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Safety Evaluation of Soil Substitutes Produced Based on Organic and Casting Waste

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ABSTRACT

Millions of tons of casting waste are generated annually worldwide, which should be subjected to recycling, as per the principles of circular economy. Spent foundry sands can be used for producing soil substitutes, but the process should yield products with guaranteed biological safety. The goal of this work was to conduct a safety evaluation of soil substitutes produced based on casting and organic waste. Toxicity tests were performed for this purpose, based on measurements involving the germination efficiency and the effect of the studied compositions on the biomass, sprout and root growth of *Sinapis alba.* In addition, an analysis of the content of chlorophyll A and B and of carotenoids was carried out, as well as a measurement of the lipid peroxidation level (content of malondialdehyde – MDA) to assess the potential oxidative stress in the tested plants. The compositions for soil formation prepared using casting waste as a mineral fraction and organic waste (compost, green waste, biogas plant digestate) had a stimulating effect on the rhizospheric and epigeal part growth of *Sinapis alba*. The germination efficiency in the prepared soil substitutes exhibited no significant difference from the germination efficiency in the control sample. However, the presence of oxidative stress (increased carotenoid and MDA contents) was found in the substitute containing green waste, which could be the result of water deficiency in the plants growing in this substrate. The complex testing of the compositions prepared based on casting waste (spent foundry sand) proved the validity of using such products as soil substitutes.

Keywords: waste management, circular economy, soil substitutes.

INTRODUCTION

Over 100 million tons of casting waste are generated annually worldwide (Díaz Pace et al. 2017; Modern Casting 2017), with about 18 millions in Europe alone (Layman's report 2018). Casting waste includes primarily spent foundry sands (FS). According to the principles of sustainable development, waste should undergo recycling at its place of generation. However, not all spent foundry sands are appropriate for reuse in the foundries themselves. Nevertheless, they can find application in highway engineering or civil engineering, but also in agriculture and horticulture. In the latter case, as toxic substances can be found in foundry sands, the use of FS is recommended, with mineral binders. According to the EPA Report (2014), only FS originating from iron, steel and aluminium foundries can be used for agricultural purposes. Material originating from non-ferrous metal foundries can contain heavy metals in quantities exhibiting toxic effects on plants (Ji et al. 2001; Dungan et al. 2009; EPA 2014; Sorvari and Wahlstrom 2014; Díaz-Pace et al. 2017). FS with mineral binders can be used as soil substitute components due to their low content of mobile forms of heavy metals as well as the structure and gradation similar to soils (Lindsay and Logan 2005, Dayton et al. 2010), whereas organic binders found in FS are more likely to exhibit a negative impact on the environment and human health (EPA 2014). Among the 600 thousand tons of waste generated annually in Poland, 500 thousand tons constitute cast iron foundry waste (Monitor Polski 2016). Using FS for extra-industrial applications is popular in the USA. Agricultural and horticultural purposes utilise 1.5 times more FS than highway engineering (except asphalt production) (EPA 2014). FS are also used for extra-industrial applications in Argentina, the RSA and Brazil. In Europe, this direction of waste repurposing is not yet common.

The soil is the living environment of numerous organisms, including plants. Apart from that, it stores nutrients for plants and is capable of water retention. Unlike the parent rock from which they originate, soils support life and are rich in organic matter formed from the remnants of plants, animals and microorganisms. Such matter accumulates primarily in the topsoil, determining its fertility and capability for agriculture (Pietr 2018). The organic matter content, and therefore the soil fertility, is influenced by the type and gradation of the soil (Šimanský et al. 2019). Sandy soils have lower organic matter content than soils rich in the clay fraction or amorphous minerals. The presence of organic matter is directly tied to the chemical properties of soils. It makes it possible to increase a soil's adsorptive and buffer capacity, and also constitutes a source of nutrients for plants. It can also frequently serve as a chelating agent for multivalent cations. One of the most important chemical properties of a soil is its pH reaction. At a low soil reaction (sour soils), the bioavailability of macronutrients (Ca, Mg, K, P, N and S) is low, while micronutrients (Zn, Mn, Fe) are available in large quantities that can be toxic to plants. Acidic pH also facilitates the bioavailability of heavy metals, whose toxicity is commonly known (Gjorgieva 2018; Chmielewski et al. 2020; Fu and Xi 2020, Borgulat et al. 2021). Due to their low nutrient content, green sands should not be applied independently as artificial soil (Dungan et al. 2009, Bożym 2018). The EPA Report (2014) also indicates that FS utilised as soil substitutes for horticultural purposes should contain an organic addition, e.g. humus or compost.

