



Research Paper

Kinetics of extraction and in situ transesterification of oils from spent coffee grounds

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ABSTRACT

Resource limits, environmental concerns and unstable petroleum costs have led to an increased effort to develop alternative liquid fuels. Purpose grown feedstocks are expensive and demand additional resources such as land and water. Spent coffee grounds (SCGs) are a good potential low-cost feedstock, however, processing times and costs must be lowered in order to be cost competitive with fossil fuels.

In this work, we investigated the kinetics of oil extraction from SCGs to explore if current methods of oil extraction could be hastened and if an integrated process which couples oil extraction and conversion to biodiesel stages in one single step (in situ transesterification) was viable.

Kinetics of oil extraction from SCGs using *n*-hexane as solvent was studied as a function of temperature, solvent to solid ratio and water content. We have found that oil extraction times could be as low as 10 min due to higher diffusion coefficients of oils from SCGs. Further, we demonstrate, for the first time, the successful in situ transesterification of SCGs using different concentrations of sodium hydroxide as a catalyst and methanol to oil mole ratios. Both of these outcomes show promise for lowering biodiesel production costs from SCGs, a ubiquitous waste product around the world.

1. Introduction

Decarbonisation of liquid fuels is necessary to avoid dangerous climate change. Biodiesel offers a viable alternative, however the growth of feedstocks render it economically uncompetitive with petroleum [1]. Moreover, the growth of biomass, both algal and terrestrial plants, requires resources that may be under pressure, for example land and water [2–4]. In addition there may be other detrimental impacts of land use change (both direct and indirect) for biodiesel feedstocks, for example increased greenhouse gas emissions [5,6]. Furthermore, the fertilisers used in biofuel production can promote eutrophication and therefore additional water treatment may be necessary [7,8].

As a result of these issues, research has focussed on the potential of waste products, transforming waste from a problem to a useful low cost resource for the production of valuable chemicals and biofuels. In the last decade an effort has been made to develop the use of agri-food waste as alternative raw materials for biodiesel production. The agri-food industry produces enormous amounts of waste which are rich in carbohydrates, proteins, pectin and bioactive compounds, all potential

sources of valuable chemicals and fuels [9]. As the biggest manufacturer in Europe, the food sector is increasingly focused on processing food waste in order to garner extra income and reduce waste and associated landfill [10]. Consequently, the potential of a number of agri-food waste products have been explored, including used cooking oil, organic municipal waste and olive stones [11–13].

With world coffee production in the region of 9.3 billion kilograms per year [14], spent coffee grounds (SCGs) is a promising biodiesel feedstock that currently has little or no commercial value. Furthermore, SCGs have a higher calorific value than many organic wastes [15]. On average, SCGs contain up to 20% of lipids [16] and lignocellulosic material, which can be used for bioenergy [17–19]. In addition SCGs could be re-purposed for various other uses such as absorbents [20], activated carbon [21–23] support for catalysts [24], antioxidants [25,26], and biofuel pellets [15]. The production of biodiesel is an especially attractive prospect given the urgent need to decarbonise liquid fuels and the limited viable sources of low carbon liquid fuel.

Oil extraction from SCGs is an important process for its successful utilisation as a biofuel. The limited studies that have assessed the oil content of SCGs report between 10 and 20% dry mass using solvent

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Table 1
Reported Soxhlet solvent extraction of oils from spent coffee grounds.

Solvent	Solvent to SCGs ratio ml g ⁻¹	Time h	Yield g oil extracted/g dry SCGs	Lit.
Hexane	15.0	8	13.0	[27]
Hexane	4.2	0.5	15.3	[28]
Hexane	8.0	6	19.7	[29]
Petroleum ether	-	6	12.5	[30]
Hexane	15.0	6	11.0	[31]
Hexane	3	1	13.4	[17]

extraction [17,27–31] (Table 1). Most of the studies are based on *n*-hexane or ether as solvents and demand excessive time periods for extraction (Table 1). The kinetics of oil extraction from SCGs is essential to determine the production rate, energy consumption and process minimization (all important parameters to determine the industrial applicability), yet this information is not reported. Key variables that may impact the extraction kinetics are: temperature, solvent to solid ratio, the diffusion capacity of solvents and solutes as well as by the size, shape and internal structure of the SCG particles. Once the oil has been extracted transesterification is necessary to produce biodiesel [17,28,32] (Table 2). As it can be observed, the yields of biodiesel from extracted oil is rarely reported, with the limited studies reporting yields of 56% to 100%; comparable with the yields from other vegetable oil feedstocks.

