




## RESEARCH ARTICLE

# Compositional and structural characterization of bioenergy digestate and its aerobic derivatives compost and vermicompost

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## Abstract

**Introduction:** In addition to produce biogas, the anaerobic digestion process releases a significant quantity of solid digestate (DG), a C-rich byproduct suitable both as a soil improver and as a biosorbent of organic and inorganic pollutants. This study considered a mixed biowaste DG and two aerobic DG-derivatives such as compost (CP) and vermicompost (VC).

**Materials and Methods:** A multianalytical approach was adopted to comprehensively characterize DG, CP and VC. Elemental composition was determined by total reflection X-ray fluorescence (TXRF) spectroscopy; micromorphological features, surface elements and surface area were evaluated by scanning electron microscopy coupled with energy dispersive X-ray (SEM-EDX) technique and Brunauer–Emmett–Teller (BET) analysis; functional properties were investigated by attenuated total reflectance-Fourier transform infrared (FTIR-ATR) spectroscopy and Raman spectroscopy.

**Results:** TXRF analysis showed that DG had the highest K content ( $\sim 8.9 \text{ g kg}^{-1}$ ), while VC had the maximum P, S and Ca contents, and appreciable levels of essential plant micronutrients, such as Mn, Fe, Cu and Zn. SEM-EDX technique evidenced heterogeneous surfaces and low porosity of all materials. BET analysis showed relevant specific surface areas of DG, CP and, especially, VC ( $\sim 1.6 \text{ m}^2 \text{ g}^{-1}$ ). FTIR-ATR and Raman spectroscopy allowed to identify various aliphatic and aromatic functional groups of the materials and provided information on their major molecular classes, such as lignocellulosic moieties and inorganic components. The latter techniques also contributed to understand the chemical transformations of the DG after the aerobic treatments.

**Conclusion:** The overall results obtained confirmed the valuable potential of these materials in improving the physical and chemical fertility of the soil and in retaining pollutants, with consequent benefits for maintaining soil productivity and preserving ecosystem health.

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**KEYWORDS**

Brunauer–Emmett–Teller analysis, biosorbent, Fourier transform infrared spectroscopy, soil amendment, total reflection X-ray fluorescence spectroscopy

## 1 | INTRODUCTION

An enormous quantity of organic waste is continuously and increasingly generated worldwide, especially in the most anthropized areas (Ellis, 2018; Wilson et al., 2012). However, landfilling and haphazard land disposal still dominate waste disposal methods, especially in low- and middle-income countries, resulting in multiple environmental issues (Feronato & Torretta, 2019). On the other hand, the urgent need to reduce greenhouse gas emissions, to search for renewable energy sources, to increase soil C sequestration and reclaim degraded areas for crop cultivation have led technicians and researchers to implement and develop technologies capable of recovering and valorizing organic waste (Singh & Kalia, 2017). In accordance with circular economy model, promising strategies have been developed to recycle organic wastes and biodegradable residues from agriculture, agri-food industry, and livestock activity, and convert them into bioenergy with consequent release of solid byproducts suitable for both agricultural and environmental applications (Appels et al., 2008; Chynoweth et al., 2001; Nkoa, 2014; Palansooriya et al., 2023; Tursi, 2019).

The anaerobic digestion (AD) process consists in the biochemical conversion of biomass operated, under low oxygen content, by four functional groups of bacterial (hydrolytic, acidogenic, acetogenic) and archaeal (methanogenic) populations that act sequentially during the process (Braguglia et al., 2018; Hassa et al., 2018). The main product of AD is biogas, which is composed of methane (50%–80% v/v), variable amount of CO<sub>2</sub> and small quantities of other gases. The raw semisolid remain of AD (approximately 90%–95% moisture), after mechanical separation, releases a solid fraction called digestate (DG) and a separate clarified liquid commonly called liquid DG (Wang & Lee, 2021). DG usually has less than 20% moisture and a C content ranging between 25% and 41% on a dry matter basis, the variability being a function of the type of ingestate (entering biomass) (Cesaro, 2021) and the configuration of the digester (Nkoa, 2014). The physicochemical properties of AD byproducts are strictly related to the type of fed substrates, and bioconversion conditions, such as the hydraulic retention time, temperature, pH, reaction and the fermentation type (Weiland, 2010). Of course, process operating conditions influence also diversity and abundance of bacterial and archaeal sub-communities, and hence the chemical properties of the digestate (Hassa et al., 2018). During the AD, easily degradable compounds are rapidly converted into biogas, while the recalcitrant lignocellulosic fraction, partially decomposed organic moieties and various byproducts (i.e., phenols, reduced N forms, volatile organic acids) remains in the solid DG (Insam et al., 2015; Nkoa, 2014).

Although the direct application of DG to agricultural soil is a common practice, it raises concern for a number of reasons, such as the emission of ammonia and nitrogen oxides into the atmosphere,

leaching of nitrates into natural waters, possible addition of agrochemicals and antibiotics (Wang & Lee, 2021), contamination by potentially toxic elements, and plant and animal pathogens (Peng and Pivato, 2019). All of these could pose serious risks to human health and ecosystem security. However, it is also true that DG contains significant amounts of organic matter and phytonutrients that suggest its use as soil amendment (Insam et al., 2015).

Due to the above-mentioned potential environmental risks arising from the incorporation of DG into soil, it is preferable to direct this material to bio-oxidative postprocess treatments, such as composting and vermicomposting, which provide stability and maturity as well as humified and safer soil improvers (Le Pera et al., 2022; Wang et al., 2022). Once added to soil, both compost (CP) and vermicompost (VC) exert multiple benefits since they represent a supply of fairly stable organic matter which improves soil fertility and stimulates plant growth and microbial activity (Blouin et al., 2019; Chen et al., 2018). Although their physicochemical properties may be quite different from each other, DG, CP and VC have similar applications, being suitable amendments for agricultural use and implementers of C sequestration which limits greenhouse gas emission into the atmosphere (De Corato, 2020; Peng and Pivato, 2019).

