15 (2022) 200083

Contents lists available at ScienceDirect

Resources, Conservation & Recycling Advances

journal homepage: www.sciencedirect.com/journal/Resources-Conservation-and-Recycling-Advances

Recycling of agricultural (orange and olive) bio-wastes into ecofriendly fertilizers for improving soil and garlic quality

Panuccio MR [a], Marra F [a], Maffia A [a], Mallamaci C [a], Muscolo A [a,*]

[a] Department of AGRARIA, Mediterranea University, Feo di Vito, 89122 Reggio Calabria, Italy

ARTICLE INFO

Keywords:
anaerobic digestion
composting
orange residue
olive pomace
soil fertility

ABSTRACT

Agricultural waste material even if free from toxic compounds or pathogens can cause environmental problems and their unsustainable use can lead to health and environmental risks. Orange and olive food processing wastes are rich in chemical compounds and could offer many opportunities of use, especially for the high level of nutritional components. This paper proposes to validate anaerobic digestion, aerobic digestion, and raw agricultural waste management as possible ecofriendly methods to turn these wastes into fertilizers. The byproducts obtained by these three different processes, have been chemically analyzed and assessed on soil, growth and antioxidant properties of garlic (*Allium sativum*). Results evidenced that the chemical properties of the soil treated with all the byproducts were positively influenced, even if the effects were different and depended on the type of the byproduct used and on the organic wastes from which the byproducts came from. The byproducts coming from orange wastes (pastazzo) were a bit more effective than those coming from olive pomace and among the byproducts the compost was the best one. Results evidenced that garlic increased its growth and antioxidant capacity when cultivated with all byproducts. The results of this study evidenced that all the byproducts obtained can be used in agriculture with success and the transformation methods used even if differently, are environmentally, economically and/or agriculturally valid.

1. Introduction

Agricultural production is considerably increased over the last 50 years due to the extension of cultivated lands, the increase in technology to enhance the productiveness, and the rise of world population (FAO, 2017; OECD/FAO, 2019). Agriculture is a sector that produces about 23.7 million food tons per day over the world contributing for more than 21% to greenhouse gases emissions (Gerber et al., 2013). The increase in agricultural production is influencing the environment, affecting negatively soil, air and water resources. Nowadays, the new global challenge is to reduce the environmental degradation adopting more ambitious and rapid measures to achieve, in the next 10 years, the goals established by the United Nations 2019 (Global Sustainable Development Report, 2019). EU Green Deal policy set out the trajectory to be climate neutral by 2050. As a milestone towards this target, the EU Commission proposed a 2030 target to reduce greenhouse gas emissions by 55 per cent compared to 1990. The European Green Deal aims to boost the efficient use of resources by moving to a clean, circular economy and stop climate change, revert biodiversity loss and cut pollution, by reducing, reusing and recycling (EU, 2020). The required measures have to consider the criteria of sustainability, focusing mainly on: an efficient reuse of wastes, a lower emission of polluting gases and a reuse of solid wastes (EC, 2019; Ferronato and Torretta, 2019; O'Connor, 2021).

Italy is the second largest European orange and olive producer after Spain, and processes approximately 800 000 tons per year (tpy) of orange, with a waste production of approximately 500 000 tpy, and 3.500.000 tpy of olives with a production of more than 2000 tpy of olive oil wastes (Prosodol, 2012). Orange and olive food processing wastes even if free from toxic elements or pathogens can negatively affect the environment for their high content of polyphenols, low pH value, and elevated salt concentration (Belligno et al., 2005; Doula et al., 2012; Ashraf et al., 2014; Khdair et al., 2019). Their unsustainable use can lead to healthy problems and environmental costs associated with illegal landfills and illegal management of wastes (export activities), as well as additional costs due to a non-realized circular economy market development (COWI, 2019). At the same time, orange and olive wastes, for their chemical composition, especially for the high level of nutritional components, could offer many opportunities of use.

* Corresponding author: Telephone number: 003909651694364, Fax number: 003909651694550
E-mail address: amuscolo@unirc.it (M. A).

https://doi.org/10.1016/j.rcradv.2022.200083

Available online 4 May 2022

Orange residues contain water (75–85%), mono- and disaccharides (6–8%), and a limited level of oils in the peel waste (Wikandari et al. 2015; Moncada et al., 2016; Restrepo-serna et al., 2018). To valorize the orange wastes (OW), new technical solutions such as pectin extraction (Fakayode and Adobi, 2018), dietary fiber extraction (Sang et al., 2021), biogas production (Rokaya et al., 2019) and essential oil (particularly D-limonene) extraction (Siddiqui et al., 2021) are nowadays utilized to convert potential environmental hazards and economic issues (Palmeros Parada et al., 2017) into resources, however, the economic viability of these alternatives is not ensured because of the high-energy costs of these processes. Ortiz-Sánchez et al. (2021) evaluated from experimental, technical, and economic perspectives the production of essential oil, pectin, and biogas from OW and their results evidenced that the value-added products can be obtained in energetic and economic feasible way, mainly at low scales.

For the above-mentioned reasons, at present, only a small part of these biowastes is used to recover bioactive compounds for food, cosmetic and pharmaceutic industries, to produce green energy and animal feed, while the majority of these wastes are delivered to landfills yet.

Olive mainly used to produce oil, moreover in Mediterranean countries, generates enormous quantity of wastes not only wood, branches, leaves but also by-products (olive pomace, olive mill wastewater, olive stones) with negative environmental impact and high costs for management and disposal (Galanakis, 2017).Olive wastes and by-products rich in nutrients phenols, hemicelluloses, fat and proteins, were in the past spread on the land, but now much researches have been done to develop newer and higher biotechnological pathways to economically valorize the byproducts, by using thermochemical (via pelleting or pyrolysis, for heat and electricity) or (bio)chemical (fractionation, extraction, anaerobic digestion, for e.g., bioethanol, biophenols, biofertilizers, biogas) processes (Negro et al., 2017). New tendency consists of using olive by-products as food additives or nutraceuticals in the food and pharmaceutical industries, but the conversion technologies and the new proposed waste valorization methods are not yet economically feasible and implemented at an industrial scale.

The competitiveness of orange and olive processing industries, that generally spend a lot of their annual budget to residue treatment, can become feasible if environmentally and economically waste system management that combines efficient low-cost technology for their treatment and valorization will be adopted. Among the different waste-valorization techniques, composting is an easy, cheap biological process that can be used to convert recalcitrant biomass as orange and olive wastes into humus like substances under controlled optimum environmental conditions. Canet et al. (2008), during 9-10 month of olive waste composting (olive waste 50% and animal manures 50%), observed a decrease in organic matter and an increase in the concentrations of nutrient and humic substances, together with large increases in pH and salinity, the latter represent a great disadvantage for agronomy. In a previous research, Muscolo et al. (2018), using olive pomace at 90% highlighted that compost maturity can be mainly linked to composting setup parameters, rather than to raw material composition. Gelsomino et al. (2010) showed that after 5-month of aerobic bioconversion, orange waste reached an acceptable degree of maturity but the addition of orange compost to the soil selectively increased pH and electrical conductivity (EC) with negative consequence on plant growth. Other authors using different typologies of organic waste-derived fertilizers showed that the addition to soil, improved soil fertility, crop and residue yields in respect to the characteristics of the fertilizer used (Rigane et al., 2011; Beeby et al., 2020; Bhunia et al., 2021).

Muscolo et al. (2019) using three different methods to transform olive pomace (coming from a two-phase olive extraction plants) into fertilizers, evidenced that the efficiency of the fertilizer produced depended mainly on its chemical properties.

Based on the above statements, in this work we have used the three different waste transformation processes (aerobic, anaerobic digestions and crude waste pelleting) with the idea that is the intricacy of the method to determine the quality of the organic fertilizer output. The novelty of this study is to use two new recalcitrant agricultural wastes, that differ for their chemical characteristics olive pomace, coming from the three-phase olive extraction plants and orange waste (pastazzo) coming from citrus transforming industry to 1) verify if the three methods were equally and universally applicable to different biomasses; 2) check if there was a biomass -method specificity 3) assess if the efficacy/efficiency of the fertilizers produced on soil properties, growth and antioxidant properties of garlic (*allium sativum*) depended on the processes or on the own chemical characteristics.

