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Impregnation of olive mill wastewater on dry biomasses: Impact on chemical properties and combustion performances

Nesrine Kraiem [a, b, c], Mejdi Jeguirim [a, *], Lionel Limousy [a], Marzouk Lajili [b], Sophie Dorge [c], Laure Michelin [a], Rachid Said [b]

[a] Institut de Sciences des Matériaux de Mulhouse, 15 rue Jean Starcky, 68057 Mulhouse, France
[b] UR Etude des Milieux Ionisés et Réactifs, IPEIM, Avenue Ibn El Jazzar, Monastir 5019, Tunisia
[c] Laboratoire Gestion des Risques, Environnement 3 bis, rue Alfred Werner, 68093 Mulhouse, France

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ABSTRACT

Mediterranean countries generate large amounts of olive oil byproducts mainly OMWW (olive mill wastewater) and EOSW (exhausted olive solid waste). Although solid residues have various valorization strategies, there is no economically viable solution for the OMWW disposal. This study aims to recover the OMWW organic contents through solid biofuels production. Hence sawdust and EOSW were used for the OMWW impregnation. The potential of the obtained samples, namely: IS (impregnated sawdust) and IEOSW (impregnated exhausted olive solid waste) were evaluated. Therefore, the physicochemical characterizations and thermogravimetric analyses of the samples were first performed. Secondly, the samples densification into pellets and their combustion in a domestic combustor were carried out. Combustion efficiencies, gaseous and PM (particulate matter) emissions as well as ash contents were evaluated.

The analysis finding shows that addition of OMWW leads to an increase of energy content through the heating values increase. An increase of the impregnated samples reactivity was observed and assigned to the potassium catalytic effect. Combustion performances show that the OMWW addition has not a negative effect on their firing quality. Moreover, a beneficial effect on the pollutant emissions is observed with IEOSW pellets. The developed strategy constitutes a promising issue for the OMWW disposal and recovery.

1. Introduction

In a context of sustainable development, the reduction of energy costs and the use of renewable energies potential based on processes that maximize energy efficiency and protect the environment are key challenges for industrial companies. With the depletion of fossil fuel resources, countries must move towards renewable energies for the development of their economy. In this framework, agro-industrial companies are prompted to reduce and/or to valorize the generated wastes from their activities. In this way, olive oil extraction industries, representing a significant economic and social activity in the Mediterranean countries, engendered two by-products: a solid residue and an aqueous effluent namely, OMSW (olive mill solid waste) and OMWW (olive mill wastewater), respectively [1].

The treatment of OMWW represents a serious ecological problem due to its high degree of organic pollution with a COD/BOD ratio (chemical oxygen demand/biological oxygen demand) evaluated to be between 2.5 and 5, its pH slightly acid [2] and its high content of recalcitrant compounds such as lignin and tannin [3]. Furthermore, OMWW contains phenolic compounds and long-chain fatty acid responsible for the phytotoxic and antibacterial effect. Tunisia like the Mediterranean countries produces large quantities of olive by-products estimated at 2009 by the Tunisian national agency for waste management, namely ANGED (Agence Nationale de Gestion des Déchets) to be annually: 600,000–1,200,000 tons of OMWW, 435,000–800,000 tons of OMSW and 120,000–170,000 tons of sludge [4]. Hence, Tunisia needs to identify an environmentally and economically viable solution for the generated waste disposal. Several OMWW disposal scenarios and methods have been studied in the literature based generally on biological or chemical-physical

* Corresponding author. Tel.: +33 3 89608661.
E-mail address: mejdi.jeguirim@uha.fr (M. Jeguirim).

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treatments, membrane filtration and evaporation. Few attempts have been tested to recover the OMWW energetic potential, but there is no economically issue for OMWW due to high moisture content [1,5]. The common disposal ways were soil spreading and evaporation ponds. Although, the latter technique is the most popular, the residual oil layer floating in the pond surface prevents water evaporation.

The treatment of the solid residue (OMSW) has been well investigated in literature [1,6]. It has been traditionally used as animal feed but nowadays it undergoes a second oil extraction by chemical processes in seed-oil factories in order to extract its residual oil, which leads to generation of EOMSW (exhausted olive mill solid waste). The thermal conversion is the main recovery process for EOMSW because of its interesting energy content with low heating value around 18 MJ kg^{-1} [7]. Separate treatments of OMWW and EOMSW have been studied by several authors [5–9], but nowadays fully combined treatment is required. Such strategy could develop a green and low cost energy in order to serve mills and seed-oil factories through small sized plants [2]. Addition of OMWW to suitable proportions of EOMSW following by biomass combustion is a promising solution which can avoid the factories to pay disposal costs [2]. Recently, Chouchene and Jeguirim established a combined method for the treatment of OMWW [10–12]. This method consisted on the impregnation of the OMWW on low cost adsorbents such as sawdust or OMSW. The impregnated samples were therefore thermally oxidized in laboratory furnace. The authors showed that the addition of OMWW to both adsorbents had not negative effect on gaseous emissions [11,12]. However, the CO (carbon monoxide) and VOC (volatile organic compounds) emissions from all the tested samples were higher due to the absence of secondary air injection in their laboratory reactor [11]. Although the obtained promising results, the validation of the OMWW treatment strategy required the investigation of the impregnated samples behavior during combustion tests in a domestic boiler.