In addition to improving the quality and fertility of soil, these materials can act as effective bioadsorbents of organic compounds and metals. This ability depends largely on their micromorphology and on the abundance and type of reactive surface functional groups. Compared to mineral adsorbents, DG and its aerobic post-process derivatives have the added value of enriching the soil with plant nutrients and effectively retaining water and micronutrients (Qian et al., 2015). CP and VC have shown excellent adsorption capacity of organic and inorganic pollutants through various mechanisms that involve their numerous sorption sites (Senesi et al., 2015). Adsorption modulates the bioavailability of pollutants in soil and limits their transfer into natural water and edible plant organs (Carnimeo et al., 2022; He et al., 2017).

Commonly, the characterization of soil amendments is based mainly on conventional techniques aimed at evaluating their supply of organic matter and plant nutrients, while their potential for retention of organic and inorganic soil components is often overlooked or not explored at all. Instead, an extensive characterization of these materials is essential because it allows to predict in broader terms the multiple functions that they perform in soil, and which go beyond the mere improvement of the physical and chemical fertility of soil, such as the potential to immobilize/release contaminants which is a crucial aspect for agricultural productivity and agroecosystem safety. It is also true that this approach requires the use of more sophisticated analytical techniques suitable to explore microstructure, surface extension, porosity and the chemical reactive sites of the adsorbents. Furthermore, a multianalytical approach allows to



investigate the evolution of the organic fraction of DG after bio-oxidative treatments such as composting and vermicomposting. The evaluation of the compositional, structural and functional changes occurred in the anaerobic DG after the two aerobic treatments (composting and vermicomposting) allows to understand if and how these treatments can improve DG performance both from the point of view of enhanced soil chemical fertility and from that of increased contaminant retention.

In addition to basic analyses such as moisture, volatile matter, fixed carbon and ash content (i.e., proximate analyses), pH and electrical conductivity (EC), more expensive and cutting-edge analyses include elemental composition (CHNS-O, i.e., ultimate analysis), total reflection X-ray fluorescence (TXRF) spectroscopy, scanning electron microscopy coupled with energy-dispersive X-ray (SEM-EDX) analysis, Brunauer–Emmett–Teller (BET) analysis, Fourier transform infrared (FTIR) spectroscopy combined with attenuated total reflection (ATR) analysis, and Raman spectroscopy. The latter techniques are rarely adopted to characterize DG samples and, especially, composted materials (CP and VC).

The TXRF technique has been used for quantifying trace and potentially toxic elements in various materials, including carbonaceous adsorbents (Abdel-Fattah et al., 2015; Taskin et al., 2019), but, as far as we know, never for the type of materials considered in this study. SEM and SEM-EDX analysis is adopted to investigate surface micromorphology and surface elements of adsorbent materials (Khare et al., 2023; Loffredo et al., 2021; Quadar et al., 2022; Yang et al., 2018). The extent and type of surface functional groups of adsorbents, which are responsible for chemical reactivity and sorption efficiency, can be studied in detail through FTIR spectroscopy (Loffredo, 2022; Quadar et al., 2022; Rueda et al., 2023).

A complete characterization of a swine manure DG has been carried out by Fierro et al. (2016), including proximate and ultimate analyses, FTIR and thermal analysis. SEM-EDX and FTIR spectroscopy have been employed to characterize DG samples produced from food waste (Opatokun et al., 2016), mixed feedstock (Loffredo et al., 2021) and olive pomace (Loffredo et al., 2024). FTIR-ATR spectroscopy was employed to characterize an olive pomace CP before and during the torrefaction process (Rueda et al., 2023). Lim and Wu (2015) carried out spectroscopic and structural characterization of a VC produced from palm oil mill effluent using FTIR spectroscopy, SEM and BET analysis. We are not aware of any studies that have applied the BET technique for DG or CP characterization.

Based on the above, this work aimed to adopt a multianalytic approach to extensively characterize a DG from mixed biowaste and two bio-oxidative post-AD derivatives of such DG, namely CP and VC. For the purpose, in addition to basic and spectroscopic analyses, two analytical techniques never or very rarely applied to this type of materials were used, namely TXRF to quantify trace and potentially toxic elements, and BET analysis to measure the specific surface area. The overall properties of the materials were examined and discussed in relation to their agricultural and environmental relevance.

## 2 | MATERIALS AND METHODS

### 2.1 | Digestate and its derivatives

The DG, CP and VC samples were all collected from a biogas-producing plant located in Capaccio, South Italy, belonging to the C&F Energy Società Agricola s.r.l. The ingestate of the AD process was a mixture of buffalo manure (80%, w/w), olive mill wastewater (15%, w/w), agrifood industry residues (3%, w/w) and poultry manure (2%, w/w). The rated power of the plant was 500 kW with hydraulic retention time of 60 days, which ensured optimal conditions for the bioconversion of the feeding biomass operating under thermophilic conditions (55–60°C). The raw semi-solid byproduct released from the AD process was mechanically treated to separate the solid fraction (DG) from the clarified liquid fraction. Before storage, the solid DG was subjected to a heat treatment at a temperature of 65°C for 5 days, which is comparable to a pasteurization process.

The CP and VC samples were produced from the above-cited DG after aerobic treatments. In particular, the CP was obtained by maintaining the DG under controlled aerobic conditions for approximately 2 months. In the case of VC, the DG was previously kept in the open air for 4 days, earthworms (*Eisenia foetida* L.) were subsequently added, and the vermicomposting process was carried out by controlling the main parameters (moisture, temperature, aeration and earthworm vitality) for 2 months.

Before starting the experiments, each material was thoroughly mixed, air-dried and ground with mortar and pestle to obtain particle dimension  $\leq 0.5$  mm.

### 2.2 | Characterization of the materials

#### 2.2.1 | Basic characterization and elemental analysis

The three tested materials (DG, CP and VC) were characterized for some basic physicochemical properties according to conventional methods. Briefly, moisture was determined after heating at 105°C overnight; the pH was potentiometrically measured in a 1:10 (w/v) material-double distilled water suspension; EC was measured at 25°C in the extract from a 1:10 (w/v) material-double distilled water suspension; the ash content was determined after burning the sample in a muffle furnace at a temperature of 550°C for 4 h. Bulk density (BD) was measured using a 1 L graduated cylinder filled with a known sample mass and tapped manually for 60 s to ensure the absence of large void spaces before measuring the final volume occupied by the sample mass. To determine the water holding capacity (WHC), the sample was firstly placed in a plastic cup and then oven-dried at 40°C overnight; after that the sample was brought to saturation with water and kept for 8 h, afterward the cup was covered at the top with parafilm and then perforated at the bottom to allow excess water to drain for 24 h.