The aim is to use the results of this study to provide company and other stakeholders with different solutions, economically and environmentally sustainable, to manage, selectively and on the basis of their potentiality the feedstock to increase the economy and the competitiveness of the agricultural sectors.

2. Materials and Methods

2.1. Olive Pomace and Orange Waste Chemical Characterization

The chemical properties of olive pomace (traditional three phases olive oil extraction process) and orange residue (pastazzo) have been detected following the methods reported in Muscolo et al. (2017). Heavy metals have been measured by using a Shimadzu model AA-680 atomic absorption spectrometer (Japan) (Pourjavid et al., 2014).

2.2. Aerobic digestion

Two separate processes to compost olive pomace and pastazzo were performed in bins, in triplicates (Muscolo et al., 2018, 2019). 1) 90% of pulp and kernel of olives and 10% of straw 2) 80% orange waste and 20% of straw were used. The choice of 90% for olive pomace and 80% for orange wastes respond to the exigence of using wastes as much as you can to verify their compostability and timing. The composting parameters used to compost both the agricultural wastes are indicated in Muscolo et al. (2018, 2019). In short, the composting parameters were setup as follow: a mesophilic temperature phase for 8 days at 29°C, a thermophilic temperature phase for 20 days at 50°C and a mesophilic temperature phase for 92 days at 27°C. The moisture was maintained at 50% and the oxygen percentage was >15%. The composting process took 4 months to obtain stable organic mixture. The time of composting was set up on the basis of results of previous experiments. Compost was air desiccated, sieved at 2 mm and homogenized.

2.3. Crude agricultural waste management

Pellets of 3/4 mm diam. were made by Steel Belt System s.r.l. as described in Muscolo et al. (2017a, 2019). The mixtures formed by 85% of liquid sulfur, 10% of bentonite clay (as support and carrier), and of 5% olive pomace or 5% pastazzo were pastilled in a special belt system.

2.4. Anaerobic digestion

Biogas energy plants (998 kWel), were in charge of Fattoria della piana s.r.l. The digester (U) 4240m^3 was loaded with olive waste 50%, animal manure and maize silage (50%) The digester (O) 3260m^3 was filled with orange waste 50%, and animal manure and maize silage (50%) (Panuccio et al., 2016; Muscolo et al., 2017b). In short, Biogas plant operators have selected process temperatures and retention times which are appropriate for the feedstock that had to be digested. Digester O: Process temperature: 40°C, pH 7.8, total volume of the digester: 7500 m^3, total volume loaded per day: 120 m^3/day, hydraulic retention time (HRT): 60 days, minimum guaranteed retention time (MGRT) 16 h at 40°C. Digester U: process temperature: 40°C, pH 8.0, total volume of the digester: 7420 m^3 total volume loaded per day: 120 m^3/day, hydraulic retention time (HRT) 60 days, minimum guaranteed retention time (MGRT) 16 h at 40°C. The digestates obtained were chemically characterized as reported in Muscolo et al. (2017b, 2019).

Composts, digestates and sulfur-based pads were analyzed as reported in Panuccio et al. (2016) and Muscolo et al. (2017a, 2019). The chemical characteristics of digestate, compost and sulfur-based pads with olive pomace or pastazzo are shown in Tables 3-4.

2.5. Soil analysis

Potted soils before treatment (CTR0) and six months after the different treatments were analyzed for physical and biological properties. Soil from Motta San Giovanni, Loc. Liso, Italy (LAT:38°0′15″12 N; LONG: 15°41′45″24 E) has a sandy-loam texture (11.85% clay, 23.21% silt, and 64.94% sand) as stated by FAO soil classification system (FAO, 1999). Moisture content were obtained drying the soil at 105°C (AOAC, 2005); pH and electric conductibility were detected in distilled water (AOAC, 2005); organic carbon was determined with Walkley–Black procedure (1934), and transformed into organic matter multiplying by 1.72; total nitrogen was assessed with Kjeldahl method (1883). Carbon content of humic and fulvic acids (Bettany et al. 1980) was assayed by Nelson and Sommers (1982) method. Water-soluble phenols were measured following the Box method (1983). Cationic exchange capacity (CEC) was determined with Mehlich methodology (1953). Fluorescein diacetate hydrolysis (FDA) reaction was determined as reported in Adam and Duncan (2001). Microbial biomass C (MBC) was detected in fresh samples (equivalent to 20 g DW) (Vance et al., 1987). Soluble organic C in the fumigated and unfumigated soil extracts were detected with the Walkley and Black method (1934). Soluble carbon was converted into biomass by using an extraction efficiency coefficient of 0.38 (Vance et al., 1987). Dehydrogenase (DH) activity was detected according von Mersi and Schinner (1991) method.

2.6. Plant material

Garlic plants were grown for six months in pots (30 cm diameter) filled with 9 kg of sandy-loam soil with the addition of: 160 g composted pastazzo or olive pomace, Pots were amended with S-bentonite + orange residue (SBOr); S-bentonite + olive pomace (SBOp) pads at the concentration of 1.4 g corresponding to 476 kg S ha^{-1} dose generally used in agriculture to lower the pH and to replenish S (Severson and Shacklette, 1988; Muscolo et al., 2017). 50% (w/w) of orange or olive digestate. Not amended soil was used as control**.** The quantity of compost, sulfur-based pads and digestate (C) have been selected on the basis of previous in vitro and in field results showing their better efficiency on soil and plant. (data not shown). The experimental design consisted of six pots for each treatment. The experiment was conducted in glass house to protect soil from rainfall, managing the irrigation system to maintain 70% of field capacity at a temperature of 25°C. After six months from sowing, the measured growth parameters of garlic plants were: leaf number and length (cm), root length (cm), bulb diameter (cm).

2.7. Preparation of garlic extracts

The extracts prepared as described by Kang (2015) were modified as reported in Muscolo et al. (2020). All extracts have been executed in triplicate.

2.8. Determination of total phenolic compounds and total flavonoids

Total phenols were measured using the Folin-Ciocalteu assay with a few changes (Muscolo et al., 2020). Sample absorbance were measured at 760 nm. Phenol content was expressed as mg gallic acid/100 g FW on the basis of a standard curve obtained with gallic acid (0-200 mg/L).

Flavonoids were tested with the colorimetric method of Djeridane et al. (2006), and expressed as rutin (R) E/g FW on the basis of a calibration curve obtained with rutin (Muscolo et al., 2020).

2.9. Antioxidant activity detection

DPPH• scavenging assay was assayed as reported in Papalia et al. (2017). DPPH activity was expressed as μM of Trolox (T) equivalents (E) using a calibration curve (1.0 to 50 μM T).

The ABTS assay (TE antioxidant capacity assay TEAC) was in accord with Re et al. (1999) method. Sample absorbance was measured at 734 nm. TEAC activity was expressed as μM Trolox (T) equivalents (E) using a reference curve (in the range from 1.0 to 50 μM) of T.

The oxygen radical absorbance capacity (ORAC) assay was carried out as in Muscolo et al. (2020). ORAC values were expressed as μmol TE mg^{-1} FW using a Trolox (10–100 μmol L^{-1}) calibration curve. All reagents were purchased from Sigma Aldrich Chemical Co.

2.10. Statistical analysis

Data are expressed as means ± standard error. Statistical analyses were processed using one-way analysis of variance (ANOVA) and means were compared with the Tukey's test ($P < 0.05$). Two-way ANOVA was used to test the effects of the factors (byproducts and wastes) on selected soil properties and on the antioxidant activity and bio-compounds of garlic. Data were processed with SYSTAT 13.0 for Windows (SPSS Inc.).