The goal of this work was to conduct a safety evaluation of soil substitutes produced based on casting and organic waste. The toxicity testing of products prepared based on spent foundry sand (FS) can confirm the validity of applying these products as soil substitutes due to their low toxicity or its absence, or completely negate it.

MATERIAL AND METHODS

Characteristics of the tested products

All the tested products were obtained according to the preparation technology included in patent No. 233754 (Głodniok et. al, 2019). On the other hand, the formulas for the soil formation compositions comprising organic and inorganic waste are included in patent No. 444410 (Głodniok, Borgulat 2023). Three compositions were prepared as part of the tests. Each was based on spent foundry sands as the inorganic waste as well as one of three types of organic waste (compost, green waste or biogas plant digestate). The basic components of the moulding compound were a quartz sand matrix, a binding agent in the form of bentonite and water. The codes and types of the waste used are presented in Table 1.

The compositions used for the testing consisted of at least 25% inorganic waste (foundry sand) and 75% organic waste (respectively: compost, green waste or biogas plant digestate). The composition contents are presented in Table 2.

The soil substitutes were prepared in a counter-current mixer that made it possible to produce a homogeneous composition. Soil originating from post-industrial land (zinc works) was used as the control sample (CS). The physicochemical composition of the substrates, final soil formation compositions and the control sample (soil) is presented in Table 3.

The physicochemical composition determination was performed at an accredited laboratory, according to standards: PN-EN 15934:2013-02, PN-ISO 10694:2002; PN-ISO 15178:2004; PN-EN 16174:2012; PN-EN ISO 11885:2009; PN-EN ISO 10390:1997. The Cd, Cr, Cu, Mn, Ni, Pb, Zn contents were determined by means of inductively coupled plasma optical emission spectroscopy (ICP-OES), whereas the mercury content was determined by means of cold vapour atomic emission spectroscopy (CV AAS) and amalgamation. The heavy metal content determination was performed according to standard PN-EN 16171:2017-02 (R/rR) by ICP-MS. All the tested products comply with the standards indicated in the Polish Regulation of 1 September 2016 on the method of conducting land contamination assessments [Dz. U. 2016, item 1395]. The soil substitutes created are used for the remediation of industrial and fossilised sites.

Table 1. Waste used in artificial soil mixtures

Type of waste	Name (abbrev.)	Waste code	Type
Inorganic waste	Foundry sand (FS)	10 09 08	Casting cores and moulds according to the casting process, other than shown in 10 09 07
Organic waste	Compost (C)	19 05 03	Compost that does not meet the requirements (unsuitable for use)
	Green waste (GW)	20 02 01	Waste from gardens and parks (including cemeteries) - biodegradable waste
	Biogas digestate (BD)	19 06 06	Digested waste from the anaerobic decomposition of animal and plant waste

Table 2. The composition of artificial soil mixtures

Mixture	Abbreviations	FS	Organic waste		
			C	GW	BD
Foundry sand + compost	FS+C	x	x		
Foundry sand + green waste	FS+GW	x		x	
Foundry sand + biogas digestate	FS+BD	x			x

Table 3. Physicochemical composition and properties of used soil substrates, final soil formation composition and control soil

Parameter	FS	C	BD	FS+C	FS+BD	FS+GW	CS
pH H_2O	7.3	7.3	7.8	8.1	7.7	7.4	5.5
DW (%)	>99	26.61	22.7	99.44	97.18	96.56	ND
OM (% DW)	1.82	43.13	58.6	23.09	23.85	30.17	ND
N_{tot} (% DW)	<0.15	1.94	7.9	1.15	1.28	0.84	ND
P (% DW)	0.01	0.55	1.92	0.32	0.54	0.16	ND
S (% DW)	<0.03	0.39	ND	0.24	0.47	0.13	ND
C (% DW)	1.48	24.52	ND	14.47	15.7	19	ND
TOC (% DW)	1.48	24.33	ND	14.24	15.62	18.85	ND
Ca (% DW)	0.26	2.4	4.6	1.72	1.16	1.46	ND
Mg (% DW)	0.17	0.38	0.6	0.35	0.29	0.31	ND
K (% DW)	0.10	1.47	ND	0.82	0.24	0.83	ND
Zn (% DW)	0.02	0.15	393	0.13	0.03	0.04	577
Fe (% DW)	0.48	1.22	ND	2.82	1.39	1.39	ND
Cd (mg kg^{-1} DW)	<1	1	<1	3	<1	1	11.6
Cr (mg kg^{-1} DW)	4	32	18.8	73	27	57	4.02
Cu (mg kg^{-1} DW)	5	44	113	112	49	54	24.6
Hg (mg kg^{-1} DW)	0.02	0.1	0.73	0.82	0.15	0.23	<0.1
Mn (mg kg^{-1} DW)	72	326	ND	470	215	313	ND
Ni (mg kg^{-1} DW)	2	27	11.8	53	18	40	3.23
Pb (mg kg^{-1} DW)	8	35	8.2	309	27	60	436