Total organic C (TOC) and N (TN) were determined on a finely ground aliquot (particle size  $< 0.5$  mm) using a CN elemental analyser



LECO CN628 (LECO Corporation). Other elements were measured by TXRF spectrometry using a S2 Picofox Spectrometer (Bruker Nano GmbH). The instrument was equipped with a Mo microfocus tube (30 W, 50 kV, 600  $\mu$ A), a multilayer monochromator, and an XFlash<sup>®</sup> silicon drift detector with a 30 mm<sup>2</sup> active area. The energy resolution, measured at the K $\alpha$  of Mn, was 150 eV (10 kcps).

For TXRF analysis, dried samples were finely powdered through a vibratory ball mill (model MM200, Retsch) for 5 min. Then, an aliquot of 10 mg of each sample was suspended in 3 mL of Triton X-100 (1:100, v/v in double-distilled water) using 10-mL plastic tubes and left for 10 min in an ultrasonic bath. After that, 10 mL of a 1000 mg L<sup>-1</sup> Yttrium standard (Sigma-Aldrich) were added to each suspension as internal standard. Finally, after vortexing for 30 s, 10  $\mu$ L of suspension were pipetted onto siliconized quartz carriers and left until complete dryness on a heating plate (50°C) under laminar flow hood. Samples were analysed for 1000 s, and TXRF spectra were elaborated using the software Spectra 7 (Bruker GmbH). All analyses were performed in triplicate.

### 2.2.2 | SEM-EDX analysis

The SEM technique was adopted to investigate surface morphology of the materials. A small aliquot of sample was fixed with an adhesive carbon tape, metallized with Au, and analysed with a high-resolution field emission scanning electron microscope VP FE-SEM SIGMA 300 (ZEISS) equipped with an EDX elemental analyser. By adopting 5-kV acceleration potential, backscattered electrons were detected, and SEM micrographs were obtained at 15,000 $\times$  magnifications.

### 2.2.3 | BET analysis

The specific surface area of the three materials was determined according to the multipoint BET methodology (Brunauer et al., 1938). This technique is based on the physisorption of an inert gas, such as N<sub>2</sub>, CO<sub>2</sub> and other gases, on the surface of the material. The theoretical model is expressed as the BET equation which allows to calculate the BET surface area of the material adopting relative pressure ( $P/P_0$ ) values ranging from 0.05 to 0.35 (multipoint) (Lowell et al., 2006).

The adsorption-desorption isotherm of N<sub>2</sub> on approximately 0.5 g of each material was determined at 77.3 K using a surface area analyser. Before the isotherm construction, the sample was out-gassed at 110°C for 4 h under N<sub>2</sub> flow to remove moisture and other impurities attached. Then, the samples were analysed with a 3Flex automated high-performance adsorption analyser (Micromeritics).

### 2.2.4 | FTIR-ATR spectroscopy

FTIR-ATR spectroscopy was used to investigate functional groups belonging to the chemical bulks of the three materials. A Perkin Elmer

Two Spectrophotometer equipped with a 2  $\times$  2 mm diamond crystal was employed. After fine pot milling 0.2 g of each dried sample, 2.5 mg of sub-samples have been spread over diamond surface and analysed (4000–400 cm<sup>-1</sup> range, 4 cm<sup>-1</sup> resolution, 32 scan, 2 cm s<sup>-1</sup> rate). Each recorded scan set was averaged, corrected against ambient air as background and treated with ATR correction correlative function. To highlight the emerging bands, the smoothing function of the workstation was not needed.

### 2.2.5 | Raman spectroscopy

Raman spectra were collected using a Horiba-Jobin Yvon LabRAM HR spectrometer with laser excitation wavelength of 532 nm under ambient conditions. Low laser power (<1 mW) was used to avoid heat-induced modification or degradation of the sample due to the focused laser light during the spectrum acquisition. The collection time was larger than 200 s. The excitation laser beam was focused through a 50 $\times$  optical microscope (1-mm spot size and 1-cm work distance). The spectral resolution was 1 cm<sup>-1</sup>.

## 3 | RESULTS AND DISCUSSION

### 3.1 | Basic characterization and elemental analysis

Results of proximate/ultimate analyses of the three materials are outlined in Table 1. All the three materials showed similar alkaline pH values, while EC values varied, being lower in CP and VC ( $\sim$  1 dS m<sup>-1</sup>) and higher in the DG sample ( $>$  2 dS m<sup>-1</sup>). Alkaline readings for DG were not unexpected, since it is known that volatile fatty acids degradation and ammonia production occurring during the AD process cause an increase in pH (Tambone et al., 2009). Likewise, the degradation of organic acids and the volatilization of ammonia during the composting process cause an increase in pH above 7.0, which characterizes most CPs (Raviv, 2013). It is also known that VCs vary in pH values depending on the parent biomass from which they are produced (Katheem et al., 2016). On the other side, EC values exceeding 2 dS m<sup>-1</sup>, which can represent a limiting factor for an organic amendment used as stand-alone substrate (Raviv, 2013), cannot be considered potentially harmful to plant growth and crop productivity when diluted in the bulk soil mass. The ash content and BD followed the same trend, being similar for DG and CP and higher for VC. The WHC of the three materials did not appear to be significantly different. Comparing the TOC values of DG, CP and VC, they seemed quite similar, while the highest TN contents were found in DG and VC.