3. Results and Discussion

3.1. Waste and fertilizer chemical characteristics

The chemical characterization of these two wastes (Table 1) evidenced substantial and significant differences ($p \leq 0.05$) showing that olive pomace contained significantly more carbon, nitrogen and total phenols, conversely pastazzo contained a greater amount of nutrients and less amount of sodium and chloride. Electric conductivity and pH were not significantly different between the two agricultural wastes (Table 1). The content of heavy metals as Pb, Ni, Cd, Cr, and Co (Table 2) were low or negligible, so the use of both wastes for the production of fertilizers would not constitute an environmental and a healthy risk due to the accumulation of these elements in soils and plants. The chemical properties of both agricultural wastes fall in any case within the ranges commonly reported in literature for these materials (Mari et al., 2003; Doymaz et al., 2004; Manios et al., 2004). Currently in Italy in the biogas process, despite the high content of nutrients and carbon, only a small part of these wastes is recycled (Hollins et al., 2017), mostly instead are uncontrollably disposed on agricultural land with environmental damage due to uncontrolled fermentations that can lead to the production of

Table 1
Chemical properties of olive pomace and orange residue. The data are the mean of three replicates ± standard error (n=6). Different letters in the same row indicate, significant differences (Tukey's test, $^*p \leq 0.05$).

Chemical properties	Olive pomace	Orange residue
pH	5.03^a ± 0.1	5.16^a±0.2
E.C (mS/cm)	12.00^a ±1.1	10.00^a±0.9
Moisture (%)	86.70^a±3.2	83.60^a±2.9
C (%)	59.62^a±1.9	48.62^b±2.5
Total N (%)	1.29^b±0.2	2.00^a± 0.3
C/N	29.81^b±1.9	37.7^a ±1.7
Na^+ (mg g^{-1} dw)	1.95^a±0.5	0.97^b±0.2
NH_4^+ (mg g^{-1} dw)	0.23^b±0.03	0.33^a±0.04
K^+(mg g^{-1} dw)	38.22^b±2.3	49.22^a±2.6
Mg^{2+} (mg g^{-1} dw)	2.03^b±0.4	4.23^a±0.7
Ca^{2+} (mg g^{-1} dw)	2.33^b±0.7	9.33^a ±1.0
Cl^- (mg g^{-1} dw)	3.73^a±0.5	2.44^b ±0.6
PO_4^{3-}(mg g^{-1} dw)	2.00^a±0.4	1.09^b ±0.3
SO_4^{2-}(mg g^{-1} dw)	nd	nd
Water soluble phenols (mg TAE g^{-1} dw)	1.80^a±0.4	0.53^b±0.2

Table 2
Heavy metals (mg/kg) in olive pomace and orange residue. The data are the mean of three replicates ± standard error (n=6). Different letters in the same row indicate, significant differences (Tukey's test, *p ≤ 0.05).

Heavy metals	Olive pomace	Orange residue
Cadmium	4.0^{a} x10^{-4}	4.5^{b} x10^{-6}
Lead	6.0^{a} x10^{-3}	5.0^{b} x10^{-5}
Zinc	2.2^{a} x10^{-2}	4.2^{b} x10^{-5}
Nichel	1.0^{a} x10^{-3}	2.0^{b} x10^{-4}
Mercury	7.0^{a} x10^{-3}	2.0^{b} x10^{-5}
Copper	nd	4.7×10^{-5}
Chromium	nd	$< 5 \times 10^{-8}$

toxic intermediate chemical compounds. (Doula et al., 2012). Slorach et al. (2019) evidenced that the application of digestate to land with the release of ammonia and nitrate leaded to higher marine eutrophication, terrestrial acidification and particulate matter formation (Agapiou et al., 2016). Satari et al. (2018) highlighted the importance to valorize citrus wastes to reduce their negative environmental impacts and, to achieve a circular bio economy creating additional profit.

The chemical analysis evidenced different chemical characteristics among compost, digestate and sulfur-based pads produced from both wastes, (Tables 3 and 4). pH was mostly alkaline in digestate. EC was low and similar in all the byproducts analyzed. Organic carbon content was significantly higher in compost and digestate than sulfur-based pads. Total nitrogen was more abundant in the digestate from olive (5.7%) and orange (5.2%) wastes followed by compost from olive (2.5%) and orange (2.7%) wastes and pads with olive (0.2%) and orange (0.9%) wastes. C/N ratio was much higher compost (18.1) than digestate and pads particularly in those coming from orange waste (Table 3). Nutrients were more concentrated in compost than in the other byproducts and mainly in compost from orange waste. The greatest amount of water-soluble phenols was in the digestate and much more in that produced by olive pomace. No significant differences in ON/TN ratio have been observed among the three byproducts coming from the transformation processes of both wastes. NH_4^+-N/NO_3^-N ratio was in absolute the highest in all the byproducts coming from the transformation process of olive pomace (Table 4). It was the lowest in the digestates and the highest in sulfur-based pads coming from both biomasses.

Table 3
Chemical characteristics of compost, digestate, and pads coming from olive wastes. The data are the mean of three replicates ± standard deviation(n=9). Different letters in the same row indicate significant differences (Tukey's test, *p ≤ 0.05).

Chemical characteristics	Compost	Digestate	Pad
pH	6.3^{b*}±0.05	8.5^{a}±0.20	6.4^{b}±0.18
Bulk Density (Kg/m^3)	598^{b}±9.0	788^{a}±8.2	nd
E.C (mS/cm)	1.3^{a}±0.25	1.3^{a}±0.20	1.0^{a}±0.10
Moisture (%)	47^{b}±3.2	64^{a}±7.1	nd
C (%)	44^{a}±2.40	45^{a}±1.40	2.5^{b}±0.14
Total N (%)	2.5^{b}±0.22	5.7^{a}±0.20	0.2^{c}±0.03
C/N	17.6^{a}±1.6	7.9^{c}±0.5	12.5^{b}±1.4
Na^+ (mg g^{-1} dw)	1.0^{a}±0.06	0.9^{a}±0.08	0.16^{b}±0.04
NH_4^+ (mg g^{-1} dw)	0.08^{a}±0.02	0.04^{a}±0.02	0.06^{a}±0.10
K^+(mg g^{-1} dw)	17^{a}±1.50	0.58^{b}±0.02	0.39^{c}±0.04
Mg^{2+} (mg g^{-1} dw)	1.40^{a}±0.06	0.58^{b}±0.08	0.49^{b}±0.03
Ca^{2+} (mg g^{-1} dw)	2.5^{a}±0.3	1.6^{b}±0.2	0.13^{c}±0.01
Cl^- (mg g^{-1} dw)	nd	0.68^{a}±0.07	0.18^{b}±0.01
NO_2^-(mg g^{-1} dw)	nd	nd	nd
NO_3^-(mg g^{-1} dw)	0.10^{b}±0.002	0.41^{a}±0.03	0.10^{b}±0.0001
PO_4^{3-}(mg g^{-1} dw)	0.43^{a}±0.03	0.47^{a}±0.06	0.13^{b}±0.02
SO_4^{2-}(mg g^{-1} dw)	0.27±0.02	nd	nd
S (%)	nd	nd	85±6
Water soluble phenols (mg TAE g^{-1} d.w)	2.44^{b}±0.06	5.24^{a}±1	1.23^{c}±0.13
ON/TN	93^{a}±5	92^{a}±8	70^{b}±3
NH_4^+-N/NO_3^--N	2.81^{a}±0.13	0.34^{c}±0.07	2.14^{b}±0.11

Table 4
Chemical characteristics of compost, digestate, and pads coming from orange wastes. The data are the mean of three replicates ± standard deviation (n=9). Different letters, in the same row indicate, significant differences (Tukey's test, *p ≤ 0.05).

