



Full Length Article

Characterization of biomass ashes produced at different temperatures from olive-oil-industry and greenhouse vegetable wastes



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HIGHLIGHTS

- Detailed characterization and comparison of novel biomass ashes were given.
- Ash from greenhouse-vegetables waste was richer in aluminosilicates and calcite.
- Ashes from olive-oil-industry wastes were richer in soluble potassium minerals.
- Increasing of ashing temperature decreased the size and surface area of particles.
- High combustion temperature increased hydrophobicity of biomass ashes.

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ABSTRACT

Reuse and recycling of biomass ashes as new resources can boost the development of a circular and sustainable economy. This requires knowledge concerning the characteristics and properties of the ash for different applications. In this study, novel biomass ashes from combustion, at different temperatures, of olive-oil wastes and from the gasification of greenhouse vegetables wastes were characterized and compared by ICP-MS, XRF, XRD, FT-IR, and SEM-EDS among other analytical techniques. All ashes were alkaline (pH between 9.9 and 13.3) and had high salinity (42–132 dS m⁻¹). They had high amounts of Si, Ca, and K; moderate of P, Mg, Al, S, and Cl; and low Na and heavy metals. By comparison, the ash from greenhouse vegetables waste was richer in aluminosilicates and calcite whereas those from olive-oil wastes showed the presence of soluble minerals such as carbonates, sulphates, and chlorides. The high K amounts (23–58 g kg⁻¹) found in the ashes from olive wastes would enable its reuse as natural K fertilizer or as a raw material in the manufacture of K chemical fertilizers. The ashes resulting from higher temperatures had lower carbon contents, higher amounts of particles <250 μm, smaller surface areas and higher hygroscopicity which would influence the use of these materials as potential catalyst or sorbents. High combustion temperatures favoured the presence of numerous spherical particles of different sizes, associated with the presence of silica, and also particle aggregates coated by laminar microcrystalline particles. The ashes produced at lower temperatures were dominated by plane crystalline particles and other large, long, carbonaceous shapes with reticular structures.

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1. Introduction

Renewable energies, especially biomass fuel, are increasing globally due to the environmental problems induced by the fossil fuels, such as the CO₂ emissions, acid rain, and the holes in the ozone layer. The worldwide annual biomass production with potential energy is estimated around 3 gigatonnes of forest wastes, 1.1 Gt of municipal solid wastes and between 1.1 and 3.1 Gt of agricultural wastes [1]. Today, biomass represents 8–15% of the

energy supplied worldwide and it is estimated to grow to 33–50% by the year 2050 [2]. Biomass ashes are the final products accumulating from the thermal combustion and gasification of biomass for heat and electricity production. The disposal of ashes in landfills is still a common practice, which causes considerable costs for the biomass-based power plants, provokes serious environmental problems, and shows disregard for the recycling the potential of the ashes. The recycling and reuse of the biomass ashes as added-value materials, for different productive sectors is essential from an economic and environmental perspective, reducing costs for biomass power producers, making it unnecessary to use non-

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Nomenclature

GV	fly ash from gasification of greenhouse vegetable wastes	CEC	cation-exchange capacity
DO	fly ash from combustion at 450 °C of dry olive cake	CCE	calcium carbonate equivalent
WOL	fly ash from combustion at 450 °C of wet olive cake mixed with olive leaves	ICP-MS	inductively coupled plasma mass spectrometry
WOB	fly ash from combustion at 850 °C of wet olive cake mixed with bagasse	XRF	X-ray fluorescence
OP	ash from combustion at 200 °C of olive pits	LOI	loss on ignition
EC	electrical conductivity	SSA	specific surface area
TC	total carbon	XRD	X-ray diffraction
TOC	total organic carbon	FT-IR	fourier transform infrared spectrometry
TKN	total kjeldhal nitrogen	SEM/EDS	scanning electron microscopy with an energy dispersive X-ray spectrometer

renewable resources and favouring the development of a circular and sustainable economy [3,4].

The region Andalusia (southern Spain) is the world's largest producer of olive oil (0.8–1 Mt olive oil/year). Wet olive cake (between 3 and 4 Mt/year) is the main waste produced during the olive oil extraction. This waste is composed of olive pulp, pits, skins, and water. After removal of the pits, part of this waste is dried and treated with solvents to make the olive-cake oil and recalcitrant waste called dry olive cake. In addition, this region is a leader in Europe in greenhouse crops of fruit and vegetables with 48 10³ hectares, which generate greenhouse vegetable wastes between 28 and 30 t/ha/year. Unlike the situation in other world regions, Andalusia has 15 power plants that use, as biomass fuel, wastes from the olive oil industry and from the greenhouse vegetables wastes, due not only to the extent of the two economic activities in this region but also to the low heating value of the wastes generated (4250 kcal kg^{−1} for wet olive cake, 4300 kcal kg^{−1} for dry olive cake, 4500 for olive pits and 3000 for greenhouse vegetable wastes) [5,6].