Overall, the results obtained were in the range of those reported in the literature for the corresponding type of material. In particular, the C content of DG ( $\sim$ 45%) was slightly higher than that averagely found in other DGs (Cesaro, 2021). Hung et al. (2017) analysed the composition of a swine manure DG and reported a C content of 37%, while Mukherjee et al. (2016) found 40% C content for a DG

**TABLE 1** Major properties of the materials tested in the present study.

Parameter	DG	CP	VC			
	mean	SD	mean	SD	mean	SD
Moisture (%) <sup>a</sup>	12.19	1.13	17.16	0.42	14.12	0.62
pH <sup>b</sup>	7.94	0.01	7.89	0.06	7.85	0.12
EC <sup>b</sup> (dS m <sup>-1</sup> at 25°C)	2.12	0.03	1.17	0.01	0.99	0.01
Ash (%) <sup>c</sup>	12.07	0.28	10.31	0.01	16.56	0.89
BD (g L <sup>-1</sup> )	220		210		270	
WHC (%)	41.8	1.9	41.6	0.4	40.0	0.8
TOC (g kg <sup>-1</sup> ) <sup>c</sup>	447.0	2.5	452.4	4.4	425.2	7.4
TN (g kg <sup>-1</sup> ) <sup>c</sup>	24.5	0.4	19.7	0.4	24.8	0.4
C/N	18.3	0.4	23.0	0.4	17.1	0.4

Note: ACP and VC are DG derivatives after 2-month composting or vermicomposting, respectively. SD, standard deviation ( $n = 3$ ).

Abbreviations: CP, compost; DG, solid digestate; VC, vermicompost.

<sup>a</sup>Air-dry basis.

<sup>b</sup>Material: distilled water 1:10 (w/v).

<sup>c</sup>Dry basis; BD: bulk density; WHC  $[(w_{\text{material}}/V_{\text{water}}) \times 100]$ : water holding capacity; TOC: total organic carbon; TN: total nitrogen.

produced from maize silage (60%) and animal residues. The relatively high organic C (~42%–45%) sequestered in all three materials contributes to reduce greenhouse gas emissions into the atmosphere and consequently to mitigate climate change. This property (high C%) is the main reason why these materials can behave as effective bio-sorbents of organic and inorganic contaminants. The N content of the materials varied from 2% to 2.5% (on dry matter basis) and was close to the average values reported in the literature for these materials (Table 1). N is a very important element for the chemical fertility of soil and is one of the main plant nutrients. A much higher TN content than that found in the present study was reported by Mukherjee et al. (2016) for a mixed feedstock DG and by Hung et al. (2017) for a swine manure DG. Obviously, DGs produced from animal waste only are richer in N than those obtained from plant residues or mixed waste. In a very recent study conducted by Kebalo et al. (2024), the authors reported organic C contents of 9% and 15% for a food waste DG and a green waste CP, respectively, while TN contents of DG and CP were, in the order, 0.72% and 0.75%.

The low C/N ratio calculated for all three materials depended on the type of biomass used in the AD process, which mainly originated from livestock waste, and indicates that, when these materials are applied to the soil, they can validly contribute to its fertility without altering the biotic equilibria already existing in the soil.

### 3.2 | TXRF spectroscopy

EDX fluorescence spectrometry is often applied for the analysis of the elemental composition of organic matrices (McWhirth et al., 2013;

López-Núñez, 2022; Taskin et al., 2019; Towett et al., 2015), providing the simultaneous and reliable quantification of several both nonmetal, such as P, S and Cl, and metal elements, e.g., K, Ca, Mn, Fe, Cu, Ni, Ti, including potentially toxic elements, such as Cr, Zn, Pb and so on. Moreover, among ED-XRF techniques, TXRF spectrometry requires limited sample quantity and has very high sensitivity; as such, this technique is valid for assessing the content of plant nutrients, as well as the possible load of pollutant elements of organic matrices intended for soil amendment, such as those considered in the present study.

Table 2 reports the concentrations of the elements detected in DG, CP and VC by TXRF analysis. As expected, macronutrients such as P, S, K and Ca were the most concentrated elements measured in all three materials. Calcium was the most abundant element (~1 wt% in DG e CP, and 1.7 wt% in VC); high Ca content may be related to its precipitation as carbonate and/or phosphate during the digestion process (Hung et al., 2017). Comparing the elemental composition of the three materials, it was found that the concentration values of the above-mentioned macronutrients were not statistically different ( $p < 0.05$ ,  $t$ -test) between DG and CP, while P and S contents were significantly higher in VC. Considering that VC also had the highest content of N (Table 1) and of important micronutrients (see Mn, Fe, Zn and Cu in Table 2), among the materials tested, VC appeared to be

**TABLE 2** Element content from TXRF analysis of the materials tested in the present study.

Element	DG		CP		VC	
	mean (mg kg <sup>-1</sup> )	SD	mean (mg kg <sup>-1</sup> )	SD	mean (mg kg <sup>-1</sup> )	SD
P	6317.8	454.9	7180.2	707.6	7692.7	137.8
S	2305.5	254.8	1940.4	125.1	2827.3	248.5
Cl	2121.0	358.2	2035.4	139.3	2308.7	56.9
K	8868.7	515.9	7765.2	861.0	4878.9	247.1
Ca	10505.6	102.5	10802.2	936.2	17508.3	316.7
Ti	101.7	7.4	55.5	0.9	101.2	3.1
Mn	114.3	8.7	96.0	1.8	139.8	2.0
Fe	1270.3	88.4	1172.5	56.1	2990.7	28.7
Ni	1.7	0.2	1.4	0.0	2.2	0.2
Cu	17.0	1.5	15.8	1.4	22.2	1.4
Zn	108.7	6.5	101.9	10.8	139.2	4.2
Br	7.6	0.2	5.6	0.1	7.6	0.4
Rb	9.5	0.5	9.1	0.3	8.7	0.8
Sr	35.8	0.8	38.7	2.9	61.2	2.4
Ba	< LOQ		20.5	0.7	23.4	7.4
Pb	3.2	0.4	3.1	0.5	4.8	0.5

Note: CP and VC are DG derivatives after 2-month composting or vermicomposting, respectively. Values are on dry basis. SD, standard deviation ( $n = 3$ ).