Chemical characteristics	Compost	Digestate	Pad
pH	7.6^{b*}±0.5	8.3^{a}±0.8	6.8^{c}±0.18
Bulk Density (Kg/m^3)	558^{b}±12	758^{a}±11	nd
E.C (mS/cm)	1.8^{a}±0.2	1.5^{a}±0.4	1.3^{a}±0.10
Moisture (%)	44^{b}±3	69^{a}±7	nd
C (%)	49^{a}±2.4	47^{a}±1.4	2.8^{b}±0.14
Total N (%)	2.7^{b}±0.8	5.2^{a}±0.9	0.9^{c}±0.03
C/N	18.1^{a}±1.6	9^{b}±0.9	9.3^{b}±1.4
Na^+ (mg g^{-1} dw)	1.0^{a}±0.2	0.8^{a}±0.1	0.12^{a}±0.04
NH_4^+ (mg g^{-1} dw)	0.03^{b}±0.01	0.03^{b}±0.01	0.09^{a}±0.01
K^+(mg g^{-1} dw)	18^{a}±1.3	3.58^{b}±0.5	1.32^{c}±0.04
Mg^{2+} (mg g^{-1} dw)	1.80^{a}±0.05	0.89^{c}±0.06	1.41^{b}±0.02
Ca^{2+} (mg g^{-1} dw)	2.9^{a}±0.2	1.8^{b}±0.1	1.1^{c}±0.01
Cl^- (mg g^{-1} dw)	nd	0.48^{a}±0.05	0.11^{b}±0.02
NO_2^-(mg g^{-1} dw)	nd	nd	nd
NO_3^-(mg g^{-1} dw)	0.1^{b}±0.01	0.32^{a}±0.02	0.2^{b}±0.0001
PO_4^{3-}(mg g^{-1} dw)	0.90^{a}±0.03	0.63^{b}±0.04	0.3^{c}±0.02
SO_4^{2-}(mg g^{-1} dw)	0.87±0.02	nd	nd
S (%)	nd	nd	85±6
Water soluble phenols (mg TAE g^{-1} dw)	1.3^{b}±0.6	2.13^{a}±0.5	1.0^{b}±0.13
ON/TN	99^{a}±3	93^{a}±5	67^{b}±3
NH_4^+-N/NO_3^--N	1.05^{b}±0.13	0.31^{c}±0.05	1.55^{a}±0.2

3.2. Fertilizer efficacy on soil

When added to soil, all treatments influenced positively the soil chemical properties compared to control, even if the effects were different and depended on the type of byproduct used and also on the starting organic wastes from which the byproducts came from. Regardless of the type of initial organic waste, the compost was the byproduct with the best effects on soil. Compost coming from orange waste was in absolute the most effective (Tables 5 and 6). Compost influenced more soil characteristics, enhancing significantly and concomitantly the amount of organic matter, nitrogen, CEC, hydrolytic and oxidative soil activities as well as MBC (Tables 5, 6). In soils treated with compost, the humification process predominated as explained by the greater value of HC compared to FC value. The increase of key soil properties due to compost addition can be related to the composition of compost itself. Compost contained more organic nitrogen, ammonium, and nutrients compared to the other byproducts, and even if the content of organic matter can be similar to that of digestate the compost contains more stable organic matter as indicated by C/N ratio and this is the reason of organic matter increase in treated soils. Additionally, when an organic matter is added to soil, microorganisms use it as energy source hence, only the less degradable fraction of OM that remains and contributes to soil organic matter. This stable fraction of the original OM is referred as the "effective organic matter" (EOM) (Veeken et al. 2017). The EOM, calculated as OM/HC ratio was the highest in soil amended with both composts and mainly with orange compost. Organic carbon and nitrogen content were significantly lower in soil treated with both typologies of sulfur-based pads than the other treatments, conversely MBC was the highest. WSP considerably enhanced in all the treatments and the highest amount was in soil treated with both digestates (Tables 5, 6).

The results evidenced that the type of byproducts, regardless of the type of waste, mostly influenced soil characteristics, while the effects due to the interaction of the two factors (byproduct × waste) were less significant (Table 7). In short, both the two types of wastes and the three recycling processes examined seemed to produce byproducts with promising fertilizing capabilities. The ranking of treatments to ameliorate soil fertility were as follow: compost, sulfur-based pads and digestate. These results evidenced a specificity between treatment and soil properties, pointing out as the effects of the single byproduct depended

Table 5
Physical and chemical properties of potted alkaline sandy-loam soils CTR0, and six months after the addtion of: composted olive pomace "A"; sulphur-bentonite + olive pomace, "B"; olive digestate "C". Not amended soil was used as control (CTR). The data are the mean of six replicates ± standard deviation (n=18). Different letters in the same row indicate, significant differences among the treatments (Tukey's test, $p \leq 0.05$).

	CTR0	CTR	A	B	C
Texture	SL	SL	SL	SL	SL
pH	8.5 ^{a} ±0.60	8.5 ^{a} ±0.60	7.5 ^{a} ±0.80	7.2 ^{a} ±0.40	8.00 ^{a} ±0.20
EC µS/cm	339 ^{c} ±11	350 ^{c} ±14	450 ^{a} ±10	419 ^{b} ±12	466 ^{a} ±8.00
WC %	21.0 ^{b} ±2.6	21.5 ^{b} ±2.81	27.4 ^{a} ±0.79	24.2 ^{ab} ±1.70	28.8 ^{a} ±1.76
WSP µg TAE g^{-1} d.s.	19 ^{c} ±2.10	14 ^{c} ±2.80	45 ^{b} ±1.60	41 ^{b} ±3.26	96 ^{a} ±4.00
TOC %	0.95 ^{b} ±0.16	0.9^{c} ±0.16	1.73 ^{a} ±0.15	1.3 ^{b} ±0.25	1.5 ^{b} ±0.30
TN %	0.15±0.01^{bc}	0.15±0.01^{bc}	0.30±0.02^{b}	0.21±0.04^{a}	0.18±0.03^{c}
C/N	6 ^{b} ±0.3	6 ^{b} ±0.3	5.8 ^{b} ±1	6.2 ^{b} ±0.5	8.3 ^{a} ±0.6
SOM %	1.63 ^{c} ±0.3	1.55 ^{c} ±0.27	2.97 ^{a} ±0.26	2.24 ^{b} ±0.13	2.58 ^{a} ±0.38
FDA µg fluorescein g^{-1} d.s.	42 ^{a} ±2	42 ^{a} ±2	47 ^{a} ±3	38 ^{b} ±2	40 ^{b} ±1
DH µg INTF g^{-1} d.s. h^{-1}	56 ^{c} ±2	57 ^{b} ±2.81	65 ^{a} ±1.86	48 ^{c} ±3.17	62 ^{ab} ±1.95
MBC µg C g^{-1} f.s.	813 ^{d} ±18	835 ^{c} ±18	1007 ^{b} ±21	1081 ^{a} ±44	861 ^{c} ±27
HC %	0.60 ^{a} ±0.05	0.60 ^{a} ±0.05	0.43 ^{b} ±0.02	0.66 ^{a} ±0.01	0.62 ^{a} ±0.03
FC %	0.40 ^{b} ±0.08	0.45 ^{b} ±0.08	0.26 ^{c} ±0.05	0.62 ^{a} ±0.03	0.60 ^{a} ±0.03
HC/FC	1.5^{b}±0.12	1.33^{b}±0.12	1.65^{a}±0.10	1.06^{c}±0.04	1.03^{c}±0.06
CSC cmol($^{+}$) Kg^{-1}	18.9 ^{b} ±1.6	18.7 ^{b} ±1.42	23.10 ^{a} ±1.58	22.3 ^{ab} ±1.23	23.4 ^{a} ±1.36

Table 6
Physical and chemical properties of potted alkaline sandy-loam soils CTR 0, and six months after the addition of: composted orange wastes "A"; sulphur-bentonite + orange wastes, "B"; orange digestate "C". Not amended soil was used as internal control (CTR). The data are the mean of six replicates ± standard deviation (n=18). Different letters in the same row indicate, significant differences among the treatments (Tukey's test, $p \leq 0.05$).