The characterization of biomass ashes is essential in order to ascertain the possible reuse of these materials for different applications. In general, the biomass-ash properties depend on the composition of the biomass fuel, the technologies used for energy production and the storage conditions [7]. Currently, ample information is available on the characteristics and properties of ash from woody biomass combustion, and to a lesser extent of ash from agroindustrial and municipal wastes [1,8–11]. However, limited and partial information is available on the characteristics of biomass ashes from the combustion of olive wastes [12,13] and there are no previous studies on ashes from greenhouse vegetable wastes.

The objective of the study was to compare the physical, chemical, mineralogical, and morphological characteristics of ashes produced from different agroindustrial wastes, which are used as biomass fuel for the production of electricity or hot water in Andalusia (Spain). For this, five novel ashes were selected according to different combustion temperatures (200 °C, 450 °C, and 850 °C) of contrasting olive-oil wastes (olive pits, dry olive cake, and wet olive cake) and from gasification at 270 °C of greenhouse vegetables wastes. The resulting information should be useful for ascertaining the properties and potential applications of these new ashes, which are commonly, stored at sites adjacent to the biomass power plants.

2. Material and methods

2.1. Biomass ash samples

Representative ashes produced at different temperatures from five different biomass fuels were assayed. Three biomass fly ashes were collected from combustion of olive-oil wastes in biomass

power plants located in Andalusia (Spain) (DO, dry olive cake at 450 °C; WOL, wet olive cake mixed with olive leaves at 450 °C, and WOB, wet olive cake mixed with bagasse at 850 °C). One bottom ash was collected from gasification at 270 °C of greenhouse vegetable wastes at a different Andalusian power plant (GV). The greenhouse vegetable wastes consisted mainly of plant debris of tomato, bean, pepper, cucumber and zucchini. Detailed information on the production characteristics of GV, DO, WOL, and WOB from the four biomass power plants is shown in Table 1. Finally, the fifth ash was obtained from low-temperature combustion (200 °C) of olive pits (OP) in a fixed-grate boiler for heating water in an olive-oil extraction plant. Five samples of 10 kg of GV, DO, WOL, and WOB were collected from various locations in the ash heaps at landfill sites adjacent to the biomass power plants. The five samples of each ash were mixed thoroughly to provide a representative ash sample. The OP ash was collected at the bottom of the boiler. All ash samples were stored in hermetically sealed plastic buckets and kept far from heat and light sources. Later, the ash samples were air-dried, sieved through a 2-mm steel sieve and analysed without any pre-treatment. The amount of the >2 mm fraction of each ash was as follows: GV: 68.7 ± 0.2%; DO: 18.1 ± 0.2%; WOL: 0.5 ± 0.04%; WOB: 6.5 ± 0.2%; OP: 44.7 ± 0.1%. All physical and chemical (except XRF analysis) analyses were run using triplicate samples of each ash.

2.2. Chemical analysis

The pH and EC were measured using mixtures of ash and water 1:2.5 and 1:5, dw:v ratio, respectively. CEC was determined by the ammonium acetate (pH 7) displacement method [14]. CCE was determined by analyzing the evolution of CO₂, after reaction with dilute hydrochloric acid, in a Bernard calcimeter [14]. TOC was determined by dichromate oxidation of the samples and subsequent titration with ferrous ammonium sulphate [14]. TKN was determined by semimicro-Kjeldahl digestion [14]. The total concentration of P, K, Ca, Mg, Na, Fe, Mn, Cu, Mn B, Cd, Ni, and Pb were determined by inductively coupled plasma mass spectrometry (ICP-MS) Perkin Elmer Optima 4300 DV after digestion of samples in concentrated aqua regia [15]. In addition, major elements were determined using an X-ray fluorescence spectrometer (XRF) type Philips MagiX PRO model PW2440 XRF with Rh 4 KW tube. Weight loss on ignition (LOI), as the measure of the amount of the unburnt carbon and carbonates remaining in the biomass ash, was calculated by the difference between the weight after the drying stage at 110 °C and after the firing stage at 950 °C.

2.3. Physical analysis

Particle size was determined after the organic matter was destroyed and the ash was dispersed with sodium hexametaphos-

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