Abbreviations: CP, compost; DG, solid digestate; LOQ, limit of quantification; VC, vermicompost.





the most suitable to improve soil chemical fertility and stimulate plant growth. It is also true that, in the case of VC, a significant reduction (~40%) of K and a marked increase (~70%) of Ca were observed, with respect to DG and CP. The increased Ca content and the higher contents of microelements such as Fe and Zn found in VC, compared to the DG from which it derived, can be related to the higher ash content of this material (Table 1). On the other hand, the lower K content observed in VC, compared to DG, might be due to the loss of this mobile element in the percolating water during vermicomposting and/or to the biological activity of earthworms.

The contents of Cu, Zn, Ni and Pb in DG and, consequently, in the DG-derivatives were all below the limits fixed by the EU (EU Regulation, 2019) for organic fertilizers and are comparable with the concentration of heavy metals (HM) reported in the scientific literature for the corresponding materials (Albuquerque et al., 2012; Quadar et al., 2022; Zhao et al., 2024). In a recent interesting review, Kovačić et al. (2022) reported the concentration of HM found in a number of DGs obtained by processing different biomasses, including cattle manure and pig slurry. In that study, Pb ranged from 0.02 to 126 mg kg<sup>-1</sup>, Cu from 1.4 to 681.0 mg kg<sup>-1</sup>, Zn from 0.81 to 4019 mg kg<sup>-1</sup>, Fe from 371 to 29837 mg kg<sup>-1</sup>, Mn from 31.5 to 96.5 mg kg<sup>-1</sup>. Such wide variability of the HM content is reasonably due to the nature and the quality of the ingestate used. This wide variability suggests the importance of quantifying HM in these matrices commonly added to the soil, since high HM contents would lead to inevitable contamination of soil and of agricultural products. In the latter case, post-AD treatments, e.g., composting, should be considered to improve the quality of DG (Albuquerque et al., 2012).

### 3.3 | SEM-EDX analysis

The micromorphological properties of DG, CP and VC, such as surface features and distribution and allocation of pores, were studied using the SEM technique, while EDX elemental analysis allowed to explore the type and distribution of elements on the material surface. SEM images were obtained at 1500× magnifications (Figure 1).

All three materials clearly revealed a heterogeneous surface due both to the heterogeneity of the starting biomass and to the biological conversion they underwent. Numerous irregularly shaped ridges, channels and cavities were observed in the SEM images along with microparticles mostly of few μm (Figure 1). Furthermore, all three samples, especially DG, showed a low porosity. It is known that a large surface area, which is typical of highly porous matrices, is of crucial importance for the retention of organic compounds and metals. The typical porosity observed in other bioenergy byproduct, such as biochar (Loffredo et al., 2022), was not evident in the materials considered here that were directly or indirectly originated mostly from animal waste at a temperature relatively low. The scarce porosity of DG could be attributed to a pore filling by decomposition products remaining on the DG surface at the end of the AD process. Hung et al. (2017) studied a swine manure DG and observed a smooth and compact surface of the material, which is typical of DG

obtained from animal waste. SEM images of a VC obtained from palm oil mill effluent showed a fine texture originated from the fragmentation and ingestion of the raw biomass in the earthworm digestive system (Lim & Wu, 2015). The overall morphological features of DG, CP and VC, as revealed by SEM suggest a more suitable use of the materials for soil improvement purposes rather than for water or sediment decontamination.

The EDX spectra showed the presence on the surface of elements such as Ca, Mg, Na, K, Fe, P and so on that are typical of materials originated from mixed biomass (Figure 1).