	CTR0	CTR	A	B	C
Texture	SL	SL	SL	SL	SL
pH	8.5 ^{a} ±0.60	8.5 ^{a} ±0.60	8.0 ^{a} ±0.80	7.2 ^{a} ±0.40	7.5 ^{a} ±0.20
EC µS/cm	339 ^{c} ±11	350 ^{c} ±14	410 ^{a} ±10	437 ^{b} ±12	479 ^{a} ±8.00
WC %	21.0 ^{b} ±2.6	21.5 ^{b} ±2.81	29.4 ^{a} ±0.79	21.2 ^{ab} ±1.70	26.8 ^{a} ±1.76
WSP µg TAE g^{-1} d.s	19 ^{c} ±2.10	14 ^{d} ±2.80	40 ^{b} ±1.60	39 ^{b} ±3.26	90 ^{a} ±4.00
TOC %	0.95 ^{b} ±0.16	0.9 ^{b} ±0.16	2.1 ^{a} ±0.15	1.5 ^{b} ±0.25	1.8 ^{a} ±0.30
TN %	0.15±0.01^{bc}	0.15±0.01^{bc}	0.33±0.02^{b}	0.25±0.04^{a}	0.19±0.03^{c}
C/N	6 ^{b} ±0.3	6 ^{b} ±0.3	6.3 ^{b} ±0.4	6 ^{b} ±0.5	9.5 ^{a} ±0.6
SOM %	1.63 ^{c} ±0.3	1.53 ^{c} ±0.3	3.62 ^{a} ±0.3	2.58 ^{b} ±0.4	3.1 ^{a} ±0.8
FDA µg fluorescein g^{-1} d.s	42 ^{a} ±2	42 ^{a} ±2	46 ^{a} ±2	48^{a} ±1	43^{a} ±3
DH µg INTF g^{-1} d.s. h^{-1}	56 ^{c} ±2	57 ^{c} ±2	69 ^{a} ±2	62 ^{b} ±3	64 ^{b} ±1.5
MBC µg C g^{-1} f.s	813 ^{d} ±18	835 ^{d} ±18	1100 ^{b} ±21	1180 ^{a} ±34	890 ^{c}±27
HC %	0.60 ^{a} ±0.05	0.60 ^{a} ±0.05	0.44 ^{b} ±0.02	0.65 ^{a} ±0.01	0.60 ^{a} ±0.03
FC %	0.40 ^{b} ±0.08	0.45 ^{b} ±0.08	0.22 ^{c} ±0.05	0.60 ^{a} ±0.03	0.58 ^{a} ±0.03
HC/FC	1.5^{b}±0.12	1.33^{b}±0.12	2^{a}±0.10	1.08^{c}±0.04	1.03^{c}±0.06
CSC cmol($^{+}$) Kg^{-1}	18.9 ^{b} ±1.6	18.7 ^{b} ±1.6	25 ^{a} ±1.5	23 ^{ab} ±1.3	24 ^{a} ±1

Table 7
Two-way ANOVA to test the effects of the factors (byproducts and wastes) on WSP, SOM, FDA, DHA and MBC of potted alkaline sandy-loam soils. ***$p<0.001$; ** $p<0.01$: *$p<0.05$.

	WSP	SOM	FDA	DHA	MBC
R^2	0.995	0.906	0.802	0.899	0.955
F-ratios					
Byproducts	1005***	42***	10**	28***	102***
Wastes	16***	19***	13**	26***	22***
B × W	n.s.	n.s	7**	10**	n.s.

on its own chemical characteristics. The results, evidencing the positive effects of all these byproducts, agreed with previous works highlighting as organic components were able to improve soil fertility (Muscolo et al., 2019; Ye et al., 2020; Cortes' et al., 2020). The efficient transformation of these wastes into useful byproducts, is in line with the directive 2008/98/EC on wastes, mainly focused on sustainable and controlled disposal of wastes to prevent soil and groundwater pollution (Doula et al., 2012).

3.3. Fertilizer efficacy on garlic growth and metabolism

The effectiveness of compost, digestate and sulfur-based pads was also determined by testing the growth and antioxidant properties of garlic, a worldwide consumed crop, to close the loop of both quality and sustainability. Results evidenced that in garlic plants, cultivated with byproducts, the number and length of leaves increased in respect to control (Table 8). No significant differences were observed in root length between control and treatments. The garlic bulb diameter increased in

Table 8
Growth parameters of garlic grown for 6 months in alkaline sandy-loam soils not amended (control, CTR) or amended with: A composted orange wastes; sulphur-bentonite + orange wastes; orange digestate; B composted olive pomace, sulphur-bentonite + olive pomace, olive digestate. The data are the mean of six replicates ± standard deviation (n=18). Different letters in the same row indicate, significant differences among the treatments (Tukey's test, $p \leq 0.05$).

A				
Garlic	CTR	Compost	SB+OR	digestate
Leaf length (cm)	28±1^{b}	42±3^{a}	39±4^{a}	33±3^{a}
Root length (cm)	20±1^{a}	21±2^{a}	21±1^{a}	20±2^{a}
Bulb diameter (mm)	10±1^{b}	15±2^{a}	14±2^{a}	14±1^{a}
Leaves (number)	4±1^{a}	7±2^{a}	6±1^{a}	6±1^{a}
B				
Garlic	CTR	Compost	SB+OP	digestate
Leaf length (cm)	28±1^{b}	35±2^{a}	34±1^{a}	30±2^{b}
Root length (cm)	20±1^{a}	20±2^{a}	21±2^{a}	20±1^{a}
Bulb diameter (mm)	10±1^{b}	14±1^{a}	13±2^{a}	13±1^{a}
Leaves (number)	4±1^{a}	6±2^{a}	5±2^{a}	5±1^{a}

