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Overcoming the caking phenomenon in olive mill wastes

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ABSTRACT

The use of olive mill wastes (*orujillo*) within coal fired power stations in the UK has led to unexpected difficulties with material caking within the fuel handling plant. This study replicated orujillo caking on a laboratory scale using a planetary ball mill and explored the impact of mill parameters (speed, volume, and duration) on the caking phenomenon. The impact of orujillo composition was examined for 4 sections of fresh and dried orujillo (whole, pulp 0–850 μm, pulp 850–3350 μm, and cluster 3350 μm+) for set milling conditions. Caking was induced by heat generation within the mill and was most prevalent in the pulp section of orujillo. Caking was brought on by a glass transition step, which was measured to be around 97–98 °C for a moisture content of 6–7% in a differential scanning calorimeter (DSC). Caking was the result of the bulk moisture content (14–18%) being higher than the standard moisture content of orujillo (<12%), and can be mitigated through drying. Thus the key to overcoming orujillo caking in fuel handling plants is through moisture content control. Additionally, as the caking issue is most prevalent in the pulp section, all fines below the required combustion particle size (typically <1 mm) should be removed prior to comminution and sent directly to the burner. This would also reduce the comminution load by nearly 50%, increase the energy potential of the fuel, and remove the most problematic section of orujillo from the fuel handling plant.

1. Introduction

Biomass co-firing and conversions offer a near term low cost solution for reducing emissions from conventional fossil fuel power plants. Agricultural residues offer a plentiful supply of material for combustion (Daioglou et al., 2015) and biodiesel (Hernández et al., 2014), and include olive mill wastes, which are often disposed of in landfill (Paraskeva and Diamadopoulos, 2006). However the agglomeration and caking of olive mill wastes in fuel handling plants of power stations is a limiting factor in its viability as a combustion fuel. This paper replicates the caking of olive mill wastes on a laboratory scale, investigates the reasons behind it, and strategies to overcome the problem.

By 2100, it has been projected that there will be an additional theoretical global bioenergy potential of 20-50 EJ/yr from agricultural residues (Daioglou et al., 2015). In Spain, 2625 ktoe/yr of primary energy could be sourced from agro-industrial residues, primarily from olive mill and wood processing residues (Gómez et al., 2010). Over 75% of global olive oil production originated from Europe in 2013–2014, with over 70% of this coming solely from Spain (International Olive Council, 2015). In Spain, the two-phase continuous centrifugal olive oil extraction system is used in approximately 90% of olive mills (Dermeche et al., 2013). In this system, the olive paste is separated into two phases; olive oil and wet pomace known as “alperujo”, which is a semi-solid combination of olive husk and olive mill waste water. Alperujo is usually treated with a secondary centrifugation to extract the residual oil (Alburquerque et al., 2004), and the resulting by-product is dried and subjected to chemical extraction in order to maximise the oil yield. The resulting dry waste from this solvent extraction is known as “orujillo”, is free of oil and usually contains 10–12% moisture content (Ollero et al., 2002). At present, there is no European legislation regulating the disposal of olive mill wastes (Rodrigues et al., 2015). Current disposal practices include landfill disposal, discharge into nearby rivers, lakes or seas and storage/evaporation in lagoons. However this has led to soil contamination and high phytotoxicity, water body pollution, underground seepage, and problems with offensive odours (Goula and Lazarides, 2015; Paraskeva and Diamadopoulos, 2006). Due to the respectable higher heating value of olive mill wastes (17–19 MJ/kg) (Álvarez et al., 2015; Williams et al., 2015),

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one method of reusing the waste is as a fuel for electricity generation (Oktay, 2006; Roig et al., 2006).

Caking can be defined as a deleterious transformation of low moisture, free flowing powders into an agglomerated solid, firstly as lumps, and ultimately as a solid sticky material, resulting in loss of material quality and function (Aguilera et al., 1995). Caking is a common issue in food, fertiliser, and pharmaceutical industries (Gabbott, 2008; Palzer, 2011; Roos, 2010), and is now being experienced with orujillo in power generation fuel handling systems. The caking rate of a powder will depend on the instantaneous moisture content and the ambient temperature and humidity, with the material's glass transition temperature being the most representative parameter of this transition (Boonyai et al., 2004). Moisture acts as an extremely good plasticiser in dried foods, and can reduce the glass transition temperature to room temperature, decrease viscosity, and result in caking (Lazou and Krokida, 2011; Roos, 2010). The glass transition temperature of the olive mill waste water is moisture dependent. The glass transition occurs around 56 °C for 9% moisture content, and reduces towards freezing as the moisture content increases (Goula and Adamopoulos, 2013). While the drying of orujillo has been investigated for bioenergy purposes (Casanova-Peláez et al., 2015; Christoforou and Fokaides, 2016; Goula et al., 2015), the caking and glass transition behaviour of orujillo has not been explored in literature. The present study explores the mechanisms of orujillo caking in relation to mill settings and material composition by replicating the phenomenon on a laboratory scale in a planetary ball mill. In addition, analytical techniques were used to examine the thermal properties of orujillo. This paper provides insight into the causation of orujillo caking and practical approaches to overcoming the issue in power generation fuel handling systems.