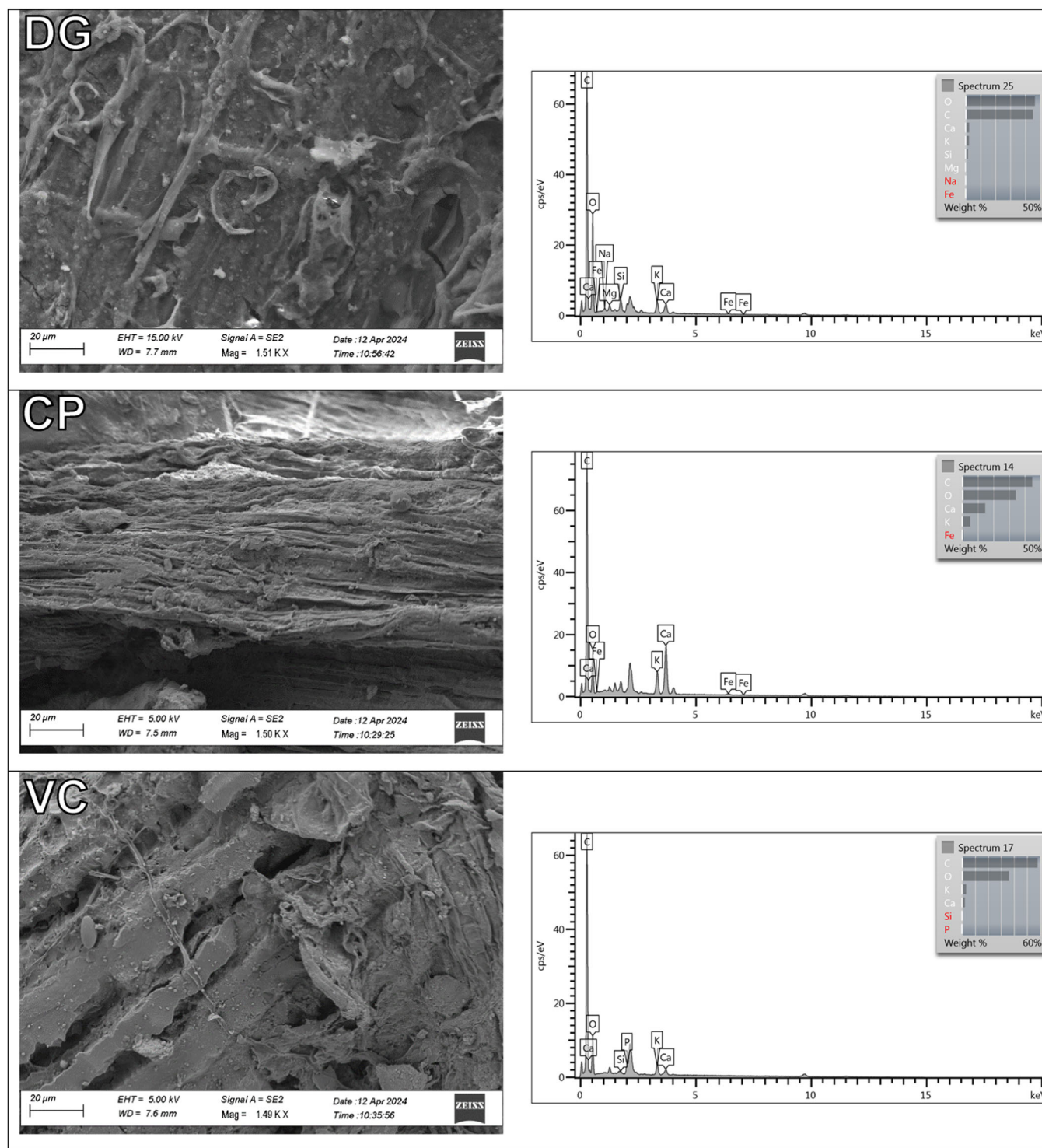
### 3.4 | BET analysis

The BET technique is based on the physical adsorption of an inert gas, such as N<sub>2</sub>, Ar or Kr, on the surface of an adsorbent material; the first layer of adsorbed molecules acts as a substrate for the adsorption of further molecules. The theoretical model on which this determination is based was first developed by Brunauer et al. (1938). This model allows to calculate the BET surface area of a material, in a range of relative pressure ( $P/P_0$ ) values of the gas ranging from 0.05 to 0.30 (multipoint) (Barrett et al., 1951; Lowell et al., 2006).

As expected, none of the materials showed values of the specific surface area comparable to other bioenergy byproduct, such as biochar, as they were mainly derived from animal waste converted through an anaerobic biological decomposition (DG), or anaerobic/aerobic decomposition (CP and VC). Results of BET analysis of the materials are shown in Table 3. The DG sample showed the lowest specific surface area (0.97 m<sup>2</sup> g<sup>-1</sup>) followed by CP (1.02 m<sup>2</sup> g<sup>-1</sup>) and VC (1.64 m<sup>2</sup> g<sup>-1</sup>). Based on these values, the VC sample proved to be a better adsorbent than the other two materials; this property is very important for the reduction of the bioavailability of contaminants present in soil pore water and consequently of their entry into agricultural plants. The BET data observed can be reasonably attributed to very small particles originated from the biomass fragmentation occurring during the composting stages or following the ingestion of biomass in the earthworm digestive system (He et al., 2017; Wu et al., 2014).

The BET data recorded for all three materials were indicative of low porosity, which in turn suggests a modest adsorption capacity. However, considering that the main destination of these materials is soil amendment, the BET surface area measured still guarantees to improve the overall adsorption capacity of the soil. This aspect is particularly relevant in contaminated soils because it limits the quantity of pollutants that are absorbed by plants and enter the food chain, and/or limits the load of pollutants transferred to surface and deep water bodies.

The BET data of DG, CP and VC referred in the scientific literature are very limited. In a recent study on the adsorption/desorption of some agrochemicals on an olive pomace DG, Loffredo et al. (2024) found a BET surface area of the DG of approximately 2 m<sup>2</sup> g<sup>-1</sup>, i.e., a double value compared to that found for the DG under study. A BET area of approximately 3.5 m<sup>2</sup> g<sup>-1</sup> was reported by Lim and Wu (2015) for a palm oil mill effluent VC.



**FIGURE 1** Scanning electron micrographs at magnifications of 1500× and energy-dispersive X-ray (EDX) spectra of the materials. Images were taken with secondary electrons. CP, compost; DG, solid digestate; VC, vermicompost.

### 3.5 | FTIR-ATR spectroscopy

The recorded FTIR-ATR spectra of the three materials are depicted in Figure 2. The FTIR-ATR spectra of DG, CP and VC featured several common IR bands, which were possibly due to their common origin (Figure 2). Since CP and VC were both produced from DG after an

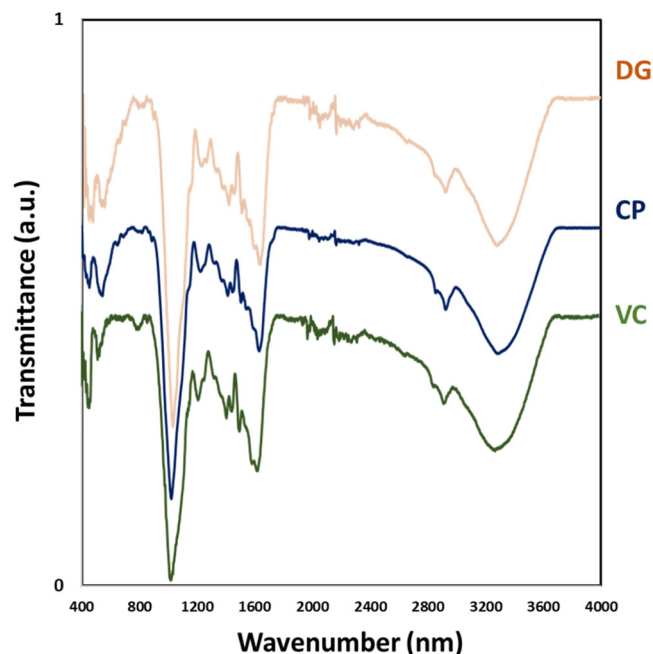
aerobic treatment, the IR analysis somehow allowed to recognize the state of chemical maturity of the intermediate metabolites via investigating the functional groups (Grigatti et al., 2004). The main transmittance bands, and common to all the three samples, were related to -OH stretching group, broadened in H-bonded mode, set around  $3350\text{--}3250\text{ cm}^{-1}$ . This is usually referred to waterish moiety,



Parameter	DG			CP			VC		
	mean	SD	R <sup>2</sup>	mean	SD	R <sup>2</sup>	mean	SD	R <sup>2</sup>
BET specific surface area (m <sup>2</sup> g <sup>-1</sup> )	0.97	0.01	0.9996	1.02	0.02	0.9996	1.64	0.01	0.9999

Note: CP and VC are DG derivatives after 2-month composting or vermicomposting, respectively. SD, standard deviation ( $n = 3$ ).

