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Original article

Integrated strategies for water removal and lipid extraction from coffee industry residues

Ioannis Efthymiopoulos[a,*], Paul Hellier[a], Nicos Ladommatos[a], Arthur Kay[b], Ben Mills-Lamptey[b]

[a] Department of Mechanical Engineering, University College London, Torrington Place, London WC1E 7JE, United Kingdom
[b] bio-bean Ltd., 6-8 Cole St., London SE1 4YH, United Kingdom

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ABSTRACT

Spent coffee grounds (SCGs) and roasted defective coffee beans (RDCBs), are a potentially sustainable source for biofuel production if the processing of these residues, and the recovery of energy-dense lipids, can be undertaken in an energy efficient way. A necessary step in solvent extraction of lipids is prior drying of the feedstock, and this can incur a significant energy cost in the case of SCGs. This study investigates solvent extraction strategies for crude lipid recovery from wet or partially dried SCG samples, with mechanical pressing used as pre-treatment and alternative to thermal drying. Dewatering of SCGs by application of pressures up to 550 bars removed 42% of the moisture present, while lipid expression from whole RDCBs was achieved, with a maximum crude lipid recovery of 77.1% relative to available oil obtained. Crude extracts removal from partially wet pressed SCGs through accelerated solvent extraction (ASE) with ethanol was not impeded by moisture presence, and the obtained extracts had high energy density (~39 MJ/kg) comparable to hexane-extracted crude lipids. SCG and RDCB crude oil removed through solvent extraction and mechanical pressing respectively had similar fatty acid (FA) compositions, but a higher proportion of free fatty acids (FFAs) in solvent-extracted oil.

Introduction

The majority of worldwide energy consumption continues to come from fossil sources [1]. However, price fluctuations, increasing energy demand, dependency on imported products and environmental concerns render the research for alternative and renewable fuels a critical matter [2–4]. For example, biodiesel has been recognized as a feasible source of energy for the transport sector as it is compatible with current diesel engine technology and existing distribution networks, and offers advantages over petroleum diesel such as negligible aromatic and sulfur content, inherent lubricity and higher flash point [4–6]. Furthermore, biodiesel is a potentially carbon neutral fuel with emissions of SO_2, SO_3, CO, unburnt hydrocarbons and particulate matter lower than that of diesel according to several studies [4,5,7–9]. Nevertheless, the high cost of biodiesel production from biomass sources has restricted its further commercialization as a sustainable fuel [5,9].

There is a high economic and indirect environmental cost of utilizing edible oils for fuels, as they have high energy requirements during cultivation, compete with food resources and are subject to potential future depletion [4,5,9–11]. The feedstock used for biodiesel production accounts for approximately 70% up to 95% of the total process cost [4,7,9,12]. Therefore, if food grade lipids could be replaced by non-edible oils, such as waste cooking oils, animal fats or other agro-industrial waste residues that contain suitable lipids, for example coffee industry residues, this would significantly reduce biodiesel costs [4,5,9,12].

SCGs are the main residual products of the coffee industry with an average annual production of 8 million tonnes worldwide, and contain a significant amount of lipids, ranging from 7 to 30.4% w/w on a dry weight basis, with most researchers reporting values between 11 and 20% w/w [9,10,13–17]. RDCBs are also residues of the coffee industry, constitute about 20% of the total mass of the coffee bean production and can be classified as black, sour and immature beans which roast to a lesser degree than other types of beans under the same roasting conditions [18–20]. RDCBs can be differentiated by non-defective ones only by an evaluation of their volatile profile [21]. According to previous studies, RDCBs have a slightly lower lipid content of 9.2–10% w/w than non-defective roasted beans, and a moisture content as low as zero immediately after roasting, which can increase up to 3% w/w as the beans tend to absorb water from surrounding air [18–20]. Table 1 shows the energy content of SCGs, defatted SCGs, SCG oil and SCG derived biodiesel found in previous studies. To the best of the authors'

Abbreviations: SCG, Spent coffee grounds; RDCB, Roasted defective coffee bean; ASE, Accelerated solvent extraction; FFA, Free fatty acid; FA, Fatty acid

* Corresponding author.
E-mail address: i.efthymiopoulos@ucl.ac.uk (I. Efthymiopoulos).

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Table 1
Higher heating values of SCGs, SCG oil, defatted SCGs and SCG derived biodiesel.

Reference	HHV of SCGs (MJ/kg)	HHV of Defatted SCGs (MJ/kg)	HHV of SCG lipids (MJ/kg)	HHV of SCG biodiesel (MJ/kg)
Al-Hamamre et al. [9]	20.79	–	35.86–39.00	39.65
Haile [10]	–	19.3–21.6	38.22	39.6
Campos-Vega et al. [14]	19.61	17.86	–	–
Silva et al. [22]	24.9	–	–	–
Tsai et al. [23]	23.5	–	–	–
Go et al. [24]	22.83–24.39	20.03–20.27	–	–
Bok et al. [25]	22.74	–	–	–
Romeiro et al. [26]	25.7	–	–	–
Zuorro and Lavecchia [27]	23.72–24.07	–	–	–
Vardon et al. [28]	23.4	20.1	–	39.6
Berhe et al. [29]	–	20.8	37.88	38.4
Abdullah and Bulent Koc [30]	–	–	43.2	–
Caetano et al. [31]	19.3	–	36.4	–
Deligiannis et al. [32]	21.16	–	–	39.49
Caetano et al. [33]	19.3	19.0	40.8	–

knowledge, data is not available in the literature regarding the higher heating value (HHV) of RDCBs.