2. Materials and methods

Orujillo has routinely been co-combusted up to 10% by weight with coal at EDF Energy plc pulverised fuel coal fired power station at Cottam in Lincolnshire, UK, for several years through a semi-direct injection system. Orujillo is milled separately in a hammer mill and then pneumatically injected into the coal pulverised fuel stream, and the coal and orujillo are then co-combusted in the boiler. Orujillo caking has led to blockages in the fuel handling system, with the resultant cake proving difficult to remove, resulting in significant operational downtime for system repairs. The caking of orujillo has been replicated in two laboratory scale mills. In a preliminary study in the planetary ball mill, the caking of fresh orujillo was replicated with the severity of caking being influenced by the mill speed, fill volume, and milling duration (Williams et al., 2013). Orujillo caking was also observed in a laboratory scale ring-roller mill, which resulted in the formation of a solid cake, and subsequently in mill overload and failure of the mill bed motors (Williams et al., 2016). In order to explore the mechanisms behind the caking effect, a full study was formulated to investigate orujillo caking on a laboratory scale. The study characterised the orujillo to establish its composition and thermal behaviour, and two sets of experimental trials were conducted in a planetary ball mill to establish the influence of environment and material composition on the caking effect. The study focuses on the use of orujillo in moisture and temperature conditions experienced in industrial processing rather than on moisture treated samples.

2.1. Materials

Spanish *orujillo* (olive cake) was provided by EDF Energy plc. Orujillo exhibits a seasonable variance of up to 8% in moisture content due to its external storage in Spain prior to shipping to the UK. Orujillo is classed as "chemically untreated fruit residues, crude olive cake (class 3.2.1.4)" according to BS EN 17225-1:2014 (The British Standards Institution, 2014).

2.2. Moisture content

Moisture content was measured in accordance with BS EN 14774-1:2009 (The British Standards Institution, 2009). 300 ± 1 g of orujillo was dried in a Thermo Scientific Heraeus UT 6 forced-air oven at 105 ± 2 °C for 24 h. After drying the weight of the sample was recorded and used to calculate the moisture content of the sample; each sample was tested in triplicate. The dried orujillo used in the milling tests were dried under the same conditions, and then placed in zip-lock sealed bags until use. To analyse the moisture re-adsorption of orujillo in atmosphere, 100 g ± 1 g of orujillo was dried according to BS EN 14774-1:2009 and then placed on a Ohaus Pioneer PA4102c balance for 24 h. The weight was logged via a laptop using the Ohaus Data Acquisition Software at 10 s intervals over a 24 h period in an air conditioned laboratory. To analyse the moisture re-adsorption of orujillo in sealed storage, 100 ± 1 g of orujillo was dried to BS EN 14774-1:2009 and then stored in zip-locked bags and weighed. After 6 days the moisture increase was found by weighing the samples and zip-lock bags again and noting the increase in mass.

2.3. Sieving

Particle size distributions were determined by sieving for the pre-milled and milled product in accordance with BS EN 15149-2:2010 (The British Standards Institution, 2010). The samples were sieved into 16 size fractions (4750, 3350, 2360, 1700, 1180, 1000, 850, 600, 425, 300, 212, 150, 75, 53, 45, 38 μm sieves). Sieving was conducted on a Retsch AS200 Control vibratory sieve shaker in two stages. In the first stage, 8 coarse sized sieves (4750–600 μm) were used, and in the second stage, 8 finer sieves (450–38 μm) were used. Each sieving stage was conducted for 15 min at a 3 mm amplitude.

2.4. Rosin-Rammler distribution analysis

The Rosin-Rammler distribution equation was originally developed to describe the distributions of coal fines from coal mills (Rosin and Rammler, 1933), and it is often used to describe the particle size distribution of comminuted biomass (Bitra et al., 2009a; Gil et al., 2012). The Rosin-Rammler equation is defined as:

$$R(d) = 100\left(1 - e^{-\left(\frac{d}{d'}\right)^{n}}\right) \qquad (1)$$

where R is cumulative percentage mass undersize (%), d is particle diameter (μm), d' is the characteristic particle size (μm) and corresponds to the 63.2% cumulative distribution particle undersize value ($1 - 1/e = 0.632$), and n is the Rosin-Rammler size distribution parameter (dimensionless). A lower value of n means a lower slope and thus a wider distribution and higher diversity of the particles sizes. The Rosin-Rammler parameters were obtained with the Matlab® GUI Tool developed by Brezáni and Zelenak (Brezáni and Zelenak, 2010) using a non-linear curve fitting technique.

2.5. Thermogravimetric analysis

The thermal composition of orujillo was obtained by using a TA Instruments Q500 Thermogravimetric Analyser (TGA). TGA runs used 10–15 mg of milled sample with a particle size range of 75–300 μm, and used the method developed by Lester et al. (Lester et al., 2007) for analysing the composition of biomass. The sample was heated in a Nitrogen atmosphere with a flow rate 100 ml/min,