In recent times, several researchers have examined the combustion of agro-industrial and agriculture residues in domestic boilers [13–17]. These biofuels were firstly densified and pelletized in order to obtain adapted fuels for the different boilers. Also, pelletization increases the biomass energy density and decreases the moisture content leading to an increase of combustion efficiency as well as a reduction of smoke during combustion [18]. Several researchers have produced pellets by blending the agro-industrial residues with wood residues. This step allows obtaining an agropellets with good quality that could be used directly in wood domestic boilers. In fact, the combustion performances as well as the gaseous emissions obtained during combustion tests could reach the European standards.

This investigation aims to validate the strategy of the OMWW treatment through its impregnation on different biomasses and the direct combustion of the impregnated samples for small-scale heat generation. Hence, the impregnation of OMWW was performed on sawdust and EOMSW for 5/1 mass fraction ratio. EOMSW is preferred to OMSW due its availability in seed-oil factories as well as its higher adsorption efficiency since the residual oil was removed. In order to reach this research purpose, firstly, characterization of the impregnated samples was performed using various analytical techniques as well through thermogravimetric analysis. Secondly, pellets from the different samples were produced and characterized according to the French and European standards. Finally, combustion tests for the different pellets were performed in a residential pellets boiler to compare their combustion efficiencies as well as gas and particle emissions.

2. Materials and methods

2.1. Samples preparation

OMWW and EOMSW used in this study were collected from olive mill (three-phase centrifugal olive mill) at the seed oil factory Zouila located in Mahdia, Tunisia. Sawdust was provided from sawmill located in Sayada, Tunisia. During impregnation tests, 20 kg of EOMSW or sawdust with 10% of moisture (in wet basis, wb) were slowly added to 100 kg of OMWW with 89% of moisture (wb) in a specific barrel. The impregnated samples were mixed regularly. In this specific investigation, in order to reduce the initial water content in the mixture which was initially 76% (wb) and therefore to accelerate the drying process, the barrel was heated, from the underside, using hot ashes provided by a combustor in the seed oil factory. The upper face of the barrel was exposed to ambient air. Currently, solar drying of the impregnated samples is examined.

At the end of drying, different samples were taken from 10 places for each mixture at time intervals of 2 h. Therefore, a homogeneous IEOSW (impregnated exhaust olive solid waste) and IS (impregnated sawdust) were obtained with less than 15% of moisture. This level 15% was chosen in order to avoid the fermentation of the different preparations. Samples of ISW and EOMSW are analyzed in the following in their raw state. During the analysis, all samples are placed in an isothermal container to maintain their properties and moisture content.

2.2. Samples characterization

2.2.1. Proximate, ultimate analyses and energetic contents

The optimization of an operating plant design for biomass combustion involves the knowledge of the fuel composition as well as its related energy properties. Hence, elemental compositions of the different prepared samples were performed by CHONS-NA 2100 protein CE instrument analyzer (Carbon, Hydrogen, Oxygen, Nitrogen, Sulfur).

Proximate analysis was obtained using different techniques. Therefore, the water content (M) is measured by weighting samples before and after drying at 105 °C (about 1 g, wb) in an oven until obtaining a constant mass according to the EN 14774-1 standard. The percentage of moisture content is calculated from the average of three measures per sample. Bulk density is carried out according the EN 15103 standard. The ash content is evaluated according to two norms in order to evaluate differences between them. Ash value is obtained after sample combustion (about 1 g, wb) in a muffle furnace during 3 h at two temperatures 550 °C and 815 °C according to EN 14775 and DIN 51719 standards respectively. The VM (volatile matter) content was obtained using the TG procedure analysis. This method consists of heating the samples under nitrogen flow at a heating rate of 10 °C min^{-1} from 20 °C to 110 °C and maintaining at this temperature during 10 min to remove the moisture. Temperature is then increased at 20 °C min^{-1} to 900 °C and kept for 10 min to obtain the weight loss corresponding to the volatile matter content. The fixed carbon content is obtained by difference.

The energy contents of the different samples were obtained using a calorimetric bomb IKA-C200 by determining the HHV (high heating values). HHV were obtained after combustion of a sample (about 0.6 g, wb) under a pure oxygen atmosphere at 35 bars according to EN 14961-1 specification. The LHV (low heating values) were calculated using the relationship between HHV and LHV given by:

$$\mathrm{LHV} = \mathrm{HHV} - \mathrm{hg}(9H + M)/100 \qquad (1)$$