Note: ND – no data, DW – dry weight, OM – organic matter, TOC – total organic carbon.

Ecotoxicological testing

As part of the basic ecotoxicological testing, germination efficiency was investigated as well as the impact of the tested compositions on sprout and root growth, based on standards: PN-EN ISO 11269-1:06 and PN-EN ISO 11269-2:06. The testing was performed on the seeds of *Sinapis alba*. 3 test samples and 1 control sample (4 test series, 4 repetitions) were prepared: control with soil originating from industrial land near a zinc works (CS), the soil formation composition with compost (FS+C), the soil formation composition with green waste (FS+GW), the soil formation composition with biogas plant digestate (FS+BD). The obtained results were averaged.

Germination efficiency

The germination efficiency was tested according to the guidelines in standard: PN-EN ISO 11269-2:2013-06, which were adapted to the purposes of this research. The test was performed in Petri dishes. The results were inspected on the 1st, 3rd, 5th and 7th day after test commencement. The impact of the studied compositions on the germination efficiency was expressed in percentages, as presented in the formula below (Eq. 1):

$$GE = \frac{S_n}{S_0} \times 100\% \quad (1)$$

where: *GE* – germination efficiency determined for the tested compositions (%)
S_0 – number of seeds in the sample (20 pcs)
S_n – number of germinated seeds in the tested samples (pcs).

Biomass growth, stimulation of the rhizospheric and epigeal part growth of higher plants

Pots with a volume of 250 ml were filled with the appropriate soil formation composition or control soil and seeded with *Sinapis alba* seeds at a uniform depth (0.5 cm). The seeds were covered with soil. The test was conducted for 28 days over a daily cycle: 16h/8h, at a temperature of 25°C under stable humidity conditions. The influence was determined relative to the control sample as % biomass growth and rhizospheric and epigeal part growth stimulation. The impact of the tested compositions on the biomass growth and the epigeal (IP) and rhizospheric (IK) part growth was estimated after 28 days of testing, per equation 2.

$$GP_{b,s,r} = -\left(\frac{CS_{b,s,r} - TS_{b,s,r}}{CS_{b,s,r}}\right) \times 100\% \quad (2)$$

where: *GP* – growth efficiency parameters: biomass (g), sprout (s) and roots (r) length; *CS* – plants growing on control soil (CS); *TS* – plants growing on the tested soil formation compositions (FS+C, FS+BD and FS+GW); *b* – biomass (g), *s* – sprout length (cm), *r* – roots lenght (cm).

Malondialdehyde (MDA) content determination in the leaves

The MDA content determination in the plant leaves was performed based on the methodology described by Hodges et al. (1999). The plant tissue samples were homogenised in a mortar with silica in an aqueous ethanol solution (8:2$_{v/v}$ of ethanol:water). Afterwards the mixture was centrifuged for 10 min (3000 g). Next, 1 ml of a solution containing 0.65% TBA and 20% TCA was added to 1 ml of the supernatant liquid. 1 ml of 20% TCA (without TBA) was added to the second test tube. Afterwards the samples were rapidly mixed and set aside at a temperature of 95°C for 20 minutes, after which they were cooled down and centrifuged (10 min, 3000 g). Next, the absorbance was determined at λ = 440, 532 and 600 nm. The MDA content was measured using the following formulas:

$$A = [(Abs\ 532_{+TBA}) - (Abs\ 600_{+TBA}) - (Abs\ 532_{-TBA}) - (Abs\ 600_{-TBA})]$$