Although the use of SCGs as a low-cost oil-rich feedstock can decrease the cost and environmental impact of biodiesel production, the processing costs remain inhibitive and must be lowered in order to be competitive with conventional oil fuels. The integration of the oil extraction and transesterification stages into one single step (in situ transesterification) has the potential to achieve this. In the in situ transesterification process, the SCGs are immersed in alcohol containing a catalyst, allowing simultaneous oil extraction and its conversion to fatty acid methyl esters (i.e. biodiesel). Prior studies have proven the viability of in situ transesterification method using various feedstocks, including sunflower seeds [33,34], soybean [35] and jatropha curcas [36]. The yields of in situ transesterification processes are influenced by various parameters, such as temperature, catalyst loading, oil to methanol ratio, particle size and moisture [37]. Further, significant differences in production yields have been observed for different feedstocks due to their varying characteristics.

Therefore, the aim of the present study was to: (1) determine if quicker oil extraction is possible by establishing the kinetics of oil extraction from SCGs using hexane as a solvent, and, (2) to determine if in situ transesterification of oils by methanol to produce fatty acid methyl esters (i.e. biodiesel) is viable and the influence of temperature, solvent to SCGs ratio and catalyst concentration.

2. Experimental section

2.1. Materials

Spent coffee grounds were provided by Pizzetta Republic, Lancaster University campus, and originated from roasted Arabica Atkinson & Co. Ltd espresso coffee. All the chemicals used are listed in Table 3. These were supplied by Sigma-Aldrich and were used without further purification.

2.2. Methods

2.2.1. Characterization of spent coffee grounds

Collected SCGs were analysed for particle size and water and oil contents. The average particle size of coffee grounds was determined by a laser diffraction analyser (Malvern Mastersizer 3000E, accuracy better than 0.6%). The water content of SCGs immediately after collection was

Table 2
Transesterification of extracted coffee oils from spent coffee grounds.

Temperature K	Oil:Methanol mol/mol	Time min	Yield%	Reference
338	1:5	150	56	[28]
338	1:5	240	69	[28]
338	1:9	240	86	[28]
358	1:5	150	63	
358	1:5	60	59	[28]
358	1:9	60	80	[28]
333	1:15	120	60.5	[32]
343	1:13	10	100	[17]

Table 3
Chemicals used in this study which were supplied by Sigma-Aldrich.

Chemical	Purity / mass%	Purpose
<i>n</i> -Hexane	99.9	Oil extraction and post-transesterification extraction of FAMES
Methyl stearate	99.5	Gas Chromatography
Methyl palmitate	99.0	
Methyl linoleate	98.5	
Methyl oleate	99.0	
Methanol	99.8	In situ transesterification
Sodium hydroxide	98	Catalyst and acid value
Ethanol	99.5	Acid value
Phenolphthalein 2% in ethanol	-	
Wij's solution (0.1 M)	-	Iodine value
Potassium iodide	99.5	Iodine and peroxide value
Sodium thiosulphate	98.5	
Starch solution 1% in water	-	
Chloroform	99.0	Peroxide value
Acetic acid	99	Peroxide value and neutralization of catalyst after in situ transesterification

determined by measuring the mass before and after the SCGs were dried in an oven at 353 K until no further changes in mass were observed (AT201 analytical balance, Mettler, UK). Solvent *n*-hexane was used to determine the oil content of the dried SCGs by performing a Soxhlet extraction at 342 K (*n*-hexane's normal boiling point). Approximately 30 g of coffee grounds were used and the extraction was carried out for six hours. Hexane was subsequently evaporated in a rotary evaporator and the content of the extracted oil was determined gravimetrically using the analytical balance. Samples of the extracted oils were further analysed by titrations to determine acid, peroxide and iodine values.

2.2.2. Kinetics of oil extraction

Samples of the SCGs, approximately 25 g, were weighed using the Mettler AT201 analytical balance with stated repeatability of $\pm 3 \times 10^{-2}$ mg and transferred to a one-litre three-neck Pyrex flask with a hemispherical base. The flask was fitted with a condenser to avoid evaporation, thermometer to monitor temperature and syringe to sample extraction mixture at regular intervals. Solvent, *n*-hexane, was used at various solvent to SCGs ratios (mLg⁻¹): 5, 8, 12, 15, 25 and 30. The known amount of *n*-hexane was heated in a flask while SCGs were heated separately at the desired extraction temperature (293.2 K, 313.2 K, 323.2 K and 333.2 K). When both materials reached the desired temperature, they were mixed and the extraction started. The flask was immersed in a temperature-controlled water bath equipped with a magnetic stirrer to ensure suspension of the SCG particles. The temperature was controlled within ± 0.1 K, monitored by a thermometer. Samples of the extraction mixture (1 mL), taken at regular intervals, were collected in pre-weighed vials. The amount of oil extracted in each time interval was determined gravimetrically by measuring the mass of the residue in the flask after evaporation of the

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