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Optimization of torrefaction conditions of coffee industry residues using desirability function approach

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ABSTRACT

The aim of the present study is to analyze the influence of independent process variables such as temperature, residence time, and heating rate on the torrefaction process of coffee chaff (CC) and spent coffee grounds (SCGs). Response surface methodology and a three-factor and three-level Box-Behnken design were used in order to evaluate the effects of the process variables on the weight loss (W_L) and the Higher Heating Value (HHV) of the torrefied materials. Results showed that the effects of the three factors on both responses were sequenced as follows: temperature > residence time > heating rate. Data obtained from the experiments were analyzed by analysis of variance (ANOVA) and fitted to second-order polynomial models by using multiple regression analysis. Predictive models were determined, able to obtain satisfactory fittings of the experimental data, with coefficient of determination (R^2) values higher than 0.95.

An optimization study using Derringer's desired function methodology was also carried out and the optimal torrefaction conditions were found: temperature 271.7 °C, residence time 20 min, heating rate 5 °C/min for CC and 256.0 °C, 20 min, 25 °C/min for SCGs. The experimental values closely agree with the corresponding predicted values.

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1. Introduction

Torrefaction is a thermal pretreatment process operating at low temperature (200–300 °C), under atmospheric conditions, in the absence of oxygen. It is interesting for upgrading ligno-cellulosic biomass to a higher quality fuel, and for its following conversion into heat or other energy carriers, such as electricity and biofuels (Poudel et al., 2015). During torrefaction, the bound and unbound moisture as well as high volatile fraction of organic components, particularly hemicellulose and some lignin, are released from biomass. They form a solid product mainly composed of cellulose and lignin (Medic et al., 2012), with lower H/C and O/C ratios and higher carbon content than raw material (Lee et al., 2012). However, the thermal decomposition behaviour of each kind of biomass can greatly vary with of the polymer structure and the ash content, that may catalyze some reactions (Lee et al., 2012).

The kinetic mechanism of the torrefaction is also influenced by the operating parameters, such as the reaction temperature, the residence time, and the heating rate (Mundike et al., 2016). Many researchers showed that the torrefaction temperature is the deter-

mining factor for obtaining the most optimized yield and quality of the final solid product (Phanphanich and Mani, 2011; Chen and Kuo, 2011; Medic et al., 2012). In general, the higher the torrefaction temperature, the more oxygenated compounds are converted into volatiles, obtaining a char-like solid product characterized by higher energy density. The effect of the residence time on the char yield and Higher Heating Value (HHV) is more difficult to interpret. Mundike et al. (2016) for Lantana camara plant, showed that increasing residence time from 25 to 80 min at 280 °C, char yield decreases from 65.97% to 52.42% and HHV increases from 22.37 MJ/kg to 24.95 MJ/kg. Chiou et al. (2015) investigated several pomaces and nut shells, and found that mass yields decrease with longer residence time along with the HHV values; in particular for apple pomace, the HHV value of char decreases from 26.1 MJ/kg to 23.0 MJ/kg by increasing residence time from 20 min to 60 min at 260 °C. As regards the influence of the heating rate, only one study analyzes its effect on char yield and HHV (Mundike et al., 2016), highlighting a minimal influence of this operating parameter on the torrefaction process.

Data in the Literature show that operating parameters should not be analyzed individually and that it is necessary to employ statistical methods taking into account the interactions between parameters. One of the most widespread methodologies to test process parameters and their interactive effects is the Response

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Surface Methodology (RSM) (Myers et al., 2009). This multivariate statistic method consists of designing a mathematical model that can exactly describe the overall process, in order to achieve best system performance (Maran and Manikandan, 2012; Cotana et al., 2015). However, if the process requires the optimization of several responses, the independent evaluation of each response cannot be the right way to find the best solution for all responses concurrently because improving one response can worsen the other one (Costa et al., 2011). For these cases, desirability function can be employed to solve this conflict, finding an optimal experimental condition to successfully fulfill the optimization of all responses (Viacava et al., 2015).

Although in the Literature there are several studies that involved torrefaction of biomass from different raw materials (e.g. oil palm waste (Aziz et al., 2012), wheat straw (Shang et al., 2012)), there had been only one work (Chen et al., 2012) that focused on the torrefaction of coffee residues, evaluating the influence of the torrefaction conditions on its properties and structures, but not defining the optimal set of the operating parameters.