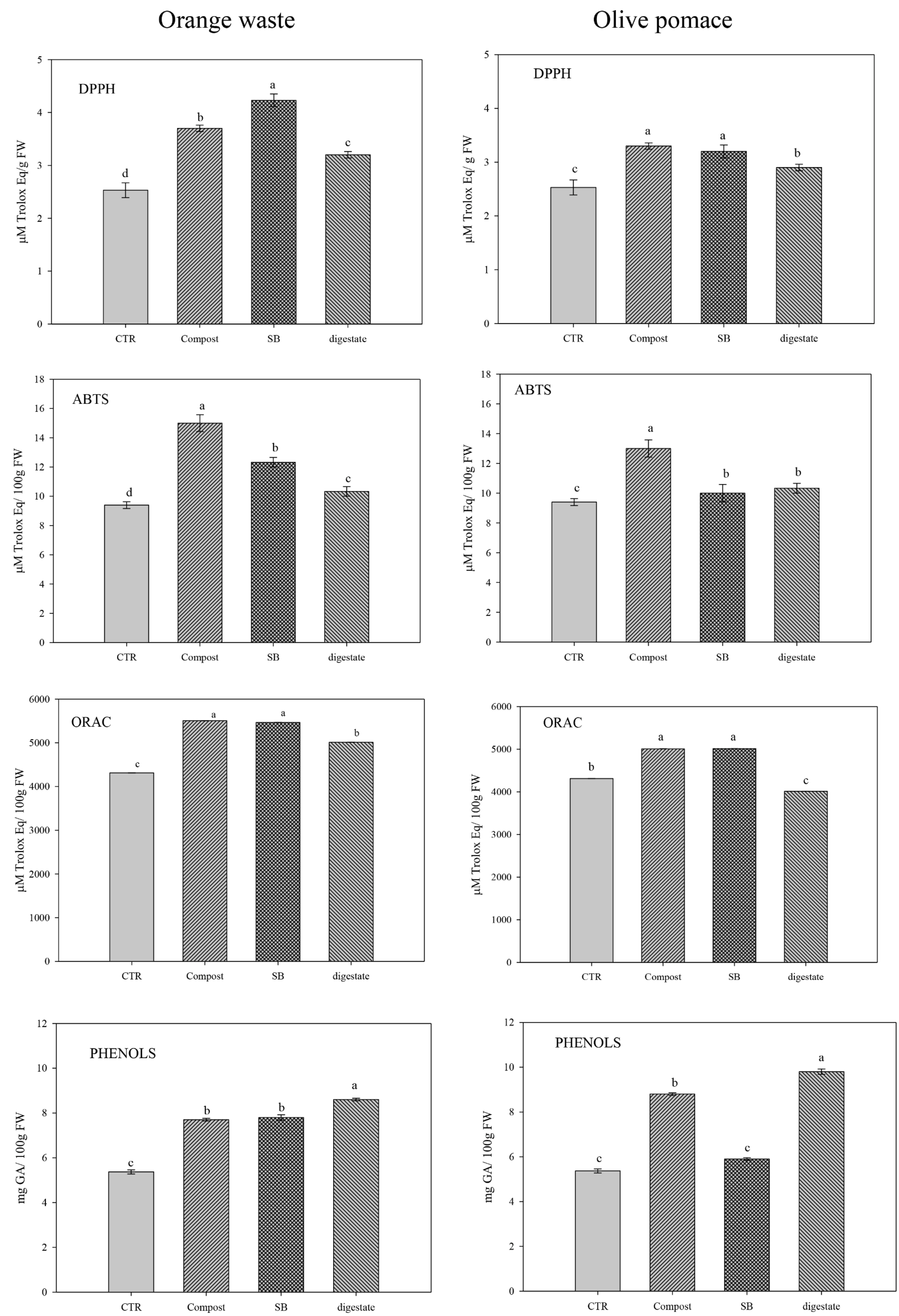

Fig. 1. Antioxidant activities, total phenols and total flavonoids in garlic bulb grown for 6 months in alkaline sandy-loam soils not amended (control, CTR) and amended with composted orange waste or olive pomace, sulfur-based pads or digestate. The data are the mean of six replicates ± standard deviation (n=18). Different letters indicate significant differences among the treatments (Tukey's test, $p \leq 0.05$). Two-way ANOVA was used to test the effects of the factors (byproducts and wastes) on antioxidants***$p<0.001$; ** $p<0.01$: *$p<0.05$.

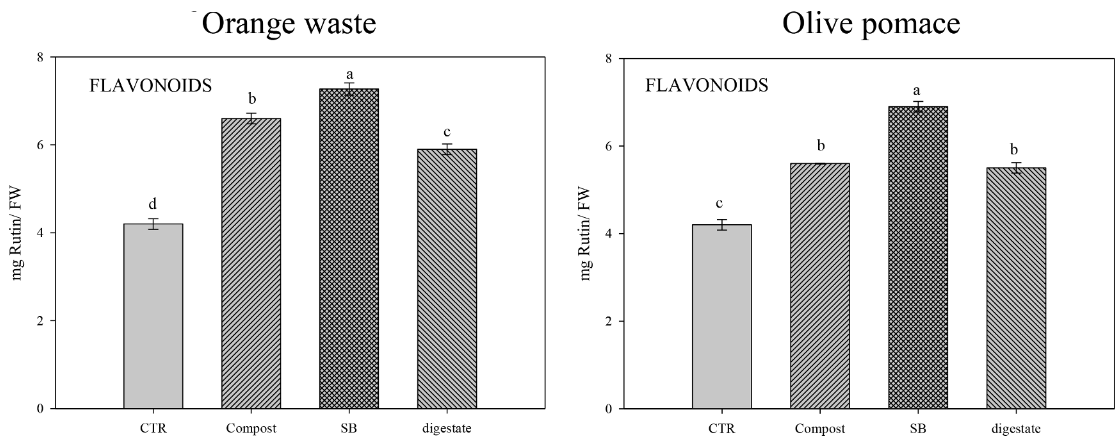

Fig. 1. (*continued*).

presence of compost, sulfur-based pads and digestate. The greatest increases were observed in presence of byproducts derived from orange waste (Table 8). These data evidenced a positive stimulatory effect of compost and sulfur-based pads. Antioxidant activities in terms of DPPH, ORAC and ABTS increased in presence of all treatments in respect to control, and the highest increment was observed with compost and sulfur-based pads coming from both orange waste and olive pomace (Fig. 1). Flavonoids increased significantly in presence of all byproducts in the following order: sulfur-based pad>compost>digestate>control. Total water-soluble phenols increased in presence of the treatments but with a different order of effectiveness: digestate>compost>sulfur-based pad>control (Fig. 1). These results evidenced as all the byproducts were able to increase the quality of garlic in terms of antioxidants, valuable inhibitors of free radical reactions, that protect cells against oxidative damage. Our results agree with previous studies reporting an increase in antioxidant compounds in broccoli (Naguib et al. 2012), sweet peppers (Del Amor et al. 2008), and tomatoes (Pieper and Barrett 2006) cultivated with bio-organic and organic fertilizers. The transformation of these agricultural wastes in resource using different processes lead to different final byproducts, all with positive effects on soil and plants. These byproducts can be used in an environmentally friendly agriculture and can positively affect the circular economy of the industrial sector and farms (Salomone et al., 2017). Each process has diverse set-up and is able to convert different amounts of wastes (90% or 80% for aerobic digestion, 50% for anaerobic digestion and 5% for sulfur-based pads) in different times (4 months aerobic digestion, 1 month for anaerobic digestion and 1 day for sulfur-based pads). The results show that each process, has a different competitivity from environmental, economic and/or agricultural standpoint. Composting (aerobic digestion process) has the greatest beneficial effects on soil and crops with low processing costs (no electricity and expenses of transport are necessary) though it has the longest production time. Anaerobic digestion is faster than aerobic digestion, produces energy or fuel, reducing the dependence of the agricultural sector on energy from fossil fuel, but it requires initial investments and conferral costs in the location of the plant. Sulfur-based pad production, despite the consumption of electricity, is a short processing cycle (one day) that disposes the crude agricultural wastes concomitantly with a great amount of another pollutant (Sulphur), reducing the emission of greenhouse gas in the atmosphere. In short, these results highlight that the different methodologies used such as recycling, valorization, and energy-producing processes, produce diverse types of products, all of high added-value that can be selectively chosen on the basis of specific farm and industry exigence to create an additional economical entry for the specific sectors.

4. Conclusion

Nowadays, converting agricultural wastes into byproducts is a priority that makes cleaner the environment, more fertile the soil, and improve farm bio economy. This study gives information on the processes that can be used to better convert these kinds of wastes into resource, evidencing as these different processes affect the chemical composition and in turn the potential added value of the end-products obtained. The recycling of these wastes can be considered a beneficial process leading to significant reductions in greenhouse gas emissions in the atmosphere for the elimination of a large amount of hazardous materials from the environment. Generally, orange and olive wastes release different amount of greenhouse gas; one ton of wet orange waste left on the ground emits 0.130 kg of CH_4, 30.900 kg of CO_2 and 0.069 kg of N_2O, while one ton of wet olive pomace produces, 1162.3 kg of CO_2, 122 kg of CH_4 and 0.12 kg of N_2O. In addition to organic wastes, Sulphur as residue of hydrocarbon refining processes generates hydrogen sulphide and Sulphur oxide causing environmental pollution, thus the use of high Sulphur percentage in the fertilizers can help to maintain a clean environment. Furthermore, the economic benefit of transforming the wastes in fertilizers will come not only from the sale of the new fertilizers produced but also by the money saved from the decrease in the production and use of chemical fertilizers and the reduction of costs for landfilling, Regarding the sustainability and profitability of compost process, the plants have to serve a local market, supplying their product within 50 km of the plant. This is equivalent at a cost of EUR 50–60 transport costs for 25 tons of biomass. The expenses are covered by end compost prices of around EUR 5/ton (EUR 125/for 25 tons). The environmental gain rises, in this case, from 60% decrease of CO_2 emission due to the reduction of landfill permanence of wastes and from the reduction in an average 9.7 tons of CO_2 equivalent emitted for every ton of mineral fertilizer less produced. Similarly, for the profitability of digestate, the plants have to be rationally distributed in the territory in order to receive short-range wastes. The production costs of digestate generally range from 10 to 30 Euro per ton for bio-waste treatment through anaerobic digestion. This value excludes the investment costs necessary for start-up, and costs are sensitive to technology used as well as input materials. In a context of high fossil fuel energy prices. the intrinsic value of the digestate can compensate the proportionately high price of fossil fertilizers quantified for nitrogen (34.5% ammonium nitrate) Euro 616 ton^{-1}, phosphate (46%)—Euro 525 ton^{-1} and • potassium (60% potassium chloride) —£534 ton^{-1}. On the basis of the above fertilizer costs, a ton of digestate was calculated to be worth a total of Euro 119,160. Sulfur-bentonite fertilizer advantage come from the selling of the pads that can be used mainly to recovery degraded lands and can be sell to 230/tons euros, to which must be added the euros saved by the reduction of CO_2, CH_4 and Sulphur emissions in the atmosphere.