$$B = [(Abs\ 440_{+TBA} - Abs\ 600_{+TBA}) \times 0.0571]$$

$$MDA = (A - B) \times 157000^{-1} \times 10^{6}\ (nmol\ ml^{-1})$$

Chlorophyll and carotenoid content determination in the leaves

The content of chlorophylls and carotenoids in the leaves of selected species was determined as an auxiliary parameter. This was accomplished using a modification of the methodology provided by Arnon (1949). 5 ml of DMSO were added to 50 mg of shredded leaves and heated in a water bath to 60°C. The samples were stored in the dark for 3h, after which the absorbance was measured at the following wavelengths: λ= 470, 646 and 665 nm. The photosynthetic pigment content was calculated using the formulas below and by conversion per gram of fresh mass:

$$Chlorophyll\ A = (Abs\ 663 \times 12.7) - (Abs\ 645 \times 2.69)\ (\mu g\ ml^{-1})$$

$$Chlorophyll\ B = (Abs\ 645 \times 22.9) - (Abs\ 645 \times 4.68)\ (\mu g\ ml^{-1})$$

$$Carotenoids = ((Abs\ 470 \times 1000) - (Chlorophyll\ A \times 2.14) - (Chlorophyll\ B \times 70.16))/220\ (\mu g\ ml^{-1})$$

Free malondialdehyde content determination

The level of MDA was determined by a colour reaction with 2-thiobarbituric acid (TBA) per a modification of the method provided by Shah et al. (2001). The material extraction was conducted in cooled mortars with an addition of 0.1% trichloroacetic acid (TCA). The homogenate was centrifuged, and afterwards 0.5% thiobarbituric acid in 20% TCA was added to the extract. The samples were heated at a temperature of 95°C for 30 min and centrifuged, after which the absorbance was measured at 532 nm. The result was corrected by discarding the value obtained at a wavelength of 600 nm as a result for non-specific reaction products with TBA.

RESULTS

Germination efficiency

By comparing the germination efficiency of *Sinapis alba* seeds growing in the soil substitutes based on foundry sands with the control group composed of seeds planted in industrial soil, it can be noted that the maximum germination efficiency was reached on the 7th day after planting.

The highest germination efficiency was found for the control sample (95%), followed by the substitutes containing compost (90%) and biogas plant digestate (90%) as the organic fraction. 75% of the seeds germinated in the composition containing green waste Figure 1.

Growth efficiency parameters

A stimulating effect on the growth of both the rhizospheric and epigeal parts of *Sinapis alba* was noted for all the tested soil substitutes, measured relative to the control group (Figure 2). The soil substitute containing biogas plant digestate as the organic fraction provided the greatest stimulation of the rhizospheric part of the plant (37%), followed by green waste (25%) and compost (18%).

In the case of the epigeal part, the highest growth relative to the control group was also characteristic of the plants growing in the substitute containing biogas plant digestate (89%), whereas the lowest was exhibited by the composition containing green waste (36%).

Plants growing in the substitute containing biogas plant digestate were characterised by the greatest biomass growth (80%) relative to the control group. Plants growing in the composition containing green waste were characterised by 13% less biomass than the *Sinapis alba* in control group growing on industrial soil (CS).

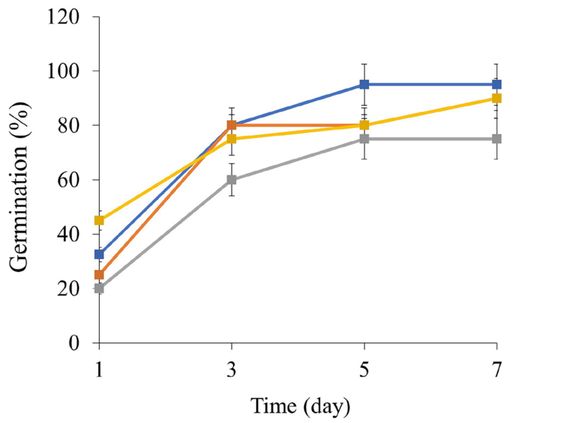

Figure 1. Germination efficiency of *Sinapis alba* seeds on various soil formation composition (mean±SD, n= 4)