Abbreviations: CP, compost; DG, solid digestate; VC, vermicompost.



**FIGURE 2** FTIR-ATR spectra of digestate (DG), compost (CP) and vermicompost (VC) samples. FTIR-ATR, attenuated total reflectance-Fourier transform infrared.

together with hydroxylated matrices including pre-oxidative catechol and polysaccharides. Other common peaks ranged from 2930 to 2830  $\text{cm}^{-1}$ , belonging to asymmetric/symmetric C-H stretching of aliphatic moieties. All samples again exhibited the main transmittance IR-active moiety in the region 1080–1030  $\text{cm}^{-1}$ , often considered as a reference peak for intensity, and assigned to C–O stretching of polysaccharides or polysaccharide-like substances, and usually associated and intensified by Si–O stretching of silicate impurities, lapped with clay minerals possibly complexed with humic acids (Senesi et al., 2003; Vona et al., 2016). Specifically, the spectra of DG, CP and VC samples showed peaks at 1635–36  $\text{cm}^{-1}$  commonly assigned to C=O stretching of amide groups (amide I band) and bonded conjugated ketones, or C=O in COO<sup>-</sup> symmetric stretching of carboxylic acids. Moreover, a possible contribution in this region was given by quinones in lignin-like molecular bulk, in which aromatic C=C  $\text{sp}^2\text{-sp}^2$  are usually found. Once compared to a reference 1030  $\text{cm}^{-1}$  peak (polysaccharides bulk and clay minerals), this set of signals slightly increased in VC, likely due to a metabolic maturation process (Carballo et al., 2008; Fuentes et al., 2007). The aerobic treatments

**TABLE 3** Specific surface area from BET analysis of the materials tested in the present study.

also revealed an increase in the intensity of aromatic overtones ( $\sim 1700\text{--}2400\text{ cm}^{-1}$ ), in lignin-like bulks, which contain syringyl and guaiacyl alcohol functionalities set from 1150 to 1220  $\text{cm}^{-1}$ , passing from low-to-medium and strong intensity, respectively, in CP, VC and DG (Figure 2). The related digitalization lines have been reported in Section 2.

### 3.6 | Raman spectroscopy

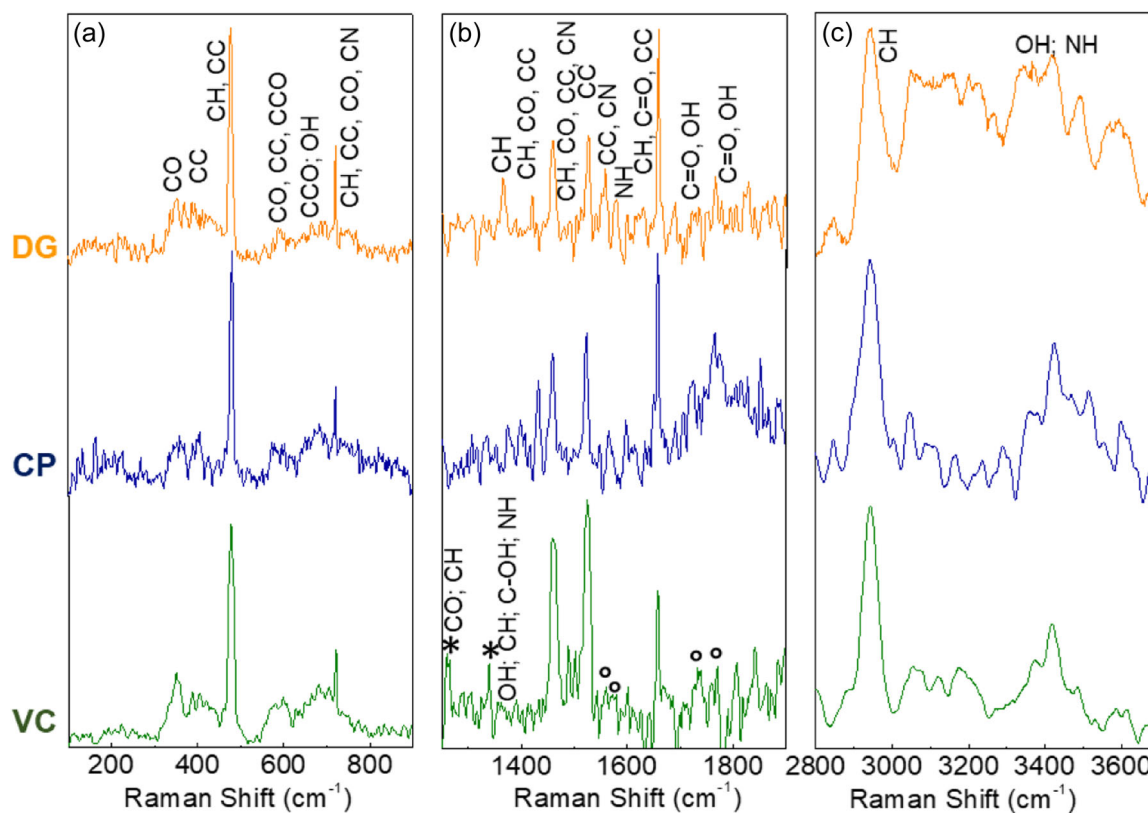
Raman spectra of DG, CP and VC were acquired in three different ranges: 100–900, 1250–1900 and 2800–3700  $\text{cm}^{-1}$  (Figure 3). In the range 100–900  $\text{cm}^{-1}$  (Figure 3a), Raman spectra showed two main bands, at  $\sim 480\text{ cm}^{-1}$  due to CH and CC bonds and at  $\sim 720\text{ cm}^{-1}$  due to CH, CC, CO and CN bonds (Miranda et al., 2014). Two broad bands at 360 and 410  $\text{cm}^{-1}$  due to, respectively, CO and CC bonds were also present, whose intensity was similar in DG and CP samples and different in VC where the band at 360  $\text{cm}^{-1}$  was more intense than that at 410  $\text{cm}^{-1}$ .

