil extraction

The first critical process in the SCG-to-biodiesel pathway is oil extraction from the SCG. The selections in the present study are based on an exhaustive literature review and comparative investigations of each relevant parameter. Oil was extracted from SCG via an integrated Soxhlet extraction system. The entire extraction process consists of three distinct steps—extraction and distilling with the means of rotary evaporator. Two solvents are used: hexane and methanol.

Saccharification process

The bioethanol pathway was investigated through a sequential alkaline pretreatment with enzymatic hydrolysis in order to maximise glucose release from SCG.
Delignification alkaline pretreatment: SCG was pretreated in dilute NaOH 0.5M at 50°C for 6 h.
Enzymatic hydrolysis: Enzymatic saccharification of untreated and pretreated solid samples was executed at 50 ºC containing 10% w/w dry solids and the cellulolytic formulation, Cellic CTec2 (Novozymes, Denmark) for 72h. A buffer solution was employed in order to adjust the pH to 5.0.

Factorial experimental procedure: The aim of the experimental procedure was to determine the influence of some basic process parameters on either the oil extraction process, or the saccharification efficiency SG. The latter was estimated as the glucose production compared to the maximum theoretical glucose production from the total conversion of carbohydrates.
The parameters studied that influence the oil extraction process are Soxhlet cycles and Solvent to feedstock ratio (controlling parameters) and the respective for the saccharification yield the NaOH concentration and the enzyme loading.
The effect of the controlling parameters on each optimization parameter was estimated by performing a 2^2 factorial experiment. In general, by using a 2^n factorial design, n controlling parameters interrelate to an optimization parameter through an appropriate linear model. Their significance can also be estimated and assessed. The levels of the controlling parameters are given in Tables 1 and 2. The experimental area of the factorial design was pre-determined in preliminary trials.

Table 1. Controlling parameters and their levels at the factorial experiment for oil extraction from SCG for each solvent (hexane and methanol)

Controlling Parameter	**Variation Intervals**		
	Low level (-)	High Level (+)	Centre
Soxhlet cycles	6	20	13
Solvent/ feedstock	2	20	10

Table 2. Controlling parameters and their levels at the factorial experiment for sugars release from SCG

Controlling Parameter	**Variation Intervals**		
	Low level (-)	High Level (+)	Centre
NaOH (M)	0,3	0,7	0,5
Cellic CTec2 (μL/ g cellulose)	25	75	50

In the 2^2 factorial designs, 4 experiments were carried out in triplicate. Five extra experiments in the centre of the designs were also conducted for statistical purposes. From these data, mathematical models were constructed whose adequacy was checked by the Fisher criterion.

Results and discussion

According to the results of the factorial experiments and by following a specific analytical procedure (Alder et al., 1995; Cochran and Cox, 1957), the following linear models were estimated, interrelating the oil extraction efficiency (OEE, %) with the selected controlling parameters of the system:

For hexane: $OEE_{hex}=90.38$ Equation 1

For methanol: $OEE_{Meth}=14.69+1.75*(\text{Soxhlet cycles}) +1.05* (\text{Solvent to feedstock ratio})$ Equation 2

The adequacy of the mathematical models derived from the factorial design was checked by the Fisher criterion and they both proved to be adequate.

When hexane was used, within the range of the controlling parameters studied, no statistically important parameter was detected, implying that a high oil extraction efficiency (around 90%) could be achieved whichever the conditions applied.

The plus (+) in equation (2) indicates that an increase of the Soxhlet cycles or/and the Solvent to feedstock ratio when methanol was used leads to a higher oil extraction efficiency and consequently to a higher biodiesel efficiency. It was shown through statistical analysis that the interactions between the two parameters were negligible.

As far as the saccharification yield is concerned, the following adequate model derived:

$SG= 18.19+0.28*(\text{Cellic CTec2 dosage})$ Equation 3

From equation (3), it is obvious that an increase in the enzyme dosage promotes saccharification and thus the feedstock is rendered more attractive for ethanol production. The alkaline solution concentration didn’t significantly affect the saccharification yield within the range studied.

Conclusions

Spent coffee grounds contain large amounts of organic compounds that justify their valorization. To this direction, an effort to remove oil and to convert the SCG carbohydrates to fermentable sugars was made. The use of hexane as solvent proved to be more efficient than methanol yielding oil extraction efficiencies over 97%. Even though delignification yields over 80% were observed, the overall alkaline chemical pretreatment combined with enzymatic hydrolysis induced moderate results. Thus, further investigation is necessary.

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