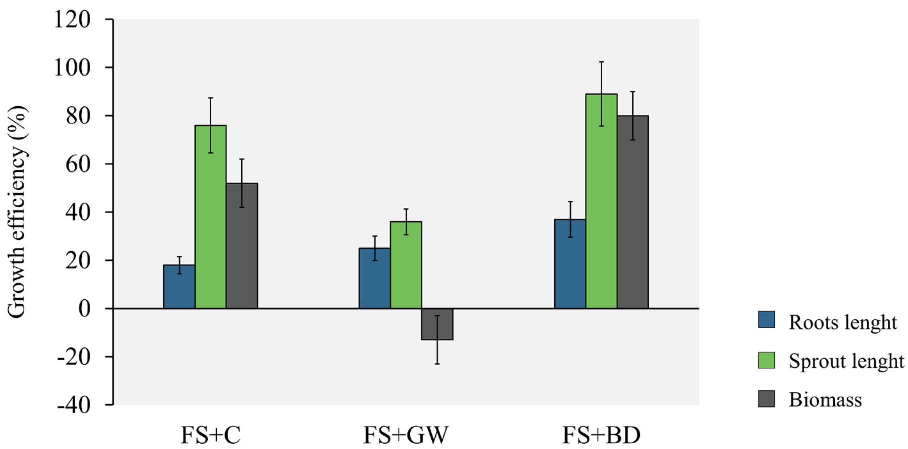

Figure 2. Selected growth efficiency parameters of *Sinapis alba* (mean±SD, n= 4). Positive values mean an increase and negative values mean a decrease in the value of a certain parameter compared to the control plants (CS)

Dry matter, photosynthetic pigment and free malondialdehyde content

Table 4 presents the differences in the dry matter, chlorophyll A and B and carotenoid contents, as well as the degree of lipid peroxidation (free malondialdehyde content) for *Sinapis alba* growing in the control soil and the 3 different soil substitutes. The greatest dry matter, much as with the biomass (Fig. 2), was characteristic of specimens growing in the substrate containing biogas plant digestate and foundry sand, whereas the lowest was found in the green waste and foundry sand composition.

In terms of the chlorophyll A and B, carotenoid and malondialdehyde contents, the plants growing in the soil substitute containing compost (FS+C) and biogas plant digestate (FS+BD) were similar to *Sinapis alba* growing in the control soil (CS). On the other hand, specimens from the soil substitute containing green waste exhibited less chlorophyll A and B but more carotenoids and malondialdehyde than the other tested plants.

Discussion

In the view of experts (Dungan and Dees 2009), based on research carried out on 43 types of spent foundry sands originating from iron, steel and aluminium foundries (green sands), the heavy metal content did not exceed the amount found naturally in soils. The waste they studied was also characterised by low waste leaching, which attested to its weak bioavailability for plants. Furthermore, the structure and gradation of the FS was similar to the soils (Dayton et al. 2010). Therefore, using them as a soil addition improves the physical properties of soil, particularly its permeability, and also decreases the mobility of heavy metals – including those found naturally in the soil (Lindsay and Logan 2005). According to the data presented in Table 3, the foundry sand intended for soil substitute production contained slight quantities of heavy metals, but it was poor in plant nutrients. For this reason, organic waste was used as an addition for increasing the nutrient content (Table 1), which resulted in a soil formation composition containing considerable quantities of organic matter as well as nitrogen, potassium and phosphorus (Table 3). The average nitrogen content in Polish soils in the case of mineral soils ranges within 0.02–0.35%, phosphorus is within 0.01–0.2%, and potassium is within 0.2–4%. The majority of these elements are present in organic compounds and are poorly available for plants. The prepared artificial soil compositions contained higher nitrogen and phosphorus (except FS+GW) contents as well as potassium compare to control post-industrial soil (Table 3).

To inspect the quality and safety of the prepared soil formation compositions, they were subjected to basic toxicity tests, and their effect on the biomass and physiology of test plants was evaluated by comparison with soil originating from an industrial area (zinc works). It was also inspected whether the prepared compositions generated any oxidative stress in the plants.

One of the biotests indicating the potential toxicity of the obtained substitutes towards plants is the measurement of germination efficiency. In the presented tests, no significant changes in germination efficiency were found between the control sample and the substitutes containing compost (FS+C) and digestate (FS+BD) (Figure 1). The lowest germination efficiency (75%) was noted for the composition containing green waste (FS+GW). Many researchers noted that soil formation compositions containing foundry sand as well as eluates from stored foundry waste had no effect on germination efficiency (Logan and Lindsay 2001, Dayton et al. 2010, Bożym 2020). These authors applied various proportions of individual composition ingredients, but without exceeding a 50% contribution of FS in the compositions.