Based on the findings of previous studies presented in Table 1, it can be seen that SCGs have a HHV greater than most agro-industrial residues and woody biomass (HHV: 19–21 MJ/kg) [25,27,28,33,34]. SCG lipids have a HHV slightly lower than that of petroleum crude oils (41–48 MJ/kg) but similar to that of other vegetable oils or animal fats [10,35]. The variation in SCG energy content can be possibly attributed to variation in lipid content and overall composition due to the origin, upstream processing and different blends of coffee varieties [9,16,36].

One disadvantage of SCGs as a source of renewable energy is the high moisture content of the grounds, which usually ranges between 50 and 60% w/w [9,13], but can be as low as 18% w/w [32], or as high as 80% w/w [37]. The water is present either as unbound excess moisture resulting from the brewing process, with coffee grounds used in the industrial production of instant coffee retaining higher moisture levels than retail, or bound moisture entrapped within the microstructure of the solid particles [9,37]. For recovery of oils from SCGs, the main extraction techniques previously reported are solvent extraction and Supercritical fluid extraction (SFE), methods that require dried materials and thus necessitate removal of moisture from SCGs [9,10,13,15,17,37,38].

Thermal drying has most commonly been used for dewatering SCGs at laboratory scale prior to further processing [9,10,13,15,28,33], however, at large scale this would likely be a time and energy intensive procedure [24,30]. Extraction of lipids from wet or partially dried SCGs through Soxhlet with *n*-hexane showed that moisture contents greater than 2% w/w inhibit oil extraction, with increasing moisture content of the grounds leading to lower crude lipid yields, while extraction at a pilot plant with countercurrent contact of *n*-hexane and SCGs was found to be less sensitive to water presence of between 5 and 10 % w/w [13]. Abdullah and Bulent Koc (2013), attempted to circumvent the necessity for water removal by extracting lipids from wet SCGs through ultrasound-assisted two-phase oil extraction and obtained a crude lipid recovery of 98% relative to total available oil in 30 min [30].

Solvent extraction of lipids at elevated temperature, commonly known as ASE, or pressurized fluid extraction, is another extraction method that partly derives from SFE but which can operate successfully with partially wet oilseeds such as rice bran and corn kernels [39,40], and one that has not been previously used for the extraction of lipids from wet or partially dried SCGs. Jalilvand et al. (2013) investigated the dynamic (i.e. continuous solvent flow) pressurized fluid extraction of oil from rice bran with a moisture content of 10.2% w/w with n-hexane at temperatures ranging between 40 and 80 °C, and achieved a 100% crude lipid recovery at 77 °C after 34 min with a flow rate of 0.2 ml/min [39]. Moreau et al. (2003) examined the extraction of oil from corn kernels with a moisture content of 14–16% at temperatures between 40 °C and 100 °C using hexane, dichloromethane, isopropanol and ethanol and obtained crude lipid yields varying between 2.9 and 5.9% w/w [40]. A correlation between increasing solvent polarity and higher crude lipid yield was observed in this study with ethanol being the most efficient solvent, while the highest crude lipid yields were achieved at 100 °C irrespective of the solvent used [40].

Mechanical expression is another method that has been extensively used for oil removal from vegetable oilseeds such as soybean [41,42], palm fruit [41], rapeseed [43], sesame seed [43] flax seed [43,44] and rubber seed [45], while it has also been used before for the recovery of lipids from RDCBs by Oliveira et al. (2006), without specifying though the pressing conditions and crude lipid yields obtained [18]. Mechanical pressing of oilseeds is usually combined with thermal drying for better results, with materials that undergo mechanical expression partially dried prior to the pressing procedure [42–44,46]. Ali and Watson (2013) investigated oil expression from flax seeds of water content between 4 and 12% w/w with a screw press, and found that the crude oil yield increased with increasing moisture within the range investigated [44]. Willems et al. (2008) investigated the expression of oil from sesame seeds with a hydraulic press at feedstock moisture contents of between 0% and 5.5% w/w and found that the highest crude oil yield was obtained at a moisture level of 2.1% w/w [43].

Generally, an increase in the mechanical pressure applied leads to a crude oil yield increase in mechanical expression from oilseeds at pressures ranging from 100 to 700 bars [43,45], while pressures greater than 450 bars can improve the crude oil recovery up to 15% w/w (oil/oil) relative to presses operating at lower pressures [43]. Santoso et al. (2014), who examined the hydraulic expression of oil from rubber seed at pressures between 80 and 120 bars, found a relationship between increasing duration of pressing (30–90 min) and higher crude oil yield [45].

Mechanical expression has also been used for water removal from SCGs, as was demonstrated by Schwartzberg (1997), who removed 63% w/w of the moisture content from SCGs by applying 600 bars of pressure (ram speed of 500 mm/min) at room temperature [47]. A previous study considering lignite, bio-solids and bagasse investigated temperatures ranging between 20 and 200 °C and pressures from 15 to 240 bars, for a constant duration of 5 min, and found that processing conditions of 150 °C and 120 bars removed approximately 55–75% of the water present [48].

In this work, SCGs, RDCBs, crude coffee lipids extracted at different conditions and defatted SCGs and RDCBs were characterized in terms of energy content, and various processing strategies investigated for energy efficient recovery of lipids. Mechanical pressing was utilized for crude lipid and water expression from coffee residues, with only one previous report of the use of pressing for water removal from SCGs [47], and none for lipid expression. Solvent extraction of oil from wet