Coffee is the second largest traded product in the world and a huge quantity and variety of residues is generated during processing from fruit to cup (Murthy and Madhava Naidu, 2012). The International Coffee Organization (ICO, 2016) estimated that about 9 million tons of coffee bean was consumed in 2016, the majority of which in the EU, USA, Brazil, and Japan [ICO]. Coffee by-products are obtained from coffee production (e.g. husk, pulp, parchment, mucilage), roasting industries (e.g. coffee silverskin) and also during soluble coffee preparation (spent coffee grounds) (Cruz et al., 2014). Two interesting coffee residues for the char production are the parchment skin, often referred to a coffee chaff (CC), that is a thin layer of endocarp, yellowish in colour, inside the coffee beans, and the spent coffee grounds (SCGs), which are mainly obtained from large facilities that process coffee bean to produce soluble coffee. CC represents about 4.2% (w/w) of coffee beans while, after brewing, 650 kg of SCGs are left per 1 ton of coffee green bean (Ballesteros et al., 2014). Most of these residues have still no special use, being mostly discharged into the environment (Santos et al., 2016). The employment of coffee wastes in value-added applications could give therefore new life to these materials. To date, several applications have been tested for coffee residues, mainly as biofuels, composts, animal feed, biosorbents and enzymes (Martinez-Saez et al., 2017). However Oliveira and Franca (2015) reported that there is still a need for significant research to make the energy recovery of coffee residues a technically and economically viable option. Since these residues are obtained at their processing facilities, the torrefaction pre-treatment can be carried out on-site, decreasing the transportation costs and improving the economic feasibility of the chain.

At the best of our knowledge, there are no papers using the desirability function approach to optimize the operating parameters of the torrefaction process. Thus, the aim of this study is to perform torrefaction for CC and SCG in a thermogravimetric analyzer, in order to find the optimization conditions based on minimizing the weight loss and maximizing the calorific gain. RSM was employed to examine the effects of torrefaction temperature, residence time, and heating rate on mass and energy yields of the solid products, investigating the chemical and physical properties of the torrefied biomass.

2. Materials and methods

2.1. Feedstock preparation

SCGs used in this study were supplied by a cafeteria in the province of Perugia (Italy) that uses a mixture of Arabica (*Coffea ara-*

bica) and Robusta (*Coffea canephora*) coffee seeds. CC was provided by a coffee company located in Pavia, Italy. Each byproduct was dried in an oven at 105 °C for 24 h until its water content was reduced to the mass fraction of about 5%. Both samples were ground using an ultra-centrifugal mill (mod. ZM200, Retsch) and sieved in order to obtain a particle size lower than 500 µm. The dried samples were then stored at room temperature in air-tight containers until use.

2.2. Torrefaction process

A thermogravimetric analyzer (TGA-701, LECO Co., USA) was employed to carry out the torrefaction tests. In each test, 0.3 g of raw material was placed in a ceramic crucible which was placed into the TGA. Nitrogen, at a flow rate of 3.5 L/min, was used as method process gas. The torrefaction test began at the temperature of 30 °C and then, by a specified heating rate, the samples were heated to the required torrefaction temperature; the materials were held at a specific residence time, depending on the experimental conditions defined in Section 2.5. The torrefied samples were extracted from the TGA when the temperature inside the furnace was lower than 100 °C, in order to avoid any oxidation of the char.

The weight loss of the torrefied biomass was calculated using the following equation:

$$W_L = \left[\frac{M_0 - M_T}{M_0} \right] * 100 \quad (1)$$

where W_L is the weight loss (%), M_0 is the initial mass of biomass before torrefaction and M_T is the residue mass after torrefaction.

2.3. Elemental analysis and energy value of biomass

Biomass properties were analyzed before and after torrefaction. In particular, raw materials were subjected to proximate, ultimate, and structural compositional analysis while torrefied samples were analyzed in terms of ultimate composition. The proximate analysis (moisture, ash, volatile matter, and fixed carbon content) was carried out in compliance with UNI EN 14774-2, UNI EN 14775, and UNI EN 15148 standard methods by using a thermogravimetric analyzer (TGA-701, LECO Co., USA). Ultimate analysis was performed by using a LECO Truspec CHN analyser, in compliance with UNI EN 15104 standard method.

The fiber compositional analysis (cellulose, hemicellulose, lignin) was carried out according to NREL laboratory analytical procedures (Sluiter et al., 2008), following the method adopted in a previous study (Buratti et al., 2015).

HHV of the samples was calculated by applying the model developed by Friedl et al. (2005), from their C, H, and N contents. In particular HHV was attained using the following equation:

$$\text{HHV (kJ/kg)} = 3.55C^2 - 232C - 2230H + 51.2C * H + 131N + 20,600 \quad (2)$$

where C, H, and N are the weight percentage obtained from the ultimate analysis.

All analytical procedures were performed in triplicate and a mean value was reported.

2.4. Thermogravimetric analysis

Thermal stability of the raw materials was evaluated by using a thermogravimetric analyzer (TGA-701, LECO Co., USA). Samples of about 0.2 g were heated from 30 °C to 900 °C under a nitrogen atmosphere, at a flow rate of 3.5 L/min and a constant heating rate of 10 °C/min.

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