Table 4. Dry matter, photosynthetic pigment and free malondialdehyde (MDA) contents for Sinapis alba growing in various soil substitutes (mean±SD, n= 4). Homogeneous groups are marked with the same letters (ANOVA, *post hoc*- LSD)

Soil	CS	FS+C	FS+GW	FS+BD
Dry matter (g)	81±7.6a	124±12.9b	65±5.8a	137±15.2b
Chlorophyll A (μg g^{-1} DM)	400±38.4ab	485±38.8b	360±41.1a	499±55.4b
Chlorophyll B (μg g^{-1} DM)	111±10.8a	140±14.7b	98±10.1a	139±12.6b
Carotenoids (μg g^{-1} DM)	18±1.6a	20±1.7a	27±2.4b	22±2.0a
MDA (nmol ml^{-1})	64±6.3ab	59±5.4a	78±9.0b	62±5.1a

The tests performed as part of this work indicate a stimulating effect of the applied compositions on the rhizospheric and epigeal parts as well as the biomass of *Sinapis alba* relative to the control group (Figure 2). The roots of *Sinapis alba* growing in the soil formation compositions based on foundry sand and organic waste were longer than the roots of the same plants growing in the control soil. In the case of the epigeal part, a stimulating effect was found in the case of *Sinapis alba* growing in a substrate containing compost (FS+C) and biogas plant digestate (FS+BD). Similarly in the case of biomass, plants growing in the composition containing compost (FS+C) were characterised by greater biomass than plants growing in the control soil (CS). The biomass of plants growing in the composition containing biogas plant digestate (FS+BD) was the greatest among all the samples tested as part of this work, whereas the lowest biomass was noted for *Sinapis alba* growing in the substitute containing green waste.

Although a stimulating effect on the rhizospheric and epigeal parts of *Sinapis alba* was found for all the obtained substitutes, further testing indicated a lower biomass growth for this plant, relative to the control group, when grown in the composition containing green waste (FS+GW). Given the above, it was decided to measure the content of chlorophyll A and B and carotenoids – pigments that determine the occurrence of photosynthesis and which thereby have a direct effect on biomass growth – as well as malondialdehyde, which is a prime indicator of oxidative stress occurring in plants.

The tests demonstrated that the level of chlorophyll A and B in the plants growing in compositions with compost (FS+C) and biogas plant digestate (FS+BD) was similar or higher to the control group, whereas the lowest values were obtained for plants growing in the soil substitute containing green waste. Numerous factors have an influence on the intensity of photosynthesis – which has a direct effect on the quantity of plant biomass. These include internal factors such as the amount of chlorophyll and the leaf structure, as well as external factors such as: the availability of water, CO_2, light, macro- and micronutrients. Considering that all the plants grew under the same temperature, light and CO_2 conditions and were provided nutrients, the reasons for the decreased biomass of plants growing in the substrate containing green waste (Figure 2) can be sought in the lower chlorophyll content. The decrease in chlorophyll content as a result of stimulated drought and heat stress was found in all the plants studied by Krzyżak et al. (2023). However, these were xerothermic plants, and thus exhibited no biomass decrease. The research by Khayatnezhad and Gholamin (2021) demonstrated that the chlorophyll content in plants susceptible to drought would decrease, whereas it would remain the same or increase in plants tolerant of drought. Furthermore, in the presented tests, the greatest level of carotenoids and MDA among all the tested samples was found in the plants growing in the substitute containing green waste (FS+GW). Carotenoids are pigments that determine the occurrence of photosynthesis, but they are also non-enzymatic antioxidants that protect chlorophylls during stress (Swapnil et al. 2021). Therefore, the high carotenoid and MDA content indicates the occurrence of oxidative stress in the plants. The MDA level is measured in response to many different stress factors: limited water supply (Rosalie et al. 2015), salinity (Rachoski et al. 2015) or heavy metal content in the soil (Juknys et al. 2012). Given the composition of the tested soil substitutes as well as their structure, it can be assumed that the substitute containing green waste did not provide adequate retentive properties. A high MDA content in the leaves in response to drought was also found in the studies by Krzyżak et al. (2023), where plants were tested for their physiological reactions to drought and the impact of high temperatures.

CONCLUSIONS

The soil formation compositions prepared based on foundry sand as the mineral fraction and organic waste (compost, green waste, biogas plant digestate) had a stimulating effect on the growth of *Sinapis alba*, which indicates the validity of using this type of waste as an important ingredient of soil formation compositions. This is significant from the perspective of circular economy, which assumes the maximum repurposing of generated waste. It should however be remembered that it is not just the chemical composition of the prepared soil substitutes that matters in terms of the plant growth, but also their structure, which needs to provide all the functions of soil, particularly its retentive capacity. Furthermore, since there is always a real risk of introducing pollutants found in the waste into the environment, control testing identifying the impact of the prepared products on the growth and condition of plants must be conducted for all of them.

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