In the range 1250–1900  $\text{cm}^{-1}$  (Figure 3b), Raman spectra were dominated by four main bands at:  $\sim 1460\text{ cm}^{-1}$  attributed to CH, CO CC and CN bonds,  $\sim 1520\text{ cm}^{-1}$  due to CC bonds,  $\sim 1660\text{ cm}^{-1}$  attributed to CH, C=O and CC bonds and  $\sim 1770\text{ cm}^{-1}$  due to C=O and OH bonds. Less intense bands were also visible at  $\sim 1360\text{ cm}^{-1}$  due to CH, 1420  $\text{cm}^{-1}$  attributed to CH, CO and CC bonds,  $\sim 1570\text{ cm}^{-1}$  ascribed to CC and CN bonds,  $\sim 1600\text{ cm}^{-1}$  due to NH bond and  $\sim 1720\text{ cm}^{-1}$  due to C=O and OH bonds (Espina et al., 2022). Comparing the spectra of the three materials, DG and CP showed bands of similar intensity, while the Raman spectrum of VC resulted quite different. Specifically, in VC spectrum, bands at 1460 and 1530  $\text{cm}^{-1}$  were more intense than the band at 1660  $\text{cm}^{-1}$  due to C=O bond, signals in the range 1720–1770  $\text{cm}^{-1}$  and CN and NH bands in the range 1560–1580  $\text{cm}^{-1}$  were less intense (indicated by ° in Figure 3), and two additional evident bands at  $\sim 1260$  and 1338  $\text{cm}^{-1}$ , due to CO, OH and C-OH bonds (indicated by \* in Figure 3), appeared.

In the range 2800–3700  $\text{cm}^{-1}$  (Figure 3c), Raman spectra showed two main broad bands at  $\sim 2900\text{ cm}^{-1}$  due to CH bonds and  $\sim 3400\text{ cm}^{-1}$  due to OH and NH bonds (Kostova et al., 2007). These bands were of comparable intensity in DG and CP, whereas in VC, the intensity of CH bonds was higher than that of OH/NH one.

Based on Raman spectra, all three materials showed aromatic and unsaturated nature, although VC presented a lower number of NH bonds and more intense CO and OH bonds which is indicative of a more intense oxidation process.





**FIGURE 3** Raman spectra of digestate (DG), compost (CP) and vermicompost (VC) samples in in three different energy ranges: 100–900  $\text{cm}^{-1}$  (a), 1250–1900  $\text{cm}^{-1}$  (b) and 2800–3700  $\text{cm}^{-1}$  (c). In VC spectrum in range b, asterisks indicate two additional bands while empty circles refer to absent bands, compared to the spectra of the other materials.

## 4 | CONCLUSIONS

Soil amendments such as DG, CP and VC are commonly characterized mainly by basic conventional techniques aiming to evaluate their contents of organic matter and plant nutrients, and to ascertain that potentially toxic elements are below the limits fixed by the national Regulation for these materials. Differently, analytical techniques suitable to study their potential as bioadsorbents of organic and inorganic soil components, such as pollutants, are often overlooked or not explored at all. The sorption potential of a soil amendment is very important because it is strictly related to the entry of pollutants in plant products and/or their transfer into natural water bodies. In addition to compositional aspects, the adsorption capacity of a material depends largely on its micromorphology, surface area, and on the abundance and type of reactive surface functional groups. The study of multiple characteristics of a soil amendment allows to predict its overall role in the soil both from the perspective of agricultural productivity and from that of agroecosystem security. Therefore, to fully characterize these matrices and to understand the structural changes possibly occurred during the bioconversion of DG into its aerobic derivatives, CP and VC, we adopted a multianalytical approach which included analytical techniques rarely or never used together for these materials. The characterization study indicated that, even in the case of a common starting biomass, the production

technology adopted can determine relevant differences in the compositional and structural properties of the processed materials. Comparatively, DG, CP and VC presented similar alkaline nature, TOC content and WHC, while EC significantly decreased after the aerobic treatment of DG. As revealed by TXRF and EDX analysis, a variety of elements were present in each material whose quantitative composition appeared different among the three types of materials, being the VC sample the richest in P, S, Ca and micronutrients such as Mn, Fe, Cu and Zn. SEM analysis and BET surface areas indicated a noteworthy potential of all three materials, especially VC, to behave as biosorbents of organic compounds, including pollutants and metals. Therefore, when used as soil amendment, their contribution to the overall adsorption capacity of the soil is expected. FTIR-ATR and Raman analysis evidenced similar spectroscopical features of DG, CP and VC, reasonably due to their common origin. The presence of numerous aliphatic and aromatic functional groups in all three materials assures stable interactions with polar and hydrophobic solutes. The evaluation of the IR signals also somehow allowed to recognize the state of chemical maturity of the material, i.e., an increase in the intensity of aromatic groups and oxidized moieties passing from DG to VC. All these findings suggest potential benefits from DG, CP and VC for soil fertility improvement and as bioadsorbents of organic and inorganic contaminants. Finally, based on the compositional, morphological and functional characteristics studied, the VC sample



appeared to be the best candidate for the improvement of soil chemical fertility and for the immobilization of soil pollutants, suggesting vermicomposting as a suitable treatment for DG valorization.

## AUTHOR CONTRIBUTIONS

**Elisabetta Loffredo:** Conceptualization; methodology; supervision; major contribution to the writing of the manuscript. **Danilo Vona:** Material preparation; investigation; data analysis; writing. **Carlo Porfido:** Investigation; data analysis; writing. **Maria Michela Giangregorio:** Investigation, data analysis; writing. **Antonio Gelsomino:** Investigation; data analysis; writing.

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## CONFLICT OF INTEREST STATEMENT

The authors declare no conflicts of interest.

## DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

## ETHICS STATEMENT

The manuscript has been approved by all authors who gave consent to publish it. The authors confirm that they have adhered to the ethical policies of the journal.

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