In short, these results highlight that the different methodologies used such as recycling, valorization, and energy-producing processes, produce diverse types of products, all of high added-value that can be selectively chosen on the basis of specific farm and industry exigence. Results evidenced also new potential applications of these two kinds of wastes considering that the derived byproducts can be used not only to recovery soil fertility but also to improve the quality of crop species stimulating the synthesis of bio compounds with pharmaceutical and nutraceutical purposes. The production processes of these byproducts, whose benefits overcome risks, are valuable and greatly could create an additional economical entry for the agricultural sector.

Declaration of Competing Interests

Regarding the manuscript entitled "Recycling of agricultural (orange and olive) bio-wastes into ecofriendly fertilizers for improving soil and garlic quality." All the authors agree that there are no interests to declare.

Acknowledgment

The authors appreciated the financial support from Calabria Region PSR 2007-2013 MIs. 124 grant number 94752165434

References

Adam, G., Duncan, H., 2001. Development of a sensitive and rapid method for the measurement of total microbial activity using fluorescein diacetate (FDA) in a range of soils. Soil Biol. Biochem. 33, 943–951.

Agapiou, A., Papadopoulos, N., Sarris, A., 2016. Detection of olive oil mill waste (OOMW) disposal areas using high resolution GeoEye's OrbView-3 and Google Earth images. Open Geosci 8, 700–710.

AOAC. 2005. Official Methods of Analysis.18th ed. Association of Official Analytical Chemists. Arlington, VA, USA.

Ashraf, M.A., Maah, J.M., Yusoff, I., 2014. Soil contamination, risk assessment and remediation. Environmental risk assessment of soil contamination. INTECH Open Science 1, 1–56 https://www.intechopen.com/books/environmental-risk-assessment of soil-contamination/soil-contamination-risk-assessment-and remediation, Accessed date: 23 May 2017.

Beeby, J., Moore, S., Taylor, L., Nderitu, S., 2020. Effects of a one-time organic fertilizer application on long-term crop and residue yields, and soil quality measurements using biointensive agriculture. Frontiers in Sustainable Food Systems 4, 67. https://doi.org/10.3389/fsufs.2020.00067.

Belligno, A., Di Leo, M.G., Marchese, M., Tuttobene, R., 2005. Effects of industrial orange wastes on soil characteristics and on growth and production of durum wheat. Agron. Sustain. Dev. 25 (1), 129–135.

Bettany, J.R., Saggar, S., Stewart, J.W.B., 1980. Comparison of the amount and forms of Sulfur in soil organic matter fractions after 65 years of cultivation. Soil Sci. Soc. Am. J. 44, 70–75.

Bhunia, S., Bhowmik, A., Mallick, R., Mukherjee, J., 2021. Agronomic efficiency of animal-derived Organic fertilizers and their effects on biology and fertility of soil: A. Review. Agronomy 11, 823. https://doi.org/10.3390/agronomy11050823.

Box, J.D., 1983. Investigation of the Folin-Ciocalteau reagent for the determination of polyphenolic substances in natural waters. Water Res 17, 511–525.

Canet, R, Pomares, F, Cabot, B, Chaves, C, Ferrer, E, Ribó, M, Albiach, MA., 2008. Composting olive mill pomace and other residues from rural southeastern Spain. Waste Manag 28 (12), 2585–2592. Dec.

Cortés, A., Oliveira, L.F.S., Ferrari, V., Taffarel, S.R., Feijoo, G., Moreira, M.T., 2020. Environmental assessment of viticulture waste valorisation through composting as a biofertilisation strategy for cereal and fruit crops. Environment. Poll. 264, 114794.

A/S, COWI, Research, Eunomia, Ltd, Consulting, 2019. Study: The costs of not implementing EU environmental law 166.

Del Amor, F.M., Serrano-Martinez, A., Fortea, I., Nunez-Delicado, E., 2008. Differential effect of organic cultivation on the levels of phenolics, peroxidase and capsidiol in sweet peppers. J. Sci. Food Agric. 88, 770–777.

Djeridane, A., Yousfi, M., Nadjemi, B., Boutassouna, D., Stocker, P., Vidal, N., 2006. Antioxidant activity of some Algerian medicinal plants extracts containing phenolic compounds. Food Chem 97, 654–660.

Doula, M.K., Tinivella, F., Ortego, L.L.M., Kavvadias, V.A., Sarris, A., Theocharopoulos, S., Sanchez-Monedero, M.A., Elaiopoulos, K., 2012. Good practice for the agronomic use of olive mill. Wastes, Application Guide. Life PROSODOL 61. Life07/ENV/GR/000280.

Doymaz, I., Gorel, O., Akgun, N.A., 2004. Drying characteristics of the solid byproduct of olive oil extraction. Biosyst. Eng. 88, 213–219.

European Commission, 2020. Circular Economy Action Plan. For a cleaner and more competitive Europe 28.

Fakayode, O.A., Abobi, K.E., 2018. Optimization of oil and pectin extraction from orange (Citrus sinensis) peels: a response surface approach. J. Anal. Sci. Technol. 9, 20. https://doi.org/10.1186/s40543-018-0151-3.

FAO. 2017. The future of food and agriculture – Trends and challenges. Rome. ISBN 978-92-5-109551-5.

FAO–UNESCO, 1999. World Soil Map, Revised Legend. Rome.

Ferronato, N., Torretta, V., 2019. Waste Mismanagement in Developing Countries: A Review of Global Issues. Int. J. Environ. Res. Public Health 16 (6), 1060. https://doi.org/10.3390/ijerph16061060.

Galanakis, C.M., Olive Mill Waste: Recent Advances for Sustainable Management; Academic Press: London, UK; Elsevier: London, UK, 2017; Preface.

Gerber, P.J., Steinfeld, H., Henderson, B., Mottet, A., Opio, C., Dijkman, J., Falcucci, A., Tempio, G., 2013. Tackling climate change through livestock – A global assessment of emissions and mitigation opportunities. Food and Agriculture Organization of the United Nations (FAO), Rome.

Global Sustainable Development Report 2019. The Future is Now – Science for Achieving Sustainable Development, (United Nations, New York).

Hollins, O., Lee, P., Sims, E., Bertham, O., Symington, H., Bell, N., Lucie Pfaltzgraff, L., Sjögren, P., 2017. Towards a circular economy–Waste management in the EU. STOA-Science and Technology Options Assessment. ISBN978-92-846-1548-3.

Kang, H.W., 2015. Antioxidant activity of ethanol and water extracts from lentil (Lens culinaris Medik). J. Food Nutr. Res.earch 3 (10), 667–669.

Khdair, A., Abu-Rumman, G., Khdair, S.I., 2019. Pollution estimation from olive mills wastewater in Jordan. Heliyon 5 e02386.

Kjeldahl, J., 1883. Neue Methode zur Bestimmung des Stickstoffs in organischen K€orpern. Anal. Chem. 22, 366e382.

Manios, T., 2004. The composting potential of different organic solid wastes: experience from the island of Crete. Environ. Int. 29, 1079–1089.

Mari, I., Ehaliotis, C., Kotsou, M., Balis, C., Georgakakis, D., 2003. Respiration profiles in monitoring the composting of byproducts from the olive oil agro-industry. Bioresour. Technol. 87, 331–336.

Mehlich, A., 1953. Rapid determination of cation and anion exchange properties and pH of soils. J. Assoc. Off Agric. Chem. 36, 445–457.

Moncada, B.J., Aristizábal, M.V., ACA, C., 2016. Design strategies for sustainable biorefineries. Biochem. Eng. J. 116, 122–134. https://doi.org/10.1016/j.bej.2016.06.009.

Muscolo, A., Mallamaci, C., Settineri, G., Calamarà, G., 2017a. Increasing soil and crop productivity by using agricultural wastes pelletized with elemental Sulphur and bentonite. Agron. J. 109, 1–11. https://doi.org/10.2134/agronj2017.03.0143.

Muscolo, A., Settineri, G., Papalia, T., Attinà, E., Basile, C., Panuccio, M.R., 2017b. Anaerobic co-digestion of recalcitrant agricultural wastes: characterizing of biochemical parameters of digestate and its impacts on soil ecosystem. Sci.Total Environ 586, 746–752.

Muscolo, A., Papalia, T., Settineri, G., Mallamaci, C., Jeske-Kaczanowska, A., 2018. Are raw materials or composting conditions and time that most influence the maturity and/or quality of composts? Comparison of obtained composts on soil properties. J. Clean. Prod. 195, 93–101.

Muscolo, A., Papalia, T., Settineri, G., Romeo, F., Mallamaci, C., 2019. Three different methods for turning olive pomace in resource: Benefits of the end products for agricultural purpose. Sci. Total Environm. 662, 1–7.

Muscolo, A., Papalia, T., Settineri, G., Mallamaci. C., Panuccio, M.R., 2020. Sulfur bentonite-organic-based fertilizers as tool for improving bio-compounds with antioxidant activities in red onion. J. Sci. Food Agric. 100, 785–793.

Naguib, A.E.-M.M., El-Baz, F.K., Salama, Z.A., Abd El Baky Hanaa, H., Ali, H.F, Gaafar, A. A., 2012. Enhancement of phenolics, flavonoids and glucosinolates of Broccoli (Brassica olaracea, var. Italica) as antioxidants in response to organic and bio-organic fertilizers. J. Saudi Soc. Agric. Sci. 11, 135–142.

Negro, M.J., Manzanares, P., Ruiz, E., Castro, E., Ballesteros, M. The biorefinery concept for the industrial valorization of residues from olive oil industry. In Olive Mill Waste: Recent Advances for Sustainable Management; Galanakis, C.M., Ed.; Academic Press: London, UK; Elsevier: London, UK, 2017; Chapter 3; pp. 57–78.

Nelson, D.W., Sommers, L.E., 1982. Total carbon, organic carbon, and organic matter. In: Page, A.L., Miller, R.H., Keeney, D.R. (Eds.), Methods of Soil Analysis. American Society of Agronomy, Madison, pp. 539–579.

O'Connor, G., 2021. Shifting the value of food and organic waste management in the food services sector in Brisbane. Australia. Resour. Conserv. Recycl. Adv. 12, 200052 https://doi.org/10.1016/j.rcradv.2021.200052. ISSN 2667-3789.

OECD/FAO 2019. OECD-FAO Agricultural Outlook 2017-2026, OECD Publishing, Paris. 10.1787/agr_outlook-2017-en.

Ortiz-Sanchez, M., Solarte-Toro, J., Orrego-Alzate, C., et al., 2021. Integral use of orange peel waste through the biorefinery concept: an experimental, technical, energy, and economic assessment. Biomass Conv. Bioref. 11, 645–659. https://doi.org/10.1007/s13399-020-00627-y.

Palmeros Parada, M., Osseweijer, P., Posada Duque, J.A., 2017. Sustainable biorefineries, an analysis of practices for incorporating sustainability in biorefinery design. Ind. Crop Prod. 106, 105–123. https://doi.org/10.1016/j.indcrop.2016.08.052.

Panuccio, M.R., Attinà, E., Basile, C., Mallamaci, C., Muscolo, A., 2016. Use of recalcitrant agriculture wastes to produce biogas and feasible biofertilizer. Waste Biomass Valoriz 7, 267–280.

Papalia, T., Barreca, D., Panuccio, M.R., 2017. Assessment of antioxidant and cytoprotective potential of jatropha (Jatropha curcas) grown in Southern Italy. Int. J. Mol. Sci. 18, 660. https://doi.org/10.3390/ijms18030660.

Pieper, JR, Barrett, DM., 2006. Effects of organic and conventional production systems on quality and nutritional tomatoes and bell peppers. J. Agric. Food Chem. 54, 8244–8252.

Pourjavid, M.R., Arabieh, M., Sehat, A.A., Rezaee, M., Hosseini, M.H., Yousefi, S.R., Jamali, M.R., 2014. Flame atomic absorption spectrometric determination of Pb(II) and Cd(II) in natural samples after column graphene oxide-based solid phase extraction using 4-Acetamidothiophenol. J. Braz. Chem. Soc. 25 (11), 2063–2072. https://doi.org/10.5935/0103-5053.20140193.

PROSODOL, 2012. Proceeding of Olive Oil Mills Wastes and Environmental Protection Symposium. October 2012 Chania, Crete 16–18. Greecehttp://refhub.elsevier.com/S0048-9697(19)30248-7/rf0180.

Re, R., Pellegrini, N., Proteggente, A., Pannala, A., Yang, M., Rice-Evans, C., 1999. Antioxidant activity applying an improved ABTS radical cation decolorization assay. Free Radic. Biol. Med. 26, 1231–1237.

Restrepo-serna, D.L., Anderson, J., Cardona-alzate, C.A., 2018. Energy Efficiency of Biorefinery Schemes Using Sugarcane Bagasse as Raw Material:1–12. 10.3390/en11123474.

Rigane, K.M., Medhioub, K., 2011. Assessment of properties of Tunisian agricultural waste composts: Application as components in reconstituted anthropic soils and their effects on tomato yield and quality. Resour. Conserv. Recycl. 55 (8), 785–792. https://doi.org/10.1016/j.resconrec.2011.03.012. ISSN 0921-3449.

Rokaya, B., Kerroum, D., Hayat, Z., Panico, A., Ouafa, A., Pirozzi, F., 2019. Biogas production by an anaerobic digestion process from orange peel waste and its improvement by limonene leaching: Investigation of H2O2 pre-treatment effect. Energy Sources, Part A: Recovery, Utilization, and Environmental Effects. https://doi.org/10.1080/15567036.2019.1692975. DOI.

Salomone, R., Saija, G., Mondello, G., Giannetto, A., Fasulo, S., Savastano, D., 2017. Environmental impact of food waste bioconversion by insects: application of Life Cycle Assessment to process using Hermetia illucens. J. Clean. Prod. 140, 890–905. https://doi.org/10.1016/j.jclepro.2016.06.154.

Sang, J., Li, L., Wen, J., Gu, Q., Wu, J., Yu, Y., Xu, Y., Fu, M., Lin, X., 2021. Evaluation of the Structural, Physicochemical and Functional Properties of Dietary Fiber Extracted from Newhall Navel Orange By-Products. Foods 10 (11), 2772. https://doi.org/10.3390/foods10112772.

Siddiqui, S.A., Pahmeyer, M.J., Assadpour, E., Jafari, S.M., 2022. Extraction and purification of d-limonene from orange peel wastes: Recent advances. Ind. Crops Prod. 177, 14484. https://doi.org/10.1016/j.indcrop.2021.114484. ISSN 0926-6690.

Vance, E.D., Brookes, P.C., Jenkinson, D.S., 1987. An extraction method for measuring soil microbial biomass. C. Soil Biol. Biochem. 19, 703–707.

Veeken, A., Adani, F., Fangueiro, D., Jensen, S., 2017. The value of recycling organic matter to soils classification as organic fertiliser or organic soil improver. EIP-AGRI Focus Group - Nutrient Recycling 10. Retrieved from http://circulairterreinbeheer.nl/wp-content/uploads/2017/10/Value-of-organic-matter-Classification-as-fertiliser-or-soil-improver_final-23-Jan-2017.pdf. Accessed 02 Dec 2019.

von Mersi, W., Schinner, F., 1991. An improved and accurate method for determining the dehydrogenase activity of soils with iodonitrotetrazolium chloride. Biol. Fertil. Soils 11, 216–220.

Walkley, A., Black, I.A., 1934. An examination of the Degtjareff method for determining soil organic matter and a proposed modification of the chromic acid titration method. Soil Sci 37, 29–38.

Wikandari, R., Nguyen. H., Millati, R., Niklasson, C., Taherzadeh, M.J., 2015. Improvement of Biogas Production from Orange Peel Waste by Leaching of Limonene. Biomed Res Int. 1–6. 10.1155/2015/494182.

Ye, L., Zhao, X., Bao, E., Li, J., Zou, Z., Cao, K., 2020. Bio-organic fertilizer with reduced rates of chemical fertilization improves soil fertility and enhances tomato yield and quality. Scientific Reports. https://doi.org/10.1038/s41598-019-56954-2, 20) 10: 177 |.